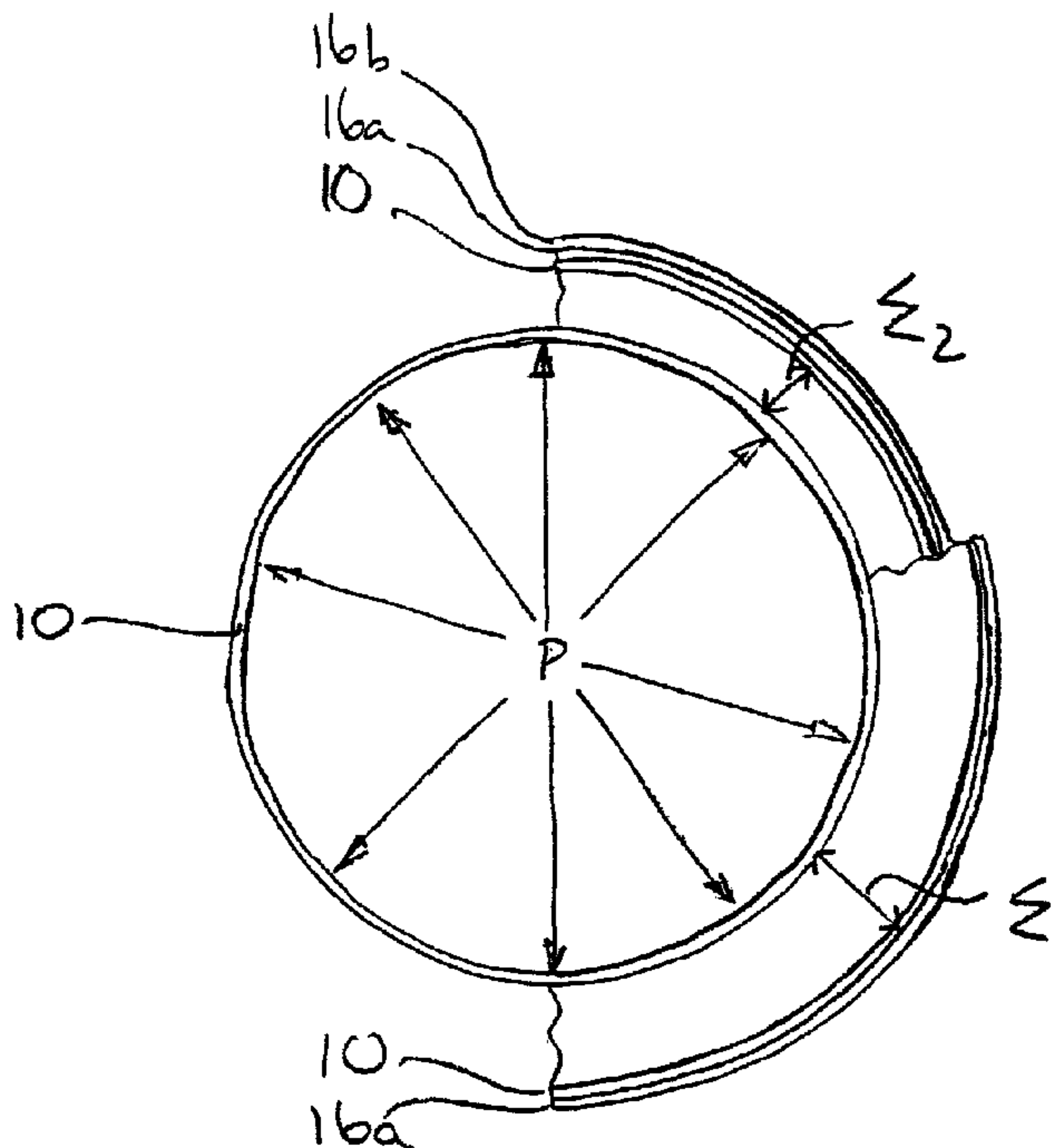




(22) Date de dépôt/Filing Date: 2000/09/12  
(41) Mise à la disp. pub./Open to Public Insp.: 2002/03/12

(51) Cl.Int.<sup>7</sup>/Int.Cl.<sup>7</sup> F17C 1/02  
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(54) Titre : CONTENEUR HAUTE PRESSION PRECONTRAIT RENFORCE DE FIBRES  
(54) Title: PRE-STRESSED FIBRE-REINFORCE HIGH PRESSURE VESSEL



(57) Abrégé/Abstract:

The invention relates to a process for the manufacture of reinforced-reinforced pressure vessels and the product produced therefrom. A ductile metal vessel liner, being formed with at least one nozzle, is wrapped with a first composite layer of resin-impregnated fibers. Pressure is applied to the vessel sufficient to yield the liner but is insufficient to cause deformation of the nozzle. The liner retains a residual compressive stress critical for withstanding cyclical service pressures. A second composite layer is applied over the first layer to increase the strength of the vessel sufficient to maintain the vessel's integrity at very high cyclical service pressures (such as about 0 - 10,000 psig). The vessel is then pressure tested at 1.5 times the intended service pressure to ensure vessel integrity.

**ABSTRACT OF THE INVENTION**

1  
2           The invention relates to a process for the manufacture of reinforced-  
3 reinforced pressure vessels and the product produced therefrom. A ductile metal  
4 vessel liner, being formed with at least one nozzle, is wrapped with a first  
5 composite layer of resin-impregnated fibers. Pressure is applied to the vessel  
6 sufficient to yield the liner but is insufficient to cause deformation of the nozzle.  
7 The liner retains a residual compressive stress critical for withstanding cyclical  
8 service pressures. A second composite layer is applied over the first layer to  
9 increase the strength of the vessel sufficient to maintain the vessel's integrity at  
10 very high cyclical service pressures (such as about 0 - 10,000 psig). The vessel is  
11 then pressure tested at 1.5 times the intended service pressure to ensure vessel  
12 integrity.

1                   **“PRE-STRESSED FIBRE-REINFORCED**  
2                   **HIGH PRESSURE VESSEL”**  
3

4                   **FIELD OF THE INVENTION**

5                   A process for the manufacture of resin-impregnated fiber-reinforced  
6 structural composite vessels and the product resulting therefrom. More  
7 particularly, pressure vessels are wrapped in stages with a resin-fibers matrix and  
8 are subjected to autofrettage pre-stressing technique to increase the vessel's  
9 cycle life and burst strength.

10  
11                   **BACKGROUND OF THE INVENTION**

12                   It is known to wrap a variety of underlying shapes with fibers  
13 embedded in a matrix of resin so as to form reinforced-reinforced plastic  
14 composite products, or FRP. The fiber acts as the structural portion wrapped  
15 over a normally weak shape such as a liner for a pressure vessel.

16                   One such example is the manufacture of reinforced-reinforced  
17 pressure vessels by wrapping hollow, substantially non-structural pressure vessel  
18 liners with fibers resulting in a vessel having burst pressure and cyclical loading  
19 capabilities beyond that of the liner alone. Fibers wrapped about the vessel liner  
20 act in tension when the vessel is pressurized.

21                   Conventionally, a multiplicity of fibers spooled into tows are passed  
22 through heated resin baths containing catalyzed resins prior to being  
23 mechanically wrapped onto the vessel liner. The configuration of the winding is  
24 dependant upon the speed of rotation of the vessel liner and the rate of travel of  
25 the tow-dispensing apparatus. The most common configurations are helical, in  
26 which the tows are at a significant angle from the axis of the object being

1 wrapped; circumferential, in which the tows are wound hoop-wise around the  
2 object; and polar, in which the tows are wrapped in the direction of the  
3 longitudinal axis of the object.

4           The resin is permitted to dry and is then cured. Curing relates to  
5 the process by which the resin is allowed to achieve its final chemical state and  
6 effect its purpose to provide reinforcement to the liner. Curing or chemical poly-  
7 condensation, is the formation of polymers from monomers with the release of  
8 water or another simple substance. Curing is usually performed at elevated  
9 temperatures however, room temperature may be sufficient for some types of  
10 resins.

11           In some cases the resulting vessel is pressed into service after  
12 curing.

13           In other known processes, the liner is pre-stressed using a  
14 technique called autofrettage wherein the liner is plastically deformed (yielded)  
15 within the fiber-wrap for imparting a compressive residual stress into the liner and  
16 thereby increasing the vessel's structural characteristics under pressure.

17           In more detail, a liner forms a fluid nozzle structure for providing  
18 access to the liner's bore. In the autofrettage process, the cured fiber-wrapped  
19 liner is placed inside a protective housing and liquid is injected through the fluid  
20 nozzle structure, over-pressurizing the liner within the wrap and causing it to  
21 yield. When the fluid pressure is released, the tension in the liner diminishes and  
22 then becomes compressive at ambient pressure (pre-stressed). Accordingly, as  
23 the pressure rises once again, in service, the stress on the liner progressively  
24 reduces from compressive, through zero, then finally becomes tensile at a peak  
25 stress less than that previously experienced at the same pressure. Cyclical

1 tensile stress is a major factor in precipitating fatigue stress failures and thus, with  
2 an initial compressive stress, each pressure cycle results in a lower maximum  
3 tensile stress in the liner and thus improves its fatigue strength.

4 It is known to apply autofrettage pressures of 6000 psig to pre-  
5 stress aluminum-lined, fiber-wrapped tanks which are safely operable at service  
6 pressures of about 3000 psig.

7 However, increased use of alternative fuels to fuel vehicles, such as  
8 compressed natural gas and hydrogen, and the requirement for ever greater fuel  
9 range, has created a need for lightweight, safe tanks with even greater capacity.  
10 One method for avoiding increasing tank size and weight, new tanks must be  
11 capable of containing fuel at higher service pressures, upwards of 10,000 psig. It  
12 is possible to provide reinforced-reinforced tanks capable of such pressures but  
13 without pre-stressing, the cyclical life is too short to be of commercial value.  
14 Accordingly a pre-stressing technique is suggested.

15 Using the prior art process, autofrettage pressures in the range of  
16 about 20,000 to 25,000 psig would be required in order to adequately yield an  
17 aluminum liner to produce a vessel capable of safely maintaining integrity at a  
18 cyclic service pressure of 10,000 psig. Unfortunately, at these high pressures the  
19 boss forming the nozzle and the internal threads used to attach various fittings to  
20 the tank liner, also yield. As the nozzle structure yields, its dimensions no longer  
21 accept conventionally sized valve bodies and fittings. The dimensional changes  
22 in this portion of the tank are not predictable, require close fitting tolerances and  
23 therefore one is unable to compensate for such deformation when originally  
24 sizing the nozzle structure. The deformation of the nozzle structure is related to  
25 the surface area of the nozzle, as provided by the nozzle's port size.

1           One approach to minimize deformation and enable high  
2 autofrettage pressures would be to make the nozzle opening much smaller,  
3 thereby reducing the service area and lowering the stress. From a practical  
4 standpoint however, this does not solve the problem as the smaller openings  
5 would no longer be able to accept current valve bodies. Note that more and more  
6 components are integrated into the valve bodies, and therefore must be of  
7 sufficient size to incorporate components such as solenoid-controlled valves,  
8 manual override valves, pressure regulators and temperature control devices.

9           Further, attempts to thicken the walls or fiber-wrap the boss have  
10 only provided marginal increases in strength of the nozzle structure, insufficient to  
11 eliminate yield.

12           US patent 4,571,969 to Tomita discloses an alternative to single  
13 high pressure autofrettage for steel liners operating at service pressures of about  
14 100-200 ksig. Tomita suggests that application of a single pressure is generally  
15 disadvantageous in that it does not always produce the required residual  
16 tangential compressive stresses in alloy steel cylinder bores. Further, Tomita  
17 states that high pressure autofrettage deforms the bore of the cylinder so that it is  
18 no longer employable in its intended use, where bore size must be maintained  
19 within narrowly prescribed ranges. Tomita teaches a cyclical autofrettage  
20 technique applicable to alloy steel cylinders in which the vessel is subjected to  
21 multiple lower pressure cycles in order to avoid large diameter dilations and the  
22 associated tolerance problems. The rate of production would diminish and costs  
23 would escalate if each cylinder were to be cyclically pressurized until a suitable  
24 strength was achieved. Further, aluminum liners have a lower yield stress than

1 alloy steel and therefore do not require the extreme pressures suggested by  
2 Tomita.

3 Thus, there remains a need for a process by which a reinforced-  
4 reinforced pressure vessel can be produced, capable of maintaining its integrity  
5 at service pressures upwards of 10,000 psig, with a nozzle structure port and  
6 threads which can be reliably sized and retain their dimensions necessary to  
7 accept a variety of valve bodies and fittings. Ideally, the process should be  
8 accomplished with a minimum number of steps for efficient rates of production.

9

#### 10 SUMMARY OF THE INVENTION

11 The current invention addresses the unsuitability of the existing  
12 single cycle autofrettage processes for manufacturing very high pressure  
13 reinforced-reinforced vessels for storing fuel gas. High pressure autofrettage is  
14 associated with disadvantages including imposing unpredictable and significant  
15 deformation of the bore of the vessels threaded nozzles. The novel process also  
16 uses a single pressure cycle, but it achieves the objectives of forming a residual  
17 compressive stress in the vessels liner using lower pressures, and standard  
18 autofrettage equipment by implementing a unique two stage resin-impregnated  
19 fiber wrapping process to produce pressure vessels capable of reliably  
20 withstanding high service pressures in the range of 10,000 psig.

1           In a broad aspect of the invention, then a process for manufacturing  
2 a reinforced-reinforced high pressure vessel comprises the steps of:

- 3           • wrapping the liner with a first composite layer of  
4           predetermined strength;
- 5           • applying a predetermined first pressure to the bore, the first  
6           pressure being greater than the design pressure and  
7           sufficient to yield the liner within the first composite layer and  
8           produce residual compressive stress therein; and
- 9           • wrapping the liner with a second composite layer so that  
10          when the design pressure is applied, the strain in the liner is  
11          constrained so that stress in the liner is below yield.

12           Preferably the first pressure and the strength of the first composite  
13 layer are complementary. A maximum pressure is set which would not damage  
14 the nozzle and further that the strength of the first composite layer is set such that  
15 the liner can be plastically deformed at a pressure less than that maximum  
16 pressure.

17           More preferably, the ductile metal liner is aluminum and the  
18 pressure used to autofrettage the liner is in the order of 15,000 psig, resulting in a  
19 product pressure vessel which, in combination with the strength provided by the  
20 second composite layer, is capable of cyclical use at design pressures of 0 -  
21 10,000 psig. More preferably, the pressure vessel is tested using cyclical  
22 pressure tests at 1.25 – 1.5 times design pressure in order to meet safety  
23 standards.

1           Using this novel autofrettage process, the user of the vessel  
2 produced thereby can be confident that the vessel has been subjected to at least  
3 1.5 times the service pressure, even with the higher service pressures now  
4 sought in the industry.

5

## 6                           BRIEF DESCRIPTION OF THE DRAWINGS

7           Figure 1 is a cross-sectional view of a cutaway end of a reinforced-  
8 reinforced pressure vessel illustrating a threaded nozzle structure fitted with a  
9 generic valve assembly;

10           Figure 2 is a flow chart describing the wrapping, drying and curing  
11 process of the prior art which may or may not include autofrettage to produce a  
12 conventional reinforced-reinforced vessel;

13           Figure 3 is a two-section graph illustrating, on the bottom a cross-  
14 section of the vessel liner and a nozzle, and on the top the stress profile resulting  
15 from autofrettage according to Fig. 2, for conventional pressures (solid line) and  
16 at very high pressures (dashed lines) for an appropriately stronger-walled vessel;

17           Figure 4 is a flow chart describing an embodiment of the present  
18 invention wherein the liner is wrapped a first time, cured, pressurized to yield,  
19 cured, and wrapped a second time;

20           Figure 5a is a graph illustrating the stress curve for the wall of the  
21 liner for autofrettage and demonstrating permanent plastic deformation;

22           Figure 5b is a graph illustrating the elastic, reversible stress curve  
23 for the pre-stressed wall of the liner during normal pressure cycling and indicating  
24 an acceptably low peak stress;

1           Figure 6 is a graphical representation of the manufacturing steps  
2 over time including staged wrapping of the vessel liner, the pressure cycles and  
3 the resulting stress curves for the liner; and

4           Figure 7 is an exaggerated and fanciful cross-sectional view of a  
5 vessel. The circle represents the unstrained liner. Two partial arcs represent the  
6 strain or deflection in the liner after the first and second wrapping, demonstrating  
7 the change in the strain and thus the reduced stress in the liner after the second  
8 wrapping.

9

#### 10           DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

11           Having reference to Fig. 1, a portion of a fiber-wrapped vessel liner  
12 10, is illustrated having a cylindrical body portion 11 (see Fig. 3), hemispherical  
13 ends 12 formed thereon and at least one boss 13 formed into a threaded nozzle  
14 structure or nozzle 14 protruding axially from a hemispherical end 12. The nozzle  
15 14 is shown fitted with a generic valve body 15. A composite layer 16  
16 comprising a matrix of fibers and resin is wrapped about the cylindrical body  
17 portion and partially around the hemispherical end 12.

18           It is understood that reference to a "wrap" or "layer" suggests as  
19 many layers of fibers and resin as necessary to obtain the desired strength  
20 between distinct process steps. Accordingly, a first wrap or composite layer  
21 could include thousands of actual wraps of fibers.

22           Having reference to Fig. 2, the prior art process is shown in which  
23 metal lined fiber-wrapped pressure vessels are produced. The ductile metal liner  
24 1 is first wrapped at 21 with heated resin impregnated fibers. The wrapped liner is

1 allowed to air dry and then is heated in a hot air oven at about 125 degrees  
2 Celsius to cause polycondensation or curing of the resin at 21.

3           Optionally, the cured, fiber-wrapped liner may then be subjected to  
4 autofrettage at 22. Autofrettage is the process of pre-stressing the ductile metal  
5 within the composite layer so as to leave a residual compressive stress therein,  
6 increasing the ultimate burst pressure and the peak tensile stress for improving  
7 fatigue strength. Referring to Fig. 5a, the autofrettage process 22 comprises  
8 placing the cured fiber-wrapped liner inside a protective housing and over-  
9 pressurizing the liner to impose stresses at 23 and causing it to yield at 24.  
10 When the pressure is released at 25, the tension in the liner diminishes and then  
11 becomes compressive 26 at ambient pressure (pre-stressed) due to the  
12 constraint imposed by the overwrap. Accordingly, as shown in Fig. 5b, on re-  
13 pressurization at 27, the stress on the liner is first progressively reduced from  
14 compressive, then passes through zero, then finally becomes tensile. Pressure  
15 cycles causing tensile stress are a major factor in precipitating fatigue stress  
16 failures. With the initial pre-compressive stress, each pressure cycle  
17 (pressurization 27 and depressurization 28) results in a lower maximum tensile  
18 stress at 29 in the liner and thus improves its fatigue strength.

19           This prior art process is performed once and the product vessel is  
20 presumed ready for service.

21           Having reference to Fig. 3, pressure  $P$ , imposed during  
22 autofrettage, strain the liner 10 and results in liner stress. The fiber-reinforcement  
23 constrains and prevents the liner from reaching the ultimate tensile strength at  
24 which the liner would lose its integrity. Due to the shape and design of the walls  
25 of the liner 10, the imposed stresses 30 in the main cylindrical body 11 are

1 greater (above yield) than in the hemispherical ends 12 or the nozzle 14.  
2 Typically, an aluminum lined pressure vessel designed for use at 3000 psig is  
3 pressurized at 6000 psig to produce sufficient yielding of the cylindrical portion 11  
4 of the aluminum liner 10.

5 The foregoing constitutes the state of the art for fiber-wrapped  
6 liners.

7 Continuing to refer to Fig. 3, in order to produce lightweight  
8 aluminum lined pressure vessels designed for use at high pressures such as  
9 about 10,000 psig, the yield pressure  $P_a$  used for autofrettage would have to be  
10 in the order of 20,000 or 25,000 psig to achieve sufficient yield in the liner. While  
11 the composite layer 16 is appropriately strengthened to prevent the cylindrical  
12 body 11 from reaching ultimate tensile strength, design constrains on the area of  
13 the nozzle 14 limit similar strengthening. Accordingly, under the prior art  
14 techniques, pressures in this range however, imposed stresses 31 at the nozzle  
15 14 result in excessive dilations and permanent deformation in the diameter of the  
16 threaded nozzle 14. Such deformation subsequently interferes with the insertion  
17 of valve body fittings 15, machined by their respective manufacturers to fixed  
18 tolerances. Further, the nozzle 14 and valve bodies 15 are typically quite large  
19 for accommodating the growing need for more and more components like  
20 solenoid operated valves to control the flow of gases out of the pressure vessel,  
21 control circuitry to operate the valves, manual override valves and temperature  
22 regulation devices.

23 The process of the present invention reduces the pressure required  
24 for autofrettage by using a unique two stage wrapping process and thus  
25 maintains the inside diameter of the threaded nozzle 14 within tolerance limits.

1           Having reference to Figs. 4 and 6, an aluminum liner 10 is first  
2 partially wrapped at 40 in a first composite layer 16a. Simply, a partial wrapping  
3 40 results in a first composite layer 16a which provides only enough  
4 reinforcement so as to enable the cylindrical liner 10,11 to be yielded at  
5 pressures which are less than those which are damaging to the nozzle 14. The  
6 amount of wrapping of the first composite layer 16a and an autofrettage or first  
7 pressure  $P_a$  are predetermined to enable the liner 10,11 to yield at the  
8 predetermined pressure but sufficiently reinforce the liner 10,11 to prevent liner  
9 failure. For a vessel having service pressures of about 10,000 psig, an  
10 autofrettage pressure used  $P_a$  (about 15,000 psig) would be lower than that  
11 which would be required using the processes of the prior art (about 25,000 psig)  
12 to produce a similarly pre-stressed pressure vessel liner. The first composite  
13 layer 16a is permitted to dry sufficiently to be subjected to autofrettage.

14           The partially wrapped liner is pressurized at 20 at the first,  
15 autofrettage pressure  $P_a$ , causing the liner 10 to yield. The lower pressure  
16 ensures that the diameter of the threaded boss is maintained within allowable  
17 tolerance limits.

18           The liner 10 is then wrapped at 42 with a second composite layer  
19 16b of resin and fibers. The subsequent second composite layer 16b, or  
20 additional layers, provides additional reinforcement to the liner 10 and additional  
21 constraint to limit the magnitude of the strain imposed on the liner 10 at testing  
22 and design pressures. The amount of wrapping in the second composite layer  
23 16b is calculated to increase the strength of the vessel sufficiently to meet the  
24 designed service pressure  $P_s$ .

1           The resin is then cured at 44 according to the known techniques of  
2 the prior art.

3           The residual stress present in the liner 10 and the overall strength  
4 provided by the second or additional and subsequent composite layers 16b  
5 increase the burst strength of the vessel and minimize the cyclical peak stress.  
6 As shown in Fig. 7, the amount of strain  $\epsilon_2$  following the second wrapping is less  
7 than the strain  $\epsilon_1$  following the first partial wrapping and autofrettage, assuming a  
8 constant pressure P.

9           Having reference to Fig. 6, after the autofrettage 42, the addition 43  
10 of the second composite layer 16b and the curing 44, the resulting product vessel  
11 45 can be subjected to repetitive pressure cycle testing PT1, PT2, PT3, and so  
12 on, to ensure that the fiber-wrapped liner meets accepted performance standards  
13 such as those set forth by the Canadian Standards Association. Thereafter, the  
14 vessel is ready for commercial cyclical service at the intended service pressure.

15           In one example of the first embodiment of the invention, an  
16 extruded aluminum liner of about 325 mm outside diameter would be partially  
17 wrapped in a 4.5 mm thick composite layer of carbon fibers pre-impregnated with  
18 epoxy resin and allowed to air dry. Such fibers include carbon or graphite fibers  
19 available from Mitsubishi Rayon JP as Grafil 34-700. A suitable epoxy resin is  
20 that obtained from Shell, as Epon 826/Epi CURE 9551. Autofrettage could be  
21 performed at approximately 15,000 psig, suitable to yield the aluminum liner. The  
22 liner would then be wrapped with a second composite layer of a further 4.5 mm in  
23 thickness using the same resin-impregnated fibers, followed by curing at 125  
24 degrees Celsius for two hours. The fiber-wrapped liner would thereafter be  
25 subjected to a certification testing program. The Canadian Standards Association

- 1 requires such vessels to be subjected to 11250 cycles at 1.5 times service
- 2 pressure. For a service pressure of 10,000 psig, the fiber-wrapped liners were
- 3 tested at 15,000 psig.

1                   THE EMBODIMENTS OF THE INVENTION FOR WHICH AN  
2 EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS  
3 FOLLOWS:  
4

5                   1. A process for manufacturing a reinforced-reinforced vessel  
6 capable of containing a design pressure, the vessel having a ductile metal liner  
7 with a composite fiber overwrap, the liner having a bore and forming at least one  
8 nozzle structure having a port contiguous with the bore for applying pressure  
9 therethrough to the bore, comprising the steps of:

10                   (a) wrapping the liner with a first composite layer of predetermined  
11 strength;

12                   (b) applying a predetermined first pressure to the bore, the first  
13 pressure being greater than the design pressure and sufficient to yield the liner  
14 within the first composite layer and produce residual compressive stress therein;  
15 and

16                   (c) wrapping the liner with a second composite layer so that when  
17 the design pressure is applied, the strain in the liner is constrained so that stress  
18 in the liner is below yield.

19  
20                   2. The process of claim 1 further comprising the steps of:

21                   (a) determining a maximum pressure in the bore which would not  
22 cause plastic deformation of the nozzle structure; and

23                   (b) setting the predetermined first pressure below the maximum  
24 pressure.  
25

1                   3. The process of claim 2 further comprising the steps of:

2                   (a) setting the strength of the first composite layer such that the  
3 liner can be plastically deformed at a pressure less than the maximum pressure;

4                   (b) determining a minimum pressure in the bore sufficient to yield  
5 the liner wrapped by the first composite layer; and

6                   (c) setting the predetermined first pressure above the minimum  
7 pressure.

8

9                   4. The process of claim 3 further wherein the predetermined first  
10 pressure is about 1.5 times the design pressure or greater.

11

12                   5. The process as recited in claim 3 wherein the liner is aluminum.

13

14                   6. A pressure vessel produced according to the process of claim 1.

15

16                   7. A pressure vessel produced according to the process of claim 3.

17

18                   8. A pressure vessel produced according to the process of claim 5.

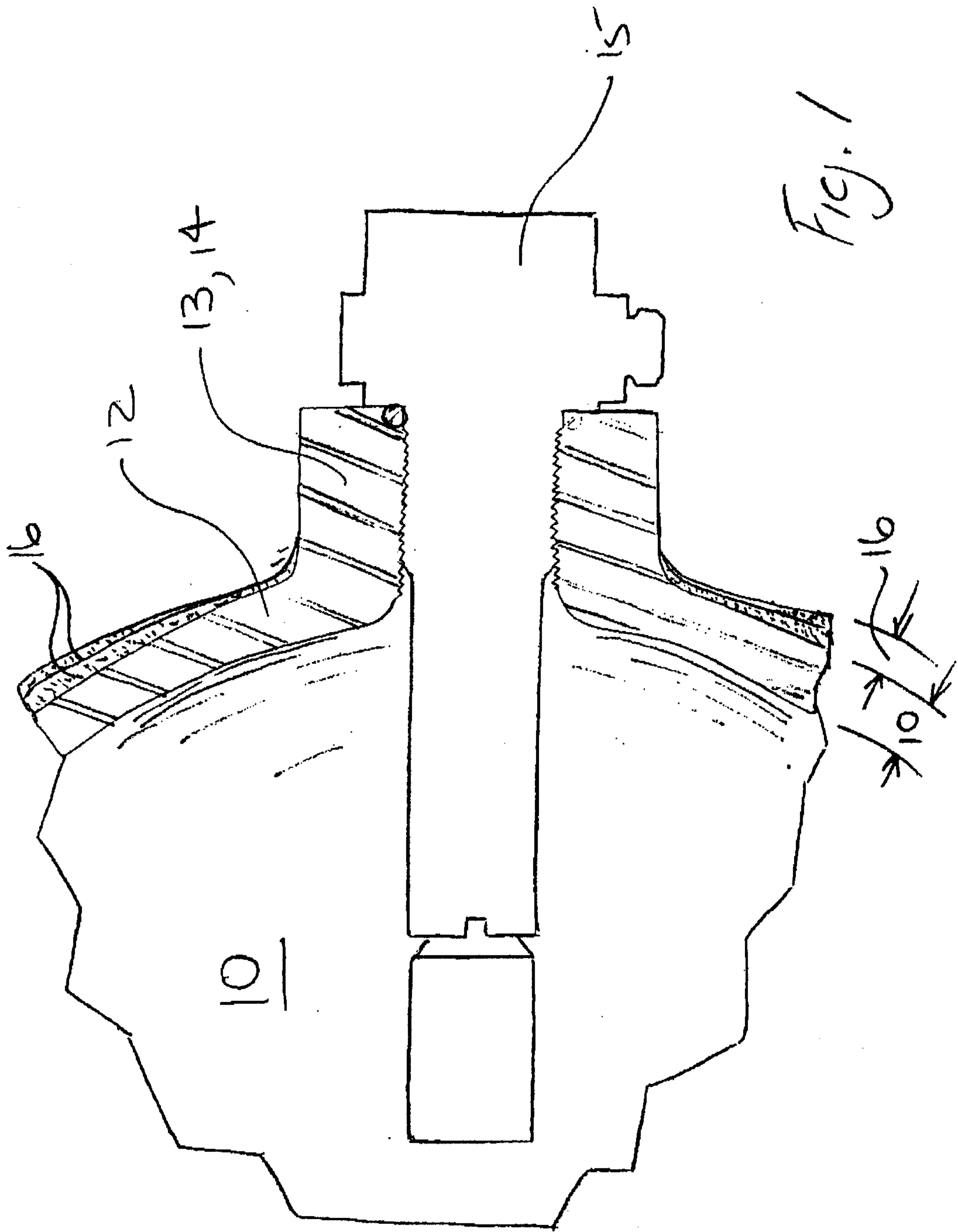
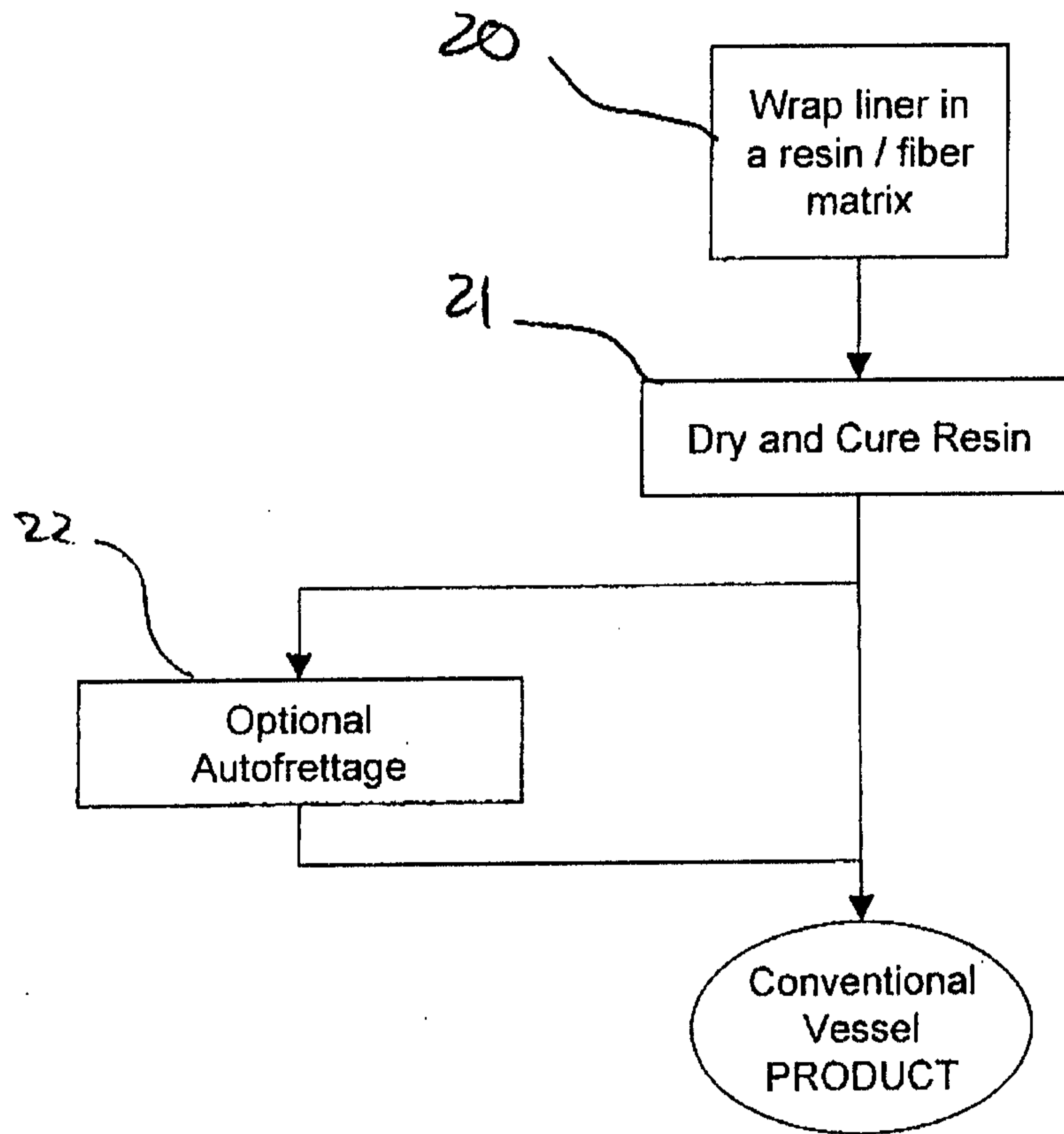


Fig. 1



*Fig. 2*

PRIOR ART

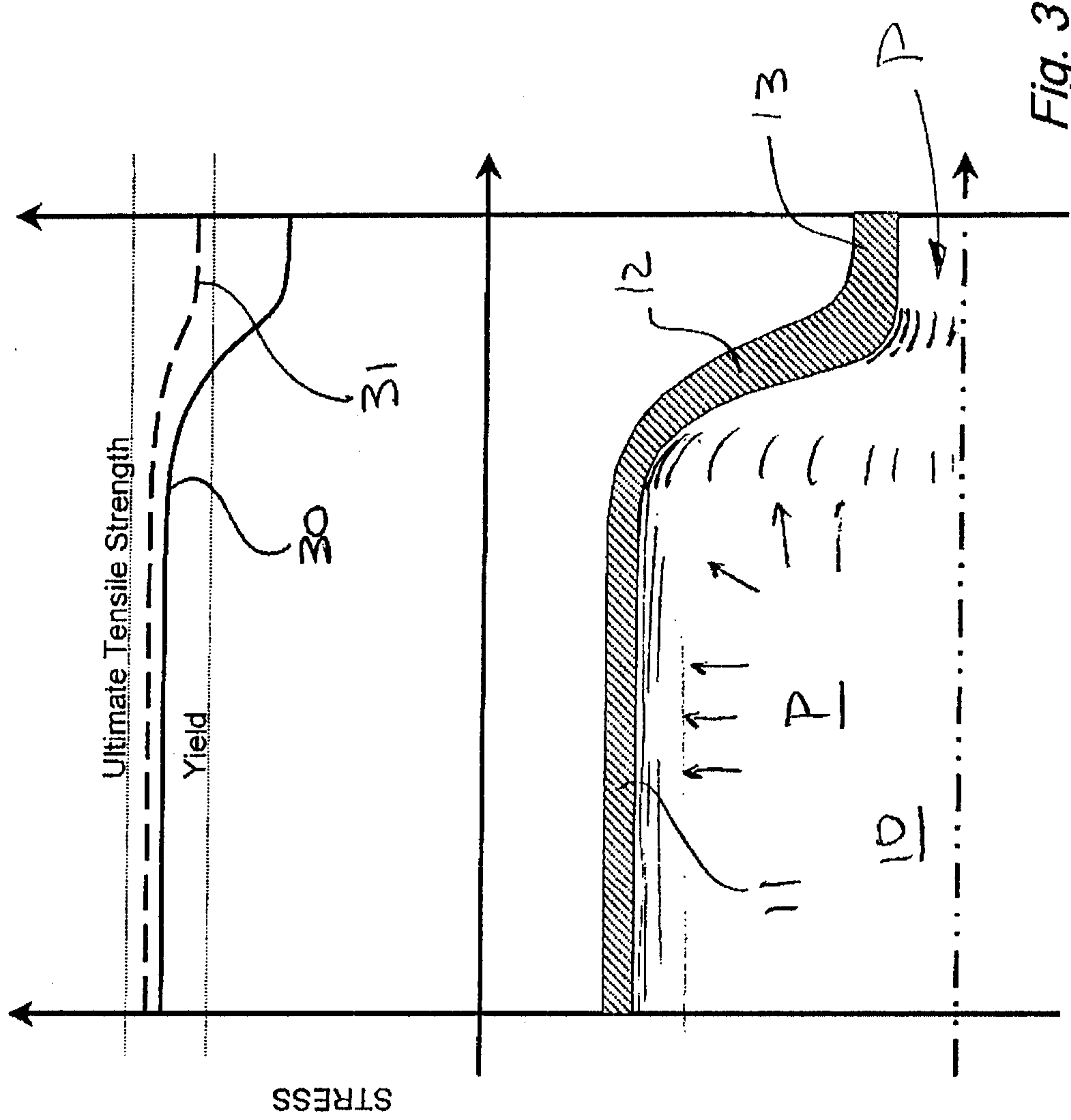


Fig. 3

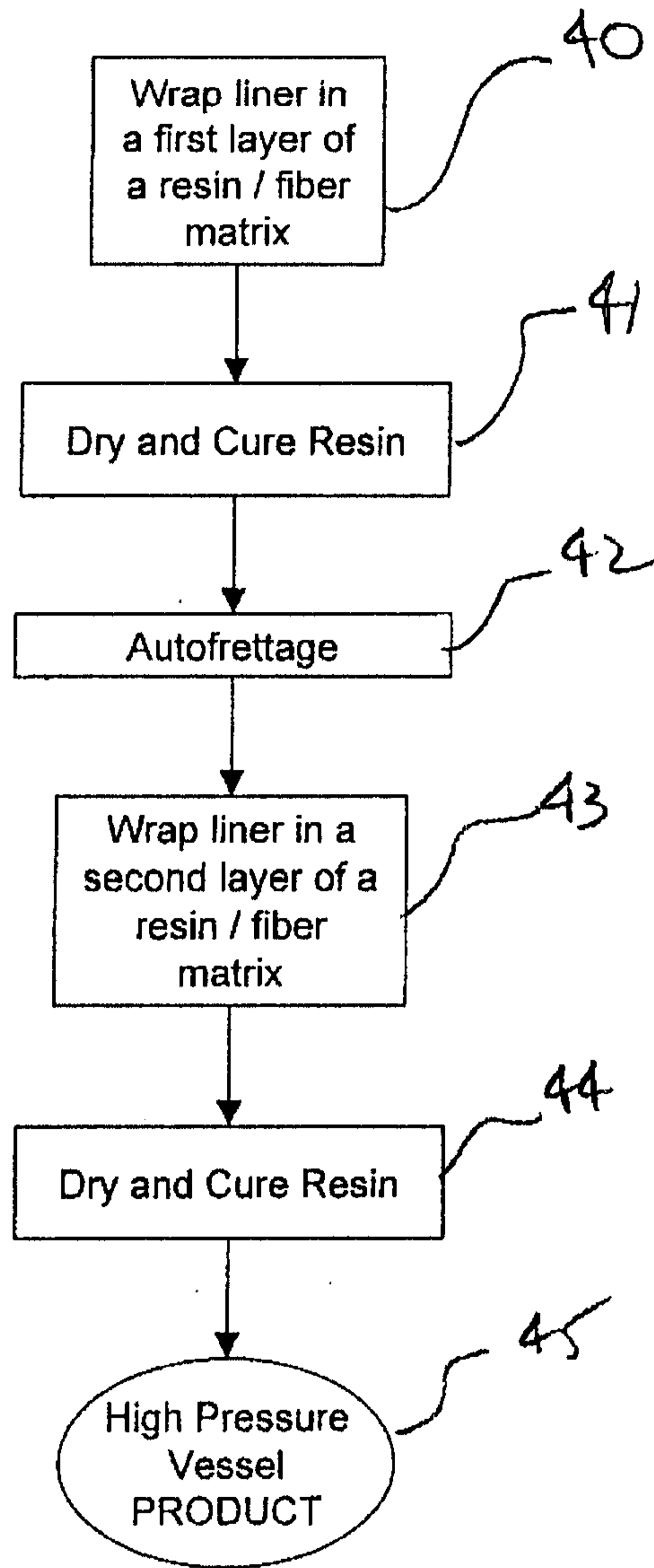
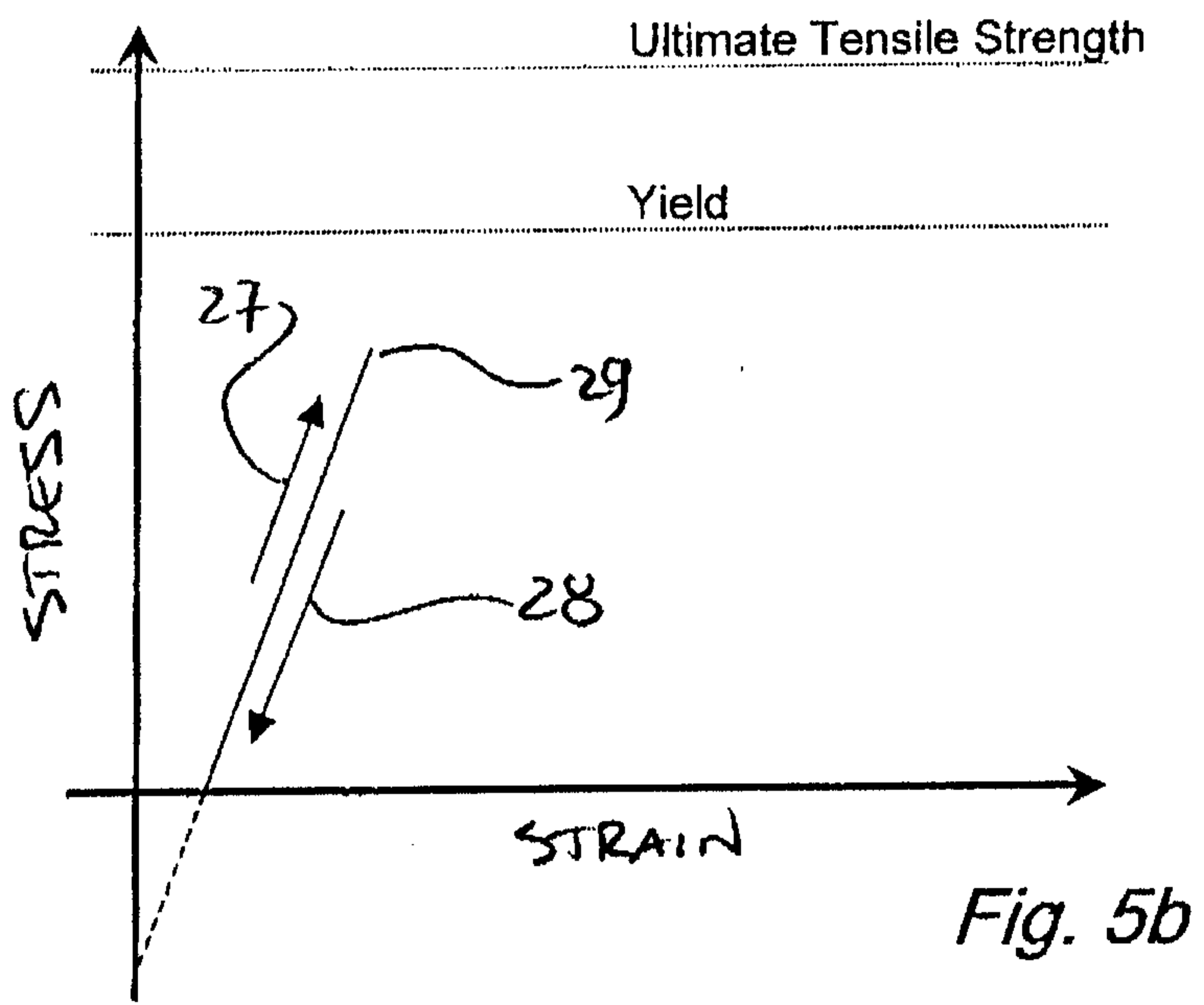
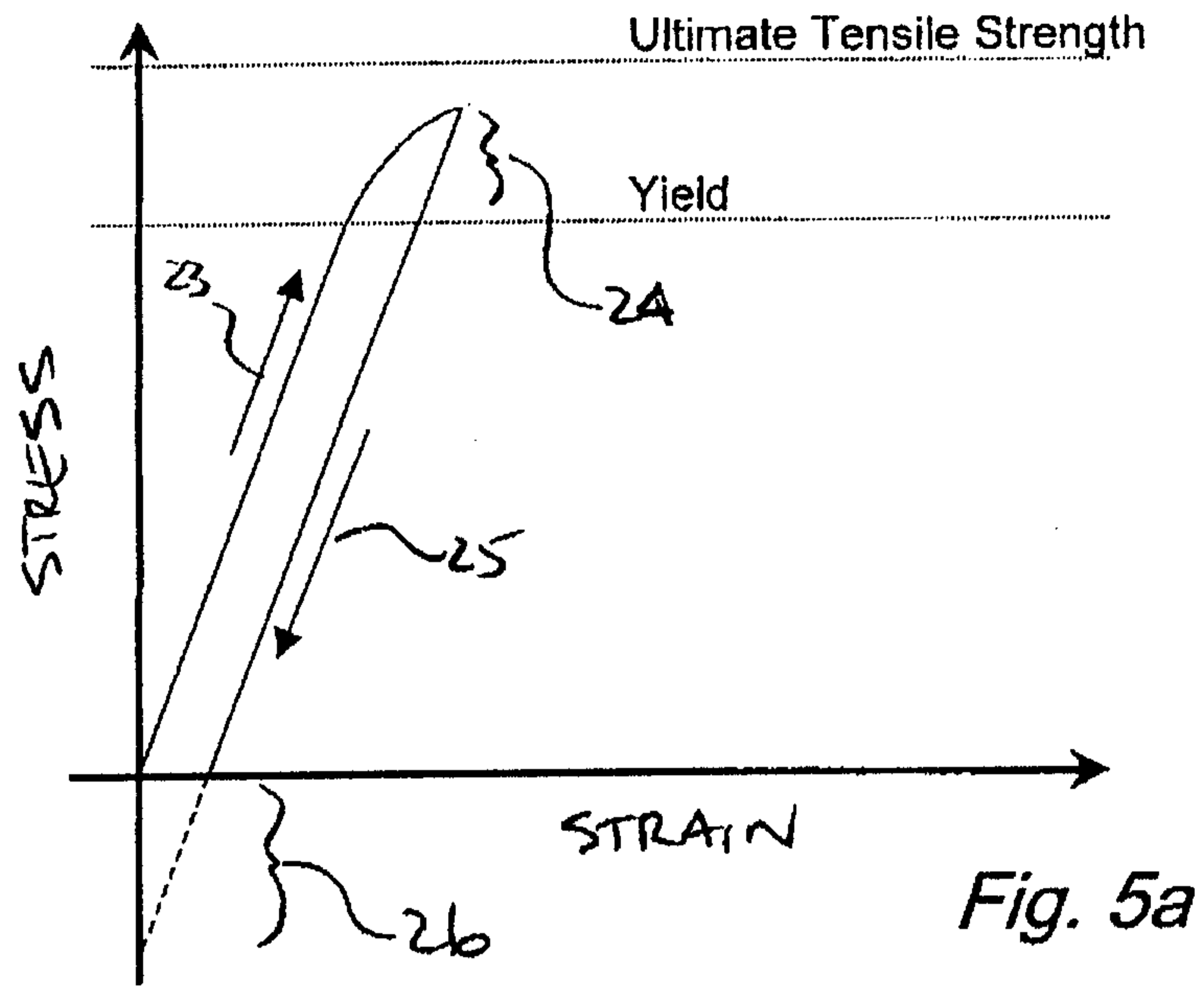
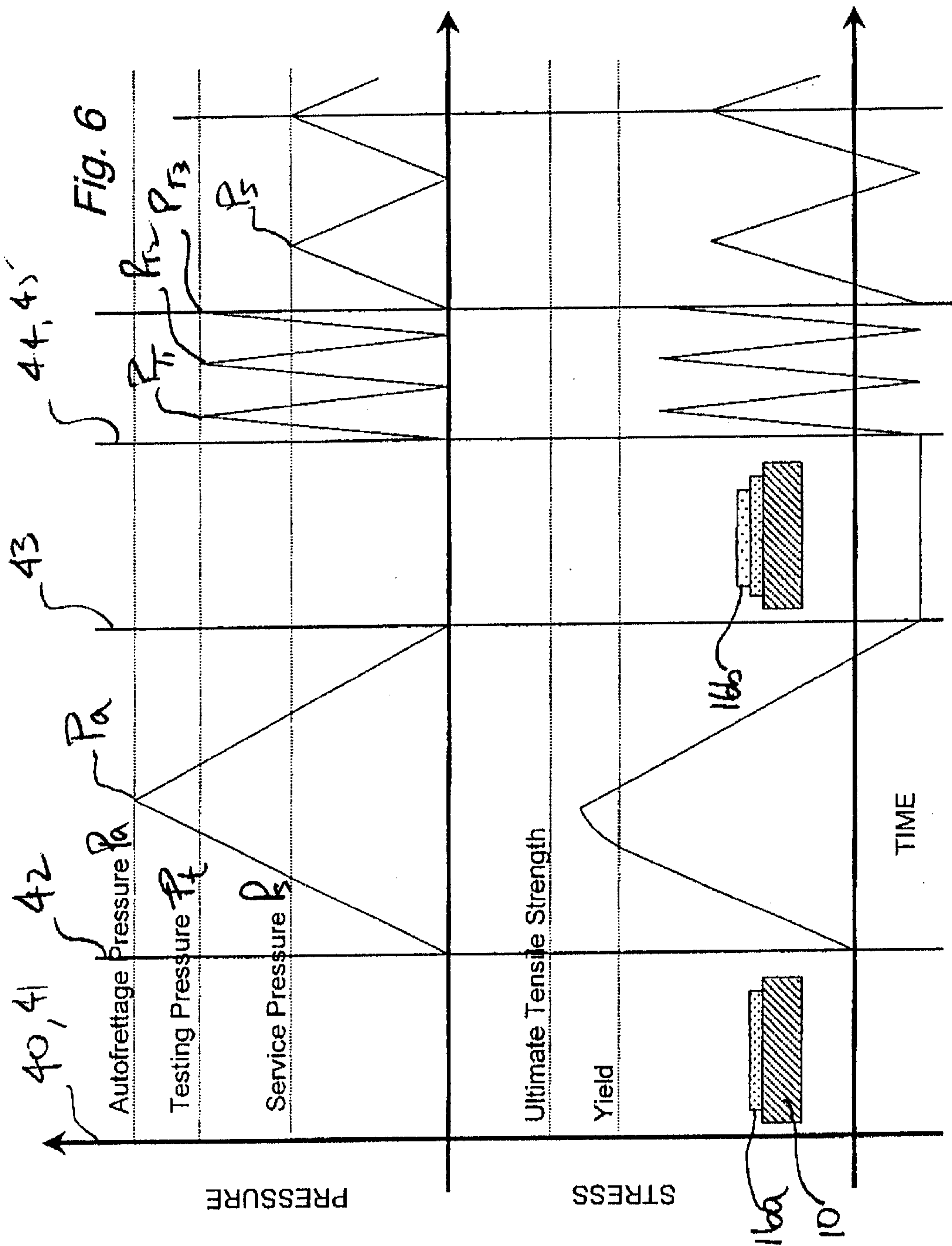


Fig. 4





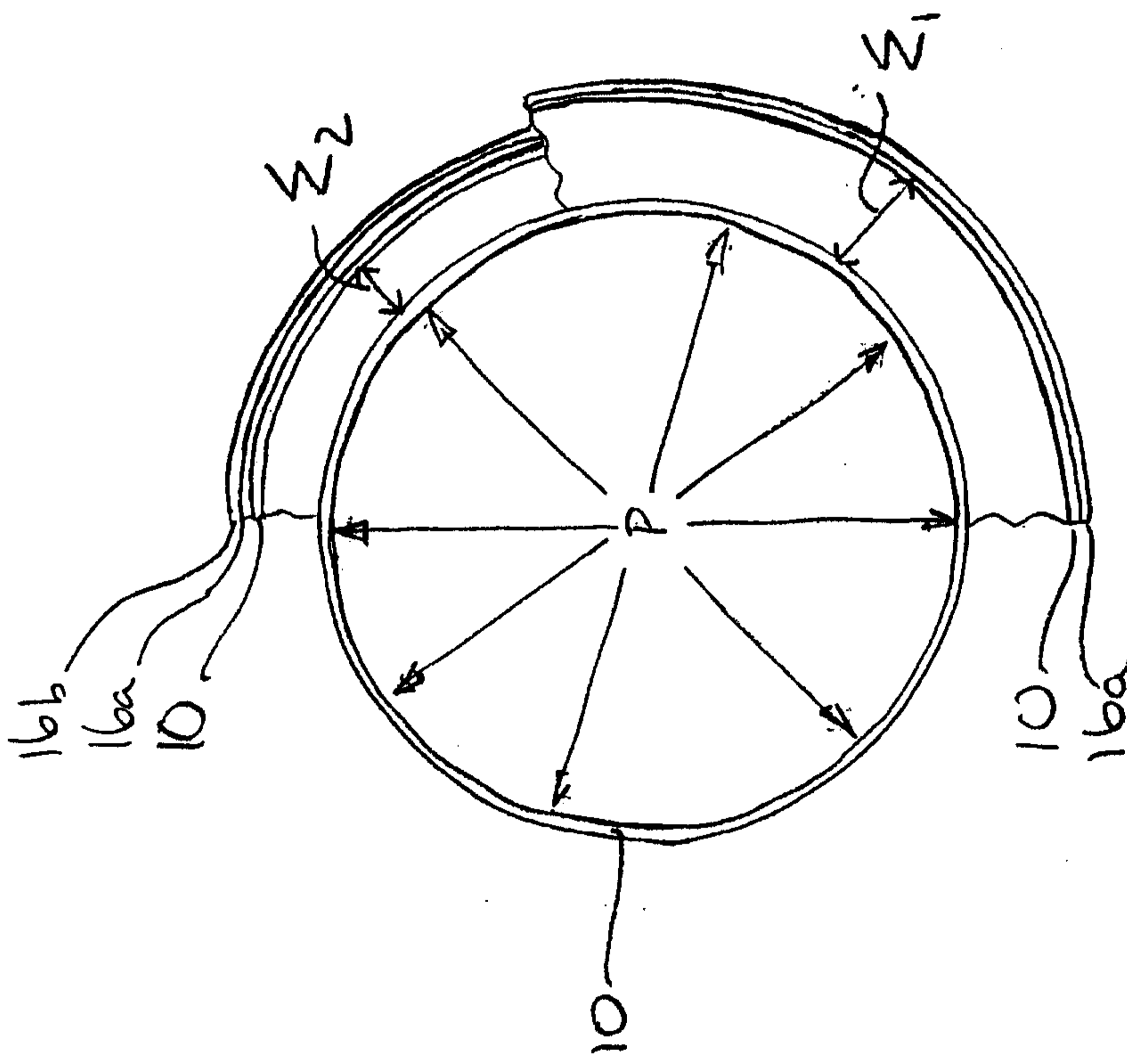


Fig. 7

