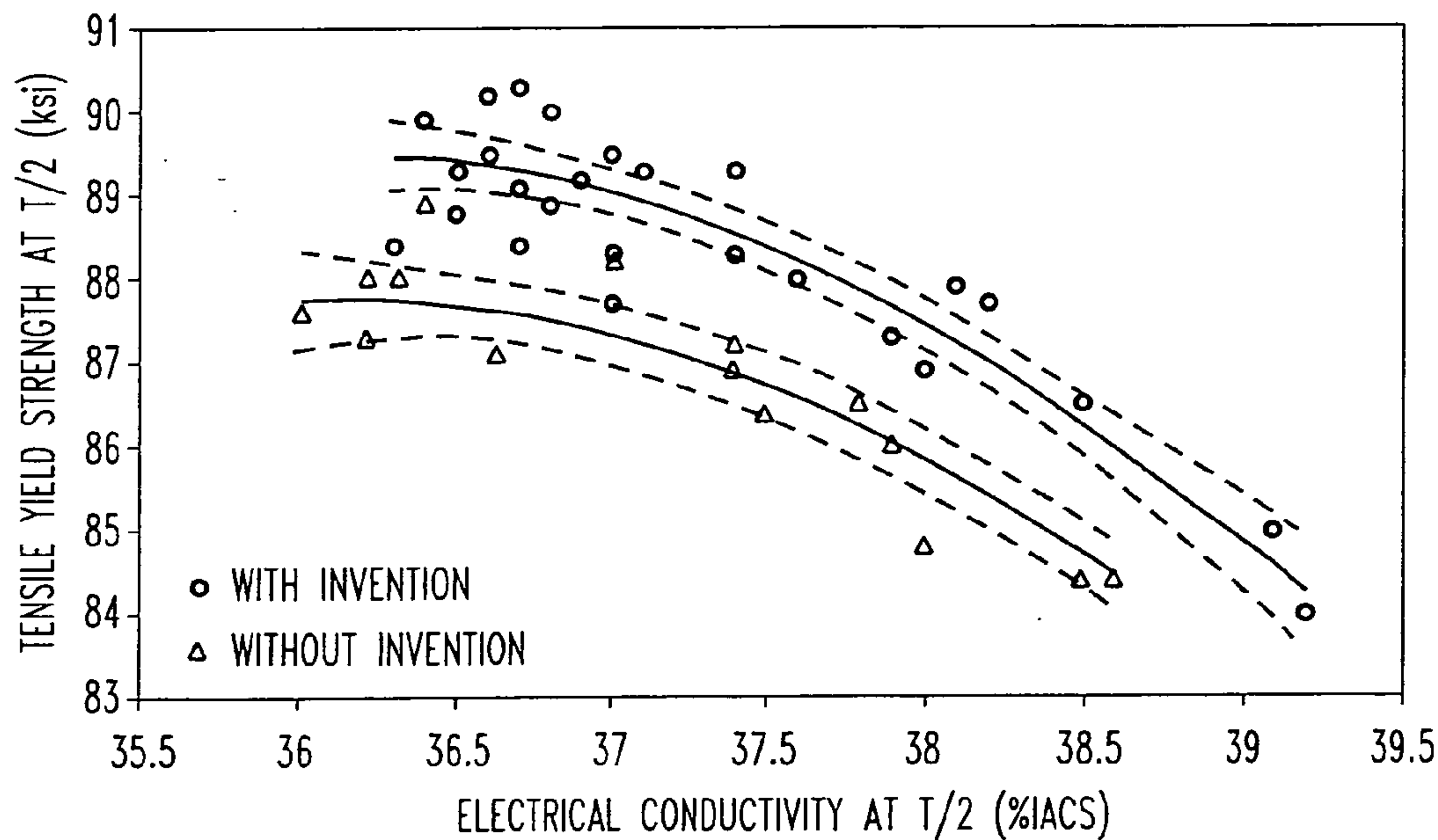
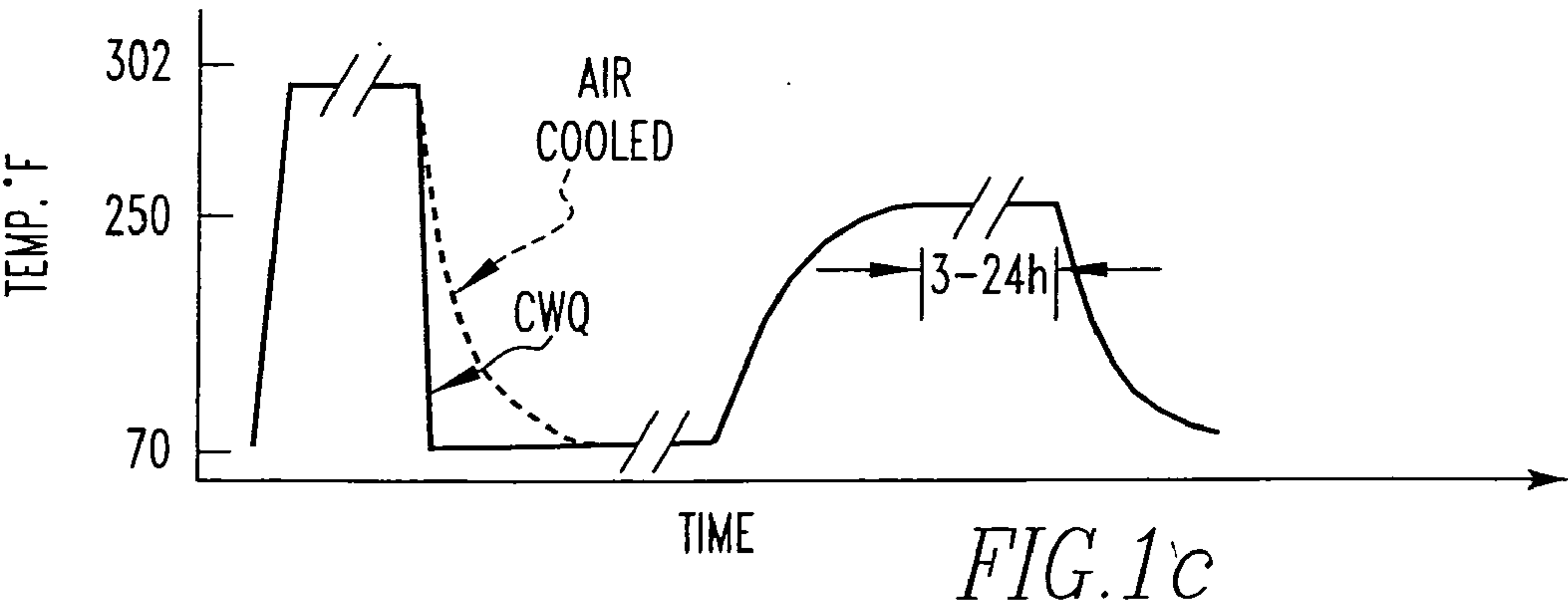
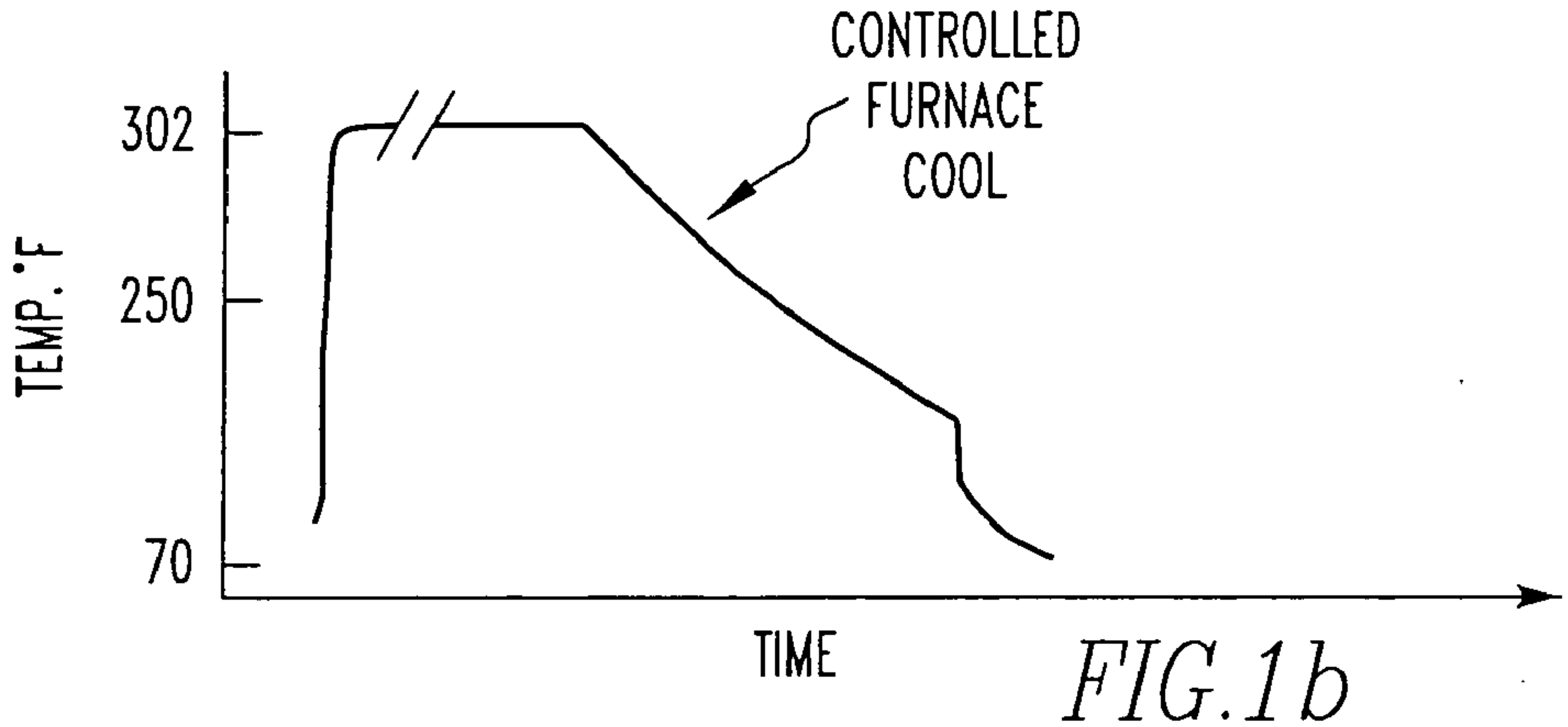
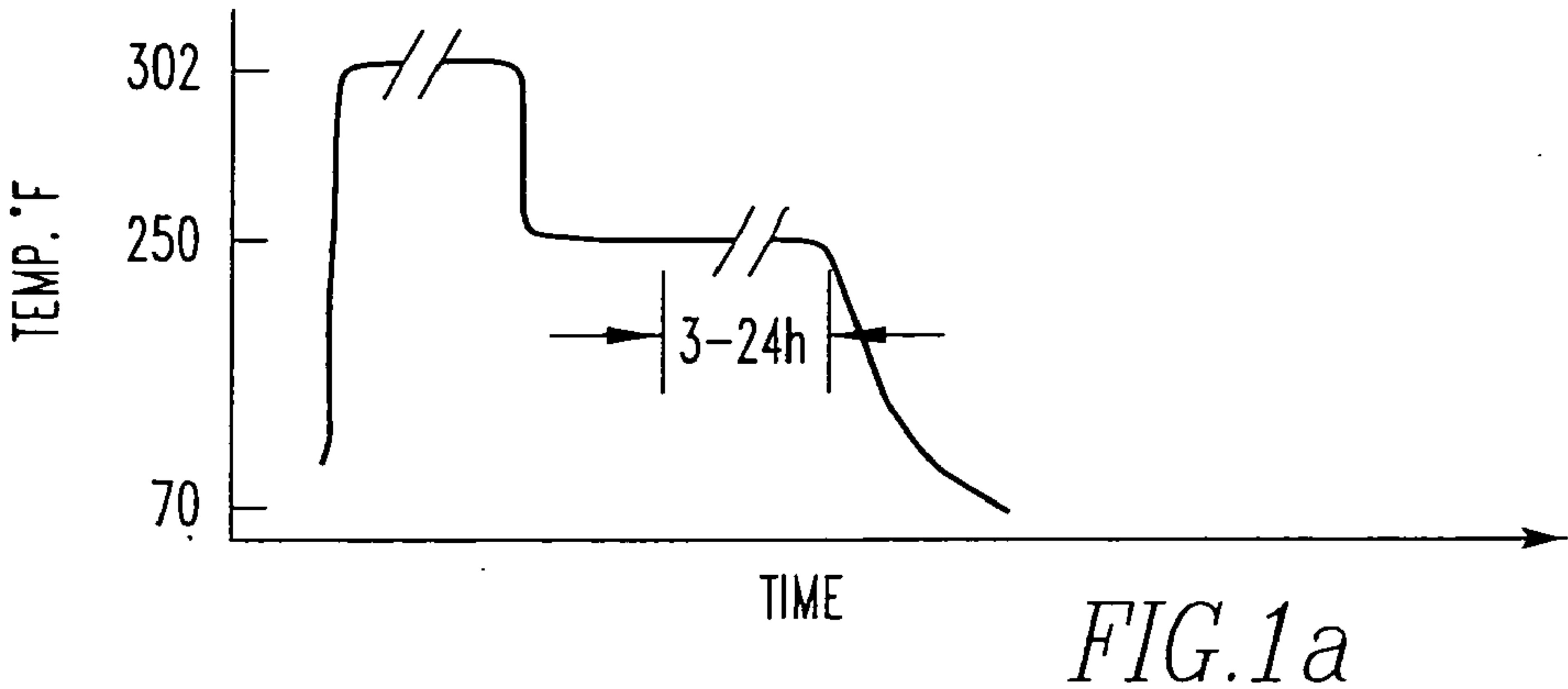


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Denzer et al.(10) **Pub. No.: US 2005/0269000 A1**(43) **Pub. Date: Dec. 8, 2005**(54) **METHOD FOR INCREASING THE  
STRENGTH AND/OR CORROSION  
RESISTANCE OF 7000 SERIES Al  
AEROSPACE ALLOY PRODUCTS****Related U.S. Application Data**(63) Continuation-in-part of application No. 10/103,273,  
filed on Mar. 20, 2002, now abandoned.(60) Provisional application No. 60/277,403, filed on Mar.  
20, 2001.(76) Inventors: **Diana K. Denzer**, Lower Burrell, PA  
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**ALCOA CENTER, PA 15069-0001 (US)**(57) **ABSTRACT**

This invention relates to a method for artificially aging 7000 Series Al aerospace alloys to impart improved strength and/or corrosion resistance performance thereto. The method purposefully adds a second aging step or stage to a one-step tempering, or a third step/stage to a low-high, two-step aging operation. The added step/stage extends at about 225-275° F. for about 3-24 hours. More preferably, the added stage extends at about 250° F. for about 6 hours or more.

(21) Appl. No.: **11/003,650**(22) Filed: **Dec. 3, 2004**



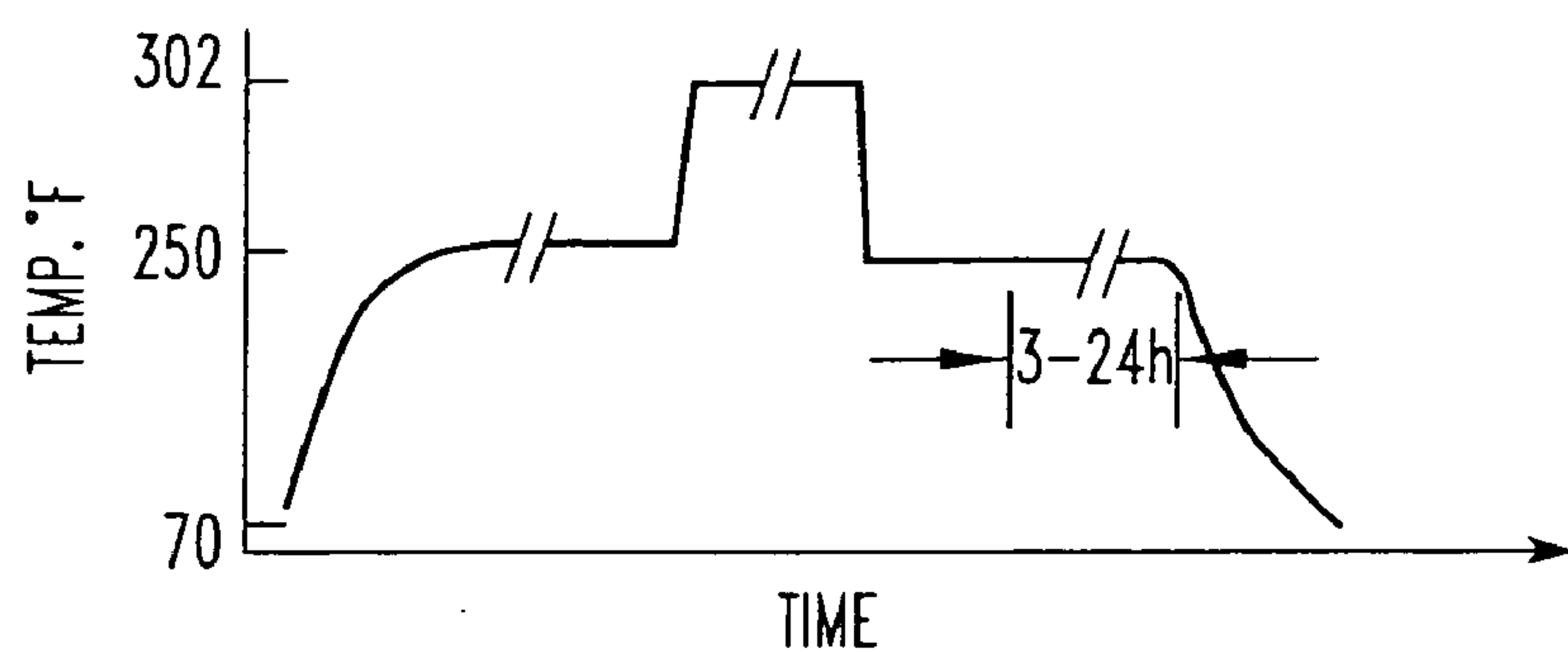


FIG. 2a

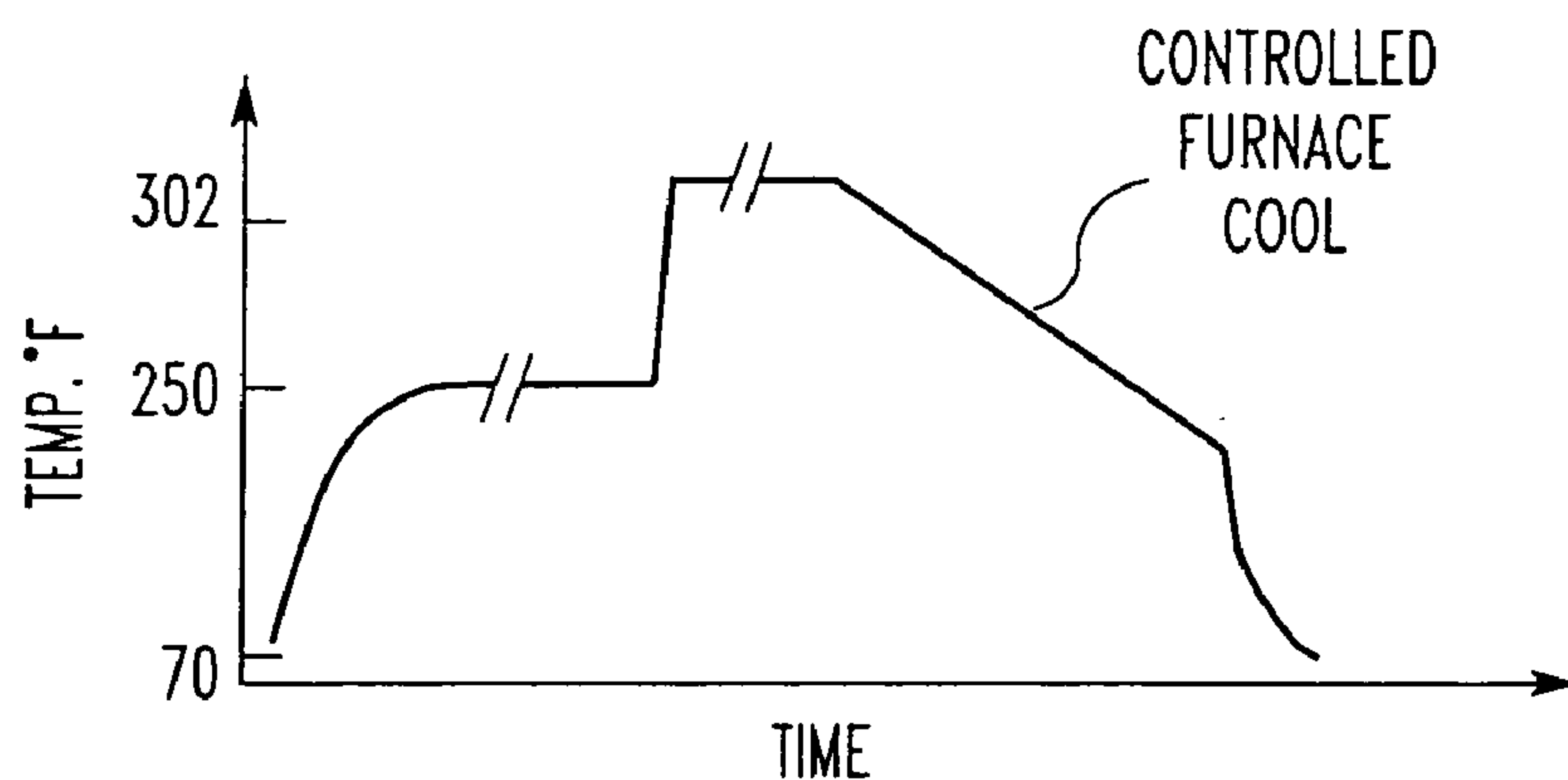


FIG. 2b

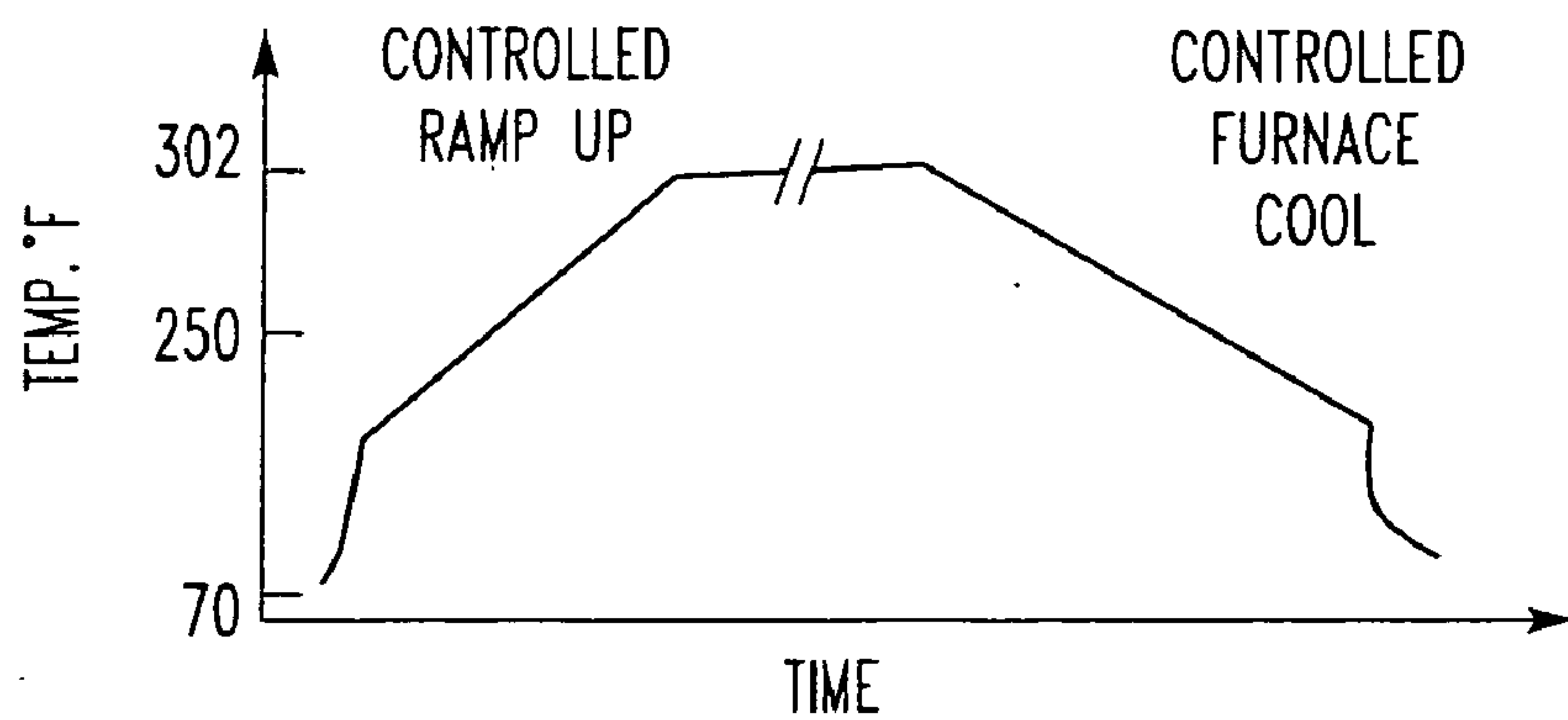
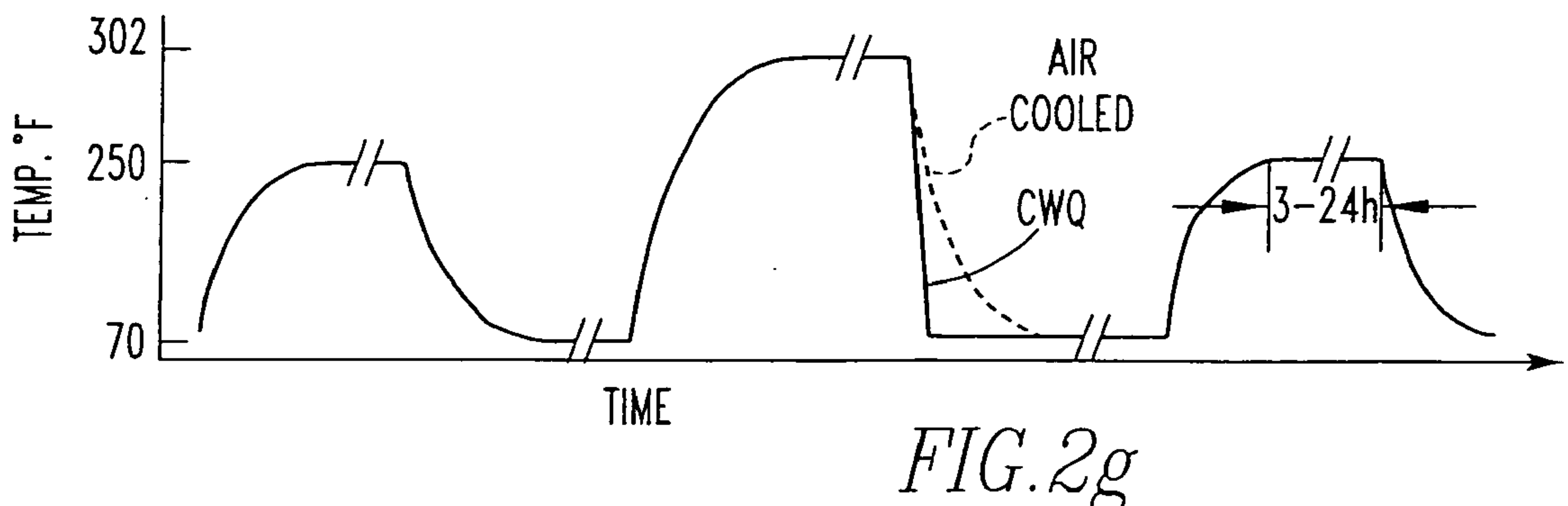
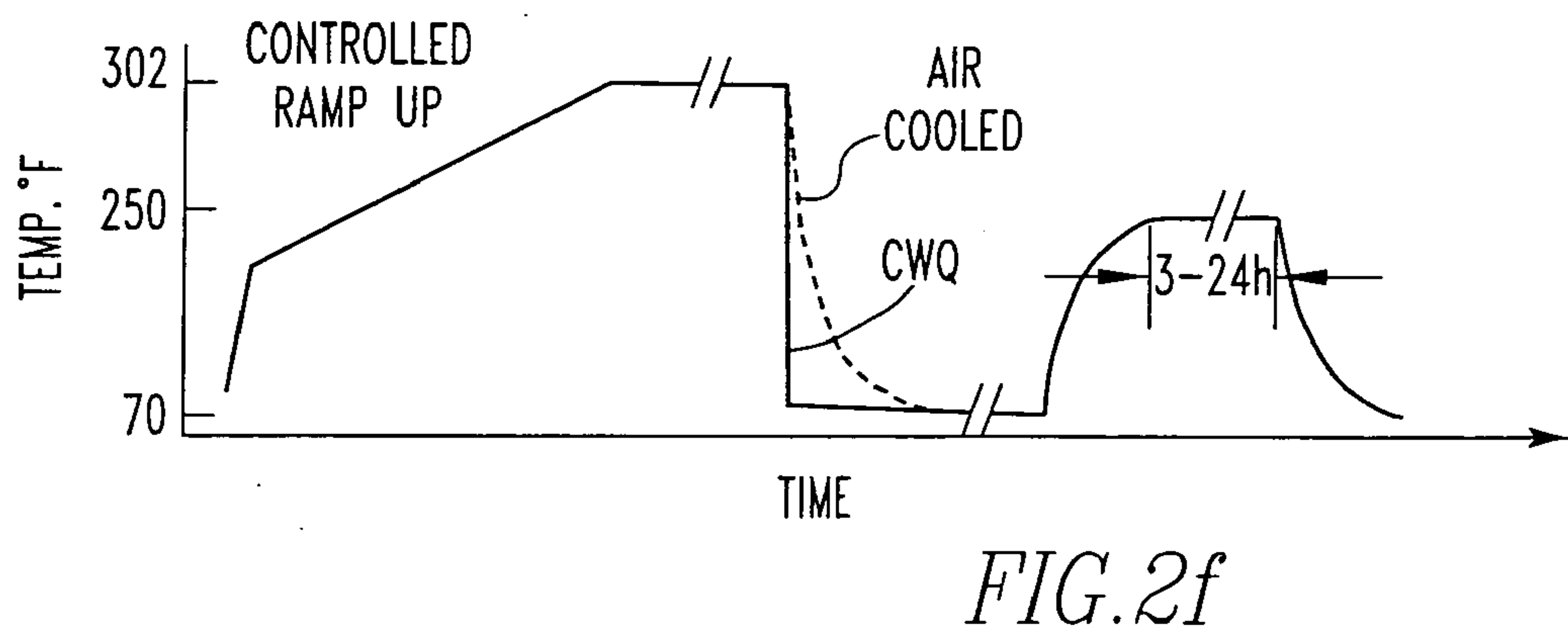
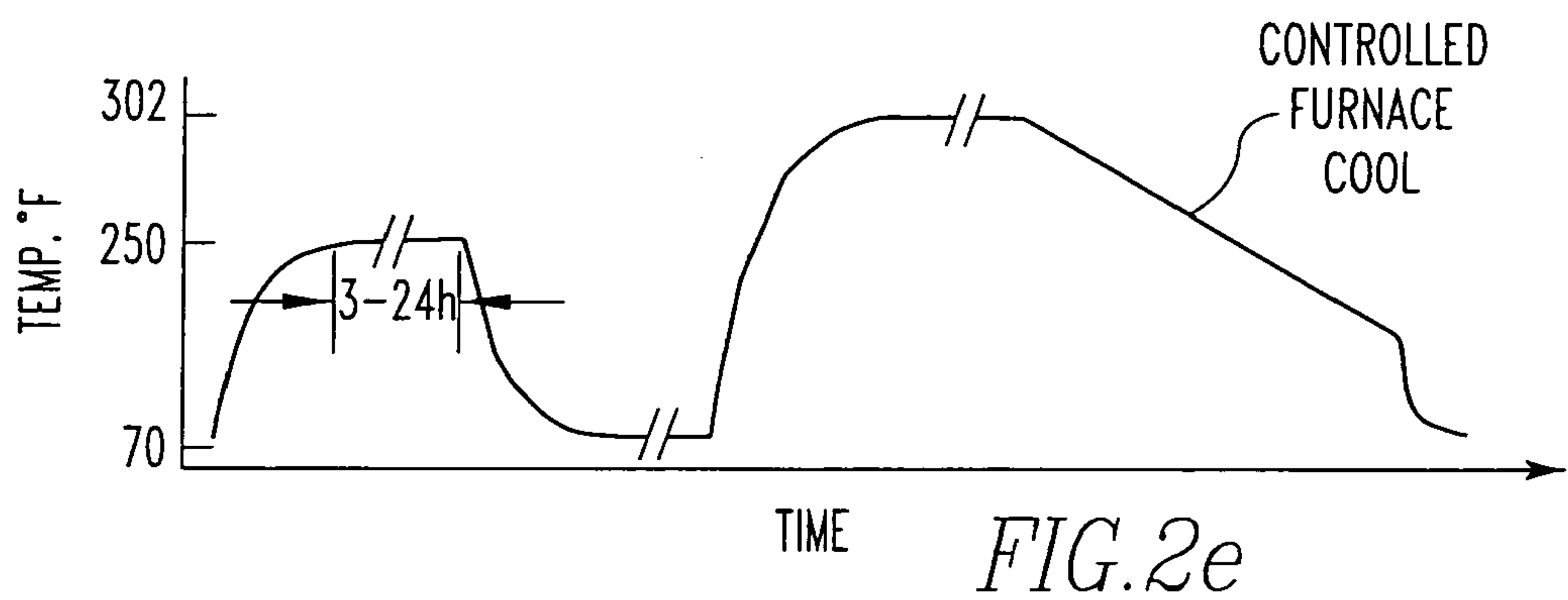
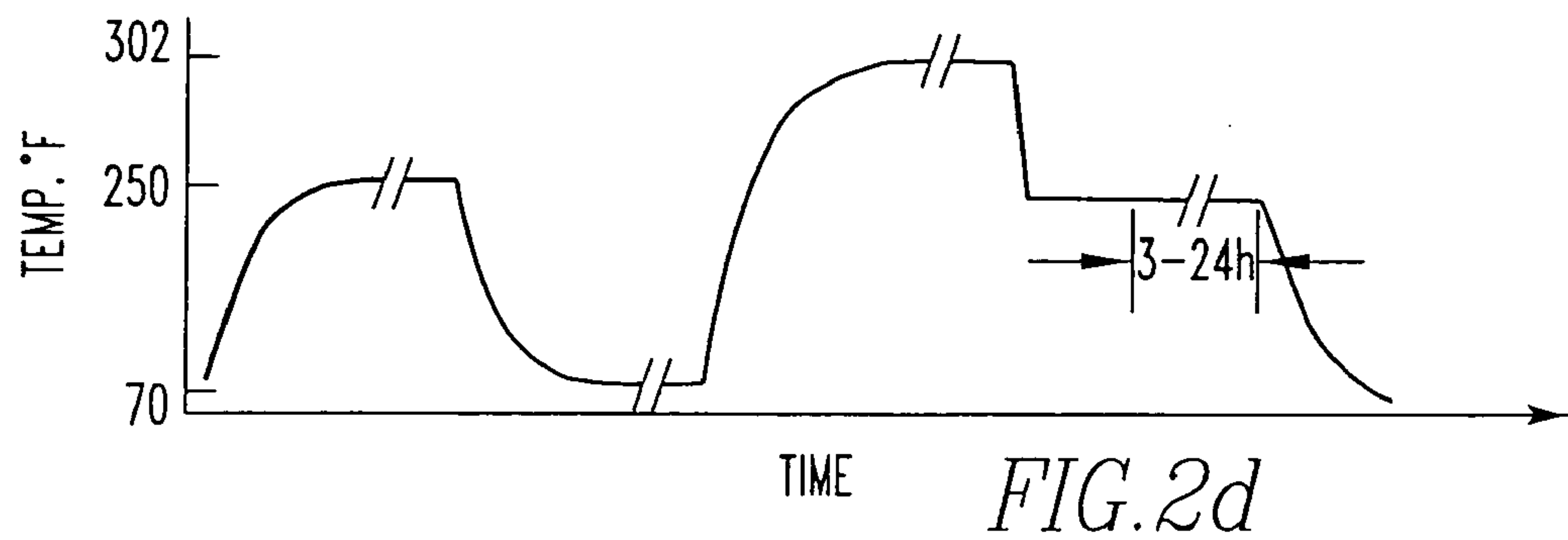


FIG. 2c



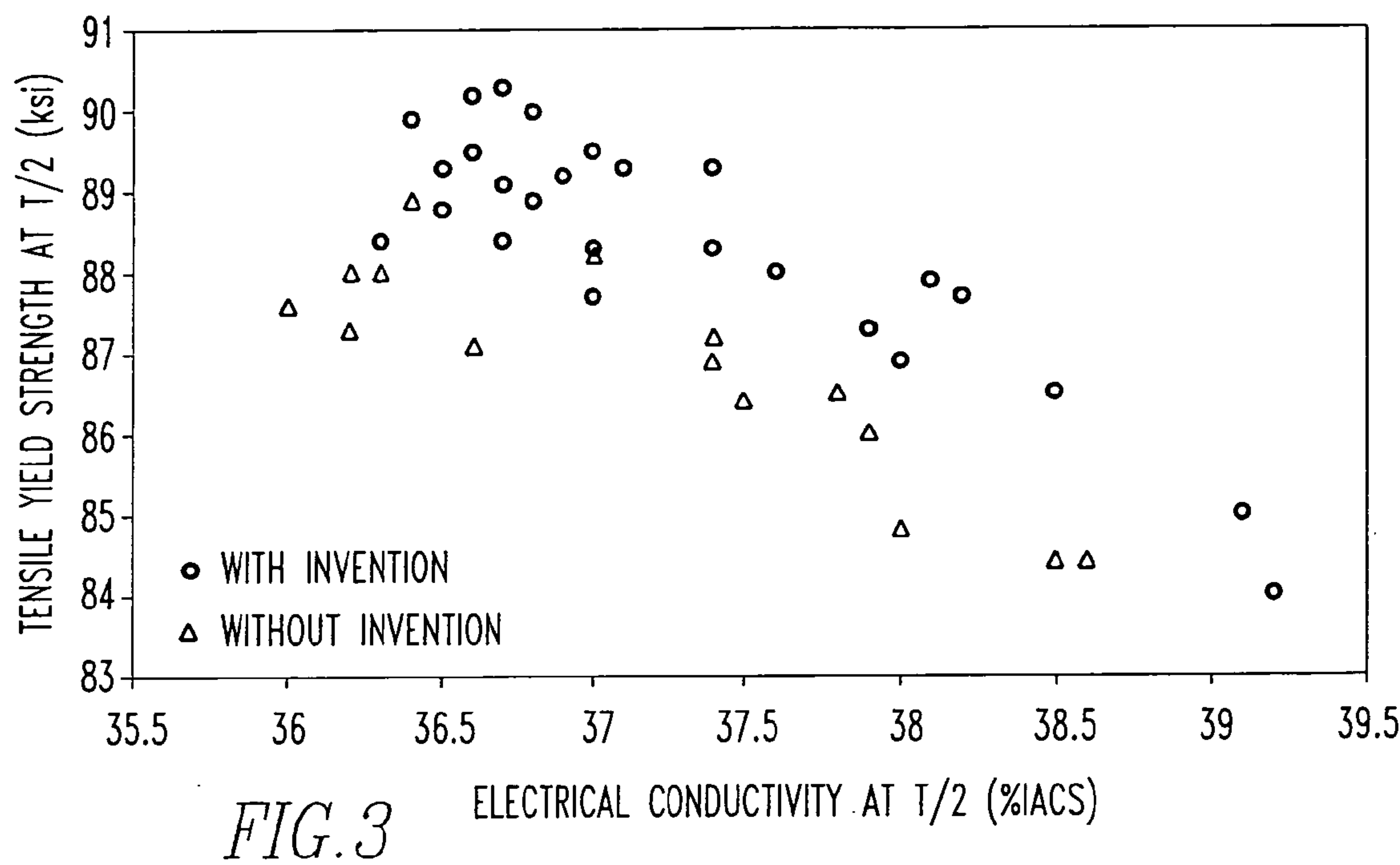


FIG. 3

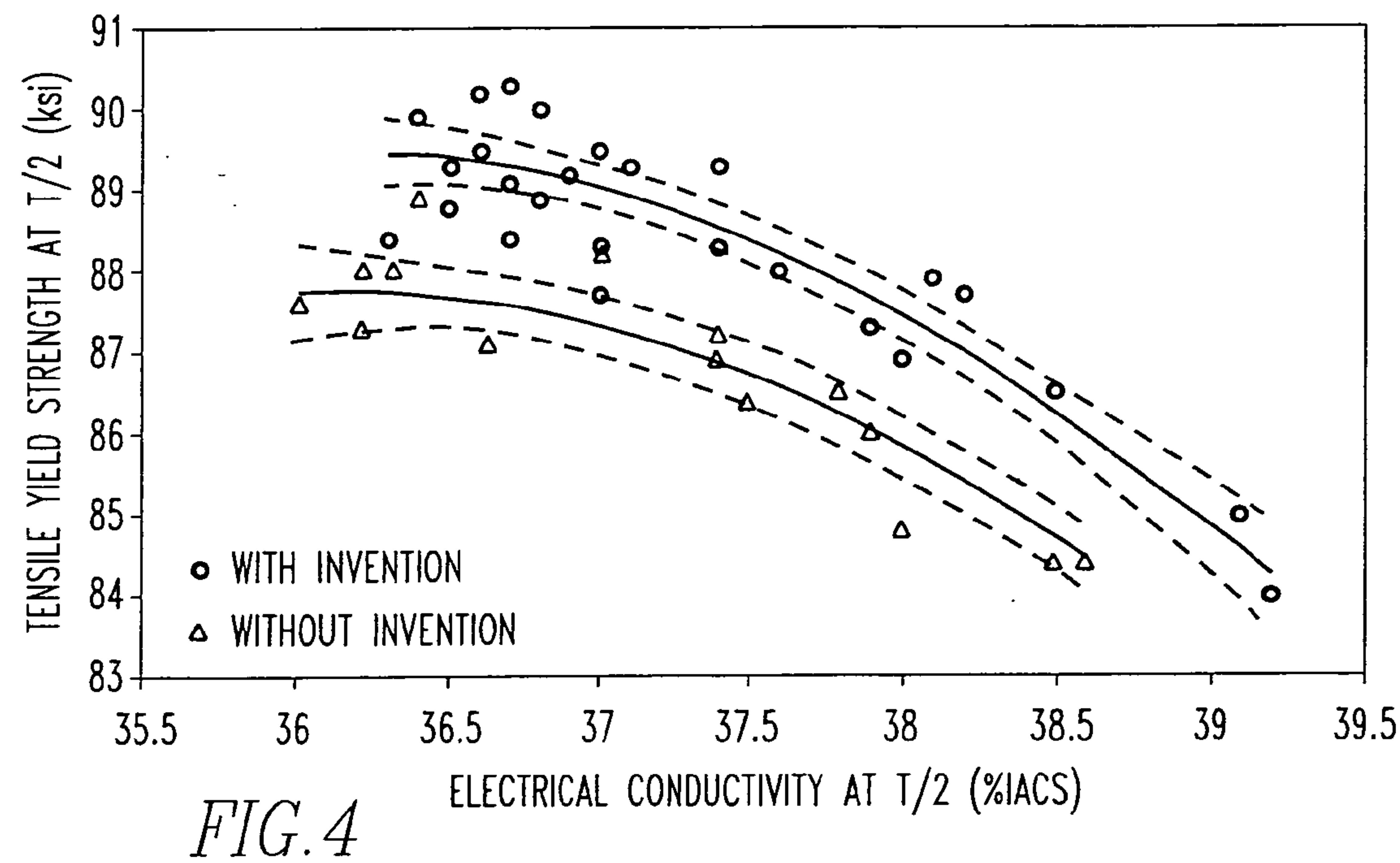


FIG. 4

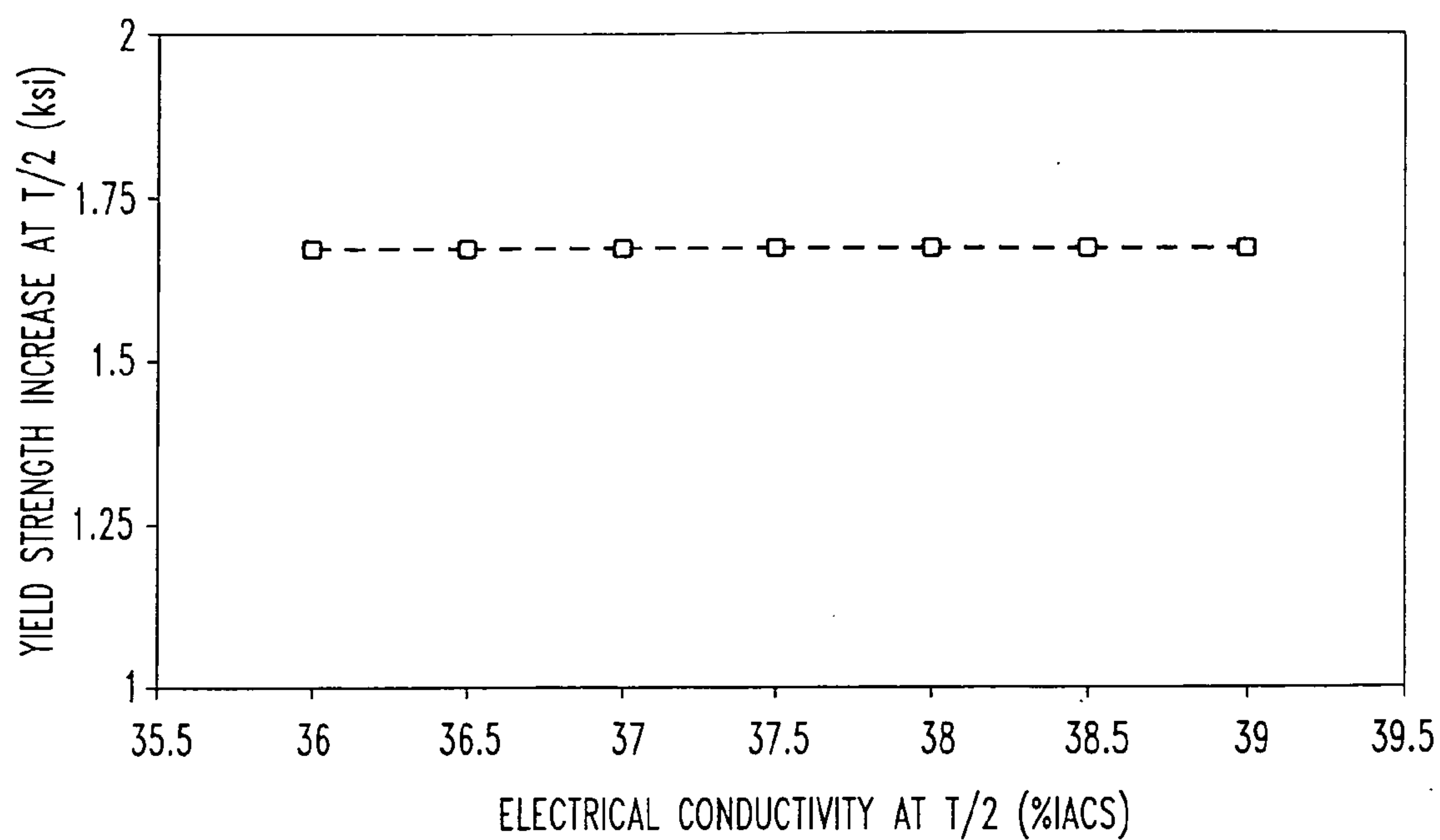


FIG. 5

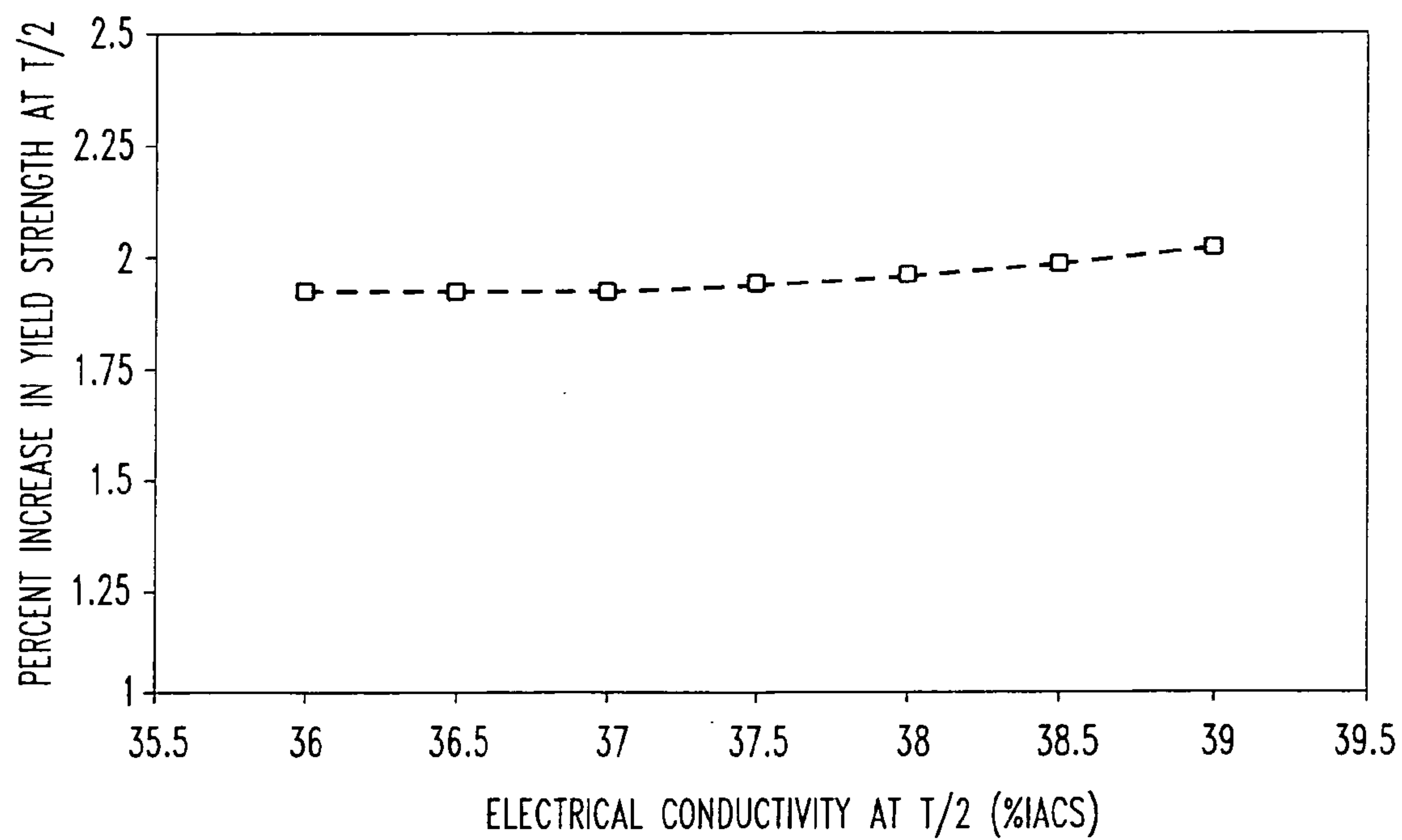


FIG. 6

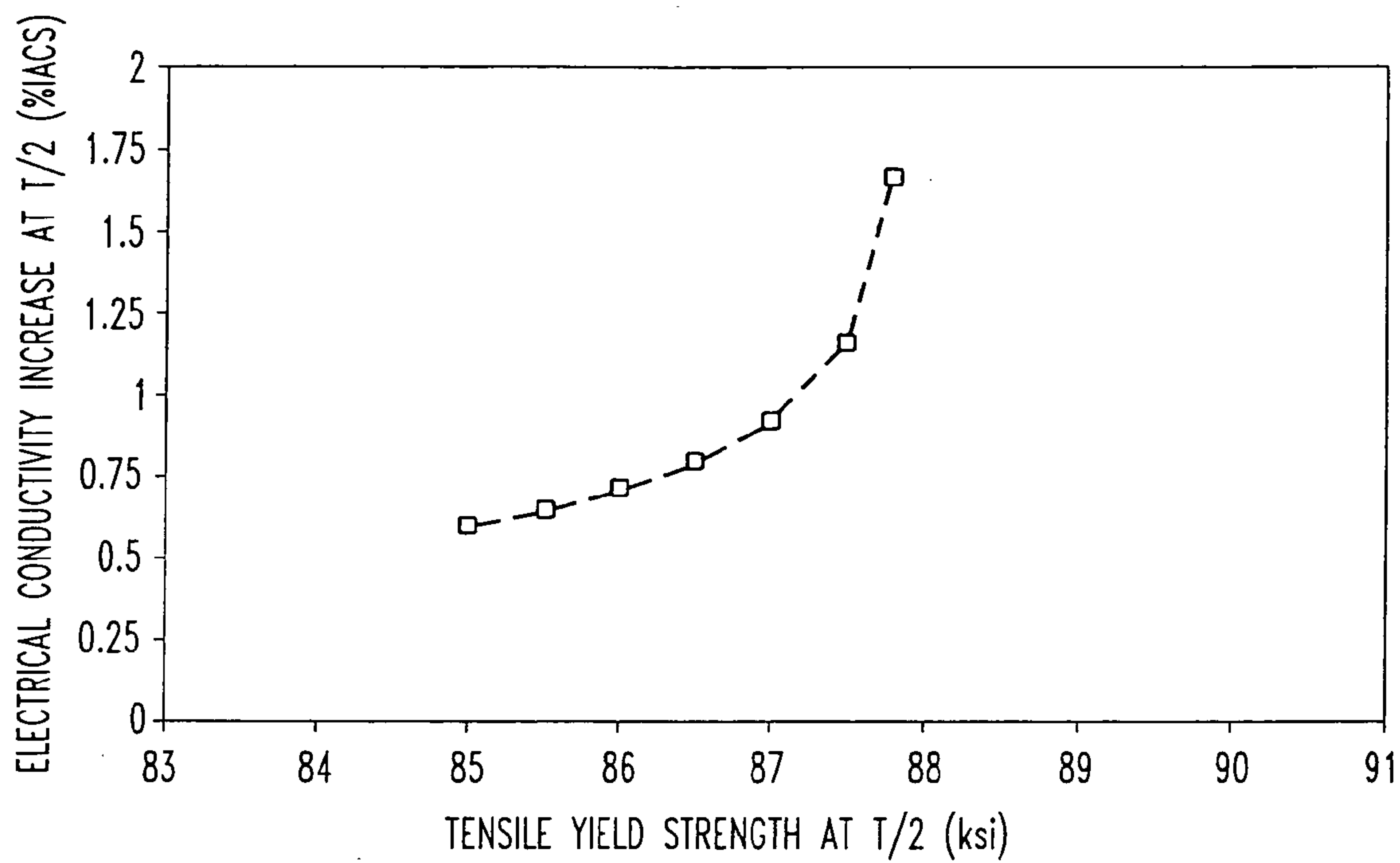


FIG. 7

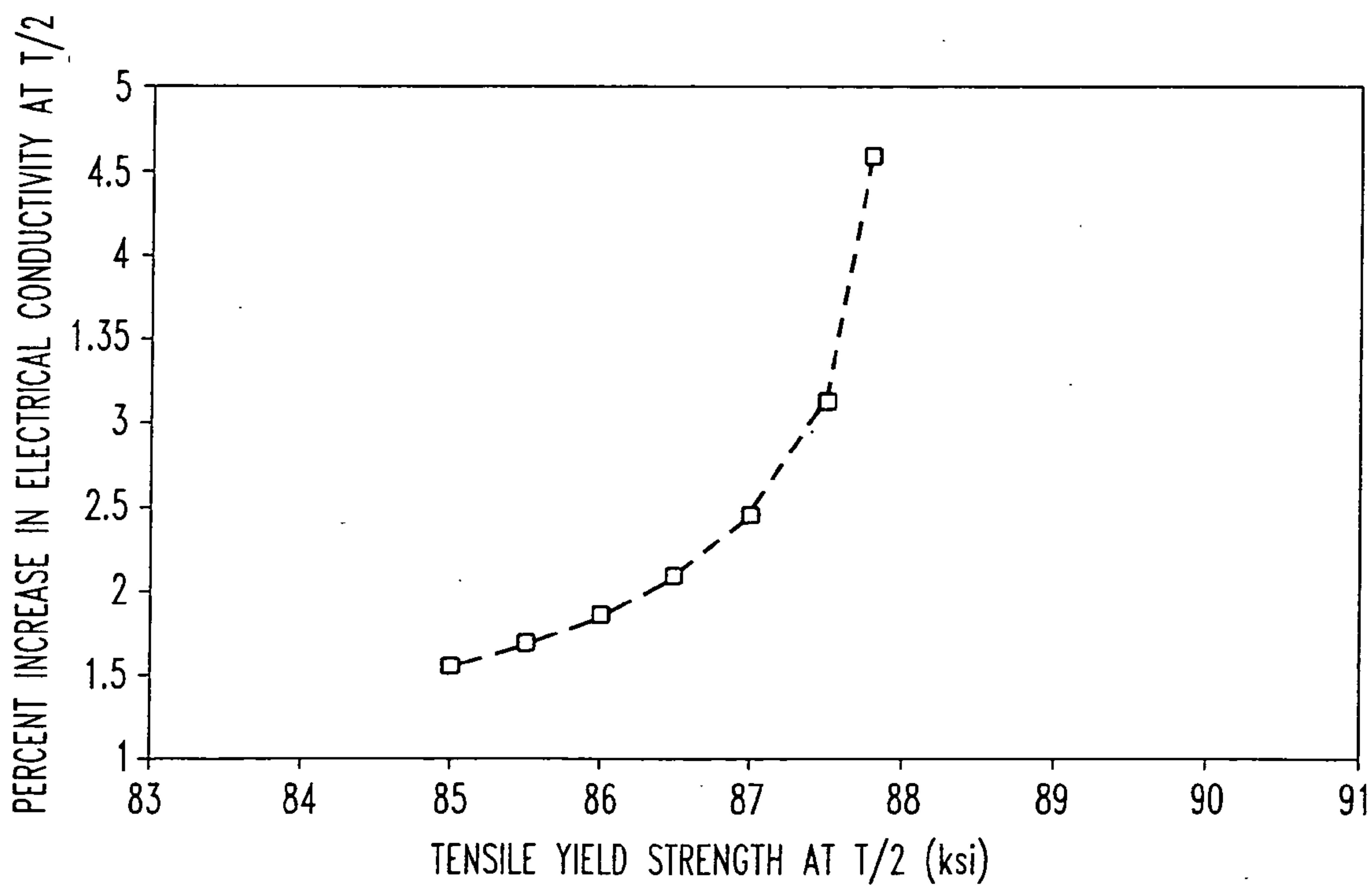


FIG. 8



# **METHOD FOR INCREASING THE STRENGTH AND/OR CORROSION RESISTANCE OF 7000 SERIES AL AEROSPACE ALLOY PRODUCTS**

## **RELATED APPLICATIONS**

[0001] This application is a continuation-in-part of U.S. patent application Ser. No. 10/103,273, filed on Mar. 20, 2002, and claims the benefit of U.S. Provisional Patent Application Ser. No. 60/277,403 filed on Mar. 20, 2001 and entitled "Age Forming Practice for Increasing Tensile Yield Strength of 7xxx-"T79" Product", the disclosures of which are fully incorporated herein by reference.

## **FIELD OF THE INVENTION**

[0002] This invention relates to the field of aluminum alloys for aerospace applications, typically 7000 Series or 7xxx alloys as designated by the Aluminum Association. More particularly, this invention relates to an improved method for imparting better yield strengths to 7000 Series aluminum alloys tempered in a known, preferred manner. This method achieves such strength improvements without detrimentally effecting corrosion resistance, particularly exfoliation corrosion resistance. Conversely, the method of this invention can be used to impart better corrosion resistance performance in these 7000 Series aluminum aerospace alloys at or about the same yield strength levels. For the sheet and plate varieties of these products, the invention may be practiced on products situated in their respective dies for further achieving some age forming improvements thereon. It is to be understood that analogous improvements in the strength/corrosion properties of 7000 Series extrusions and forgings should also take place.

## **BACKGROUND OF THE INVENTION**

[0003] The manufacturers of large commercial jetliners have been attempting to improve the performance of their current and future lines of passenger aircraft for some time. They are currently considering new plate and extrusion products for the upper wing portions of these plane models. One manufacturer has been actively seeking to improve the strength and corrosion performance of next generation materials, especially over incumbent 7150-"T79" plate products. That temper, "T79", is produced by age-forming individual pre-machined panels, typically to the desired contour part shape during artificial aging.

[0004] A typical age forming practice for large aircraft wing panels usually involves starting with a W51 tempered (solution heat treated and stress relieved) plate product. Alternately, that same W51-tempered part may be subjected to the first of several multiple step tempering practices while still flat, either by the material supplier, an intermediate distributor/handler, or the end user/customer, i.e. the ultimate aircraft manufacturer/assembler. Note that this first artificial aging step is not typically performed while the alloy material is kept in its ultimate forming die. Instead, the latter plate product is sawed and machined to a desired shape and thickness for a making given wing panel component part therefrom. That machined panel is then aligned over a forming die whereupon pressure is applied to force said panel to assume its final or near-final shape, that of the die itself. The die and panel may then be artificially aged together per prescribed practices. Alternately, this first tem-

pering in a multiple step aging practice could take place with a sawed and machined part situated "in" its forming die, after which both part and die are further artificially aged together.

[0005] A typical 7xxx age forming practice entails one or two steps. If a two step practice is used, the first step is usually performed at a lower temperature than the second. That first step is typically about 200-250° F. for about 3 to 12 hours. The second step of that two-step practice targets one or more temperatures between about 280-350° F. for about 6 to 24 hours, and in some instances for as high as 30 hours. If only a one step practice is used, that typically transpires at one or more target temperatures between about 280-320° F. for about 6 to 24 hours.

[0006] For the upper wing panels of most large aircraft, both high strength and exfoliation corrosion resistance are critical. In the typical age form practice, exfoliation corrosion resistance is known to improve with progressive over-aging. There is a corresponding decrease, or trade-off, in strength, however. As such, there is a clear industry-driven need for an improved aging practice that would provide higher strengths at about the same level of corrosion resistance, or a higher level of corrosion resistance performance at about the same strength level. This invention addresses both such industry needs.

[0007] Numerous 3-step aging practices are known for enhancing corrosion resistance without degrading the strength of 7000 Series aluminum aerospace alloys. Among these are the prior art disclosures of U.S. Pat. Nos. 3,856,584, 3,957,542; 4,477,292; 4,863,528 and 5,108,520. For some of these disclosures, a first aging step was performed at about 250° F. with a second step above about 350 or 360° F. That second step is then followed by a third step similar to their first step temperature of about 250° F. Some of these references state that their observed benefits diminish at lower, second step temperatures. A two-step practice of note is also shown and described in U.S. Pat. No. 3,881,966. By contrast, the preferred first of two, or second of three, aging practice steps of this invention proceed at a significantly lower, first or second step temperature as compared to the prior art temperings described above, lower by about 40 to 50° F. As such, the results of this invention were even more surprising since strength increases were not expected using a lower temperature aging treatment following the 300°+ practices of the preferred embodiments herein.

## **SUMMARY OF THE INVENTION**

[0008] Briefly stated, this invention relates to an improved method for artificially aging 7000 Series aluminum aerospace alloys. This method imparts improved strength performance at the same corrosion resistance performance level, or improved corrosion resistance performance at the same strength level. It accomplishes these property improvements by purposefully adding a second aging step or stage to a typical one-step tempering process, or a purposeful third step/stage to a known two-step aging operations. The purposefully added step/stage (second of two or third of three) extends at about 225-275° F. for about 3-24 hours, or more preferably at about 250° F. for about 6 hours or more. The invention especially imparts improved combinations of strength and exfoliation corrosion resistance to 7055 aluminum alloy products (Aluminum Association designation) in sheet, plate, extrusion or even forged product forms.



[0009] The invention also imparts a crystal grain size that is substantially larger than  $45\text{ }\mu\text{m}$ , ranging in size from about  $50\text{ }\mu\text{m}$  to predominantly more than  $500\text{ }\mu\text{m}$ . In addition, the aspect ratios of most of the crystals in the alloy products treated according to the invention can be seen to be substantially larger than 4:1, and range from about 4:1 for recrystallized grain in the surface layers, to predominantly on the order of about 50:1 for the unrecrystallized interior.

[0010] One commercial jetliner manufacturer's specification for 7xxx age formed upper wing panels refers to the "-T7951" temper. As of the filing date for this patent application, that temper is still not officially registered with the Aluminum Association. The standard practice for "-T7951", described above, involves a one- or two-step aging practice. In the present invention, a second step is purposefully added to the known, typical one-step aging practice for "-T79". That second step extends at about  $225\text{--}275^{\circ}\text{ F.}$  for about 3-24 hours, or more preferably at about  $250^{\circ}\text{ F.}$  for about 6 hours. With the addition of that second aging step, the inventors herein observed a surprising and significant increase in strength at the same level of corrosion resistance, especially exfoliation corrosion resistance. Another way or restating this observed improvement is that the addition of the second aging step above imparted a significant increase in corrosion resistance, especially exfoliation corrosion resistance, at about the same strength level.

[0011] Alternately, this same invention entails adding a third step to the two-step aging practice for "-T7951". That third step likewise extends at about  $225\text{--}275^{\circ}\text{ F.}$  for about 3-24 hours, or more preferably at about  $250^{\circ}\text{ F.}$  for about 6 hours. With the addition of that third aging step following a lower than usual second temperature aging practice, a surprising and significant increase in strength was observed at the same level of corrosion resistance, especially exfoliation corrosion resistance. Or restated once more, the addition of this third aging step above imparts a significant increase in corrosion resistance, especially exfoliation corrosion resistance, at about the same strength level.

[0012] In either instance, adding a second step to a one-step aging practice for 7000 Series aluminum alloys, or adding a third step to a known two-step aging practice, it should be duly noted that the "additional step" of this invention is: (1) always lower than the aging step that it follows; and (2) that preceding step, itself, whether the first of now two aging steps; or the second of now THREE aging steps, takes place at temperatures lower than what is otherwise known to be practiced for other T77 aging practices for 7000 Series alloys.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0013] FIGS. 1 (a) through (c) are graphic representations of three, 2-step aging schemes according to the invention;

[0014] FIGS. 2 (a) through (g) are graphic representations of seven representative 3-step aging schemes according to the invention;

[0015] FIG. 3 is a graph depicting the relative improvement in strength, particularly longitudinal tensile yield strength (TYS), versus electrical conductivity (in % IACS) both measured at T/2 as representative of exfoliation corrosion resistance performance, for various samples of 0.75

inch thick, 7055 plate after artificial aging by known 1- and 2-step practices (hollow triangular data points) versus the preferred aging practice of this invention to which a controlled second or third step, as appropriate was purposefully added to the aforesaid known practices (shown with solid circular data points);

[0016] FIG. 4 is the same graph of FIG. 3 through which solid curves A-A and B-B were drawn using a quadratic statistical equation approach for predicting the strength/EC slopes of the Invention versus known (1- and 2-step aged) 7055 plate product and around which 95% confidence bands were drawn in dotted lines;

[0017] FIG. 5 is a graph depicting the numerical increase in tensile yield strength (ksi) values predicted for 7055 Plate aged by the invention over its known (1- and 2-step aged) counterparts per the quadratic curves in FIG. 4 above;

[0018] FIG. 6 is a graph depicting the increase in tensile yield strength values predicted (by percent improvement) for 7055 Plate aged by the invention over its known (1- and 2-step aged) counterparts;

[0019] FIG. 7 is a graph numerically depicting the improvement in electrical conductivity (% IACS) predicted for 7055 Plate aged by the invention over its known (1- and 2-step aged) counterparts; and

[0020] FIG. 8 is a graph depicting that same improvement in predicted electrical conductivity values (by percentages) for 7055 Plate aged by the invention versus its known (1- and 2-step aged) counterparts.

#### DETAILED DESCRIPTION OF THE INVENTION

[0021] Numerous variations of aging practices according to the invention are depicted in accompanying FIGS. 1 and 2. Particularly, FIGS. 1 (a) through (c) are graphic representations of three, 2-step aging schemes according to the invention, with 1(a) representing a 2-step or staged method with a partial (air) cooling between controlled steps/stages. In FIG. 1(b), there is shown a representative 2-step method that has a controlled, furnace ramping down between first and second steps/stages. Finally, FIG. 1(c) schematically depicts a 2-step or staged method with a distinct, fully separated cooling (via air or cold water quenching "CWQ") between steps/stages.

[0022] FIGS. 2 (a) through (g) are graphic representations of seven representative 3-step aging schemes according to the invention. In FIG. 2(a), a 3-step or staged method is shown with a partial (air) cooling between controlled steps 2 and 3. FIG. 2(b) illustrates a 3-step method that has a controlled, furnace ramping down to achieve the same effect as the isothermal 3<sup>rd</sup> step described earlier. FIG. 2(c) represents a variation on 2(b) with a controlled temperature ramping up as step 1. In FIG. 2(d), a variation on 2(a) is shown with a controlled interrupted cool down between steps 1 and 2. Similarly, FIG. 2(e) depicts a variation on 2(b) with a full cool down between steps 1 and 2 and a controlled, furnace ramping down to achieve the same effect as the isothermal 3<sup>rd</sup> step described earlier. FIG. 2(f) illustrates a variation on the 3 step practice of 2(c) above, but with a distinct, fully separated cooling (via air or cold water quenching "CWQ") between steps 2 and 3. Finally, representative FIG. 2(g) shows still another variation on 2(f) with



distinct, fully separated cooling (via air or cold water quenching “CWQ”) between each of steps 1, 2 and 3. It is important to note that in each of the foregoing aging examples, both **FIGS. 1 and 2**, that the latter stages of any such practice according to the invention can be performed either in or out of a forming die.

[0023] The following examples illustrate the relative TYS strength increases observed in the practice of this invention on 7055 plate product. Samples of 0.75-inch thick 7055 plate were given various combinations of first- and second-step aging practices. [Note that when only a one step practice was supplemented per this invention, the data in Table 1 that follows actually lists a “1<sup>st</sup> Step” time and temperature as “None”. That, in effect, makes the Table 1 “2<sup>nd</sup> Step” so listed a 1<sup>st</sup> step of two, which is then followed by the 40-50° F. lower, second (of two) steps or stages per the present invention.] Some of the Table 1 samples were given an additional aging step for performance comparison purposes. Those treated samples always list this added step

in the “3<sup>rd</sup> Step” column of accompanying Table 1. But that step is meant to be the second of two, or third of three aging treatments, depending on whether a true 1<sup>st</sup> step aging was performed thereon.

[0024] Tensile yield strength, electrical conductivity and exfoliation corrosion resistance (or “EXCO”) values were measured for each Table 1 sample, the latter EXCO data per ASTM Standard No. G-34, the disclosure of which is incorporated herein. With respect to that table, it should be noted that electrical conductivity “EC” serves as an indicator of corrosion resistance, i.e., the higher the EC value measured (as a % IACS value), the more corrosion resistant that product ought to be. Ultrasonic depth of attack data gathered in conjunction with EXCO corrosion testing is also listed in accompanying Table 1. A small (or shallow) depth of attack indicates improved corrosion resistance. In almost all cases, both strength and corrosion resistance improved with the added aging practice of this invention.

TABLE 1

| Effect of Invention (added Aging Practice) on Strength & Exfoliation Resistance<br>7055, 0.75 inch plate at T/2 |                                 |                                 |                                 |                |                       |   |                                 |   |
|---|---------------------------------|---------------------------------|---------------------------------|----------------|-----------------------|---|---------------------------------|---|
| Identification<br>of<br>Experiments   | 1 <sup>st</sup> Step<br>° F./hr | 2 <sup>nd</sup> Step<br>° F./hr | 3 <sup>rd</sup> Step<br>° F./hr | EC<br>(% IACS) | Long.<br>TYS<br>(ksi) | EXCO<br>Avg. Depth of<br>Attack<br>After 48 hrs<br>(inch) | Visual Rating<br>after 48 hours | Δ TYS (ksi)<br>with invention step<br>minus w/o<br>invention step |
| 1   | 250/3                           | 300/10                          | None                            | 36.3           | 88.0                  | 0.0090  | EC                              |   |
| 1A  | 250/3                           | 300/10                          | 250/6                           | 36.7           | 89.1                  | 0.0083  | EC                              | 1.1   |
| 2   | 250/3                           | 300/17.5                        | None                            | 37.4           | 87.2                  | 0.0037  | EB                              |   |
| 2A  | 250/3                           | 300/17.5                        | 250/6                           | 37.6           | 88.0                  | 0.0047  | EC                              | 0.8   |
| 3   | 250/3                           | 310/5.5                         | None                            | 36.0           | 87.6                  | 0.0063  | EC                              |   |
| 3A  | 250/3                           | 310/5.5                         | 250/6                           | 36.4           | 89.9                  | 0.0057  | EC                              | 2.3   |
| 4   | 250/3                           | 310/9.6                         | None                            | 37.5           | 86.4                  | 0.0030  | EB                              |   |
| 4A  | 250/3                           | 310/9.6                         | 250/6                           | 38.2           | 87.7                  | 0.0023  | EB                              | 1.3   |
| 5   | None                            | 300/10                          | None                            | 36.2           | 87.3                  | 0.0040  | EC                              |   |
| 5A  | None                            | 300/10                          | 250/6                           | 36.7           | 88.4                  | 0.0060  | EC                              | 1.1   |
| 6   | None                            | 300/17.5                        | None                            | 37.8           | 86.5                  | 0.0023  | EB                              |   |
| 6A  | None                            | 300/17.5                        | 250/6                           | 37.9           | 87.3                  | 0.0017  | EB                              | 0.8   |
| 7   | None                            | 310/5.5                         | None                            | 36.6           | 87.1                  | 0.0030  | EC                              |   |
| 7A  | None                            | 310/5.5                         | 250/6                           | 36.3           | 88.4                  | 0.0063  | EC                              | 1.3   |
| 8   | None                            | 310/9.6                         | None                            | 38.0           | 84.8                  | 0.0003  | EA                              |   |
| 8A  | None                            | 310/9.6                         | 250/6                           | 38.0           | 86.9                  | 0.0030  | EB                              | 2.1   |
| 9   | None                            | 300/14                          | None                            | 37.4           | 86.9                  | 0.0043  | EC                              |   |
| 9A  | None                            | 300/14                          | 250/6                           | 38.1           | 87.9                  | 0.0027  | EC                              | 1.0   |
| 10  | None                            | 305/16                          | None                            | 38.6           | 84.4                  | 0.0030  | EB                              |   |
| 10A   | None                            | 305/16                          | 250/6                           | 39.2           | 84.0                  | 0.0027  | EB                              | -0.4  |
| 11  | 250/3                           | 305/14                          | None                            | 37.9           | 86.0                  | 0.0030  | EC                              |   |
| 11A   | 250/3                           | 305/14                          | 250/6                           | 38.5           | 86.5                  | 0.0027  | EB                              | 0.5   |
| 12  | 250/3                           | 310/14                          | None                            | 38.5           | 84.4                  | 0.0020  | EB                              |   |
| 12A   | 250/3                           | 310/14                          | 250/6                           | 39.1           | 85.0                  | 0.0017  | EB                              | 0.6   |
| 13  | 250/3                           | 302/10                          | None                            | 37.0           | 88.2                  | 0.0057  | EC                              |   |
| 13A   | 250/3                           | 302/10                          | 250/3                           | 37.0           | 89.5                  | 0.0080  | EC                              | 1.3   |
| 13  | 250/3                           | 302/10                          | None                            | 37.0           | 88.2                  | 0.0057  | EC                              |   |
| 14A   | 250/3                           | 302/10                          | 250/6                           | 37.0           | 89.5                  | 0.0057  | EC                              | 1.3   |
| 13  | 250/3                           | 302/10                          | None                            | 37.0           | 88.2                  | 0.0057  | EC                              |   |
| 15A   | 250/3                           | 302/10                          | 250/12                          | 37.4           | 89.3                  | 0.0070  | EC                              | 1.1   |
| 13  | 250/3                           | 302/10                          | None                            | 37.0           | 88.2                  | 0.0057  | EC                              |   |
| 16A   | 250/3                           | 302/10                          | 250/24                          | 36.7           | 90.3                  | 0.0070  | EC                              | 2.1   |
| 17  | 250/6                           | 302/10                          | None                            | 36.4           | 88.9                  | 0.0070  | EC                              |   |
| 17A   | 250/6                           | 302/10                          | 250/6                           | 36.6           | 90.2                  | 0.0077  | EC                              | 1.3   |
| 17  | 250/6                           | 302/10                          | None                            | 36.4           | 88.9                  | 0.0070  | EC                              |   |
| 18A   | 250/6                           | 302/10                          | 250/24                          | 36.8           | 90.0                  | 0.0070  | EC                              | 1.1   |
| 19  | None                            | 302/10                          | None                            | 36.2           | 88.0                  | 0.0057  | EC                              |   |
| 19A   | None                            | 302/10                          | 250/3                           | 36.8           | 88.9                  | 0.0083  | EC                              | 0.9   |
| 19  | None                            | 302/10                          | None                            | 36.2           | 88.0                  | 0.0057  | EC                              |   |
| 20A   | None                            | 302/10                          | 250/6                           | 36.6           | 89.5                  | 0.0080  | EC                              | 1.5   |
| 19  | None                            | 302/10                          | None                            | 36.2           | 88.0                  | 0.0057  | EC                              |   |
| 21A   | None                            | 302/10                          | 250/12                          | 37.4           | 88.3                  | 0.0063  | EC                              | 0.3   |

TABLE 1-continued

| Effect of Invention (added Aging Practice) on Strength & Exfoliation Resistance<br>7055, 0.75 inch plate at T/2 |                                 |                                 |                                 |                |                       |   |                                 |   |
|---|---------------------------------|---------------------------------|---------------------------------|----------------|-----------------------|---|---------------------------------|---|
| Identification<br>of<br>Experiments   | 1 <sup>st</sup> Step<br>° F./hr | 2 <sup>nd</sup> Step<br>° F./hr | 3 <sup>rd</sup> Step<br>° F./hr | EC<br>(% IACS) | Long.<br>TYS<br>(ksi) | EXCO<br>Avg. Depth of<br>Attack<br>After 48 hrs<br>(inch) | Visual Rating<br>after 48 hours | Δ TYS (ksi)<br>with invention step<br>minus w/o<br>invention step |
| 19  | None                            | 302/10                          | None                            | 36.2           | 88.0                  | 0.0057  | EC                              |   |
| 22A   | None                            | 302/10                          | 250/24                          | 36.9           | 89.2                  | 0.0077  | EC                              | 0.2   |
| 19  | None                            | 302/10                          | None                            | 36.2           | 88.0                  | 0.0057  | EC                              |   |
| 23A   | None                            | 302/10                          | 275/3                           | 36.5           | 88.8                  | 0.0057  | EC                              | 0.8   |
| 19  | None                            | 302/10                          | None                            | 36.2           | 88.0                  | 0.0057  | EC                              |   |
| 24A   | None                            | 302/10                          | 275/6                           | 37.0           | 88.3                  | 0.0070  | EC                              | 0.3   |
| 19  | None                            | 302/10                          | None                            | 36.2           | 88.0                  | 0.0057  | EC                              |   |
| 25A   | None                            | 302/10                          | 275/12                          | 37.0           | 87.7                  | 0.0063  | EC                              | -0.3  |
| 19  | None                            | 302/10                          | None                            | 36.2           | 88.0                  | 0.0057  | EC                              |   |
| 26A   | None                            | 302/10                          | 225/6                           | 36.5           | 89.3                  | 0.0083  | EC                              | 1.3   |
| 19  | None                            | 302/10                          | None                            | 36.2           | 88.0                  | 0.0057  | EC                              |   |
| 27A   | None                            | 302/10                          | 225/24                          | 37.1           | 89.3                  | 0.0073  | EC                              | 1.3   |

[0025] One main means for evaluating the data of Table 1 is to compare relative sample strengths at a constant electrical conductivity EC value. Accompanying FIGS. 3 through 7 facilitate such a comparison. At any given electrical conductivity value, it was noted from FIG. 3 that TYS values ran about 1.5 ksi higher when another step (the second of two or third of three steps) was employed per the present invention. An alternative evaluation from Table 1/FIG. 3 leads to another conclusion about this invention, namely that at a constant TYS value, relatively higher electrical conductivity values (and hence, relatively improved corrosion resistance performances) were observed per the added step or stage of this invention (again, the second of two or third of three steps).

[0026] Some of the data included in accompanying Table 1/FIG. 3 was based on tests performed after the filing of the U.S. provisional from which this application claims priority. In accompanying FIGS. 4 through 8, all of the foregoing comparative data was plotted for performing statistical analyses thereon using the quadratic statistical methodology commonly referred to as Analysis of Covariance (ANCOVA). The fit for this quadratic equation evaluation is summarized in the following Tables 2(a) through (c):

TABLE 2a

| Summary of Fit Quadratic Equation |           |
|-----------------------------------|-----------|
| Adjusted R <sup>2</sup>           | 86.12%    |
| Root Mean Square Error            | 0.614 ksi |

[0027]

TABLE 2b

| Analysis of Variance |    |                |             |          |
|----------------------|----|----------------|-------------|----------|
| Source               | DF | Sum of Squares | Mean Square | F Ratio  |
| Model                | 3  | 96.926         | 32.309      | 85.829   |
| Error                | 38 | 14.304         | 0.376       | Prob > F |
| C. Total             | 41 | 111.230        |             | <.0001   |

[0028]

TABLE 2C

| Parameter Estimates   |           |           |         |           |
|-----------------------|-----------|-----------|---------|-----------|
| Term                  | Estimate  | Std Error | t Ratio | Prob >  t |
| Intercept             | -633.1809 | 189.995   | -3.33   | 0.0019    |
| Invention With        | 0.8392    | 0.099     | 8.46    | <.0001    |
| Without               | -0.8392   | 0.099     | 8.46    | <.0001    |
| EC Slope              | 39.9710   | 10.135    | 3.94    | 0.0003    |
| EC <sup>2</sup> Slope | -0.55335  | 0.135     | -4.10   | 0.0002    |

[0029] Predicted TYS =  $-632.3417 + 39.9710 \cdot \text{EC} - 0.55335 \cdot \text{EC}^2$  With Invention

[0030] Predicted TYS =  $-634.0201 + 39.9710 \cdot \text{EC} - 0.55335 \cdot \text{EC}^2$  Without

[0031] TYS Increase due to Inv. 1.678 ksi over range of EC (36.0 to 39.2% IACS)

[0032] The 95% confidence intervals for these quadratically predicted strength versus EC curves, items A-A and B-B in FIG. 4, were then drawn with dotted lines in that Figure. Statistically noteworthy from those two predicted curves, A-A (and its 95% band) for the Invention versus curve B-B for the known 1- and 2-step comparative data (and its 95% band) is the lack of overlap between 95% confidence bands. That distancing between quadratically calculated curves for flat 7055 plate product further evidences the IMPROVEMENT over the prior art observed through the practice of this invention.

[0033] Using the A-A and B-B curves of FIG. 4, accompanying FIG. 5 shows the numerical increase in tensile yield strength (ksi) values predicted for 7055 Plate aged by the invention over its known (1- and 2-step aged) counterparts. FIG. 6 predicts that same improvement in strength as a function of electrical conductivity by percentage rather than in actual ksi values observed. The data supporting FIGS. 5 and 6 is found in Table 3 that follows:



TABLE 3

| Predicted Increase in Tensile Yield Strength due to Invention |                 |                       |
|---|-----------------|-----------------------|
| EC (% IACS)   | Quadratic Model |                       |
|   | (Numerical ksi) | (Percentage Increase) |
| 36  | 1.678           | 1.91                  |
| 36.5  |                 | 1.91                  |
| 37  |                 | 1.92                  |
| 37.5  |                 | 1.93                  |
| 38  |                 | 1.96                  |
| 38.5  |                 | 1.98                  |
| 39  |                 | 2.02                  |

[0034] Using electrical conductivity (“EC”) as the standard for side-by-side comparative statistical analyses, **FIG. 7** shows the numerical EC improvement predicted (in % IACS values) for the invention over its known (1- and 2-step aged) counterparts. **FIG. 8** predicts that same improvement in strength as a function of electrical conductivity by percentage rather than in actual EC (% IACS) values observed. Note that for both **FIGS. 7 and 8**, EC increase could not be determined over the entire range of tensile yield strengths due to the mathematical consequence of inverting quadratic calculations. The data supporting **FIGS. 7 and 8** is found in Table 4 that follows:

TABLE 4

| Predicted Increase in Electrical Conductivity due to Invention |                    |                       |
|--|--------------------|-----------------------|
| TYS (ksi)  | Quadratic Model    |                       |
|  | (Numerical % IACS) | (Percentage Increase) |
| 85   | 0.595              | 1.55                  |
| 85.5   | 0.642              | 1.68                  |
| 86   | 0.703              | 1.85                  |
| 86.5   | 0.787              | 2.09                  |
| 87   | 0.913              | 2.45                  |
| 87.5   | 1.152              | 3.13                  |
| 87.8 (max)   | 1.663              | 4.59                  |

[0035] In aerospace, marine, or other structural applications, it is for structural and materials engineers to select a material for a particular part based on a “weakest link” failure mode. For example, the upper wing alloy of a large aircraft is predominantly subjected to compressive stresses. There, then, stress corrosion cracking (or “SCC”) resistance is not as big a design issue. As such, upper wing skin alloys are usually made from higher strength Al alloys having relatively lower SCC resistance levels. Within that same wing box assembly, the spar members that get subjected to greater tensile stresses than compressive stresses. Such spar members are traditionally made from more corrosion resistant but lower strength temper materials such as those aged by known T74-type practices.

[0036] Wing skins are typically made from thinner gauge plates as compared to the wing spars made from thick plate products. Thinner gauge plate products possess thin, narrow width grains brought about by greater rolling reductions. Such grains tend to be highly laminated. Unfortunately, corrosion induces delamination along these grain boundaries during service. Hence, resistance to exfoliation corrosion is an important requirement for the upper wing skins of today’s

larger aircrafts. As with SCC, exfoliation resistance improves with progressive overaging. This invention attempts to maintain exfoliation corrosion resistance performance while still managing to improve strength values, particularly those of a TYS variety. Alternately, this invention will impart improved exfoliation corrosion resistance performance at or about the same strength value levels.

[0037] Microstructural properties two samples of 7055 alloy treated according to the invention were also observed. Two plates were prepared in accordance with the invention from two 7055 alloy plates, 1.0 inch and 1.4 inches thick respectively. These plates were produced using standard commercial practices for casting, homogenization, rolling, solution heat-treating and quenching. The plates were then artificially aged according to the invention. Specifically, the samples were subjected to logarithmic heating up to 302° F. over 5 hours, held at 302° F. for 10 hours, cooled to 250° F. in 2 hours, and held at 250° F. for 6 hours. The plates were then allowed to air cool to room temperature.

[0038] Samples of the plates were then prepared for analysis in the L-ST and LT-ST planes using standard polishing procedures. The samples were polished and electro-etched at 35 V for 90 seconds using Barkers reagent (3% HBF<sub>3</sub>), and observed at various magnifications using an Axiomat Metallograph (Zeiss, West Germany). Observations were made at the sample surface and at T/2 using 50× and 100× magnification using the metallograph’s scale.

[0039] The microstructure depicted in these exhibits was understood to be typical of 7055 plates produced under commercial conditions and subjected to the artificial aging process of the invention. It was observed that the structure of the samples was predominately un-recrystallized except at the surface, and the grains are elongated in both the LT and the L directions.

[0040] In sample 1, the typical grain was observed to be about 500 μm or more in the L direction, both at the surface and in the interior (T/2) of the sample. Similarly, in the LT direction, the majority of grains were about 50 μm to 200 μm at the surface, and about 200 μm to about 500 μm in the interior (T/2). In short, the predominant size of the grain in this sample was substantially greater than 45 μm.

[0041] In sample 2, the typical unrecrystallized grain was about 500 μm or more in the L direction, both at the surface and in the interior (T/2) of the sample. In the LT direction, the majority of grains at the surface are above about 50 μm, and 50 μm to 200 μm in the interior. In short, the predominant size of the grains in this sample was substantially greater than 45 μm.

[0042] The observed aspect ratio of the length of grain in the L direction to the length in the ST direction was also substantially greater than 4 to 1 in most cases, and, for both samples, was estimated to range from about 10:1 to about 50:1.

[0043] While most of the data herein was performed on 7055 aluminum (Aluminum Association designation), particularly that artificially aged per known “T79” practices, the method of this invention is also suitably practiced on still other 7xxx or 7000 Series, aluminum aerospace alloys, including but not limited to: 7050, 7150, 7085, 7449, even 7075 aluminum. Restated, this invention would best be practiced on an aluminum alloy containing about 5 to 10 wt.



% Zn, about 1 to 3 wt. % Mg and about 1 to 3 wt. % Cu as its main alloying constituents, with supporting elements, like Zr, Cr and/or Sc, and grain refining additives like Ti, B and/or C added thereto.

[0044] It should be further noted that when the method of this invention includes adding a third aging step to a known two step aging practice, like "T79" tempering, it is not always necessary to practice the invention in separate, distinct stages. In other words, the method of this invention may just as easily be practiced on an aging operation that includes slowly ramping up, in a controlled manner, through one or more, first stage temperatures without any true stopping, or holding point. By gradually passing through the first "stage", one may still accomplish the effects of a first heat treatment temperature without really imposing a separately distinct furnace operation thereon.

[0045] Conversely, the same effect of this method may be achievable by slowly, yet controllably, ramping down from the first of two, or second of three heat treatment steps/stages without having a purposeful cooling off period or quench (air, cold water or otherwise) thereafter. The same relative property improvements may be observed ramping controllably down from the higher, preceding heat treatment (either the first of two; or second of three) stage and through the preferred added heat treatment times and temperatures of this invention ultimately achieving a total, cumulative effect of 7000 Series aluminum alloy product exposure of about 225-275° F. for about 3-24 hours.

[0046] While the present invention is satisfied by embodiments in many different forms, there is shown in the drawings and, described herein in detail, the preferred embodiments of the invention, with the understanding that the present disclosure is to be considered as exemplary of the principles of the invention and is not intended to limit the invention to the embodiments illustrated. Various other embodiments will be apparent to and readily made by those skilled in the art without departing from the scope and spirit of the invention. The scope of the invention will be measured by the appended claims and their equivalents. Having described the presently preferred embodiments, it is to be understood that the invention may be otherwise embodied within the scope of the appended claims.

What is claimed is:

1. A method for imparting improved strength at about the same corrosion resistance performance level to an aluminum aerospace alloy product selected from the group consisting of 7050, 7055, 7075, 7085, and 7150 (Aluminum Association designation) that has been artificially aged at one or more temperatures between about 290-330° F. for about 2-30 hours, said method comprising:

(a) performing an additional aging equivalent to about 225-275° F. for about 3-24 hours after the preceding, higher temperature artificial aging,

wherein the crystal grain size that is substantially larger than about 50  $\mu\text{m}$ .

2. The method of claim 1 wherein said preceding, artificial aging includes heating the alloy product between about 295-310° F. for about 4-18 hours.

3. The method of claim 1 wherein said preceding, artificial aging includes a typical "T79" tempering.

4. The method of claim 3, wherein the majority of grains are from about 50  $\mu\text{m}$  to 200  $\mu\text{m}$  at the surface of the alloy product.

5. The method of claim 2, wherein the majority of the grains range in size from about 200  $\mu\text{m}$  to about 500  $\mu\text{m}$  in the interior (T/2).

6. An aluminum alloy product produced by the method of claim 1.

7. The method of claim 1 wherein step (a) includes heating the alloy product for at least about 6 hours at about 250° F.

8. The method of claim 1 wherein step (a) is preceded by an air or cold water quenching.

9. The method of claim 1 wherein said preceding, artificial aging ramps down controllably through additional aging step (a).

10. The method of claim 1 wherein said alloy product is sheet or plate.

11. The method of claim 3 wherein said alloy product is an aerospace extrusion.

12. An alloy product produced by the method of claim 3.

13. The method of claim 1 wherein step (a) is performed with the alloy product in a forming die.

14. A method for imparting improved corrosion resistance performance at about the same strength level to an aluminum aerospace alloy product of the 7XXX series of alloys (Aluminum Association designation) artificially aged at one or more temperatures between about 290-330° F., said method comprising:

(a) performing an additional aging equivalent to about 225-275° F. for about 3-24 hours after the preceding, higher temperature artificial aging,

wherein, the aspect ratios of crystals in the alloy product are substantially larger than 4:1 in the recrystallized grain in the surface layer to predominantly on the order of about 50:1 for the unrecrystallized interior of the product.

15. The method of claim 14 wherein the preceding, artificial aging includes heating the alloy product between about 295-310° F. for about 4-18 hours.

16. An aluminum alloy product produced by the method of claim 14.

17. The method of claim 14, wherein the crystal grains range in size from about 200  $\mu\text{m}$  to about 500  $\mu\text{m}$  in the interior (T/2).

18. The method of claim 14 wherein step (a) includes heating the alloy product for at least about 6 hours at about 250° F.

19. The method of claim 14 wherein said higher temperature, artificial aging step ramps gradually down and through said additional aging step (a).

20. A method for improving the strength and/or corrosion resistance performance of aluminum alloy plate product comprising about 5 to 10 wt. % Zn, about 1 to 3 wt. % Mg and about 1 to 3 wt. % Cu, said method comprising:

(a) artificially aging and die forming said plate product at one or more temperatures between about 290-330° F. for about 2-30 hours, and

(b) performing an additional aging on said plate product equivalent to about 225-275° F. for about 3-24 hours, wherein the crystal grains range in size from about 200  $\mu\text{m}$  to about 500  $\mu\text{m}$  in the interior (T/2) and the aspect ratios of crystals in the alloy product are substantially

larger than 4:1 in the recrystallized grain in the surface layer to predominantly on the order of about 50:1 for the unrecrystallized interior of the product.

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