Note: Within nine months of the publication of the mention of the grant of the European patent in the European Patent Bulletin, any person may give notice to the European Patent Office of opposition to that patent, in accordance with the Implementing Regulations. Notice of opposition shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).
The present invention relates to a method for reducing the amount of die buildup associated with the production of polyolefin based elastic fiber and for improving the unwind or release characteristics of such fiber. In particular the method involves the use of polydimethylsiloxane (PDMSO) in the polyolefin resin.

There has been much recent publicity related to cross-linked polyolefin-based elastic fibers, including the news of the Federal Trade Commission establishing a new generic term, "Lastol", to cover such fibers. The production and make-up of these fibers are known in the art, for example as described in US Patents 6,048,935; 6,140,442; 6,194,532; 6,248,851; 6,448,355; 6,436,534; 6,500,540; and 6,559,208.

In the production of these fibers, it has been observed that deposits gradually build up on the dies during melt spinning. Periodically, the production must be shut down in order to remove these deposits. It is therefore desirable to reduce or eliminate these buildups.

After the fiber has been formed, the fiber is typically wound on bobbins or spools for later use. Another problem which has been observed with some of these packages is that the fiber tends to stick to the package during unwinding leading to tangles and breaks. It is a goal of the present invention to improve the release or unwind characteristics of the fiber from these packages.

It has been discovered that the addition of small amounts of polydimethylsiloxane (PDMSO) to the polyolefin fiber prior to fiber formation aids in both of these goals. This result is unexpected as silicones are generally applied to the fiber externally by means of a metering pump or, a kiss roll. Such oils are commonly called spin finishes.

Figure 1 is a graph demonstrating the frequency dies needed to be cleaned using different levels of PDMSO. Figure 2 is a graph demonstrating the effect of PDMSO and lubricating oil on the releasability factor. Figure 3 demonstrates the load at break and elongation at break of fibers made in accordance with the present invention.

The present invention is a way of improving the fiber spinning and unwinding properties for any polyolefin-based fiber. The olefin polymer for use in the present invention can be any olefin based material capable of forming a fiber, including ethylene-alpha olefin interpolymers, substantially hydrogenated block polymers, propylene alpha olefin interpolymer (including propylene ethylene copolymers), styrene butadiene styrene block polymers, styrene-ethylene/butene-styrene block polymers, ethylene styrene interpolymers, polypropylenes, polyamides, polyurethanes and combinations thereof. The homogeneously branched ethylene polymers described in US 6,437,014, particularly the substantially linear ethylene polymers, are particularly well suited for use in this invention.

The present invention is particularly useful for fibers made form linear low density ethylene alpha-olefin copolymers, most preferably those having a density in the range of 0.80 to 0.89 g/cc (as determined by ASTM 1505) and a melt index from 0.5 dg/min to 10 dg/min (as determined by ASTM D1238). Preferred polyolefin materials are sold under the AFFINITY trade name by The Dow Chemical Company, the EXACT trade name sold by Exxon-Mobil Company, or the TAFMER trade name sold by Mitsui Chemical Company. The most preferred polymer is polyethylene with an octene comonomer content of 5 to 30 percent by weight, having a density of about 0.875 g/cc and a melt index of about 3 dg/min.

The PDMSO used is a hydroxyl-terminated, ultra high molecular weight poly(dimethylsiloxane). The PDMSO preferably has a molecular weight of approximately 2 million. The PDMSO is conveniently added to the polymer composition in the form of a masterbatch, in a polyethylene based carrier material, in an amount so that the final composition contains from 0.1 percent to 5 percent PDMSO by weight, more preferably from 0.3 percent to 2 percent by weight. Most preferably the final composition contains should contain no less than 0.5 percent by weight PDMSO.

The PDMSO can be added to the polymer in any way known to the art. The PDMSO is added prior to extrusion/fiber formation in order to get the benefit of both reduce die build-up and improved unwinding characteristics. Conveniently, the PDMSO may be added to the polyolefin material via a masterbatch with additional polyolefin material as the carrier medium.

The PDMSO reduces die build-up in the extrusion of the polymer and also improves the release characteristics of the fiber from the packages or spools of the fiber. This latter effect can be further improved with the external application of a spin finish. The spin finish can be any lubricating oil and is preferably selected from the group comprising silicones, mineral oils, ester oils with a viscosity range of 1 to 120 cSt, and blends thereof.

The method of the present invention leads to spools or packages having improved unwinding or release characteristics. In order to demonstrate this improvement a releasability test was created. For this test, the package/spool was placed in contact with a feed or drive roller (friction roller) rotating at a set speed (as provided by spinning the given roller at a speed of 30 rpm), and the filament was unwound onto a winding roller initially rotating at a speed set at three times the speed of the feed roller (that is the draft was 3x). At this speed and draft, no sticking was observed. The winding roller rotational speed was then gradually reduced until the point where the unwinding fiber sticks as indicated by the fiber no longer being removed tangentially from the package/spool. A releasability factor ("RF") was then calculated according to the following formula where "r.s." means rotational speed:

\[ RF = \frac{r.s. \times \text{Speed} \times \text{Length}}{100} \]
Fibers with the best releasability have very low values on this test (that is they do not stick even at low speeds). It is preferred that the method of the present invention reduce the release-ability value by at least twenty percent (for example if the release-ability factor for a particular fiber was 80 percent without the use of PDMSO, then the method of the present invention will preferably result in a releasability factor of 66 percent or less).

Another aspect of the present invention is an elastic polyolefin-based fiber package having a releasability of less than 100 percent, more preferably less than 50 percent, still more preferably less than 30 percent most preferably less than 20 percent, using the test just described.

EXAMPLES

For these Examples fiber was prepared from a base polymer prepared using an INSITE Constrained Geometry Metallocene catalyst. The base polymer was polyethylene with an octene comonomer content of about 35 percent to give a density of 0.875 g/cc with a melt index of 3.0 +/- 0.20 dg/min as determined using ASTM D1238.

A masterbatch of 40 percent by weight PDMSO in an ethylene styrene interpolymer was prepared. The master batch was then dry blended into the base polymer to provide the desired level of PDMSO in the fiber. For these examples 3 levels of PDMSO were evaluated, 0 percent, 0.5 percent and 1 percent. The polymer was then melt spun into fibers at a melt temperature of 270°C. Each polymer formulation was run for 8 hours and the number of times the spinnerets (dies) had to be cleaned is reported in Figure 1.

As can be seen from Figure 1, the frequency of cleaning required dropped significantly with the presence of PDMSO.

As the fiber was spun, it was wrapped onto packages. In some cases an external spin finish of silicone lubricating oil (Takemoto DELION 9535) was applied in an amount of 1.6 percent by weight to the fiber prior to winding onto the package. These packages where then evaluated for their release-ability using the test method described above. An improvement of 35 - 40 percent in the release value was obtained with PDMSO (indicated as "Si" in the figure) alone compared to filament with no lubrication and an improvement of up to 70 percent was observed for PDMSO in combination with external spin finish oil. The results of these experiments are reported in Figure 2.

The load at break (as determined by DIN 53 835 Part 1 and elongation at break (as determined by DIN 53 835 Part 1) of these fibers were measured at 100 mm grip distance at 500 mm/min separation speed. There was no significant loss of physical properties when using this internal and external lubrication method, as demonstrated in Figure 3.

Claims

1. A method for the production of elastic polyolefin-based fibers, wherein a polyolefin-based material is extruded and melt spun into a fiber, and wherein an amount of from 0.1 to 5.0% by weight of a hydroxyl-terminated ultra high molecular weight polydimethylsiloxane (PDMSO) is incorporated into the polyolefin-based material.

2. The method of Claim 1 wherein the polyolefin-based material comprises an ethylene alpha-olefin copolymer produced using a Metallocene catalyst.

3. The method of Claim 2 wherein the alpha olefin is octene.

4. The method of Claim 3 wherein the polyolefin-based material has a density in the range of 0.8 to 0.89 g/cc and a melt index in the range of 0.5 to 10 dg/min.

5. The method of Claim 2 wherein the PDMSO has an average molecular weight of approximately 2 million.

6. The method of Claim 1 wherein the PDMSO is added in an amount such that the PDMSO comprises 0.5 to 1 percent by weight of the fiber.

7. The method of Claim 2 wherein the PDMSO is added via a masterbatch.

8. The method of Claim 2 further comprising an additional step of adding a lubricating oil to the formed fiber via a spin
9. The method of Claim 1 characterized in that the method results in a releasability factor which is at least 20 percent less than a similar fiber without PDMSO.

10. A fiber package comprising fibers made according to the method of Claim 1, wherein the fiber package is characterized by having a releasability factor less than 50 percent.

Patentansprüche

1. Verfahren zur Herstellung von elastischen Polyolefinfasern, wobei ein Polyolefinmaterial extrudiert und zu einer Faser schmelzgesponnen wird, und wobei eine Menge von 0,1 bis 5,0 Gew.-% eines ultrahochmolekularen Polydimethylsiloxans (PDMSO) mit endständigen Hydroxylgruppen dem Polyolefinmaterial beigemengt wird.

2. Verfahren nach Anspruch 1, wobei das Polyolefinmaterial ein unter Verwendung eines Metallocenkatalysators hergestelltes Ethylen/α-Olefine-Copolymer umfasst.

3. Verfahren nach Anspruch 2, wobei das α-Olefin Octen ist.

4. Verfahren nach Anspruch 3, wobei das Polyolefinmaterial eine Dichte im Bereich von 0,8 bis 0,89 g/cm³ und einen Schmelzindex im Bereich von 0,5 bis 10 dg/min hat.

5. Verfahren nach Anspruch 2, wobei das PDMSO ein durchschnittliches Molekulargewicht von ungefähr 2 Millionen hat.

6. Verfahren nach Anspruch 1, wobei das PDMSO in einer solchen Menge zugesetzt wird, dass das PDMSO 0,5 bis 1 Gew.-% der Faser ausmacht.

7. Verfahren nach Anspruch 2, wobei das PDMSO über ein Masterbatch zugesetzt wird.


9. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass das Verfahren in einem Trennbarkeitsfaktor resultiert, der mindestens 20 Prozent niedriger ist als bei einer ähnlichen Faser ohne PDMSO.

10. Faserpackung aus Fasern, die nach dem Verfahren von Anspruch 1 hergestellt sind, wobei die Faserpackung dadurch gekennzeichnet ist, dass sie einen Trennbarkeitsfaktor kleiner als 50 Prozent hat.

Revendications

1. Procédé de production de fibres élastiques à base de polyoléfine, dans lequel on extrude et l’on file à l’état fondu un matériau à base de polyoléfine pour en faire une fibre, et dans lequel on incorpore dans le matériau à base de polyoléfine un polydiméthylsiloxane (PDMSO) à groupes terminaux hydroxyle et à masse molaire ultra-haute, en une proportion pondérale de 0,1 à 5,0 %.

2. Procédé conforme à la revendication 1, dans lequel le matériau à base de polyoléfine comprend un copolymère d’éthylène et d’alpha-oléfine préparé à l’aide d’un catalyseur de type métallocène.

3. Procédé conforme à la revendication 2, dans lequel l’alpha-oléfine est de l’octène.

4. Procédé conforme à la revendication 3, dans lequel le matériau à base de polyoléfine présente une masse volumique de 0,8 à 0,89 g/cm³ et un indice de fluidité à chaud de 0,5 à 10 dg/min.

5. Procédé conforme à la revendication 2, dans lequel le PDMSO présente une masse molaire moyenne d’environ 2 000 000.
6. Procédé conforme à la revendication 1, dans lequel on ajoute du PDMSO en une quantité telle que ce PDMSO représente de 0,5 à 1 % du poids de la fibre.

7. Procédé conforme à la revendication 2, dans lequel on ajoute le PDMSO au moyen d’un mélange-maître.

8. Procédé conforme à la revendication 2, qui comporte en outre une étape supplémentaire où l’on applique une huile lubrifiante à la fibre formée, au moyen d’un produit d’ensimage.

9. Procédé conforme à la revendication 1, caractérisé en ce qu’il permet d’obtenir une fibre dont l’indice d’aptitude au dévidage est inférieur d’au moins 20 % à celui d’une fibre similaire, mais sans PDMSO.

10. Enroulement de fibres comprenant des fibres produites selon un procédé conforme à la revendication 1, lequel enroulement de fibres se caractérise en ce qu’il présente un indice d’aptitude au dévidage inférieur à 50 %.
Figure 1

Figure 2
Physical Properties of Fiber

<table>
<thead>
<tr>
<th></th>
<th>OIl 0% Si 0%</th>
<th>OIl 0% Si 0.5%</th>
<th>OIl 0% Si 1.0%</th>
<th>OIl 1.6% Si 0%</th>
<th>OIl 1.6% Si 0.5%</th>
<th>OIl 1.6% Si 1.0%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Load @ break cN</td>
<td>71.24</td>
<td>72.76</td>
<td>78.07</td>
<td>75.17</td>
<td>79.08</td>
<td>81.22</td>
</tr>
<tr>
<td>Elongation at Break (%)</td>
<td>506</td>
<td>508</td>
<td>502</td>
<td>522</td>
<td>502</td>
<td>542</td>
</tr>
</tbody>
</table>

Figure 3
REFERENCES CITED IN THE DESCRIPTION

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