## (11) EP 4 394 082 A1

#### (12)

## **EUROPEAN PATENT APPLICATION**

(43) Date of publication: 03.07.2024 Bulletin 2024/27

(21) Application number: 23214207.5

(22) Date of filing: 05.12.2023

(51) International Patent Classification (IPC):

C23C 24/08 (2006.01) C23C 26/00 (2006.01)

C23C 28/04 (2006.01)

(52) Cooperative Patent Classification (CPC):C23C 28/04; C23C 24/08; C23C 26/00;C23C 28/042; C23C 28/048

(84) Designated Contracting States:

AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC ME MK MT NL NO PL PT RO RS SE SI SK SM TR

Designated Extension States:

BA

**Designated Validation States:** 

KH MA MD TN

(30) Priority: 27.12.2022 US 202218146802

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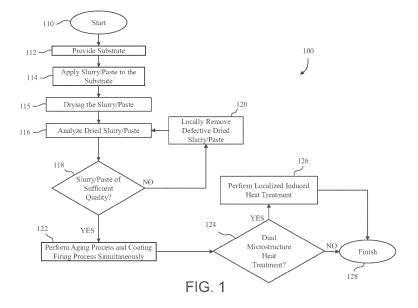
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# (54) SUPERALLOY COMPONENTS HAVING COATINGS THEREON AND METHODS FOR PRODUCING THE SAME

(57) Methods are provided for producing components. The methods include applying a slurry or paste to a surface of a substrate, wherein the substrate includes a nickel-based superalloy, drying the slurry or paste applied to the surface to define a coated substrate, and performing a heat treatment on the coated substrate to simultaneously densify and/or crystallize the dried slurry or paste to thereby form a coating that is bonded to the

surface and to age the substrate to achieve a specific hardness therein by controllably forming precipitates of an intermetallic phase within the substrate and/or controllably modifying a size distribution of the precipitates of the intermetallic phase within the substrate, wherein residual stress within the substrate are preserved during the heat treatment.



## TECHNICAL FIELD

**[0001]** The present invention generally relates to methods for producing components comprising superalloy substrates with coating systems thereon, and more particularly relates to methods that include combination heat treatments that simultaneously age the substrate and densify and/or crystallize the coating thereon.

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## **BACKGROUND**

[0002] Various processes are used during the production of components that include superalloys, such as certain components of turbine engines, to achieve a balance of various desired properties. For example, nickel-based superalloy components for turbine applications commonly undergo precipitation hardening, also known as age hardening or aging, to increase the yield strength of the superalloy. The precipitation hardening treatment involves heat treating the superalloy to form precipitates therein. If properly controlled, the formed precipitates will be configured to allow a hardening of the superalloy according to a desired hardness. Specifically, the formed precipitate particles may act as obstacles to dislocation movement and thereby strengthen the superalloy. Thereafter, a coating system may be applied to the aged superalloy to provide, for example, environmental protection (e.g., resistance to high temperatures, oxidation, and/or corrosion).

**[0003]** These processes may have various shortcomings. For example, the aging process can form an oxide scale on surfaces of the superalloy that typically must be removed (e.g., mechanically). In addition, formation of the coating system typically exposes the superalloy substrate to high temperatures for an extended period of time that may cause removal of residual stress therein, which in certain applications may be desirable.

**[0004]** Hence, there is a need for methods capable of producing superalloy components that are efficient and, optionally, preserve residual stress therein. Furthermore, other desirable features and characteristics of the present invention will become apparent from the subsequent detailed description and the appended claims, taken in conjunction with the accompanying drawings and the foregoing technical field and background.

## **BRIEF SUMMARY**

**[0005]** This summary is provided to describe select concepts in a simplified form that are further described in the Detailed Description. This summary is not intended to identify key or essential features of the claimed subject matter, nor is it intended to be used as an aid in determining the scope of the claimed subject matter.

**[0006]** A method is provided for producing a component. The method comprises applying a slurry or paste

to a surface of a substrate, wherein the substrate includes a nickel-based superalloy, drying the slurry or paste applied to the surface to define a coated substrate, and performing a heat treatment on the coated substrate to simultaneously densify and/or crystallize the dried slurry or paste to thereby form a coating that is bonded to the surface and to age the substrate to achieve a specific hardness therein by controllably forming precipitates of an intermetallic phase within the substrate and/or controllably modifying a size distribution of the precipitates of the intermetallic phase within the substrate, wherein residual stress within the substrate are preserved during the heat treatment.

[0007] A component is provided that comprises a substrate comprising a nickel-based superalloy having precipitates of an intermetallic phase, the substrate having a specific hardness therein, and a coating bonded to a surface of the substrate, the coating configured for resistance to hot corrosion caused by molten salts of sodium, magnesium, vanadium, and/or sulfur dioxide gas, wherein the coating is configured to react with the molten salts to increase a melting point of the molten salts thereby reducing deposition thereof on the component, wherein the substrate includes residual stress therein, and wherein the coating has an as-formed structure free of mechanically induced defects created during production of the component.

**[0008]** Furthermore, other desirable features and characteristics of the method and component will become apparent from the subsequent detailed description and the appended claims, taken in conjunction with the accompanying drawings and the preceding background.

### BRIEF DESCRIPTION OF THE DRAWINGS

**[0009]** The present invention will hereinafter be described in conjunction with the following drawing figures, wherein like numerals denote like elements, and wherein:

FIG. 1 is a flowchart of a method for producing a component in accordance with an embodiment;

FIG. 2 schematically represents cross-sectional views of nickel-based superalloy substrates with a coating thereon before and after heat treatments in accordance with an embodiment; and

FIG. 3 includes a component that includes a nickelbased superalloy substrate and a coating thereon and illustrates a method of performing a heat treatment on the component to produce a microstructure having a grain size gradient in accordance with an embodiment of the invention.

#### DETAILED DESCRIPTION

**[0010]** The following detailed description is merely exemplary in nature and is not intended to limit the invention

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or the application and uses of the invention. As used herein, the word "exemplary" means "serving as an example, instance, or illustration." Thus, any embodiment described herein as "exemplary" is not necessarily to be construed as preferred or advantageous over other embodiments. All of the embodiments described herein are exemplary embodiments provided to enable persons skilled in the art to make or use the invention and not to limit the scope of the invention which is defined by the claims. Furthermore, there is no intention to be bound by any expressed or implied theory presented in the preceding technical field, background, brief summary, or the following detailed description.

**[0011]** Methods disclosed herein that generally provide for producing components, such as gas turbine components, having a substrate with a protective coating system on a surface thereof. The methods provide for producing the components in a manner that is more efficient than certain prior art methods and that may preserve residual stress within the substrate imparted prior to forming the coating system.

[0012] The substrates may include or be made from any type of alloy. For aerospace applications, the substrates may be made from a superalloy material due to their mechanical strength and resistance to high temperatures. For convenience, the method is described herein in reference to substrates comprising a nickel-based superalloy. However, it should be understood that the substrates may include or be formed of various other types of superalloys (e.g., cobalt-based, nickel-iron-based) or alloys (e.g., stainless steel). Nonlimiting examples of nickel-based superalloys include Ni-Cr-Fe-based (e.g., Inconels), Ni-Mo-Fe-based (e.g., Hastelloy A and B), and Ni-Cr-Mo-Fe-based (e.g., Hastelloy C and Hastelloy X). The substrates may have any regular or irregular shape and size.

[0013] The coating system may include any number of layers each having different compositions, microstructures, and/or intended purposes. In various embodiments, one or more of the layers of the coating system may be configured to provide environmental protection to the underlying substrate, such as resistance to high temperatures and/or corrosion. In various embodiments, the coating system is configured to provide for resistance to hot corrosion caused by molten salts of sodium, magnesium, vanadium, and/or sulfur dioxide gas. In such embodiments, one or more layers of the coating system may be configured to react with the molten salts to increase a melting point of the molten salts thereby reducing deposition thereof on the component. In various embodiments, the coating system includes a glass material having different ratios of metal oxides such as, but not limited to, barium oxide, silicon oxide, strontium oxide, aluminum oxide, magnesium oxide, calcium oxide, cobalt oxide, and titanium oxide. The thickness of the coating system and/or individual layers thereof can vary. For example, the coating system may have a total thickness of between about 0.5 and 10 µm. Nonlimiting examples of coating

systems that may be applied to the substrate are disclosed in U.S. Patent No. 11,415,004 B2 to Mohajeri et al., the contents of which are incorporated herein in their entirety by reference.

[0014] Referring now to FIG. 1, a flow chart is provided illustrating an exemplary method 100 for producing a component. The method 100 may start at 110. At 112, a substrate is provided, such as a superalloy substrate. In various embodiments, the substrate may be near net shape, that is, substantially similar in size and shape to the intended final size and shape of the component being produced. Various processes may be used to produce the substrate, including but not limited to various forging, milling, machining, casting, and/or powder metallurgy processes. In certain embodiments, the substrate may include residual stress imparted by one or more of these production processes.

**[0015]** Various material processing processes may be performed on the substrate. For example, the method 100 may include performing a solution heat treatment on the substrate to dissolve precipitates in the substrate and to homogenize a microstructure of the substrate. The method 100 may include performing a stabilization heat treatment on the substrate to optimize a size and a morphology of precipitates therein, such as gamma prime phase precipitates, within the substrate.

[0016] After the substrate has been provided, the method 100 may include applying, at 114, a slurry or paste to the substrate that is configured to form a coating thereon. The slurry or paste may be formed by various processes. For example, the slurry or paste may be made by mixing a fine glass powder with a carrier fluid and, optionally, additional components (e.g., binder, dispersant, etc.). Nonlimiting examples of slurries and pastes, as well as application methods thereof, are disclosed in Mohajeri et al. The slurry or paste may be applied onto desired surfaces of the substrate by, for example, brush painting, doctor-blading, screen printing, or spray painting. Low temperature volatile components of the slurry or paste may be removed during a drying step at 115, for example, in an oven. If present, organic compounds of the slurry or paste may be burned-out by a binder burnout process.

**[0017]** At 116, the method 100 may include analyzing the coated substrate to determine whether the dried slurry or paste applied thereto is of sufficient quality to proceed. If the slurry or paste is insufficient (e.g., includes defects), an entirety or a portion of the slurry or paste may be locally removed to repair the slurry or paste at 120. Once a determination is made at 118 that the slurry or paste is of sufficient quality, the method 100 may include simultaneously performing an aging process and a coating firing process in a single heat treatment at 122, referred to herein as a combination heat treatment.

**[0018]** The combination heat treatment may have various parameters, including various temperatures and durations, depending on the materials and desired result. Notably, the parameters of the combination heat treat-

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ment must be sufficient to both age the substrate to a desired degree (e.g., to achieve a specific hardness therein by controllably forming and/or modifying a size distribution of precipitates therein) as well as densifying and/or crystallizing the dried slurry or paste to a sufficient degree (e.g., to form a coating bonded to the surface of the substrate). In addition, the parameters of the combination heat treatment may be configured to avoid elimination of residual stress in the substrate (e.g., sufficiently low temperature and/or short duration). The precipitates formed in the substrate may be precipitates of an intermetallic phases, such as but not limited to a secondary gamma prime phase. In various embodiments, the combination heat treatment may include heating the substate having the dried slurry or paste to a temperature within a range of between about 750 and 850 °C and maintaining such temperature for a duration of between 8 to 32 hours. The combination heat treatment may be performed in a vacuum (e.g., less than about 1330 Pa), an inert atmosphere (e.g., argon, helium, hydrogen), and/or atmosphere having a different partial pressure of oxygen with respect to air.

[0019] In various embodiments, the combination heat treatment may be performed by an induction heating process. The process may include generating a magnetic field around the substrate with an inductor connected to an electrical power source sufficient to induce eddy currents within the substrate and dissipate heat by Joule effect within the substrate. The electrical power source may be controlled while generating the magnetic field to provide the magnetic field for a duration of application of the magnetic field sufficient to directly induce the eddy currents within the substrate so as to produce a heat level therein to form and/or modify the size distribution of the precipitates. Exemplary but nonlimiting induction heating processes that may be adapted to perform the combination heat treatment are disclosed in U.S. Patent No. 11,136,634 to Bridier et al., the contents of which are incorporated herein in their entirety by reference.

**[0020]** In various embodiments, the method 100 may include performing the combination heat treatment in a manner, or performing a subsequent heat treatment, to produce a dual microstructure within the substrate. For example, the method 100 may include performing a localized induced heat treatment at 126 to controllably generate a coarse grain microstructure region within the component from a fine grain microstructure metallic component. In various embodiments, the component may be heat treated such that a core region thereof includes fine metallurgical grains configured to provide a desired fatigue resistance and a peripheral region thereof includes course metallurgical grains configured to provide a desired creep resistance. Exemplary but nonlimiting methods for producing such dual microstructures are disclosed in Bridier et al.

**[0021]** In various embodiments, the dual microstructure may be formed by generating a magnetic field around the coated substrate with an inductor connected to an

electrical power source sufficient to induce eddy currents within the coated substrate and dissipate heat by Joule effect within the coated substrate, and controlling the electrical power source, by a processor, while generating the magnetic field to provide the magnetic field for a duration of application of the magnetic field sufficient to directly induce the eddy currents within the coated substrate so as to produce a heat level within the coated substrate to grow the grains. In general, the heat level may be lower to produce the fine grains and greater to produce the coarse grains. The grain growth may be further controlled by applying the slurry or paste in a manner such that a thickness of the coating on the surface varies across the surface. For example, the coating may be thicker over areas of the surface having the coarse grains and thinner over areas of the surface having the fine grains.

**[0022]** Subsequent to producing the dual microstructure, or if such dual microstructure is not desired, the method 100 may finish at 128.

**[0023]** Referring now to FIG. 2, a portion of a component is represented during production thereof which illustrates changes that occur during the heat treatment described at 122 of the method 100 in FIG. 1. Initially, a substrate 210 is provided that is coated with a slurry or paste 230. During the heat treatment, the substrate 210 is aged to produce the substrate 220 and the slurry or paste 230 is converted into a coating 240. Importantly, an oxide layer is not formed on the substrate 220 as is common during aging processes performed on uncoated substrates.

[0024] Referring now to FIG. 3, an exemplary component 300 is represented that includes a substrate 310 having a coating 312 thereon. The substrate 310 includes a nickel-based superalloy having precipitates of a gamma prime phase therein and having a specific hardness. The coating 312 is bonded to exterior surfaces of the substrate 310 and is configured to provide resistance to hot corrosion caused by molten salts of sodium, magnesium, vanadium, and/or sulfur dioxide gas. In particular, the coating 312 is configured to react with the molten salts to increase a melting point of the molten salts thereby reducing deposition thereof on the component 300. The substrate 310 includes residual stress therein that were imparted prior to forming the coating 312. Since the residual stress was preserved, mechanical treatments were unnecessary subsequent to forming the coating 312. Therefore, the coating 312 has an as-formed structure free of mechanically induced defects.

[0025] In this example, the component 300 includes a dual microstructure produced by controlling the induction heat treatment and the thickness of the coating 312. Portions of the component 300 directly treated by induction are represented as being located between a series of solid circles 330. The coating 312 includes coating portions 314, 316, 318, 320, 322, and 324 having various thicknesses. As represented, the combination of the controlled heat treatment and the varying thickness of the

coating 312 results in a dual microstructure wherein the component 300 includes a coarse grain region 326 and a fine grain region 328. As a nonlimiting example, the coarse grain region 326 may have grains with a minimum or average grain size of greater than 20  $\mu m$  and the fine grain region 328 may have grains with a maximum or average grain size of less than 20  $\mu m$ . The component 300 may include additional grain regions between the coarse grain region 326 and the fine grain region 328 having grain sizes that transition from the grain sizes of the coarse grain region 326 to the grain sizes of the fine grain region 328.

**[0026]** The component 300 is configured to be installed in a gas turbine engine. For example, the component 300 may be a shroud.

[0027] The methods disclosed herein and components formed therewith provide various benefits over certain existing methods and components. For example, use of the combination heat treatment combines two heat treatment processes into one heat treatment process which reduces the steps necessary to produce the components, thereby potentially reducing the cost and time required for production thereof. Further, aging the substrate with the coating thereon prevents or reduces the likelihood of the formation of an oxide layer on the substrate. This in turn avoids the necessity of removing such oxide layer and therefore further reduces the number of steps necessary to produce the component.

**[0028]** The methods may provide for producing components having novel properties. For example, the methods allow for maintaining residual stress within the substrate during the coating formation process unlike certain prior art process, for example, that cause diffusion between the coating and the surface. In such embodiments, subsequent mechanical treatments may be omitted and therefore, the coating may be free of surface defects (e.g., scratches, abrasions, etc.) that may be created by such treatments. This may allow for the coating to exhibit improved performance relative to comparable coatings having such defects.

[0029] In this document, relational terms such as first and second, and the like may be used solely to distinguish one entity or action from another entity or action without necessarily requiring or implying any actual such relationship or order between such entities or actions. Numerical ordinals such as "first," "second," "third," etc. simply denote different singles of a plurality and do not imply any order or sequence unless specifically defined by the claim language. The sequence of the text in any of the claims does not imply that process steps must be performed in a temporal or logical order according to such sequence unless it is specifically defined by the language of the claim. The process steps may be interchanged in any order without departing from the scope of the invention as long as such an interchange does not contradict the claim language and is not logically nonsensical.

[0030] Furthermore, depending on the context, words such as "connect" or "coupled to" used in describing a

relationship between different elements do not imply that a direct physical connection must be made between these elements. For example, two elements may be connected to each other physically, electronically, logically, or in any other manner, through one or more additional elements.

[0031] While at least one exemplary embodiment has been presented in the foregoing detailed description of the invention, it should be appreciated that a vast number of variations exist. It should also be appreciated that the exemplary embodiment or exemplary embodiments are only examples, and are not intended to limit the scope, applicability, or configuration of the invention in any way. Rather, the foregoing detailed description will provide those skilled in the art with a convenient road map for implementing an exemplary embodiment of the invention. It being understood that various changes may be made in the function and arrangement of elements described in an exemplary embodiment without departing from the scope of the invention as set forth in the appended claims.

#### Claims

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 A method for producing a component, the method comprising:

applying a slurry or paste to a surface of a substrate, wherein the substrate includes a nickel-based superalloy;

drying the slurry or paste applied to the surface to define a coated substrate; and

performing a heat treatment on the coated substrate to simultaneously densify and/or crystal-lize the dried slurry or paste to thereby form a coating that is bonded to the surface and to age the substrate to achieve a specific hardness therein by controllably forming precipitates of an intermetallic phase within the substrate and/or controllably modifying a size distribution of the precipitates of the intermetallic phase within the substrate,

wherein residual stress within the substrate are preserved during the heat treatment.

- 2. The method of claim 1, wherein the coating is configured for resistance to hot corrosion caused by molten salts of sodium, magnesium, vanadium, and/or sulfur dioxide gas, wherein the coating is configured to react with the molten salts to increase a melting point of the molten salts thereby reducing deposition thereof on the component.
- The method of claim 1, wherein the coating comprises a mixture of two or more metal oxides, which are selected from the group consisting of: barium oxide, silicon oxide, strontium oxide, aluminum oxide, mag-

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nesium oxide, calcium oxide, cobalt oxide, boron oxide, iron oxide, zirconium oxide, nickel oxide, and titanium oxide, wherein the coating is in fully crystalline form and/or a mixture of crystalline and glass phases.

- **4.** The method of claim 1, wherein the component is configured to be installed in a gas turbine engine.
- **5.** The method of claim 1, wherein performing the heat treatment on the substrate comprises:

generating a magnetic field around the substrate with an inductor connected to an electrical power source sufficient to induce eddy currents within the substrate and dissipate heat by Joule effect within the substrate; and controlling the electrical power source while generating the magnetic field to provide the magnetic field for a duration of application of the

magnetic field for a duration of application of the magnetic field sufficient to directly induce the eddy currents within the substrate so as to produce a heat level within the substrate to form the precipitates.

- 6. The method of claim 1, further comprising producing the substrate such that the substrate has a final net shape and dimension prior to applying the slurry or coating to the surface of the substrate, wherein producing the substrate results in the residual stress within the substrate.
- **7.** The method of claim 6, further comprising:

performing a solution heat treatment on the substrate to dissolve other precipitates in the component and to homogenize a microstructure of the substrate; and

performing a stabilization heat treatment to optimize a size and a morphology of the precipitates of the intermetallic phase within the substrate.

- 8. The method of claim 1, wherein performing the heat treatment comprises increasing and maintaining the temperature of the substrate to a temperature range of between 750 and 850 °C for a duration of between 8 to 32 hours in a vacuum, an inert atmosphere, and/or atmosphere having a different partial pressure of oxygen with respect to air.
- 9. The method of claim 1, wherein performing the heat treatment produces a microstructure in the substrate having a grain size gradient that transitions from fine grains having a grain size of less than 20  $\mu$ m to coarse grains having a grain size of greater than 20  $\mu$ m.

**10.** The method of claim 9, wherein performing the heat treatment includes controlling the grain sizes of the microstructure of the substrate by:

generating a magnetic field around the substrate with an inductor connected to an electrical power source sufficient to induce eddy currents within the substrate and dissipate heat by Joule effect within the substrate;

controlling the electrical power source, by the processor, while generating the magnetic field to provide the magnetic field for a duration of application of the magnetic field sufficient to directly induce the eddy currents within the substrate so as to produce a heat level within the substrate to grow the grains,

wherein the heat level is lower to produce the fine grains and greater to produce the coarse grains; and

applying the slurry or paste in a manner such that a thickness of the coating on the surface varies across the surface, wherein the coating is thicker over areas of the surface having the coarse grains and thinner over areas of the surface having the fine grains.

## **11.** A component comprising:

a substrate comprising a nickel-based superalloy having precipitates of an intermetallic phase, the substrate having a specific hardness therein; and

a coating bonded to a surface of the substrate, the coating configured for resistance to hot corrosion caused by molten salts of sodium, magnesium, vanadium, and/or sulfur dioxide gas, wherein the coating is configured to react with the molten salts to increase a melting point of the molten salts thereby reducing deposition thereof on the component,

wherein the substrate includes residual stress therein,

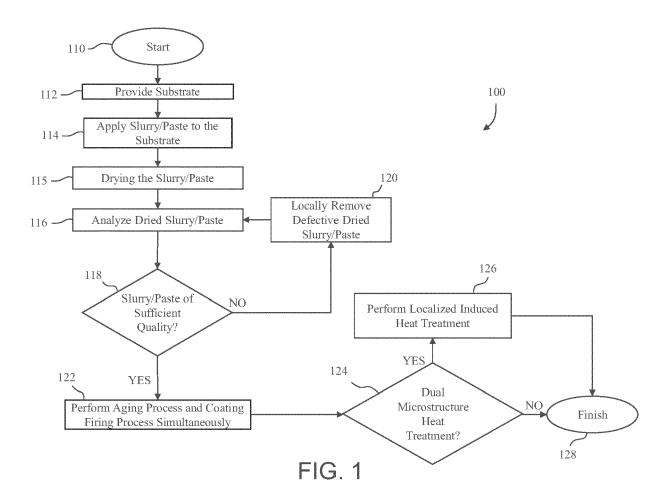
wherein the coating has an as-formed structure free of mechanically induced defects created during production of the component.

- 12. The component of claim 11, wherein the coating comprises a mixture of two or more metal oxides, which are selected from the group consisting of: barium oxide, silicon oxide, strontium oxide, aluminum oxide, magnesium oxide, calcium oxide, cobalt oxide, boron oxide, iron oxide, zirconium oxide, nickel oxide, and titanium oxide, wherein the coating is in fully crystalline form and/or a mixture of crystalline and glass phases.
- 13. The component of claim 11, wherein the component is configured to be installed in a gas turbine engine.

- 14. The component of claim 11, wherein a microstructure of the substrate has a grain size gradient that transitions from fine grains having a grain size of less than 20  $\mu$ m to coarse grains having a grain size of greater than 20  $\mu$ m.
- **15.** The component of claim 11, wherein the component is formed by a method comprising:

applying a slurry or paste to the surface of the substrate; drying the slurry or paste applied to the surface to form a coated substrate; and performing a heat treatment on the dried slurry or paste to simultaneously densify and/or crystallize the dried slurry or paste to thereby form the coating and to age the substrate to achieve the specific hardness therein by controllably forming the precipitates of the intermetallic phase within the substrate and/or controllably modifying a size distribution of the precipitates of the intermetallic phase within the substrate, wherein the residual stress within the substrate

are preserved during the heat treatment.



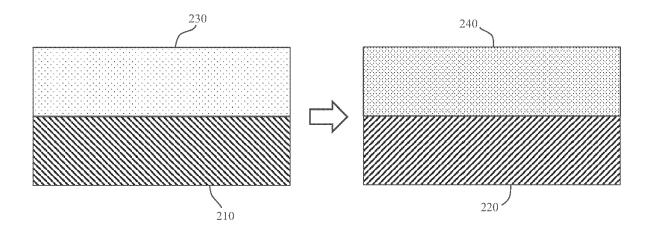


FIG. 2

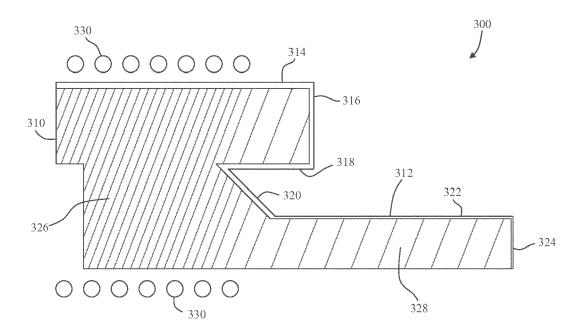


FIG. 3



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## EP 4 394 082 A1

## ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 23 21 4207

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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