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METHODS TO HEAT SEAL FILMS

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the benefit to Serial No. 62/669,463, filed May 10, 2018,
5 the disclosure of which is hereby incorporated by reference in its entirety.

FIELD OF THE INVENTION

[0002] The present disclosure relates methods to heat seal films to produce heat sealed packaging.

BACKGROUND OF THE INVENTION

10 [0003] Films made from polyolefin polymers are widely used in packaging applications, such as pouches for dry food mixes, pet foods, snack foods, seeds, etc. In many film applications, it is desirable to seal the film during the packaging operation. This may be accomplished by the use of adhesives or by using heat sealing techniques. When heat sealing is used, it is important that the plastic film be readily heat sealable while also possessing other
15 good physical and mechanical properties such as resistance to tearing, high tensile strength, and good processability in high-speed equipment.

[0004] For example, the objective of a seal is to protect products inside the packaging against spoilage and leakage (e.g., air and moisture ingress) from its surrounding. Seal failure can be caused by many things including improper sealing conditions, defects in the film (e.g.,
20 holes), defects in the film-film alignment (e.g., a warp or twist that causes a fold or kink in one or both of the films), and contaminants within the seal. Seal failure can decrease the product shelf-life, cause packaging lines to be shut down for cleaning to remove contaminants, and lead to consumer complaints.

[0005] Quality control for seal integrity can be performed using a variety of methods from
25 visual inspection to a widely used water bath test described in ASTM D3078-02 (2013).

[0006] Briefly, the water bath test determines the presence or absence of large leaks. Generally, a sealed package is placed in a water tank filled with water with some headspace remaining. The tank is closed, thereby immersing the sealed package in the water at room temperature. The air pressure in the headspace is reduced. As the air is evacuated, the package
30 expands and puts pressure on the seals. The package is then visually inspected for signs of bubble emission from the seals, which indicates seal failure. If a package has emitted a bubble, the entire package is deemed to be not hermetic. However, if one does not see a bubble that does not necessarily mean the package is hermetic. The size of the leak that can be detected is dependent upon the film compositions and the test parameters selected. Typically, the leak

size has to be about 100 microns in diameter or larger for the water bath test to detect it. However, a seal with a leak as small as 2-20 microns can be considered in the industry to be a failed seal.

5 [0007] While the water bath test is easy and inexpensive, the results are not quantitative and can be highly subjective and dependent on the operator.

[0008] Another seal integrity test is a dye penetration test described in ASTM F1929-15. Briefly, the seal of a sealed package is maintained in contact with a liquid dye for a specified amount of time. Then, the seal is visually inspected for locations where the dye penetrated through the seal. Generally, the dye penetration test can identify leaks of about 50 microns or
10 larger. While the dye penetration test can detect the presence or absence of holes smaller than the water bath test, the dye penetration test is still not quantitative and is more time consuming and expensive than the water bath test.

[0009] Therefore, a need exists for improved methods for heat sealing films.

SUMMARY OF THE INVENTION

15 [0010] In a class of embodiments, the invention provides for a method comprising heat sealing a portion of a first sealing layer of a first film to a portion of a second sealing layer of a second film to produce a seal of a sealed packaging; increasing an internal pressure of the sealed packaging via an orifice extending through a portion of the sealed packaging; and measuring a pressure decay for the internal pressure of the sealed packaging, wherein the
20 pressure decay is a change in pressure (ΔP) over a measurement time (t_m).

[0011] In another class of embodiments, the invention provides for forming a plurality of sealed packagings each under different heat sealing parameters, wherein each sealed packaging is produced by: placing a wire between a portion of a first sealing layer of a first film and a portion of a second sealing layer of a second film; heat sealing the portion of the first sealing
25 layer to the portion of the second sealing layer to produce a seal of the sealed packaging; testing a hermeticity of the plurality of sealed packagings by: increasing an internal pressure of each sealed packaging via an orifice extending through a portion of each sealed packaging; and measuring a pressure decay for the internal pressure of the each sealed packaging, wherein the pressure decay is a change in pressure (ΔP) over a measurement time (t_m).

BRIEF DESCRIPTION OF THE DRAWINGS

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[0012] The following figures are included to illustrate certain aspects of the embodiments, and should not be viewed as exclusive embodiments. The subject matter disclosed is capable of considerable modifications, alterations, combinations, and equivalents in form and function, as will occur to those skilled in the art and having the benefit of this disclosure.

[0013] FIG. 1 is a cross-sectional illustration of an example heat sealable film that is a monolayer structure comprising a heat sealable layer.

[0014] FIG. 2 is a cross-sectional illustration of another example heat sealable film that is a multilayer structure comprising, in order from top to bottom of the illustration, a paper
5 substrate, a tie layer, an aluminum foil, and a heat sealable layer.

[0015] FIG. 3A is a top view illustration of an example of a packaging.

[0016] FIG. 3B is a cross-sectional illustration of the example of the packaging along 3B of FIG. 3A.

[0017] FIG. 3C is a cross-sectional illustration of the example of the packaging along 3C
10 of FIG. 3A.

[0018] FIG. 3D is a zoom view of a portion of FIG. 3C.

[0019] FIGS. 4A and 4B illustrate making a packaging by folding a single film.

[0020] FIG. 4C is a top view of the packaging illustrating the location of the seal.

[0021] FIG. 5 is an illustration of an apparatus suitable for performing the internal pressure
15 decay methods of the present disclosure.

[0022] FIG. 6A and 6B are photographs of inflated sealed bags in an improper, tilted position and a proper, straight position, respectively.

[0023] FIG. 7 illustrates corresponding plots for the internal pressure and internal pressure decay over time in an example internal pressure decay method of the present disclosure.

20 [0024] FIGS. 8A (top view) and 8B (cross-sectional view) illustrate placement of the wire before heat sealing a packaging.

[0025] FIGS. 8C (top view) and 8D (cross-sectional view) illustrate the wire, seal, and leaks the sides of the wire after heat sealing the packaging.

25 [0026] FIGS. 9A (no tint) and 9B (tint added) are light microscopy images of seals at the wire where leaks have been created.

[0027] FIG. 10 is a cross-sectional illustration of a wire between two films before heat sealing.

[0028] FIG. 11 is a plot of the measured internal pressure decay over time for three heat sealed packages.

30 [0029] FIG. 12 is a plot of the ΔP as a function of leak width.

[0030] FIG. 13 is a plot of the seal strength and ΔP ($t_m = 30$ minute) for the first packaging without a wire and the first packaging with a 100 micron copper wire in the seal as a function of sealing temperature.

[0031] FIG. 14 is a plot of the seal strength and ΔP ($t_m = 30$ minute) for the second packaging without a wire and the second packaging with a 100 micron copper wire in the seal as a function of sealing temperature.

DETAILED DESCRIPTION OF THE INVENTION

5 [0032] The present disclosure relates an internal pressure decay method for detecting leaks, preferably microleaks, in sealed packagings. The internal pressure decay method of the present disclosure can be used in many methods and processes including correlating leak width to internal pressure decay, determining sealing parameters that provide hermetically sealed packages and/or determining film compositions that provide hermetically sealed packages, and
10 performing quality control on packagings of sealed articles.

Definitions

[0033] The various descriptive elements and numerical ranges disclosed herein can be combined with other descriptive elements and numerical ranges to describe preferred embodiments of the compositions and processes relating to the internal pressure decay method
15 of the present disclosure; further, any upper numerical limit of an element can be combined with any lower numerical limit of the same element to describe preferred embodiments. In this regard, the phrase “within the range from X to Y” is intended to include within that range the “X” and “Y” values.

[0034] Unless otherwise noted, if an amount of a component is stated, that amount is
20 understood to be an aggregate amount if two or more different species of that component are present together.

[0035] As used herein, the term “copolymer” is meant to include polymers having two or more monomers. The term “polymer” as used herein includes, but is not limited to, homopolymers, copolymers, terpolymers, etc., and alloys and blends thereof. The term
25 “terpolymer” as used herein refers to a polymer synthesized from three different monomers. Terpolymers, in some embodiments, may be produced (1) by mixing all three monomers at the same time or (2) by sequential introduction of the different comonomers. The mixing of comonomers may be done in one, two, or possible three different reactors in series and/or in parallel. The term “polymer” as used herein also includes impact, block, graft, random, and
30 alternating copolymers. The term “polymer” shall further include all possible geometrical configurations unless otherwise specifically stated. Such configurations may include isotactic, syndiotactic and random (*i.e.*, atactic) symmetries.

[0036] The term “monomer” or “comonomer,” as used herein, can refer to the monomer used to form the polymer, *i.e.*, the unreacted chemical compound in the form prior to

polymerization, and can also refer to the monomer after it has been incorporated into the polymer, also referred to herein as a “[monomer]-derived unit”. Different monomers are discussed herein, including propylene monomers, ethylene monomers, and diene monomers.

[0037] The term “blend” or “polymer blend” as used herein refers to a mixture of two or more polymers. Blends may be produced by, for example, solution blending, melt mixing, or compounding in a shear mixer. Solution blending is common for making adhesive formulations comprising baled butyl rubber, tackifier, and oil. Then, the solution blend is coated on a fabric substrate, and the solvent evaporated to leave the adhesive.

[0038] As used herein, the term “linear” relative to a polymer means a polymer having no detectable branching (quantitatively or qualitatively), preferably a long-chain branching factor of 1.0 (+/-0.02).

[0039] As used herein, the term “ α -olefin copolymers” refers to a polymer having two or more monomer units selected from C₂, C₃, C₄, C₅, and C₆.

[0040] As used herein, the term “heat sealing” refers to the joining portions of two abutting layers as a result of the application of heat and/or pressure. The heat can be direct heat, heat generated from applying pressure, heat generated by an additive when exposed to pressure and/or electromagnetic radiation, ultrasonic sealing, or the like.

[0041] As used herein, the term “heat seal” refers to the joined portions of two layers as a result of the application of heat and/or pressure.

[0042] As used herein, the term “seal direction” refers to the longest dimension of the seal.

[0043] As used herein, the term “heat sealable layer” refers to a layer composed of a material that when exposed to heat and/or pressure forms a seal with an abutting material.

[0044] As used herein, the term “heat sealable film” refers to any film (single layer or multilayer) where at least one surface layer is a heat sealable layer.

[0045] As used herein, the term “sealed packaging” refers to a packaging that is sealed but not necessarily hermetically sealed. That is, the term “sealed packaging,” unless otherwise indicated, encompasses packagings that have been sealed and may be hermetic or may have one or more leak.

[0046] As used herein, the term “hermetic seal” refers to a seal having no leaks greater than about 5 microns in diameter.

[0047] As used herein, the term “microleak” refers to a leak having a leak width of about 5 microns to about 50 microns.

[0048] As used herein, the term “wire” is used generically to encompass long, solid objects that can be placed between sealing layers to create a leak in the seal. The term “wire”

encompasses strips, strands, threads, cables, and the like. The diameter of a wire refers to the dimension of the wire perpendicular to the seal direction, which if a strip is used as a wire would be the thickness of the strip.

Heat Sealable Films and Packaging

5 [0049] Heat sealable films can be any film (single layer or multilayer) where at least one surface layer is a heat sealable layer. Examples of materials that the heat sealable layer can comprise include, but are not limited to, low density polyethylene (LDPE), linear LDPE (e.g., metallocene linear LDPE), medium density polyethylene (MDPE), polypropylenes, polybutylenes, α -olefin copolymers (e.g., propylene-ethylene copolymers, ethylene-butylene-
10 propylene terpolymers, and propylene-butylene copolymers), ethylene vinyl acetate (EVA) copolymers, ethylene acrylic acid (EAA) copolymer, ethylene vinyl alcohol (EVOH) copolymers, ionomers, and blends thereof. Examples of materials that can be used in other layers of the heat sealable films include, but are not limited to, LDPE, linear LDPE, MDPE, polypropylenes, polybutylenes, α -olefin copolymers, EVA copolymers, EAA copolymers,
15 EVOH copolymers, ionomers, polyethylene terephthalate (PET), polyamides (PA), aluminum foil, and paper.

[0050] Examples of commercially available materials suitable for use in the films as the heat sealable layer and/or as another layer include, but are not limited to, AFFINITY™ GA (a polyolefin elastomer, available from Dow Chemical Co.), AFFINITY™ GP (a polyolefin
20 elastomer, available from Dow Chemical Co.), ENGAGE™ (a polyolefin elastomer, available from Dow Chemical Co.), DOWLEX™ (a polyethylene resin, available from Dow Chemical Co.), DOW™ LDPE (low density polyethylene, available from Dow Chemical Co.) (e.g., DOW™ LDPE 722), ELITE™ (a high α -olefin polyethylene resin, available from Dow Chemical Co.) (e.g., ELITE™ 5815, a metallocene LLDPE), EVOLUE™ (metallocene
25 LLDPE, available from Mitsui Chemicals, Inc.), EXCEED™ (a polyethylene or polyethylene copolymer resin, available from ExxonMobil Chemical Company) (e.g., EXCEED™ 0019XC, ethylene 1-hexene copolymers), EXCEED™ XP (a polyethylene or polyethylene copolymer resin, available from ExxonMobil Chemical Company), ENABLE™ (a polyethylene or polyethylene copolymer resin, available from ExxonMobil Chemical Company), and blends
30 thereof.

[0051] Heat sealable films include, but are not limited to, monolayer films, multilayer polyethylene films, paper-board coating, PET-containing laminates (e.g., PE-PET films), PA-containing laminates (e.g., PE-PA films), aluminum-polyethylene laminates, and paper-aluminum-polyethylene laminates. For example, FIG. 1 is a cross-sectional illustration of an

example heat sealable film 100 that is a monolayer structure comprising a heat sealable layer 102. In another example, FIG. 2 is a cross-sectional illustration of an example heat sealable film 200 that is a multilayer structure comprising, in order from top to bottom of the illustration, a paper substrate 204, a tie layer 206, an aluminum foil 208, and a heat sealable layer 202.

5 [0052] FIG. 3A is a top view illustration of an example of a packaging 310. FIG. 3B is a cross-sectional illustration of the example of the packaging 310 along 3B of FIG. 3A. FIG. 3C is a cross-sectional illustration of the example of the packaging 310 along 3C of FIG. 3A. FIG. 3D is a zoom view of a portion of FIG. 3C.

[0053] The packaging 310 illustrated in FIG. 3A-3D is composed of two films (a first film 10 312 and a second film 314) sealed together at heat seal 316, which is near the edges where the two films 312, 314 overlap. As shown in FIG. 3D, the first film 312 is a monolayer film composed of a first heat sealing layer 318, and the second film 314 is a multilayer film composed of three layers including a second heat sealing layer 320, which is a surface layer of the second film 314. The seal 316 is a heat seal formed between a portion of the first sealing 15 layer 318 of the first film 312 and a portion of the second sealing layer 320 of the second film 314.

[0054] The seal 316, the first film 312, and the second film 314 define an internal portion 322 of the packaging 310. As used herein, for a sealed packaging, the films that define the internal portion can also be referred to as walls.

20 [0055] Further, the internal pressure decay methods described further herein refer to a central portion 324 of a film or wall and is, therefore, illustrated in FIG. 3A. The central portion 324 of a film is within about 25% of the center of the film relative to the seal and in the plane of the film.

[0056] FIGS. 4A-4B illustrates making a packaging 410 by folding 426 a single film 428. 25 FIG. 4C is a top view of the packaging 410 illustrating the location of the seal 416.

[0057] FIG. 4A is a cross-sectional view of the single film 428 with an arrow indicating folding 426. Once folded, the film 428 acts as two films (a first film 412 and a second film 414 that need to be sealed on only three sides as illustrated in the top view of FIG. 4C.

[0058] For reference, the central portion 424 of a film is illustrated in FIG. 4C, which for 30 packagings like bags is where a needle punctures the wall during the internal pressure decay methods of the present disclosure.

[0059] As described in the methods herein, the internal pressure decay test of the present disclosure can be used to determine and/or adjust compositions of the films and/or sealing layers of the films.

Sealing Parameters

[0060] Heat sealing abutting layers to form seals and, consequently, packagings can be performed using a variety of parameters that depend on the compositions of the abutting layers. Examples of sealing parameters include, but not limited to, sealing temperature, sealing time,
5 and sealing pressure.

[0061] For example, the sealing temperature can be from about 90°C to about 190°C, about 95°C to about 135°C, about 90°C to about 110°C, about 100°C to about 120°C, about 110°C to about 130°C, about 120°C to about 140°C, about 130°C to about 150°C, about 140°C to about 160°C, about 155°C to about 175°C, about 160°C to about 180°C, or about 175°C to
10 about 190°C.

[0062] For example, the sealing time can be from about 0.2 seconds to about 5 second, about 0.2 seconds to about 3 seconds, about 0.2 seconds to about 1 second, about 0.5 seconds to about 3 seconds, about 0.5 seconds to about 1 second, or about 0.2 seconds to about 0.5 seconds.

15 [0063] For example, the sealing pressure can be about 100 kPa to about 500 kPa, about 100 kPa to about 200 kPa, about 200 kPa to about 300 kPa, about 300 kPa to about 400 kPa, about 400 kPa to about 500 kPa, about 250 kPa to about 375 kPa.

[0064] As described in the methods herein, the internal pressure decay test of the present disclosure can be used to determine and/or adjust heat sealing parameters. Such methods may
20 be alone or in combination with determining and/or adjusting compositions of the films and/or sealing layers of the films.

Internal pressure decay method

[0065] Generally, internal pressure decay methods of the present disclosure involve puncturing a wall of a sealed packaging with a needle, increasing the internal pressure of the
25 sealed packaging (i.e., inflating the sealed packaging), stabilizing the internal pressure of the sealed packaging, and measuring a change pressure (ΔP) over time (t).

[0066] More specifically, for example, FIG. 5 is an illustration of an example apparatus 530 suitable for performing the internal pressure decay methods of the present disclosure. The apparatus 530 includes a needle 532 mounted in a support 534 and connected to a gas supply
30 536. The sealed packaging 510 is placed under the needle 532. A septum 538 is placed between the needle 532 and the sealed packaging 510. Optionally, the septum 538 can be adhered to the sealed packaging 510 with an adhesive. The septum 538 mitigates leaks at the puncture site. The needle is preferably sharp, short, and has a small inner diameter to mitigate forming a leak at the injection site. The septum can comprise a material like polyethylene,

polypropylene, silicone, and the like. The septum, for example, can have a thickness of about 1 mm to about 5 mm, preferably about 3 mm.

[0067] The needle is carefully inserted through a central portion of the wall (e.g., as illustrated in FIGS. 3A and 4C) of a sealed packaging. An object can be included in the sealed packaging to ensure the needle punctures only one wall and does not damage the opposing wall. For example, a piece of cardboard, plastic, or other material may be used.

[0068] After puncturing the sealed packaging 510, gas flows (arrows 540) from the gas supply 536 into the sealed packaging 510 to inflate the sealed packaging 510. Any gas that is nonreactive with the packaging 510 and components of the apparatus 530 can be used. Examples of suitable gases can include, but are not limited to, air, nitrogen, argon, carbon dioxide, helium, and combinations thereof.

[0069] The inflation rate of the sealed packaging 510 depends on the seal strength and film compositions. The inflation rate should not be too fast to stress the seals before the pressure decay measurements can occur. The inflation rate will depend on the packaging size, seal dimensions, and film materials.

[0070] After inflation, the sealed packaging 510 should be in solid, straight contact with the workspace surface 542. For example, FIG. 6A and 6B are photographs of inflated sealed bags in an improper, tilted position and a proper, straight position, respectively. When the sealed packaging is tilted, the tilt of the packaging applies pressure to the needle at the puncture site and can create a leak that alters the results of the test.

[0071] Referring again to FIG. 5, after inflation, the sealed packaging 510, the septum 538, and a lower portion 544 of the support 534 should be in good contact so that the septum 538 forms a seal with the sealed packaging 510 and with the lower portion 544 of the support 534. However, the support 534 should be positioned at a proper height so that too much pressure is not applied between the three components where tension is applied to the bag.

[0072] After inflation, the internal pressure of the sealed packaging 510 is maintained for a period of time, referred to herein as the stabilizing time. For more accurate measurements, stabilizing times of 1 minute or greater are preferred. While shorter or longer stabilizing times can be used, the stabilizing time can, for example, be about 1 minute to about 10 minutes, about 1.5 minute to about 5 minutes, or about 2 minute to about 4 minutes.

[0073] The pressure to which the sealed packaging 510 is inflated depends on the seal strength and the composition of the films. As used herein, this pressure is referred to as an initial pressure (P_0). In FIG. 5 the pressure sensor is illustrated at 546.

[0074] P_0 should be sufficiently high to force gas out of a leak but sufficiently low, to not create or expand leaks in the sealed packaging and to not burst the sealed packaging. While lower or higher pressures can be used, the initial pressure can, for example, be about 10 kPa to about 100 kPa, about 20 kPa to about 40 kPa, about 30 kPa to about 50 kPa, about 40 kPa to about 60 kPa, about 50 kPa to about 70 kPa, about 60 kPa to about 80 kPa, or about 70 kPa to about 90 kPa.

[0075] After the settling time, the gas supply 536 is sealed off (e.g., with valve 548) upstream of the pressure sensor 546. The pressure sensor 546 then measures the internal pressure over time ($\Delta P/t$) (or pressure decay) of the sealed packaging 510.

10 [0076] The lower limit of the leak width that can be detected with the internal pressure decay methods described herein depends on the sensitivity of the pressure sensor being used. The internal pressure decay methods described herein can be used to detect leaks of about 5 microns or larger (e.g., about 5 microns to about 250 microns, about 10 microns to about 50 microns, about 25 microns to about 100 microns, about 50 microns to about 150 microns, or about 100 microns to about 250 microns) including, advantageously, microleaks.

[0077] FIG. 7 illustrates corresponding plots for the internal pressure over time (top plot) and internal pressure decay over time (bottom plot) in an example internal pressure decay method of the present disclosure. As described above, the sealed packaging is inflated during an inflation time 750 to a desired initial pressure (P_0) and maintained at P_0 for the stabilizing time 752. Then, the gas supply is closed, and the pressure within the internal portion of the sealed packaging is monitored from the initial time (t_0) to a second time (t_1), where the pressure at t_1 is referred to herein as P_1 . The time from t_0 to t_1 is also referred to herein as the measurement time (t_m). The pressure decay is $\Delta P/t_m$, where $\Delta P = |P_1 - P_0|$ and $t_m = t_1 - t_0$.

25 [0078] While any length of time can be used for t_m , preferably the time is long enough to see the pressure change and short enough to not be unnecessarily time consuming. The measurement time can, for example, be about 1 minute to about 1 hour, about 5 minutes to about 45 minutes, or about 10 minutes to about 30 minutes.

[0079] The t_m is highly dependent on P_0 . For higher P_0 , the pressure will reduce more quickly and a shorter t_m can be used with accurate results. However, as discussed above, P_0 should not be too high to create or expand a leak. Further, a longer t_m provides more opportunity to create or expand a leak. Therefore, a balance between P_0 and t_m should be reached.

[0080] A suitable P_0 and a suitable t_m that provides quick and accurate results can be determined experimentally by comparing the pressure decay for a sealed packaging and a sealed packaging with a leak.

[0081] The leak can be introduced by including a defect and/or a contaminant in the seal
5 when forming the packaging. For example, creating the leak in the seal can be achieved by placing a wire between the two heat sealable films before creating the heat seal in a location such that after sealing the wire extends from inside the packaging to outside the packaging between the two films. FIGS. 8A (top view) and 8B (cross-sectional view) illustrate placement of the wire 854 before heat sealing 856 a packaging 810. FIGS. 8C (top view) and 8D (cross-
10 sectional view) illustrate the wire 854, seal 816, and leaks 858 the sides 860 of the wire after heat sealing the packaging 810. The sealing layers create seals 816 at the top 862 and bottom 864 of the wire.

[0082] In the present methods, the size of the leak is defined as the leak width according to EQ. 1.

15

$$\text{leak width} = (\text{border distance} - \text{wire diameter})/2 \quad \text{EQ. 1.}$$

[0083] The border distance is the distance between the edges of the seals on either side of the wire. The border distances can be measured by light microscopy looking through the seal
20 (e.g., a top view). Looking through the seal in this manner is advantageously non-destructive and does not affect the leak size, where a side view that requires cutting is destructive and potentially alters the dimension of the leak. The wire diameter should be known. Optionally, a tint can be used to better distinguish the seal borders.

[0084] FIGS. 9A (no tint) and 9B (tint added) are light microscopy images of seals at the
25 wire where leaks have been created. Overlaid on each are several border distance measurements.

[0085] Both FIGS. 9A and 9B are the same magnification and use a 100 micron wire. Because the tint coats the wire, the wire in FIG. 9B appears larger, which is why the wire diameter in EQ. 1 is based on the known wire diameter and not a wire diameter measured by
30 light microscopy.

[0086] Any wire can be used to form the leaks. Preferably, the material thereof should be nonreactive with the sealing layer. Further, the material should seal well to the sealing layer of the film so that the leak is formed at the sides of the wire and not the top and bottom of the wire, as illustrated in FIG. 8D, which will make measuring the leak width more accurate.

Examples of materials the wire can comprise include, but are not limited to, copper, nickel, iron, aluminum, lead, gold, platinum, palladium, stainless steel, brass, fluorocarbon polymers, and any combination thereof. Nylon and cotton wires have been tried, but the results were inconclusive, which is believed to be due to the water absorption properties of such wires. Such materials may be suitable if coated with a suitable material to mitigate the water absorption properties.

[0087] The wire can have a diameter of about 50 microns to about 200 microns, about 75 microns to about 150 microns, or about 50 microns to about 100 microns. The diameter of the wire can be chosen based on the thickness of the films. FIG. 10 is a cross-sectional illustration of a wire 1054 between two films 1012, 1014. The wire has a radius 1070. The first film 1012 has a thickness 1072, and the second film 1014 has a thickness 1074. The diameter of the wire can be chosen based on a ratio of the wire radius to the thinnest of the two film thicknesses 1072, 1074 (which is thickness 1072 in FIG. 10) as illustrated in EQ. 2.

$$k = (h_{wire}) / (h_{thinnest\ film}) \quad \text{EQ. 2,}$$

where h_{wire} is the radius of the wire and $h_{thinnest\ film}$ is the thickness of the thinnest film.

[0088] k can be larger than, smaller than, or equal to 1. Preferably, k is preferably less than or equal to about 1 (e.g., about 0.02 to about 10, preferably about 0.05 to about 5, and more preferably about 0.1 to about 3). k is useful when comparing films of different thicknesses and/or wires of different thicknesses because EQ. 2 allows for a method of normalizing the values for comparison.

[0089] When experimentally determining a suitable P_0 and a suitable t_m , the ΔP in a sealed packaging can be compared to the ΔP in a sealed packaging with a wire (i.e., having a leak) each over the same measurement time using EQ. 3.

$$r = (\Delta P_{wire}) / (\Delta P_{no\ wire}) \quad \text{EQ. 3.}$$

[0090] Differentiation between hermetically and not hermetically sealed packaging is more accurate with higher r values. Preferably, r has a value of about 2 or greater, for example, about 2 to about 4 or about 2 to about 3.

[0091] The leak width for the sealed packaging with a wire is preferably about 250 microns or less, for example, about 5 microns to about 250 microns, about 5 microns to about 100

microns, about 5 microns to about 50 microns, about 10 to about 50 microns, about 15 microns to about 30 microns, or about 5 to about 25 microns.

Correlating leak width to internal pressure decay

[0092] The pressure decay can be correlated to the size of the leak. Such a method involves creating a leak in a seal (e.g., with a wire as described above), measuring the width of the leak, repeating several times, and correlating the leak width with the pressure decay. The diameter of the wire and/or the sealing parameters can be varied to create leaks of different sizes. Further, because of sample and experimental variability, using the same diameter of the wire and the same sealing parameters can result in different sized leaks.

[0093] The correlation between leak width and pressure decay can be an equation, a graph, a table, or the like. The correlation can optionally also indicate the hermeticity of the packaging, that is, if the pressure decay is small enough for the packaging to be considered hermetic, not hermetic, and, optionally, likely hermetic. For example, the hermeticity for a specific packaging may be that a pressure decay of 0.35 kPa/30 minutes or less is hermetic, a pressure decay of 0.55 kPa/30 minutes or greater is hermetic, and a pressure decay of between 0.35 kPa/30 minutes and 0.55 kPa/30 minutes is likely hermetic.

Sealing parameter determination methods

[0094] The internal pressure decay method can be used to determine proper sealing parameters for different films. For example, methods can include producing a plurality of sealed packagings with a first set of sealing parameters, puncturing a wall of the sealed packagings with a needle, increasing the internal pressure of the sealed packagings, stabilizing the internal pressure of the sealed packagings, measuring the pressure decay in the sealed packagings, and optionally identifying the hermeticity of sealed packagings. This can be repeated for one or more additional sets of sealing parameters. The sealing parameters (or a variation thereof) that produce the most hermetically sealed packagings can be used to produce packagings containing articles.

Film composition determination methods

[0095] The internal pressure decay method can be used to determine proper film compositions for different sealing parameters. For example, methods can include producing a plurality of sealed packagings with a set of sealing parameters wherein the sealed packagings are composed of a first film and a second film, puncturing a wall of the sealed packagings with a needle, increasing the internal pressure of the sealed packagings, stabilizing the internal pressure of the sealed packagings, measuring the pressure decay in the sealed packagings, and optionally identifying the hermeticity of sealed packagings. This can be repeated for one or

more additional film compositions. The film compositions (or a variation thereof) that produce the most hermetically sealed packagings can be used to produce packagings containing articles.

Quality control methods

[0096] The internal pressure decay method can be used for quality control of packagings during production. For example, methods can include selecting a sealed packaging (e.g., from a packaging line), puncturing a wall of the sealed packaging with a needle, increasing the internal pressure of the sealed packaging, stabilizing the internal pressure of the sealed packaging, measuring the pressure decay in the sealed packaging, and optionally identifying the hermeticity of sealed packaging. This can be repeated for several packagings. If the pressure decay and/or hermeticity are unacceptable for several packagings, the sealing parameters can be changed to provide hermetically sealed packagings.

[0097] Optionally, a correlation between leak width and pressure decay (either known or measured) can be used to estimate the leak width, which may aid in identifying possible contamination sources.

[0098] For quality control methods, the packaging typically contains materials that are oxygen-sensitive and/or water-sensitive. Examples of such materials include, but are not limited to, food, beverages, medications, medical devices, electronics, and adhesives.

[0099] Unless otherwise indicated, all numbers expressing quantities of ingredients, properties such as molecular weight, reaction conditions, and so forth used in the present specification and associated claims are to be understood as being modified in all instances by the term “about.” Accordingly, unless indicated to the contrary, the numerical parameters set forth in the following specification and attached claims are approximations that may vary depending upon the desired properties sought to be obtained by the embodiments of the present invention. At the very least, and not as an attempt to limit the application of the doctrine of equivalents to the scope of the claim, each numerical parameter should at least be construed in light of the number of reported significant digits and by applying ordinary rounding techniques.

[00100] One or more illustrative embodiments incorporating the invention embodiments disclosed herein are presented herein. Not all features of a physical implementation are described or shown in this application for the sake of clarity. It is understood that in the development of a physical embodiment incorporating the embodiments of the present invention, numerous implementation-specific decisions must be made to achieve the developer's goals, such as compliance with system-related, business-related, government-related and other constraints, which vary by implementation and from time to time. While a

developer's efforts might be time-consuming, such efforts would be, nevertheless, a routine undertaking for those of ordinary skill in the art and having benefit of this disclosure.

[00101] While compositions and methods are described herein in terms of “comprising” various components or steps, the compositions and methods can also “consist essentially of”
5 or “consist of” the various components and steps.

[00102] To facilitate a better understanding of the embodiments of the present invention, the following examples of preferred or representative embodiments are given. In no way should the following examples be read to limit, or to define, the scope of the invention.

EXAMPLES

10 [00103] Example 1 – Three heat sealed packages were produced: (1) control, (2) 100 micron copper wire in the seal, and (3) 200 micron copper wire in the seal.

[00104] The internal pressure decay method described herein was used with a stabilizing time of 20 minutes and a P_0 of 20 Pa. The internal pressure decay was measured over 90 minutes. FIG. 11 is a plot of the measured internal pressure decay over time for the three
15 samples.

[00105] The water bath test was also performed on the packages where the control packaging produced no bubble, the 100 micron copper wire in the seal packaging produced about four bubbles, and the 200 micron copper wire in the seal packaging produced a very large number of bubbles.

20 [00106] Example 2 – Several heat sealed packages were produced with a 100 micron copper wire in the seal using a seal time of 0.4 seconds and varying the seal temperature from about 105°C to about 140°C. The internal pressure decay method described herein was used with a stabilizing time of 1 minute and a P_0 of 20 kPa. The internal pressure decay was measured over 30 minutes. The leak widths were determined using light microscopy and EQ. 1. FIG. 12
25 is a plot of the ΔP as a function of leak width. The plot also includes (1) an approximate scatter range that accounts for influences like machine and sensor inaccuracy, external disturbances, and other factors that impact the leak test and (2) areas where the hermeticity of the packaging is considered hermetic, likely hermetic, and not hermetic.

[00107] Example 3 – The internal pressure decay methods was used to compare sealing
30 temperature. The first packaging is formed from two three-layer films comprising a first layer comprising polyethylene, a middle layer comprising polyethylene, and a sealing layer comprising EXCEED™. The second packaging is formed from two three-layer films comprising a first layer comprising polyethylene, a middle layer comprising polyethylene, and a sealing layer comprising ELITE™.

[00108] FIG. 13 is a plot of the seal strength and ΔP ($t_m = 30$ minute) for the first packaging without a wire and the first packaging with a 100 micron copper wire in the seal as a function of sealing temperature. FIG. 14 is a plot of the seal strength and ΔP ($t_m = 30$ minute) for the second packaging without a wire and the second packaging with a 100 micron copper wire in the seal as a function of sealing temperature.

[00109] For both samples, when a contaminant (the wire) is introduced, the temperature required to achieve a hermetic seal increases. Further, while the seal has reached a maximum strength (i.e., where the seal force plateaus), higher temperatures are required to achieve hermeticity with a contaminant, especially in the second packaging. As packaging in manufacturing situations is typically only based on achieving the strongest seal, this example illustrates that hermeticity should also be considered before determining sealing parameters.

[00110] Therefore, the present invention is well adapted to attain the ends and advantages mentioned as well as those that are inherent therein. The particular embodiments disclosed above are illustrative only, as the present invention may be modified and practiced in different but equivalent manners apparent to those skilled in the art having the benefit of the teachings herein. Furthermore, no limitations are intended to the details of construction or design herein shown, other than as described in the claims below. It is therefore evident that the particular illustrative embodiments disclosed above may be altered, combined, or modified and all such variations are considered within the scope and spirit of the present invention. The invention illustratively disclosed herein suitably may be practiced in the absence of any element that is not specifically disclosed herein and/or any optional element disclosed herein. While compositions and methods are described in terms of “comprising,” “containing,” or “including” various components or steps, the compositions and methods can also “consist essentially of” or “consist of” the various components and steps. All numbers and ranges disclosed above may vary by some amount. Whenever a numerical range with a lower limit and an upper limit is disclosed, any number and any included range falling within the range is specifically disclosed. In particular, every range of values (of the form, “from about a to about b,” or, equivalently, “from approximately a to b,” or, equivalently, “from approximately a-b”) disclosed herein is to be understood to set forth every number and range encompassed within the broader range of values. Also, the terms in the claims have their plain, ordinary meaning unless otherwise explicitly and clearly defined by the patentee. Moreover, the indefinite articles “a” or “an,” as used in the claims, are defined herein to mean one or more than one of the element that it introduces.

CLAIMS

What is claimed is:

1. A method comprising:
heat sealing a portion of a first sealing layer of a first film to a portion of a second sealing layer of a second film to produce a seal of a sealed packaging;
increasing an internal pressure of the sealed packaging via an orifice extending through a portion of the sealed packaging; and
measuring a pressure decay for the internal pressure of the sealed packaging, wherein the pressure decay is a change in pressure (ΔP) over a measurement time (t_m).
2. The method of claim 1, further comprising:
disposing a wire between the portion of the first sealing layer and the portion of the second sealing layer.
3. The method of claim 2, wherein a leak forms at one or more sides of the wire.
4. The method of claim 3, further comprising:
determining a leak width; and
correlating $\Delta P/t_m$ to the leak width.
5. The method of claim 3, further comprising:
producing another sealed packaging wherein at least one heat sealing parameter is changed based on the $\Delta P/t_m$.
6. The method of any one of the preceding claims, further comprising:
stabilizing the internal pressure of the sealed packaging for about 1 minute to about 10 minutes prior to measuring the $\Delta P/t_m$.
7. The method of any one of the preceding claims, further comprising:
puncturing a wall of the sealed packaging with a needle to create the orifice extending through the sealed packaging; and

increasing the internal pressure (P_0) of the sealed packaging by introducing a gas to the sealed packaging via the needle.

8. The method of any one of the preceding claims, wherein the P_0 is about 100 mbar to about 1000 mbar before measuring the $\Delta P/t_m$.

9. The method of any one of the preceding claims, wherein measuring the t_m is about 1 minute to about 1 hour.

10. The method of any one of the preceding claims, further comprising:

determining experimentally the P_0 and/or the t_m by a method comprising:

wherein the sealed packaging is a first sealed packaging and the pressure decay is a first pressure decay,

producing a second sealed packaging with a wire between the sealing layers;

producing a third sealed packaging without the wire between the sealing layers;

increasing an internal pressure of the second and third sealed packagings via an orifice extending through a portion of the second and third sealed packagings; and

measuring a second and a third pressure decay the second and third sealed packagings, respectively, and

choosing the P_0 and/or the t_m such that $r = (\Delta P_{wire})/(\Delta P_{no\ wire})$ is greater than or equal to 2.

11. The method of any one of the preceding claims, further comprising:

estimating a leak width based on the $\Delta P/t_m$.

12. The method of any one of the preceding claims, further comprising:

identifying a hermeticity of the sealed packaging based on $\Delta P/t_m$.

13. The method of any one of the preceding claims, further comprising:

changing at least one heat sealing parameter when producing an additional sealed packaging based on the $\Delta P/t_m$.

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14. The method of any one of the preceding claims, wherein the sealed packaging comprises a material that is oxygen-sensitive and/or water-sensitive.
15. A method comprising:
forming a plurality of sealed packagings each under different heat sealing parameters, wherein each sealed packaging is produced by:
placing a wire between a portion of a first sealing layer of a first film and a portion of a second sealing layer of a second film;
heat sealing the portion of the first sealing layer to the portion of the second sealing layer to produce a seal of the sealed packaging;
testing a hermeticity of the plurality of sealed packagings by:
increasing an internal pressure of each sealed packaging via an orifice extending through a portion of each sealed packaging; and
measuring a pressure decay for the internal pressure of the each sealed packaging, wherein the pressure decay is a change in pressure (ΔP) over a measurement time (t_m).
16. The method of claim 15, further comprising:
evaluating the $\Delta P/t_m$ to produce revised heat sealing parameters; and
forming a plurality of hermetically sealed packagings under the revised heat sealing parameters, wherein each hermetically sealed packaging is produced by heat sealing a portion of a third sealing layer to a portion of a fourth sealing layer to produce the hermetically sealed packaging.
17. The method of claim 15 or claim 16, wherein the plurality of sealed packagings comprise a first subset of sealed packagings comprising films of a first composition and a second subset of sealed packagings comprising films of a second composition.
18. The method of claim 17, further comprising:
evaluating the $\Delta P/t_m$ to produce revised heat sealing parameters;
evaluating the $\Delta P/t_m$ to produce a preferred film composition; and
forming a plurality of hermetically sealed packagings under the revised heat sealing parameters, wherein each hermetically sealed packaging is produced by heat sealing a portion

of a third sealing layer having the preferred film composition to a portion of a fourth sealing layer having the preferred film composition to produce the hermetically sealed packaging.

19. The method of any one of claims 15-18, further comprising:
stabilizing the internal pressure of the sealed packaging for about 1 minute to about 10 minutes prior to measuring the $\Delta P/t_m$.
20. The method of any one of claims 15-19, further comprising:
puncturing a wall of each sealed packaging with a needle to create the orifice extending through each sealed packaging; and
increasing the internal pressure of each sealed packaging by introducing a gas to the sealed packaging via the needle.
21. The method of any one of claims 15-20, further comprising:
forming a second plurality of sealed packagings without the wire and that contain food using heat sealing conditions and a film composition that are based on the different heat sealing parameters with corresponding first or second compositions of the plurality of sealed packagings that produced a hermetic seal.
22. The method of any one of claims 15-21, wherein the sealed packaging comprises a material that is oxygen-sensitive and/or water-sensitive.

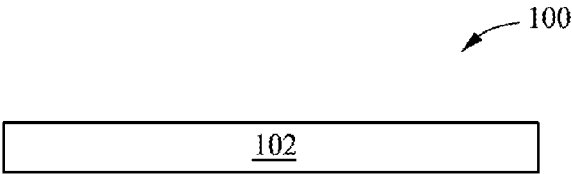


FIG. 1

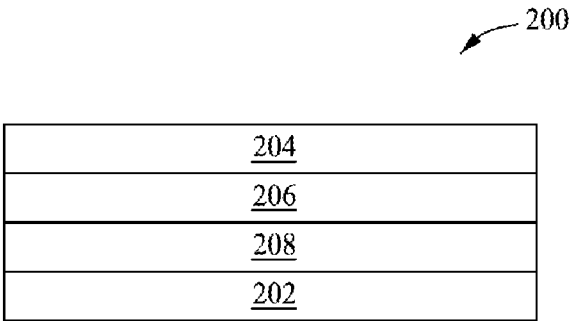


FIG. 2

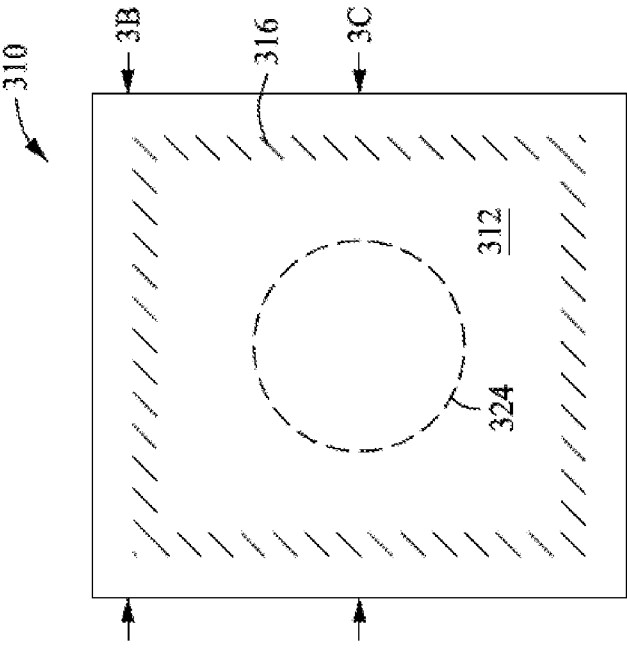


FIG. 3A

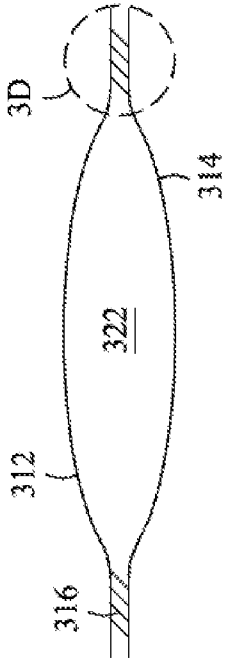


FIG. 3C

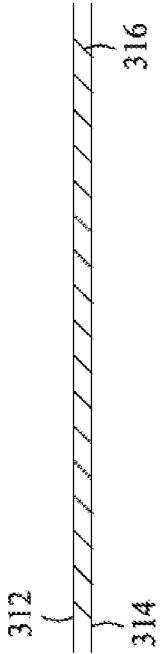


FIG. 3B

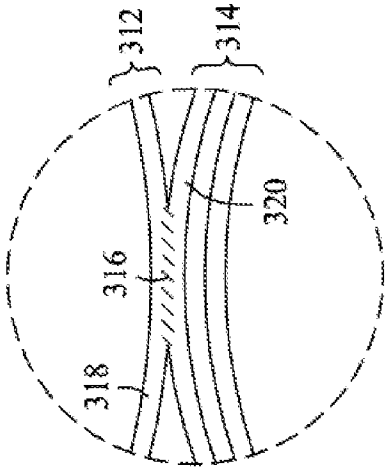


FIG. 3D

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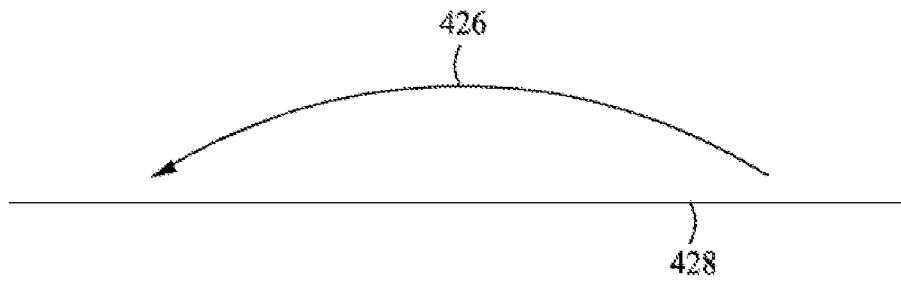


FIG. 4A

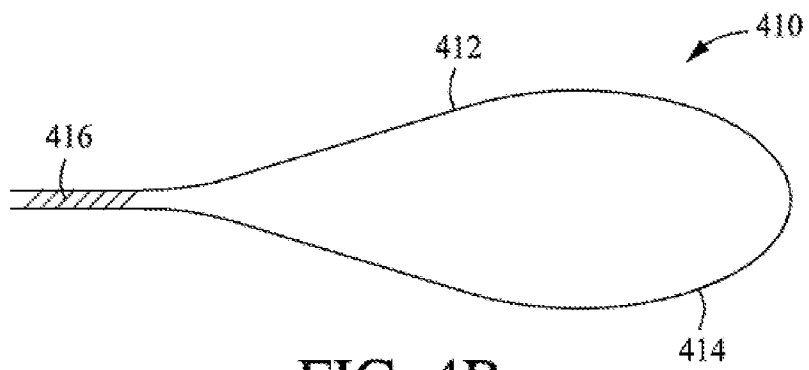


FIG. 4B

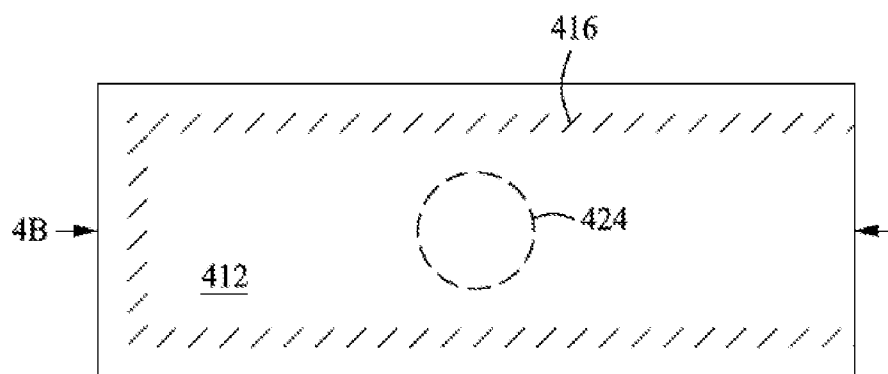


FIG. 4C

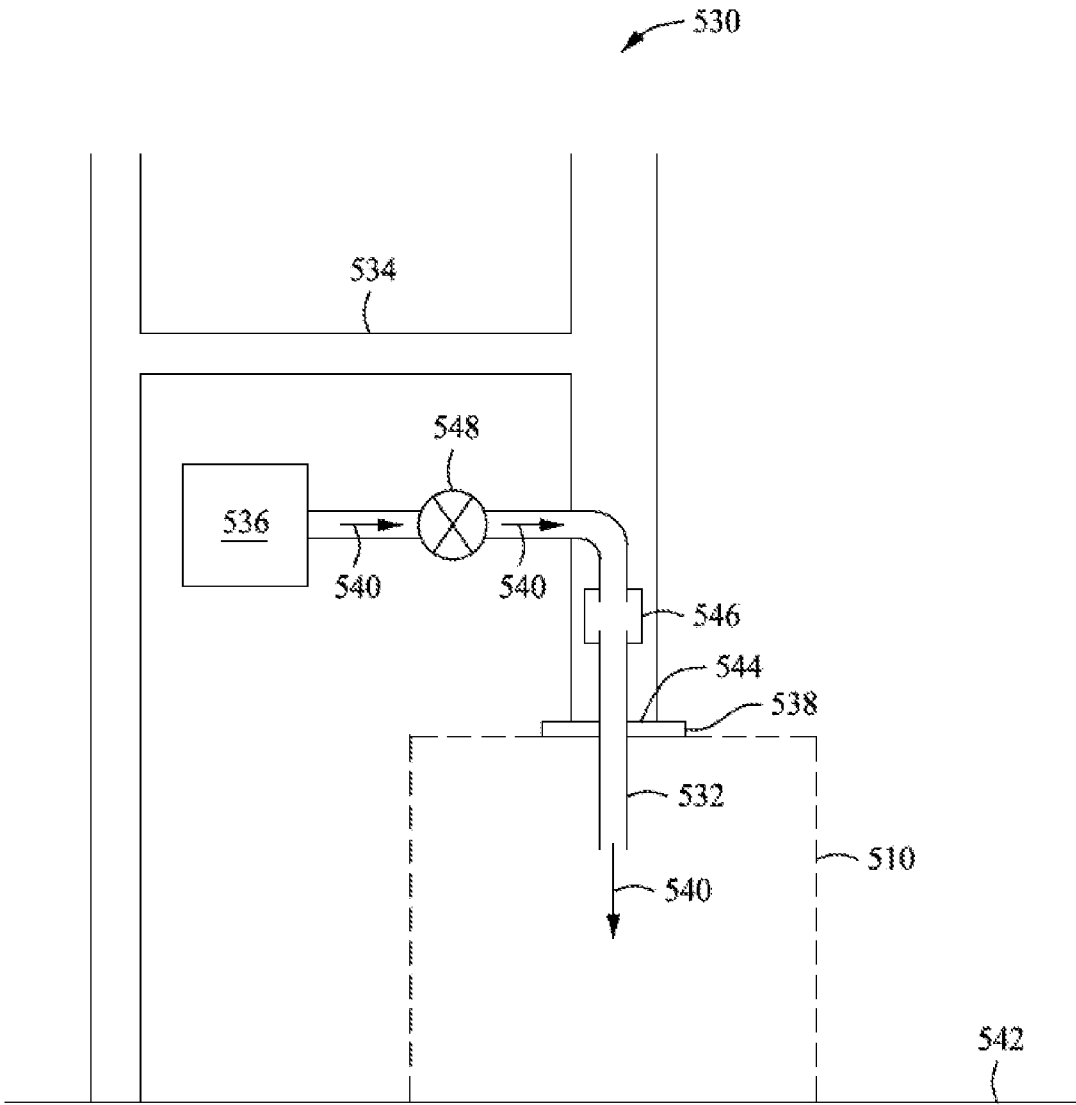


FIG. 5

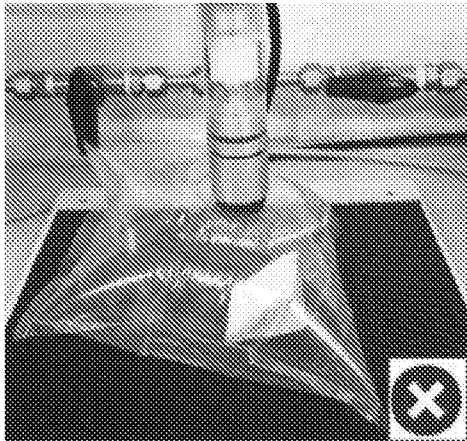


FIG. 6A

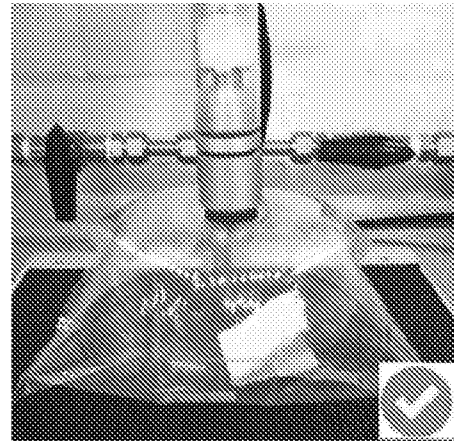


FIG. 6B

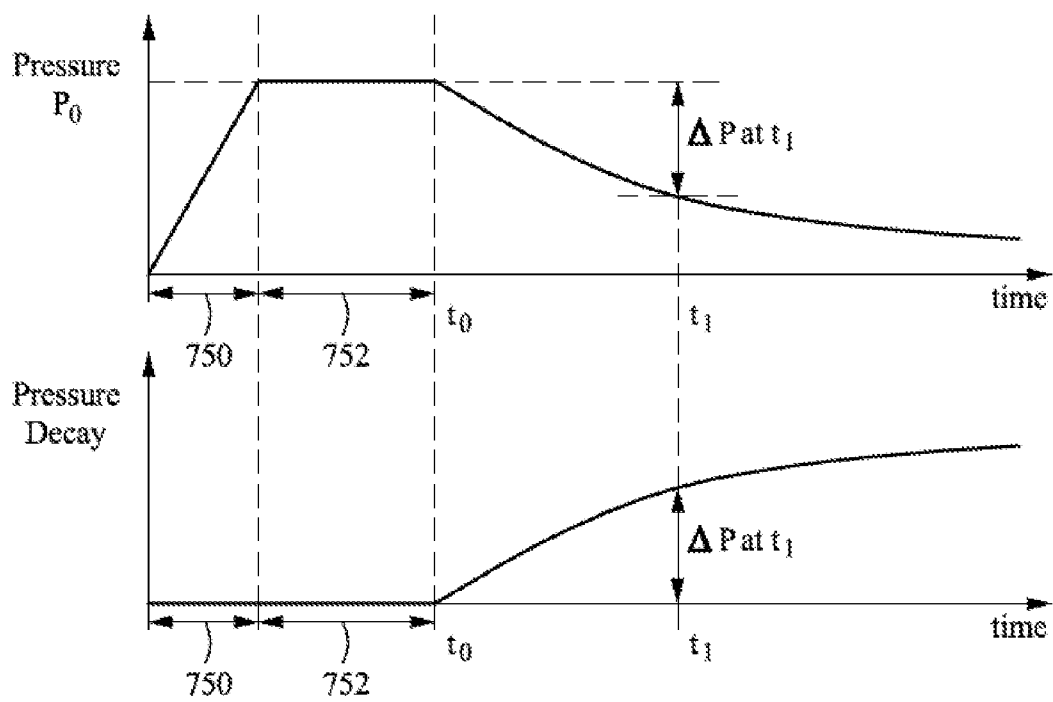


FIG. 7

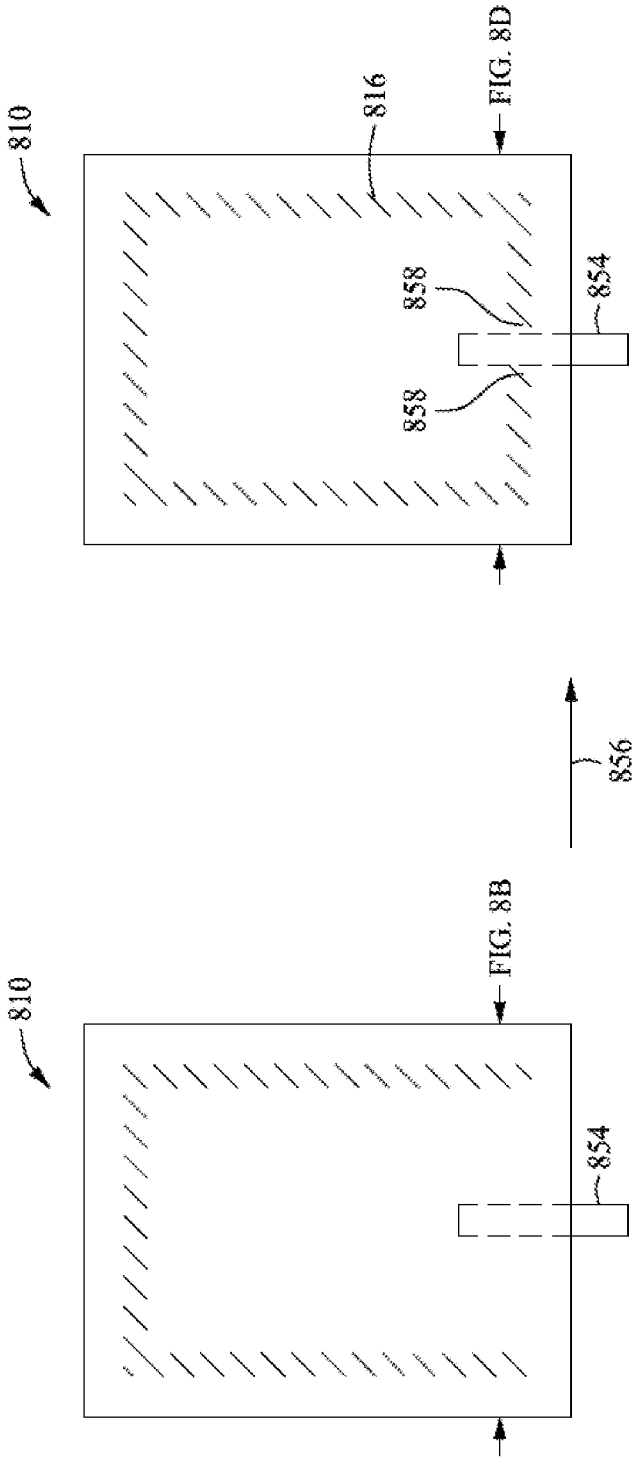


FIG. 8C

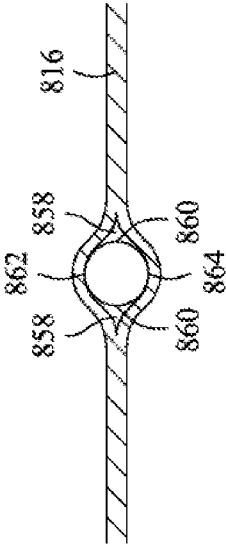


FIG. 8D

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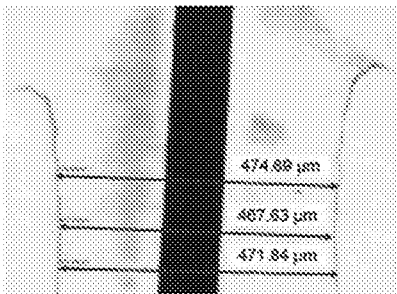


FIG. 9A

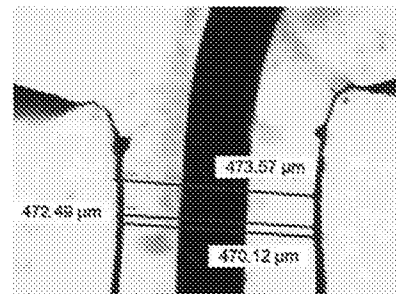


FIG. 9B

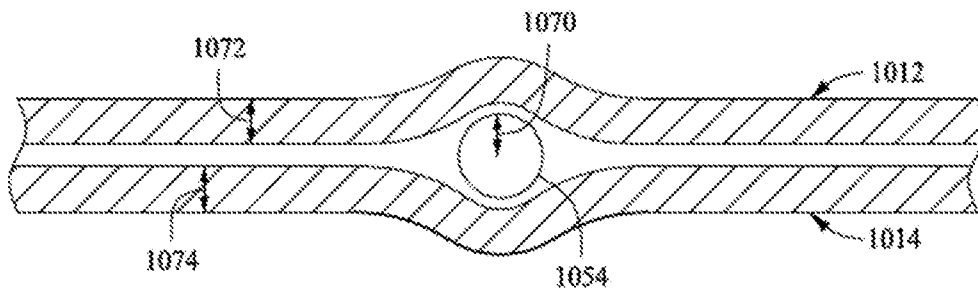


FIG. 10

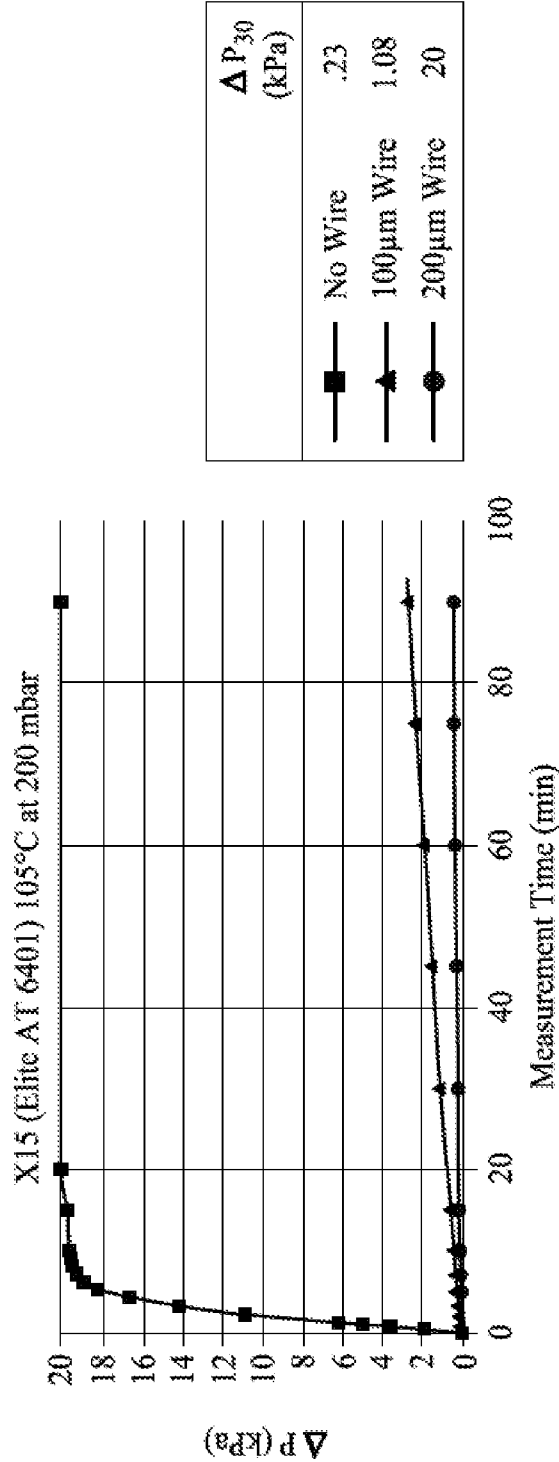


FIG. 11

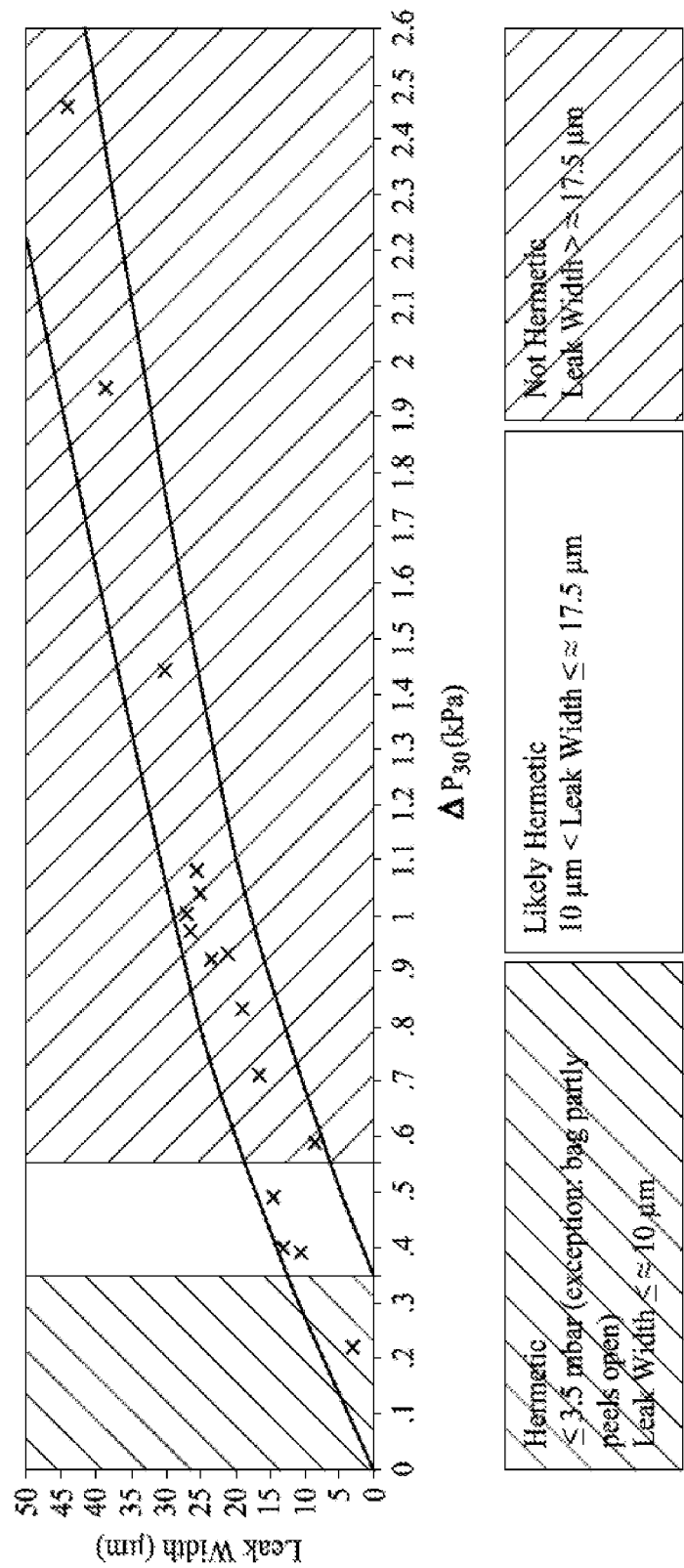


FIG. 12

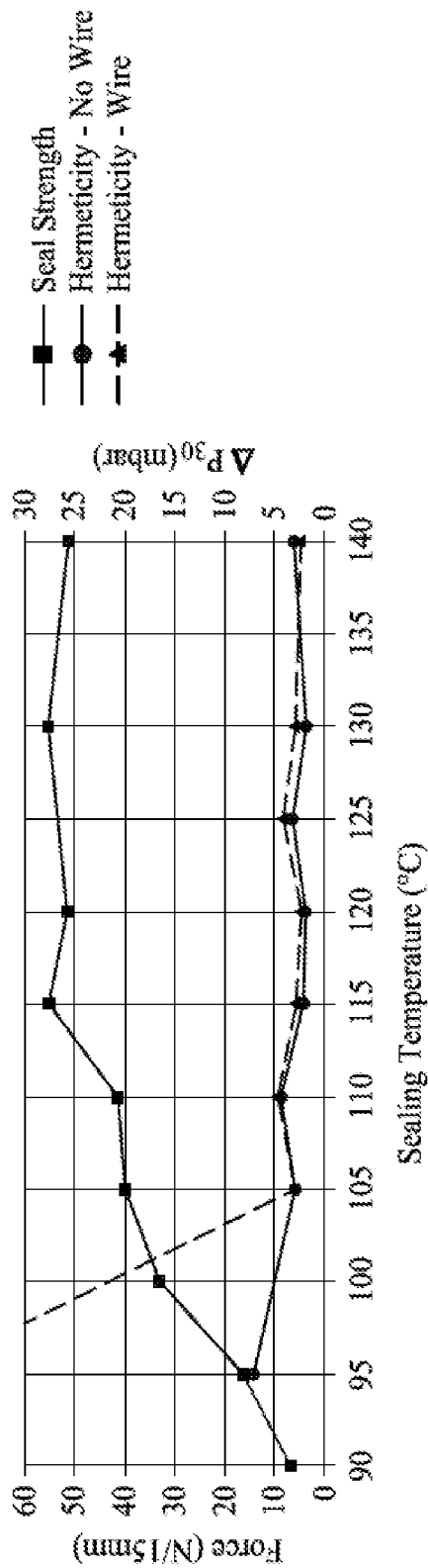


FIG. 13

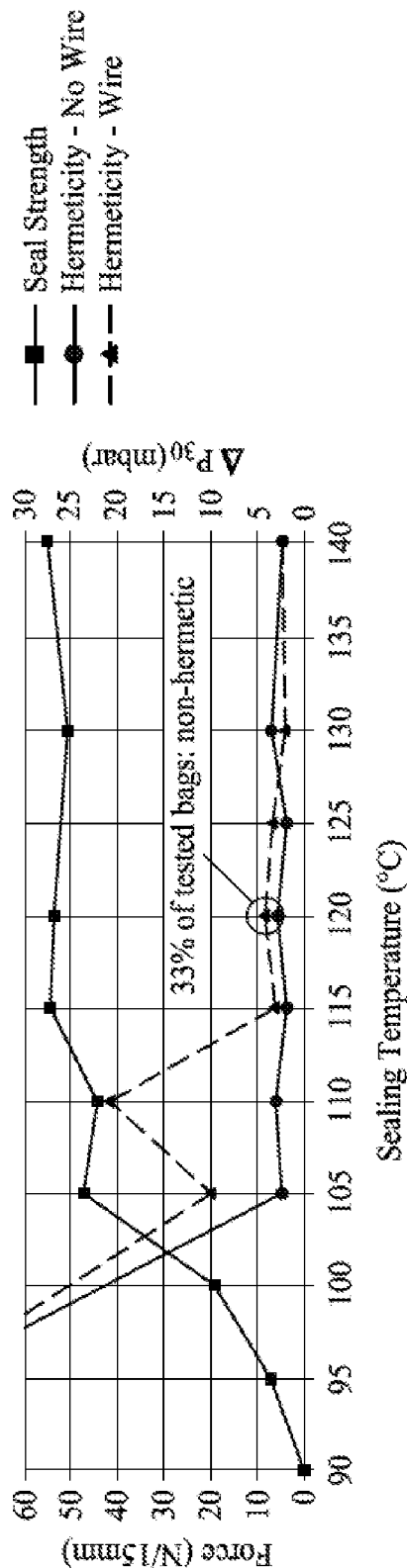


FIG. 14

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2019/031485

A. CLASSIFICATION OF SUBJECT MATTER
INV. B29C65/02 B29C65/82 G01M3/32
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 B29L G01M

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A	WO 2016/146950 A1 (SARTORIUS STEDIM FMT SAS [FR]) 22 September 2016 (2016-09-22) abstract figure 4 page 9, last paragraph claim 17	1,6-14 2-5, 15-22
X A	----- "ASTM F2095- 07e1 Standard test methods for pressure decay leak test for flexible packages with and without restraining plates", ASTM D6556 - 10, ASTM INTERNATIONAL, US, vol. 15.10, 1 January 2007 (2007-01-01), pages 1-6, XP008096350, ISSN: 0192-2998, DOI: 10.1520/F2095-01 the whole document ----- -/-	1,6-14 2-5, 15-22



Further documents are listed in the continuation of Box C.



See patent family annex.

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

18 July 2019

Date of mailing of the international search report

26/07/2019

Name and mailing address of the ISA/

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Authorized officer

Taillandier, Sylvain

INTERNATIONAL SEARCH REPORT

International application No

PCT/US2019/031485

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 733 555 A (FRANKS STEPHEN H [US]) 29 March 1988 (1988-03-29) abstract figures 1-3 column 4, lines 23-42 -----	1,6-14

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2019/031485

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 2016146950	A1	22-09-2016	EP 3270866 A1 24-01-2018
			FR 3033696 A1 23-09-2016
			US 2018087997 A1 29-03-2018
			WO 2016146950 A1 22-09-2016

US 4733555	A	29-03-1988	NONE
