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(54) Title: METHOD AND APPARATUS FOR THE PRODUCTION OF HIGH TENACITY POLYOLEFIN SHEET

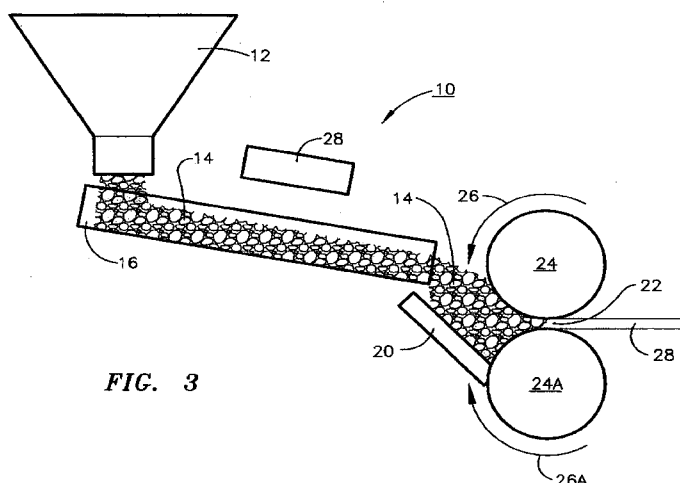


FIG. 3

(57) Abstract: A process for the production of virtually full density poly olefin suitable for further processing by drawing to form a high tenacity, highly oriented polyolefin sheet comprising: a) feeding a metered amount of polyolefin powder into the nip between two heated calender rolls initially set at a gap smaller than the size of the smallest polyolefin powder particle and at a temperature above the melting point of the powder; b) rolling the powder through the nip under these conditions until a coherent sheet of polyolefin is produced; and c) once a coherent sheet of polyolefin exits the nip lowering the temperature in the nip to a temperature below the melting point of the polyolefin powder and increasing the gap to a desired level above the thickness of the largest powder particle. Apparatus for the performance of such a process is also described.



Method and Apparatus for the Production of High Tenacity Polyolefin Sheet

Field of the Invention

01) The present invention relates to ultra high molecular weight polyethylene (UHMWPE) and other high molecular weight polyolefin materials useful for ballistic applications and more particularly to a novel and highly economical process for their production.

Background of the Invention

02) The processing of ultra high molecular weight polyethylene (UHMWPE), i.e. polyethylene having a molecular weight in excess of about 2 million, is known in the polymer arts to be extremely difficult. Products made from such materials are, however, very strong, tough and durable.

03) In the following series of U.S. Patents filed by Kobayashi et al and assigned to Nippon Oil Co., Ltd. a number of inventions related to the fabrication of fibers and films of polyolefins generally and UHMWPE specifically, are described: U.S Patents Nos. 4,996,011, 5,002,714, 5,091,133, 5,106,555, 5,200,129, and 5,578,373. The processes described in these patents generally describe the continuous production of high strength and high modulus polyolefin films by feeding polyolefin

powder between a combination of endless belts disposed in an up and down opposing relationship , compression molding the polyolefin powder at a temperature below its melting point between the endless belts and then rolling and stretching the resultant compression molded polyolefin into an oriented film. As compression molded, the sheet is relatively friable thus requiring the subsequent calendering or drawing operations to provide an oriented film that exhibits very good strength and durability properties. In fact, the strength of such materials produced by these processes can be 3 times that of steel on a weight basis and they exhibit very low creep.

04) Enhanced processes for the production of such materials have also been described in the following U.S Patents and patent applications: U.S. Patent No. 7,348,053 and U.S Patent Application Serial No. 11/217,279 filed September 1, 2005.

05) A common element of all of these prior art processes is that they require compaction of an UHMWPE powder as the initial step in the production process. Until now, it has been the thinking of the UHMWPE manufacturing community that such powder compaction was necessary in order to place the material in a form that it could be subsequently rolled and drawn as described in the referenced prior art. Stated differently, it has been the thinking that in order to produce the product in a process involving the subsequent rolling and drawing steps to obtain the orientation required for the production of ballistically useful UHMWPE, the powder had to first be placed in the form of a sheet that demonstrated sufficient

tenacity to be successfully processed in such subsequent rolling and drawing processes. In the prior art, such a form was obtained by compacting the powder into a relatively friable sheet that could be introduced into the rolling operation for subsequent processing.

06) The performance of this compaction process step, particularly in the production of UHMWPE sheets wider than 1-2 inches in width, requires the use of relatively massive, quite complex and very expensive equipment (measured in the millions of dollars for installed such equipment). Such equipment thus requires high levels of capital expenditures for installation and due to its complexity ongoing high operating and maintenance expenses.

07) U.S. Patent No. 4,436,682 to Knopp, issued March 13, 1984 describes a process for compacting polymer powders into fully dense products. According to this patent, a polymer powder is fed from a hopper into the nip between two rolls, compacted therein at a temperature below the melting point of the polymer powder and withdrawn from the nip under tension to form a "fully dense" polymer sheet. According to Knopp, when his process is applied to an UHMWPE powder, the resulting sheet has a density of about 0.82g/cc which he designates as "substantially fully dense". It is well known that the density of UHMWPE is on the order of above 0.945g/cc. Hence, the product of Knopp's process is hardly "fully dense" and is unsuited to further processing by calendering or drawing, since it will tear or break when subjected to such processes.

08) It would thus be of great benefit to the producer of such UHMWPE materials, particularly in widths greater than a couple of inches, if a much simpler, smaller and less expensive first process step could be substituted for the powder compaction step, without negatively affecting the either the product thus produced or significantly affecting the kinetics of the process, i.e. it did not, for example, slow production to an uneconomical rate.

Object of the Invention

09) It is therefore an object of the present invention to provide an enhanced process for the production of UHMWPE sheet that eliminates the need for the previously described compaction step and uses a much more cost effective and simpler process for the production of a high tenacity UHMWPE sheet that can undergo subsequent processing by drawing.

Summary of the Invention

10) According to the present invention, there is provided a process for the production of virtually full density polyolefin suitable for further processing by drawing to form a high tenacity, highly oriented polyolefin sheet comprising: a) feeding a metered amount of polyolefin powder into the nip between two heated

calender rolls initially set at a gap smaller than the size of the smallest polyolefin powder particle and at a temperature above the melting point of the powder; b) rolling the powder through the nip under these conditions until a coherent sheet of polyolefin is produced: and c) once a coherent sheet of polyolefin exits the nip lowering the temperature in the nip to a temperature below the melting point of the polyolefin powder and increasing the gap to a desired level above the thickness of the largest powder particle. Such a process not only eliminates the need for a separate and costly compaction step, but yields a coherent polyolefin sheet that is ready for drawing in accordance with prior art processes for the production of a high tenacity, highly oriented polyolefin sheet having a high heat of fusion. According to a preferred embodiment of the present invention, the polyolefin of choice is ultra high molecular weight polyethylene (UHMWPE).

Description of the Drawings

- 11) Figure 1 is a schematic representation of the production processes of the prior art.**

- 12) Figure 2 is a schematic diagram of a preferred embodiment of the apparatus used to implement the drawing portion of the preferred process of the present invention.**

13) Figure 3 is a schematic side view of the apparatus used to produce coherent UHMWPE sheet in accordance with the present invention.

Detailed Description

14) In the description that follows, operating parameters, material properties etc. are presented in the context of those for ultra high molecular weight polyethylene (UHMWPE), but it will be readily understood by the skilled artisan in the polymer field that the invention described herein is readily applicable to other polyolefin polymers such as high molecular weight polypropylene through the judicious selection of materials and process conditions appropriate for these other polyolefin materials.

15) The term “tape” as used herein refers to products having widths on the order of or greater than about 1/2 inch and preferably greater than 1 inch. The term “fiber” as used herein is meant to define a “narrow” tape, i.e. an element narrower than about 1/2 inch. The term “slit film fiber” refers specifically to a “fiber” or narrow tape made in accordance with the present invention that exhibits a generally rectangular cross-section and smooth, i.e. non-serrated or ragged edges. The terms “sheet” and “film” as used herein is meant to refer to thin sections of the materials of the present invention in widths up to and exceeding 160 inches in width as could be produced in large commercial equipment specifically designed for production in such widths. According to a preferred embodiment, such sheets and tapes have a

generally rectangular cross-section and smooth edges. Hence, the fundamental difference between a “tape”, a “slit film fiber”, a “fiber”, a “film” and a “sheet” as used to describe the products of the processes described herein relates to the width thereof and is generally independent of the thickness thereof.

16) Referring now to Figure 1, the processes described in the prior art and depicted schematically in Figure 1 comprised the continuous production of high strength and high modulus polyolefin films by feeding polyolefin powder between a combination of endless belts disposed in an up and down opposing relationship, compacting the polyolefin powder at a temperature below its melting point between the endless belts and then rolling and stretching the resultant compression molded polyolefin into an oriented film. To the extent of their relevance to the modified processes described herein, the aforementioned prior art descriptions contained in U.S Patents Nos. 4,996,011, 5,002,714, 5,091,133, 5,106,555, 5,200,129, and 5,578,373. are incorporated herein by reference in their entirety.

17) A major difference between the processes of the prior art and those of the present invention is that the present invention obviates the need for the compaction step and its related high cost entirely. Thus, the method described herein begins with heated polyolefin powder introduced as described hereinafter directly into a pair of heated, counter rotating calender rolls under very specific temperature and gap conditions to produce a coherent polyolefin sheet suitable for subsequent

further drawing to orient the polyolefin and to produce a ballistically useful high tenacity, highly oriented polymeric material.

18) According to a preferred embodiment of the present invention, the polyolefin processed in accordance with the process of the present invention is an UHMWPE that exhibits high crystallinity (above about 80% as determined by differential scanning calorimetry), a heat of fusion equal to or greater than 220 joules/gram and low levels of entanglement. Thus, it is preferred that the input starting material UHMWPE possess the degree of crystallinity and heat of fusion and meet the low entanglement requirements stated above. Such commercially available materials as Ticona X-168 from Ticona Engineering Polymers, 2600 Updike Road, Auburn Hills MI 48236 and type 1900 CM from Basell Corp., 2801 Centerville Road, Wilmington, DE 19808 are useful in the successful practice of the present invention.

19) Referring now to the accompanying drawings, as depicted in Figure 3, the initial step in the process of the present invention utilizes a direct roll apparatus 10 comprising a polymer powder hopper 12 that feeds a metered amount of polymer powder 14 into a vibratory chute 16 via a metering device 18 and thence to a containment plate 20. At containment plate 20 the powder is introduced into the gap 22 between two counter rotating heated calender rolls 24 and 24A rotating in the directions shown by arrows 26 and 26A. A heater 27 preferably an infrared heater, imparts heat to powder 14 as described more fully below. Heater 27 is preferably located from about 2 to about 8 inches above powder 14 in vibratory

chute 16 and set at a temperature of between about 160 and 220° F. These distances and temperatures will, of course, be variable depending upon the particular polymer powder 14 being processed, and the type of heater used, but have been found suitable for the processing of the preferred UHMWPE. As powder 14 cascades down vibratory chute 16 onto containment plate 20 it builds to a point where it is drawn into gap 22.

20) The successful practice of the present invention requires that at the start of the direct roll process, gap 22 be set narrower than the size of the smallest individual polymer powder particle, for example at about 50 μ . Gap 22 may, of course, be widened if the minimum particle size of polymer powder 14 is greater than 50 μ . Similarly, at start up of the direct rolling process described herein, heated calender rolls 24 and 24A are heated to a temperature above the melting point of polymer powder 14. While this melting point will be dependent upon the particular material being processed, in the case of the preferred UHMWPE starting materials described elsewhere herein this initial temperature is about 149° C or about 3° C above the melting point of the preferred UHMWPE. Lower temperatures could, of course, be appropriate for lower melting polyolefin materials. At this point, rolling of powder 14 is initiated. As soon as a coherent sheet of polymer 28 begins to emerge from gap 22 the temperature of calender rolls 24 and 24A is reduced to below the melting point of polymer powder 14 and gap 22 is increased to that desired for the final product thickness for coherent sheet 28. As used herein, the term "coherent sheet" is meant to define a polymer sheet that is suitable for further

processing by drawing without tearing, ripping or otherwise becoming unusable in such additional processing. For all practical purposes, such a sheet will be virtually fully dense such as in the case of the preferred UHMWPE materials described herein having a density above about 0.945g/cc. For the preferred UHMWPE powders 14 described elsewhere herein the operating temperature (the temperature after formation of a coherent sheet 28 is in the range of from about 136 to about 144° C and preferably between about 139 and about 141° C, and the operating gap is on the order of 100 μ and 230 μ and preferably at about 140 μ . It should be noted that the initial and operating temperatures recited herein are not necessarily set points for the polymer powder/sheet in nip 22, but rather surface temperatures of heated calender rolls 14 and 24A.

21) While the operating speed of the apparatus just described will vary with the particular polyolefin being processed, using the preferred UHMWPE materials described above, start up roll speeds of between about 1.9 to about 4.0 meters per minute have been found acceptable. Steady state operation of the apparatus is generally within the range of between about 2.0 and about 12.0 meters per minute. It should be noted that these operating speeds are based primarily on ones ability to take up coherent sheet 28 and the size of heated calendar rolls 24 and 24A, since larger rolls will generally tend to increase the surface in contact with the polymer in nip 22. Thus, if downstream operations or take up apparatus are capable of faster speeds, or larger diameter rolls are used, higher operating speeds for the direct roll process just described are possible.

22) The product of the just described process is a virtually full dense and translucent UHMWPE sheet, i.e. an UHMWPE sheet having a density of about 0.95 to about 0.98 g/cc.

23) While the apparatus used to practice the process of the present invention is depicted herein as horizontally oriented, the process will operate equally well in a vertical configuration, i.e. with the polymer powder being fed to gap 22 between two horizontally parallel calender rolls 24 and 24A. In this orientation, powder 14 is metered from a heated hopper located above horizontally parallel calender rolls 24 and 24A so that powder 24 is fed from above into gap 22 and the product sheet 28 is drawn from below gap 22. All other operating procedures, i.e. temperature control and gap setting variations remain the same.

24) While not critical to the successful practice of the present invention, and clearly variable depending upon the particular polyolefin being processed, roll surface roughnesses of from about 4 to about 8 RMS have been found suitable for the processing of the preferred UHMWPE materials described herein.

25) Post-processing of coherent sheet 28 to obtain a highly useful UHMWPE ballistic sheet, film, tape or fiber is performed in much the same fashion as and in apparatus similar to that described in issued U.S. Patent No. 7,348,053, issued

March 25, 2008, i.e. by drawing coherent sheet 28 which are referred to and incorporated herein in their entirety.

26) Referring now to Figure 2, the drawing apparatus utilized to achieve the thickness reductions of the coherent sheet produced as just described that result in production of the preferred UHMWPE products of the present invention 10 comprises:

a payoff 42, a godet stand 44 including heated godet rolls 16 (to anneal the product) and nip rolls 48 for establishing and maintaining tension in the line, a first draw zone 50, a first in-line tension sensor 52, a second godet stand 54, a second draw stand 56, a second in-line tension sensor 58, a third godet stand 60 and according to the preferred embodiment, a fibrillation unit 62, a nip roll stand 64 for maintaining tension and godet station 66 comprised of unheated take-up rolls 68.

As seen from Figure 1, the input or starting material of this process is generally the thick, compacted and rolled but unoriented product of the compaction step of the prior art production process. According to the preferred process of the present invention, the input or starting material in the drawing/calendaring process steps described below is, of course, coherent sheet 28 that emerges from gap 22 in the process described above.

27) Each of the elements of the apparatus just described and utilized in the successful practice of the present invention are well known in the film and fiber drawing arts as is their combination in a line of the type just described.

Consequently, no detailed description of such a line is required or will be made herein and the reader is referred to the numerous design manuals and descriptions of such apparatus commonly available in the art.

28) Maintaining a constant tension of between about 0.5 and about 5g/ denier, and preferably between about 0.8 and 3 g/denier during drawing is also important to the production of product having the required "thinness" and other enhanced properties specified herein. The term "denier" as used herein is defined as the weight in grams of 9000 meters of the product film, tape, sheet or fiber. At tension levels below 0.5g/denier no significant drawing or reduction will be obtained while at tension levels above about 5g/denier the material will tend to separate. In the case of drawing, tension is a function of the feed polymer and can vary broadly depending thereon and the ranges just specified refer to those found useful with particular preferred UHMWPE commercial starting materials.

29) Total reductions achieved during drawing and calendaring will generally be between about 50:1 and about 170:1 or more depending again upon the input raw material and the end use to which the product is to be applied. Such total drawing and calendaring is computed as the multiple of each of the individual reductions achieved by each of the combined process steps.

30) According to a highly preferred embodiment of the present invention, drawing is performed in line with direct rolling as described hereinabove. In such a

continuous process, calender rolls 24 and 24A become payoff 72 of drawing apparatus 70. Such an arrangement provides a highly efficient method for practicing the novel production process of the present invention.

31) After thickness reduction by drawing in the apparatus shown in Figure 2 according to the processing parameters just described, the UHMWPE films, sheets, fibers or tapes thus produced exhibit heats of fusion at or above about 243 joules/gram, tenacities in the range of from about 18 and 20 g/, tensile moduli between about 1200 and about 1800 g/d and elongations in the range of from about 1.6 to about 2.0 percent.

32) There has thus been described a novel process for the production of coherent polyolefin, preferably UHMWPE, sheet and high tenacity, highly oriented polyolefin, preferably UHMWPE, sheet, film, tape or fiber that eliminates the need for the prior art compaction step which, until the development described herein, was considered necessary for the successful production of such materials.

37) As the invention has been described, it will be apparent to those skilled in the art that the same may be varied in many ways without departing from the spirit and scope of the invention. Any and all such modifications are intended to be included within the scope of the appended claims.

What is claimed is:

- 1) A process for the production of high tenacity, highly oriented polyolefin sheet having a high heat of fusion comprising:**

 - a) feeding a metered amount of polyolefin powder into the nip between two counter rotating heated calender rolls set at an initial gap smaller than the size of the smallest polyolefin powder particle and at an initial temperature above the melting point of the polyolefin powder;**
 - b) rolling the powder through the gap under these conditions until a coherent sheet of polyolefin is produced:**
 - c) once a coherent sheet of polyolefin exits the gap lowering the temperature in the gap to an operating temperature below the melting point of the polyolefin powder and increasing the gap to a desired operating level above the thickness of the largest powder particle; and**
 - d) drawing the coherent sheet under controlled tension at a temperature of between about 140 and 158° C to produce a high tenacity, highly oriented polyolefin sheet having a high heat of fusion.**
- 2) The process of claim 1 wherein the polyolefin powder comprises an UHMWPE powder.**
- 3) The process of claim 2 wherein the UHMWPE exhibits high crystallinity, a heat of fusion equal to or greater than 220 joules/gram and low levels of entanglement.**

4) The process of claim 3 wherein the initial gap is below about 50 μ and the initial temperature is between about 146 and 150° C.

5) The process of claim 4 wherein the operating gap is between about 100 and about 230 μ and the operating temperature is between about 136 and about 144° C.

6) The process of claim 5 wherein the counter rotating calender rolls rotate at a rate of between about 2 and about 12 meters/minute.

7) The process of claim 1 wherein drawing is performed in-line and continuously with the formation of the coherent sheet.

8) The process of claim 6 wherein drawing is performed in-line and continuously with the formation of the coherent sheet.

9) A process for the production of a coherent virtually full density polyolefin sheet suitable for further processing by drawing to produce a high tenacity, highly oriented film, tape, fiber or sheet from a polyolefin powder comprising:

a) feeding a metered amount of polyolefin powder into the nip between two counter rotating heated calender rolls initially set at a gap smaller than the size of the smallest polyolefin powder particle and at a temperature above the melting point of the polyolefin powder;

b) rolling the powder through the nip under these conditions until a coherent polyolefin sheet is produced; and

c) once a coherent sheet of polyolefin exits the nip lowering the temperature in the nip to a temperature below the melting point of the polyolefin powder and increasing the gap to a desired level above the thickness of the largest powder particle.

10) The process of claim 9 wherein the polyolefin powder comprises an UHMWPE powder.

11) The process of claim 10 wherein the UHMWPE exhibits high crystallinity, a heat of fusion equal to or greater than 220 joules/gram and low levels of entanglement.

12) The process of claim 11 wherein the initial gap is below about 50 μ and the initial temperature is between about 146 and 150° C.

13) The process of claim 12 wherein the operating gap is between about 100 and about 230 μ and the operating temperature is between about 136 and about 144° C.

14) The process of claim 13 wherein the counter rotating calender rolls rotate at a rate of between about 2 and about 12 meters/minute.

15) A process for the production of high tenacity, highly oriented polyolefin sheet comprising:

a) feeding a metered amount of polyolefin powder into the nip between two counter rotating heated calender rolls set at a temperature below the melting point of the polyolefin powder;

b) rolling the powder through the nip to produce a coherent sheet of polyolefin; and

c) drawing the coherent sheet under controlled tension at a temperature of between about 140 and 158° C to produce a high tenacity, highly oriented polyolefin sheet having a high heat of fusion.

16) The process of claim 15 wherein the two counter rotating heated calender rolls are stacked vertically.

17) Apparatus for the production of a coherent virtually full density polyolefin sheet suitable for further processing by drawing to produce a high tenacity, highly oriented film, tape, fiber or sheet from a polyolefin powder comprising:

- a) a hopper including a metering mechanism;**
- b) a vibratory chute;**
- c) a containment plate;**
- d) a pair of counter rotating heated calender rolls defining a nip**

there between; and

- e) a heater above the vibratory chute;**

wherein powder is delivered from the hopper to the vibratory chute via the metering mechanism where the powder is preheated by the heater and thence to the containment plate where it accumulates and is introduced into the nip.

18) The apparatus of claim 16 wherein the counter rotating heated calender rolls are stacked vertically.

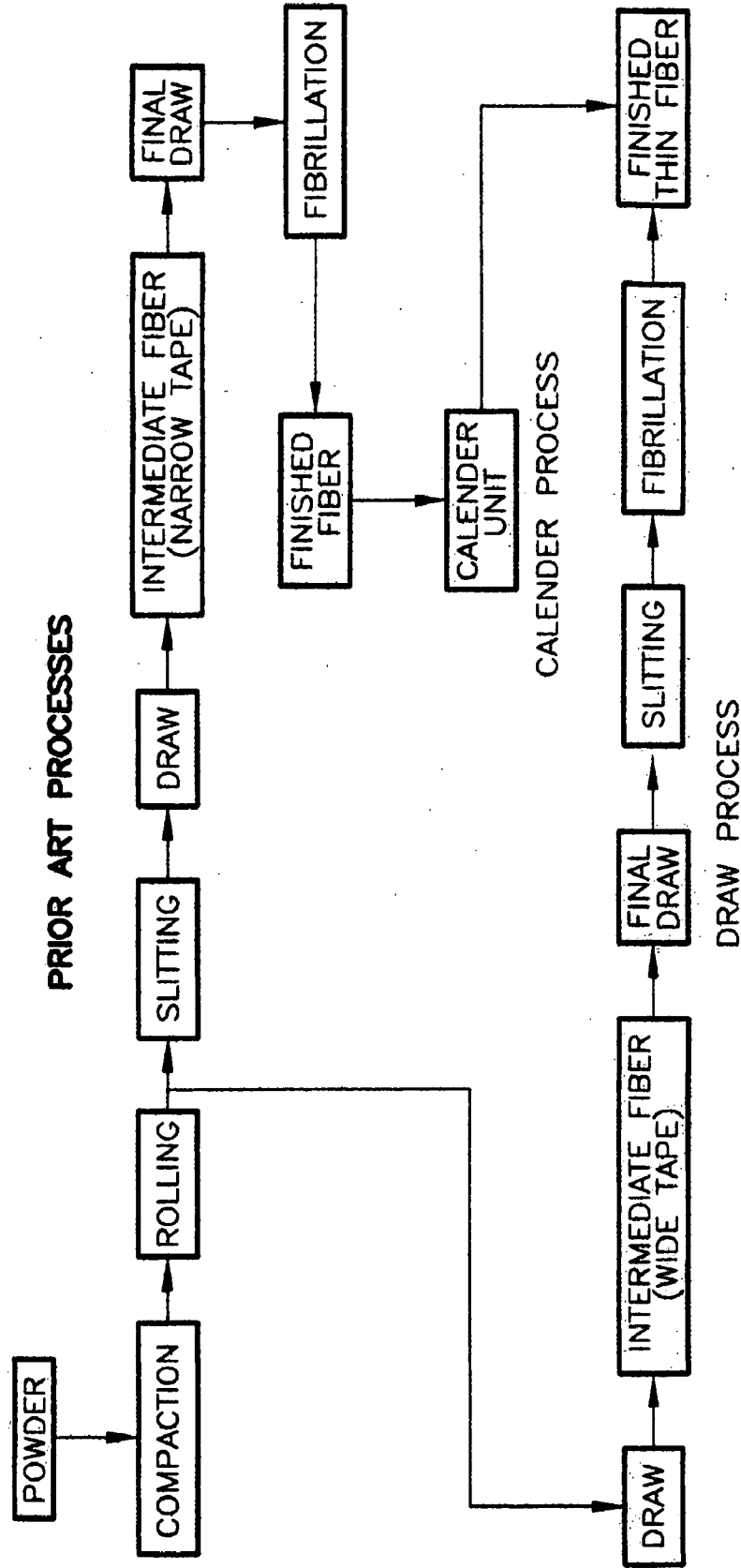


FIG. 1

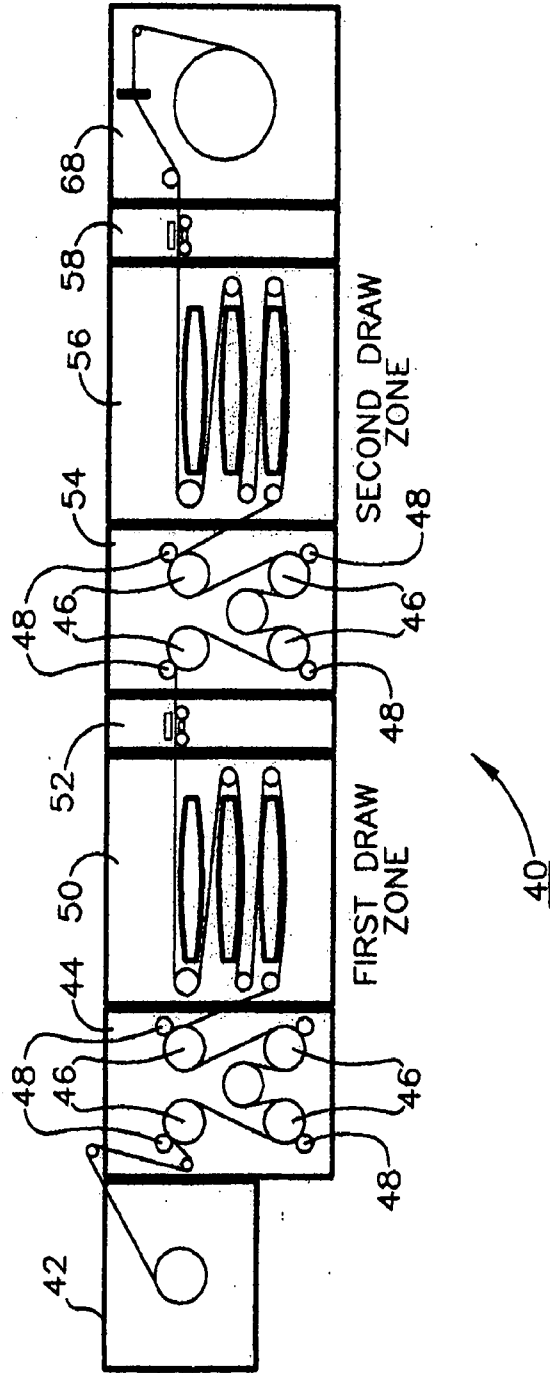


FIG. 2

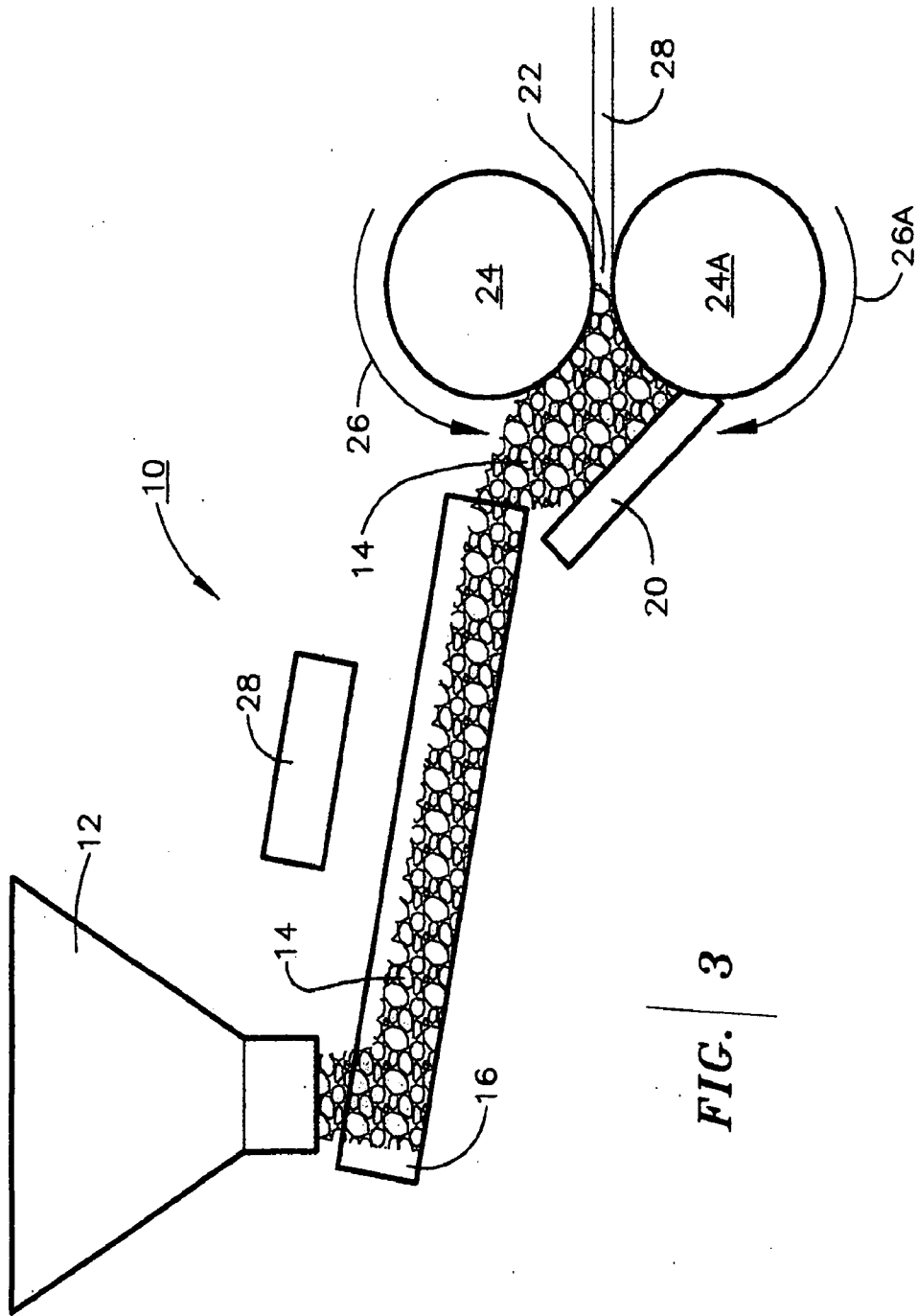


FIG. 3

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 09/02026

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - B29C 55/18 (2009.01)

USPC - 264/288.4; 156/324; 156/495; 428/93

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)

USPC Classifications search - 264/288.4

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
USPC - 264/288.4; 156/324; 156/495; 428/93(TERMS-see below and cursory search)[04 January 2008(04.01.2008)]

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

USPC, PubWest, Google Patent, Google

polyolefin/powder, UHMWPE, calendar rolls, nip, gap/initial gap, heat of fusion/fusion, temperature, coherent, joules-joules/gram, crystallinity, continuously, entanglement, counter rotating, [04 January 2008(04.01.2008)]

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4,436,682 A (Knopp) 13 March 1984(13.03.1984) Col. 3, ln 1-10; Col. 4, ln 4-7, Col. 3, ln 8-11; Col. 9, ln 3-4, Col. 8, ln 42-43; Col. 2, ln 45-48; Col. 3, ln 25-27; Col. 6, ln 41-48; Col. 9, ln 16-17; Col. 4, ln 23-34; Col. 8, ln 65-66; Col. 2, ln 68-Col. 3, ln 5; Col. 3, ln. 18-34; Col. 8, ln 46; Col. 3, ln 5-7; Col. 3, ln 53-59; Col. 3, ln 35-43; Col. 6, ln 49-Col. 7, ln 7; Col. 6, ln 52;	15 1-14, 16-18
Y	US 6,951,685 B1 (Weedon et al.) 04 October 2005(04.10.2005) Col. 3, ln 56-62; Col. 4, ln 34-38; Col. 4, ln 11-13; Col. 1, ln 20; Col. 3, ln 8; Col. 8, ln 31-33; Col. 4, ln 16-17; Col. 4, ln 12-17;	1-14, 16-18
Y	US 5,160,470 A (Graville et al.) 03 November 1992(03.11.1992) Col. 3, ln 24-32; Col. 3, ln 25-30; Col. 3, ln 35-38, Fig. 1	17 and 18

 Further documents are listed in the continuation of Box C.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search

10 May 2009(10.05.2009)

Date of mailing of the international search report

22 MAY 2009

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