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(54) **Precipitation hardening steel with improved toughness and method**

Ausscheidungshärtender Stahl mit verbesserter Festigkeit und Verfahren

Acier à durcissement par précipitation avec ténacité améliorée et procédé

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- **Davis J.R., Ed.: "ASM Specialty Handbook-Stainless Steels", 1996, ASM International, XP002732565, ISBN: 0-87170-503-6 page 34-37, 300-302, * figure 20; tables 18,20 ***

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Description

CLAIM OF PRIORITY

[0001] This application claims priority to U.S. Patent Application Serial No. 14/038,229 filed on September 26, 2013.

BACKGROUND

[0002] The following description relates to precipitation hardening steel with improved toughness and a heat treatment process for making the same.

[0003] Precipitation hardening (PH) steels are steel alloys which utilize an aging treatment to form additional phases which improve the alloy's mechanical properties. Precipitation hardening steel is widely used in industry for fittings, shafts, pins, aircraft components, and chemical applications. Precipitation hardening steel is typically heat treated using standard processes. To improve toughness of precipitation hardening steel, higher temperature heat treatments can be used. Also, refined chemistry can be used.

[0004] GB 1 056 561 discloses a precipitation-hardenable, stainless steel that comprises:- .Cr 7.- 18% .Ni 6.- 12% .Al 0.5.- 2.5% .Mn up to.1% .Si up to.1% .C up to.0.05% .P up to.0.015% .S up to.0.1% .N up to.0.05% the balance being iron. The steels may also optionally contain:- .Mo up to.8% .Ti up to.0.1% .B up to.0.003% and one half of the nickel may be substituted by Co in the ratio of three parts Co for each part of Ni replaced. The steel may be hot worked, annealed at 980-1093 DEG C., quenched in air, oil or water and then fabricated. It is then austenitized at 704-955 DEG C., cooled to between 150 DEG C. and - 129 DEG C. and tempered at 370 DEG - 650 DEG C. followed by cooling in air, oil or water.

[0005] US 2011/232809 discloses a precipitation-hardened stainless steel alloy that comprises, by weight: about 14.0 to about 16.0 percent chromium; about 6.0 to about 8.0 percent nickel; about 1.25 to about 1.75 percent copper; greater than about 1.5 to about 2.0 percent molybdenum; about 0.001 to about 0.025 percent carbon; niobium in an amount greater than about twenty times that of carbon; and the balance iron and incidental impurities. The alloy has an aged microstructure and an ultimate tensile strength of at least about 1100 MPa and a Charpy V-notch toughness of at least about 69 J. The aged microstructure includes martensite and not more than about 10% reverted austenite. Alternatively, the alloy includes substantially all martensite and substantially no reverted austenite. The alloy is useful for making turbine airfoils.

[0006] Davis J.R., Ed.: "ASM Speciality Handbook-Stainless Steels", 1996, ASM International, ISBN: 0-87170-503-6, p34-37, 300-302 discloses methods for annealing of precipitation hardening stainless steels in hydrogen, argon or helium atmospheres to obtain a scale free surface.

[0007] According to a first aspect of the invention, there is provided a heat treatment process for precipitation hardening steel, as claimed in claim 1.

[0008] According to a second aspect of the invention, there is provided a vertical lift aircraft component as claimed in claim 6.

DESCRIPTION OF DRAWINGS

10 **[0009]**

FIG. 1 is a diagram of an example heat treatment system;

15 FIG. 2 is a flowchart diagram of an example heat treatment process for precipitation hardening steel; and

20 FIG. 3 is an example plot based on exemplary data of material characteristics vs. an extended aging time.

[0010] Like reference symbols in the various drawings indicate like elements.

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DETAILED DESCRIPTION

[0011] FIG. 1 is a schematic diagram of an example heat treatment system 100. The heat treatment system 100 is a collection of equipment, chambers, and components that can be used to perform a heat treatment process on a precipitation hardening steel part. The heat treatment process can be controlled to alter or enhance the material characteristics of the precipitation hardening steel. For example, the process can be controlled to increase the toughness of a precipitation hardening steel part while maintaining strength. The precipitation hardening steel part could be any suitable part, component, tool or other (item?) used in any applicable industry such as aerospace, aviation, nuclear, or automotive. In aviation, precipitation hardening steel may, for example, be used for fixed-wing aircraft, rotor-winged aircraft such as helicopters, vertical-lift aircraft and tilt-rotor aircraft. The precipitation hardening steel part could be composed of any suitable steel, such as 13Cr-8Ni (PH 13-8, UNS S13800, XM-13).

[0012] Referring to FIG. 1, the heat treatment system 100 includes a solution treatment furnace 102, a protective atmosphere cooling chamber 104, a low temperature cooling chamber 106, an aging treatment furnace 108, and an air cooling chamber 110. Though, the system 100 may not include a separate protective atmosphere cooling chamber 104 or an air cooling chamber 110 without departing from the scope of the disclosure. For example, the solution treatment furnace 102 may be a vacuum furnace where both the solution treatment and the protective atmosphere cooling occur. In instances that do not include a separate air cooling chamber 110, air cool-

ing may be accomplished by removing a part from the aging treatment furnace 108 or opening the furnace doors and cooling in ambient air. The components of the heat treatment system 100 may vary in their configuration or arrangement. For example, the protective atmosphere cooling chamber 104 can be incorporated into the solution treatment furnace 102. As another example, the protective atmosphere cooling chamber 104 and the low temperature cooling chamber 106 could be the same chamber or equipment. One or more components may be connected as part of a workflow or may be separate. For example, conveyors, rollers, or other mechanisms could convey a part from one component to another. In some implementations, one or more of the components can be omitted, or other components can be included.

[0013] The solution treatment furnace 102 is a furnace capable of heating a precipitation hardening steel part to the austenitizing temperature. The austenitization temperature is a temperature at which, given sufficient time, will ensure transformation of the steel to an austenitic structure. The austenitization temperature may vary depending on a steel's particular chemical composition. As an example, the austenitization temperature for PH 13-8 is typically a temperature in the range of 930 °C (1700 °F). In a typical implementation, the precipitation hardening steel part is held at each of one or more stages at a temperature setting until that temperature is substantially or otherwise uniform throughout the part. For example, the precipitation hardening steel part could be heated in the furnace to a temperature of 930 °C (1700 °F) and held at that temperature for one hour after the part has uniformly reached 930 °C (1700 °F). A different austenitizing temperature or a different period of time could be used. Any suitable treatment furnace like those found in the industry can be used. For example, the solution treatment furnace 102 can have a gas or electric heat source, or may process the part in air, a vacuum, or protective atmosphere.

[0014] The protective atmosphere cooling chamber 104 is a chamber or apparatus in which the steel part can cool or quench in a protective atmosphere after solution treatment heating. The protective atmosphere can be a gas such as nitrogen or argon, or it can be a gas mixture such as a mixture of nitrogen and hydrogen. The protective atmosphere cooling chamber 104 can cool the precipitation hardening steel part to a given temperature such as 300 °F or room temperature. In some instances, the protective atmosphere cooling chamber 104 is incorporated into the solution treatment furnace 102. For example, the temperature setting of the solution treatment furnace 102 could be lowered without removing the steel part, or a protective atmosphere could be introduced into the solution treatment furnace 102. As previously mentioned, the system 100 may not include a separate protective atmosphere cooling chamber 104.

[0015] The low temperature cooling chamber 106 is a chamber or apparatus in which the precipitation hardening steel part can cool or quench to a relatively low tem-

perature for conversion of the steel structure to martensite. For example, the low temperature cooling chamber 106 could cool a precipitation hardening steel part to a temperature below room temperature, such as below 16 °C (60 °F) or -73 °C (-100 °F). For example, the precipitation hardening steel part may be cooled below 0 °C (32 °F). In some implementations, the cooling process may improve fatigue performance. In some instances, the part in the low temperature cooling chamber 106 is maintained at a low temperature by a liquid nitrogen bath or a deep freeze chiller. In some instances, the low temperature cooling chamber 106 may be combined with the protective atmosphere cooling chamber 104. For example, the part could be cooled to room temperature and then down to -73 °C (-100 °F) in a single chamber.

[0016] The aging treatment furnace 108 is a furnace for applying an aging heat treatment to a precipitation hardening steel part. In some instances, the aging treatment furnace 108 is the same furnace as the solution treatment furnace 102. The aging treatment furnace 108 can be configured to hold a steel part at a set temperature for a period of time. The set temperature can be a temperature that will create reverted austenite in the precipitation hardening steel part, such as a temperature in the range of 510- 620 °C (950-1150 °F). After the steel part has uniformly reached the set temperature, the aging treatment furnace 108 can maintain the part at that temperature for a period of time. For example, the aging treatment furnace 108 can be configured to maintain the precipitation hardening steel part at a set temperature of 540 °C (1000 °F) for more than four hours, for example, for five or more hours.

[0017] The example air cooling chamber 110 is a chamber within which a precipitation hardening steel part can cool to room temperature after an aging treatment. The example air cooling chamber 110 uses a cool or room temperature environment to allow the part to cool to room temperature. In some cases, other cooling techniques such as a water bath or an oil bath could be implemented instead of or in tandem with air cooling. The example air cooling chamber 110 can be incorporated into the aging treatment furnace 108. For example, the aging treatment furnace 108 can cool to room temperature after the aging treatment. As previously mentioned, the system 100 may not include a separate air cooling chamber 110.

[0018] FIG. 2 shows an example heat treatment process 200. The heat treatment process 200 is an example process to toughen a precipitation hardening steel part. In one embodiment, the toughness of the precipitation hardening steel part may be improved while maintaining tensile strength, other strength or other properties of the precipitation hardening steel above desired levels. In some cases, sub processes of the heat treatment process 200 may be omitted or combined. For example, the heat treatment process 200 may not include at least one of cooling in a protective atmosphere or warming to ambient temperature. The heat treatment process 200 can

use some or all of the system described in FIG. 1.

[0019] At 202, the precipitation hardening steel part is subjected to a solution treatment. This solution treatment is a heat treatment that holds the steel part at an austenitization temperature for a period of time. The steel part to be treated can be heated in a furnace such as the solution treatment furnace 102 in FIG. 1. In some cases, the precipitation hardening steel part is placed in the furnace when the furnace is at room temperature, and the temperature of the furnace is subsequently increased to the austenitization temperature. In this manner, the temperature of the steel part can ramp along with the temperature of the furnace. Once the precipitation hardening steel part has uniformly reached the set austenitization temperature, the part is held at that temperature until the part has formed a substantially or otherwise homogenous austenite structure. The length of time that the precipitation hardening steel part is held at the austenitization temperature can depend on the size and shape of the part. For example, a part can be ramped to 930 °C (1700 °F) and held at 930 °C (1700 °F) for 1 hour.

[0020] At 204, the solution-treated part is cooled or quenched in a protective atmosphere. The precipitation hardening steel part can be allowed to cool in the solution treatment furnace or in a separate environment such as the protective atmosphere chamber 104 of FIG. 1. Cooling the precipitation hardening steel part in a protective atmosphere can reduce oxidation and other undesired chemical reactions. In some cases, the precipitation hardening steel part is allowed to cool to room temperature.

[0021] At 206, the precipitation hardening steel part is cooled to a low temperature to facilitate martensitic conversion. The low quenching temperature can be a temperature below room temperature, such as at or below 16 °C (60 °F), 0 °C (32 °F), or -73 °C (-100 °F). A lower temperature (such as -73 °C (-100 °F)) can increase the driving force for martensitic conversion and create a substantially or otherwise uniform martensitic microstructure. The precipitation hardening steel part can be cooled with a variety of techniques, including placement in a deep freeze chiller or a liquid nitrogen bath. The temperature of the part can be ramped down, or the part can be placed directly in a low-temperature environment (such as a liquid nitrogen bath). In some cases, the precipitation hardening steel part is held in the cooling environment long enough for the entire part to reach the desired temperature. At 208, the precipitation hardening steel part is allowed to return to an ambient temperature (such as room temperature).

[0022] At 210, the precipitation hardening steel part is subjected to an aging treatment. An aging treatment is a prolonged heat treatment in a furnace such as the aging treatment furnace 108 of FIG. 1. An aging treatment can increase fracture toughness in a precipitation hardening steel part. In some implementations, the precipitation hardening steel part is placed in a room temperature furnace, and the furnace temperature is increased to the

set aging temperature. In some implementations, the precipitation hardening steel part is placed into a furnace that is already at the set aging temperature. After the temperature of the part has reached the set aging temperature such that it is substantially or otherwise uniform at the temperature, the precipitation hardening steel part is maintained at that temperature for a period of time. For example, the set aging temperature can be a temperature in the range of 510-620 °C (950-1150 °F). In the illustrated embodiment, the precipitation hardening steel part is held or heated at a temperature of 510- 620 °C (950-1150 °F) for five or more hours. The heat treatment time may, for example, be 5.5 hours, 6 hours, 6.5 hours, 7 hours, 7.5 hours, 8 hours, or some other suitable time. Holding the precipitation hardening steel part at the aging temperature of 540 °C (1000 °F) for five or more hours can additionally increase fracture toughness and additionally enhance resistance to hydrogen embrittlement. An extended age time of five or more hours can, for example, form retained austenite and allow martensite to revert to austenite in an even manner.

[0023] At 212, the precipitation hardening steel part is cooled to room temperature. The precipitation hardening steel part can be cooled in situ by bringing the aging treatment furnace to room temperature. The precipitation hardening steel part can also be cooled by removing the part from the furnace to a cooler environment, such as a chiller or the ambient temperature of the room. In some cases, the precipitation hardening steel part is cooled in a protective atmosphere. These and other cooling techniques can be implemented.

[0024] FIG. 3 shows an example plot 300 based on exemplary data, showing material properties curves 302, 304, and 306 for precipitation-hardened 13-8 stainless steel (PH-13-8) after undergoing extended aging treatments for more than four hours. The PH-13-8 stainless steel characterized in FIG. 3 was treated using the example process described in FIG. 2. The PH-13-8 stainless steel was solution treated at 930 °C (1700 °F), cooled at -73 °C (-100 °F), and aged at 540 °C (1000 °F) for four to eight hours. In FIG. 3, curve 302 represents the ultimate tensile strength (UTS) of the steel, curve 304 represents the yield strength (YS), and curve 306 represents the fracture toughness (K_q). In FIG. 3, the x-axis is the aging time of the PH-13-8 stainless steel in hours. The left y-axis is the tensile strength measured in ksi, corresponding to curves 302 and 304. The right y-axis is the fracture toughness measured in ksi√in, corresponding to curve 306. The horizontal line 308 represents an example minimum UTS standard of 1410 MPa (205 ksi). The horizontal line 310 represents an example minimum fracture toughness standard of 130 MPa√m (120 ksi√in) at room temperature. Room temperature is typically in the range of 18 °C to 24 °C (65 °F to 75 °F).

[0025] FIG. 3 shows that aging more than four hours, such as for five or more hours, can have a material effect on material properties of PH 13-8 stainless steel. For example, the fracture toughness curve 306 shows that

increasing the aging time more than 4 hours can result in increased fracture toughness of 155 MPa√m (140 ksi√in). At six or more hours, the example fracture toughness is 140 MPa√m (125 ksi√in), greater than the minimum standard fracture toughness of line 310. The ultimate tensile strength (curve 302) and yield strength (curve 304) of the 13-8 stainless steel is reduced as the aging time increases. However, for example, in one or more embodiments, the extended heat treatment aging at standard temperature allows improved toughness while limiting reduction in ultimate tensile strength and yield strength and/or maintaining ultimate tensile strength and yield strength above minimum or other desired limits. After 8 hours of aging, the example ultimate tensile strength remains greater than the minimum standard ultimate tensile strength of line 308. In specific embodiments, the extended aging time may yield material properties for the precipitation hardening steel of a fracture toughness greater than 125 MPa√m (115 ksi√in), 130 MPa√m (120 ksi√in), or 145 MPa√m (130 ksi√in) at room temperature, a yield strength greater than 1345 MPa (195 ksi) or 1380 MPa (200 ksi), and an ultimate tensile strength greater than 1450 MPa (210 ksi) or 1480 MPa (215 ksi). For example, the properties may include one or more of the following: about 1515 (220 ksi) strength and 95 MPa√m (85 ksi√in) toughness (H950); about 1275 MPa (185 ksi) strength and 155 MPa√m (140 ksi√in) toughness (H1025); about 1205 MPa (175 ksi) strength and 180 MPa√m (165 ksi√in) toughness (H1050); or others.

[0026] While this specification contains many details, these should not be construed as limitations on the scope of what may be claimed, but rather as descriptions of features specific to particular examples. Certain features that are described in this specification in the context of separate implementations can also be combined. Conversely, various features that are described in the context of a single implementation can also be implemented in multiple embodiments separately or in any suitable sub-combination.

[0027] A number of examples have been described. Nevertheless, it will be understood that various modifications can be made. Accordingly, other implementations are within the scope of the following claims.

Claims

1. A heat treatment process for precipitation hardening steel comprising:

a solution treatment for a precipitation hardening steel part, wherein the precipitation hardening steel part is heated to an austenitizing temperature to form a substantially homogenous austenite structure of the precipitation hardening steel part;
a quenching treatment including cooling the pre-

cipitation hardening steel part in a protective atmosphere;

a cooling treatment following the quenching treatment for a precipitation hardening steel part, wherein the precipitation hardening steel part is cooled to a martensitizing temperature to form a substantially uniform martensitic microstructure of the precipitation hardening steel part; and

an aging treatment for a precipitation hardening steel part, including heating the precipitation hardening steel part in a furnace at a temperature of at least 510 °C (950 °F) and at most 620 °C (1150 °F) for at least 6 hours until a fracture toughness of the precipitation hardening steel part is greater than 130 MPa√m (120 ksi√in) and until martensite reverts to austenite in an even manner; and

a cooling treatment following the ageing treatment, wherein the precipitation hardened steel is cooled to room temperature;

wherein the precipitation hardening steel part is composed of precipitation-hardened 13Cr-8Mo stainless steel (PH 13-8, UNS S13800, XM-13).

2. The heat treatment process of claim 1, wherein the austenitizing temperature is a temperature of 930°C (1700 °F).
3. The heat treatment process of claim 1, or of claim 2, wherein the martensitizing temperature is a temperature of 0 °C (32 °F) or lower.
4. The heat treatment process of claim 1, or of any of claim 2 to 3, wherein the temperature for the aging treatment is 537.5 °C plus or minus 7.5 °C (1000 °F plus or minus 15 °F).
5. The heat treatment process of claim 1, or of any of claims 2 to 4, wherein the aging treatment is for at least 7 hours; or is for at least 8 hours.
6. A vertical lift aircraft component of precipitation hardening steel formed by the process of any of claims 1 to 5.

Patentansprüche

1. Wärmebehandlungsprozess für ausscheidungshärtenden Stahl, umfassend:

eine Lösungsbehandlung für ein ausscheidungshärtendes Stahlteil, wobei der ausscheidungshärtende Stahl auf eine Austenitisiertemperatur erwärmt wird, um eine im Wesentlichen homogene Austenitstruktur des ausscheidungshärtenden Stahlteils zu bilden;

- ein Abschreckbehandlung, einschließlich Küh-
len des ausscheidungshärtendes Stahlteils in
einer Schutzatmosphäre;
eine Kühlungsbehandlung nach der Abschreck-
behandlung für ein ausscheidungshärtendes 5
Stahlteil, wobei der ausscheidungshärtende
Stahl auf eine Martensittemperatur zum Bil-
den einer im Wesentlichen gleichförmigen Mar-
tensitmikrostruktur des ausscheidungshärten-
den Stahlteils gekühlt wird; und 10
eine Alterungsbehandlung für ein ausschei-
dungshärtendes Stahlteil, einschließlich des Er-
wärmens des ausscheidungshärtenden
Stahlteils in einem Ofen bei einer Temperatur
von mindestens 510°C (950°F) und höchstens 15
620°C (1150°F) für mindestens 6 Stunden, bis
eine Bruchzähigkeit des ausscheidungshärten-
den Stahlteils größer als 130 MPa√m (120
ksi√in) ist und bis Martensit auf gleichmäßige 20
Weise zu Austenit rückgängig wird; und
eine Kühlungsbehandlung nach der Alterungs-
behandlung, wobei der ausscheidungshärtende
Stahl auf Raumtemperatur abgekühlt wird;
wobei das ausscheidungshärtende Stahlteil aus
ausscheidungsgehärtetem 13Cr-8Mo-Edel- 25
stahl (PH 13-8, UNS S13800 XM-13) besteht.
2. Wärmebehandlungsprozess nach Anspruch 1, wo-
bei die Austenittemperatur eine Temperatur von
930°C (1700°F) ist. 30
3. Wärmebehandlungsprozess nach Anspruch 1 oder
Anspruch 2, wobei die Martensittemperatur eine
Temperatur von 0°C (32°F) ist oder kleiner ist. 35
4. Wärmebehandlungsprozess nach Anspruch 1 oder
nach einem der Ansprüche 2 bis 3, wobei die Tem-
peratur für die Alterungsbehandlung 537,5°C plus
oder minus 7,5°C (1000°F plus oder minus 15°F)
beträgt. 40
5. Wärmebehandlungsprozess nach Anspruch 1 oder
nach einem der Ansprüche 2 bis 4, wobei die Alte-
rungsbehandlung für mindestens 7 Stunden ist; oder
für mindestens 8 Stunden ist. 45
6. Flugzeugbauteil mit vertikalem Hub aus ausschei-
dungshärtendem Stahl, das durch das Verfahren
nach einem der Ansprüche 1 bis 5 gebildet wird. 50

Revendications

1. Procédé de traitement thermique pour de l'acier à
durcissement par précipitation comprenant : 55
- un traitement en solution pour une pièce en acier
à durcissement par précipitation, dans lequel la

pièce en acier à durcissement par précipitation
est chauffée à une température d'austénitisation
pour former une structure d'austénite pratique-
ment homogène de la pièce en acier à durcis-
sement par précipitation ;
un traitement de trempe consistant à refroidir la
pièce en acier à durcissement par précipitation
dans une atmosphère protectrice ;
un traitement de refroidissement à la suite du
traitement de trempe pour une pièce en acier à
durcissement par précipitation, dans lequel la
pièce en acier à durcissement par précipitation
est refroidie à une température de martensiti-
sation pour former une microstructure martensi-
tique pratiquement uniforme de la pièce en acier
à durcissement par précipitation ; et
un traitement de vieillissement pour une pièce
en acier à durcissement par précipitation, con-
sistant à chauffer la pièce en acier à durcis-
sement par précipitation dans un four à une tem-
pérature d'au moins 510 °C (950 °F) et d'au
maximum 620 °C (1150 °F) pendant au moins
6 heures jusqu'à ce que la ténacité à la rupture
de la pièce en acier à durcissement par précipi-
tation soit supérieure à 130 MPa√m (120 ksi√po)
et jusqu'à ce que la martensite se reconvertisse
en austénite d'une manière égale ; et
un traitement de refroidissement à la suite du
traitement de vieillissement, dans lequel l'acier
durci par précipitation est refroidi à température
ambiante;
dans lequel la pièce en acier à durcissement par
précipitation est composée d'acier inoxydable
13Cr-8Mo durci par précipitation (PH 13-8, UNS
S13800, XM-13).

2. Procédé de traitement thermique selon la revendi-
cation 1, dans lequel la température d'austénitisation
est une température de 930 °C (1700 °F).
3. Procédé de traitement thermique selon la revendi-
cation 1, ou selon la revendication 2, dans lequel la
température de martensitisation est une tempéra-
ture inférieure ou égale à 0 °C (32 °F).
4. Procédé de traitement thermique selon la revendi-
cation 1, ou selon l'une quelconque des revendica-
tions 2 à 3, dans lequel la température pour le trai-
tement de vieillissement est de 537,5 °C plus ou
moins 7,5 °C (1000 °F plus ou moins 15 °F).
5. Procédé de traitement thermique selon la revendi-
cation 1, ou selon l'une quelconque des revendica-
tions 2 à 4, dans lequel le traitement de vieillissement
se fait pendant au moins 7 heures ; ou se fait pendant
au moins 8 heures.
6. Élément d'aéronef de poussée verticale constitué

d'acier à durcissement par précipitation formé par le procédé selon l'une quelconque des revendications 1 à 5.

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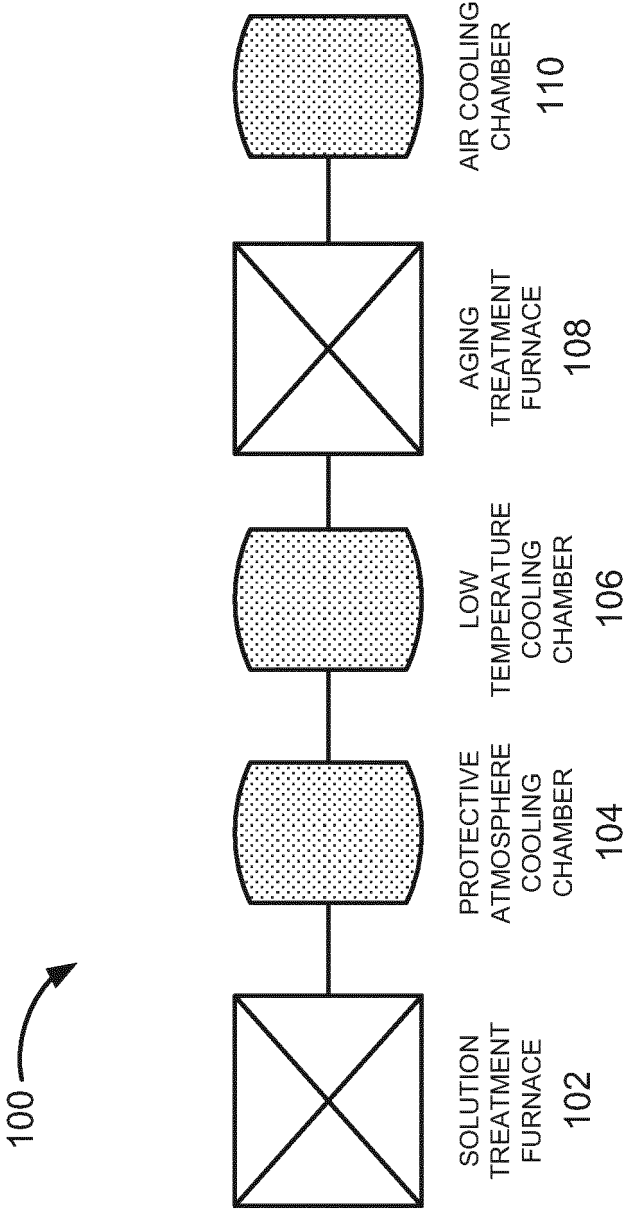


FIG. 1

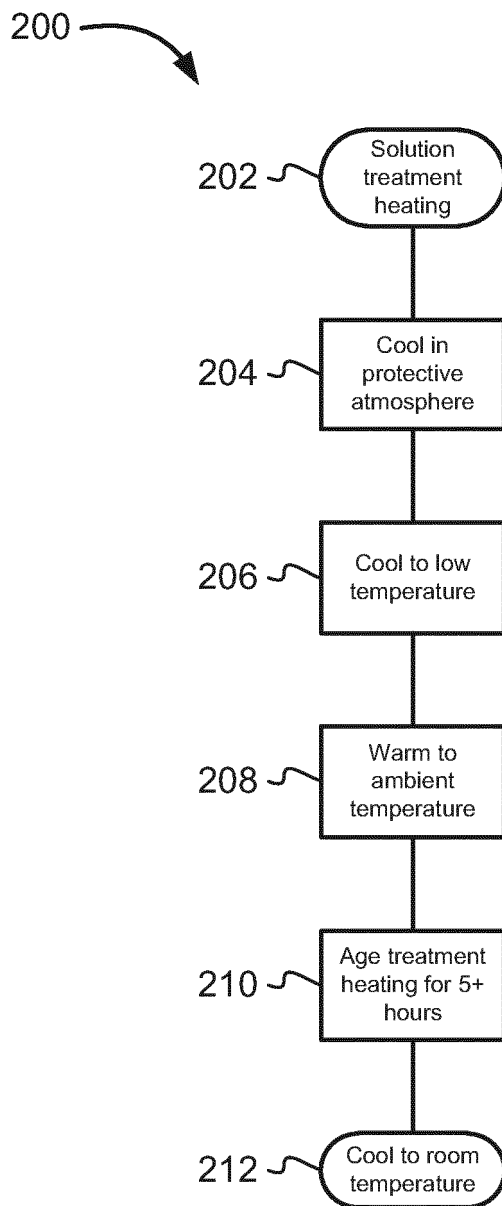


FIG. 2



REFERENCES CITED IN THE DESCRIPTION

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