

CONVENTION

AUSTRALIA

Patents Act

609736

APPLICATION FOR A STANDARD PATENT

☒ We The Dow Chemical Company

of 2030, Dow Center,
Abbott Road,
Midland,
Michigan 48640,
UNITED STATES OF AMERICA.

hereby apply for the grant of a standard patent for an invention
entitled:

PROCESS AND APPARATUS FOR PRODUCING EXTRUDED THERMOPLASTIC
FOAM BODIES

which is described in the accompanying complete specification.

Details of basic application

Number of basic application: 909,442

Convention country in which
basic application was filed: UNITED STATES OF AMERICA

Date of basic application : 19 September 1986

Address for Service:

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Dated: 31 August 1987

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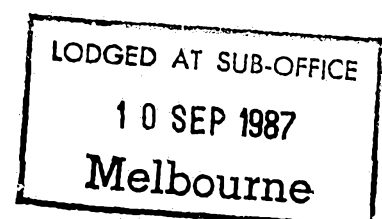
David B. Fitzpatrick

Our Ref : 66676
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APPLICATION ACCEPTED AND AMENDMENTS

ALLOWED 13.2.91

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Patent Declaration

DECLARATION FOR A PATENT APPLICATION

7 INSTRUCTIONS

(a) Insert "Convention" if applicable
(b) Insert FULL name(s) of applicant(s)

(c) Insert "of addition" if applicable
(d) Insert TITLE of invention

(e) Insert FULL name(s) AND address(es) of declarant(s) (See headnote*)

(f) Insert FULL name(s) AND address(es) of actual inventor(s)

(g) Recite how applicant(s) derive(s) title from actual inventor(s) (See headnote**)

(h) Insert country, filing date, and basic applicant(s) for the/or EACH basic application

(k) Insert PLACE of signing

(l) Insert DATE of signing

(m) Signature(s) of declarant(s)

Note: No legalization or other witness required

In support of the (a) convention application made by

(b) THE DOW CHEMICAL COMPANY
2030 Dow Center, Abbott Road,
Midland, Michigan 48640, United States of America.

(hereinafter called "applicant(s)" for a patent (c) for an invention entitled (d)

PROCESS AND APPARATUS FOR PRODUCING EXTRUDED THERMOPLASTIC FOAM BODIES

I/~~we~~ (e) Richard G. Waterman, General Patent Counsel
THE DOW CHEMICAL COMPANY
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do solemnly and sincerely declare as follows:

1. ~~I am/We are the applicant(s).~~
(or, in the case of an application by a body corporate)
1. I am/~~We are~~ authorized to make this declaration on behalf of the applicant(s).
2. ~~I am/We are the actual inventor(s) of the invention.~~
(or, where the applicant(s) is/are not the actual inventor(s))
2. (f) Kyung Won Suh, 1533 Welsh Hills Road, Granville, Ohio 43023, U.S.A.;
Norihiro Sakata, 9-21 Yabase 3-chome, Suzuka-shi, Mie Pref, 513, Japan

is/are the actual inventor(s) of the invention and the facts upon which the applicant(s) is/are entitled to make the application are as follows:

- (g) The applicant Company is the assignee of the said invention from the said actual inventor(s).

(Note: Paragraphs 3 and 4 apply only to Convention applications)

3. The basic application(s) for patent or similar protection on which the application is based is/~~are~~ identified, by country, filing date, and basic applicant(s) as follows:

- (h) September 19, 1986, Kyung Won Suh and Norihiro Sakata
United States of America.

4. The basic application(s) referred to in paragraph 3 hereof was/~~were~~ the first application(s) made in a Convention country in respect of the invention the subject of the application.

Declared at (k) Midland, Michigan, 48640,

Dated (l) August 24 1987 U.S.A.

THE DOW CHEMICAL COMPANY

SIGNATURE
AS TYPED

CORP.
SEAL

To: The Commissioner of Patents

By:

RICHARD G. WATERMAN
General Patent Counsel

Agent: Phillips Ormonde & Fitzpatrick

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PROCESS AND APPARATUS FOR PRODUCING EXTRUDED THERMOPLASTIC FOAM BODIES

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(56) Prior Art Documents
AU 452935 44320/72 B29D 27/00
AU 570483 30610/84 B29C 67/22

(57) Claim

1. A process for producing a thermoplastic foam body comprising the steps:

(a) extruding continuously a foamable thermoplastic resin into a first zone at subatmospheric pressure to form a continuous extruded thermoplastic foam body, the first zone having a liquid removal means and a seal means for entry into a second zone;

(b) passing the continuous extruded thermoplastic foam body from the first zone at subatmospheric pressure to the second zone through the seal means, the second zone being a liquid reservoir, with the liquid open to the atmosphere, and the liquid present in such volume so as to completely submerge the seal means;

(c) whereby liquid leaks between the seal means and the continuous extruded thermoplastic foam body into the first zone and is removed therefrom by said liquid removal means; and

(d) passing the continuous extruded thermoplastic foam body from the second zone to the atmosphere.

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COMPLETE SPECIFICATION
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Complete Specification for the invention entitled:

PROCESS AND APPARATUS FOR PRODUCING EXTRUDED THERMOPLASTIC
FOAM BODIES

Our Ref : 66676
POF Code: 1037/1037

The following statement is a full description of this invention, including
the best method of performing it known to applicant(s):

PROCESS AND APPARATUS FOR PRODUCING
EXTRUDED THERMOPLASTIC FOAM BODIES

5 The present invention relates to controlled environment extrusion technology for producing extruded thermoplastic foam bodies and more particularly, to vacuum, steam or steam and vacuum extrusion processes and an apparatus for these processes.

10 Foamed bodies of a thermoplastic synthetic resin are produced generally by injecting a blowing agent into a molten resin within an extruder, cooling and kneading the mixture in the extruder to prepare a uniform composition having a melt viscosity suitable for foaming and extruding the composition from a die
15 into the atmosphere to foam the molten thermoplastic resin by virtue of the difference between the vapor pressure of the blowing agent and the atmospheric pressure.

20 As is known, the formation of extruded foam bodies in the form of boards, planks, billets, etc., can be enhanced by the employment of a vacuum chamber wherein expansion of continuously extruded foamable
25 material is accomplished under subatmospheric pres-

sure. This, however, necessitates removal of the foamed extrudate from the vacuum chamber, and to do so without adversely affecting the reduced pressure controlled environment in such chamber has presented a difficult problem for solution.

One known solution to this problem involves the employment of an inclined barometric leg which permits continuous removal of the extrudate from a vacuum chamber at the top of the leg in either continuous or discrete lengths to the atmosphere without affecting the reduced pressure within the vacuum chamber. For further details of this technique, reference may be had to U.S. Patents 3,704,083; 4,044,084; 4,199,310; and 4,271,107.

Another solution to the extraction problem is set forth in U.S. Patent 4,487,731. This solution generally involves the continuous extrusion of foamable material into a reduced pressure chamber or zone, cutting the foamed extrudate to length in such reduced pressure chamber, transferring the cut length into a second contiguous chamber or zone which cyclically alternates between such reduced pressure and atmospheric pressure, such transfer being effected when the second chamber is at reduced pressure, and then discharging the cut length from the second chamber to the atmosphere when such second chamber is at atmospheric pressure. This extraction procedure requires a large number of vacuum cycles per hour in order to achieve a desired level of output.

U.S. Patent 4,486,369 provides for continuous extrusion of material, such as foam, into a controlled environment, such as a vacuum chamber or zone at

reduced pressure, and cutting the extrudate to length within such vacuum chamber. The cut lengths are extracted from the vacuum chamber by inventorying a plurality of the cut lengths in an extraction chamber or zone then at reduced pressure and thereafter discharging the inventory from the extraction chamber to the atmosphere without affecting the reduced pressure in the vacuum chamber. This extraction procedure decreases the number of required vacuum cycles due to the discharging at atmospheric pressure of a plurality of cut lengths, or boards.

The process of the present invention is characterized by continuously extruding a foamable thermoplastic resin into a first zone at subatmospheric pressure to form a continuous extruded thermoplastic foam body. The first zone has a liquid removal means and a seal means for entry into a second zone. The continuous thermoplastic foam body is passed from the first zone to the second zone through the seal means. The second zone is a liquid reservoir, with the liquid open to the atmosphere and present in such volume to completely submerge the seal means. Liquid leaks between the seal means and the continuous extended thermoplastic foam body into the first zone and is removed therefrom by said liquid removal means. The continuous extruded thermoplastic foam body is then passed from the second zone to the atmosphere.

The process of the present invention is advantageously practiced with the apparatus of the present invention. Such an apparatus comprises a first chamber, means to extrude a foamable thermoplastic resin into the first chamber to form a continuous extruded thermoplastic foam body and means to maintain

the first chamber at subatmospheric pressure. The first chamber has a means for liquid removal and a seal means for passing the continuous extruded thermoplastic foam body from the first chamber to a liquid reservoir.

5 The liquid reservoir has the liquid open to the atmosphere and the liquid present in such volume so as to completely submerge the seal means. After entering the liquid reservoir, there is a means for passing the continuous extruded thermoplastic foam body from the
10 liquid reservoir to the atmosphere.

The process and apparatus of the present invention provide a subatmospheric pressure for the expansion of a continuously extruded thermoplastic foam
15 body where the subatmospheric pressure is easily maintained even with continuous removal of the extruded thermoplastic foam body from a zone of subatmospheric pressure, and the subatmospheric pressure is
20 independent of the liquid level in the zone of subatmospheric pressure.

In the Drawings

Figure 1 is a schematic view illustrating one embodiment apparatus for carrying out a method according to the invention;

Figure 2 is a schematic view illustrating a second embodiment apparatus for carrying out a method according to the invention;

Figure 3 is a side view illustrating a third embodiment apparatus for carrying out a method according to the invention; and

Figure 4 is a top view of Figure 3 and also illustrates liquid recirculation.

Referring to Figure 1, the extruder partially represented by 5, is coupled to an extrusion die 7 in such a way so that the foamable thermoplastic resin leaving the extrusion die enters the first zone 10 which is at subatmospheric pressure. Entry by the foamable thermoplastic resin into the first zone at subatmospheric pressure must be through appropriate vacuum seals 8 so as to prevent vacuum leaks.

Vacuum will be applied and maintained in the first zone through connection 12 with a suitable vacuum device, such as a pump.

The continuous extruded thermoplastic foam body (foamed extrudate) 50 may be shaped in a forming section 9 after leaving the extrusion die and prior to entering the first zone at subatmospheric pressure. The forming section may be parallel belts, parallel plates or other appropriate dies, devices or machinery.

The foamed extrudate will have a conveyor assembly 22 to guide and optionally transport the foamed extrudate away from the extrusion die and forming section. Such conveyor assembly is constructed of rollers, endless belts or the like, and may be rotatably driven to advance the foamed extrudate through the first zone at subatmospheric pressure.

The foamed extrudate is optionally cooled by a fluid spray 28 so that the foamed extrudate is sufficiently rigid to withstand the pressure difference across the seal means which could cause foam collapse and the frictional and pull force exerted by the seal means which could cause foam breakage. This optional cooling will not be necessary if the foamed extrudate is sufficiently rigid due to the distance the foamed extrudate must travel in the first zone at subatmospheric pressure.

Seal means 16 are used to remove the foamed extrudate from the first zone at subatmospheric pressure into the second zone 20. The seal means must provide a satisfactory vacuum seal and a low frictional force.

In the case of a continuous foamed extrudate in the shape of a board having two major faces substantially parallel to each other and two minor faces also substantially parallel to each other, the seal means includes sealing elements 18 and 19 for the two major faces and other sealing elements for the minor faces of the foamed extrudate.

The sealing elements are rollers, roller conveyors, moving belts or belt conveyors which are

mechanically spring loaded or pneumatically operated and which sealing elements extend through the seal between the first zone and the second zone.

5 Preferably, the two major faces of the foamed extrudate are supported and guided between two belt conveyors which are rotatably driven to convey the foamed extrudate from the first zone at subatmospheric pressure to the second zone. Optionally, one or both
10 belt conveyors will have a means for moving the sealing element normal to a plane of the belt in contact with the foamed extrudate. This ability to move will minimize leakage from the second zone into the first zone at subatmospheric pressure and will accommodate
15 thickness variations between the two substantially parallel major faces of the foamed extrudate. To further decrease leakage belt follower plates may optionally be used.

20 Preferably, the sealing element for the two minor faces of the foamed extrudate is an inflatable roller or a cam seal.

25 An inflatable roller is constructed of a hard material on both ends of the roller so as to fit intimately with the belt conveyors, thus minimizing leakage through the belt conveyors, and a pliable inflatable skin between the hard material ends of the roller to
30 maintain intimate contact with the two minor faces of the foamed extrudate. This pliable inflatable skin will also compensate foamed extrudate width changes, the width being the distance between the two substantially parallel minor faces of the foamed extrudate.
35 To minimize leakage around the pliable inflatable skin, a means for sealing the pliable inflatable skin while

still allowing free movement of the inflatable roller is necessary. Such a means for sealing is generally placed on the side of the inflatable roller opposite the minor faces of the foamed extrudate. One example of such a sealing means is a rigid steel roller housed snugly in a frame.

A cam seal for the minor faces of the foamed extrudate has a stack of cam followers which contact the belt conveyors and the minor faces of the foamed extrudate, move independently about a pivot shaft and are housed in a frame which minimizes leakage at points substantially opposite the minor face of the foamed extrudate.

While specific sealing elements for the major and minor faces of the foamed extrudate have been described, such descriptions are illustrative and not intended to be limiting as to the seal means employed in passing the foamed extrudate from the first zone at subatmospheric pressure to the second zone.

After passing the foamed extrudate through the seal means, the foamed extrudate enters the second zone. The second zone is a liquid reservoir, with the liquid open to the atmosphere and the liquid present in such volume so as to completely submerge the seal means. More particularly, the liquid is present in such volume so as to completely submerge the sealing elements in intimate contact with the major and minor faces of the foamed extrudate.

Preferably, the liquid is water. Introduction of a liquid as opposed to the atmosphere around the sealing elements of the seal means will allow for a

controlled and manageable leak rate of liquid from the second zone 20 into the first zone 10 at subatmospheric pressure.

5 Liquid that does enter the first zone through the sealing elements will fall to the lowest point of the first zone, and excessive accumulation of liquid will be prevented by a liquid removal means, such as a drain 14 or a recirculation system 34.

10 Preferably, the liquid removal means is a recirculation system which would include a level control switch 32 for the liquid level in the first zone, piping to carry the liquid from the first zone to 15 a pump 30 and piping from the pump to the second zone 20.

20 Optionally, the second zone can include a liquid supply tank 42. This liquid supply tank will accept water in excess of the level desired 40 in the second zone, and will supply liquid 36 to the second zone as a level control switch 37 for the second zone is activated. The liquid will be supplied by pump 38 25 and the appropriate piping from the liquid supply tank to the pump and from the pump to the second zone.

30 The foamed extrudate is removed from the second zone. This removal of foamed extrudate is optionally assisted by support means 24, for example, rollers, conveyors or a ramp in the second zone, and a foamed extrudate pulling means 26, for example, rotatably driven belt conveyors.

35 In Figure 2, an alternative embodiment of the present invention, a heated gas 80 is also introduced into the first zone and, more particularly, sprayed on

the foamed extrudate to further expand the foamed extrudate. The heated gas is preferably steam. Optionally, the first zone 10 can be physically or functionally divided into a separate heating zone 82 and a separate cooling zone 84 both of which are maintained at subatmospheric pressure.

In Figure 3, the extrusion die is enclosed by a die housing 110 able to be maintained at subatmospheric pressure. Access to the extrusion die is through one or more access ports 112, and the process may be viewed through one or more viewing ports 114 both located on the die housing.

From the die housing the foamed extrude is passed to the vacuum chamber 120 which is attached to the die housing by an angled spool piece 122. The vacuum chamber is linear sections 124 joined by spool pieces 126. The linear sections preferably have a hinged portion for easy opening and access to the process and the hinged portion has an inflatable rubber seal or other sealing means to allow vacuum operation. Heated gas, such as steam; and cooling fluid, such as water, can be supplied at any point within the vacuum chamber through inlet nozzles, emitters, sprayers or the like.

The foamed extrudate is then passed from the vacuum chamber to a vacuum tank 130. The vacuum chamber is attached to the vacuum tank by another angled spool piece 128. Again, a viewing port 132 and access ports 134 are located on the vacuum tank.

The foamed extrudate is passed to upper 140 and lower 145 belt conveyors which are the sealing elements

for two of the faces of the foamed extrudate located between the belt conveyors. The lower belt conveyor is rigidly held in place, while the upper belt conveyor will move slidably normal to the plane of the belt to adjust for the thickness of the foamed extrudate. For example, the upper belt conveyor is supported by a frame 142 connected to air cylinders. Belt conveyor rotation is provided by a rotating means such as a motor, which rotates one or more rollers on which the belt rests. Also, leakage of liquid past the upper belt conveyor may be minimized by an adjustable follower plate.

Side sealing elements are as previously described.

The vacuum tank is connected to the liquid reservoir 160 and the foamed extrudate is passed from the vacuum tank to the liquid reservoir through the sealing elements.

The foamed extrudate is then passed through the liquid in the liquid reservoir and passed up the exit ramp 162 to the atmosphere. Figure 4 is an illustrative top view of Figure 3 and includes the liquid removal means 136 from the vacuum tank. This removed liquid is recirculated into the liquid reservoir through a liquid entry by means of piping 164 and a pump 166. Also, as can be seen in Figure 4, the foamed extrudate traveling through the liquid up the exit ramp 162 will emerge into the atmosphere, dripping liquid, prior to leaving the tank perimeter. This dripping liquid from the foamed extrudate enters a liquid supply tank, 170, through an opening 168. Piping 172 from the liquid supply tank 170 to the vacuum tank 130 will

allow for liquid from the liquid supply tank to enter the vacuum chamber and thus the liquid reservoir.

Operating conditions for the first zone are a subatmospheric pressure of from 1 to 14.7 pounds psig (7 to 100 kPa) and a temperature of from 20°C to 5 to 10°C greater than the softening point of the glassy polymers or crystallization temperature of the crystalline thermoplastic polymers. Preferably, the subatmospheric pressure is from 3 to 10 psig (20-70 kPa) and the temperature is from 30°C to the softening point or crystallization temperature of the thermoplastic resins.

Thermoplastic resins used in the process of the present invention are not particularly limited but include various homopolymers and copolymers. These include, but are not limited to, styrenic polymers, olefinic polymers and other thermoplastic polymers that are extrudable.

Useful blowing agents are not limited either and include those compounds which are gas or liquid in a normal state and those blowing agents which are thermally decomposable.

Example 1

5 An extruded thermoplastic foamed body is made according to the process of the present invention using the apparatus of the present invention.

10 A polystyrene with a weight average molecular weight of about 200,000 is fed to a 2½ inch (6.4 cm) extruder at a rate of about 100 pounds (45 kg) per hour and melted.

15 Dichlorodifluoromethane at a rate of about 11.0 parts per hundred of polystyrene by weight is injected into the molten polystyrene and mixed.

20 This mixture is then passed through a slit extrusion die and then expanded and formed between substantially parallel plates in the first zone at subatmospheric pressure. The extrusion die pressure is from 500 to 800 psig (3.5-5.6 MPa) and the foaming temperature is from 129 to 134°C. The expanded foam is then cooled with a water spray, passed through the seal means into the water reservoir and removed from the water reservoir. The extruded foamed body has a
25 thickness of about one inch (2.5 cm).

30 Table I illustrates further conditions and the results of four runs.

TABLE I
EFFECT OF VACUUM EXPOSURE

Run Number	1	2	3	4*
Vacuum pishg (kPa)	8.2 (56.5)	11.0 (75.8)	12.7 (87.6)	14.7 (101)
Line Speed ft/min (m/min)	15 (4.6)	15 (4.6)	15 (4.6)	15 (4.6)
Cross-sectional Area in ² (cm ²)	7.9 (51)	7.6 (49)	5.6 (36.1)	5.0 (32.3)
Density lbs/ft ³ (kg/m ³)	2.08 (3.33)	2.58 (4.13)	2.97 (4.76)	3.32 (5.32)
Cell Size (millimeters)				
V (vertical or thickness)	1.12	1.16	1.30	1.20
H (horizontal or width)	1.16	1.08	1.12	1.08
E (extrusion or longitudinal)	1.62	1.54	1.54	1.95

*No vacuum utilized, not an example of the present invention.

Example II

Example 1 is repeated with the addition of steam as a heated gas into the first zone at subatmospheric pressure.

Table II illustrates conditions and results for the four runs of this example.

TABLE II
EFFECT OF CONCURRENT STEAM AND VACUUM EXPOSURE

Run Number	5*	6	7	8
Steam Exposure Time (seconds)	88	88	88	88
Steam Regulator Pressure psig (kPa)	12 (184)	12 (184)	12 (184)	12 (184)
Vacuum psig (kPa)	14.7 (101)	12.8 (88.3)	11.0 (75.8)	8.2 (56.5)
Line Speed ft/min (m/min)	15 (4.6)	15 (4.6)	15 (4.6)	15 (4.6)
Cross-sectional Area in ² (cm ²)	6.3 (40.6)	6.3 (40.6)	6.5 (41.9)	7.9 (51)
Density lbs/ft ³ (kg/m ³)	2.68 (4.29)	2.71 (4.34)	2.97 (3.97)	2.08 (3.33)
Cell Size (millimeters)				
V (vertical or thickness)	1.12	1.25	1.12	.98
H (horizontal or width)	1.05	1.30	1.30	1.20
E (extrusion or longitudinal)	2.43	2.12	1.54	2.12

*No vacuum utilized, not an example of the present invention.

As can be seen in Examples 1 and 2, steam and moderate vacuum conditions produce a lower density foam than a foam produced only under a moderate vacuum condition.

Although the invention has been shown and described with respect to certain embodiments, it is obvious that equivalent alterations and modifications will occur to others skilled in the art upon the reading and understanding of this specification. The present invention includes all such equivalent alterations and modifications, and is limited only by the scope of the claims.

The claims defining the invention are as follows:

1. A process for producing a thermoplastic foam body comprising the steps:

(a) extruding continuously a foamable thermoplastic resin into a first zone at subatmospheric pressure to form a continuous extruded thermoplastic foam body, the first zone having a liquid removal means and a seal means for entry into a second zone;

(b) passing the continuous extruded thermoplastic foam body from the first zone at subatmospheric pressure to the second zone through the seal means, the second zone being a liquid reservoir, with the liquid open to the atmosphere, and the liquid present in such volume so as to completely submerge the seal means;

(c) whereby liquid leaks between the seal means and the continuous extruded thermoplastic foam body into the first zone and is removed therefrom by said liquid removal means; and

(d) passing the continuous extruded thermoplastic foam body from the second zone to the atmosphere.

2. A process as claimed in Claim 1, further comprising the step of recirculating the liquid removed from the first zone into the second zone.



3. A process as claimed in Claim 1; further comprising the step of discharging liquid present in a first section of the first zone into a second section of the first zone.

5

4. A process as claimed in Claim 3, wherein the discharging step is performed by sufficiently angling the first section of the first zone so that liquid present in the first section of the first zone
10 flows into the second section of the first zone.

5. A process as claimed in any one of the preceding claims, further comprising the step of cooling the extruded thermoplastic foam body with a
15 fluid spray prior to passing the extruded thermoplastic foam body through the seal means into the second zone.

6. A process as claimed in any one of the preceding claims, further comprising the step of
20 introducing a heated gas into the first zone.

7. A process as claimed in Claim 6, wherein the heated gas introduced into the first zone is steam.

25 8. A thermoplastic foam body extrusion apparatus comprising:

(a) a first chamber;

(b) means to extrude a foamable thermoplastic resin into the first chamber to form a continuous
30 extruded thermoplastic foam body;

(c) means to maintain the first chamber at subatmospheric pressure;

(d) means for liquid removal from the first chamber;

35 (e) seal means for passing the continuous extruded thermoplastic foam body from the first chamber

to a liquid reservoir;

(f) the liquid reservoir, with the liquid open to the atmosphere and the liquid present in such volume so as to completely submerge the seal means; and

5 (g) means for passing the continuous extruded thermoplastic foam body from the liquid reservoir to the atmosphere.

9. An apparatus as claimed in Claim 8, further
10 comprising means for recirculating the liquid removed from the first chamber into the liquid reservoir.

10 An apparatus as claimed in Claim 8, further
comprising means for discharging liquid present in a
15 first section of the first chamber into a second section of the first chamber having the means for liquid removal from the first chamber.

11. An apparatus as claimed in Claim 10,
20 wherein the first section of the first chamber is an angled first section, sufficiently angled so that liquid in the angled first section flows into the second section of the first chamber having the means
25 for liquid removal from the first chamber.

12. An apparatus as claimed in any one of Claims 8 to 11, further comprising means for cooling the extruded thermoplastic foam body with a fluid spray prior to passing the extruded thermoplastic foam body through the seal means into the liquid reservoir.

13. An apparatus as claimed in any one of Claims 8 to 12, further comprising means for introducing a heated gas into the first chamber.

14. A process according to Claim 1 substantially as hereinbefore described with reference to any one of the Examples.

15. An apparatus according to Claim 8 substantially as hereinbefore described with reference to any one of the Examples.

DATED: 7 February 1991

PHILLIPS ORMONDE & FITZPATRICK

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(9243h)



