



(11) **EP 3 676 412 B1**

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention  
of the grant of the patent:  
**14.08.2024 Bulletin 2024/33**

(21) Application number: **18766496.6**

(22) Date of filing: **28.08.2018**

(51) International Patent Classification (IPC):  
**C22F 1/047** <sup>(2006.01)</sup> **B22D 11/00** <sup>(2006.01)</sup>  
**B21B 3/00** <sup>(2006.01)</sup> **C22C 21/08** <sup>(2006.01)</sup>  
**C22F 1/053** <sup>(2006.01)</sup>

(52) Cooperative Patent Classification (CPC):  
**C22F 1/053; B22D 7/005; C22C 21/10;**  
**C22F 1/047; B22D 11/003**

(86) International application number:  
**PCT/US2018/048305**

(87) International publication number:  
**WO 2019/046275 (07.03.2019 Gazette 2019/10)**

(54) **7XXX SERIES ALUMINUM ALLOY PRODUCTS IN A STABILIZED T4 TEMPER AND METHODS OF MAKING THE SAME**

PRODUKTE AUS ALUMINIUMLEGIERUNG DER SERIE 7XXX IN STABILISIERTEM  
T4-HÄRTEGRAD UND VERFAHREN ZU IHRER HERSTELLUNG

PRODUITS EN ALLIAGE D'ALUMINIUM DE SÉRIE 7XXX DANS UN ÉTAT DE REVENU T4  
STABILISÉ ET LEURS PROCÉDÉS DE FABRICATION

(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 MK MT NL NO**  
**PL PT RO RS SE SI SK SM TR**

(30) Priority: **29.08.2017 US 201762551497 P**

(43) Date of publication of application:  
**08.07.2020 Bulletin 2020/28**

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**CN-A- 106 906 435 US-A1- 2017 121 802**

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**Description****PRIORITY CLAIM**

**[0001]** This application claims the benefit of and priority to U. S. Provisional Application No. 62/551,497, filed on August 29, 2017.

**FIELD**

**[0002]** The present disclosure generally provides 7xxx series aluminum alloy products in a stable T4 temper. The disclosure provides methods of making such products, for example, using processes that include a combination of casting, rolling, solutionizing, quenching, reheating, and slow cooling. The disclosure also provides various end uses of such products, such as in automotive, transportation, electronics, and industrial applications.

**BACKGROUND**

**[0003]** Aluminum alloys of the 7xxx series are used in a number of contexts, especially where high strength and light weight are particularly desirable. For that reason, such alloys are frequently used in the aerospace industry and as encasements for various electronic products, such as cellular phones. Notwithstanding these advantages, the process of forming 7xxx series aluminum alloys can pose certain challenges, which can increase the costs of using such alloys in certain manufactured goods.

**[0004]** For example, 7xxx series aluminum alloy products are often supplied to users in an F temper, meaning that it is supplied in an as-fabricated form, where, for example, no solutionizing is performed after the rolling process. Users must therefore solutionize the product themselves and form it into a manufactured product by a hot forming process. In some other instances, 7xxx series aluminum alloy products are supplied to users in a T6 temper, where the aluminum alloy product undergoes solutionizing followed by artificial aging. Users can generally form such products at room temperature, but their formability is quite low.

**[0005]** It is possible to make 7xxx series aluminum alloy products using processes consistent with the production of aluminum alloy products in a T4 temper, where the products are subjected to solutionizing and allowed to age naturally. Such products would presumably exhibit desirable formability at room temperature compared to products supplied in a T6 temper. US 2017/0121802 A1 refers to a method for producing an aluminum alloy product comprising casting, homogenizing, hot rolling, cold rolling, optionally solution heat treating at 430 to 600°C, optionally cooling the sheet to a temperature of 25-120°C and subsequent aging. But such processes generally yield products in an unstable T4 temper that hardens within a few days and ceases to exhibit desirable formability properties. Because aluminum alloy products cannot easily be supplied and used in such a tight time win-

dow, aluminum alloy manufacturers have not typically supplied such 7xxx series aluminum alloy products to the market.

5 **SUMMARY**

**[0006]** Covered embodiments of the invention are defined by the claims, not this summary. This summary is a high-level overview of various aspects of the invention and introduces some of the concepts that are further described in the Detailed Description section below. This summary is not intended to identify key or essential features of the claimed subject matter, nor is it intended to be used in isolation to determine the scope of the claimed subject matter. The subject matter should be understood by reference to appropriate portions of the entire specification, any or all drawings and each claim.

**[0007]** The present disclosure provides novel 7xxx series aluminum alloy products in a T4 temper that is stable for a substantial period of time, for example, up to 6 months, before beginning to harden and thereby losing formability. Because these products are able to retain the formability benefits of this T4 temper for such a long period of time, they can be readily incorporated into manufacturing production cycles. This was impossible for previously known 7xxx series aluminum alloy products in a T4 temper, as those products generally began to harden and lose formability within mere days of their production. By the time the aluminum alloy product arrived at a buyer's plant, the material would likely have lost the beneficial formability properties it once possessed. Thus, the discovery of a 7xxx series aluminum alloy product in a stable T4 temper offers a significant advancement in the state of the art, and allows such materials to be incorporated into products of manufacture with greater ease and at much lower costs.

**[0008]** In a first aspect, the disclosure provides a method of making a rolled aluminum alloy product, comprising providing a 7xxx series aluminum alloy, wherein the 7xxx series aluminum alloy is a molten 7xxx series aluminum alloy; casting the molten 7xxx series aluminum alloy to provide an aluminum alloy cast product; homogenizing the aluminum alloy cast product to provide a homogenized aluminum alloy cast product; rolling the homogenized aluminum alloy cast product to form a rolled aluminum alloy product; solutionizing and, then, pre-aging, the rolled aluminum alloy product, wherein the pre-aging comprises heating the rolled aluminum alloy product to a pre-aging temperature ranging from 60 °C to 130 °C, wherein the pre-aging is conducted for a period of time up to 24 hours; and coiling the rolled aluminum alloy product after the pre-aging. In some embodiments, the aluminum alloy product is a strip, a shate, a sheet, a plate, a billet, or other aluminum alloy product. In some such embodiments, the rolled aluminum alloy product exhibits an increase in its yield strength (Rp) of no more than 25 MPa during a post-production period immediately following the pre-aging, wherein the post-production period

ranges from 15 days to 180 days.

**[0009]** Additional aspects and embodiments are set forth in the detailed description, claims, non-limiting examples, and drawings, which are included herein.

#### BRIEF DESCRIPTION OF THE DRAWINGS

**[0010]**

FIG. 1 shows the change in the yield strength (Rp) as a function of the number of days following initial production for aluminum alloy samples prepared with and without pre-aging (PX).

FIG. 2 shows the change in the yield strength (Rp) as a function of the number of days following initial production for aluminum alloy samples prepared without pre-aging (PX) and with pre-aging under various conditions.

FIGS. 3A-C show the change in yield strength (Rp) for aluminum alloy samples as a function of pre-aging time, pre-aging temperature, and whether a paint bake cycle was used.

FIGS. 4A-C show the change in elongation strength for aluminum alloy samples as a function of pre-aging time, pre-aging temperature, and whether a paint bake cycle was used.

FIGS. 5A-C show the change in uniform elongation for aluminum alloy samples as a function of pre-aging time, pre-aging temperature, and whether a paint bake cycle was used.

FIGS. 6A-C show the change in total elongation for aluminum alloy samples as a function of pre-aging time, pre-aging temperature, and whether a paint bake cycle was used.

FIGS. 7A-D show the change in critical fracture strain for aluminum alloy samples as a function of pre-aging time, pre-aging temperature, and whether a paint bake cycle was used.

FIGS. 8A-D show the change in n-value for aluminum alloy samples as a function of pre-aging time, pre-aging temperature, and whether a paint bake cycle was used.

FIGS. 6A-C show the change in total elongation for aluminum alloy samples as a function of pre-aging time, pre-aging temperature, and whether a paint bake cycle was used.

FIGS. 7A-D show the change in critical fracture strain for aluminum alloy samples as a function of pre-aging time, pre-aging temperature, and whether a paint

bake cycle was used.

FIGS. 8A-D show the change in n-value for aluminum alloy samples as a function of pre-aging time, pre-aging temperature, and whether a paint bake cycle was used.

#### DETAILED DESCRIPTION

**[0011]** The present disclosure provides methods of making 7xxx series aluminum alloy products that are in a stable T4 temper following solutionizing and pre-aging. These products can be easily formed at room temperature for a substantial period of time following pre-aging.

#### Definitions and Descriptions

**[0012]** In this description, reference is made to alloys identified by AA numbers and other related designations, such as "series" or "7xxx." For an understanding of the number designation system most commonly used in naming and identifying aluminum and its alloys, see "International Alloy Designations and Chemical Composition Limits for Wrought Aluminum and Wrought Aluminum Alloys" or "Registration Record of Aluminum Association Alloy Designations and Chemical Compositions Limits for Aluminum Alloys in the Form of Castings and Ingot," both published by The Aluminum Association.

**[0013]** As used herein, a "plate" generally has a thickness of greater than about 15 mm. For example, a plate may refer to an aluminum product having a thickness of greater than about 15 mm, greater than about 20 mm, greater than about 25 mm, greater than about 30 mm, greater than about 35 mm, greater than about 40 mm, greater than about 45 mm, greater than about 50 mm, or greater than about 100 mm.

**[0014]** As used herein, a "shate" (also referred to as a sheet plate) generally has a thickness of from about 4 mm to about 15 mm. For example, a shate may have a thickness of about 4 mm, about 5 mm, about 6 mm, about 7 mm, about 8 mm, about 9 mm, about 10 mm, about 11 mm, about 12 mm, about 13 mm, about 14 mm, or about 15 mm.

**[0015]** As used herein, a "sheet" generally has a thickness of less than 4 mm. For example, a sheet may have a thickness of less than about 4 mm, less than about 3 mm, less than about 2 mm, less than about 1 mm, less than about 0.5 mm, less than about 0.3 mm, or less than about 0.1 mm.

**[0016]** Reference may be made in this application to alloy temper or condition. For an understanding of the alloy temper descriptions most commonly used, see "American National Standards (ANSI) H35 on Alloy and Temper Designation Systems." An F condition or temper refers to an aluminum alloy as fabricated. An O condition or temper refers to an aluminum alloy after annealing. An Hxx condition or temper, also referred to herein as an H temper, refers to an aluminum alloy after cold rolling

with or without thermal treatment (e.g., annealing). Suitable H tempers include HX1, HX2, HX3 HX4, HX5, HX6, HX7, HX8, or HX9 tempers, along with Hxxx temper variations (e.g., H111), which are used for a particular alloy temper when the degree of temper is close to the Hxx temper. A T1 condition or temper refers to an aluminum alloy cooled from hot working and naturally aged (e.g., at ambient temperature). A T2 condition or temper refers to an aluminum alloy cooled from hot working, cold worked and naturally aged. A T3 condition or temper refers to an aluminum alloy solution heat treated, cold worked, and naturally aged. A T4 condition or temper refers to an aluminum alloy solution heat treated and naturally aged. A T5 condition or temper refers to an aluminum alloy cooled from hot working and artificially aged (at elevated temperatures). A T6 condition or temper refers to an aluminum alloy solution heat treated, quenched, and artificially aged. A T61 condition or temper refers to an aluminum alloy solution heat treated, quenched, naturally aged for a period of time, and then artificially aged. A T7 condition or temper refers to an aluminum alloy solution heat treated and artificially overaged. A T8x condition or temper (e.g., T8) refers to an aluminum alloy solution heat treated, cold worked, and artificially aged. A T9x condition or temper refers to an aluminum alloy solution heat treated, artificially aged, and cold worked.

**[0017]** The following aluminum alloys are described in terms of their elemental composition in weight percentage (wt. %) based on the total weight of the alloy. In certain examples of each alloy, the remainder is aluminum, with a maximum wt. % of 0.15 % for the sum of the impurities.

**[0018]** As used herein, the meaning of "room temperature" can include a temperature of from about 15 °C to about 30 °C, for example about 15 °C, about 16 °C, about 17 °C, about 18 °C, about 19 °C, about 20 °C, about 21 °C, about 22 °C, about 23 °C, about 24 °C, about 25 °C, about 26 °C, about 27 °C, about 28 °C, about 29 °C, or about 30 °C.

**[0019]** All ranges disclosed herein are to be understood to encompass any and all subranges subsumed therein. For example, a stated range of "1 to 10" should be considered to include any and all subranges between (and inclusive of) the minimum value of 1 and the maximum value of 10; that is, all subranges beginning with a minimum value of 1 or more, e.g. 1 to 6.1, and ending with a maximum value of 10 or less, e.g., 5.5 to 10.

**[0020]** As used herein, terms such as "cast product," "cast metal product," "cast aluminum product," "cast aluminum alloy product," and the like refer to a product produced by direct chill casting (including direct chill co-casting) or semi-continuous casting, continuous casting (including, for example, by use of a twin belt caster, a twin roll caster, a block caster, or any other continuous caster), electromagnetic casting, hot top casting, or any other casting method.

### Aluminum Alloy Compositions

**[0021]** The methods disclosed herein generally use 7xxx series aluminum alloys. In general, such alloys are aluminum alloys that include Zn as the major alloying element. Any suitable 7xxx series aluminum alloy can be used in the methods and products disclosed herein. For example, such 7xxx series aluminum alloys include, but are not limited to, the following 7xxx series aluminum alloys: AA7011, AA7019, AA7020, AA7021, AA7039, AA7072, AA7075, AA7085, AA7108, AA7108A, AA7015, AA7017, AA7018, AA7019A, AA7024, AA7025, AA7028, AA7030, AA7031, AA7033, AA7035, AA7035A, AA7046, AA7046A, AA7003, AA7004, AA7005, AA7009, AA7010, AA7011, AA7012, AA7014, AA7016, AA7116, AA7122, AA7023, AA7026, AA7029, AA7129, AA7229, AA7032, AA7033, AA7034, AA7036, AA7136, AA7037, AA7040, AA7140, AA7041, AA7049, AA7049A, AA7149, AA7204, AA7249, AA7349, AA7449, AA7050, AA7050A, AA7150, AA7250, AA7055, AA7155, AA7255, AA7056, AA7060, AA7064, AA7065, AA7068, AA7168, AA7175, AA7475, AA7076, AA7178, AA7278, AA7278A, AA7081, AA7181, AA7185, AA7090, AA7093, AA7095, and AA7099.

**[0022]** In some embodiments, the 7xxx series aluminum alloys used herein have an elemental composition as set forth in Table 1.

**Table 1**

Element	Weight Percentage (wt. %)
Zn	4.0 - 15.0
Cu	0.1 - 3.5
Mg	1.0 - 4.0
Fe	0.05 - 0.50
Si	0.05 - 0.30
Zr	0 - 0.50
Mn	0 - 0.25
Cr	0 - 0.20
Ti	0 - 0.15
Impurities	0 - 0.15
Al	Remainder

**[0023]** In some cases, the aluminum alloys used herein have an elemental composition as set forth in Table 2.

**Table 2**

Element	Weight Percentage (wt. %)
Zn	5.6 - 9.3
Cu	0.2 - 2.6
Mg	1.4-2.8

(continued)

Element	Weight Percentage (wt. %)
Fe	0.10 - 0.35
Si	0.05 - 0.20
Zr	0 - 0.25
Mn	0 - 0.05
Cr	0 - 0.05
Ti	0 - 0.05
Impurities	0 - 0.15
Al	Remainder

**[0024]** In some cases, the aluminum alloy includes zinc (Zn) in an amount of from 4% to 15% (e.g., from 5.4% to 9.5%, from 5.6% to 9.3%, from 5.8% to 9.2%, or from 4.0% to 5.0%) based on the total weight of the alloy. For example, the aluminum alloy can include about 4.0%, about 4.1%, about 4.2%, about 4.3 %, about 4.4 %, about 4.5%, about 4.6 %, about 4.7 %, about 4.8 %, about 4.9 %, about 5.0 %, about 5.1%, about 5.2 %, about 5.3%, about 5.4%, about 5.5%, about 5.6%, about 5.7%, about 5.8%, about 5.9%, about 6.0%, about 6.1%, about 6.2 %, about 6.3%, about 6.4%, about 6.5%, about 6.6%, about 6.7%, about 6.8%, about 6.9%, about 7.0%, about 7.1%, about 7.2%, about 7.3%, about 7.4%, about 7.5%, about 7.6%, about 7.7%, about 7.8%, about 7.9%, about 8.0%, about 8.1%, about 8.2%, about 8.3%, about 8.4%, about 8.5%, about 8.6%, about 8.7%, about 8.8%, about 8.9%, about 9.0%, about 9.1%, about 9.2%, about 9.3%, about 9.4%, about 9.5%, about 9.6%, about 9.7%, about 9.8%, about 9.9%, about 10.0%, about 10.1%, about 10.2%, about 10.3%, about 10.4%, about 10.5%, about 10.6%, about 10.7%, about 10.8%, about 10.9%, about 11.0%, about 11.1%, about 11.2%, about 11.3%, about 11.4%, about 11.5%, about 11.6%, about 11.7%, about 11.8%, about 11.9%, about 12.0%, about 12.1%, about 12.2%, about 12.3%, about 12.4%, about 12.5%, about 12.6%, about 12.7%, about 12.8%, about 12.9%, about 13.0%, about 13.1%, about 13.2 %, about 13.3%, about 13.4 %, about 13.5%, about 13.6%, about 13.7%, about 13.8%, about 13.9 %, about 14.0%, about 14.1%, about 14.2%, about 14.3%, about 14.4%, about 14.5%, about 14.6%, about 14.7%, about 14.8%, about 14.9%, or about 15.0% Zn. All are expressed in wt. %.

**[0025]** In some cases, the aluminum alloy includes copper (Cu) in an amount of from 0.1% to 3.5% (e.g., from 0.2% to 2.6%, from 0.3% to 2.5%, or from 0.15% to 0.6%) based on the total weight of the alloy. For example, the aluminum alloy can include about 0.1%, about 0.11%, about 0.12%, about 0.13%, about 0.14%, about 0.15%, about 0.16%, about 0.17%, about 0.18%, about 0.19%, about 0.20%, about 0.21%, about 0.22%, about 0.23%, about 0.24%, about 0.25%, about 0.26%, about 0.27%, about 0.28%, about 0.29%, about 0.30%, about 0.35%,

about 0.40%, about 0.45%, about 0.50%, about 0.55%, about 0.60%, about 0.65%, about 0.70%, about 0.75%, about 0.80%, about 0.85%, about 0.90%, about 0.95%, about 1.0%, about 1.1%, about 1.2%, about 1.3%, about 1.4%, about 1.5%, about 1.6%, about 1.7%, about 1.8%, about 1.9%, about 2.0%, about 2.1%, about 2.2%, about 2.3%, about 2.4%, about 2.5%, about 2.6%, about 2.7%, about 2.8%, about 2.9%, about 3.0%, about 3.1%, about 3.2%, about 3.3%, about 3.4%, or about 3.5% Cu. All are expressed in wt. %.

**[0026]** In some cases, the aluminum alloy includes magnesium (Mg) in an amount of from 1.0% to 4.0% (e.g., from 1.0% to 3.0%, from 1.4% to 2.8%, or from 1.6% to 2.6%). For example, the aluminum alloy can include about 1.0%, about 1.1%, about 1.2%, about 1.3%, about 1.4%, about 1.5%, about 1.6%, about 1.7%, about 1.8%, about 1.9%, about 2.0%, about 2.1%, about 2.2%, about 2.3%, about 2.4%, about 2.5%, about 2.6%, about 2.7%, about 2.8%, about 2.9%, about 3.0%, about 3.1%, about 3.2%, about 3.3%, about 3.4%, about 3.5%, about 3.6%, about 3.7%, about 3.8%, about 3.9%, or about 4.0% Mg. All are expressed in wt. %.

**[0027]** In some cases, the aluminum alloy includes a combined content of Zn, Cu, and Mg ranging from about 5% to 14% (e.g., from 5.5% to 13.5%, from 6% to 13%, from 6.5% to 12.5%, or from 7% to 12%). For example, the combined content of Zn, Cu, and Mg can be about 5.1%, about 5.5%, about 6.0%, about 6.5%, about 7.0%, about 7.5%, about 8.0%, about 8.5%, about 9.0%, about 9.5%, about 10.0%, about 10.5%, about 11.0%, about 11.5%, about 12.0%, about 12.5%, about 13.0%, about 13.5%, or about 14.0%. All are expressed in wt. %.

**[0028]** In some cases, the aluminum alloy includes iron (Fe) in an amount of from 0.05% to 0.50% (e.g., from 0.10% to 0.35% or from 0.10% to 0.25%) based on the total weight of the alloy. For example, the aluminum alloy can include about 0.05%, about 0.06%, about 0.07%, about 0.08%, about 0.09%, about 0.10%, about 0.11%, about 0.12%, about 0.13%, about 0.14%, about 0.15%, about 0.16%, about 0.17%, about 0.18%, about 0.19%, about 0.20%, about 0.21%, about 0.22%, about 0.23%, about 0.24%, about 0.25%, about 0.26%, about 0.27%, about 0.28%, about 0.29%, about 0.30%, about 0.31%, about 0.32%, about 0.33%, about 0.34%, about 0.35%, about 0.36%, about 0.37%, about 0.38%, about 0.39%, about 0.40%, about 0.41%, about 0.42%, about 0.43%, about 0.44%, about 0.45%, about 0.46%, about 0.47%, about 0.48%, about 0.49%, or about 0.50% Fe. All are expressed in wt. %.

**[0029]** In some cases, the aluminum alloy includes silicon (Si) in an amount of from 0.05% to 0.30% (e.g., from 0.05% to 0.25% or from 0.07% to 0.15%) based on the total weight of the alloy. For example, the aluminum alloy can include about 0.05%, about 0.06%, about 0.07%, about 0.08%, about 0.09%, about 0.10%, about 0.11%, about 0.12%, about 0.13%, about 0.14%, about 0.15%, about 0.16%, about 0.17%, about 0.18%, about 0.19%, about 0.20%, about 0.21%, about 0.22%, about 0.23%,

about 0.24%, about 0.25%, about 0.26%, about 0.27%, about 0.28%, about 0.29%, or about 0.30% Si. All are expressed in wt. %.

**[0030]** In some cases, the aluminum alloy includes zirconium (Zr) in amounts up to 0.50% (e.g., from 0.01% to 0.25%, from 0.03% to 0.20%, or from 0.05% to 0.15%) based on the total weight of the alloy. For example, the aluminum alloy can include about 0.01%, about 0.02%, about 0.03%, about 0.04%, about 0.05%, about 0.06%, about 0.07%, about 0.08%, about 0.09%, about 0.10%, about 0.11%, about 0.12%, about 0.13%, about 0.14%, about 0.15%, about 0.16%, about 0.17%, about 0.18%, about 0.19%, about 0.20%, about 0.21%, about 0.22%, about 0.23%, about 0.24%, about 0.25%, about 0.26%, about 0.27%, about 0.28%, about 0.29%, about 0.30%, about 0.31%, about 0.32%, about 0.33%, about 0.34%, about 0.35%, about 0.36%, about 0.37%, about 0.38%, about 0.39%, about 0.40%, about 0.41%, about 0.42%, about 0.43%, about 0.44%, about 0.45%, about 0.46%, about 0.47%, about 0.48%, about 0.49%, or about 0.50% Zr. In other examples, the alloys can include Zr in an amount less than 0.05% (e.g., about 0.04%, about 0.03%, about 0.02%, or about 0.01%) based on the total weight of the alloy. In some cases, Zr is not present in the alloy (i.e., 0%). All are expressed in wt. %.

**[0031]** In some cases, the aluminum alloy includes manganese (Mn) in an amount of up to 0.25% (e.g., from 0.01% to 0.10% or from 0.02% to 0.05%) based on the total weight of the alloy. For example, the aluminum alloy can include about 0.01%, about 0.02%, about 0.03%, about 0.04%, about 0.05%, about 0.06%, about 0.07%, about 0.08%, about 0.09%, about 0.10%, about 0.11%, about 0.12%, about 0.13%, about 0.14%, about 0.15%, about 0.16%, about 0.17%, about 0.18%, about 0.19%, about 0.20%, about 0.21%, about 0.22%, about 0.23%, about 0.24%, or about 0.25% Mn. In some cases, Mn is not present in the alloy (i.e., 0%). All are expressed in wt. %.

**[0032]** In some cases, the aluminum alloy includes chromium (Cr) in an amount of up to 0.20% (e.g., from 0.01% to 0.10%, from 0.01% to 0.05%, or from 0.03% to 0.05%) based on the total weight of the alloy. For example, the aluminum alloy can include about 0.01%, about 0.02%, about 0.03%, about 0.04%, about 0.05%, about 0.06%, about 0.07%, about 0.08%, about 0.09%, about 0.10%, about 0.11%, about 0.12%, about 0.13%, about 0.14%, about 0.15%, about 0.16%, about 0.17%, about 0.18%, about 0.19%, or about 0.20% Cr. In some cases, Cr is not present in the alloy (i.e., 0%). All are expressed in wt. %.

**[0033]** In some cases, the aluminum alloy includes titanium (Ti) in an amount of up to 0.15% (e.g., from 0.001% to 0.10%, from 0.001% to 0.05%, or from 0.003% to 0.035%) based on the total weight of the alloy. For example, the alloy can include about 0.001%, about 0.002%, about 0.003%, about 0.004%, about 0.005%, about 0.006%, about 0.007%, about 0.008%, about 0.009%, about 0.010%, about 0.011%, about 0.012%,

about 0.013%, about 0.014%, about 0.015%, about 0.016%, about 0.017%, about 0.018%, about 0.019%, about 0.020%, about 0.021%, about 0.022%, about 0.023%, about 0.024%, about 0.025%, about 0.026%, about 0.027%, about 0.028%, about 0.029%, about 0.030%, about 0.031%, about 0.032%, about 0.033%, about 0.034%, about 0.035%, about 0.036%, about 0.037%, about 0.038%, about 0.039%, about 0.040%, about 0.041%, about 0.042%, about 0.043%, about 0.044%, about 0.045%, about 0.046%, about 0.047%, about 0.048%, about 0.049%, about 0.050%, about 0.055%, about 0.060%, about 0.065%, about 0.070%, about 0.075%, about 0.080%, about 0.085%, about 0.090%, about 0.095%, about 0.100%, about 0.110%, about 0.120%, about 0.130%, about 0.140%, or about 0.150% Ti. In some cases, Ti is not present in the alloy (i.e., 0%). All are expressed in wt. %.

**[0034]** In some cases, the aluminum alloy includes one or more elements selected from the group consisting of Y, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu in an amount of up to 0.10% (e.g., from 0.01% to 0.10%, from 0.01% to 0.05%, or from 0.03% to 0.05%), based on the total weight of the alloy. For example, the aluminum alloy can include about 0.01%, about 0.02%, about 0.03%, about 0.04%, about 0.05%, about 0.06%, about 0.07%, about 0.08%, about 0.09%, or about 0.10% of one or more elements selected from the group consisting of Y, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu. All are expressed in wt. %.

**[0035]** In some cases, the aluminum alloy includes one or more elements selected from the group consisting of Mo, Nb, Be, B, Co, Sn, Sr, V, In, Hf, Ag, Sc, and Ni in an amount of up to 0.10% (e.g., from 0.01% to 0.10%, from 0.01% to 0.05%, or from 0.03% to 0.05%), based on the total weight of the alloy. For example, the aluminum alloy can include about 0.01%, about 0.02%, about 0.03%, about 0.04%, about 0.05%, about 0.06%, about 0.07%, about 0.08%, about 0.09%, or about 0.10% of one or more elements selected from the group consisting of Mo, Nb, Be, B, Co, Sn, Sr, V, In, Hf, Ag, Sc, and Ni. All are expressed in wt. %.

**[0036]** In some cases, the aluminum alloy includes other minor elements, sometimes referred to as impurities, in amounts of 0.15% or below, 0.14% or below, 0.13% or below, 0.12% or below, 0.11% or below, 0.10% or below, 0.09% or below, 0.08% or below, 0.07% or below, 0.06% or below, 0.05% or below, 0.04% or below, 0.03% or below, 0.02% or below, or 0.01% or below. In some embodiments, these impurities include, but are not limited to, Ga, Ca, Bi, Na, Pb, or combinations thereof. Accordingly, in some embodiments, one or more elements selected from the group consisting of Ga, Ca, Bi, Na, and Pb may be present in the aluminum alloy in amounts of 0.15% or below, 0.14% or below, 0.13% or below, 0.12% or below, 0.11% or below, 0.10% or below, 0.09% or below, 0.08% or below, 0.07% or below, 0.06% or below, 0.05% or below, 0.04% or below, 0.03% or below, 0.02% or below, or 0.01% or below. The sum of all impurities

does not exceed 0.15% (e.g., 0.10%). All expressed in wt. %. The remaining percentage of the alloy is aluminum.

#### Aluminum Alloy Products and Properties Thereof

**[0037]** Through the use of the pre-aging step following the solutionizing, the aluminum alloy products obtained by the claimed method and disclosed herein are able to resist hardening for up to about 6 months, and, during that time, exhibit desirable bendability and formability. By comparison, 7xxx series aluminum alloy products prepared without the pre-aging step begin to age naturally within a few days of their production and quickly harden and become more difficult to bend and form. Thus, the aluminum alloy products disclosed herein exhibit stable and desirable strength and bendability following solutionizing. In certain embodiments, the aluminum alloy products disclosed herein can be easily formed at a temperature as low as those in the range of about 200 °C to 250 °C for up to about six months following their production. In other embodiments, the aluminum alloy products are cold-formable at room temperature for up to six months following their production.

**[0038]** The aluminum alloy products disclosed herein can have any suitable shape or physical configuration. The aluminum alloy products are rolled aluminum alloy products, which are formed by reducing the thickness of the material using a series of rollers. Such rolling can be carried out by hot rolling, cold rolling, or any combination thereof. In some such embodiments, the rolled aluminum alloy product is an aluminum alloy sheet or an aluminum alloy shate. Such shates or sheets can have a thickness of no more than 15 mm, no more than 14 mm, no more than 13 mm, no more than 12 mm, no more than 11 mm, no more than 10 mm, no more than 9 mm, no more than 8 mm, no more than 7 mm, no more than 6 mm, no more than 5 mm, no more than 4 mm, no more than 3 mm, no more than 2 mm, no more than 1 mm, no more than 0.5 mm, no more than 0.3 mm, or no more than 0.1 mm.

**[0039]** In embodiments where the aluminum alloy product is a sheet, it is possible to measure certain strength and bendability characteristics of the sheet. For example, in some embodiments, such an aluminum alloy sheet has a yield strength (Rp), measured according to the ISO 6892-1 test, of at least 100 MPa, at least 120 MPa, at least 140 MPa, at least 160 MPa, at least 180 MPa, at least 200 MPa, at least 220 MPa, at least 240 MPa, at least 260 MPa, at least 280 MPa, at least 300 MPa, at least 320 MPa, at least 340 MPa, and up to about 360 MPa, up to about 380 MPa, or up to about 400 MPa, immediately following pre-aging. In some further embodiments, such aluminum alloy sheets exhibit a "stable strength," which, for example, means that the yield strength (Rp), measured according to the ISO 6892-1 test, of the aluminum alloy sheet increases by no more than 25 MPa, no more than 20 MPa, no more than 15 MPa, no more than MPa, or no more than 5 MPa during a post-production period (i.e., the period immediately fol-

lowing the pre-aging). The post-production period generally ranges from about 7 days to about 180 days, from about 14 days to about 180 days, from about 21 days to about 180 days, from about 90 days to about 180 days, or from about 120 days to about 180 days. In some embodiments, the post-production period is about 7 days, about 14 days, about 21 days, about 30 days, about 40 days, about 50 days, about 60 days, about 70 days, about 80 days, about 90 days, about 100 days, about 110 days, about 120 days, about 130 days, about 140 days, about 150 days, about 160 days, about 170 days, or about 180 days.

#### Methods of Making Aluminum Alloy Products

**[0040]** The disclosed aluminum alloy products are the products of the disclosed method. Without intending to limit the scope of the inventions set forth herein, the properties of the aluminum alloy products set forth herein are partially determined by the formation of certain microstructures during the preparation thereof. In certain embodiments, the method of preparation can influence certain resulting properties in the aluminum alloy product.

**[0041]** The aluminum alloy products disclosed herein can be prepared and processed by, for example, casting, homogenization, rolling, solutionizing, and quenching. The aluminum alloy products described herein is also pre-aged and coiled.

#### Casting

**[0042]** The methods disclosed herein comprise a step of casting a molten aluminum alloy to provide an aluminum alloy cast product. Such aluminum alloy cast products can be provided using any casting process. For example, in some embodiments, the alloys are cast using a direct chill (DC) casting process to provide a cast ingot. In some other embodiments, the alloys are cast using a continuous casting (CC) process that may include, but is not limited to, the use of twin-belt casters, twin-roll casters, or block casters. In some embodiments, the casting process is performed by a CC process to provide a cast product in the form of a billet, a plate, a shate, a strip, and the like. In some embodiments, the molten alloy may be treated before casting. The treatment can include one or more of degassing, inline fluxing, and filtering.

**[0043]** The cast product can then be subjected to further processing steps, as described in further detail below. In some embodiments, the processing steps can be used to prepare aluminum alloy sheets. The processing steps can be suitably applied to any cast product, including, but not limited to, ingots, billets, plates, strips, etc., using modifications and techniques as known to those of ordinary skill in the art.

#### Homogenization

**[0044]** The homogenization step can include heating

a cast aluminum alloy product prepared from an alloy composition described herein to attain a peak metal temperature (PMT) of at least 450 °C (e.g., at least 450 °C, at least 460 °C, at least 470 °C, at least 480 °C, at least 490 °C, at least 500 °C, at least 510 °C, at least 520 °C, at least 530 °C, at least 540 °C, at least 550 °C, at least 560 °C, at least 570 °C, or at least 580 °C). For example, the cast aluminum alloy product can be heated to a temperature of from 460 °C to 640 °C, from 480 °C to 620 °C, from 500 °C to 600 °C, from 520 °C to 580 °C, from 530 °C to 575 °C, from 535 °C to 570 °C, from 540 °C to 565 °C, from 545 °C to 560 °C, from 530 °C to 560 °C, or from 550 °C to 580 °C. In some embodiments of any of the foregoing embodiments, the heating rate to the PMT is 100 °C/hour or less, 75 °C/hour or less, 50 °C/hour or less, 40 °C/hour or less, 30 °C/hour or less, 25 °C/hour or less, 20 °C/hour or less, or 15 °C/hour or less. In some other such embodiments, the heating rate to the PMT is from 10 °C/min to 100 °C/min (e.g., from 10 °C/min to 90 °C/min, from 10 °C/min to 70 °C/min, from 10 °C/min to 60 °C/min, from 20 °C/min to 90 °C/min, from 30 °C/min to 80 °C/min, from 40 °C/min to 70 °C/min, or from 50 °C/min to 60 °C/min).

**[0045]** In most instances, the cast aluminum alloy product is then allowed to soak (i.e., held at the indicated temperature) for a period of time. In some embodiments, the cast aluminum alloy product is allowed to soak for up to 24 hours (e.g., from 30 minutes to 6 hours, inclusively). For example, in some embodiments, the cast aluminum alloy product is soaked at a temperature of at least 450 °C for about 30 minutes, for about 1 hour, for about 2 hours, for about 3 hours, for about 4 hours, for about 5 hours, for about 6 hours, for about 7 hours, for about 8 hours, for about 9 hours, for about 10 hours, for about 11 hours, for about 12 hours, for about 13 hours, for about 14 hours, for about 15 hours, for about 16 hours, for about 17 hours, for about 18 hours, for about 19 hours, for about 20 hours, for about 21 hours, for about 22 hours, for about 23 hours, for about 24 hours, or for any time period in between.

**[0046]** In some embodiments, following homogenization, the cast aluminum alloy product is allowed to cool to room temperature in the air.

#### Hot Rolling

**[0047]** Following the homogenization step, one or more hot rolling passes are performed. In certain cases, the aluminum alloy products are laid down and hot rolled at a temperature ranging from about 250 °C to about 550 °C (e.g., from about 300 °C to about 500 °C or from about 350 °C to about 450 °C).

**[0048]** In certain embodiments, the aluminum alloy product is hot rolled to an about 4 mm to about 15 mm gauge (e.g., from 5 mm to 12 mm gauge). For example, the aluminum alloy product can be hot rolled to an about 15 mm gauge, an about 14 mm gauge, an about 13 mm gauge, an about 12 mm gauge, an about 11 mm gauge,

an about 10 mm gauge, an about 9 mm gauge, an about 8 mm gauge, an about 7 mm gauge, an about 6 mm gauge, or an about 5 mm gauge.

**[0049]** In other cases, the aluminum alloy product can be hot rolled to a gauge no more than 4 mm (i.e., a sheet). In some such embodiments, the aluminum alloy product is hot rolled to an about 1 mm to an about 4 mm gauge. For example, the aluminum alloy product can be hot rolled to an about 4 mm gauge, an about 3 mm gauge, an about 2 mm gauge, or an about 1 mm gauge.

#### Optional Cold Rolling

**[0050]** Following the hot rolling, one or more cold rolling passes are optionally performed. In certain embodiments, the rolled product from the hot rolling step (e.g., the plate, shate, or sheet) can be cold rolled to a thin gauge shate or sheet. In some embodiments, this thin-gauge shate or sheet is cold rolled to have a thickness up to 12.0 mm, such as a thickness ranging from about 1.0 mm to about 12.0 mm, from about 2.0 mm to about 8.0 mm, from about 3.0 mm to about 6.0 mm, or from about 4.0 mm to about 5.0 mm. In some embodiments, this thin-gauge shate or sheet is cold rolled to have a thickness of about 12.0 mm, about 11.9 mm, about 11.8 mm, about 11.7 mm, about 11.6 mm, about 11.5 mm, about 11.4 mm, about 11.3 mm, about 11.2 mm, about 11.1 mm, about 11.0 mm, about 10.9 mm, about 10.8 mm, about 10.7 mm, about 10.6 mm, about 10.5 mm, about 10.4 mm, about 10.3 mm, about 10.2 mm, about 10.1 mm, about 10.0 mm, about 9.9 mm, about 9.8 mm, about 9.7 mm, about 9.6 mm, about 9.5 mm, about 9.4 mm, about 9.3 mm, about 9.2 mm, about 9.1 mm, about 9.0 mm, about 8.9 mm, about 8.8 mm, about 8.7 mm, about 8.6 mm, about 8.5 mm, about 8.4 mm, about 8.3 mm, about 8.2 mm, about 8.1 mm, about 8.0 mm, about 7.9 mm, about 7.8 mm, about 7.7 mm, about 7.6 mm, about 7.5 mm, about 7.4 mm, about 7.3 mm, about 7.2 mm, about 7.1 mm, about 7.0 mm, about 6.9 mm, about 6.8 mm, about 6.7 mm, about 6.6 mm, about 6.5 mm, about 6.4 mm, about 6.3 mm, about 6.2 mm, about 6.1 mm, about 6.0 mm, about 5.9 mm, about 5.8 mm, about 5.7 mm, about 5.6 mm, about 5.5 mm, about 5.4 mm, about 5.3 mm, about 5.2 mm, about 5.1 mm, about 5.0 mm, about 4.9 mm, about 4.8 mm, about 4.7 mm, about 4.6 mm, about 4.5 mm, about 4.4 mm, about 4.3 mm, about 4.2 mm, about 4.1 mm, about 4.0 mm, about 3.9 mm, about 3.8 mm, about 3.7 mm, about 3.6 mm, about 3.5 mm, about 3.4 mm, about 3.3 mm, about 3.2 mm, about 3.1 mm, about 3.0 mm, about 2.9 mm, about 2.8 mm, about 2.7 mm, about 2.6 mm, about 2.5 mm, about 2.4 mm, about 2.3 mm, about 2.2 mm, about 2.1 mm, about 2.0 mm, about 1.9 mm, about 1.8 mm, about 1.7 mm, about 1.6 mm, about 1.5 mm, about 1.4 mm, about 1.3 mm, about 1.2 mm, about 1.1 mm, about 1.0 mm, about 0.9 mm, about 0.8 mm, about 0.7 mm, about 0.6 mm, about 0.5 mm, about 0.4 mm, about 0.3 mm, about 0.2 mm, or about 0.1 mm.



### Solutionizing

**[0051]** The solutionizing step can include heating the sheet, plate, or shate from room temperature to a temperature of from 430 °C to 510 °C (e.g., from 440 °C to 500 °C, from 450 °C to 490 °C, from 460 °C to 480 °C). The sheet, plate, or shate can soak at the temperature for a period of time. In certain aspects, the alloy is allowed to soak for up to approximately 5 minutes (e.g., from 5 seconds to 5 minutes, inclusively). For example, the sheet, plate, or shate can soak at the temperature of from 430 °C to 510 °C for about 5 seconds, about 10 seconds, about 15 seconds, about 20 seconds, about 25 seconds, about 30 seconds, about 35 seconds, about 40 seconds, about 45 seconds, about 50 seconds, about 55 seconds, about 60 seconds, about 65 seconds, about 70 seconds, about 75 seconds, about 80 seconds, about 85 seconds, about 90 seconds, about 95 seconds, about 100 seconds, about 105 seconds, about 110 seconds, about 115 seconds, about 120 seconds, about 125 seconds, about 130 seconds, about 135 seconds, about 140 seconds, about 145 seconds, about 150 seconds, about 3 minutes, about 4 minutes, or about 5 minutes, or anywhere in between.

### Quenching

**[0052]** In certain embodiments, the plate, shate, or sheet can then be cooled to a temperature of 25 °C to 65 °C at a quench speed that can vary between about 50 °C/s to about 400 °C/s in a quenching step that is based on the selected gauge. For example, the quench rate can be from about 50 °C/s to about 375 °C/s, from about 60 °C/s to about 375 °C/s, from about 70 °C/s to about 350 °C/s, from about 80 °C/s to about 325 °C/s, from about 90 °C/s to about 300 °C/s, from about 100 °C/s to about 275 °C/s, from about 125 °C/s to about 250 °C/s, from about 150 °C/s to about 225 °C/s, or from about 175 °C/s to about 200 °C/s.

**[0053]** In the quenching step, the sheet, plate, or shate is rapidly quenched with a liquid (e.g., water) and/or gas or another selected quench medium. In certain aspects, the sheet, plate, or shate can be rapidly quenched with water. In certain aspects, the sheet, plate, or shate is quenched with a gas or a liquid.

### Pre-Aging (Re-Heating)

**[0054]** The methods disclosed herein generally include a pre-aging step following the solutionizing and quenching steps. The pre-aging step includes heating the alloy after the solutionizing step to a temperature ranging from 60 °C to 130 °C (e.g., from about 65 °C to about 125 °C, from about 70 °C to about 120 °C, from about 75 °C to about 115 °C, from about 80 °C to about 120 °C, or from about 85 °C to about 115 °C). In some examples, the pre-aging step can include heating the alloy after solutionizing from about 80 °C to about 120 °C (e.g., from

about 90 °C to about 110 °C). The pre-aging step is conducted for a period of time up to 24 hours (e.g., for a period of time up to about 20 hours, up to about 15 hours, up to about 12 hours, up to about 10 hours, up to about 9 hours, up to about 8 hours, up to about 7 hours, up to about 6 hours, up to about 5 hours, up to about 4 hours, up to about 3 hours, up to about 2 hours, up to about 1 hours, or up to about 30 minutes). The alloy can soak at the temperature for a period of time. In certain aspects, the alloy is allowed to soak for a period of time up to approximately 2 hours (e.g., for a period of time up to about 1 minute, up to about 2 minutes, up to about 3 minutes, up to about 4 minutes, up to about 5 minutes, up to about 6 minutes, up to about 7 minutes, up to about 8 minutes, up to about 9 minutes, up to about 10 minutes, up to about 20 minutes, up to about 30 minutes, up to about 40 minutes, up to about 45 minutes, up to about 60 minutes, or up to about 90 minutes). The time between the post-solutionizing quench and the pre-aging can be any length of time ranging from about 0 minutes up to about 60 minutes. For example, the time between the post-solutionizing quench and the pre-aging can be any length of time ranging from about 5 minutes up to about 45 minutes or from about 10 minutes up to about 35 minutes.

**[0055]** Following the heating of the aluminum alloy product in the pre-aging step, the heated product is generally cooled from the peak pre-aging temperature back to room temperature slowly and without the use of quenching the product with a gas or a liquid. In some other embodiments, however, the cooling to room temperature is assisted by forced cooling using, for example, air, a cool liquid, and the like, or any combination thereof. In some embodiments, the cooling from the peak pre-aging temperature back to room temperature occurs over the course of about 48 hours, about 36 hours, about 24 hours, about 18 hours, about 12 hours, or for any range of time in between. The aluminum alloy product can be cooled from the pre-aging temperature to room temperature in any suitable physical configuration. In some embodiments, the aluminum alloy product is coiled at the pre-aging temperature (or at a temperature no more than 5 °C below the pre-aging temperature), and cooled to room temperature over the course of about 48 hours, about 36 hours, about 24 hours, about 18 hours, about 12 hours, or for any range of time in between.

**[0056]** Following the cooling, the aluminum alloy product is in a form that is ready for delivery, and suitable for use in various cold forming and warm forming processes. In this state, the aluminum alloy product in a stable T4 temper, which is retained for a period of up to about 6 months, after which point the formed material ages and hardens.

### Articles of Manufacture

**[0057]** The disclosure provides an article of manufacture, which is comprised of a 7xxx series aluminum alloy

product obtained according to the claimed method and disclosed herein. The article of manufacture comprises a rolled aluminum alloy product, such as a rolled aluminum alloy sheet. Examples of such articles of manufacture include, but are not limited to, an automobile, a truck, a trailer, a train, a railroad car, an airplane, a body panel or part for any of the foregoing, a bridge, a pipeline, a pipe, a tubing, a boat, a ship, a storage container, a storage tank, an article of furniture, a window, a door, a railing, a functional or decorative architectural piece, a pipe railing, an electrical component, a conduit, a beverage container, or a food container.

**[0058]** In some other embodiments, the aluminum alloy products disclosed herein can be used in automotive and/or transportation applications, including motor vehicle, aircraft, and railway applications, or any other desired application. In some examples, the aluminum alloy products disclosed herein can be used to prepare motor vehicle body part products, such as bumpers, side beams, roof beams, cross beams, pillar reinforcements (e.g., A-pillars, B-pillars, and C-pillars), inner panels, outer panels, side panels, inner hoods, outer hoods, or trunk lid panels. The aluminum alloys and methods described herein can also be used in aircraft or railway vehicle applications, to prepare, for example, external and internal panels.

**[0059]** In some other embodiments, the aluminum alloy products disclosed herein can be used in electronics applications. For example, the aluminum alloy products disclosed herein can also be used to prepare housings for electronic devices, including mobile phones and tablet computers. In some examples, the alloys can be used to prepare housings for the outer casing of mobile phones (e.g., smart phones) and tablet bottom chassis.

**[0060]** In some other embodiments, the aluminum alloy products disclosed herein can be used in industrial applications. For example, the aluminum alloy products disclosed herein can be used to prepare products for the general distribution market.

**[0061]** In some other embodiments, the aluminum alloy products disclosed herein can be used as aerospace body parts. For example, the aluminum alloy products disclosed herein can be used to prepare structural aerospace body parts, such as a wing, a fuselage, an aileron, a rudder, an elevator, a cowl, or a support. In some other embodiments, the aluminum alloy products disclosed herein can be used to prepare non-structural aerospace body parts, such as a seat track, a seat frame, a panel, or a hinge.

**[0062]** The following examples serve to further illustrate certain embodiments of the present disclosure without, at the same time, however, constituting any limitation thereof. On the contrary, it is to be clearly understood that resort may be had to various embodiments, modifications and equivalents thereof which, after reading the description herein, may suggest themselves to those of ordinary skill in the art without departing from the spirit of the disclosure.

## EXAMPLE 1 - Strength Testing

**[0063]** Two samples of AA7075 aluminum alloy sheets of 1.4 mm thickness were prepared according to identical processing methods, including solutionizing at 480 °C followed by a full water quench, except for the pre-aging step. One sample was subjected to pre-aging (PX) at 100 °C for less than 1 minute, and then cooled to room temperature over 24 hours. The other sample did not undergo pre-aging (comparative). FIG. 1 shows the change in the yield strength (Rp) as a function of the number of days following initial production for each of the samples, where the yield strength is measured according to the ISO 6892-1 test. The sample prepared with pre-aging shows a significantly more stable yield strength over a period of about 30 days.

**[0064]** Six samples of clad AA7075/AA5182 aluminum alloy sheets of 2.0 mm thickness were prepared according to methods as described herein, with variations only in the pre-aging step. Certain samples were subjected to pre-aging at different temperatures, and some samples were subjected to pre-aging in combination with simulated coil cooling. FIG. 2 shows the change in the yield strength (Rp) as a function of the number of days following initial production for each of the samples.

## EXAMPLE 2

**[0065]** Samples of AA7075 aluminum alloy sheets of 1.4 mm thickness were prepared according to identical processing methods, including solutionizing at 480 °C with a five minute soak time, a full water quench at a quench rate of 350 °C/s, a pre-aging step, and natural aging. The pre-aging temperature and time were varied, as was the natural aging time. The pre-aging time was either 1 hour, 4 hours, or 8 hours. The pre-aging temperature was either 70 °C or 100 °C. Natural aging (NA) was conducted for 1 week, 2 weeks, 3 weeks, or 4 weeks. The samples were tested with and without being subjected to a paint-bake cycle (PB). When subjected to a paint-bake cycle, the samples were tested with and without 2% pre-strain FIGS. 3A-D show the change in the yield strength for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. FIG 3D shows the effect of pre-strain when the samples were subjected to paint-baking. The yield strength (MPa) was measured according to the ISO 6892-1:2016 test. FIGS. 4A-C show the change in elongation strength for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. FIG 4D shows the effect of pre-strain when the samples were subjected to paint-baking. The elongation strength (MPa) was measured according to the ISO 6892-1:2016 test. FIGS. 5A-C show the change in uniform elongation for the sample as a function of the pre-aging time and temperature and as a function

of whether the samples were subjected to a paint-bake cycle. FIG 5D shows the effect of pre-strain when the samples were subjected to paint-baking. The uniform elongation (%) was measured according to the ISO 6892-1:2016 test. FIGS. 6A-C show the change in total elongation for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. FIG 6D shows the effect of pre-strain when the samples were subjected to paint-baking. The total elongation (%) was measured according to ISO 6892-1:2016 test. FIGS. 7A-D show the change in critical fracture strain for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. FIGS 7C and D show the effect of pre-strain when the samples were subjected to paint-baking. The critical fracture strain (%) was measured according to the ISO 6892-1:2016 test. FIGS. 8A-D show the strain hardening exponent (n-value) for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. FIGS 8C and D show the effect of pre-strain when the samples were subjected to paint-baking. The critical fracture strain (%) was measured according to the ISO 6892-1:2016 test.

**[0066]** As shown by FIGS. 3-8, pre-aging at 70 °C resulted in increasing yield strength as the length of natural aging increased. For pre-aging at 100 °C, the yield strength was relatively stable when pre-aged for 4 to 8 hours, regardless of the length of time of natural aging. Yield strengths of greater than 450 MPa were achievable after the samples were subjected to a paint-bake cycle. The samples subjected to a 2% pre-strain prior to the paint-bake cycle showed a slight increase in yield strength.

**[0067]** Various embodiments of the invention have been described in fulfillment of the various objectives of the invention. It should be recognized that these embodiments are merely illustrative of the principles of the present invention. Numerous modifications and adaptations thereof will be readily apparent to those of ordinary skill in the art without departing from the invention as defined in the following claims.

**[0068]** Samples of AA7075 aluminum alloy sheets of 1.4 mm thickness were prepared according to identical processing methods, including solutionizing at 480 °C with a five minute soak time, a full water quench at a quench rate of 350 °C/s, a pre-aging step, and natural aging. The pre-aging temperature and time were varied, as was the natural aging time. The pre-aging time was either 1 hours, 4 hours, or 8 hours. The pre-aging temperature was either 70 °C or 100 °C. Natural aging (NA) was conducted for 1 week, 2 weeks, 3 weeks, or 4 weeks. The samples were tested with and without being subjected to a paint-bake cycle (PB). When subjected to a paint-bake cycle, the samples were tested with and without 2% pre-strain FIGS. 3A-C show the change in the yield strength for the sample as a function of the pre-aging

time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. The yield strength (MPa) was measured according to the ISO 6892-1:2016 test. FIGS. 4A-C show the change in elongation strength for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. The elongation strength (MPa) was measured according to the ISO 6892-1:2016 test. FIGS. 5A-C show the change in uniform elongation for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. The uniform elongation (%) was measured according to the ISO 6892-1:2016 test. FIGS. 6A-C show the change in total elongation for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. The total elongation (%) was measured according to ISO 6892-1:2016 test. FIGS. 7A-D show the change in critical fracture strain for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. FIGS 7C and D show the effect of pre-strain when the samples were subjected to paint-baking. The critical fracture strain (%) was measured according to the ISO 6892-1:2016 test. FIGS. 8A-D show the strain hardening exponent (n-value) for the sample as a function of the pre-aging time and temperature and as a function of whether the samples were subjected to a paint-bake cycle. FIGS 8C and D show the effect of pre-strain when the samples were subjected to paint-baking. The critical fracture strain (%) was measured according to the ISO 6892-1:2016 test.

**[0069]** As shown by FIGS. 3-8, pre-aging at 70 °C resulted in increasing yield strength as the length of natural aging increased. For pre-aging at 100 °C, the yield strength was relatively stable when pre-aged for 4 to 8 hours, regardless of the length of time of natural aging. Yield strengths of greater than 450 MPa were achievable after the samples were subjected to a paint-bake cycle. The samples subjected to a 2% pre-strain prior to the paint-bake cycle showed a slight increase in yield strength. Various embodiments of the invention have been described in fulfillment of the various objectives of the invention. It should be recognized that these embodiments are merely illustrative of the principles of the present invention. Numerous modifications and adaptations thereof will be readily apparent to those of ordinary skill in the art.

## Claims

1. A method of making a rolled aluminum alloy product, comprising:

providing a 7xxx series aluminum alloy, wherein the 7xxx series aluminum alloy is provided as a

- molten 7xxx series aluminum alloy;  
 casting the molten 7xxx series aluminum alloy  
 to provide an aluminum alloy cast product;  
 homogenizing the aluminum alloy cast product  
 to provide a homogenized aluminum alloy cast  
 product;  
 rolling the homogenized aluminum alloy cast  
 product to form a rolled aluminum alloy product;  
 solutionizing and pre-aging the rolled aluminum  
 alloy product,  
 wherein the pre-aging comprises heating the  
 rolled aluminum alloy product to a pre-aging  
 temperature ranging from 60 °C to 130 °C,  
 wherein the pre-aging is conducted for a period  
 of time up to 24 hours; and  
 coiling the rolled aluminum alloy product after  
 the pre-aging.
2. The method of claim 1, wherein the 7xxx series alu-  
 minum alloy is selected from the group consisting of  
 AA7011, AA7019, AA7020, AA7021, AA7039,  
 AA7072, AA7075, AA7085, AA7108, AA7108A,  
 AA7015, AA7017, AA7018, AA7019A, AA7024,  
 AA7025, AA7028, AA7030, AA7031, AA7033,  
 AA7035, AA7035A, AA7046, AA7046A, AA7003,  
 AA7004, AA7005, AA7009, AA7010, AA7011,  
 AA7012, AA7014, AA7016, AA7116, AA7122,  
 AA7023, AA7026, AA7029, AA7129, AA7229,  
 AA7032, AA7033, AA7034, AA7036, AA7136,  
 AA7037, AA7040, AA7140, AA7041, AA7049,  
 AA7049A, AA7149, AA7204, AA7249, AA7349,  
 AA7449, AA7050, AA7050A, AA7150, AA7250,  
 AA7055, AA7155, AA7255, AA7056, AA7060,  
 AA7064, AA7065, AA7068, AA7168, AA7175,  
 AA7475, AA7076, AA7178, AA7278, AA7278A,  
 AA7081, AA7181, AA7185, AA7090, AA7093,  
 AA7095, and AA7099.
3. The method of claim 1, wherein the 7xxx series alu-  
 minum alloy comprises:
- from 4.0 to 15.0 wt. % Zn;  
 from 0.1 to 3.5 wt % Cu;  
 from 1.0 to 4.0 wt. % Mg;  
 from 0.05 to 0.50 wt. % Fe;  
 from 0.05 to 0.30 wt. % Si;  
 up to 0.50 wt. % Zr;  
 up to 0.25 wt. % Mn;  
 up to 0.20 wt. % Cr;  
 up to 0.15 wt. % Ti; and  
 up to 0.15 wt. % impurities;
- with the remainder being Al.
4. The method of any of claims 1 or 3, wherein the 7xxx  
 series aluminum alloy comprises:
- from 5.6 to 9.3 wt. % Zn;
- from 0.2 to 2.6 wt. % Cu;  
 from 1.4 to 2.8 wt. % Mg;  
 from 0.10 to 0.35 wt. % Fe;  
 from 0.05 to 0.20 wt. % Si;  
 up to 0.25 wt. % Zr;  
 up to 0.05 wt. % Mn;  
 up to 0.05 wt. % Cr;  
 up to 0.05 wt. % Ti; and  
 up to 0.15 wt. % impurities;
- with the remainder being Al.
5. The method of claim 1, wherein the 7xxx series alu-  
 minum alloy comprises:
- from 4.0 to 15.0 wt. % Zn;  
 from 0.1 to 3.5 wt % Cu;  
 from 1.0 to 4.0 wt. % Mg;  
 from 0.05 to 0.50 wt. % Fe;  
 from 0.05 to 0.30 wt. % Si;  
 up to 0.50 wt. % Zr;  
 up to 0.25 wt. % Mn;  
 up to 0.20 wt. % Cr;  
 up to 0.15 wt. % Ti;  
 up to 0.10 wt. % of one or more elements se-  
 lected from the group consisting of Mo, Nb, Be,  
 B, Co, Sn, Sr, V, In, Hf, Ag, Sc, Ni, Y, La, Ce,  
 Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm,  
 Yb, and Lu; and  
 up to 0.15 wt. % impurities;
- with the remainder being Al.
6. The method of any of claims 1-5, wherein the rolled  
 aluminum alloy product is an aluminum alloy sheet  
 or an aluminum alloy shate and in particular,  
 wherein the rolled aluminum alloy product has a  
 thickness of no more than 15 mm.
7. The method of any of claims 1-6, wherein the casting  
 comprises direct chill (DC) casting or continuous  
 casting and/or,  
 wherein the rolling comprises hot rolling, cold rolling,  
 or any combination thereof.
8. The method of any of claims 1-7, further comprising  
 quenching the rolled aluminum alloy product after  
 the solutionizing and preceding the pre-aging.
9. The method of any of claims 1-8, further comprising,  
 after the pre-aging, cooling the rolled aluminum alloy  
 product to about room temperature over a period of  
 time ranging from 12 hours to 48 hours.
10. The method of any of claims 1-9, further comprising,  
 after the pre-aging and coiling the rolled aluminum  
 alloy product, then, cooling the rolled aluminum alloy  
 product to room temperature.

11. The method of claim 10, wherein the coiling is carried out at a temperature of no more than 5 °C below the pre-aging temperature and in particular, wherein the cooling is carried out over a period of time ranging from 12 hours to 48 hours.
12. The method of any of claims 1-11, wherein the rolled aluminum alloy product has a yield strength ( $R_p$ ) of at least 240 MPa immediately following pre-aging, wherein the yield strength ( $R_p$ ) is measured according to ISO 6892-1 and/or, wherein the rolled aluminum alloy product exhibits an increase in its yield strength ( $R_p$ ) of no more than 25 MPa during a post-production period immediately following the pre-aging, wherein the post-production period ranges from 7 days to 180 days.

### Patentansprüche

1. Verfahren zur Herstellung eines gewalzten Aluminiumlegierungs-Produkts, umfassend:

Bereitstellen einer Aluminiumlegierung der Serie 7xxx, wobei die Aluminiumlegierung der Serie 7xxx als eine geschmolzene Aluminiumlegierung der Serie 7xxx bereitgestellt wird;  
Gießen der geschmolzenen Aluminiumlegierung der Serie 7xxx, um ein Aluminiumlegierungs-Gussprodukt bereitzustellen;  
Homogenisieren des Aluminiumlegierungs-Gussprodukts, um ein homogenisiertes Aluminiumlegierungs-Gussprodukt bereitzustellen;  
Walzen des homogenisierten Aluminiumlegierungs-Gussprodukts, um ein gewalztes Aluminiumlegierungs-Produkt zu bilden;  
Lösungsglühen und Voraltern des gewalzten Aluminiumlegierungs-Produkts, wobei die Voralterung das Erhitzen des gewalzten Aluminiumlegierungs-Produkts auf eine Voralterungstemperatur im Bereich von 60 °C bis 130 °C umfasst, wobei die Voralterung über eine Zeitspanne von bis zu 24 Stunden durchgeführt wird; und  
Aufwickeln des gewalzten Aluminiumlegierungs-Produkts nach der Voralterung.

2. Verfahren nach Anspruch 1, wobei die Aluminiumlegierung der Serie 7xxx ausgewählt ist aus der Gruppe bestehend aus AA7011, AA7019, AA7020, AA7021, AA7039, AA7072, AA7075, AA7085, AA7108, AA7108A, AA7015, AA7017, AA7018, AA7019A, AA7024, AA7025, AA7028, AA7030, AA7031, AA7033, AA7035, AA7035A, AA7046, AA7046A, AA7003, AA7004, AA7005, AA7009, AA7010, AA7011, AA7012, AA7014, AA7016, AA7116, AA7122, AA7023, AA7026, AA7029, AA7129, AA7229, AA7032, AA7033, AA7034,

AA7036, AA7136, AA7037, AA7040, AA7140, AA7041, AA7049, AA7049A, AA7149, AA7204, AA7249, AA7349, AA7449, AA7050, AA7050A, AA7150, AA7250, AA7055, AA7155, AA7255, AA7056, AA7060, AA7064, AA7065, AA7068, AA7168, AA7175, AA7475, AA7076, AA7178, AA7278, AA7278A, AA7081, AA7181, AA7185, AA7090, AA7093, AA7095 und AA7099.

3. Verfahren nach Anspruch 1, wobei die Aluminiumlegierung der Serie 7xxx umfasst:

von 4,0 bis 15,0 Gew.-% Zn;  
von 0,1 bis 3,5 Gew.-% Cu;  
von 1,0 bis 4,0 Gew.-% Mg;  
von 0,05 bis 0,50 Gew.-% Fe;  
von 0,05 bis 0,30 Gew.-% Si;  
bis zu 0,50 Gew.-% Zr;  
bis zu 0,25 Gew.-% Mn;  
bis zu 0,20 Gew.-% Cr;  
bis zu 0,15 Gew.-% Ti; und  
bis zu 0,15 Gew.-% Verunreinigungen; wobei der Rest Al ist.

4. Verfahren nach einem der Ansprüche 1 oder 3, wobei die Aluminiumlegierung der Serie 7xxx umfasst:

von 5,6 bis 9,3 Gew.-% Zn;  
von 0,2 bis 2,6 Gew.-% Cu;  
von 1,4 bis 2,8 Gew.-% Mg;  
von 0,10 bis 0,35 Gew.-% Fe;  
von 0,05 bis 0,20 Gew.-% Si;  
bis zu 0,25 Gew.-% Zr;  
bis zu 0,05 Gew.-% Mn;  
bis zu 0,05 Gew.-% Cr;  
bis zu 0,05 Gew.-% Ti; und  
bis zu 0,15 Gew.-% Verunreinigungen; wobei der Rest Al ist.

5. Verfahren nach Anspruch 1, wobei die Aluminiumlegierung der Serie 7xxx umfasst:

von 4,0 bis 15,0 Gew.-% Zn;  
von 0,1 bis 3,5 Gew.-% Cu;  
von 1,0 bis 4,0 Gew.-% Mg;  
von 0,05 bis 0,50 Gew.-% Fe;  
von 0,05 bis 0,30 Gew.-% Si;  
bis zu 0,50 Gew.-% Zr;  
bis zu 0,25 Gew.-% Mn;  
bis zu 0,20 Gew.-% Cr;  
bis zu 0,15 Gew.-% Ti;  
bis zu 0,10 Gew.-% eines oder mehrerer Elemente, ausgewählt aus der Gruppe bestehend aus Mo, Nb, Be, B, Co, Sn, Sr, V, In, Hf, Ag, Sc, Ni, Y, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb und Lu; und  
bis zu 0,15 Gew.-% Verunreinigungen; wobei der Rest Al ist.

6. Verfahren nach einem der Ansprüche 1 bis 5, wobei das gewalzte Aluminiumlegierungs-Produkt ein Aluminiumlegierungs-Blech oder eine Aluminiumlegierungs-Shate ist und insbesondere, wobei das gewalzte Aluminiumlegierungs-Produkt eine Dicke von nicht mehr als 15 mm hat. 5
7. Verfahren nach einem der Ansprüche 1 bis 6, wobei das Gießen direktes Kokillen-(direct chill - DC-) Gießen oder Stranggießen umfasst und/oder wobei das Walzen Warmwalzen, Kaltwalzen oder eine Kombination davon umfasst. 10
8. Verfahren nach einem der Ansprüche 1 bis 7 ferner umfassend das Quenchen des gewalzten Aluminiumlegierungs-Produkts nach dem Lösungsglühen und vor der Voralterung. 15
9. Verfahren nach einem der Ansprüche 1 bis 8, ferner umfassend, nach der Voralterung, das Abkühlen des gewalzten Aluminiumlegierungs-Produkts auf etwa Raumtemperatur über einen Zeitraum von 12 bis 48 Stunden. 20
10. Verfahren nach einem der Ansprüche 1 bis 9 ferner umfassend, nach der Voralterung und dem Wickeln des gewalzten Aluminiumlegierungs-Produkts, das Abkühlen des gewalzten Aluminiumlegierungs-Produkts auf Raumtemperatur. 25
11. Verfahren nach Anspruch 10, wobei das Wickeln bei einer Temperatur von nicht mehr als 5 °C unter der Voralterungstemperatur durchgeführt wird und insbesondere wobei das Abkühlen über einen Zeitraum von 12 Stunden bis 48 Stunden durchgeführt wird. 30
12. Verfahren nach einem der Ansprüche 1-11, wobei das gewalzte Aluminiumlegierungs-Produkt eine Streckgrenze ( $R_p$ ) von mindestens 240 MPa unmittelbar nach der Voralterung aufweist, wobei die Streckgrenze ( $R_p$ ) gemäß ISO 6892-1 gemessen wird, und/oder wobei das gewalzte Aluminiumlegierungs-Produkt einen Anstieg seiner Streckgrenze ( $R_p$ ) von nicht mehr als 25 MPa während einer Postproduktionsperiode unmittelbar nach der Voralterung aufweist, wobei die Postproduktionsperiode im Bereich von 7 Tagen bis 180 Tagen liegt. 35
- Revendications** 40
1. Procédé de fabrication d'un produit en alliage d'aluminium laminé, consistant à : 45
- fournir un alliage d'aluminium de la série 7xxx, l'alliage d'aluminium de la série 7xxx étant fourni sous forme d'alliage d'aluminium de la série 7xxx en fusion ; 50
- couler l'alliage d'aluminium de la série 7xxx en fusion pour obtenir un produit coulé en alliage d'aluminium ;  
homogénéiser le produit coulé en alliage d'aluminium pour obtenir un produit coulé en alliage d'aluminium homogénéisé ;  
laminer le produit coulé en alliage d'aluminium homogénéisé pour former un produit en alliage d'aluminium laminé ;  
mettre en solution et faire pré-vieillir le produit en alliage d'aluminium laminé,  
le pré-vieillissement consistant à chauffer le produit en alliage d'aluminium laminé à une température de pré-vieillissement comprise entre 60 °C et 130 °C,  
le pré-vieillissement étant effectué pendant une période de temps pouvant aller jusqu'à 24 heures ; et  
enrouler le produit en alliage d'aluminium laminé après le pré-vieillissement.
2. Procédé selon la revendication 1, dans lequel l'alliage d'aluminium de la série 7xxx est choisi dans le groupe constitué par AA7011, AA7019, AA7020, AA7021, AA7039, AA7072, AA7075, AA7085, AA7108, AA7108A, AA7015, AA7017, AA7018, AA7019A, AA7024, AA7025, AA7028, AA7030, AA7031, AA7033, AA7035, AA7035A, AA7046, AA7046A, AA7003, AA7004, AA7005, AA7009, AA7010, AA7011, AA7012, AA7014, AA7016, AA7116, AA7122, AA7023, AA7026, AA7029, AA7129, AA7229, AA7032, AA7033, AA7034, AA7036, AA7136, AA7037, AA7040, AA7140, AA7041, AA7049, AA7049A, AA7149, AA7204, AA7249, AA7349, AA7449, AA7050, AA7050A, AA7150, AA7250, AA7055, AA7155, AA7255, AA7056, AA7060, AA7064, AA7065, AA7068, AA7168, AA7175, AA7475, AA7076, AA7178, AA7278, AA7278A, AA7081, AA7181, AA7185, AA7090, AA7093, AA7095 et AA7099.
3. Procédé selon la revendication 1, dans lequel l'alliage d'aluminium de la série 7xxx comprend :
- de 4,0 à 15,0 % en poids de Zn ;  
de 0,1 à 3,5 % en poids de Cu ;  
de 1,0 à 4,0 % en poids de Mg ;  
de 0,05 à 0,50 % en poids de Fe ;  
de 0,05 à 0,30 % en poids de Si ;  
jusqu'à 0,50 % en poids de Zr ;  
jusqu'à 0,25 % en poids de Mn ;  
jusqu'à 0,20 % en poids de Cr ;  
jusqu'à 0,15 % en poids de Ti ; et  
jusqu'à 0,15 % en poids d'impuretés ;  
le reste étant constitué d'Al.

4. Procédé selon l'une des revendications 1 à 3, dans lequel l'alliage d'aluminium de la série 7xxx comprend :

de 5,6 à 9,3 % en poids de Zn ;  
de 0,2 à 2,6 % en poids de Cu ;  
de 1,4 à 2,8 % en poids de Mg ;  
de 0,10 à 0,35 % en poids de Fe ;  
de 0,05 à 0,20 % en poids de Si ;  
jusqu'à 0,25 % en poids de Zr ;  
jusqu'à 0,05 % en poids de Mn ;  
jusqu'à 0,05 % en poids de Cr ;  
jusqu'à 0,05 % en poids de Ti ; et  
jusqu'à 0,15 % en poids d'impuretés ;  
le reste étant constitué d'Al.

5. Procédé selon la revendication 1, dans lequel l'alliage d'aluminium de la série 7xxx comprend :

de 4,0 à 15,0 % en poids de Zn ;  
de 0,1 à 3,5 % en poids de Cu ;  
de 1,0 à 4,0 % en poids de Mg ;  
de 0,05 à 0,50 % en poids de Fe ;  
de 0,05 à 0,30 % en poids de Si ;  
jusqu'à 0,50 % en poids de Zr ;  
jusqu'à 0,25 % en poids de Mn ;  
jusqu'à 0,20 % en poids de Cr ;  
jusqu'à 0,15 % en poids de Ti ;  
jusqu'à 0,10 % en poids d'un ou plusieurs éléments choisis dans le groupe constitué de Mo, Nb, Be, B, Co, Sn, Sr, V, In, Hf, Ag, Sc, Ni, Y, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, et Lu ; et  
jusqu'à 0,15 % en poids d'impuretés ;  
le reste étant constitué d'Al.

6. Procédé selon l'une des revendications 1 à 5,

dans lequel le produit en alliage d'aluminium laminé est une feuille en alliage d'aluminium ou une plaque-tôle en alliage d'aluminium, et en particulier le produit en alliage d'aluminium laminé a une épaisseur maximale de 15 mm.

7. Procédé selon l'une des revendications 1 à 6,

dans lequel la coulée comprend la coulée à refroidissement direct (DC) ou la coulée continue, et/ou le laminage comprend le laminage à chaud, le laminage à froid ou toute combinaison de ceux-ci.

8. Procédé selon l'une des revendications 1 à 7, comprenant en outre la trempe du produit en alliage d'aluminium laminé après la mise en solution et

avant le pré-vieillissement.

9. Procédé selon l'une des revendications 1 à 8, comprenant en outre, après le pré-vieillissement, le refroidissement du produit en alliage d'aluminium laminé jusqu'à la température ambiante sur une période de temps allant de 12 à 48 heures.

10. Procédé selon l'une des revendications 1 à 9, comprenant en outre, après le pré-vieillissement et l'enroulement du produit en alliage d'aluminium laminé, le refroidissement du produit en alliage d'aluminium laminé à la température ambiante.

11. Procédé selon la revendication 10,

dans lequel l'enroulement est effectué à une température qui n'est pas inférieure de plus de 5 °C à la température de pré-vieillissement, et en particulier le refroidissement est effectué sur une période de temps allant de 12 heures à 48 heures.

12. Procédé selon l'une des revendications 1 à 11,

dans lequel le produit en alliage d'aluminium laminé a une limite d'élasticité ( $R_p$ ) d'au moins 240 MPa immédiatement après le pré-vieillissement, la limite d'élasticité ( $R_p$ ) étant mesurée conformément à la norme ISO 6892-1, et/ou le produit en alliage d'aluminium laminé présente une augmentation de sa limite d'élasticité ( $R_p$ ) ne dépassant pas 25 MPa au cours d'une période de post-production suivant immédiatement le pré-vieillissement, la période de post-production étant comprise entre 7 jours et 180 jours.

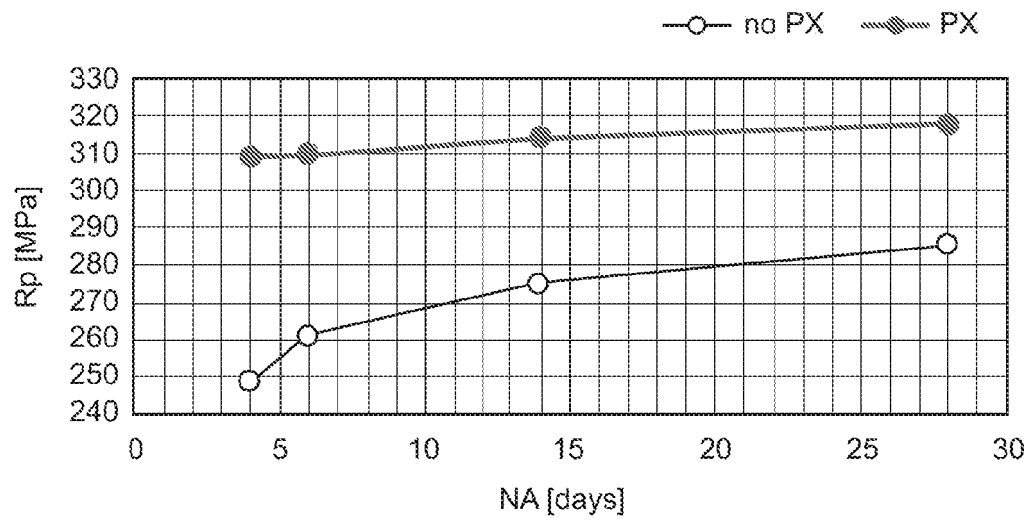


FIG. 1



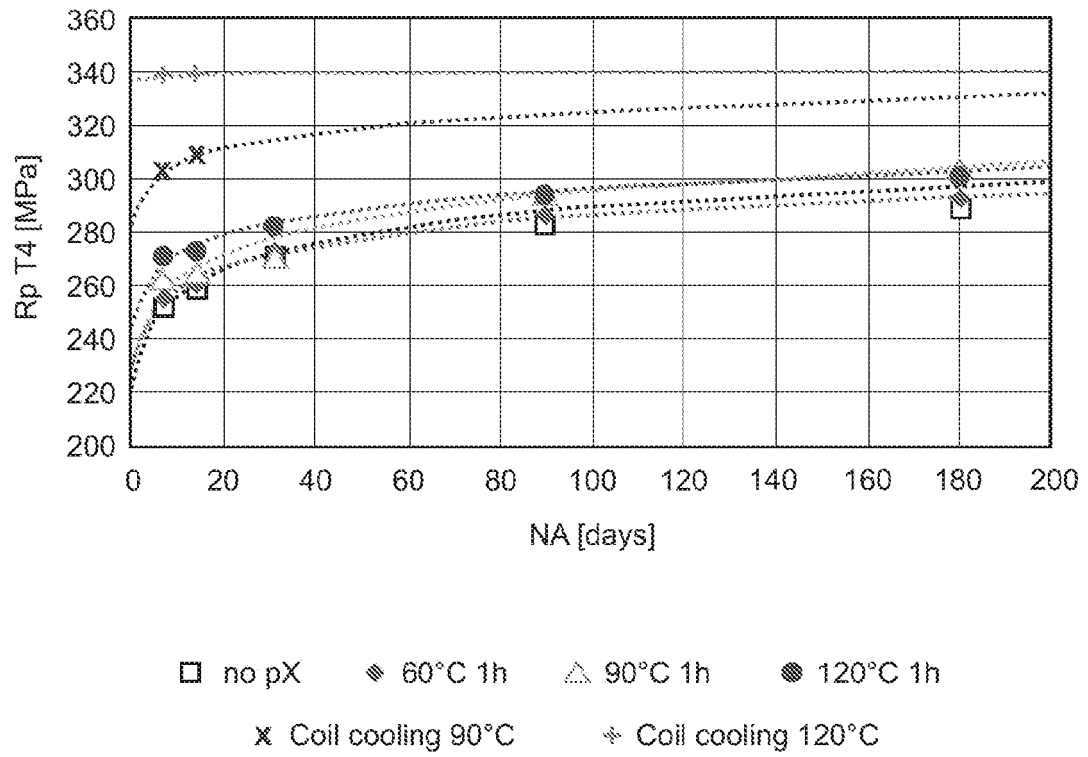


FIG. 2

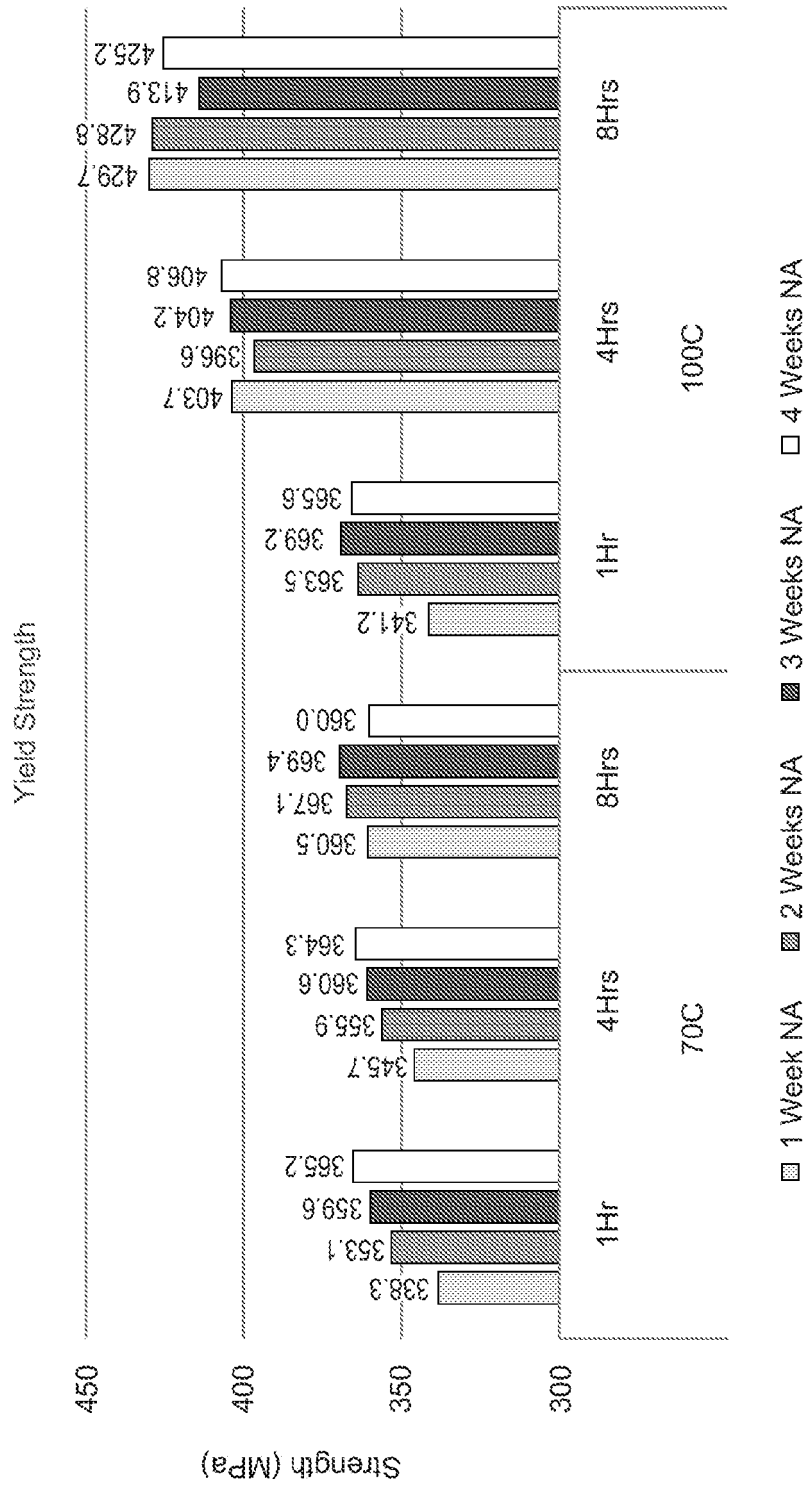


FIG. 3A

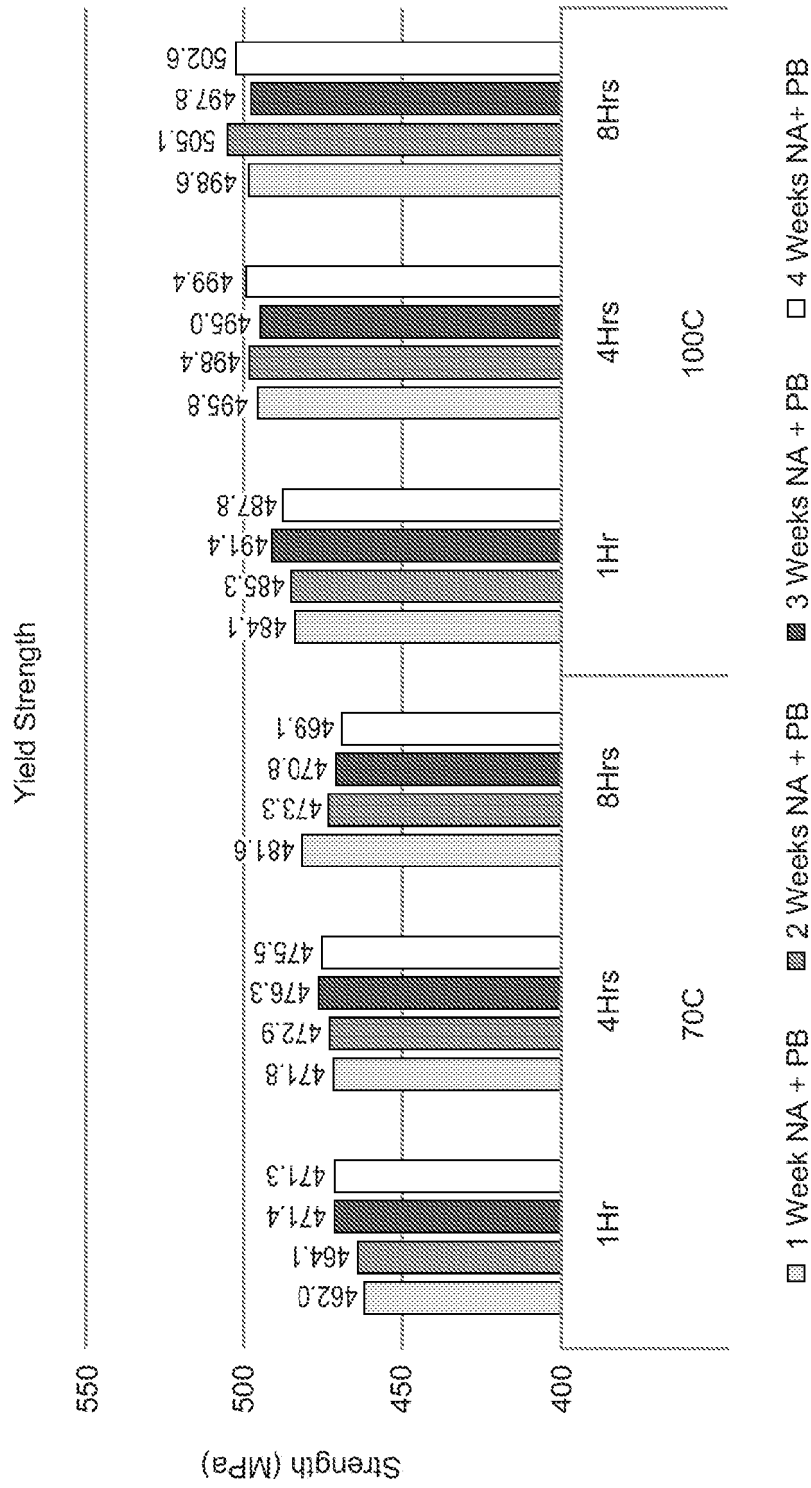


FIG. 3B

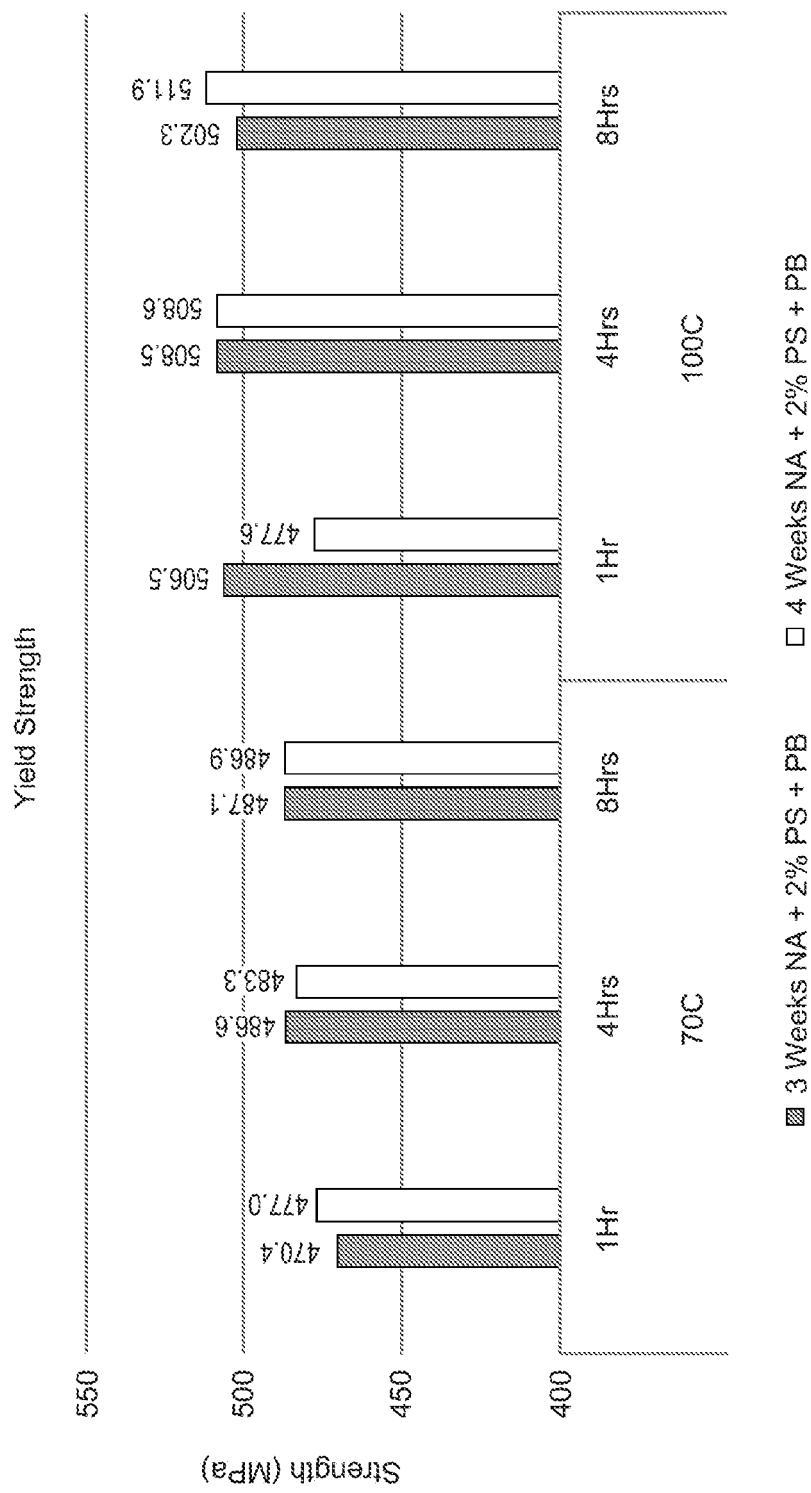


FIG. 3C

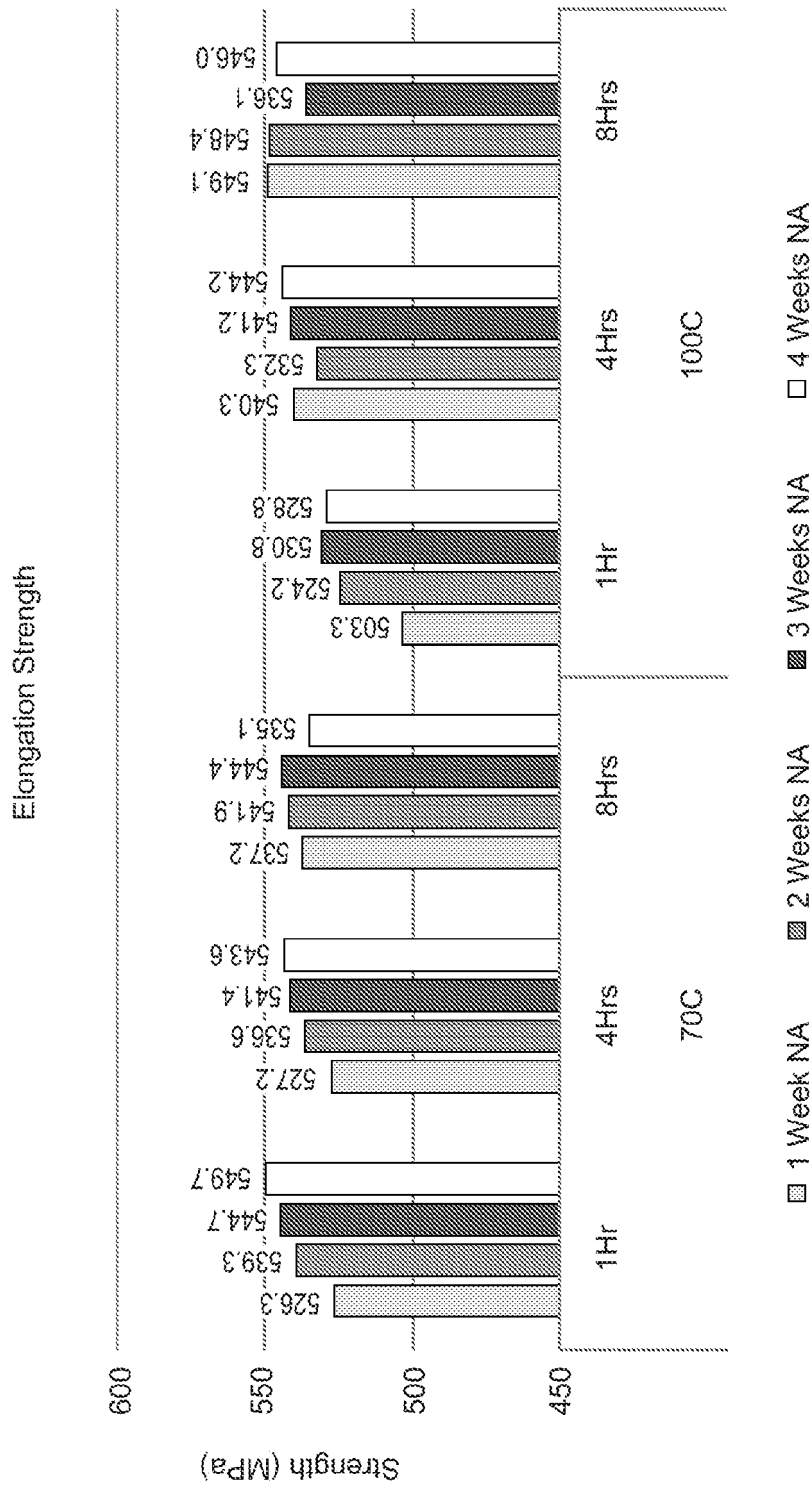


FIG. 4A

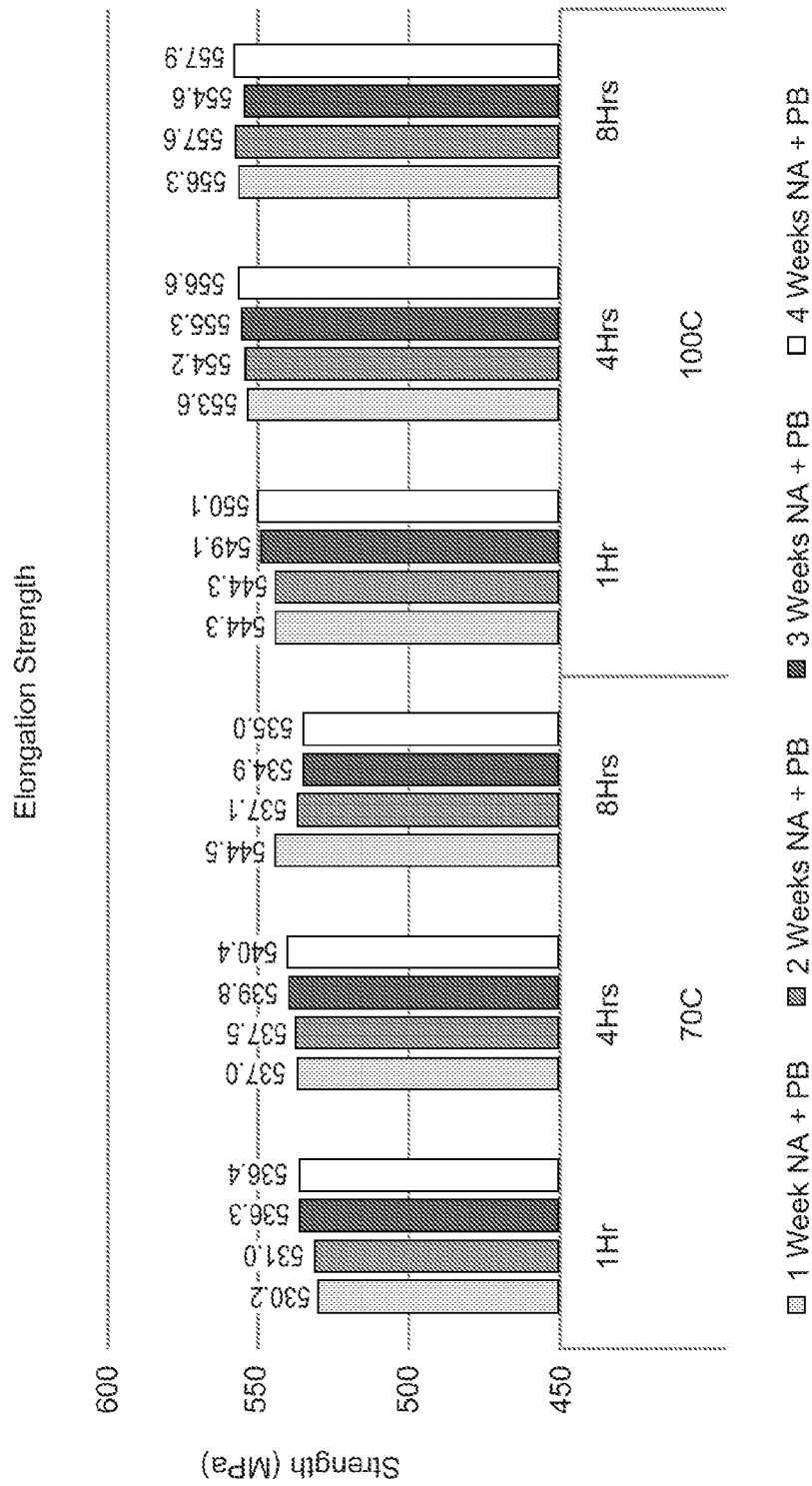


FIG. 4B

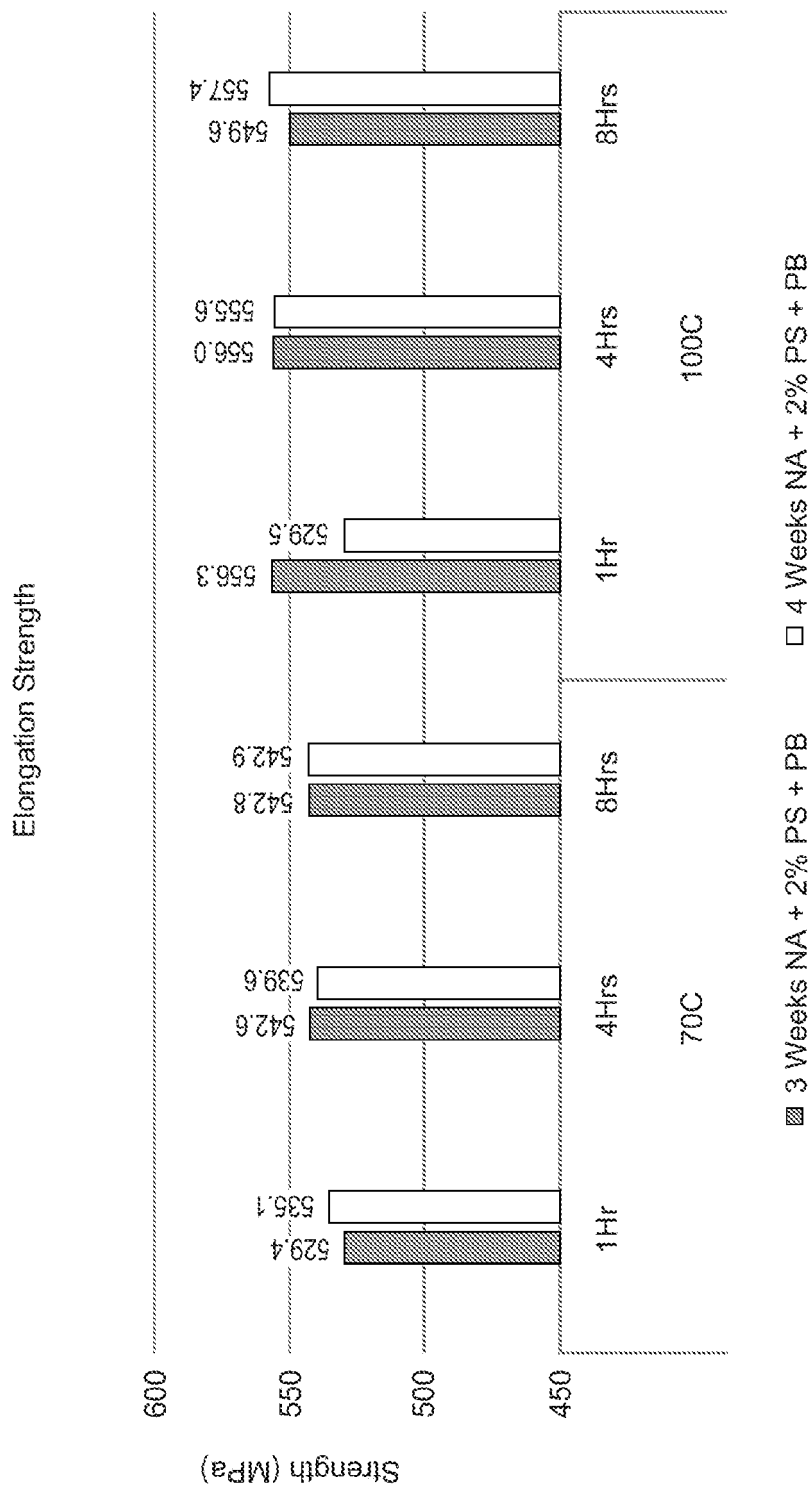


FIG. 4C

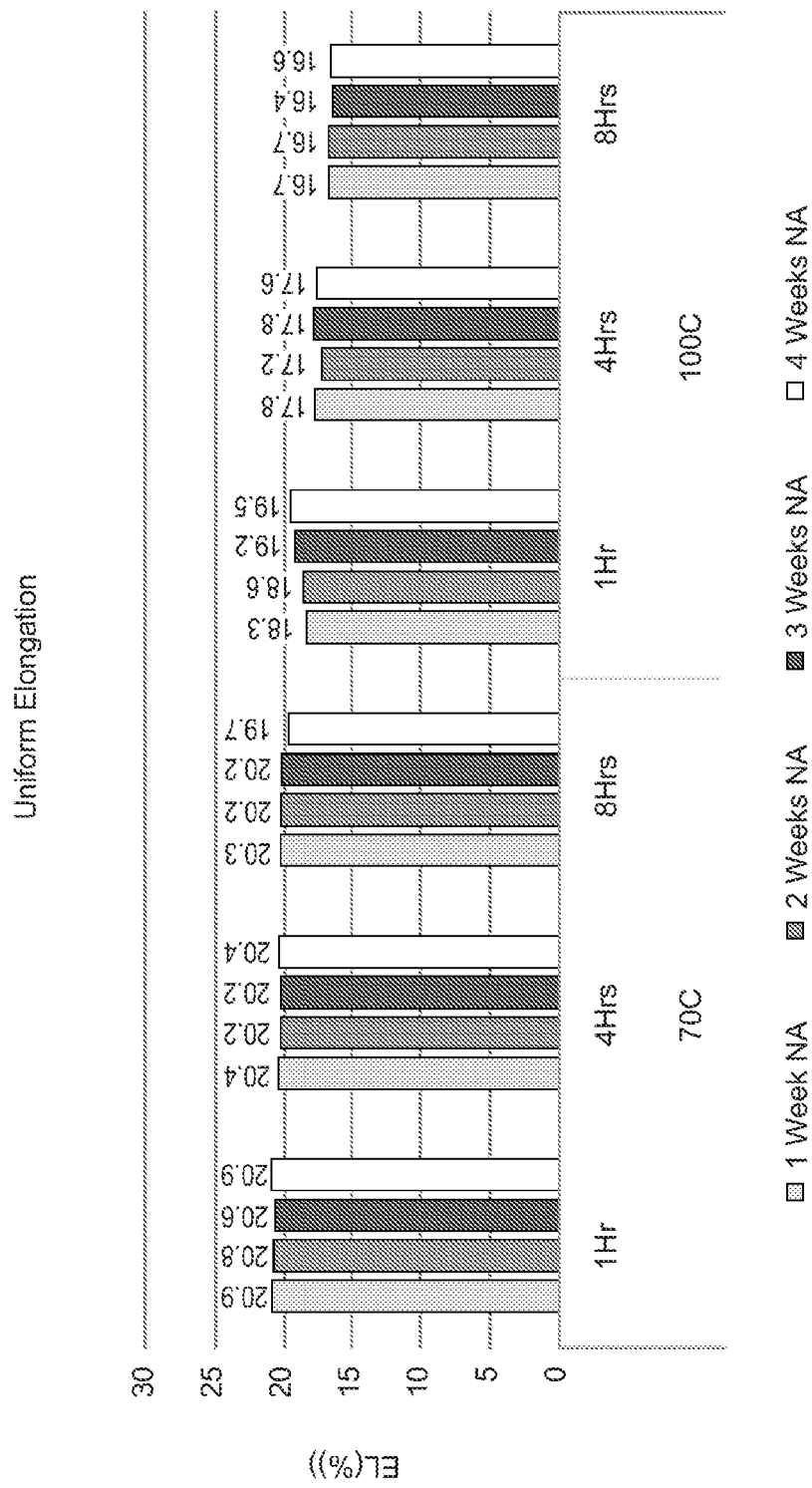


FIG. 5A



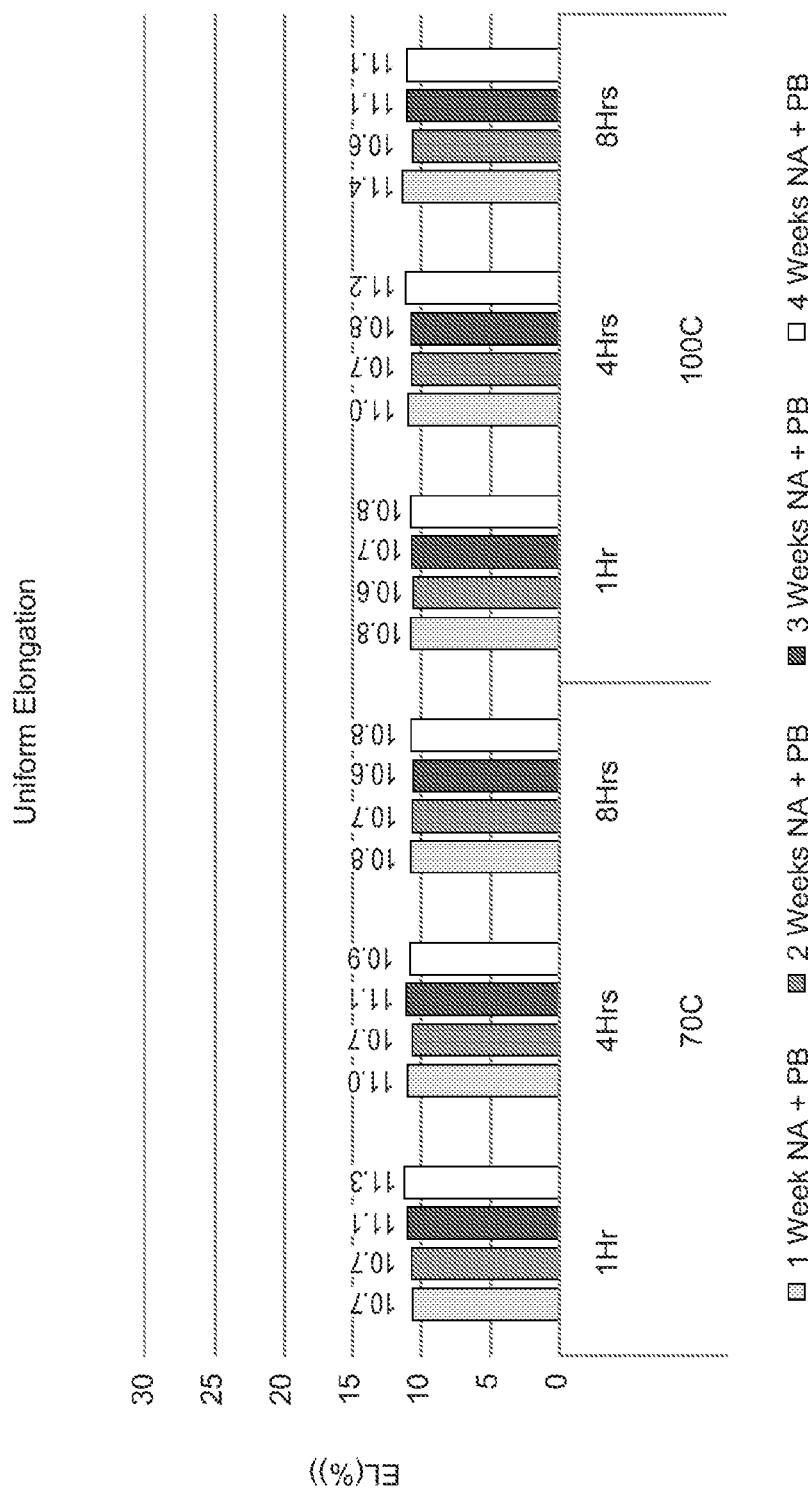


FIG. 5B

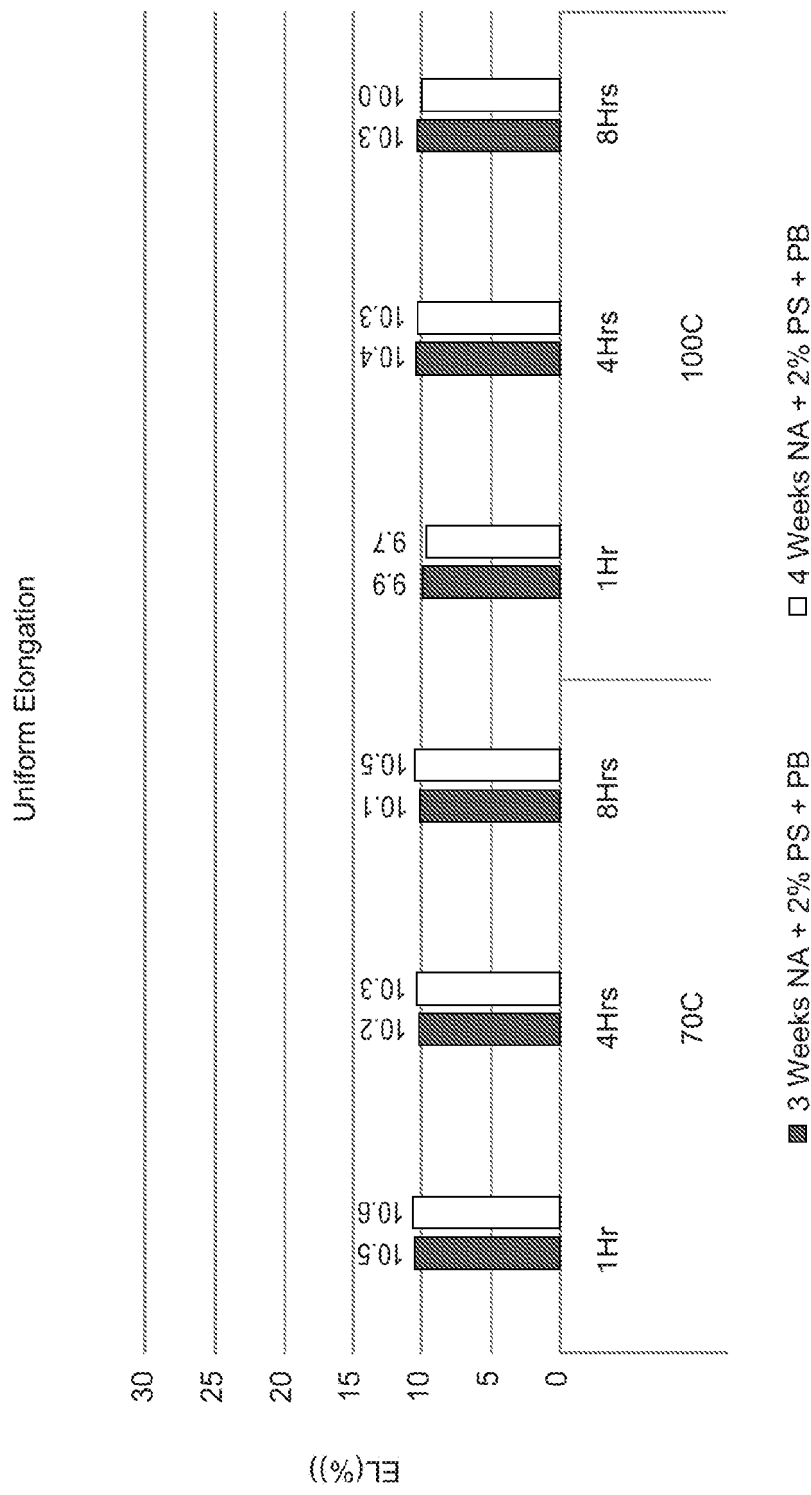


FIG. 5C

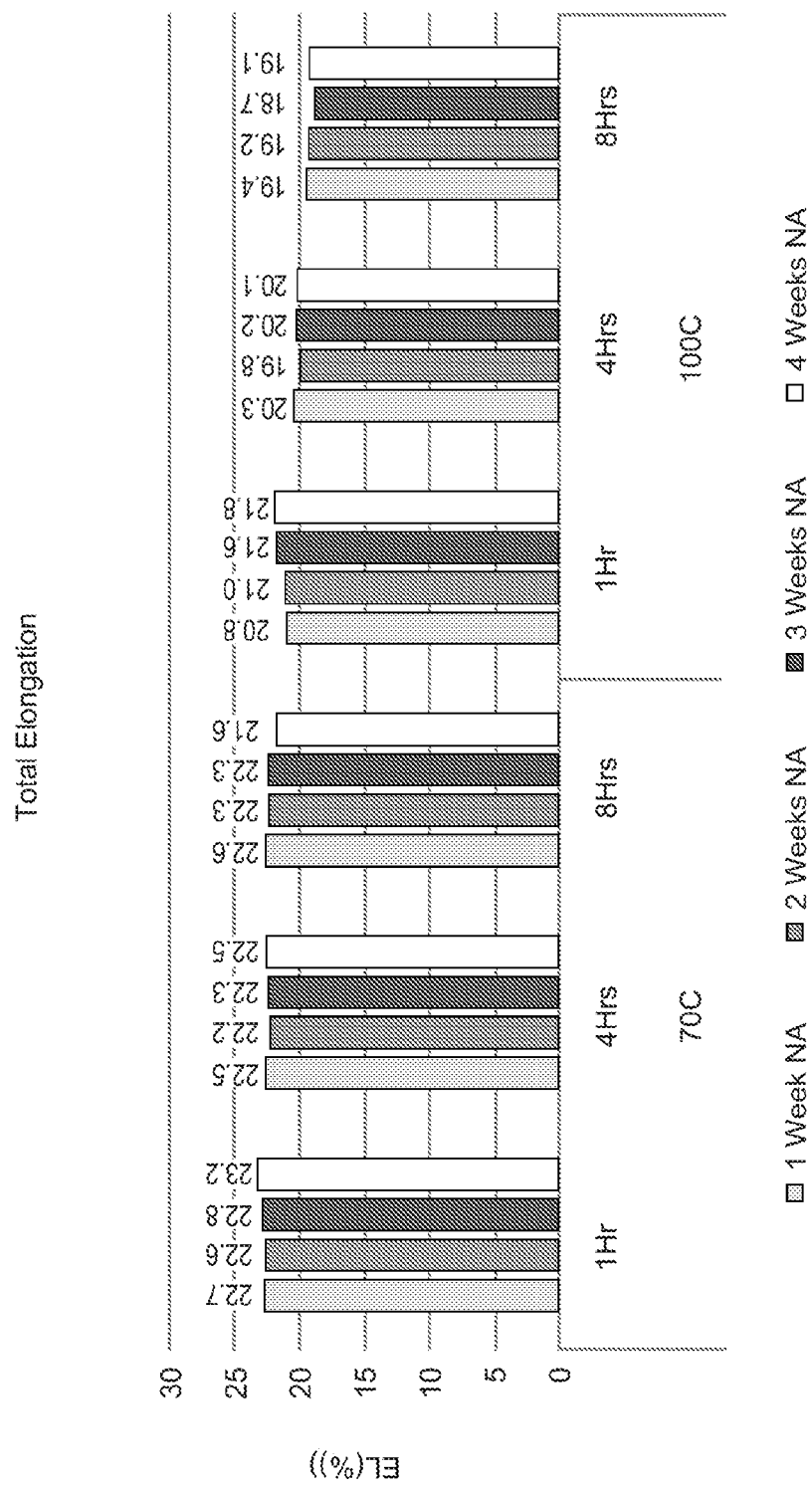


FIG. 6A

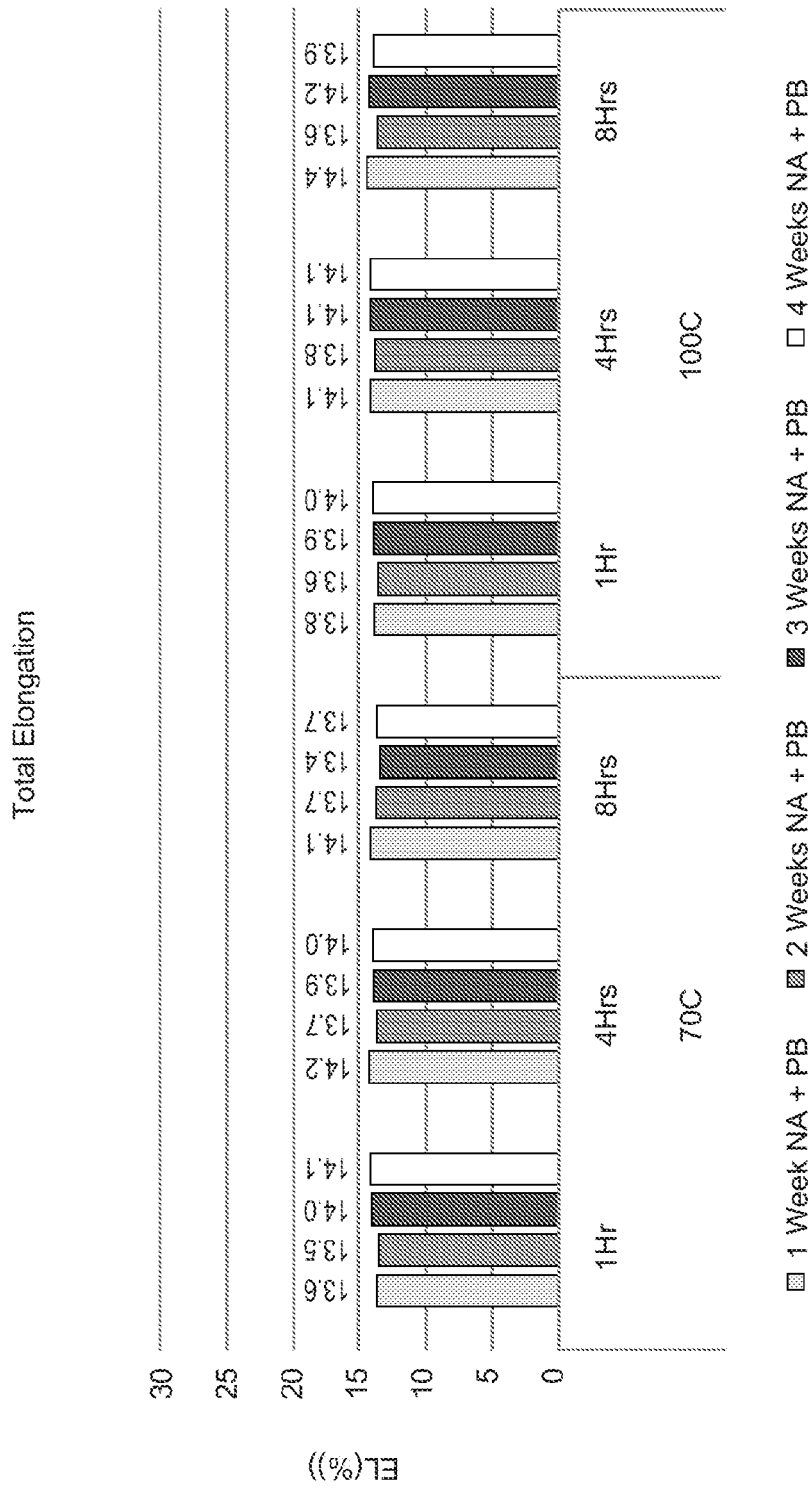


FIG. 6B

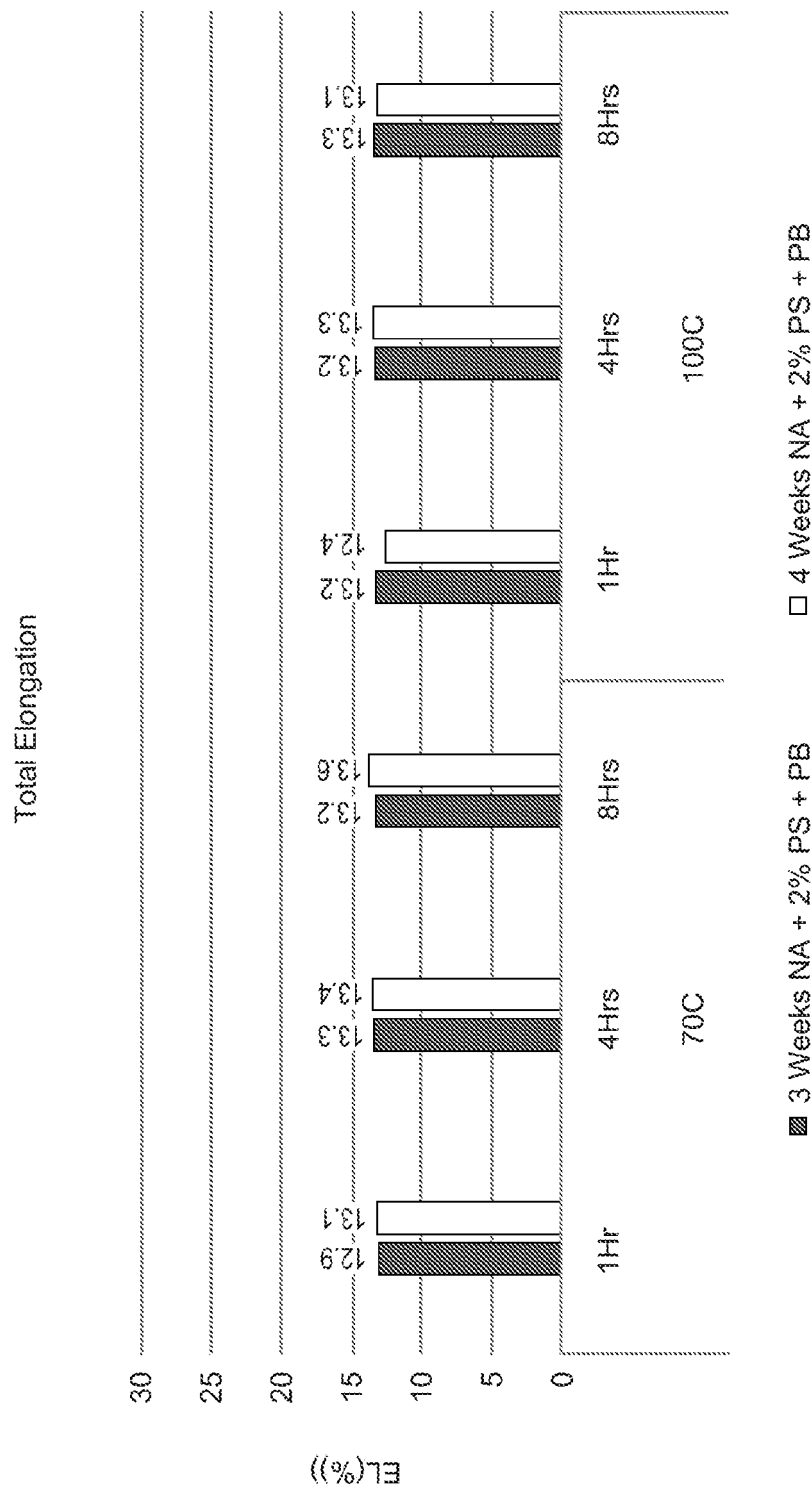


FIG. 6C

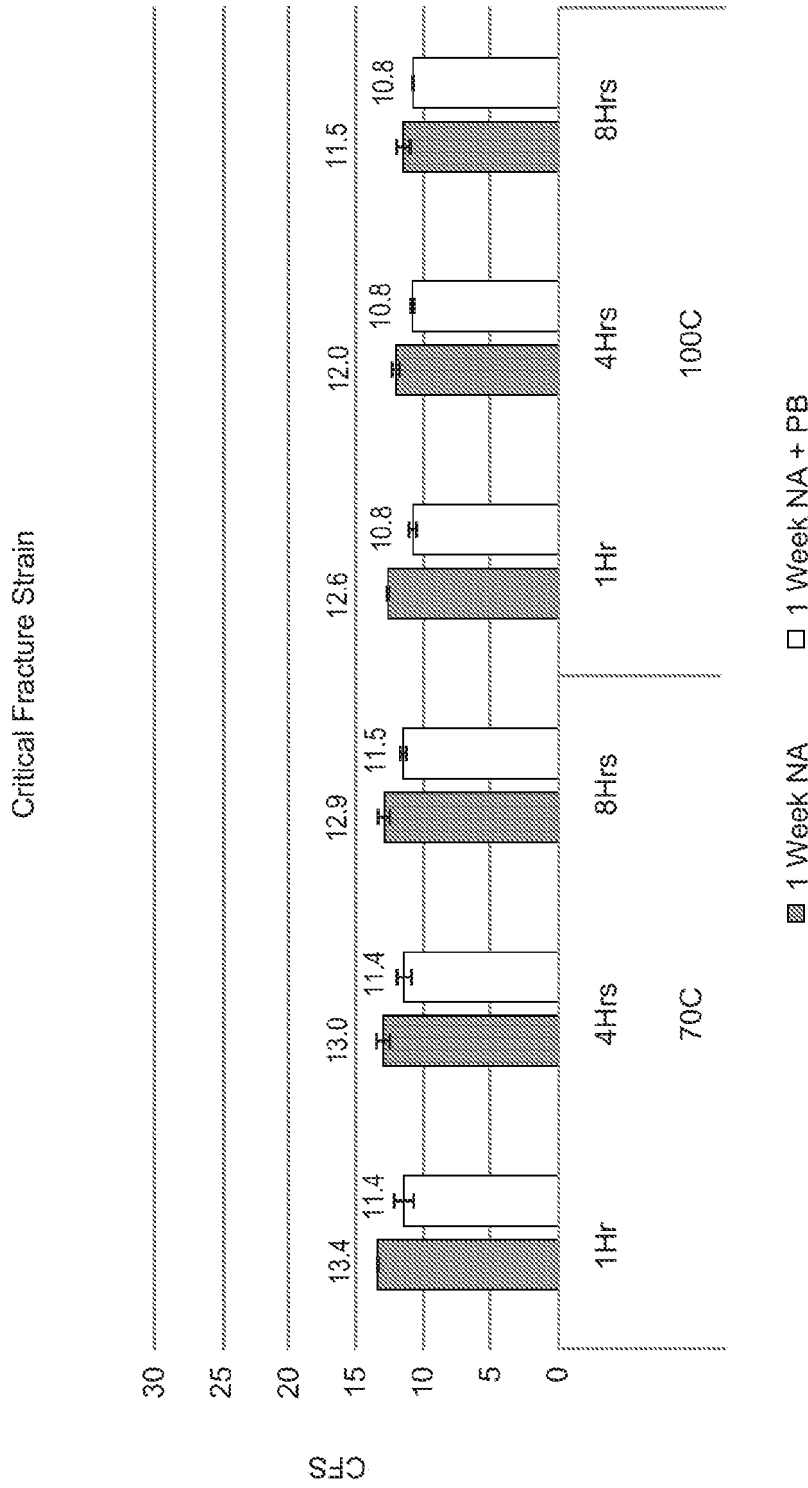


FIG. 7A

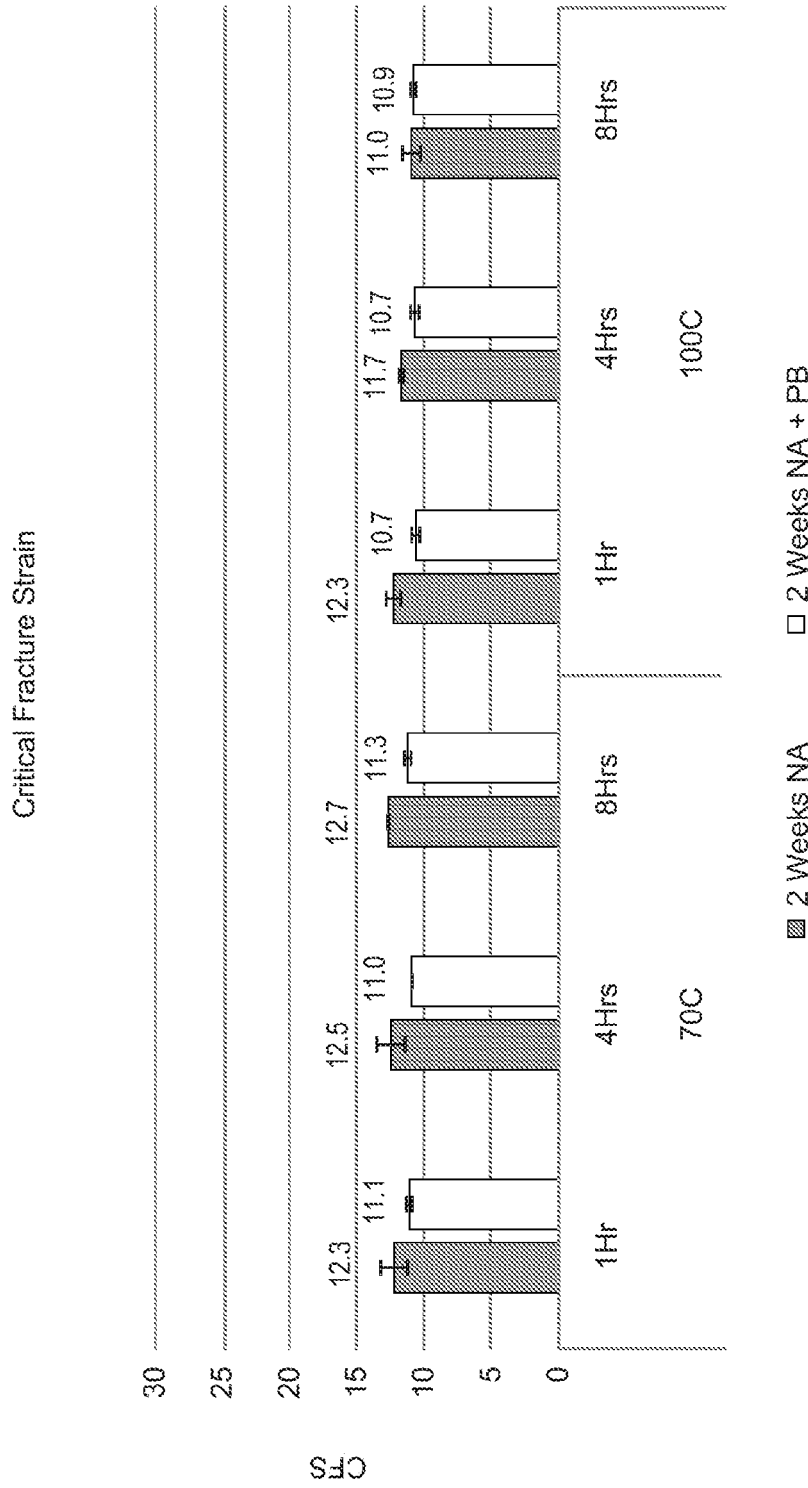


FIG. 7B

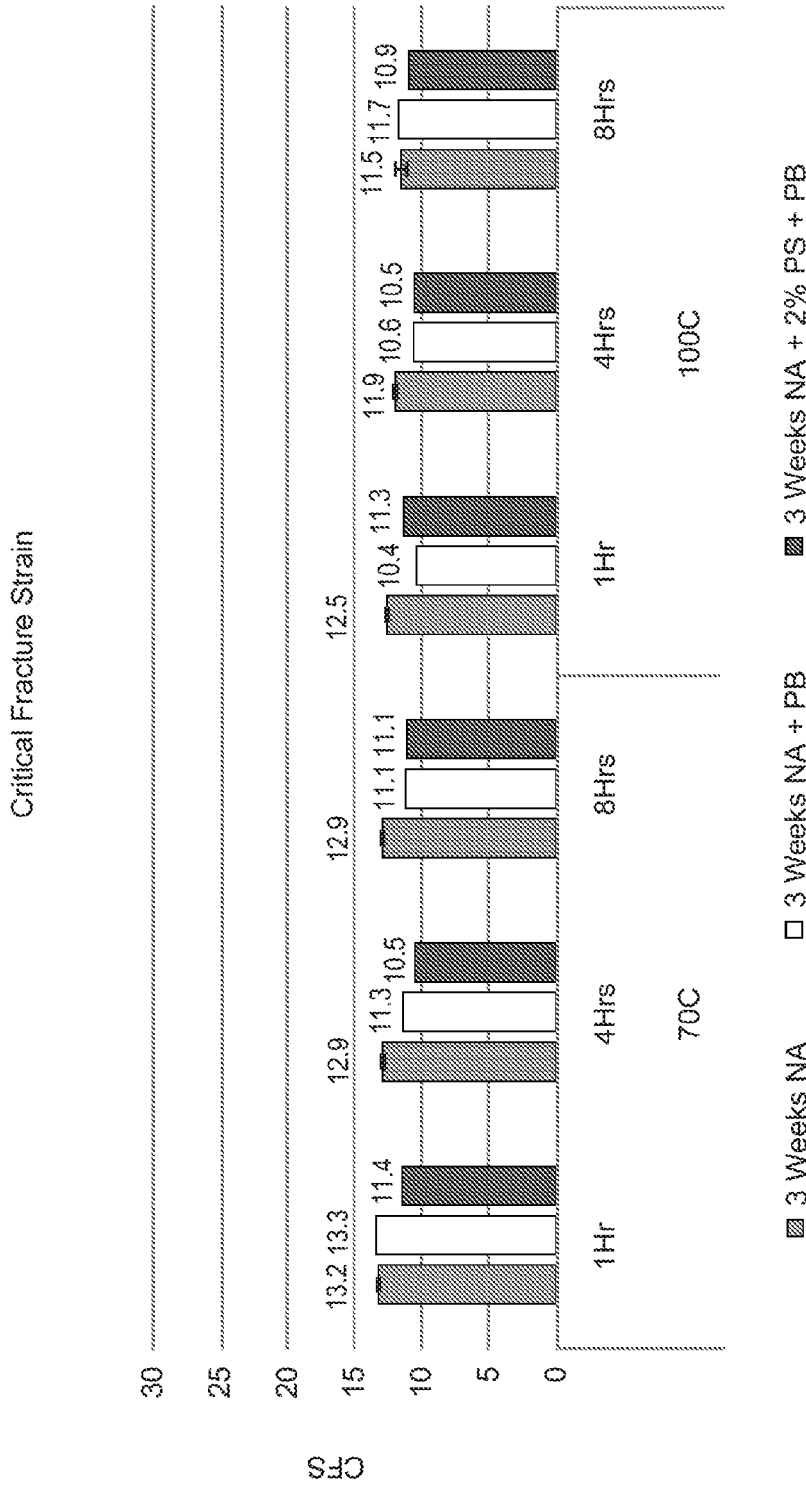


FIG. 7C



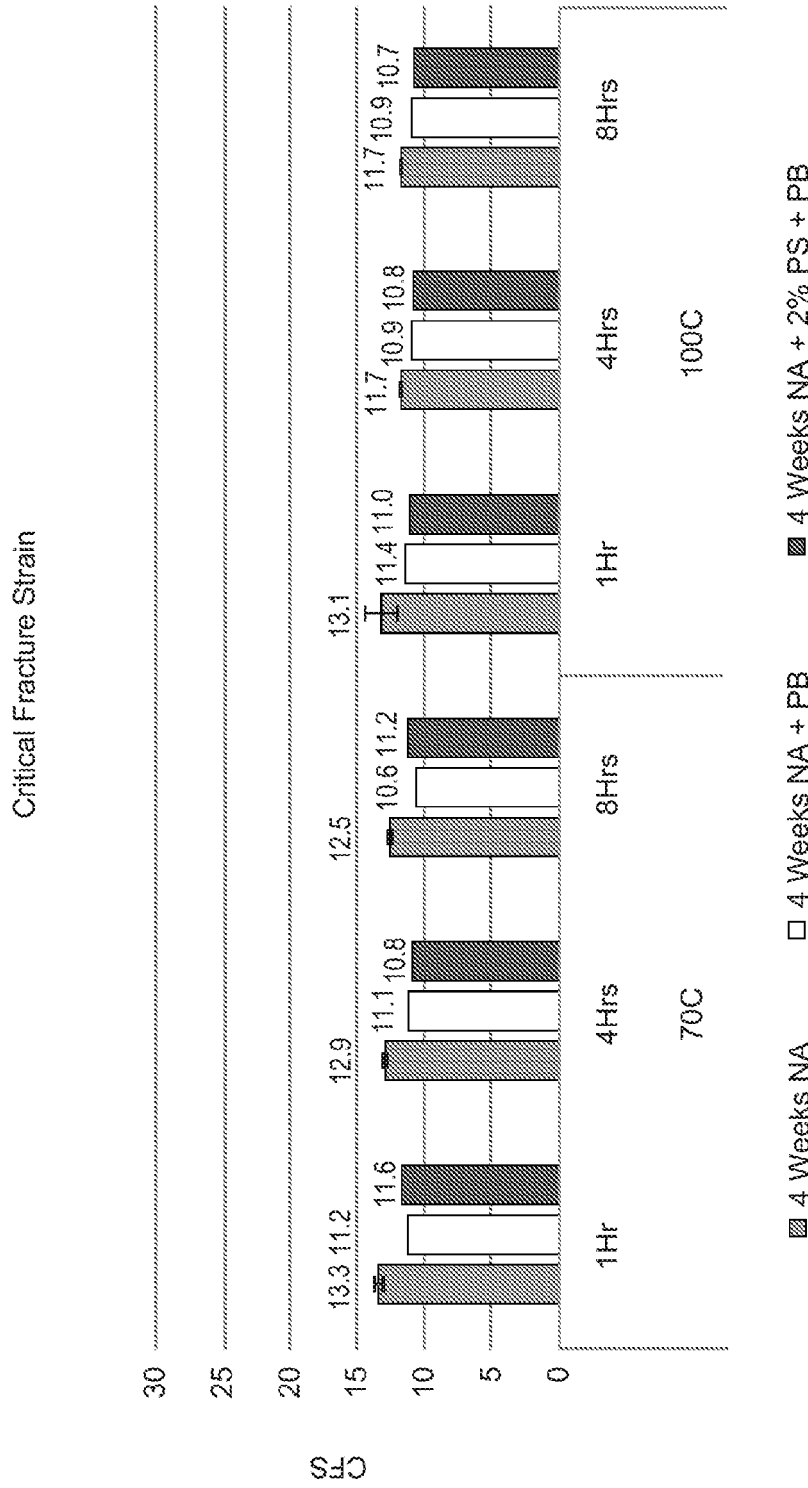


FIG. 7D

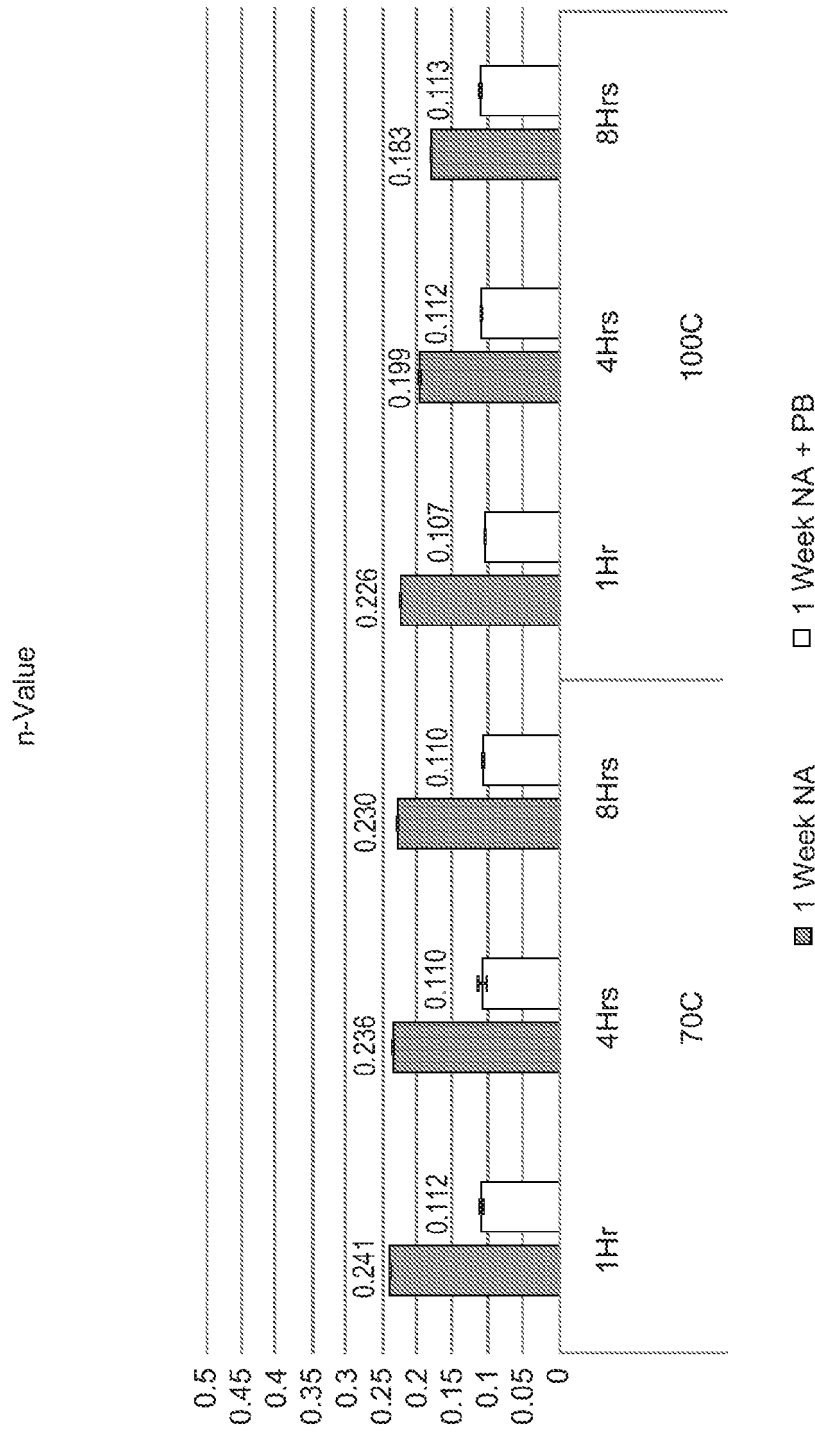


FIG. 8A

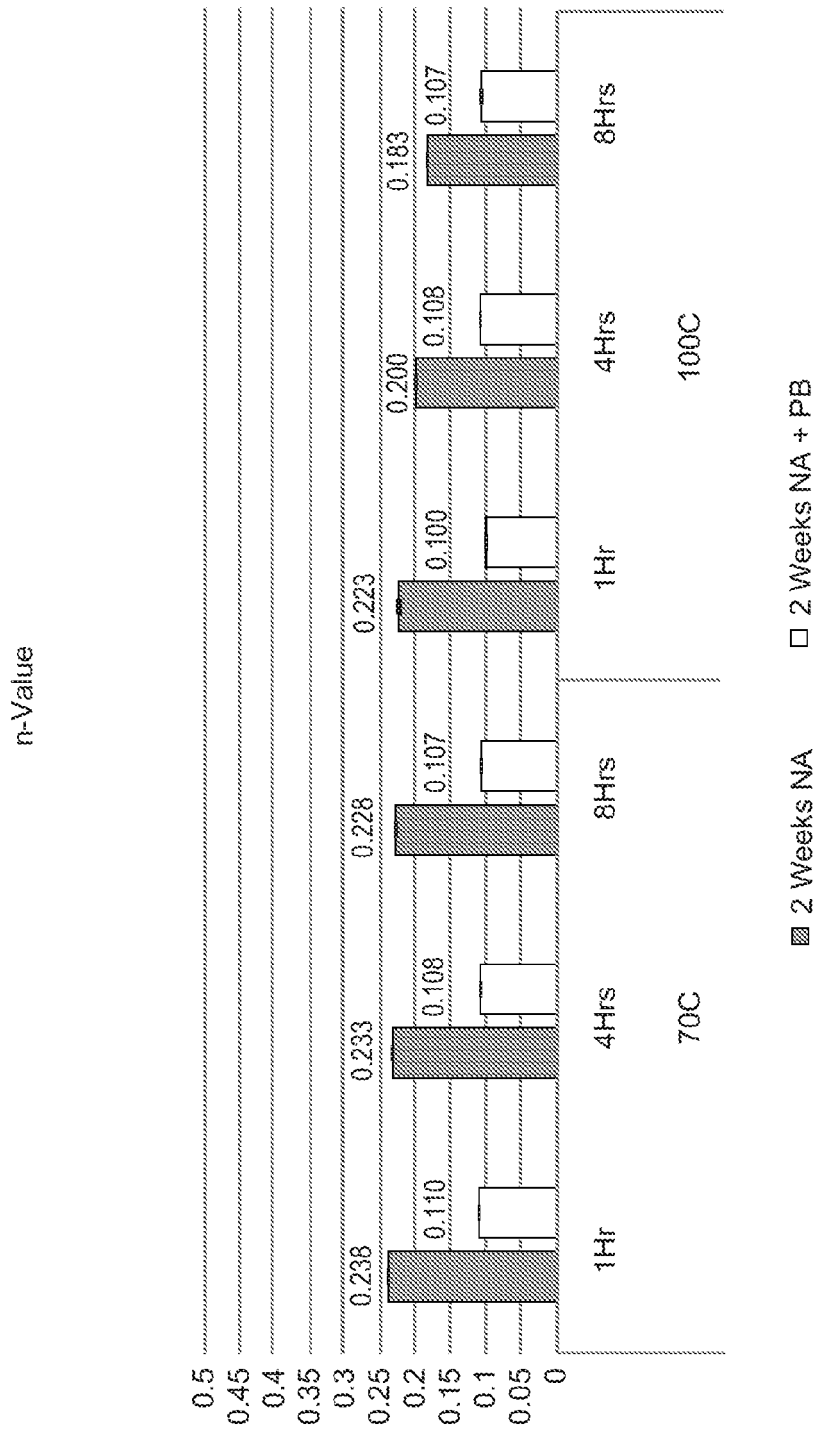


FIG. 8B

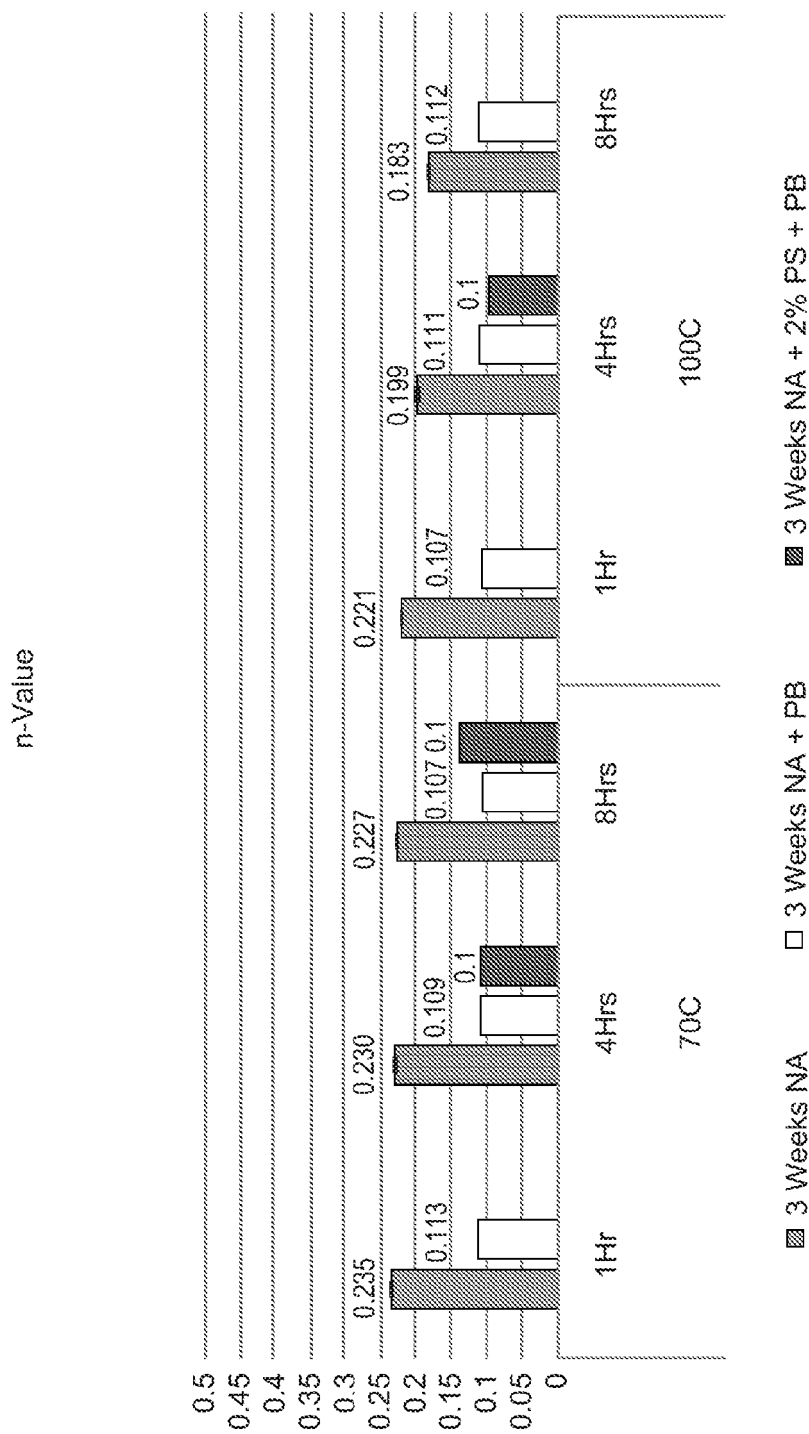


FIG. 8C

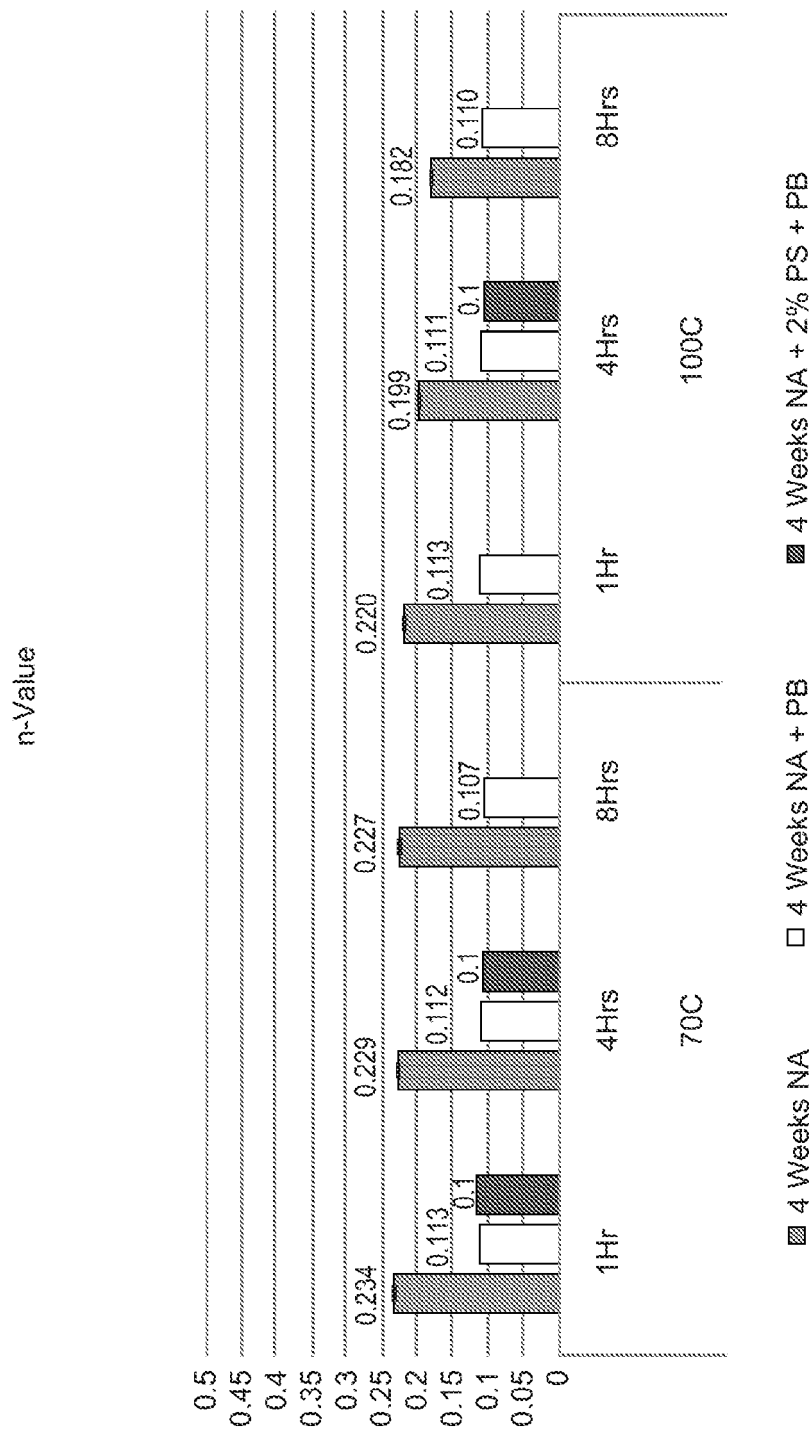


FIG. 8D

**REFERENCES CITED IN THE DESCRIPTION**

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