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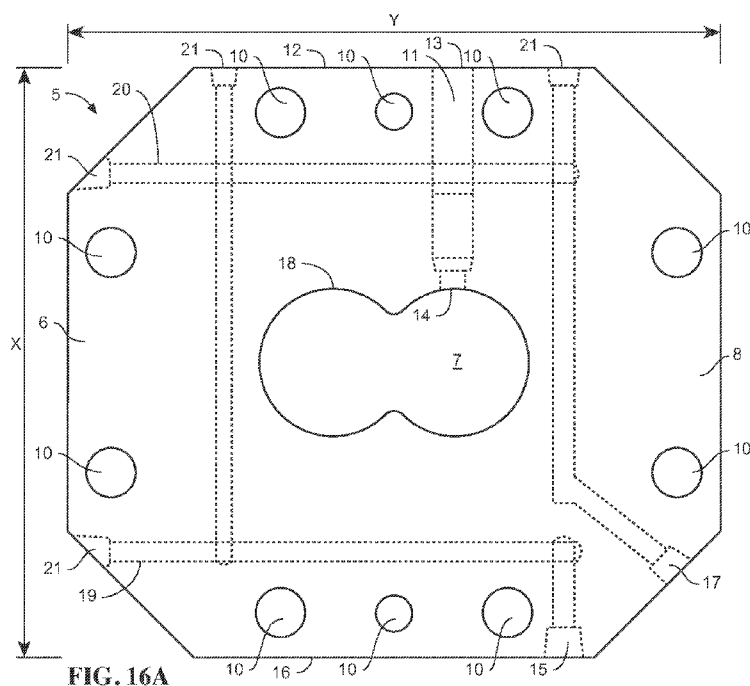


FIG. 16A

(57) Abstract: Described herein is a liquid injector for a barrel extruder as well as methods and processes of manufacturing irradiation crosslinked polypropylene foam. In some embodiments, this includes a liquid injection barrel element (5) that is incorporated in an extruder barrel that includes at least one injection port (11), a temperature sensor well (22), and cooling channels (19, 20).

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LIQUID INJECTION BARREL ELEMENT FOR BARREL EXTRUDER

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims priority benefit to U.S. Non-Provisional Patent Application No. 16/370,223, filed on March 29, 2019, the disclosure of which is hereby incorporated herein by reference in its entirety.

FIELD OF THE INVENTION

[0002] The present invention relates to a liquid injector for a barrel extruder. More specifically, the invention relates to a liquid injection barrel element incorporated in an extruder barrel and comprising at least one injection port, a temperature sensor well, and cooling channels.

BACKGROUND OF THE INVENTION

[0003] Specialty foams may be manufactured using a process that includes grinding materials, blending materials, extrusion, irradiation, and foaming. This process may produce irradiation crosslinked polypropylene foam, which can be used for automotive interior trim applications, for example.

[0004] FIG. 1 shows auxiliary components used in a conventional process to manufacture irradiation crosslinked polypropylene foam. The process shown in FIG. 1 includes components for grinding materials, mixing a blend, extrusion, irradiation and foaming. Although any combination of these steps may be performed at a single location, they may also be performed at different locations to minimize costs. For example, manufacturing foam at the same production site where material ingredients are blended and extruded is often more costly than simply shipping extruded structures for subsequent foaming elsewhere. Extruded

structures are more compact for transport to another site where foaming can be performed by, for example, an automobile manufacturer.

[0005] In step 100 of FIG. 1, resins are initially transported to a production site and fed to a grinder that pulverizes the resins into a powder form. The resins are formed as pellets, granules, chips, flakes, beads, cylinders, tubes, or the like, before being pulverized into a powder form. In step 102, powdered resins are stored in a raw materials silo along with other material ingredients. In step 104, the powdered resin and other material ingredients are blended together by using a Henschell type mixer, for example. The other material ingredients may include an antioxidant package, a crosslinking agent, a blowing agent (i.e., foaming agent), and the like. In step 106, the blended material mixture is extruded into structures by using a die on a parallel twin screw extruder that has a short length to diameter (L/D) ratio, which minimizes shear and residence time. In step 108, an electron beam is used to produce irradiation crosslinked structures. The foaming process of step 110 includes pre-heating the irradiation crosslinked structure, followed by a salt bath that is used as a heat conducting medium to activate the blowing agent in the irradiation crosslinked structure to create foam. In step 112, the foam is cooled, washed, and wound.

[0006] Other conventional methods for manufacturing crosslinked foam may use chemical crosslinking, instead of radiation to produce irradiation crosslinked polypropylene or polyethylene foam. However, chemical crosslinking yields foam that may not be used for automotive interior trim applications because the foam is not smooth. A smooth surface is typically used for automotive interior trim because these applications include a bi-laminate of foam and a laminated foil of TPO or PVC. Consequently, chemical crosslinking is of limited use for these types of applications because it does not yield uniform foam cells. Other known

methods include reactive extrusion, where a chemical reaction or crosslinking takes place during the extrusion process.

[0007] During the extrusion process, additives have commonly been introduced via injection of liquids into the extruder barrel. These injections can be done at various locations along the extruder barrel and at varying levels of injection pressure. In some instances, the extrusion process can be in or can create a high temperature environment. As such, it is possible that the temperature of the extrusion environment may be higher than the boiling point of a liquid additive to be injected. This can create high heat related reactions within the injected liquid such as premature boiling, decomposition, and polymerization, among others.

[0008] In addition, some liquids injected into the extruder barrel may be flammable. If the temperature of the extrusion environment is too high, these flammable liquids can produce extreme safety risks.

SUMMARY OF THE INVENTION

[0009] As discussed above, there are many issues that can arise if the temperature of the liquid being injected into an extruder barrel becomes too high. As such, it can be important to maintain the temperature of the liquid being injected below a certain temperature (e.g., its boiling point). Controlling and/or maintaining the temperature of the liquid being injected into the extruder barrel can prevent high heat related reactions within the injected liquid such as premature boiling, decomposition, and/or polymerization. In addition, controlling the temperature of the injected liquid can also maintain/control the flow characteristics of the liquid, thereby allowing better control over the volume flow rate of the liquid. Furthermore, if the liquid is a flammable liquid, controlling the temperature can improve the safety of the extrusion process as it can minimize the risk for the creation of flammable fumes in the presence of high heat from an adjacent extruder barrel.

[0010] Applicants have discovered a liquid injection barrel element that can be incorporated into an extruder barrel system that includes cooling channels within barrel element to control the element temperature. These cooling channels can allow the liquid being injected into the barrel through the barrel element to be maintained a certain temperature (preferably below the liquid's boiling point). This liquid injection barrel element can also include a temperature sensor well in order to measure the temperature of the barrel element close to the port where the liquid will be injected. Based on the temperature reading from the temperature sensor well, the flowrate of cooling fluid through the cooling channels can be adjusted such that the temperature of the barrel element is appropriate for liquid injection for a given injection liquid.

[0011] In some embodiments, a barrel element for a barrel extruder includes a body having an interior opening extending through a width of the body, wherein the body comprises: at least one injection port extending from a first external side of the body to the interior opening; a temperature sensor well extending from the first external side of the body towards the interior opening; a cooling inlet channel extending from a second external side of the body in a first direction; a cooling outlet channel extending from a third external side of the body in the first direction; and a plurality of connecting cooling channels extending in a second direction, wherein the cooling inlet channel and the cooling outlet channel are fluidly connected by the plurality of connecting cooling channels. In some embodiments, the at least one injection port is fluidly connected between the first external side of the body and the interior opening. In some embodiments, the at least one injection port extends from the first external side of the body in a direction that is perpendicular to a tangent of an outer perimeter of the interior opening. In some embodiments, the body comprises a second injection port extending from the first external side of the body to the interior opening. In some

embodiments, the second injection port is fluidly connected between the first external side of the body and the interior opening. In some embodiments, a distal end of the temperature sensor well is closed. In some embodiments, the distal end of the temperature sensor well is adjacent to an outlet of the at least one injection port. In some embodiments, the second external side of the body and the third external side of the body are the same. In some embodiments, the cooling inlet channel and the cooling outlet channel are parallel. In some embodiments, the cooling inlet channel and the cooling outlet channel are on opposite sides of the interior opening. In some embodiments, the plurality of connecting cooling channels are perpendicular to the cooling inlet channel and/or cooling outlet channel. In some embodiments, the cooling inlet channel extends towards the first external side of the body. In some embodiments, the cooling outlet channel extends towards the first external side of the body. In some embodiments, the plurality of connecting cooling channels comprises a first group of connecting cooling channels and a second group of connecting cooling channels. In some embodiments, the first group of connecting cooling channels and the second group of connecting cooling channels are on opposite sides of the interior opening. In some embodiments, the width of the body is 20-60 mm.

[0012] In some embodiments, a method of manufacturing an extruded structure includes introducing resin into a feeder of an extruder; introducing a liquid crosslinking agent into the extruder through a barrel element at a location downstream of the feeder, wherein the barrel element comprises: a body having an interior opening extending through a width of the body, wherein the body comprises: at least one injection port extending from a first external side of the body to the interior opening; a temperature sensor well extending from the first external side of the body towards the interior opening; a cooling inlet channel extending from a second external side of the body in a first direction; a cooling outlet channel extending from a

third external side of the body in the first direction; a plurality of connecting cooling channels extending in a second direction, wherein the cooling inlet channel and the cooling outlet channel are fluidly connected by the plurality of connecting cooling channels; introducing a chemical blowing agent into the extruder at a location downstream of the location where the liquid crosslinking agent is introduced into the extruder; and extruding a structure from the extruder. In some embodiments, the method includes irradiating the extruded structure to produce a crosslinked extruded structure. In some embodiments, the method includes foaming the crosslinked extruded structure to produce a foam structure. In some embodiments, the foaming includes pre-heating the crosslinked extruded structure. In some embodiments, the foaming includes using a salt bath as a heat source to activate the chemical blowing agent in the crosslinked extruded structure. In some embodiments, the foaming includes using at least one of a radiant heater, a hot air oven, or microwave energy as a heat source to activate the chemical blowing agent in the crosslinked extruded structure. In some embodiments, the chemical blowing agent is introduced into the extruder through a side stuffer downstream of a location where the liquid crosslinking agent is introduced into the extruder. In some embodiments, the resin introduced into the feeder has a non-powder form. In some embodiments, the resin is formed as pellets, granules, chips, flakes, beads, cylinders, or tubes. In some embodiments, the resin comprises a polypropylene based polymer comprising homopolymer polypropylene, MAH-g-polypropylene, impact modified polypropylene, polypropylene-ethylene copolymer, MAH-g-polypropylene-ethylene copolymer, metallocene polypropylene, metallocene polypropylene-ethylene copolymer, metallocene polypropylene olefin block copolymer with a controlled block sequence, polypropylene based polyolefin plastomer, polypropylene based polyolefin elasto-plastomer, polypropylene based polyolefin elastomer, polypropylene based thermoplastic polyolefin

blend and polypropylene based thermoplastic elastomeric blend. In some embodiments, the extruded structure has a sheet-like profile and is at least one of a film, a web or a sheet. In some embodiments, the extruding is performed with a co-rotating, twin screw extruder. In some embodiments, the extruder has a length to screw diameter ratio of 36:1 to 52:1. In some embodiments, the extruder has a length greater than 20 screw diameters. In some embodiments, the feeder is located within an initial 4 screw diameters, the liquid crosslinking agent is introduced into the barrel element within 4 to 8 screw diameters and the blowing agent is introduced into the extruder within 16 to 20 screw diameters. In some embodiments, each twin screw has a diameter of 27 to 135 mm. In some embodiments, a temperature within the extruder is maintained at least 10 degrees Celsius below a thermal decomposition initiation temperature of the chemical blowing agent. In some embodiments, the chemical blowing agent has domains each of a radius size less than 16 μm in the extruded structure. In some embodiments, the liquid crosslinking agent is divinylbenzene. In some embodiments, the chemical blowing agent is azodicarbonamide. In some embodiments, the foam has a density of 20 to 250 kg/m^3 .

[0013] In some embodiments, a method for manufacturing an extruded structure includes introducing resin at a first zone of the extruder; introducing a liquid crosslinking agent at a barrel element between the first zone and a second zone of an extruder, wherein the second zone is downstream of the first zone and the barrel element comprises: a body having an interior opening extending through a width of the body, wherein the body comprises: at least one injection port extending from a first external side of the body to the interior opening; a temperature sensor well extending from the first external side of the body towards the interior opening; a cooling inlet channel extending from a second external side of the body in a first direction; a cooling outlet channel extending from a third external side of the body in the first

direction; a plurality of connecting cooling channels extending in a second direction, wherein the cooling inlet channel and the cooling outlet channel are fluidly connected by the plurality of connecting cooling channels; introducing a chemical blowing agent at a third zone of the extruder; and extruding a structure at a fourth zone of the extruder. In some embodiments, the method includes irradiating the extruded structure to crosslink the resin of the extruded structure. In some embodiments, the method includes foaming the irradiated crosslinked structure to produce a foam structure. In some embodiments, the third zone is downstream of the second zone. In some embodiments, the extruded structure comprises 30 to 80% polypropylene. In some embodiments, the crosslinked composition comprises a crosslinking degree of 20 to 75%. In some embodiments, the extruding is performed with a co-rotating, twin screw extruder having a length greater than 20 screw diameters, the first zone is within the initial 4 screw diameters, the second zone is within 4 to 8 screw diameters, and the third zone is within 16 to 20 screw diameters.

[0014] In some embodiments, an extruded structure is produced by a method that includes introducing resin into a feeder of an extruder; introducing a liquid crosslinking agent into the barrel element at a location downstream of the feeder, wherein barrel element comprises: a body having an interior opening extending through a width of the body, wherein the body comprises: at least one injection port extending from a first external side of the body to the interior opening; a temperature sensor well extending from the first external side of the body towards the interior opening; a cooling inlet channel extending from a second external side of the body in a first direction; a cooling outlet channel extending from a third external side of the body in the first direction; a plurality of connecting cooling channels extending in a second direction, wherein the cooling inlet channel and the cooling outlet channel are fluidly connected by the plurality of connecting cooling channels; introducing a chemical blowing

agent into the extruder; and extruding a structure from the extruder, wherein the chemical blowing agent has domains each of a radius size less than 16 μm in the extruded structure.

[0015] As used herein, the singular forms “a,” “an,” and “the” are intended to include the plural forms as well, unless the context clearly indicates otherwise. It is also to be understood that the term “and/or” as used herein refers to and encompasses any and all possible combinations of one or more of the associated listed items. It is further to be understood that the terms “includes,” “including,” “comprises,” and/or “comprising,” when used herein, specify the presence of stated features, integers, steps, operations, elements, components, and/or units but do not preclude the presence or addition of one or more other features, integers, steps, operations, elements, components, units, and/or groups thereof.

BRIEF DESCRIPTION OF THE DRAWINGS

[0016] Exemplary embodiments of the invention will now be described with reference to the accompanying drawings, in which:

[0017] FIG. 1 shows auxiliary components used in a conventional process to manufacture irradiation crosslinked polypropylene foam;

[0018] FIG. 2 is a flow diagram showing a conventional process to manufacture irradiation crosslinked polypropylene foam;

[0019] FIG. 3 is a flow diagram showing a process to manufacture irradiation crosslinked polypropylene foam without the pulverizing and blending steps used in conventional processes, according to embodiments of the invention;

[0020] FIG. 4 is a diagram of a co-rotating extruder used for a direct compounding extrusion process, according to embodiments of the invention;

[0021] FIG. 5 shows auxiliary components used in the processes to produce compounded extruded structures, according to embodiments of the invention;

[0022] FIG. 6 shows internal parts of a co-rotating extruder, according to embodiments of the invention;

[0023] FIG. 7 illustrates the locations of sections and zones in an extruder in relation to the barrel element, according to embodiments of the invention;

[0024] FIG. 8 illustrates heating and cooling features of a barrel segment of extruder, according to embodiments of the invention;

[0025] FIG. 9 shows injectors used for injecting a crosslinking agent into an extruder, according to embodiments of the invention;

[0026] FIG. 10 shows an injector used for injecting a crosslinking agent into a barrel element, according to embodiments of the invention.

[0027] FIG. 11 shows an example of a liquid diaphragm pump, according to embodiments of the invention;

[0028] FIG. 12 includes images of poor and good mixing of DVB depending on injection location in an extruder barrel, according to embodiments of the invention;

[0029] FIG. 13 includes images of cross-sections of sheet structures examined under 100X magnification, according to embodiments of the invention.

[0030] FIG. 14 illustrates an embodiment of a barrel element acting as a spacer between two barrel sections of the extruder.

[0031] FIG. 15 is an image of a front view of an embodiment of a barrel element disclosed herein.

[0032] FIG. 16A is an illustration of a front view depicting internal components of an embodiment of a barrel element disclosed herein.

[0033] FIG. 16B is an illustration of a side view depicting internal components of an embodiment of a barrel element disclosed herein.

[0034] FIG. 17A is an illustration of a front view depicting internal components of an embodiment of a barrel element disclosed herein.

[0035] FIG. 17B is an illustration of a top view depicting internal components of an embodiment of a barrel element disclosed herein.

[0036] FIG. 17C is an illustration of a side view depicting internal components of an embodiment of a barrel element disclosed herein.

[0037] FIG. 18 is an illustration of a front view depicting internal components of an embodiment of a barrel element disclosed herein.

[0038] FIG. 19 is an image of a front view of an embodiment of a barrel element disclosed herein.

[0039] FIG. 20 is an image of a top view of an embodiment of a barrel element disclosed herein.

[0040] FIG. 21 is an image of a side view of an embodiment of a barrel element disclosed herein.

[0041] In the Figures, like reference numbers correspond to like components unless otherwise stated.

DETAILED DESCRIPTION OF THE INVENTION

[0042] Described herein are methods and processes of manufacturing irradiation crosslinked polypropylene foam. In some embodiments, this includes extrusion of all material components, including a liquid crosslinking agent, to manufacture extruded structures for production of irradiation crosslinked polypropylene foam. In addition, this liquid crosslinking agent can be added to the extrusion process through a liquid injection barrel element that can be incorporated into the extruder barrel system. This barrel element can

include cooling channels to control the temperature of the barrel element as well as a temperature sensor well in order to measure the temperature of the barrel element.

[0043] Extruded structures may be produced at one location, irradiated at a different location, and converted to foam at yet another location. An extruded structure, as referred to herein, has a sheet-like profile and may be a film, a web, a sheet, or the like. The process of producing an extruded sheet structure may also be referred to herein as sheeting.

Accordingly, this disclosure describes the manufacture of extruded structures as a separate process from irradiation and foaming, although they can all be part of the same process in some embodiments.

[0044] Conventional methods to produce irradiation crosslinked polypropylene foam include separate steps of pulverizing, blending all material components together in a Henschel mixer, extruding the blended material using a counter-rotating extruder with a short length to diameter ratio (L/D), crosslinking the extruded structure using an electron beam, and then foaming the crosslinked structure using a heat conducting medium to activate the chemical blowing agent.

[0045] In conventional methods, the resin pellets are pulverized into powder before mixing the powdered resin together with other material ingredients. A powder form of resin facilitates adsorption of the liquid crosslinking liquid agent, such as divinylbenzene (DVB), in the mixture. Adsorption is a process of a liquid adhering to the surface of the adsorbant, which in this case is the pulverized resin. Using non-powdered resin can prevent the liquid crosslinking agent from being adsorbed by the mixture because it settles to the bottom of a mixer or storage container. Powdering the resin also facilitates creation of a homogenous blend of material components in the mixture because many additives are also formed as fine

powder. For example, the chemical blowing agent azodicarbonamide (ADCA) is 18-30 microns in size.

[0046] FIG. 2 is a flow diagram showing a conventional process to manufacture irradiation crosslinked polypropylene foam. At 200, resins are pulverized from pellets to a powder form. At 202, the powdered resins and other material ingredients are blended into a mixture. At 204, an extruder is used to produce an extruded structure, such as a sheet. At 206, an electron beam is used to produce an irradiation crosslinked structure. At 208, the irradiation crosslinked structure is converted into a foam product.

[0047] Unlike conventional methods, the described methods and processes allow for the manufacturing of extruded structures from a mixture of material ingredients that includes a resin, a crosslinking agent, and a chemical blowing agent that are all inputted directly into an extruder. This is advantageous because it eliminates the need to pulverize resins, and eliminates the need for a separate mixer to blend the pulverized resin with other material ingredients. This is also advantageous because extruded structures can be produced without requiring those additional steps, labor, and capital to create powdered resin in a blend with other materials. Operating costs are further reduced because eliminating the need to pulverize resin eliminates the need for cryogenic liquid nitrogen to pulverize softer olefinic material.

[0048] FIG. 3 is a flow diagram showing a process to manufacture irradiation crosslinked polypropylene foam without the conventional pulverizing and blending steps in the process shown in FIG. 2. At 300, direct compounding is used for sheet extrusion by introducing all material ingredients directly into an extruder to produce an extruded structure. At 302, an electron beam is used for crosslinking extruded structure. At 304, a foaming process converts the irradiation crosslinked structure into polypropylene foam.

PULVERIZING AND BLENDING

[0049] Suppliers usually provide resins in pellet form. The pellets may range in size from 2 to 10 mm. The resin pellets may be pulverized as part of an overall manufacturing process, or pulverized at a remote location before extrusion and sheeting. Cryogenic grinders use liquid nitrogen to facilitate grinding of soft olefin materials without heat generation and clumping. Although using liquid nitrogen facilitates grinding, it is not necessary required to yield powdered resins. Resin may also be obtained as flakes or in powder form, directly resulting from a polymerization reactor. Reactor flakes or powder can have the particle size distribution shown in TABLE 1 below, as measured according to ASTM D5644. In some cases, resins of smaller sizes do not need to be pulverized and are ready for used in blending.

Particle Size %							Total =
10 mesh	20 mesh	35 mesh	60 mesh	100 mesh	140 mesh	Pan	100%
3.65	39.49	26.57	17.40	6.95	2.94	2.99	100

TABLE 1

[0050] After pulverizing pellets, resin generally has a particle size distribution shown in TABLE 2. Thus, the size of the resin can be reduced to less than approximately 2 mm. For olefinic materials, such as polypropylene or polyethylene, the bulk density after pulverizing is generally between 25-55 g/100cc, as measured per ASTM D1895.

Particle Size %							Total =
10 mesh	20 mesh	35 mesh	60 mesh	100 mesh	140 mesh	Pan	100%
0.20	12.16	38.95	36.75	9.65	2.35	-0.06	100

TABLE 2

[0051] Pulverized resin can be combined with other ingredients and additives into a mixer to blend all material ingredients together. The mixer may be, for example, a Henschell type mixer. The material ingredients may include, for example, pulverized resins or reactor flakes, a liquid crosslinking agent, a chemical blowing agent, an antioxidant package, and any other types of additives. The material ingredients are scaled gravimetrically to the mixer in a precise sequence of steps so that no clumping occurs when blending the batch of material ingredients. This batch of materials is mixed at a low speed for a period of time, then at a higher speed. Generally, the mixing time involves a total of approximately 9-10 minutes per blend batch.

EXTRUSION

[0052] Extrusion is a process used to create structures having a fixed cross-sectional profile. An extruder is used to create extruded structures. The methods and processes described herein can eliminate the steps of pulverizing resins and pre-blending material ingredients because all material ingredients can be fed directly into an extruder. Material ingredients can be fed through a single port of the extruder, or each ingredient can be individually fed to the extruder through separate ports, or combinations thereof. Pre-blending ingredients of a foam composition may be performed to facilitate their dispersal, although it is not necessary. A Henschell mixer may be used for such pre-blending.

[0053] Described herein is an extrusion process that includes the step of injecting a liquid crosslinking agent directly into an extruder for manufacture of an extruded structure, such as foam sheets or films. Although the majority of this discussion will be with respect to a liquid crosslinking agent, various other liquid additives can be injected using the liquid injection barrel element described here. In some embodiments, these types of compounded sheets are produced without grinding materials and blending a mixture of the materials before

introducing the blend into the extruder. Thus, an extrusion process can be used to make irradiation crosslinked polypropylene compounded foam sheet products without pre-grinding or pre-blending materials.

[0054] FIG. 4 depicts a diagram of a co-rotating extruder used for a direct compounding extrusion process. In some embodiments, different combinations of raw materials are fed to different ports of the co-rotating extruder 400 shown in FIG. 4. Depending on a foam product being produced, different raw materials can be fed directly to the co-rotating extruder to achieve a compounded extruded structure used for the manufacture of an irradiation crosslinked polypropylene foam with a specific target thickness, density, and crosslinking value. Thus, various factors affect the extrusion process, including a targeted thickness, density, foam type, and the like.

[0055] The different ports in FIG. 4 include a feeder 402 for resin, a port for an injection liquid 404 (e.g., a crosslinking agent such as DVB injector), a port for a chemical blowing agent 408 (e.g., an ADCA side stuffer), and a vacuum port 410. Extruder 400 may also include an atmosphere vent 406.

[0056] Extruder 400 includes multiple barrel sections or zones. Each barrel section represents an axial length of, for example, 4 diameters (4D) of the extruder. In particular, extruder 400 depicts a feed section and ten other sections that are downstream of the feed section. Although the number of barrels may vary according to different embodiments, the total extruder length should be sufficient for mixing material components to yield a blend with uniform dispersive and distributive properties. In some embodiments, extruder 400 includes a barrel element 5. The barrel element itself will be discussed in more detail later. However, the barrel element can include the port for an injection liquid 404. In addition, the barrel element can be between any two adjacent barrel sections or zones of the extruder. As

such, the barrel element can act as a spacer between adjacent barrel sections or zones. For example, as shown in Figure 14, barrel element 5 is a spacer between two adjacent barrel sections or zones of the extruder. In some embodiments, the barrel element can be between the feed barrel section and the first barrel section or zone as depicted in Figure 4. In some embodiments, the barrel element can have an axial length of, for example, 0.1 diameter (0.1D), 0.25 diameter (0.25D), 0.5 diameter (0.5D), 0.75 diameter (0.75D), 1 diameter (1D), 2 diameters (2D), 3 diameters (3D), or 4 diameters (4D) of the extruder. In some embodiments, the barrel element can have an axial length of about less than 4 diameters, about less than 3 diameters, about less than 2 diameters, about less than 1 diameter, about less than 0.5 diameter of the extruder. In some embodiments, the width of the barrel element in the direction of the axial length of the extruder can be at least about 20 mm, at least about 25 mm, at least about 30 mm, at least about 35 mm, at least about 40 mm, at least about 45 mm, at least about 50 mm, or at least about 60 mm. In some embodiments, the width of the barrel element in the direction of the axial length of the extruder can be at most about 60 mm, at most about 55 mm, at most about 50 mm, at most about 45 mm, at most about 40 mm, at most about 35 mm, or at most about 30 mm. In some embodiments, the width of the barrel element in the direction of the axial length of the extruder can be about 20-60 mm or about 25-50 mm.

[0057] FIG. 5 shows auxiliary components used in the overall process to produce compounded extruded structures. The overall process includes a blending level and a sheeting level, for manufacturing extruded structures. Extruder 500 shown in FIG. 5 corresponds to another embodiment of extruder 400 shown in FIG. 4, but FIG. 5 shows additional auxiliary components that are also part of the manufacturing process. Many of the additional auxiliary components shown in FIGS. 4 and 5 are commercially available.

[0058] The sheeting level process includes a high speed and high energy input co-rotating twin screw extruder 500. Material ingredients are fed to extruder 500 by using gravimetric loss-in-weight feeders 502. A chemical blowing agent (e.g., ADCA) is fed through side stuffer 504 of extruder 500. Side stuffer 504 may be a standard unit that is sized for particular extruders. Vacuum port 506 is applied to extruder 500 to remove volatiles or air entrapment in the polymer melt. Removing these impurities helps prevent air bubbles or voids in extruded structures that manifests into defects during foaming. Typical vacuum pressures may be 18 to 25 torr, in an absolute vacuum. The vacuum pump can be of dry type or liquid ring type pump which uses chilled process water to maintain a seal.

[0059] The melt is filtered through breaker plate 508 that contains filter 510 at the downstream end of extruder 500. Typical filters can be as coarse as 20 mesh or as fine as 250 mesh, which are industry standard screens for plastic extrusion processes. After melt filtration, gear pump 512 (i.e., melt pump) is used to pressurize and maintain a uniform output to extrusion die 514. In some embodiments, extrusion die 514 has a coat-hanger design that makes a sheet profile of uniform thickness and width that is subsequently cast onto a chill roll stack. The liquid crosslinking agent can be injected into the upstream end of extruder 500 through an injector. In some embodiments, the injector can be fluidly connected with the liquid injection port of the barrel element. The liquid crosslinking agent can be delivered to the injector from a low pressure or high pressure liquid pump that can be a piston or diaphragm type pump specified for a particular liquid, viscosity, and injector.

[0060] Extruded structures, such as sheets, are produced without grinding and blending steps by feeding pellets of polypropylene or other resins directly to extruder 500. A chemical blowing agent is introduced into extruder 500, and a liquid crosslinking agent is injected directly into extruder 500. This extrusion process adequately mixes and compounds material

ingredients together to achieve a homogenous blend that is well mixed with the blowing agent. The blowing agent is effectively dispersed and distributed throughout the polymer matrix. During the extrusion process, the melt temperature of the polymer is kept below an activation or decomposition temperature of the blowing agent to prevent premature foaming.

[0061] The processes and methods described herein can use a co-rotating twin screw extruder that has a longer length to diameter (L/D) ratio than extruders used for conventional methods. That is, the equipment required for pulverizing, blending, and mixing are replaced with additional segments or zones in extruder 500 that can perform these specific operations. For example, when using pulverized resins and pre-blended materials, the L/D is commonly between 20:1 and 24:1 L/D. Unlike conventional processes, an extruder length for direct compounding of an extruded sheet may have an L/D ratio of 36:1 to 52:1, such as 44:1. Thus, pellet resins are fed to a throat of extruder 500, chemical blowing agent is side stuffed into extruder 500, and liquid crosslinking agent is injected directly into extruder 500.

[0062] A common way to quantify lengths of extruders is a ratio of barrel length to screw diameter (L/D). For example, if an extruder has an internal screw diameter of 100 mm and a length of 2000 mm, the L/D ratio of this extruder is 20:1 L/D. Axial length of an extruder in addition to a location along the axis can also be represented and reported in diameters. For example, a 100 mm diameter extruder with a 20:1 L/D that is referenced at a location 400 mm from the feed port could be represented as having four screw diameters of length or 4D (4X100 mm). This is a common and convenient way to reference certain locations along an extruder, since many extruders use multiple modular barrels built in lengths of four diameters.

[0063] A counter rotating extruder may be used in conventional methods that process a blend comprising the foaming agent to minimize shear and heat so as not to cause premature

foaming by inadvertently activating the chemical blowing agent. This corresponds to a premature decomposition of the foaming agent. For example, the extrusion melt temperature of the sheet-like profile can be at least 10°C below the thermal decomposition initiation temperature of the chemical blowing agent.

[0064] FIG. 6 shows internal parts of a typical co-rotating extruder, which, in this example, has a 36:1 (L/D). FIG. 7 depicts detailed about locations and sections or zones in relation to the barrel element pointed out as an injection spacer.

[0065] A diameter or size of the extruder can vary, for example, from about 27 mm to 100 mm. An extruder diameter that is small, such as 27 mm, may not produce sufficient output for commercial production. On the other hand, a diameter that is too large (e.g., much greater than 100 mm) may compromise mixing because the number of melt divisions per kilogram of material processed is reduced at a same RPM, because more material is passing through extruder 500. Accordingly, in some embodiments, the diameter of the extruder is about 27-235 mm, about 40-100 mm, about 40-80 mm, or about 60-100 mm, for commercial production of extruded structures. In some embodiments, the diameter of the extruder is about 40 mm, about 50 mm, or about 87 mm.

[0066] Using extruders with L/D ratios that are too low or high may produce defective foams. For example, an L/D ratio of 20:1 may not allow for enough mixing elements in conjunction with metering elements to pump the material. This may result in sheet products that include unmelted materials and/or sheet products of reduced size because of additives that were not uniformly dispersed in the sheet products. These defecting sheet products can manifest into other defects during the final foaming process. On the other hand, for example, an extruder L/D ratio of 60:1 may be too long. An extruder length that is too long may result

in too much residence time of the polymer in the extruder. Increased residence time may lead to degradation and premature activation of the chemical foaming agent.

[0067] The extruder may have a screw design to sufficiently melt polymer components, as shown in FIG. 7. The extruder achieves a consistent mixture with uniform dispersive and distributive characteristics to obtain a homogenous blend without unmelted portions, large particles, and the like. Removing these defects from the mixture helps eliminate defects that would otherwise manifest during the foaming process.

[0068] FIG. 13 includes images of cross-sections of sheet structures examined under 100X magnification. The image on the left is an example of “good mixing” during extrusion. The radius size of each powder blowing agent domain is equal to or less than about 16 μm , or 32 μm in diameter. The image on the right is an example of “poor mixing” during extrusion. In this case, the domains of the chemical blowing agent are much larger, as large as 46 μm in radius. These large domains of blowing agent cause defects when the sheet is subsequently foamed.

[0069] The extrusion process includes different parameters that can be controlled. These parameters include a feed rate, percentage of recipe material components, extruder screw RPM, barrel zone temperatures, and head pressure. Head pressure, as referred to herein, is pressure at the discharge of extruder 500. FIG. 8 depicts heating and cooling features of a barrel segment of extruder 500 that can be used to control barrel temperatures, for example.

[0070] Regardless of how the ingredients are fed into the extruder, the shearing force and mixing within the extruder must be sufficient to produce a homogenous an extruded structure having a sheet-like profile. The sheet-like profile may be a film, a web, or a sheet. In some embodiments, a co-rotating twin screw extruder provides sufficient shearing force and mixing through the extruder barrel to extrude a sheet with uniform properties.

[0071] The extrusion temperature of the sheet-like profile can be at least 10°C below the thermal decomposition initiation temperature of the chemical blowing agent. If the extrusion temperature exceeds the thermal decomposition temperature of the chemical blowing agent, then the blowing agent will decompose, which will result in undesirable premature foaming.

RESINS

[0072] Resins fed to extruder 500 may comprise polypropylene, or a blend of polypropylene with other compatible materials. Polyethylene is commonly used when blends are desired.

In some embodiments, these two materials are blended together to achieve desired physical properties for which either base resin cannot achieve by itself. The composition can have a synergistic effect by blending two or more materials together. In some embodiments, several polypropylenes may be blended together with multiple polyethylene type materials.

[0073] In some embodiments, the extruded structure comprises about 25 to 95 parts by weight of at least one polypropylene based polymer having a melt flow index (MFI) from about 0.1 to 25 grams per 10 minutes at 230°C and/or at least one polyethylene based polymer having a melt flow index from about 0.1 to 25 grams per 10 minutes at 190°C. In some embodiments, the resin includes 30-80% polypropylene with a balance of polyethylene.

[0074] Notably, shear stress is proportional to shear rate viscosity. A melt flow index that is too high causes viscosity of resin to be too low, which results in less shear stress to disperse ingredients during the extrusion process, and which ultimately results in defects during foaming. On the other hand, a melt index that is too low results in viscosity that is too high, which causes shear heating to occur and high extrusion pressure. This can cause premature activation of the foaming agent and poor extrusion output. Therefore, it is preferred to have the melt flow index of polypropylene and polyethylene between 0.5 and 8 grams per 10 minutes, or more preferably between 0.5 and 3.5 grams per 10 minutes.

[0075] Resins fed to extruder 500 may be in pellet form because grinding is unnecessary. Typical pellet size varies from about 2 mm in average dimension $((\text{length} + \text{width})/2)$ to about 10 mm, and a common pellet size is approximately 4 mm. Pellet size can also be represented in pellets per gram, and for olefins can vary between 200 and 10 for the given size of 2 mm to 10 mm, respectively. Using smaller pellet sizes can increase the cost of pelletizing. On the other hand, using larger pellets requires more dispersive energy to breakdown and fully melt and homogenize materials during the extrusion process.

[0076] Resins are fed to extruder 500 in main feed port 502, or the first barrel segment of extruder 500, within a length of 4D, as shown in FIG. 5. As such, the feeder and/or feed port can be located within an initial 4 screw diameters of the extruder. Feeding resins further downstream of extruder 500 may cause unmelted resin in the extruded structures because the resins will bypass the plasticating (i.e., melting and mixing) section of the feedscrews of extruder 500. Therefore, unmelted resin will result in the sheets, which cause defects in the foam.

[0077] Polypropylene, as referred to herein, is a polymer resin that includes homopolymer polypropylene, MAH-g-polypropylene, impact modified polypropylene, polypropylene-ethylene copolymer, MAH-g-polypropylene-ethylene copolymer, metallocene polypropylene, metallocene polypropylene-ethylene copolymer, metallocene polypropylene olefin block copolymer with a controlled block sequence, polypropylene based polyolefin plastomer, polypropylene based polyolefin elasto-plastomer, polypropylene based polyolefin elastomer, polypropylene based thermoplastic polyolefin blend and polypropylene based thermoplastic elastomeric blend. Another example of polypropylene is an isotactic homopolypropylene.

[0078] Polyethylene, as referred to herein, is a polymer based resin that includes LDPE, MAH-g-LDPE, LLDPE, MAH-g-LLDPE, VLDPE, VLLDPE, HDPE, MAH-g-HDPE,

polyethylene-propylene copolymer, MAH-g-polyethylene-propylene copolymer, metallocene polyethylene, MAH-g-metallocene polyethylene, metallocene ethylene-propylene copolymer, MAH-g-metallocene ethylene-propylene copolymer, metallocene polyethylene olefin block copolymer (with a controlled block sequence), EVOH, EVA, MAH-g-EVA, EMA, MAH-g-EMA, EGMA, MAH-g-EGMA, EBA, MAH-g-EBA, EEA, MAH-g-EEA, EAA, and MAH-g-EAA.

[0079] An impact modified polypropylene is a homopolypropylene or polypropylene-ethylene-copolymer (random PP copolymer) with ethylene-propylene copolymer rubber. The rubber can be amorphous or semicrystalline but is not in sufficient quantities to render the material any plastomeric or elastomeric properties. Examples of commercially available impact modified polypropylene include, but are not limited to, TI4003F, TI4015F, and PRISMA(R) 6910 from Braskem and PRO-FAX® 8623 and PRO-FAX® SB786 from LyondellBasell.

[0080] Polypropylene-ethylene copolymer is polypropylene with random ethylene units. Examples of commercially available polypropylene-ethylene copolymer include, but are not limited to, 6232, 7250FL, and Z9421 from Total Petrochemicals, 6D20 and DS6D81 from Braskem, and PRO-FAX® RP311H and ADSYL™ 7415 XCP from LyondellBasell.

CROSSLINKING AGENT & BARREL ELEMENT

[0081] A crosslinking agent can be used to crosslink polypropylene and blends with polyethylene under electron beam irradiation because polypropylene resins will not crosslink on their own. In contrast, certain types of polyethylene, for example, LDPE, VLDPE, and other copolymers, can crosslink without using a crosslinking agent. In some embodiments, polypropylene based foams may include olefin foams containing some portion of PP resin that is crosslinked and could include blends with polyethylene, EVA, or the like.

[0082] In some embodiments, a liquid crosslinking agent is injected into extruder at the barrel element directly after the feed barrel section or zone of the extruder. As such, the liquid crosslinking agent can be injected into the extruder through a barrel element between the feed barrel section and the first zone of the extruder. Figures 15, 19, 20, and 21 are images of a barrel element used for injecting a liquid into the extruder.

[0083] Figure 16 illustrates a front view (16A) and a side view (16B) of an embodiment of a barrel element that also includes illustrates internal channels and ports. Barrel element 5 can include a body 6 having an interior opening 7. The interior opening can extend through the width of the barrel element (i.e., the axial length of the barrel element) from a first face 8 to a second face 9 of the barrel element. The interior opening can be the same size as the barrel openings of the various barrel sections of the extruder such that the extruder screw(s) can fit through the opening.

[0084] In addition, the barrel element can include connection bores 10. These connection bores can extend through the width of the barrel element (i.e., the axial length of the barrel element) from the first face to the second face of the barrel element. These connection bores can be used to attach barrel sections of the extruder on both sides of the barrel element. For example, one barrel section (e.g., a feed zone) can be attached to the first face of the extruder and a second barrel section (e.g., a first zone) can be attached to the second face of the extruder. These barrel sections can be attached to the barrel element by many means including screws.

[0085] The barrel element can also include at least one injection port 11. In Figures 16, 17, and 18 the at least one injection port is shown as dotted lines indicating that the port is a channel inside of the body of the barrel element. The at least one injection port can extend from an external first side 12 of the barrel element towards the interior opening 7. In

addition, the at least one injection port can include an inlet 13 at the external first side of the barrel element and an outlet 14 opening up to the internal opening of the barrel element. As such, the at least one injection port can be fluidly connected between the first external side of the body and the interior opening. Accordingly, the at least one injection port can be used to introduce liquids such as a liquid crosslinking agent into the extruder.

[0086] As shown in Figures 17 and 18, the barrel element can also include a second injection port 11A. Similar to injection port 11, the second injection port 11A can extend from an external first side 12 of the barrel element towards the interior opening 7. In addition, the second injection port can include an inlet 13A at the external first side of the barrel element and an outlet 14A opening up to the internal opening of the barrel element. As such, the second injection port can be fluidly connected between the first external side of the body and the interior opening. Accordingly, the second injection port can be used to introduce liquids such as a liquid crosslinking agent into the extruder. These injection ports can be formed from drilling into the body of the barrel element. If the barrel element includes multiple injection ports, at least one of the injection ports may be plugged. As such, the at least one injection port that is plugged may not be in used during that particular extrusion process.

[0087] In some embodiments, at least one of the injection ports can extend towards the internal opening in a direction that is perpendicular to the external first side of the body as shown in Figure 16A and 17A. In other embodiments, at least one of the injection ports can extend towards the internal opening in a direction that is angled from the external first side of the body as shown in Figures 17A and 18. In some embodiments, all the injection ports can extend towards the internal opening in a direction that is angled from the external first side of the body as shown in Figure 18. In some embodiments, at least one of the injection ports extends from an external side of the body in a direction that is perpendicular to a tangent of

the outer perimeter (18) of the internal opening as shown by injection ports 11 and 11A in the figures.

[0088] Examples of crosslinking agents can include divinylbenzene (DVB) or other composition with similar properties. In some embodiments, a liquid crosslinking agent, such as divinylbenzene (DVB), is injected into the barrel element of the extruder, between the axial location of 4D to 8D, 4D to 7D, 4D to 6D, 4D to 5D, or 4D to 4.5D of the extruder. DVB can be used with varying degrees of purity and sourced from different commercial suppliers such as Dow or Nippon. Commercially available DVB usually includes a percentage of DVB with the balance being primarily ethylvinylbenzene (EVB). When using a DVB liquid containing 80% purity (i.e., 80% by wt. is divinylbenzene and 20% ethylvinylbenzene) and making polypropylene foam, the amount injected is usually between 2-4 phr (parts per hundred parts resin). This amount varies depending on the level of crosslinking desired. In some embodiments, the DVB is 2-3% by weight of the material ingredients.

[0089] In some embodiments, DVB is added into the extruder thru an injector. FIG. 9 shows examples of various types of commercially available injectors from Specialty Extrusion Solutions. These include a spring loaded valve injector, a manual valve injector, and a pneumatic valve injector. Spring loaded valves open thru pressure activated injection. Standard spring loaded valves function over a pressure range of 50 to 1500 psig and open at preset pressures. Manual valves can be adjusted from fully open to closed to adjust the pressure and flowrate of the injected DVB. Pneumatic valves use air pressure to control the position of the valve, thus allowing for various pressures and flowrates as required.

[0090] In some embodiments, DVB is added into the extruder thru an injector that has a specified hole sufficient to pump the liquid DVB into the extruder under low pressure (≤ 50

psig) or high pressure (>50 psig). FIG. 10 shows a low pressure injector. Regardless of the injector type and intended injection pressure, the injector is inserted into an injection port of the barrel element to allow for the addition of crosslinking agent into the extruder.

[0091] In some embodiments, the liquid crosslinking agent can be pumped into the extruder using a diaphragm pump. The internal components and seals of the diaphragm pump may have resistivity to chemicals such as DVB. Diaphragm pumps are commercially available, such as the Lewa LDC diaphragm pump. FIG. 12 shows an example of a liquid diaphragm pump.

[0092] Insufficient dispersion of the liquid DVB may occur if it is injected after the plasticating section of the screw, such as after 16D. This may result in localized concentrations of liquid DVB in extruded structures that will manifest into inconsistent levels of crosslinking in during irradiation and defects during foaming such as bubbles or blisters.

[0093] FIG. 12 includes images of poor and good mixing of DVB depending on the injection location of an extruder barrel. The two upper pictures show poorly mixed DVB. In these examples, the liquid DVB was dyed with a black dye and injected after 16D without any blowing agent present. The resulting compounded sheet was then examined under a microscope at 400X magnification to qualitatively ascertain how well the DVB was mixed (i.e., dispersed) within the base polymer. It is clear that injecting after 16D resulted in large domains of DVB that were not dispersed or mixed very well within the base polymers.

[0094] In addition, if the liquid is injected into a section of the extruder under pressure, the pump must overcome this melt pressure to prevent the injector from clogging or plugging with polymer. If the DVB is injected between 4D and 12D, good mixing and dispersion of the DVB will be achieved. The image on the bottom of FIG. 12 shows good mixing of DVB,

as a result of injecting between 4D and 12D. No large domains of dyed DVB can be seen in this example.

[0095] If the DVB is added directly into the feed between 0 and 4D, good dispersion may be achieved, however, it can wet the feed area causing polymer and other materials to stick and break off in clumps. In addition, it can have a tendency to pool and accumulate at the bottom of the barrel. Therefore, in some embodiments, injecting the liquid DVB into extruder between 4D and 12D, between 4D and 8D, between 4D to 7D, between 4D to 6D, between 4D to 5D, or between 4D to 4.5D can allow proper dispersion of the liquid within the polymer by subjecting it to the high shear plasticating section of the screws and not allowing the liquid to pool in the feed area.

[0096] Other suitable crosslinking monomers include commercially available difunctional, trifunctional, tetrafunctional, pentafunctional, and higher functionality monomers. Such crosslinking monomers are available in liquid, solid, pellet, and powder forms. Examples include, but are not limited to, acrylates or methacrylates such as 1,6-hexanediol diacrylate, 1,6-hexanediol dimethacrylate, ethylene glycol diacrylate, ethylene glycol dimethacrylate, trimethylol propane trimethacrylate, tetramethylol methane triacrylate, 1,9-nonanediol dimethacrylate and 1,10-decanediol dimethacrylate; allyl esters of carboxylic acid (such as trimellitic acid triallyl ester, pyromellitic acid triallyl ester, and oxalic acid diallyl ester); allyl esters of cyanuric acid or isocyanuric acid such as triallyl cyanurate and triallyl isocyanurate; maleimide compounds such as N-phenyl maleimide and N,N'-m-phenylene bismaleimide; compounds having at least two tribonds such as phthalic acid dipropargyl and maleic acid dipropargyl; and divinylbenzene. These crosslinking monomers may be used alone or in combinations thereof.

[0097] Different methods may be used to compound and extrude structures, especially when using crosslinking agents other than DVB. For example, crosslinking agents may be solids rather than liquids, which may not be added to a mixture of material ingredients. If a solid crosslinking agent is added to the mixture, it is still desirable to pulverize the resin to get a homogenous blend between the resin and powder blowing agent. However, using a DVB liquid as a crosslinking agent provides a more uniform and controlled crosslinking compared to using solid crosslinking agents.

[0098] The barrel element can also include cooling channels. The cooling channels are configured to allow a cooling fluid to enter the barrel element and exit the barrel element removing heat that may build up in the barrel element. Similar to the injection ports, internal cooling channels are shown in dotted lines in Figures 16, 17, and 18. For example, a cooling inlet channel 15 can extend from a second external side 16 of the body in a first direction. In some embodiments, the cooling inlet channel can extend from a second external side of the body towards the first external side of the body. In addition, the barrel element can include a cooling outlet channel 17 that extends from a third external side of the body. In some embodiments, the second external side of the body and the third external side of the body are the same. In some embodiments, the cooling outlet channel can extend in the first direction (i.e., the same as the cooling inlet channel). In other embodiments, the cooling outlet channel can extend in a different direction than the first direction. In some embodiments, the cooling inlet channel and/or the cooling inlet channel can extend in a direction from one side of the barrel element towards the opposite side. In some embodiments, the cooling inlet channel and/or the cooling outlet channel can extend in the direction of the height X of the barrel element. In some embodiments, the cooling inlet channel can extend in a direction that is parallel to the direction the cooling outlet channel extends. In some embodiments, the

cooling inlet channel and the cooling outlet channel are on opposite sides of the internal opening.

[0099] Figure 16A also shows connecting cooling channels 19 and 20. These connecting cooling channels can be fluidly connected to the inlet cooling channel and the outlet cooling channel such that a cooling fluid can flow through the inlet cooling channel, through the connecting cooling channels, and then through the cooling outlet channel to exit the device. As such, the connecting cooling channels are fluidly connected between the cooling inlet channel and the cooling outlet channel. Connecting cooling channels 19 and 20 can extend in the direction of the length Y of the barrel element. In some embodiments, the connecting cooling channels can extend internally from a third external side of the body. In some embodiments, connecting cooling channel 19 and connecting cooling channel 20 can extend in a direction from one side of the barrel element towards the opposite side. In some embodiments, the connecting cooling channel 19 and connecting cooling channel 20 can extend in a direction that is parallel to each other. In some embodiments, connecting cooling channel 19 and connecting cooling channel 20 can extend in a direction that is perpendicular to the cooling inlet channel and/or the cooling outlet channel. In some embodiments, connecting cooling channel 19 and connecting cooling channel 20 are on opposite sides of the internal opening.

[0100] Unlike Figure 16A which shows a single connecting cooling channels 19 and 20 on opposite sides of the internal opening, Figure 17A and Figure 18 show two groups of a plurality of connecting cooling channels 19A and 20A. Figure 17A illustrates a first group 19A having three connecting cooling channels and a second group 20A having three connecting cooling channels. Figure 18 illustrates a first group 19A having four connecting cooling channels and a second group 20 having four connecting cooling channels. Each

group can have any number of connecting cooling channels. In addition, each group of the plurality of connecting cooling channels can be fluidly connected to the inlet cooling channel and the outlet cooling channel such that a cooling fluid can flow through the inlet cooling channel, through each of the connecting cooling channels in the plurality of connecting cooling channels, and then through the cooling outlet channel to exit the device. As such, each group of a plurality of connecting cooling channels is fluidly connected between the cooling inlet channel and the cooling outlet channel. A group of a plurality of connecting cooling channels can extend in the direction of the length Y of the barrel element. In some embodiments, a group of a plurality of connecting cooling channels can extend internally from a third external side of the body. In some embodiments, a group of connecting cooling channels can extend in a direction from one side of the barrel element towards the opposite side. In some embodiments, a group of connecting cooling channels can extend in a direction that is parallel to another group of connecting cooling channels. In some embodiments, a group of connecting cooling channels can extend in a direction that is perpendicular to the cooling inlet channel and/or the cooling outlet channel. In some embodiments, two groups of connecting cooling channels can be on opposite sides of the internal opening.

[0101] The groups of plurality of cooling channels can allow for the barrel element to more easily maintain and control the temperature. If the temperature of the barrel element gets too high, the injection liquid can polymerize, thereby clogging the at least one injection port. As such, it can be important to control and maintain a temperature below the boiling point of the liquid that is to be injected in the at least one injection port.

[0102] The cooling channels (e.g., inlet, outlet, connecting) can be formed by drilling into the body of the barrel element. As such, some of the cooling channels may have plugs 21. These

plugs are used to prevent any cooling fluid from exiting the device anywhere other than the cooling channel outlet.

[0103] In order to determine whether the temperature of the barrel element is properly maintained for injecting liquid, the barrel element can include a temperature sensor well 22. Similar to the cooling channels and the injection ports, the temperature sensor well can be shown as dotted lines indicating that the well is inside the body of the barrel element. The temperature sensor well can be configured to receive a temperature measuring device such as a temperature probe in order to determine the temperature of the barrel element near the site of injection. The temperature sensor well can extend from an external side of the barrel element towards the interior opening. In some embodiments, the external side of the barrel element can be the first side 12. Besides extending towards the internal opening, the temperature sensor well can also extend towards the outlet of the at least one injection port. Thus, the distal end 23 of the temperature sensor well can be in close proximity to or adjacent to the outlet of the at least one injection port. This distal end can be closed (i.e., it does not have access to the internal opening). In some embodiments, the temperature sensor well extends in the height wise direction X of the barrel element. In some embodiments, the temperature sensor well can extend in a direction that is parallel to the inlet cooling channel, outlet cooling channel, and/or the at least one injection port. In some embodiments, the temperature sensor well can extend from an external side of the body in a direction that is perpendicular to a tangent of the outer perimeter (18) of the internal opening.

[0104] The closer the distal end of the temperature sensor well is to the outlet of the at least one injection port, the better as that location provides a more accurate temperature reading of the temperature at the point of liquid injection. In some embodiments, the temperature sensor well can be located between a first injection port and a second injection port. In some

embodiments, the barrel element can have multiple temperature sensor wells. For example, if the barrel element has multiple injection ports, each injection port can have a corresponding temperature sensor well extending towards it.

[0105] As shown in Figure 16B, 17B, and 17C, the injection port(s), the cooling channels (there is intersection between at least some of the various cooling channels to allow a cooling fluid to flow through the device), and the temperature sensor wells do not intersect.

Accordingly, any fluid or device (e.g., a temperature measurement device) traveling through a port would not come into contact with a cooling channel or the temperature sensor well; any fluid or device traveling through a cooling channel would not come into contact with a port or the temperature sensor well; and any fluid or device traveling in the temperature sensor well would not come into contact with a port or a cooling channel.

BLOWING AGENT

[0106] In some embodiments, a chemical blowing agent (i.e., foaming agent) is fed through side stuffer 504 to extruder 500 at a fifth barrel segment, between 16D and 20D along the axial length. In particular, the chemical blowing agent can be fed above by a gravimetric loss-in-weight feeder. In some embodiments, side stuffing could be bypassed by introducing the chemical blowing agent into a throat or feed section of the extruder, which corresponds to the first barrel segment between 0 and 4D. However, bypassing the side stuffer increases the likelihood of prematurely activating the foaming agent in plasticating (melting and mixing) section. The plasticating section of extruder 500 provides aggressive kneading discs that create dispersive shear to melt the polymer in barrel sections two and three, and in turn create viscous generation of heat. If the mixing elements of the screw are altered to lessen heat generation, inadequate melting of the resin can occur for high melting point and high viscosity resins, in addition to creating a “poor mixing” condition with respect to the blowing

agent, as shown in FIG. 13. Therefore, adding the chemical blowing agent in the feed section (e.g., first barrel section) is not preferred.

[0107] On the other hand, adding the chemical blowing agent too far downstream between 24D and 32D (e.g., seventh or eighth segment) does not allow for proper distributive mixing of the agent, resulting in large concentrations or clumps that will manifest into defects during the foaming process. Therefore, side stuffing the chemical blowing agent is preferred between 16D and 26D, or more preferably between 16D and 20D to achieve “good mixing” and proper incorporation into the polymer melt without premature activation or decomposition. Introducing the foaming agent in these zones of the extruder facilitates “good mixing” and incorporation without premature activation or decomposition to avoid the “poor mixing” shown in FIG. 13.

[0108] Any type of chemical blowing agent may be used in the extrusion process disclosed herein. Examples of chemical blowing agents include azo compounds, hydrazine compounds, carbazides, tetrazoles, nitroso compounds and carbonates. A chemical blowing agent may be employed alone or in any combination. Other examples of chemical blowing agents include powdered exothermic azodicarbonamide (ADCA), endothermic acid-carbonate system, or combinations thereof.

[0109] In some embodiments, azodicarbonamide (ADCA) is used as the chemical blowing agent. The thermal decomposition of ADCA typically occurs at temperatures between 190 to 230°C. Accordingly, extruding temperature is maintained at or below 190°C to prevent ADCA from thermally decomposing in the extruder. The particle size of ADCA is usually between 9-50 microns depending on grade.

[0110] ADCA is a preferred blowing agent over acid-carbonate systems due to a higher gas volume generated. As a result, less blowing agent is required for the same expansion

compared to an endothermic acid-carbonate system. In some embodiments, a percentage of blowing agent used is between 1-30 parts per hundred parts resin (phr) for the production of irradiation crosslinked polypropylene foam. In some embodiments, the amount of ADCA is 3-20% parts by weight of the material ingredients.

[0111] A lower temperature limit for extrusion is that of the polymer with the highest melting point. If the extrusion temperature drops below the melting temperature of the polymer with the highest melting point, then undesirable “unmelts” appear in the sheet-like profile. Upon foaming, an extruded sheet-like profile that was extruded below this lower temperature limit will exhibit uneven thickness, a non-uniform cell structure, pockets of cell collapse, and other undesirable attributes.

[0112] If the difference between a decomposition temperature of a thermally decomposable blowing agent and a melting point of a polymer with a highest melting point is large, then a catalyst for a blowing agent decomposition may be used. Exemplary catalysts include, but are not limited to, zinc oxide, magnesium oxide, calcium stearate, glycerin, and urea.

IRRADIATION

[0113] In the irradiation step, the extruded structure is subjected to irradiation with ionizing radiation at a given exposure to crosslink the composition, thereby obtaining a crosslinked sheet.

[0114] Crosslinking is a process of bonding polymer chains to each other. Crosslinks may be generated using a variety of different techniques and can be formed both intermolecularly, between different polymer molecules, and intramolecularly between portions of a single polymer molecule. Such techniques include providing crosslinking monomers which are separate from a polymer chain and providing polymer chains which incorporate a

crosslinking monomer containing a functional group that can form a crosslink or be activated to form a crosslink.

[0115] An extruded structure is subjected to irradiation with ionizing radiation at a given exposure to crosslink the composition, thereby obtaining a crosslinked extruded structure. Ionizing radiation produces an extruded structure with an excellent surface appearance and uniform cells. A crosslinking monomer may be added to promote crosslinking because ionizing radiation alone is unable to produce a sufficient degree of crosslinking on polypropylene(s), polypropylene based materials, some polyethylene(s), and some polyethylene based materials.

[0116] Examples of ionizing radiation include, but are not limited to, alpha, beta (electron beams), x-ray, gamma, and neutron. Among them, an electron beam having uniform energy is preferably used to prepare irradiated extruded structures. Exposure time, frequency of irradiation and acceleration voltage upon irradiation with an electron beam can vary widely depending on an intended crosslinking degree and thickness of the extruded structure. However, it should generally be in the range of about 10 to 500 kGy, and preferably from about 20 to 300 kGy, and more preferably from about 20 to 200 kGy.

[0117] If the exposure to radiation is too low, then cell stability is not maintained upon foaming. If the exposure is too high, the moldability of the resulting foam sheet may be poor. Moldability, as referred to herein, is a desirable property when the foam sheet is used in thermoforming applications. The polymer components will also be degraded from excessive polymer chain scission. Also, the extruded structure profile may be softened by exothermic heat release upon exposure to electron beam radiation such that the structure can deform with excessive exposure.

[0118] The irradiation frequency is preferably no more than four times, more preferably no more than two times, and even more preferably just one time. If the irradiation frequency is more than about 4 times, then the polymer components may suffer degradation so that upon foaming, for example, uniform cells will not be created in the resulting foam composition.

[0119] When the thickness of the sheet-like profile is greater than about 4 mm, irradiating each primary surface of the profile with an ionized radiation is preferred to make the degree of crosslinking of the primary surface(s) and the inner layer more uniform.

[0120] Irradiation with an electron beam provides an advantage in that sheet-like profiles having various thicknesses can be effectively crosslinked by controlling an acceleration voltage of electrons. The acceleration voltage is generally in the range of about 200 to 3000 kV, and preferably from about 400 to 1200 kV, and more preferably from about 600 to 1000 kV.

[0121] If the acceleration voltage is less than about 200 kV, then radiation cannot reach the inner portion of the sheet-like profile. As a result, the cells in the inner portion may be coarse and uneven upon foaming. Additionally, acceleration voltage that is too low for a given thickness profile will cause arcing, resulting in “pinholes” or “tunnels” in the foamed sheet. On the other hand, the polymers may degrade, and the proper dose as a function of cross section may not be achieved, if the acceleration voltage is greater than about 1500 kV.

[0122] Regardless of the type of ionizing radiation selected, crosslinking is performed so that the composition is crosslinked from 20 to 75% and more preferably from 30 to 60%, as measured by the “Toray Gel Fraction Method” and detailed in the sections below.

FOAMING

[0123] Foaming may be performed by heating the crosslinked structure with molten salt, radiant heaters, vertical hot air oven, horizontal hot air oven, microwave energy, or any

combinations thereof, to activate the foaming agent (i.e., chemical blowing agent) in the extruded structure to expand the polymer of the structure. In some embodiments, the foaming may be conducted in an impregnation process using, for example, nitrogen in an autoclave, followed by a free foaming via molten salt, radiant heaters, vertical hot air oven, horizontal hot air oven, microwave energy or any combinations thereof.

[0124] In some embodiments, a combination of molten salt and radiant heaters is used to heat a crosslinked sheet-like profile. In some embodiments, the crosslinked sheet-like profile can be softened with preheating before foaming. Pre-heating helps stabilize the expansion of the sheet-like material upon foaming.

[0125] Foaming is performed by heating the crosslinked extruded structure to a temperature higher than the decomposition temperature of the thermally decomposable blowing agent. For the thermally decomposable blowing agent ADCA, foaming is performed in a continuous process at a temperature of about 200 to 260°C or preferably at about 220 to 240°C. A continuous foaming process is preferred over a batch process for production of a continuous foam sheet.

[0126] Foams produced using the methods and processes described herein can have a section or overall density of about 20 to 250 kg/m³, or preferably from about 30 kg/m³ to 125 kg/m³, as measured by JIS K6767. The section density can be controlled by an amount of blowing agent and the thickness of the extruded structure profile. If the density of a sheet is less than about 20 kg/m³, then the sheet does not foam efficiently due to a large amount of chemical blowing agent needed to attain the desired density. Consequently, it becomes increasingly difficult to control expansion, control uniform section density and thickness, and prevent cell collapse.

[0127] Foam compositions are not limited to a section density of 250 kg/m³. A foam composition of 350 kg/m³, 450 kg/m³, or 550 kg/m³ may also be produced. However, a foam composition having a density of less than 250 kg/m³ is preferred because greater densities are cost prohibitive when compared to other materials which can be used for the same applications.

[0128] Foam compositions discussed herein have closed cells. Preferably, at least 90% of the cells have undamaged cell walls, more preferably at least 95%, even more preferably more than 98%. The average cell size is preferably from 0.05 to 1.0 mm, and most preferably from 0.1 to 0.7 mm. If the average cell size is lower than 0.05 mm, then the foam density is typically greater than 250 kg/m³. If the average cell size is larger than 1 mm, the foam will have an uneven surface. There is also a possibility of the foam composition being undesirably torn if the population of cells in the foam does not have a preferred average cell size where the foam is stretched or portions are subjected to a secondary process. In some embodiments, the cell size in a foam composition may have a bimodal distribution representing a population of cells in the core of the foam composition which are relatively round and a population of cells in the skin, near the surfaces of the foam composition, which are relatively flat, thin, and/or oblong.

[0129] In some embodiment, the thickness of the foam composition can be about 0.2 mm to 50 mm, preferably from about 0.4 mm to about 40 mm, more preferably from 0.6 mm to 30 mm, and even more preferably from 0.8 mm to 20 mm. If thickness is less than 0.2 mm, then foaming is not efficient due to significant gas loss from primary surfaces. If thickness is greater than about 50 mm, it is increasingly difficult to control expansion, and produce a structure with uniform section density and thickness.

[0130] In some embodiments, the extruded structure may be subjected to secondary processes, including but not limited to, embossing, corona or plasma treatment, surface roughening, surface smoothing, perforation or microperforation, splicing, slicing, skiving, layering, bonding, hole punching, and the like. A desired thickness of an extruded structure, such as a sheet, can be obtained by slicing, skiving, or bonding to produce a thickness of 0.1 mm to about 100 mm.

[0131] The foam composition may also contain other compatible additives. Common additives include, but are not limited to, organic peroxides, antioxidants, lubricants, thermal stabilizers, colorants, flame retardants, antistatic agents, nucleating agents, plasticizers, antimicrobials, antifungals, light stabilizers, UV absorbents, anti-blocking agents, fillers, deodorizers, thickeners, cell size stabilizers, metal deactivators, and combinations thereof.

MEASUREMENTS

[0132] Properties of materials described herein are measured according to the following.

[0133] Crosslinking is measured by using the “Toray Gel Fraction Method.” According to this method, tetralin solvent dissolves non-crosslinked material and a crosslinking degree is expressed as weight percentage of crosslinked material.

[0134] An apparatus used to determine a percent of polymer crosslinking includes a 100 mesh, 0.0045 inch wire diameter; Type 304 stainless steel bags; numbered wires and clips; a Miyamoto thermostatic oil bath apparatus; an analytical balance; a fume hood; a gas burner; a high temperature oven; an anti-static gun; and three 3.5 liter wide mouth stainless steel containers with lids. Reagents and materials used to determine a percent of crosslinking include tetralin high molecular weight solvent, acetone, and silicone oil.

[0135] In particular, the weight of an empty wire mesh bag is measured and recorded. For each sample, about 100 milligrams $\pm$ about 5 milligrams of sample is weighed and transferred

to the wire mesh bag. The weight of the wire mesh bag and the sample, typically in the form of foam cuttings, is recorded. Each bag is attached to the corresponding number wire and clips. When the solvent temperature reaches 130°C, the combination of bag and sample is immersed in the solvent. The samples are shaken up-and-down about 5 or 6 times to loosen any air bubbles and fully wet the samples. The samples are attached to an agitator and agitated for three hours for the solvent to dissolve the foam. The samples are then cooled in a fume hood.

[0136] The samples are washed by shaking up-and-down about 7 or 8 times in a container of primary acetone. The samples are washed a second time in a second acetone wash. The washed samples are washed once more in a third container of fresh acetone. The samples are then hanged in a fume hood for about 1 to about 5 minutes to evaporate the acetone. The samples are then dried in a drying oven for about 1 hour at 120°C. The samples are cooled for a minimum of about 15 minutes. The wire mesh bag is weighed on an analytical balance and the weight is recorded.

[0137] Crosslinking is then calculated using the formula $100 \cdot (C - A) / (B - A)$, where A = empty wire mesh bag weight; B = wire bag weight + foam sample before immersion in tetralin; and C = wire bag weight + dissolved sample after immersion in tetralin.

[0138] The melt flow index (MFI) value for a polymer is defined and measured according to ASTM D1238 at 230°C for polypropylenes and polypropylene based materials, and at 190°C for polyethylene and polyethylene based materials using a 2.16 kg plunger for 10 minutes. The test time may be reduced for relatively high melt flow resins.

[0139] MFI provides a measure of flow characteristics of a polymer and is an indication of the molecular weight and processability of a polymer material. If MFI values are too high, which corresponds to a low viscosity, extrusion according to the present disclosure cannot be

satisfactorily carried out. Problems associated with MFI values that are too high include low pressures during extrusion, problems setting the sheet thickness profile, uneven cooling profile due to low melt viscosity, poor melt strength and/ or machine problems. MFI values that are too low include high pressures during melt processing, calendering, sheet quality and profile problems, and higher extrusion temperatures which cause a risk of foaming agent decomposition and activation.

[0140] MFI is a good measurement for foaming because it reflects viscosity of a material, and viscosity has an effect on the foaming. Achieving a particular MFI value renders an effective foam composition. A lower MFI material may improve physical properties because molecular chain length is higher, which creates more energy needed for chains to flow when a stress is applied. Moreover, a longer molecular chain (MW) has more components that can crystallize to provide more strength through intermolecular ties. However, an MFI that is too low will cause viscosity to become too high.

[0141] Polymers with higher MFI values have shorter chains. Consequently, a volume of a material with higher MFI values has more chain ends relative to polymers having a lower MFI, which can rotate and create free volume due to the space needed for such rotation. For example, rotation occurring above the T_g or glass transition temperature of the polymer. This increases the free volume and enables easy flow under stress forces. For this disclosure, the MFI should be within the described ranges to provide an appropriate compromise between these properties.

[0142] Particle size distribution is measured according to ASTM D5644. In particular, material is sieved and amounts that are captured in a particular mesh size (e.g., 10, 16, 30, 50, 80 and 100 mesh, and what is captured in a bottom pan) are reported as percentages.

[0143] Specific Energy is defined as energy applied in kilowatts to material being processed by extruder 500, normalized per kilogram. Essentially, specific energy is an indicator of how much

work is being applied during extrusion and the intensity of the extrusion process. Applied and specific energy are calculated as follows:

$$\text{Specific Energy} = \frac{KW(\text{applied})}{\text{feedrate} \left(\frac{\text{kg}}{\text{hr}} \right)}$$

$$KW(\text{applied}) = \frac{KW(\text{motor rating}) \times 1.5 \times \text{torque from maximum allowable (in decimal form)} \times RPM(\text{actual running RPM}) \times 0.87 (\text{gearbox efficiency})}{\text{Max RPM (capability of extruder)}}$$

[0144] A density of an extruded structure, such as a sheet, is defined and measured using section or overall density, rather than a core density, according to JIS K6767.

Example

[0145] An extruded sheet is directly compounded on a 44:1 L/D co-rotating twin screw extruder using the raw material recipe shown in the TABLE labeled "Example: Recipe" below that will be irradiated and foamed to produce a foam with a density of 67 kg/m³, a thickness of 2.5 mm, and a crosslinking value of 40%. The polypropylene, polyethylene, antioxidant masterbatch, and additive are fed into the feed section (i.e., the first barrel segment or location 0-4D) using loss-in-weight feeders at a rate that is proportional to the size of the extruder being used. The ADCA is fed between 16D and 20D using a side stuffer, and the DVB injected into the barrel at 4D using the injector shown in Figure 10 inserted into the disclosed barrel element of the present invention at pressure below 20 psig.

[0146] The conditions to extrude the sheet in Example 1 are shown in the following TABLE labeled "Example: Extrusion Parameters." A 70 dutch weave (14 x 88) stainless steel wire mesh filter was used in this example, and the melt extruded through a 1.400 mm die gap opening to produce a compounded sheet with a thickness of 1.325 mm. The temperatures of

each barrel zone, the melt adaptors, gear pump, and die are also indicated in the Table. The inlet pressure to the gear pump is set to maintain a pressure of 550 psi. The extruder RPM is set to a value of 84 to achieve a sufficient number of melt divisions and achieve sufficient dispersive and distributive mixing so that defects do not occur during foaming. Under these operating conditions the melt temperature of the resin during extrusion is approximately 350 Fahrenheit as measured after the filter pack and before the gear pump, and load on the drive is 58%. The specific energy imparted to the polymer blend in this example is 0.149 kW/(kg/hr).

Example: Recipe

Polypropylene: commercially available random copolymer w/MFI of 1.85 g/10 min (230 Celsius, 2.16 kg) and a melting temperature of about 140 Celsius	34.3%
Polypropylene: commercially available random copolymer w/MFI of 0.75 g/10 min (230 Celsius, 2.16 kg) and a melting temperature of about 147 Celsius	34.3%
Polyethylene: commercially available LDPE-butene random copolymer w/MFI of 5 g/10 min (190 Celsius, 2.16 kg) and a melting temperature of about 125 Celsius	17.1%
Antioxidant masterbatch: LDPE carrier resin containing 14% active antioxidant blend comprised of basic commercially available phenolic and phosphite type antioxidants for plastics extrusion and 0.35% calcium stearate. MFI 9-17 g/10 min (190 Celsius, 2.16 kg)	4.7%
ADCA (azodicarbonamide): chemical foaming agent	5.5%
DVB (divinylbenzene): commercially available 80% purity	2.4%
Additive: commercially available process aid compounded into an LDPE-butene copolymer, active ingredients 3.0% of masterbatch w/MFI of 5.0 g/10 min (190 Celsius, 2.16 kg)	1.7%

Example: Extrusion Parameters

Filter	70 dutch weave
Initial Die Lip Gap	1.4 mm
Extruded Sheet	1.325 mm
Feed Zone	-
Zone #1	380°F
Zone #2	385°F
Zone #3	385°F
Zone #4	365°F
Zone #5	305°F
Zone #6	275°F
Zone #7	265°F
Zone #8	250°F
Zone #9	235°F
Zone #10	235°F
Adaptors	315°F
GP (Gear Pump)	330°F
Die	345°F
Extruder RPM	84
Target GP Inlet Pressure	550 psi

OTHER APPLICATIONS

[0147] Foam compositions disclosed herein can be used for a variety of additional applications. A soft and flexible foam composition can be used as a tape or gasket to seal double-pane windows, handheld electronic devices, and household appliances. A firm and rigid foam may be thermoformed for applications such as an automobile air duct.

[0148] In some embodiments, foam compositions can be combined with different layers such as, for example, a film and/or foil layer. The foam may be laminated on one or both sides with these materials and may comprise multiple layers. Layers may be joined by chemical bonds, mechanical means, electromagnetic charges, hydrophobic or hydrophilic attractions, or combinations thereof. The foam composition and laminate composition can be used for automobile interior parts such as door panels, armrests, center consoles, seats, and the like.

[0149] In some embodiments, foam compositions can also be used in furniture, room partitions, casing, flooring, and the like.

[0150] In some embodiments, pressure sensitive adhesives such as acrylic polymers, polyurethanes, thermoplastic elastomers, block copolymers, polyolefins, silicones, rubber based adhesives, copolymers of ethylhexylacrylate and acrylic acid, copolymers of isooctyl acrylate and acrylic acid, blends of acrylic adhesives, rubber based adhesives, and combinations thereof may be applied to at least one of the foam surfaces to facilitate attachment of the foam to objects.

[0151] In some embodiments, the foam can be die cut into useful articles such as gaskets.

[0152] The term “consist essentially of” means that the composition consists almost exclusively of the specified components except that additional unspecified component substances which do not materially affect the basic and novel characteristics of this invention can also be present. For example, the blended mixture may include stabilizers, colorants, antioxidants and other additives as required in such amounts that do not impair inherent performance thereof.

[0153] This application discloses several numerical ranges in the text and figures. The numerical ranges disclosed inherently support any range or value within the disclosed numerical ranges, including the endpoints, even though a precise range limitation is not stated verbatim in the specification because this disclosure can be practiced throughout the disclosed numerical ranges.

[0154] The above description is presented to enable a person skilled in the art to make and use the disclosure, and is provided in the context of a particular application and its requirements. Various modifications to the preferred embodiments will be readily apparent to those skilled in the art, and the generic principles defined herein may be applied to other embodiments and applications without departing from the spirit and scope of the disclosure. Thus, this disclosure is not intended to be limited to the embodiments shown, but is to be

accorded the widest scope consistent with the principles and features disclosed herein.

Finally, the entire disclosure of the patents and publications referred in this application are hereby incorporated herein by reference.

CLAIMS

1. A barrel element for a barrel extruder comprising:
 - a body having an interior opening extending through a width of the body,wherein the body comprises:
 - at least one injection port extending from a first external side of the body to the interior opening;
 - a temperature sensor well extending from the first external side of the body towards the interior opening;
 - a cooling inlet channel extending from a second external side of the body in a first direction;
 - a cooling outlet channel extending from a third external side of the body in the first direction; and
 - a plurality of connecting cooling channels extending in a second direction, wherein the cooling inlet channel and the cooling outlet channel are fluidly connected by the plurality of connecting cooling channels.
2. The barrel element of claim 1, wherein the at least one injection port is fluidly connected between the first external side of the body and the interior opening.
3. The barrel element of claim 1, wherein the at least one injection port extends from the first external side of the body in a direction that is perpendicular to a tangent of an outer perimeter of the interior opening.
4. The barrel element of claim 1, wherein the body comprises a second injection port extending from the first external side of the body to the interior opening.
5. The barrel element of claim 4, wherein the second injection port is fluidly connected between the first external side of the body and the interior opening.

6. The barrel element of claim 1, wherein a distal end of the temperature sensor well is closed.
7. The barrel element of claim 6, wherein the distal end of the temperature sensor well is adjacent to an outlet of the at least one injection port.
8. The barrel element of claim 1, wherein the second external side of the body and the third external side of the body are the same.
9. The barrel element of claim 1, wherein the cooling inlet channel and the cooling outlet channel are parallel.
10. The barrel element of claim 1, wherein the cooling inlet channel and the cooling outlet channel are on opposite sides of the interior opening.
11. The barrel element of claim 1, wherein the plurality of connecting cooling channels are perpendicular to the cooling inlet channel and/or cooling outlet channel
12. The barrel element of claim 1, wherein the cooling inlet channel extends towards the first external side of the body.
13. The barrel element of claim 1, wherein the cooling outlet channel extends towards the first external side of the body.
14. The barrel element of claim 1, wherein the plurality of connecting cooling channels comprises a first group of connecting cooling channels and a second group of connecting cooling channels.
15. The barrel element of claim 14, wherein the first group of connecting cooling channels and the second group of connecting cooling channels are on opposite sides of the interior opening.

16. The barrel element of claim 1, wherein the width of the body is 20-60 mm.
17. A method of manufacturing an extruded structure, comprising:
introducing resin into a feeder of an extruder;
introducing a liquid crosslinking agent into the extruder through a barrel element at a location downstream of the feeder, wherein the barrel element comprises:
a body having an interior opening extending through a width of the body,
wherein the body comprises:
at least one injection port extending from a first external side of the body to the interior opening;
a temperature sensor well extending from the first external side of the body towards the interior opening;
a cooling inlet channel extending from a second external side of the body in a first direction;
a cooling outlet channel extending from a third external side of the body in the first direction; and
a plurality of connecting cooling channels extending in a second direction,
wherein the cooling inlet channel and the cooling outlet channel are fluidly connected by the plurality of connecting cooling channels;
introducing a chemical blowing agent into the extruder at a location downstream of the location where the liquid crosslinking agent is introduced into the extruder; and
extruding a structure from the extruder.
18. The method of claim 17, further comprising irradiating the extruded structure to produce a crosslinked extruded structure.
19. The method of claim 18, further comprising foaming the crosslinked extruded structure to produce a foam structure.

20. The method of claim 19, wherein the foaming includes pre-heating the crosslinked extruded structure.

21. The method of claim 20, wherein the foaming includes using a salt bath as a heat source to activate the chemical blowing agent in the crosslinked extruded structure.

22. The method of claim 19, wherein the foaming includes using at least one of a radiant heater, a hot air oven, or microwave energy as a heat source to activate the chemical blowing agent in the crosslinked extruded structure.

23. The method of claim 17, wherein the chemical blowing agent is introduced into the extruder through a side stuffer downstream of a location where the liquid crosslinking agent is introduced into the extruder.

24. The method of claim 17, wherein the resin introduced into the feeder has a non-powder form.

25. The method of claim 24, wherein the resin is formed as pellets, granules, chips, flakes, beads, cylinders, or tubes.

26. The method of claim 25, wherein the resin comprises a polypropylene based polymer comprising homopolymer polypropylene, MAH-g-polypropylene, impact modified polypropylene, polypropylene-ethylene copolymer, MAH-g-polypropylene-ethylene copolymer, metallocene polypropylene, metallocene polypropylene-ethylene copolymer, metallocene polypropylene olefin block copolymer with a controlled block sequence, polypropylene based polyolefin plastomer, polypropylene based polyolefin elasto-plastomer, polypropylene based polyolefin elastomer, polypropylene based thermoplastic polyolefin blend and polypropylene based thermoplastic elastomeric blend.

27. The method of claim 17, wherein the extruded structure has a sheet-like profile and is at least one of a film, a web or a sheet.

28. The method of claim 17, wherein the extruding is performed with a co-rotating, twin screw extruder.

29. The method of claim 28, wherein the extruder has a length to screw diameter ratio of 36:1 to 52:1.

30. The method of claim 28, wherein the extruder has a length greater than 20 screw diameters.

31. The method of claim 30, wherein the feeder is located within an initial 4 screw diameters, the liquid crosslinking agent is introduced into the barrel element within 4 to 8 screw diameters and the blowing agent is introduced into the extruder within 16 to 20 screw diameters.

32. The method of claim 29, wherein each twin screw has a diameter of 27 to 135 mm.

33. The method of claim 17, wherein a temperature within the extruder is maintained at least 10 degrees Celsius below a thermal decomposition initiation temperature of the chemical blowing agent.

34. The method of claim 17, wherein the chemical blowing agent has domains each of a radius size less than 16 μm in the extruded structure.

35. The method of claim 17, wherein the liquid crosslinking agent is divinylbenzene.

36. The method of claim 17, wherein the chemical blowing agent is azodicarbonamide.

37. The method of claim 19, wherein the foam has a density of 20 to 250 kg/m^3 .

38. A method for manufacturing an extruded structure, comprising:
introducing resin at a first zone of the extruder;

introducing a liquid crosslinking agent at a barrel element between the first zone and a second zone of an extruder, wherein the second zone is downstream of the first zone and the barrel element comprises:

a body having an interior opening extending through a width of the body,
wherein the body comprises:

at least one injection port extending from a first external side of the body to the interior opening;

a temperature sensor well extending from the first external side of the body towards the interior opening;

a cooling inlet channel extending from a second external side of the body in a first direction;

a cooling outlet channel extending from a third external side of the body in the first direction; and

a plurality of connecting cooling channels extending in a second direction,
wherein the cooling inlet channel and the cooling outlet channel are fluidly connected by the plurality of connecting cooling channels;

introducing a chemical blowing agent at a third zone of the extruder; and

extruding a structure at a fourth zone of the extruder.

39. The method of claim 38, further comprising irradiating the extruded structure to crosslink the resin of the extruded structure.

40. The method of claim 39, further comprising foaming the irradiated crosslinked structure to produce a foam structure.

41. The method of claim 38, wherein the third zone is downstream of the second zone.

42. The method of claim 38, wherein the extruded structure comprises 30 to 80% polypropylene.

43. The method of claim 38, wherein the crosslinked composition comprises a crosslinking degree of 20 to 75%.

44. The method of claim 38, wherein the extruding is performed with a co-rotating, twin screw extruder having a length greater than 20 screw diameters, the first zone is within the initial 4 screw diameters, the second zone is within 4 to 8 screw diameters, and the third zone is within 16 to 20 screw diameters.

45. An extruded structure produced by a method, comprising:
introducing resin into a feeder of an extruder;
introducing a liquid crosslinking agent into the barrel element at a location downstream of the feeder, wherein barrel element comprises:
a body having an interior opening extending through a width of the body,
wherein the body comprises:
at least one injection port extending from a first external side of the body to the interior opening;
a temperature sensor well extending from the first external side of the body towards the interior opening;
a cooling inlet channel extending from a second external side of the body in a first direction;
a cooling outlet channel extending from a third external side of the body in the first direction; and
a plurality of connecting cooling channels extending in a second direction,
wherein the cooling inlet channel and the cooling outlet channel are fluidly connected by the plurality of connecting cooling channels;
introducing a chemical blowing agent into the extruder; and
extruding a structure from the extruder, wherein the chemical blowing agent has domains each of a radius size less than 16 μm in the extruded structure.

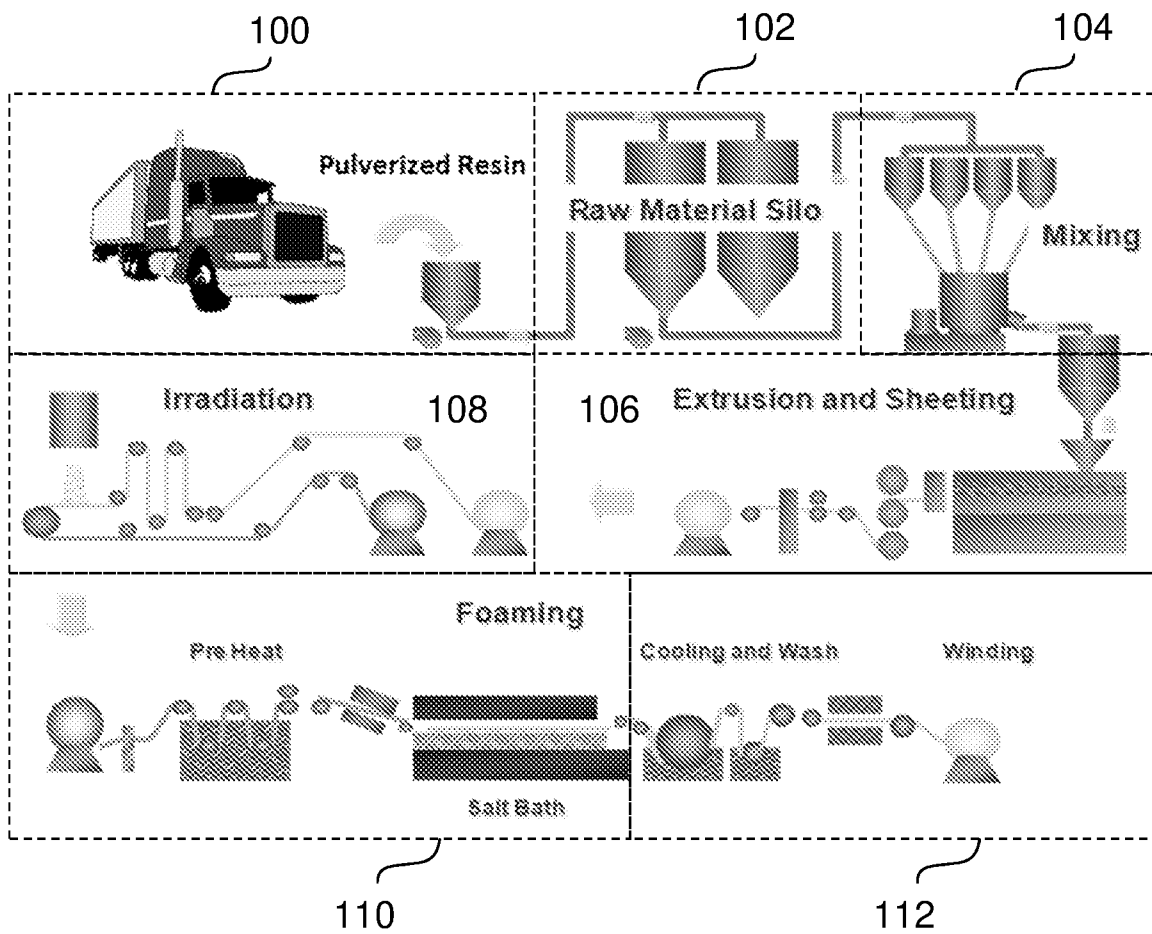


FIG. 1

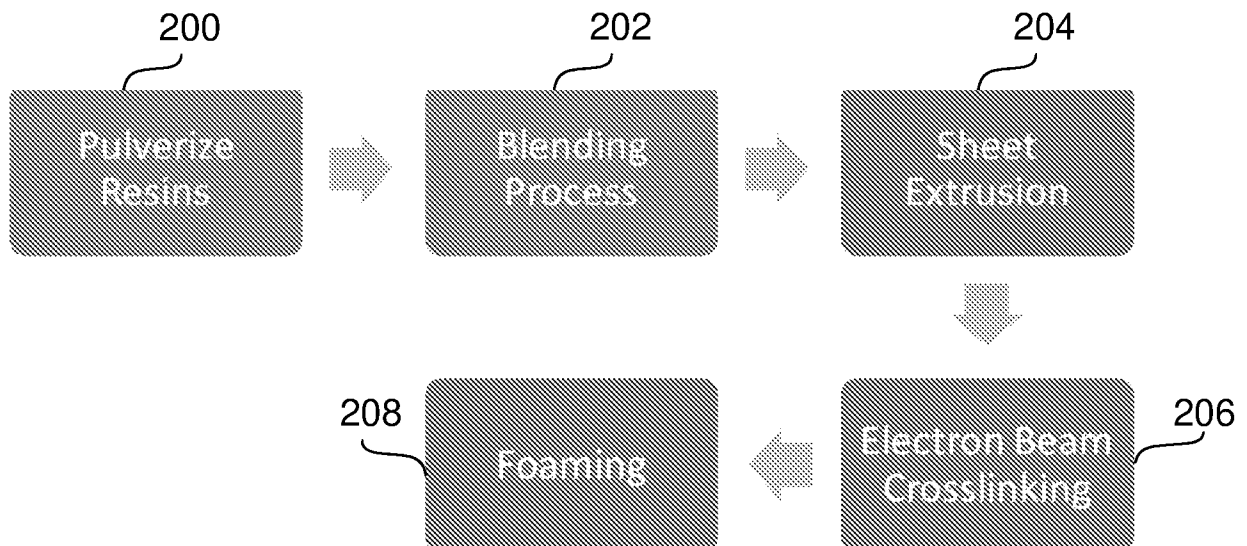


FIG. 2

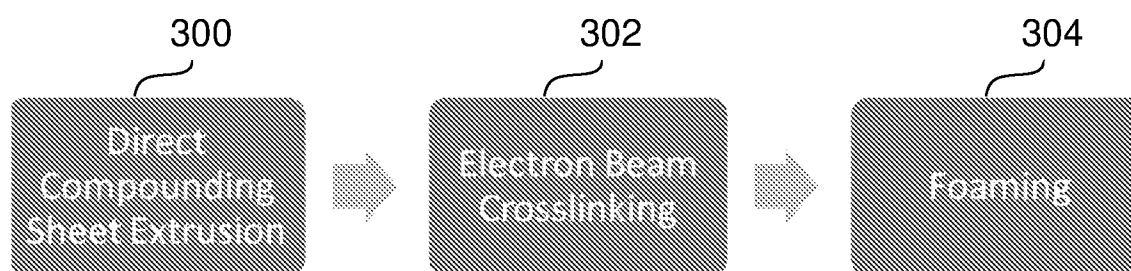


FIG. 3

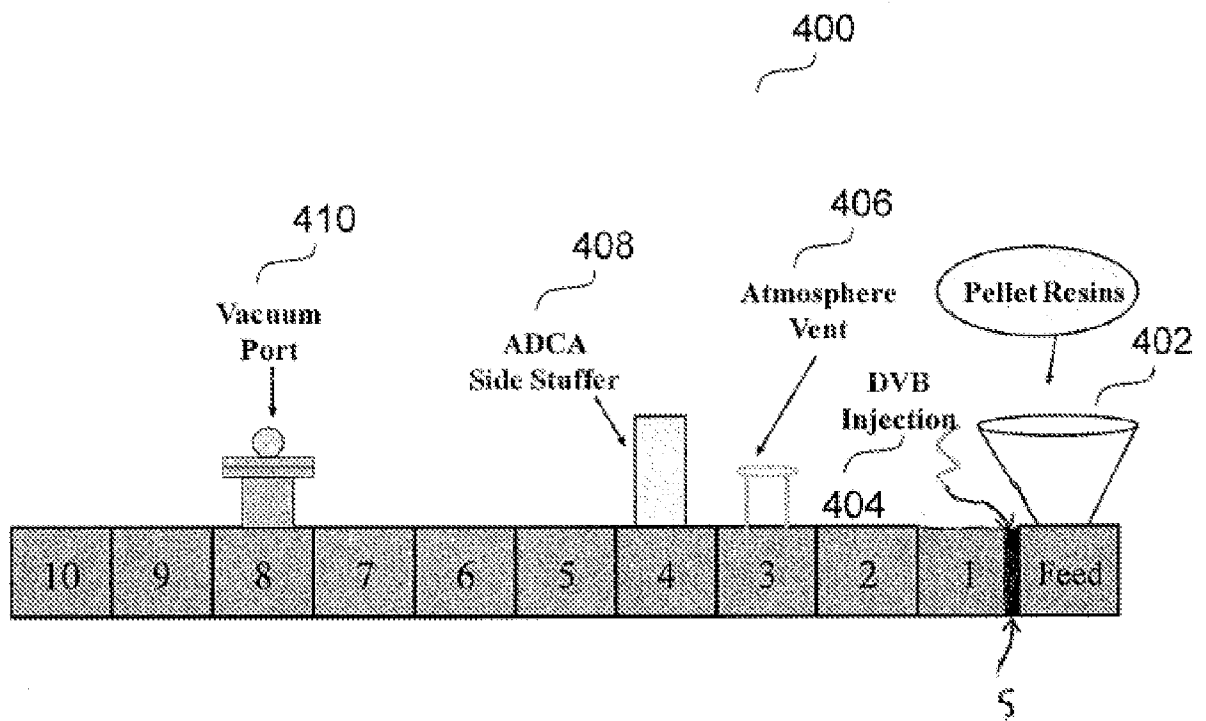


FIG. 4

5/21

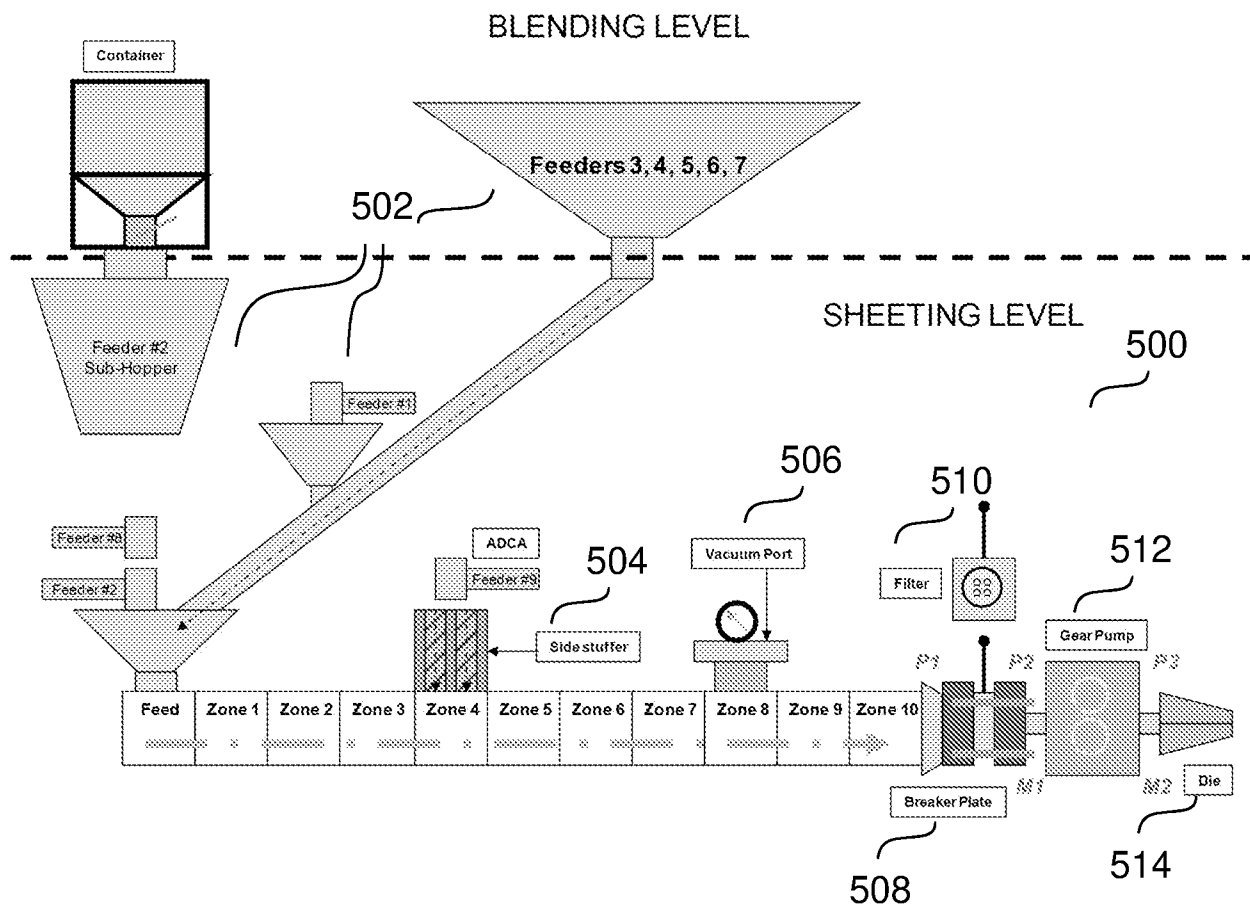


FIG. 5

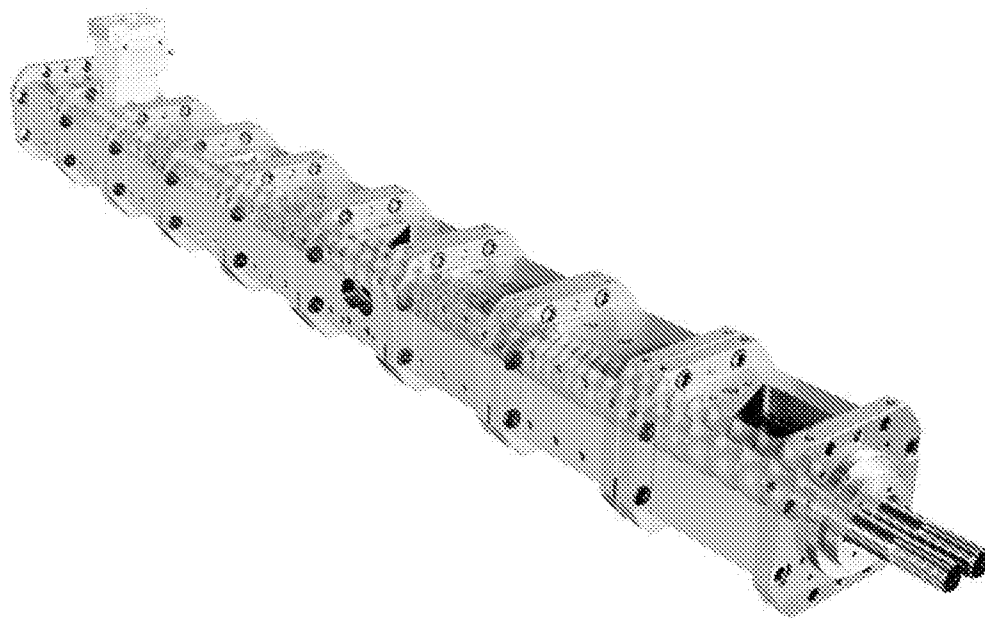


FIG. 6

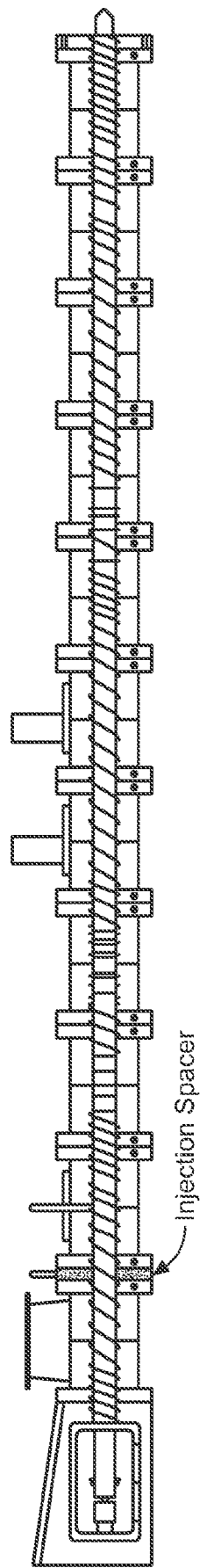


FIG. 7

Barrel Section Temperature Control

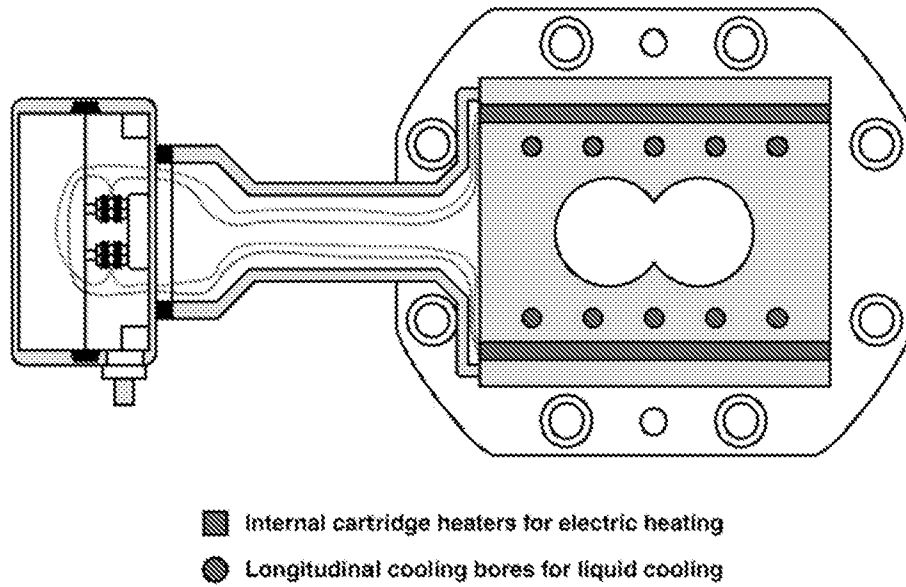


FIG. 8

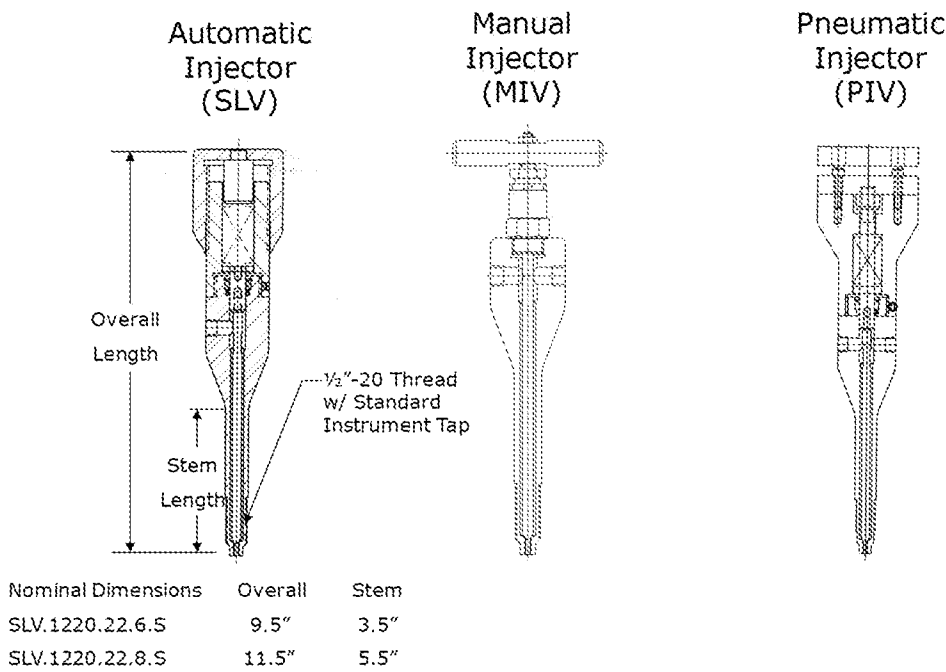


FIG. 9

10/21

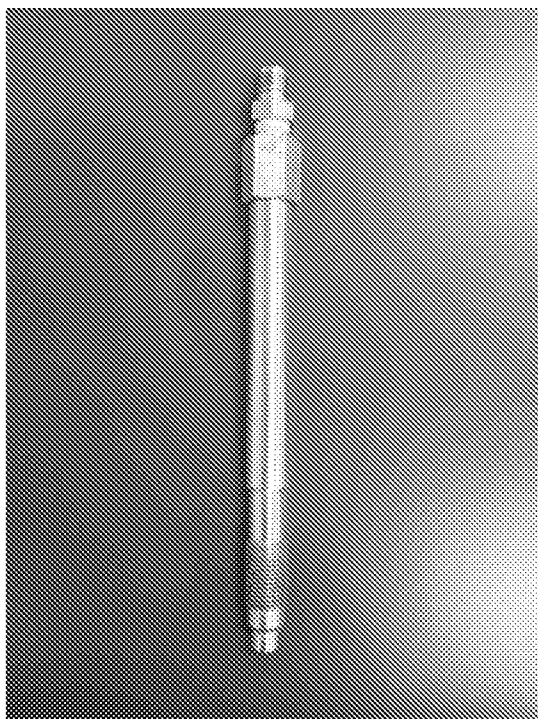
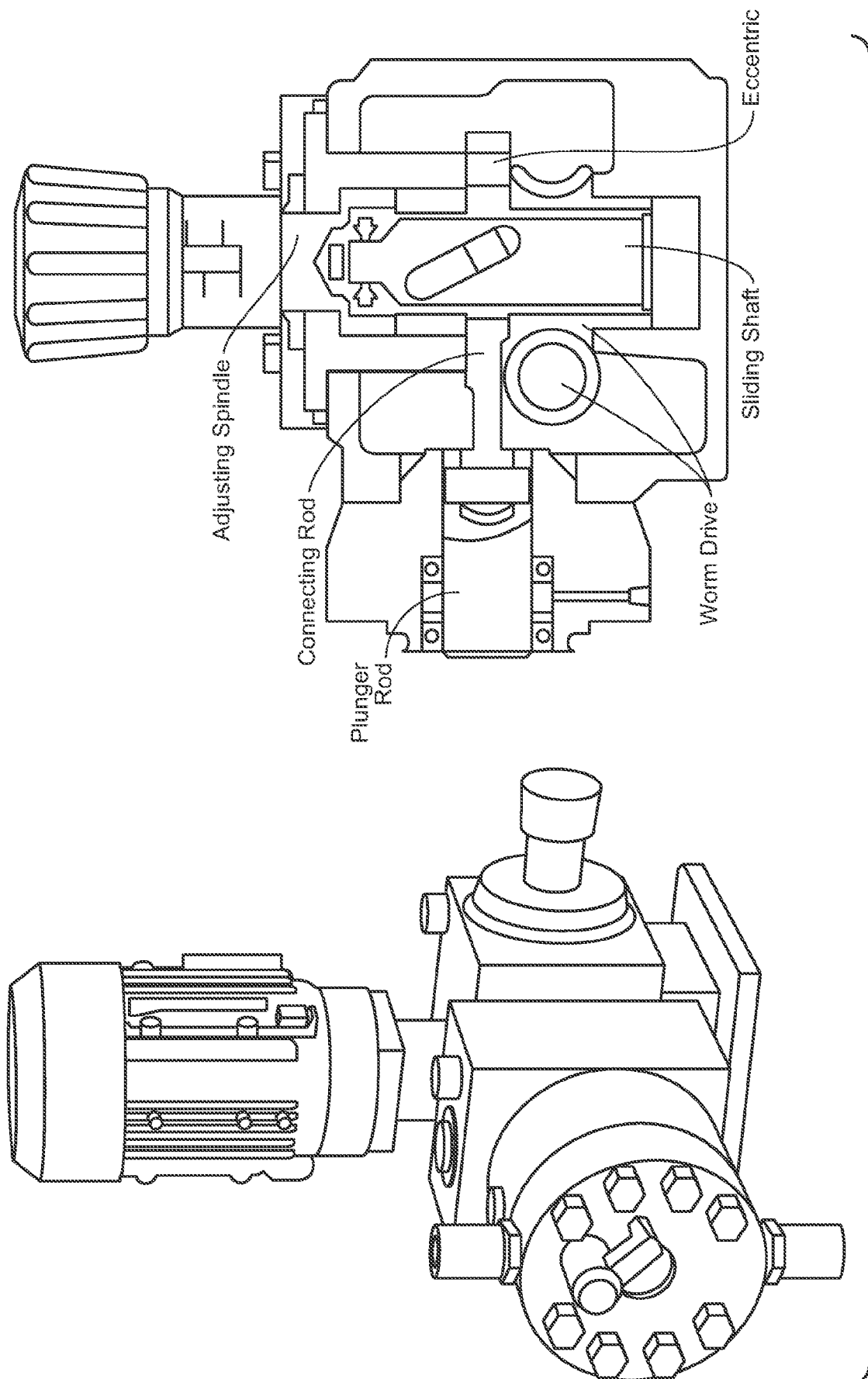


FIG. 10



12/21

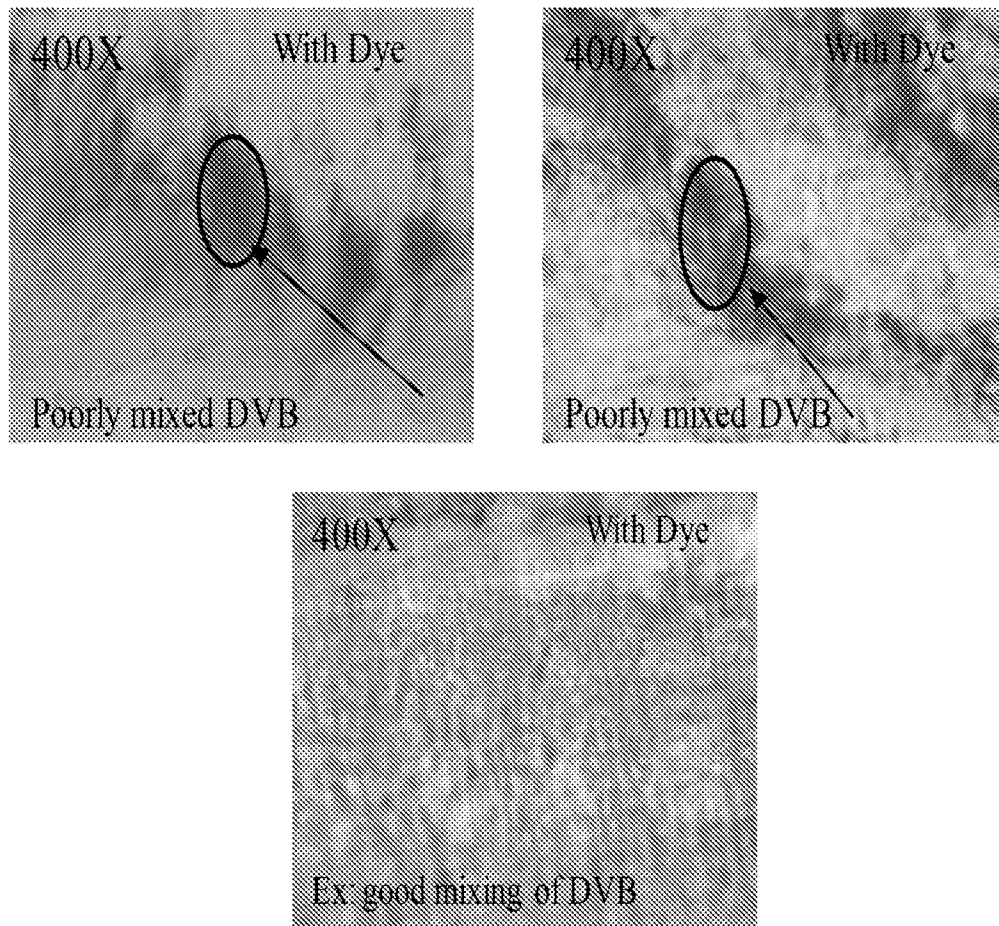


FIG. 12

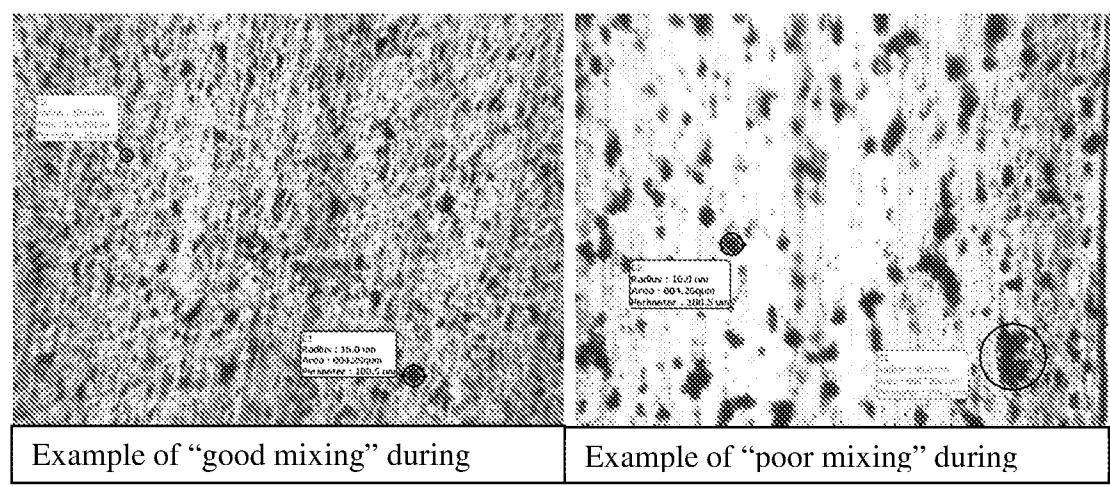


FIG. 13

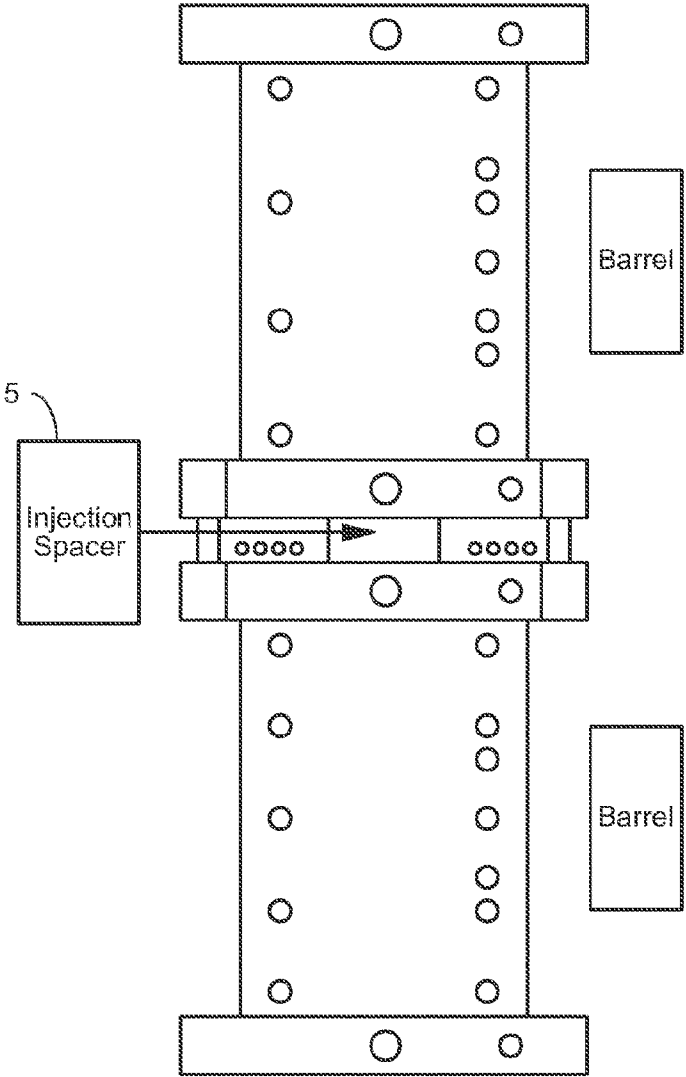


FIG. 14

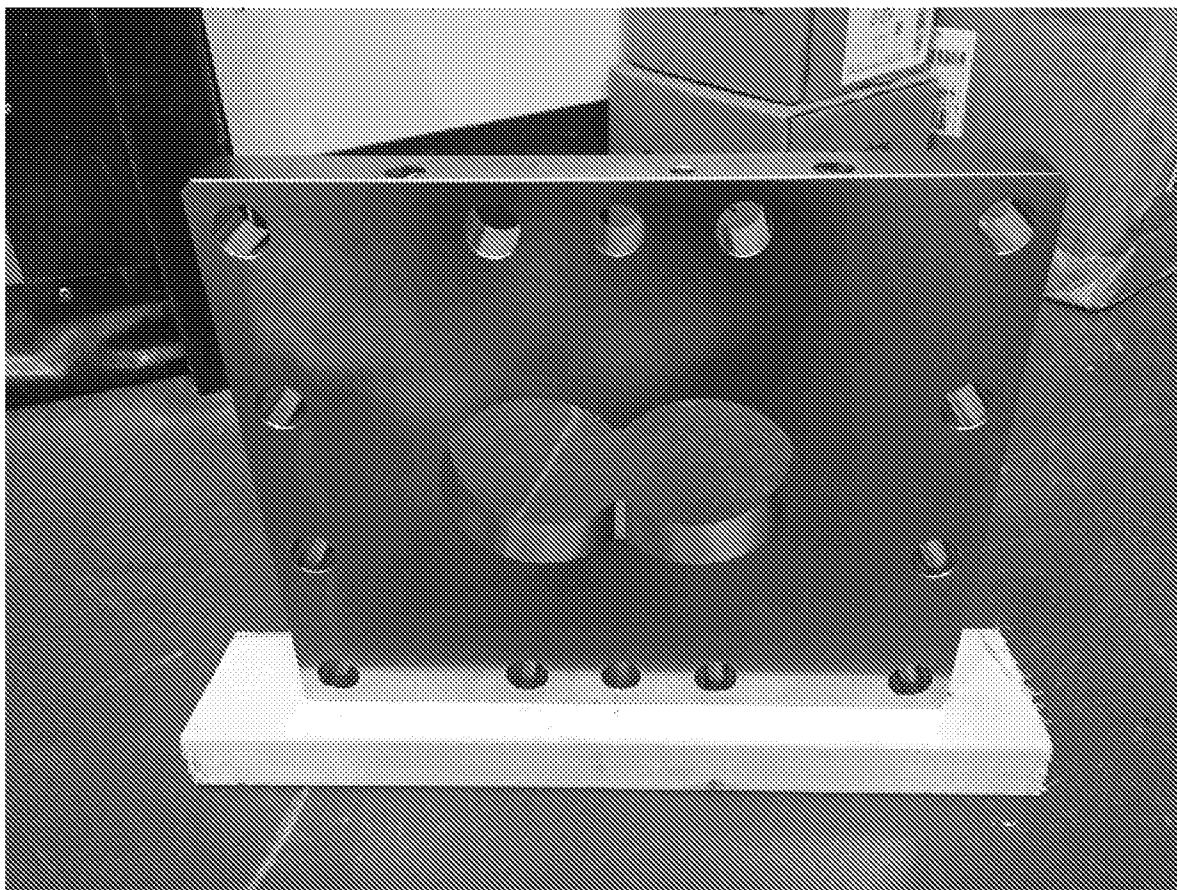
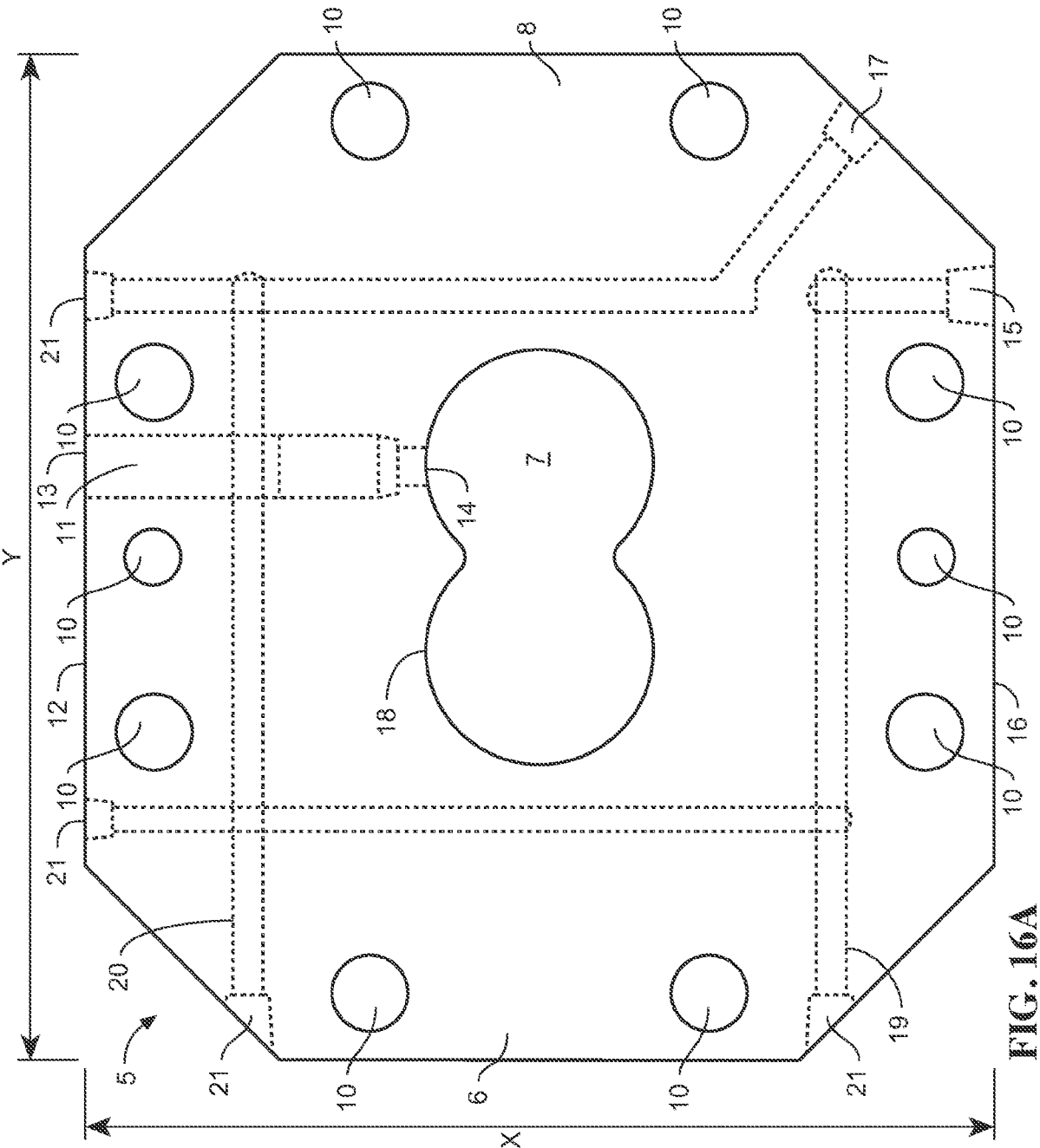
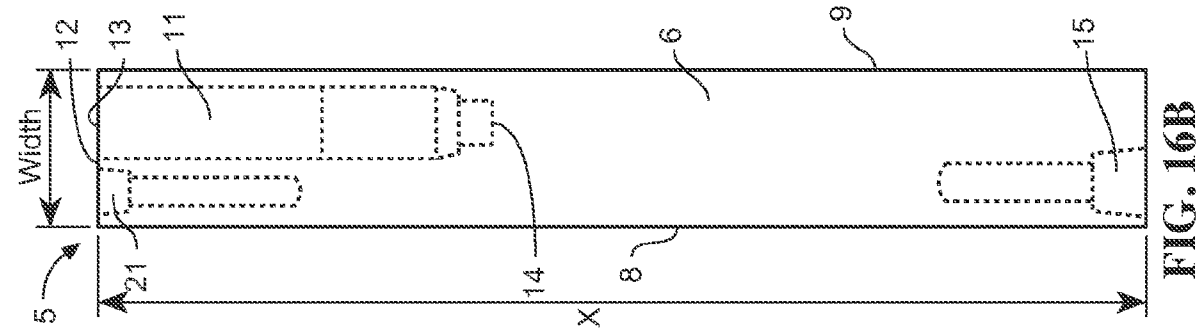


FIG. 15



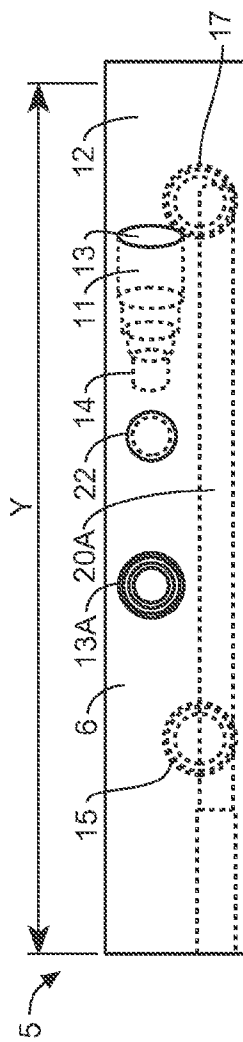


FIG. 17B

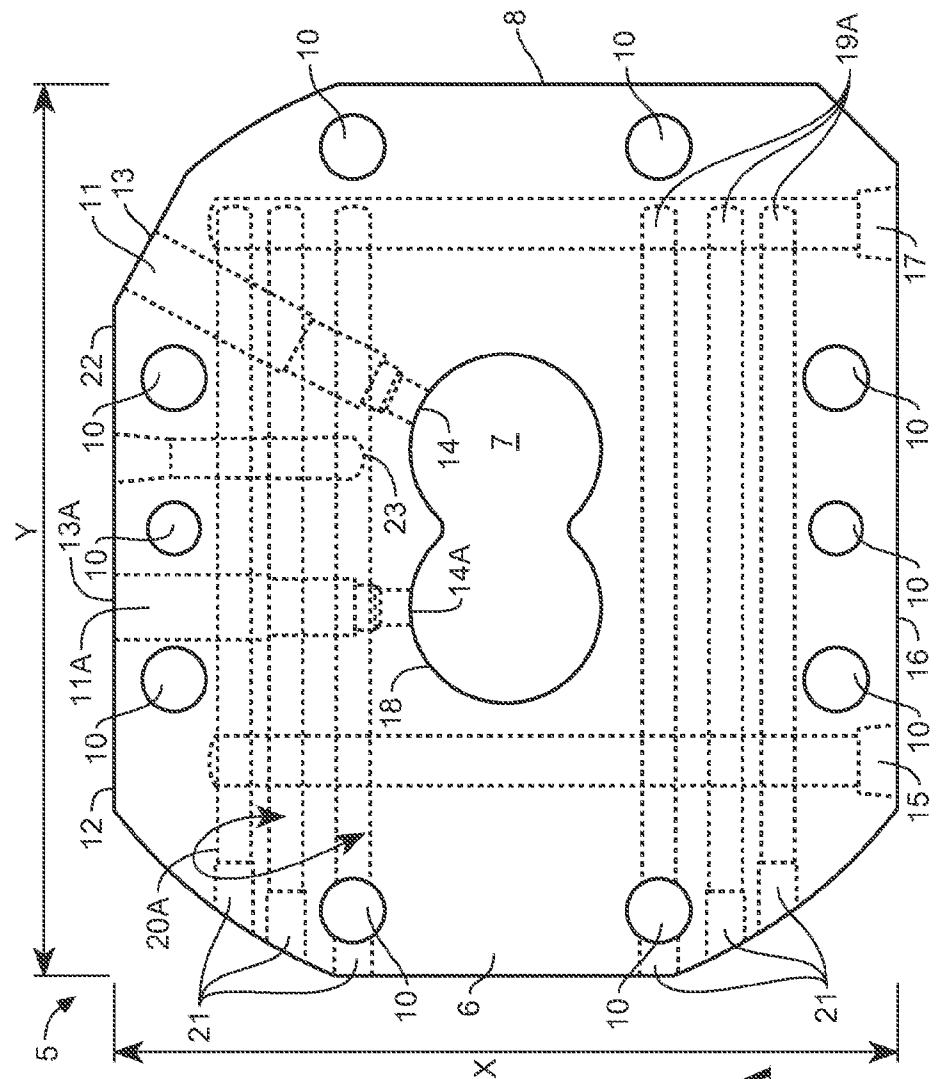


FIG. 17A

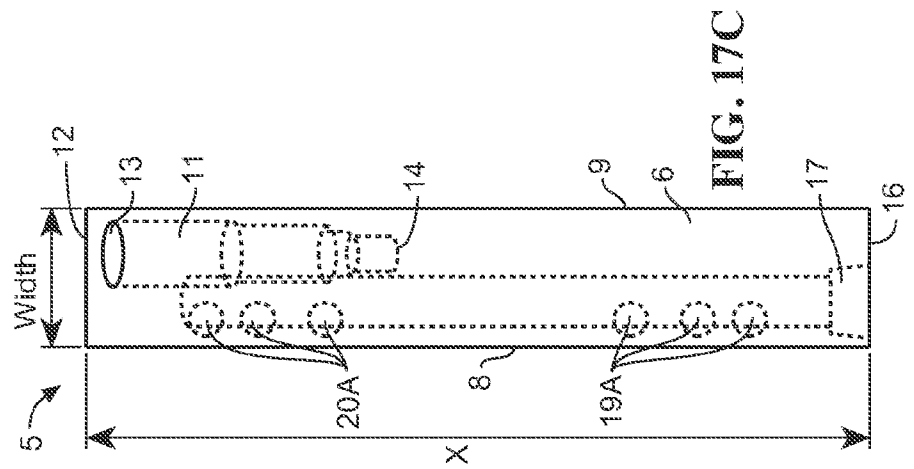


FIG. 17C

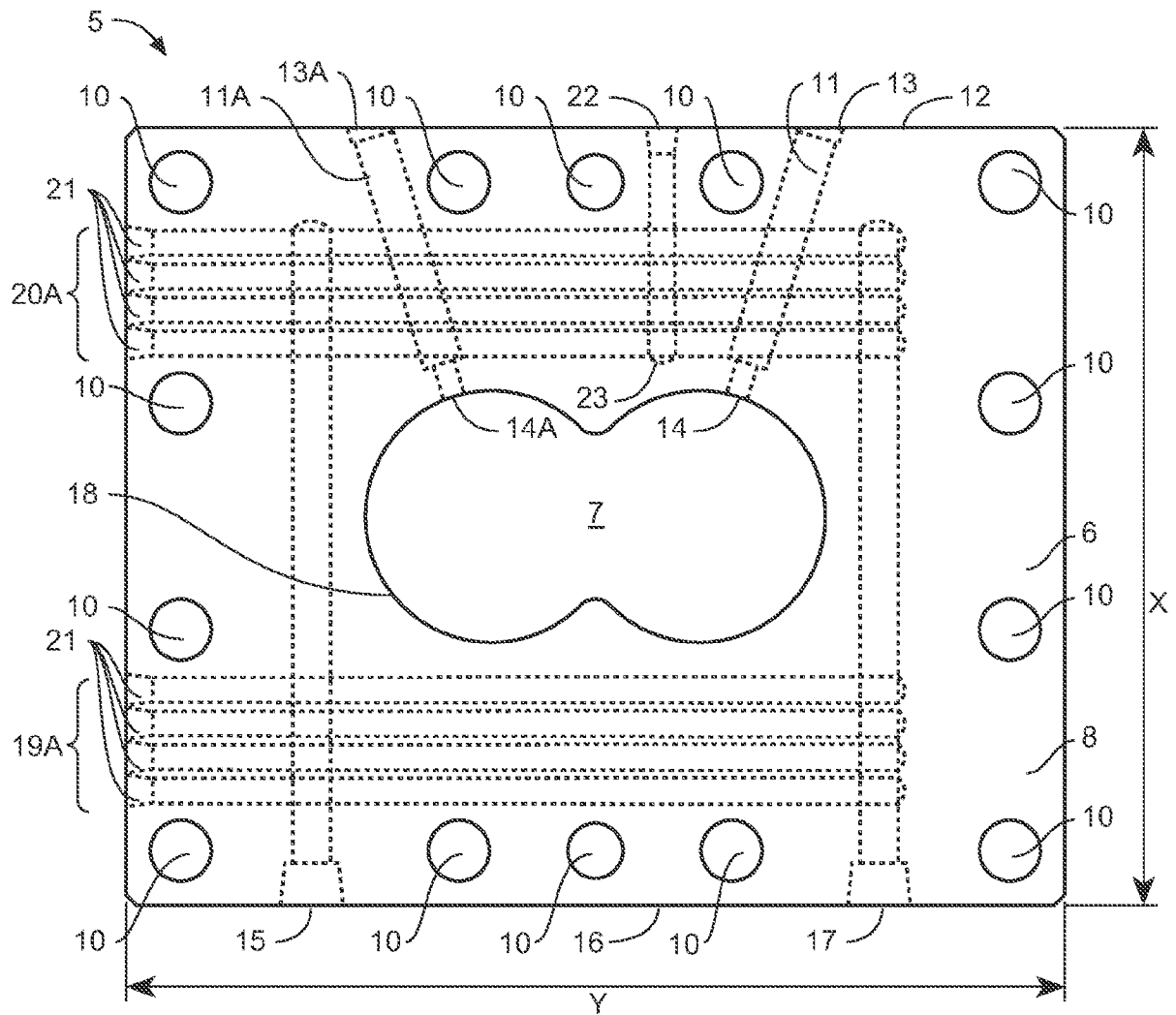


FIG. 18

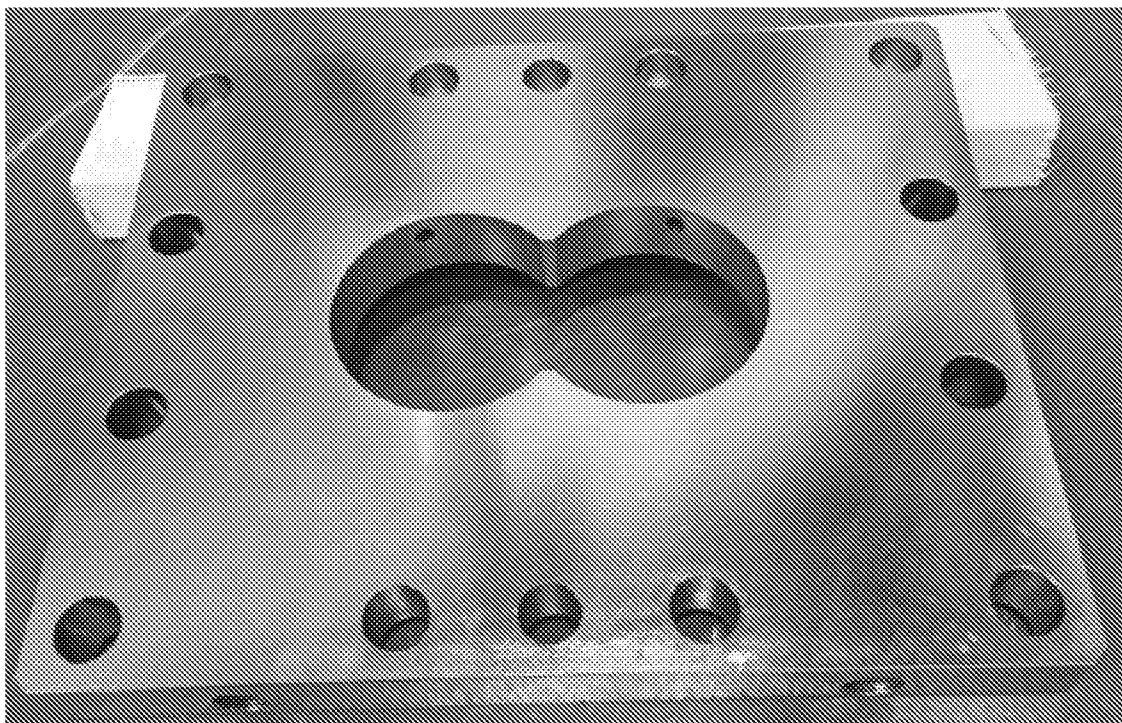


FIG. 19

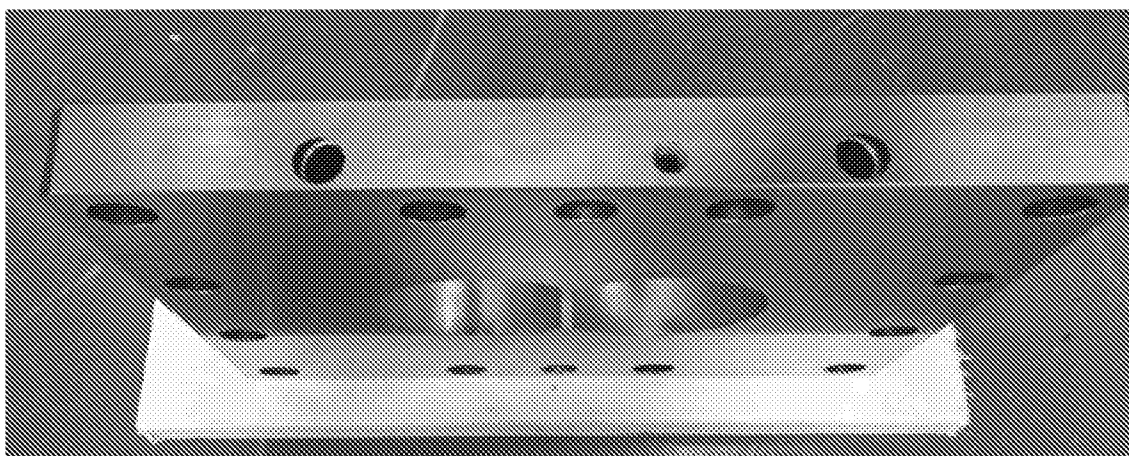


FIG. 20

21/21

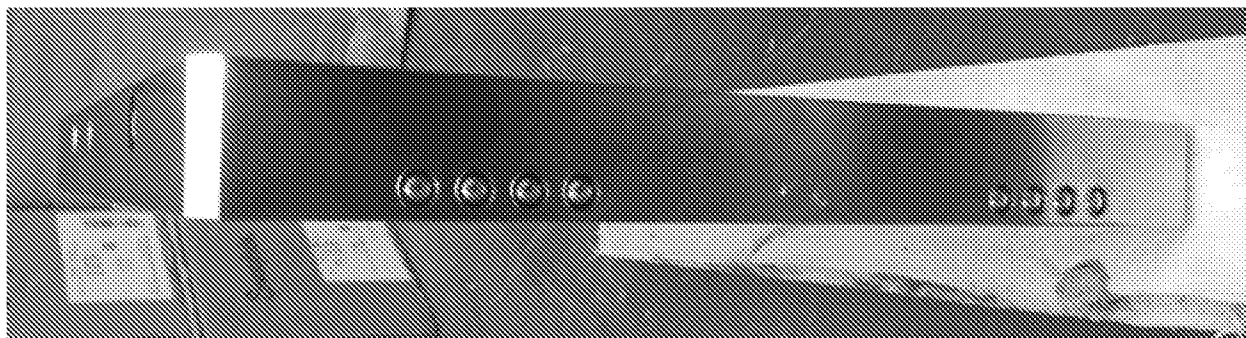


FIG. 21

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2020/025484

A. CLASSIFICATION OF SUBJECT MATTER INV. B29C44/50 B29C44/34 B29C35/16 B29C44/60 B29C48/00 B01J19/20 ADD. According to International Patent Classification (IPC) or to both national classification and IPC																							
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) B29C B29K B29B B01F B01J F28D F28F Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal, WPI Data																							
C. DOCUMENTS CONSIDERED TO BE RELEVANT <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="width: 10%;">Category*</th> <th style="width: 70%;">Citation of document, with indication, where appropriate, of the relevant passages</th> <th style="width: 20%;">Relevant to claim No.</th> </tr> </thead> <tbody> <tr> <td style="text-align: center; vertical-align: top;">X</td> <td>US 2015/183142 A1 (BALDWIN JESSE [US] ET AL) 2 July 2015 (2015-07-02) paragraphs [0001], [0045] - [0047], [0059], [0066], [0076], [0083], [0088] figures 4,8 -----</td> <td style="text-align: center; vertical-align: top;">1-45</td> </tr> <tr> <td style="text-align: center; vertical-align: top;">X</td> <td>US 5 639 159 A (SATO TAKUYA [JP]) 17 June 1997 (1997-06-17)</td> <td style="text-align: center; vertical-align: top;">1-16</td> </tr> <tr> <td style="text-align: center; vertical-align: top;">A</td> <td>the whole document -----</td> <td style="text-align: center; vertical-align: top;">17-45</td> </tr> <tr> <td style="text-align: center; vertical-align: top;">X</td> <td>DE 10 2009 034565 A1 (SMK METALL UND KUNSTSTOFFWAREN [AT]) 4 February 2010 (2010-02-04)</td> <td style="text-align: center; vertical-align: top;">1-16</td> </tr> <tr> <td style="text-align: center; vertical-align: top;">A</td> <td>the whole document -----</td> <td style="text-align: center; vertical-align: top;">17-45</td> </tr> <tr> <td style="text-align: center; vertical-align: top;">A</td> <td>US 2002/167102 A1 (YAMAGUCHI SATORU [JP] ET AL) 14 November 2002 (2002-11-14) the whole document -----</td> <td style="text-align: center; vertical-align: top;">1-45</td> </tr> </tbody> </table>			Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	X	US 2015/183142 A1 (BALDWIN JESSE [US] ET AL) 2 July 2015 (2015-07-02) paragraphs [0001], [0045] - [0047], [0059], [0066], [0076], [0083], [0088] figures 4,8 -----	1-45	X	US 5 639 159 A (SATO TAKUYA [JP]) 17 June 1997 (1997-06-17)	1-16	A	the whole document -----	17-45	X	DE 10 2009 034565 A1 (SMK METALL UND KUNSTSTOFFWAREN [AT]) 4 February 2010 (2010-02-04)	1-16	A	the whole document -----	17-45	A	US 2002/167102 A1 (YAMAGUCHI SATORU [JP] ET AL) 14 November 2002 (2002-11-14) the whole document -----	1-45
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X	US 2015/183142 A1 (BALDWIN JESSE [US] ET AL) 2 July 2015 (2015-07-02) paragraphs [0001], [0045] - [0047], [0059], [0066], [0076], [0083], [0088] figures 4,8 -----	1-45																					
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A	the whole document -----	17-45																					
A	US 2002/167102 A1 (YAMAGUCHI SATORU [JP] ET AL) 14 November 2002 (2002-11-14) the whole document -----	1-45																					
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Date of the actual completion of the international search 21 July 2020		Date of mailing of the international search report 28/07/2020																					
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Ullrich, Klaus																					

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2020/025484

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2015183142 A1	02-07-2015	CN 106163792 A	23-11-2016
		EP 3089867 A1	09-11-2016
		JP 6533526 B2	19-06-2019
		JP 2017503889 A	02-02-2017
		KR 20160105450 A	06-09-2016
		US 2015183142 A1	02-07-2015
		US 2018126617 A1	10-05-2018
		WO 2015103134 A1	09-07-2015

US 5639159 A	17-06-1997	BR 9604427 A	23-06-1998
		CA 2173767 A1	24-08-1997
		CN 1157774 A	27-08-1997
		EP 0791449 A1	27-08-1997
		JP 2781772 B2	30-07-1998
		JP H09225992 A	02-09-1997
		KR 970062020 A	12-09-1997
		TW 319735 B	11-11-1997
		US 5639159 A	17-06-1997

DE 102009034565 A1	04-02-2010	AT 11399 U1	15-10-2010
		DE 102009034565 A1	04-02-2010

US 2002167102 A1	14-11-2002	NONE	
