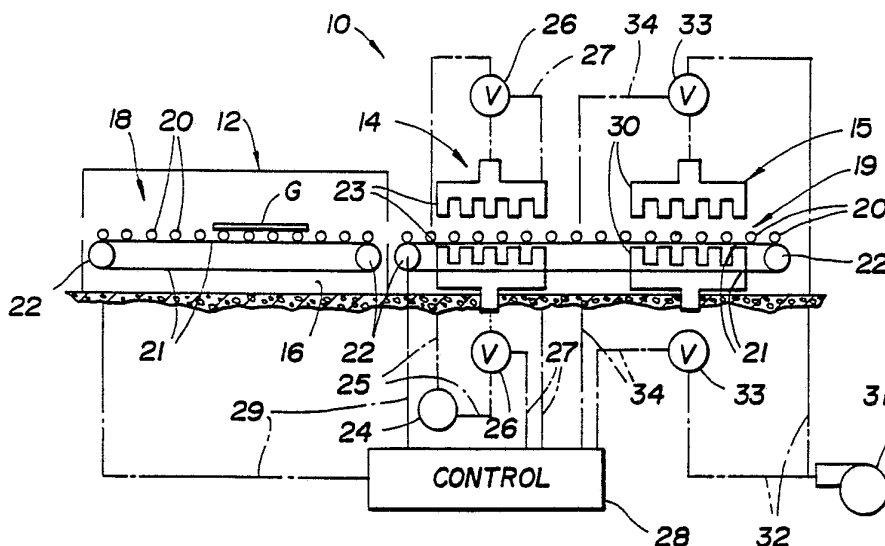




INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<p>(21) International Application Number: PCT/US89/04021 (22) International Filing Date: 15 September 1989 (15.09.89) (30) Priority data: 251,194 29 September 1988 (29.09.88) US (71) Applicant: GLASSTECH, INC. [US/US]; 995 Fourth Street, Ampoint Industrial Park, Perrysburg, OH 43551 (US). (72) Inventors: GARDON, Robert ; 28405 Eastbrook Court, Farmington Hills, MI 48018 (US). BUENO, Alejandro, G. ; 7209 Manore Road, Whitehouse, OH 43571 (US). SHETTERLY, Donovan, M. ; 3303 Gallatin Road, Toledo, OH 43606 (US).</p>		<p>(74) Agent: KUSHMAN, James, A.; Brooks & Kushman, 2000 Town Center, Suite 2000, Southfield, MI 48075 (US). (81) Designated States: AT (European patent), AU, BE (European patent), BR, CH (European patent), DE (European patent), DK, FI, FR (European patent), GB (European patent), IT (European patent), JP, KR, LU (European patent), NL (European patent), NO, SE (European patent). Published <i>With international search report.</i> <i>With amended claims and statement.</i></p>

(54) Title: **GLASS SHEET MODULATED QUENCHING**



(57) Abstract

A glass sheet tempering method and resultant glass sheet are disclosed as being provided by modulated quenching that initially cools oppositely facing surfaces of a conveyed glass sheet (G) at a first cooling station (14) with a first rate of heat transfer for a finite time to cool the surfaces from tempering temperature to below the strain point without cooling the center of the glass sheet below the strain point. Thereafter the oppositely facing surfaces of the conveyed glass sheet are cooled within a second cooling station (15) at a second rate of heat transfer less than the first rate to initially cool the center of the glass sheet below the strain point without corresponding surface cooling, and thereafter to cool the center and the surface. The modulated quenching provides a relatively large ratio of surface compression to center tension without excessive magnitudes of stress.

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GLASS SHEET MODULATED QUENCHING**TECHNICAL FIELD**

This invention relates to tempering of glass sheets.

5

BACKGROUND ART

Glass sheets are tempered to provide resistance to breakage. This tempering is performed by first heating the glass sheets to the tempering temperature which is normally a minimum of about 590°C but preferably 600 to 620°C or higher without being so hot that the glass sheet becomes too supple to control shape or too soft so as to be easily marked. The heated glass sheet is then suddenly cooled to establish a thermal gradient between the glass surfaces and the center. This thermal gradient is maintained during cooling below the strain point where stresses are essentially permanent, normally about 520°C, and require time periods of minutes for any stress to be relieved. Upon final cooling of the entire glass sheet to ambient temperature, the glass surfaces are in compression and the center is in tension. The surface compression makes the tempered glass sheet more resistant to breakage since most fractures occur in tension at the glass surface and thus must first overcome the compressive stress at the surface before breakage can take place.

Conventionally tempered glass sheets upon breakage fracture into many small fragments that are dull and relatively harmless as opposed to breaking into long shards. Recently, there has been a demand by the architectural industry for glass sheets with resistance to breakage as well as the capability of being main-

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tained within a peripheral frame after breakage as can be done with annealed glass that breaks into shards as opposed to many fragments. Such a glass sheet that is resistant to breakage and does not break into small
5 fragments may also be desirable for vehicle windshields so that the broken windshield will still permit viewing of the roadway so that the vehicle can be operated before the windshield is replaced.

While the ratio of the surface compression to
10 center tension in glass sheets has often been regarded as being more or less fixed at about 2.0 for tempered glass, values of 2.6 have been experimentally obtained with air tempering but require that the glass be heated substantially higher than the normal tempering
15 temperature. Also, the surface compression and center tension are both relatively high with such higher values of the ratio such that the glass dices upon breakage due to its high center tension. Thus, merely tempering glass with a high surface compression to center tension
20 ratio without individually controlling surface compression and center tension is of less value than when those stresses can be individually controlled.

DISCLOSURE OF INVENTION

An object of the present invention is to
25 provide an improved glass sheet tempering method and a resultant tempered glass sheet with surface compression and center tension that cannot be achieved with conventional tempering.

In carrying out the above object, the improved
30 method provides tempering of a glass sheet heated to tempering temperature and is performed by initially conveying the heated glass sheet through a first cooling

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station that cools the oppositely facing surfaces of the glass sheet with a first rate of heat transfer for a finite time to cool the surfaces below the strain point without cooling the center of the glass sheet below the strain point. Thereafter, the glass sheet is conveyed through a second cooling station that cools the oppositely facing surfaces of the glass sheet at a second rate of heat transfer less than the first rate to cool the center of the glass sheet below the strain point and also further cool the surfaces. By such a modulated quenching, the glass sheet upon cooling to ambient temperature has surface compression and center tension that cannot be achieved with conventional tempering at a uniform rate of cooling. A quick change from the first rate of heat transfer to the second lesser rate of heat transfer can be achieved by the glass sheet conveyance as described between the first and second cooling stations.

In the preferred practice, the second rate of heat transfer at the second cooling station is of a magnitude such that the surface temperature initially increases without going substantially above the strain point prior to subsequently cooling.

The first rate of heat transfer and finite time thereof and the second rate of heat transfer are preferably selected with respect to the thickness of the heated glass sheet and its tempering temperature to provide center tension sufficiently low so as to prevent dicing of the glass sheet upon fracture and to also prevent spontaneous fracturing of the cooled glass sheet such as can take place due to nickel sulfide inclusions with conventionally tempered glass sheets having a greater center tension.

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The present invention also contemplates the resultant glass sheet which is conveyed for heating and quenching by this modulated quenching method to provide the resultant stress pattern.

5 The objects, features, and advantages of the present invention are readily apparent from the following detailed description of the best mode for carrying out the invention as described in connection with the drawings.

10

BRIEF DESCRIPTION OF DRAWINGS

FIGURE 1 is a schematic view of a glass sheet tempering system for performing the method of the present invention to produce the tempered glass sheets thereof;

15

FIGURE 2 is a diagrammatical view illustrating the steps of the method;

20 FIGURE 3 is a graph illustrating the rates of heat transfer during the tempering wherein a first stage of cooling is at a greater rate than a second stage during modulated quenching that tempers the glass sheet;

25 FIGURE 4 is a diagrammatical graph superimposed on the thickness of the tempered glass sheet to illustrate the stresses which result from the modulated quenching with the initial greater rate of cooling and the subsequent lesser rate of cooling as illustrated in Figure 3; and

FIGURE 5 is a graph with solid line curves that illustrate the temperature of the glass sheet surfaces and center as the cooling is performed initial-

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ly at the greater rate and subsequently at the reduced rate and also shows a phantom line curve that represents the center temperature with cooling at a uniform rate in accordance with conventional tempering.

5

BEST MODE FOR CARRYING OUT THE INVENTION

With reference to Figure 1 of the drawings, a schematically illustrated glass sheet tempering system is generally indicated by 10 and includes a furnace 12 as well as first and second quench or cooling stations 14 and 15 mounted adjacent each other on the factory floor 16. Furnace and quench roller conveyors 18 and 19 of the system each include rolls 20 that are rotatively driven by friction with associated continuous drive loops 21 trained over wheels 22 in accordance with the teachings of U.S. Patents 3,806,312; 3,934,970; 3,947,242; and 3,994,711, the disclosures of which are hereby incorporated by reference, to provide conveyance of glass sheets G through the furnace 12 and the cooling stations 14 and 15 from the left toward the right as illustrated. Within the furnace 12, the conveyed glass sheet G is heated from ambient temperature to the tempering temperature, which normally must be at least 590°C and preferably above 600°C in the most preferred range of 620°C to 630°C or above without being too hot so the glass sheet is easily marked or too supple to maintain its shape. Thereafter, the heated glass sheet is conveyed from the furnace conveyor 18 to the quench conveyor 19 for conveyance through the first cooling station 14 between upper and lower blastheads 23 having nozzles that are fed compressed gas from a source 24 through conduits 25 controlled by valves 26 operated by connections 27 from a suitable control 28. This control 28 also has connections 29 to the furnace and quench conveyors 18 and 19 to coordinate the conveyance during

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indexing from the furnace 12 to the first cooling station 14 and to also permit independent operation of the two conveyors before and after such indexing. During the indexing into and conveyance through the first cooling station 14, the compressed gas fed to the blastheads 23 provides cooling gas to the upper and lower surfaces of the glass sheet with a sweep type motion during a first stage of quenching as is hereinafter more fully described. The second cooling station 15 has upper and lower blastheads 30 having nozzles that are fed pressurized quenching gas of a lower pressure from a blower 31 through conduits 32 controlled by valves 33 operated by connections 34 to the control 28.

The glass sheet tempering of the present invention is performed by the steps illustrated in the diagrammatical view of Figure 2 where the tempering cycle is begun by an initial heating step 42 which is performed in the furnace 12 as previously described. After the heating of the glass sheet to the tempering temperature so that the tempering can be performed, the glass sheet is then initially conveyed through the first cooling station 14 for a first stage of cooling 44 and is thereafter quickly conveyed from the first cooling station 14 to the second cooling station 15 for rapidly switching to a second stage of cooling 46. During the initial stage of cooling 44, the oppositely facing surfaces of the conveyed glass sheet are cooled with a first rate of heat transfer for a finite time, about 1 to 3 seconds for thin glass of approximately 3 millimeter thickness and as great as about 3-10 seconds for thicker glass, to cool the surfaces below the strain point without cooling the center of the glass sheet below the strain point. Thus, after completion of the first stage of cooling 44, the glass sheet surfaces will be hardened so as to act essentially as an elastic solid

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with respect to maintaining stresses, while the glass center at a higher temperature above the strain point will still act essentially as a viscous liquid with respect to relieving so as to relieve any stresses that may be caused by differences in cooling due to the thermal gradient between the surfaces and the center. Subsequently, after a quick transition that happens almost instantaneously by the conveyance, the oppositely facing surfaces of the conveyed glass sheet are cooled during the second stage at a second rate of heat transfer less than the first rate to cool the center of the glass sheet below the strain point without corresponding cooling of the surfaces. Subsequently, the second stage cooling further cools the center and the surfaces. During this second stage cooling, the quench conveyor can oscillate the glass sheet back and forth in the second cooling station for the required cooling time. Thereafter, a final cooling step provides cooling of the glass sheet to ambient temperature such that the glass sheet has surface compression and center tension that cannot be achieved with conventional tempering at a uniform rate of cooling. This final cooling step can be performed by after-coolers downstream from the cooling station or by natural convection of ambient air.

Adjustment of the modulated quench is done by changing the first greater rate of cooling, the second rate of cooling, and the finite time of the first greater rate of cooling, the latter of which is controlled by the speed of conveyance and the length of the blastheads of the first cooling station. Such adjustment enables the modulated quenching to provide compression at the glass surfaces and tension at the glass center as shown in Figure 4 by the solid line indicated stress curve which is shown in comparison

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to the phantom line indicated stress curve 56 that results from cooling at a uniform rate. It will be noted that the stress curve 54 that results from the modulated quenching has surface compression of the same
5 magnitude as with conventional quenching but has a lesser center tension so as to have less tendency to break into small fragments upon fracture than is the case with conventional quenching at a uniform rate
10 throughout cooling of both the surfaces and the center from the tempering temperature to below the strain point. Also, it will be noted that the neutral planes of no stress are closer to the respectively adjacent surfaces than with conventional quenching.

It is believed that maintaining the center
15 tension below about 35 MegaPascals prevents dicing of 3 millimeter thick glass and also prevents spontaneous fracturing such as can occur from nickel sulfide inclusions in glass of that thickness. It is also believed that even better results can be achieved when the center
20 tension is maintained at or below about 30 MegaPascals for 3 millimeter thick glass in order to prevent dicing and spontaneous fracturing. For 6 millimeter thick glass, the center tension is preferably maintained below 30 MegaPascals and most preferably below 25 MegaPascals
25 to prevent dicing and spontaneous fracturing. The center tension thus must be lower for thicker glass. It is also possible for any given center tension to obtain a greater magnitude of surface compression with the modulated quenching than can be achieved with conventional
30 quenching using a uniform rate of cooling.

As shown in Figure 5, the temperature of the glass sheet surfaces 50 during the first stage of cooling 44 rapidly decreases from the tempering temperature which is illustrated at about 630°C. This rapid

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decrease in the surface temperature proceeds until the transition 45 to the second stage of cooling 46 where the rate of cooling is quickly decreased as permitted by the switching function resulting from the conveyance of the glass sheet from the first cooling station 14 to the second cooling station 15. In an ideal situation, the surfaces would be cooled just down to or slightly below the strain point by the first stage of cooling and would be maintained there during the initial part of the second stage cooling as the center continues to cool. This cooling of the surfaces without concomitant cooling of the center reduces the ultimate center tension in relationship to the surface compression. However, such ideal processing cannot be easily performed. Rather, the first stage cooling in practice cools the surfaces slightly below the strain point, about 20°C as illustrated, and thereafter the second rate of heat transfer is of a magnitude such that the center continues cooling while the surface temperature increases from the point 58 for a short period of time. This interim surface temperature increase proceeds without going back substantially above the strain point 59 so that the glass surfaces do not relieve their stresses and thus lose the temper that has been induced into the glass sheet. Upon the interim surface temperature increase back up to a point 60, the surface temperature again begins to decrease but at a much slower rate than is the case with conventional tempering at a uniform cooling rate as shown by phantom line curve 62. During the initial stage of cooling 44, the temperature of the glass sheet center 52 decreases but is still above the strain point 59 upon reaching the transition 45 unlike the surfaces 50 which are below the strain point as previously discussed. During the second stage of cooling 46 at a lower rate of heat transfer, the center 52 of the glass sheet continues to cool but has a slower rate as shown

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by curve 64 than is the case with conventional tempering at a uniform cooling rate as shown by the phantom line curve 66.

5 It should be appreciated that the modulated
quenching performed in accordance with the present
invention to produce the types of glass sheets thereof
as described above differs from the prior art in the
manner in which the surface compression and center
10 tension can be individually adjusted with respect to
each other in order to achieve the desired result. As
previously mentioned, the cooling of the surfaces from
tempering temperature to below the strain point must
take place with the center still above the strain point
as the transition takes place from the first rate of
15 cooling to the second rate of lesser cooling. The
parameters which affect the final stress distribution
include: the magnitudes of the first and second rates
of cooling, the temperature of the glass prior to the
initial cooling, the time period of the initial cooling
20 as controlled by the speed of conveyance and the length
of the first cooling station, the thickness of the
glass, and the type of glass insofar as its chemistry
affects the strain point range and the heat transfer
through the thickness of the glass sheet.

25 In testing conducted on 30 centimeter by 30
centimeter glass samples 6 millimeter thick heated to
640°C and 625°C, the first stage of tempering was
performed for times between 1 and 3.5 seconds with the
best results achieved at about 2 seconds. First stage
30 cooling rates of 100 to 135 x 10⁻⁴ cal/cm²-sec-°C were
used for the 6 millimeter thick glass at 640°C and of 80
to 100 x 10⁻⁴ cal/cm²-sec-°C were used for the 6 millime-
ter thick glass at 625°C. Second stage cooling rates of
about 14 x 10⁻⁴ cal/cm²-sec-°C were used for the 6

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millimeter thick glass at 640°C and of about 15×10^{-4} cal/cm²-sec-°C for the 6 millimeter thick glass at 625°C.

In testing conducted on 30 centimeter by 30 centimeter glass samples 3 millimeter thick heated to 5 640°C, first and second stage cooling rates of 120 and 30×10^{-4} cal/cm²-sec-°C were used with the first stage lasting for 1 to 2.5 seconds.

Both the 6 millimeter and 3 millimeter thick glass processing had the first stage cooling provided by 10 compressed gas at about .20 to .35 MegaPascals and second stage cooling provided by blower gas pressurized at about 1 to 1.25 KiloPascals.

While the best mode for carrying out the invention has been described in detail, those familiar 15 with the art to which this invention relates will recognize various alternative designs and embodiments for carrying out the invention as described by the following claims.

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WHAT IS CLAIMED IS:

1. A method for tempering a glass sheet heated to tempering temperature, the method comprising:
initially conveying the heated glass sheet
5 through a first cooling station that cools the oppositely facing surfaces of the glass sheet with a first rate of heat transfer for a finite time to cool the surfaces below the strain point without cooling the center of the glass sheet below the strain point; and
10 thereafter conveying the glass sheet through a second cooling station that cools the oppositely facing surfaces of the glass sheet at a second rate of heat transfer less than the first rate to cool the center of the glass sheet below the strain point and
15 further cool the surfaces, whereby the glass sheet upon ultimate cooling to ambient temperature has surface compression and center tension that cannot be achieved with conventional tempering at a uniform rate of cooling.
- 20 2. A method as in claim 1 wherein the second rate of cooling at the second cooling station is of a magnitude such that the surface temperatures initially increase without going substantially above the strain point prior to subsequently cooling.
- 25 3. A method as in claim 1 or 2 wherein the first rate of heat transfer and finite time thereof and the second rate of heat transfer are selected with respect to the thickness of the heated glass sheet and its temperature to provide center tension sufficiently
30 low so as to prevent dicing of the glass sheet upon fracture.

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4. A method as in claim 1 or 2 wherein the first rate of heat transfer and finite time thereof and the second rate of heat transfer are selected with respect to the thickness of the heated glass sheet and
5 its temperature to provide center tension sufficiently low to prevent spontaneous fracturing of the cooled glass sheet.

5. The invention comprising: a tempered glass sheet having surface compression and center
10 tension provided by:

heating the glass sheet to tempering temperature;

subsequently cooling the oppositely facing surfaces of the glass sheet with a first rate of heat
15 transfer for a finite time to cool the surfaces below the strain point without cooling the center of the glass sheet below the strain point; and

thereafter cooling the oppositely facing surfaces of the glass sheet at a second rate of heat
20 transfer less than the first rate to cool the center of the glass sheet below the strain point and further cool the surfaces, the second rate of heat transfer being of a magnitude such that the surface temperature initially increases without going back substantially above the
25 strain point prior to subsequently cooling, whereby the glass sheet upon ultimate cooling to ambient temperature has surface compression and center tension that cannot be achieved with conventional tempering at a uniform rate of cooling.

AMENDED CLAIMS

[received by the International Bureau
on March 1990 (08.03.90);
original claims 1-5 replaced by amended claims 1-4 (2 pages)]

1. A method for tempering a glass sheet heated to tempering temperature, the method comprising: initially conveying the heated glass sheet
5 through a first cooling station that cools the oppositely facing surfaces of the glass sheet with a first rate of heat transfer for a finite time to cool the surfaces below the strain point without cooling the center of the glass sheet below the strain point; and
10 thereafter conveying the glass sheet through a second cooling station that cools the oppositely facing surfaces of the glass sheet at a second rate of heat transfer less than the first rate to cool the center of the glass sheet below the strain point and
15 further cool the surfaces, the second rate of cooling at the second cooling station being of a magnitude such that the surface temperatures initially increase without going substantially above the strain point prior to subsequently cooling, whereby the glass sheet upon
20 ultimate cooling to ambient temperature has surface compression and center tension that cannot be achieved with conventional tempering at a uniform rate of cooling.

2. A method as in claim 1 wherein the first
25 rate of heat transfer and finite time thereof and the second rate of heat transfer are selected with respect to the thickness of the heated glass sheet and its temperature to provide center tension sufficiently low so as to prevent dicing of the glass sheet upon
30 fracture.

3. A method as in claim 1 wherein the first rate of heat transfer and finite time thereof and the second rate of heat transfer are selected with respect to the thickness of the heated glass sheet and its
5 temperature to provide center tension sufficiently low to prevent spontaneous fracturing of the cooled glass sheet.

4. The invention comprising: a tempered glass sheet having surface compression and center
10 tension provided by:

heating the glass sheet to tempering temperature;

subsequently cooling the oppositely facing surfaces of the glass sheet with a first rate of heat
15 transfer for a finite time to cool the surfaces below the strain point without cooling the center of the glass sheet below the strain point; and

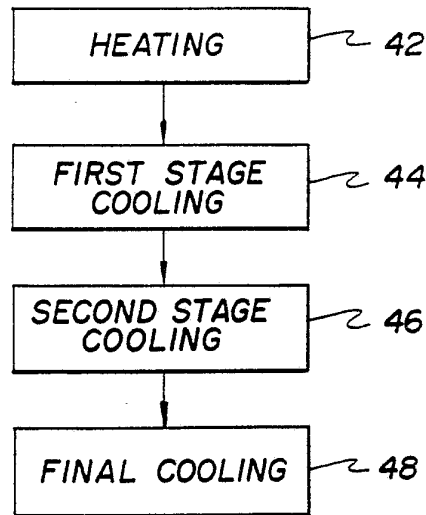
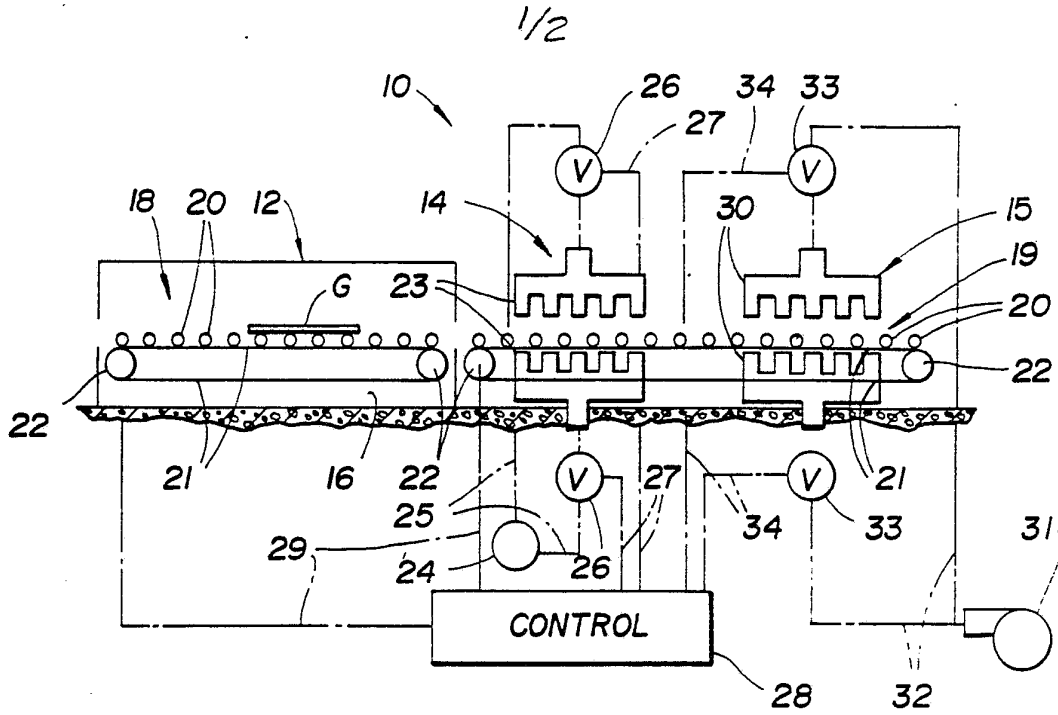
thereafter cooling the oppositely facing surfaces of the glass sheet at a second rate of heat
20 transfer less than the first rate to cool the center of the glass sheet below the strain point and further cool the surfaces, the second rate of heat transfer being of a magnitude such that the surface temperature initially increases without going back substantially above the
25 strain point prior to subsequently cooling, whereby the glass sheet upon ultimate cooling to ambient temperature has surface compression and center tension that cannot be achieved with conventional tempering at a uniform rate of cooling.

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STATEMENT UNDER ARTICLE 19

This response to the International Search Report for the present international application under the Patent Cooperation Treaty has revised the claims to correspond with the allowed claim coverage of the corresponding United States application for which priority has been claimed. More specifically, the replacement base method claim 1 and product claim 4 now each recite the manner in which the first and second cooling steps are performed with the second rate of cooling being of a magnitude such that the surface temperatures initially increase without going substantially above the strain point prior to subsequent cooling. The prior art cited in the International Search Report including United States Patents 4,204,845 Shields et al; 4,236,909 Thomas et al; and 2,311,846 Littleton et al do not teach or in any way suggest this concept as recited in a glass sheet tempering process. As discussed in the paragraph beginning on page 8 and continuing through page 9 and concluding on page 10, the relationship of the first and second rates of cooling provides the large surface compression to center tension ratio in a manner that can be effectively performed and which is not taught or suggested in any way by the prior art.

It is thus respectfully submitted that the present PCT application now defines a novel invention involving an inventive step as well as having industrial applicability so as to thereby be patentable. An indication to that effect upon the Preliminary Examination to be conducted in the future is thus respectfully solicited.



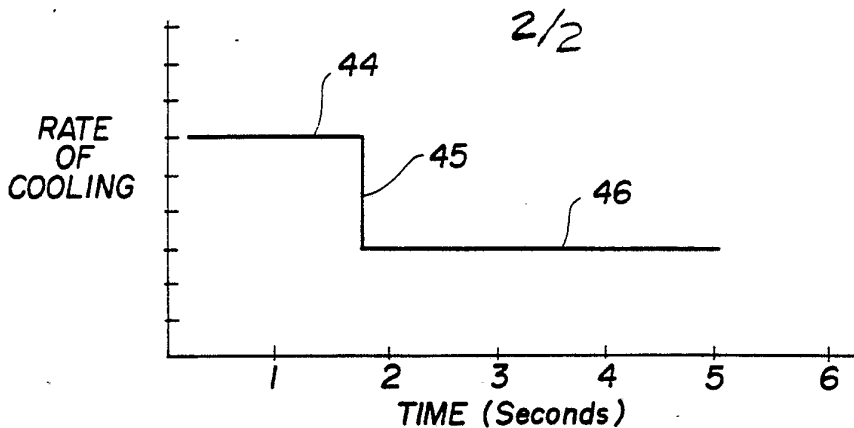


Fig. 3

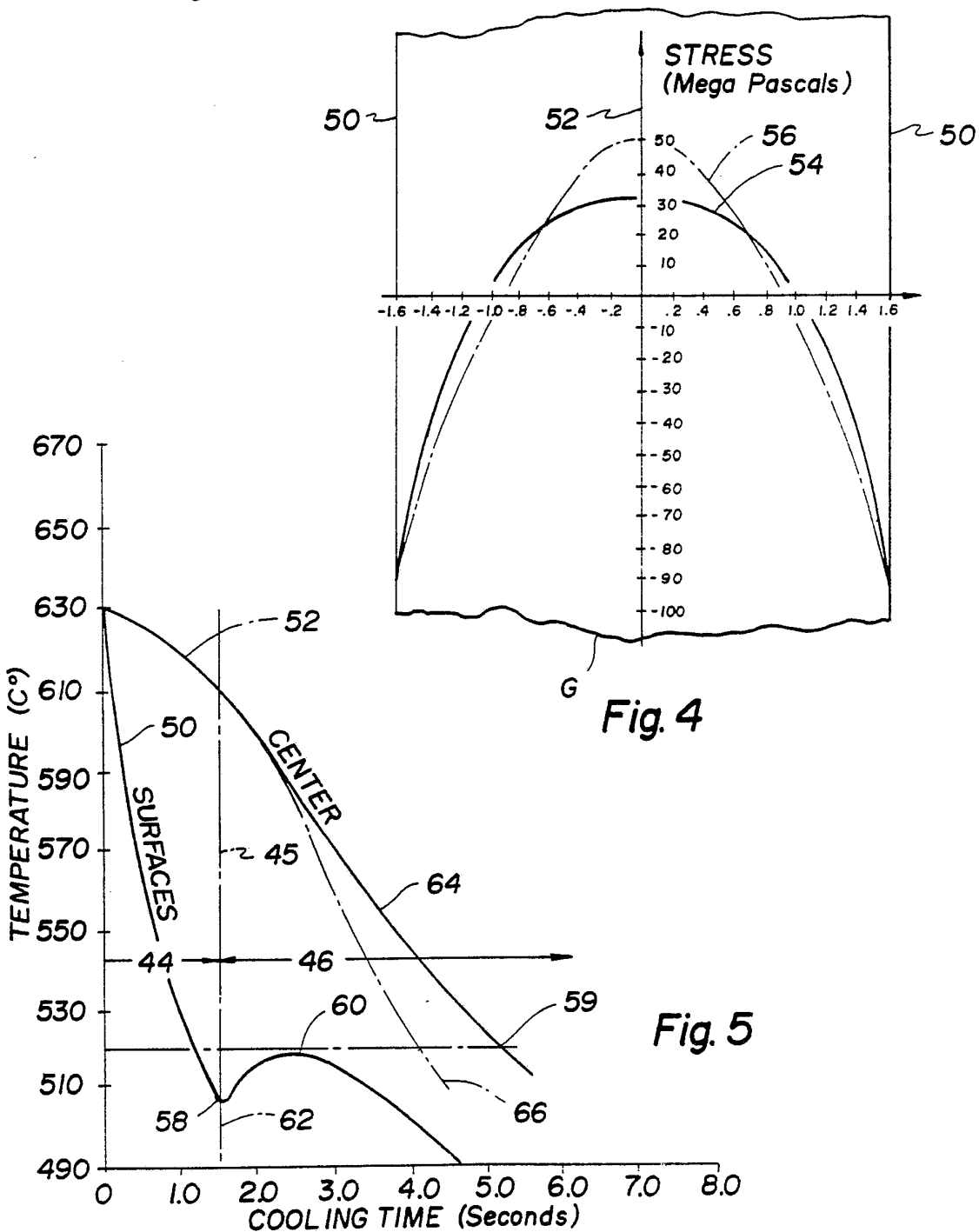


Fig. 5

INTERNATIONAL SEARCH REPORT

International Application No. **PCT/US89/04021**

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC IPC(4): C03B 27/04; B32B 17/00 U.S.CL.: 65/114; 428/410		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
U.S.	65/104, 115, 117, 119; 428/410	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹		
Category ⁹	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X	US, A, 4,204,845 (SHIELDS ET AL) 27 May 1980 See the entire document.	1-5
X	US, A, 4,236,909 (THOMAS ET AL) 02 December 1980 See the entire document.	1-5
X	US, A, 2,311,846 (LITTLETON ET AL) 23 February 1943 See the entire document.	1-5
<p>⁹ Special categories of cited documents: ¹⁰</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
07 December 1989	16 JAN 1990	
International Searching Authority	Signature of Authorized Officer	
ISA/US	<i>Kenneth M. Schor</i> KENNETH M. SCHOR, PRIMARY EXAMINER	