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(54) EXTRUSION OF POLYMERS

EXTRUSION VON POLYMEREN

EXTRUSION DE POLYMERES

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- **PATENT ABSTRACTS OF JAPAN vol. 013, no. 189 (M-821), 8 May 1989 & JP 01 016422 A (ISUZU MOTORS LTD), 19 January 1989**

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Description

[0001] This invention relates to an apparatus for producing a melt-extruded article of a polymer and to a method of making an article composed of a polymer according to the pre-characterising part of claim 1 and claim 8 respectively.

[0002] An apparatus and a method of this type is known from e.g. US-A-3 078 513.

[0003] Polymeric compositions are often shaped by melt extrusion. A conveying means conveys the molten polymeric composition as a continuous stream under pressure to an extrusion orifice, and forces it through the orifice to form an extrudate of desired shape. In many cases, the composition is fed as a solid to a conveying means which melts the composition through a combination of shearing and external heating. Well known conveying means include single screw and twin screw extruders. Unfortunately, conventional conveying means often fail to deliver the molten composition to the extrusion orifice at a constant rate, as a result of pressure variations within the conveying means, particularly when the composition has high viscosity. This results in an extrudate of variable quality. It is well known to mitigate this problem by placing a gear pump (alternatively known as a melt pump) between the conveying means and the extrusion orifice. The gear pump helps to deliver a substantially constant volume of molten polymeric composition to the extrusion orifice, and in consequence to produce an extrudate of improved quality. However, the gear pump must be correctly sized and the pressure variations must be small enough to ensure that the gear pump is neither starved nor overfed.

[0004] We have found that the problems outlined above are particularly serious when conductive polymers are melt extruded. The term "conductive polymer" is used herein to denote a composition which comprises a polymeric component and, dispersed therein, a particulate conductive filler, e.g. carbon black, graphite, a metal, a conductive ceramic, or a metal carbide or nitride. Particularly when the conductive polymer contains a high concentration of particulate filler (which may be only the conductive filler or a mixture of conductive and non-conductive fillers), pressure variations during the extrusion process can cause undesirable variation in the electrical properties of the extrudate, even when a gear pump is placed between the conveying means and the extrusion orifice.

[0005] It is the object of the present invention to provide an apparatus and a method which bleeds molten polymer from the system. ensure that the gear pump is not overfed and, therefore, continue to deliver a constant volume of molten polymeric composition to the extrusion orifices.

[0006] We have discovered, in accordance with the present invention, that when a gear pump is used in combination with a conveying means and an extrusion orifice (as outlined above) greatly improved results can be obtained through the use of a polymer relief means, which, when the pressure in the gear pump exceeds a selected level,

[0007] The present invention is particularly suitable for use in the extrusion of conductive polymers and will be chiefly described by reference to such use. However, it is to be understood that the invention can also be used in the extrusion of other polymeric compositions, and that the information given herein for conductive polymers is applicable to other polymeric compositions (with, if appropriate, modifications which will be apparent to those skilled in the extrusion of polymers having regard to their own knowledge and the information herein).

[0008] The above object is achieved in one aspect of the present invention by the apparatus for producing a melt-extruded article composed of a polymer as defined in claim 1.

[0009] As discussed below, the polymer relief means is a gear pump, and for this reason the gear pump **(3)** is often referred to herein as the "first" gear pump, even though it is the only gear pump when the polymer relief means is a component other than a gear pump.

[0010] The above object is achieved in a second aspect of the present invention by the method of making an article composed of a polymer as defined in claim 8.

[0011] Particular embodiments of the invention are the subject of the respective dependent claims.

[0012] This invention is particularly useful when (a) the molten polymeric composition is of high viscosity (since this tends to increase the pressure variations in the extruder or other conveying means); and/or (b) the product is adversely affected by pressure variations during the extrusion process, e.g. if it has at least one dimension which is very small, or if it has electrical or other properties which are dependent on the shear which has been exerted on the composition during extrusion; and/or (c) the extruder (or other conveying means) is of a type which inherently tends to produce pressure variations. Thus, preferred processes of the invention have one or more of the following features:--

(a) The polymeric composition

(i) comprises a polymer having a melting point T_m°C;
(ii) has a viscosity of 2 to 15 K.Pa.sec., particularly 8 to 11 K.Pa.sec., at a shear rate of 50 sec^{-1} at a temperature of 1.5 times T_m; and
(iii) is extruded at a temperature of not more than 1.5 times T_m.

Such a composition can contain, for example, at least 35% by volume of one or more particulate fillers, preferably at least 40% by volume of a conductive filler, e.g. carbon black.

(b)(i) The polymeric composition is a conductive polymer exhibiting PTC behavior, particularly such a composition having a resistivity less than 10 ohm-cm, particularly less than 1 ohm-cm.

(b)(ii) The polymeric composition is extruded as a sheet having a thickness less than 0.25 mm (0.010 inch), e.g. 0.13 mm (0.005 inch) or less; or is extruded as a strand or sheet which has a thickness greater than 0.25 mm (0.010 inch) and is then calendered to a sheet having a thickness less than 0.25 mm (0.0 10 inch), e.g. 0.13 mm (0.005 inch).

(c) The polymer conveying means is (i) a reciprocating single screw extruder, or (ii) a non-reciprocating screw extruder having a relatively short length to diameter (1/d) ratio, e.g. less than 15:1, for example 8:1 to 15:1.

[0013] As noted above, the polymer relief means is preferably a second gear pump, but it can be any component which will permit the polymeric composition to escape from the system when the pressure becomes excessive and will prevent it from doing so when the pressure has dropped to the desired level. Suitable components include gear pumps, pressure relief valves, and suitable reclosable orifices. When, as is preferred, a second gear pump is used as the polymer relief means, it can be substantially smaller than the first gear pump. Thus, it is preferred that the ratio of the maximum capacity of the first gear pump to the maximum capacity of the second gear pump is 5:1 to 50:1, e.g. 1 to 25:1. Preferably, the operation of the second gear pump (or other polymer relief means) is controlled by a control system which continuously monitors the pressure exerted on the polymeric composition and operates the second gear pump in response to that pressure. The control system can monitor the pressure in the first gear pump directly or it can monitor pressure at some other part of the system where the pressure is representative of the pressure in the first gear pump, e.g. in the passageway between the conveying means and the first gear pump.

[0014] In order for the first gear pump to deliver a constant volume of molten polymeric composition to the extrusion orifice, the pressure within the gear pump should be between a lower value, P_{lower}, and an upper value, P_{upper}. The first selected level, P_1 (above which the polymer relief means removes molten polymer), should be less than P_{upper}, preferably 0.25 to 0.9 times The second selected level, P_2 (below which the polymer relief means does not remove molten polymer) is (a) equal to or less than P_1, preferably from 0.4 to 1.0 times P_1, and (b) greater than P_{lower}, preferably 1.2 to 1.6 times P_{lower}. One of the advantages of the present invention is that because the polymer relief means ensures that pressure peaks in the extruder are not transmitted to the extrusion orifice, it is possible to operate at higher rates of extrusion than would be possible if the pressure peaks had to be below P_{upper} for the first gear pump.

[0015] This invention is particularly suitable for melt extruding conductive polymers of the kind used in circuit protection devices, i.e. PTC compositions which comprise a crystalline polymer, e.g. polyethylene or polyvinylidene fluoride, and a conductive particulate filler, preferably carbon black, in amount sufficient to reduce the room temperature resistivity to a low level, e.g. less than 5 ohm-cm.

[0016] Suitable conductive polymer compositions are disclosed in U.S. Patent Nos. 4,237,441, 4,388,607,4,534,889, 4,545,926, 4,560,498, 4,591,700, 4,724,417, 4,774,024, 4,935,156, 5,049,850, and 5,250,228, 5,378,407, 5,451,919, 5,582,770 and 5,747,147 and International Publication No. WO 96/29711.

[0017] The conductive polymer can be fed to the conveying means in any appropriate way, including

(a) in the form of a solid, e.g. pellets, prepared by a previous melt mixing process in which the conductive filler is dispersed in the polymer component; or

(b) in the form of a dry blend of the polymeric component and the conductive filler, in which case the conveying means must be one which will melt-mix these ingredients, as well as convey them to the first gear pump; or

(c) in the form of a molten dispersion of the conductive filler in the polymer component, prepared for example in an internal batch mixer.

For example, the present invention can be used to modify a process of the type disclosed in International Patent Publication No. WO 98/05503. That process comprises making a conductive polymer laminate by

(A) loading a dry blend of the polymeric component and the conductive filler into a mixing apparatus;

(B) mixing the polymeric component and the conductive filler in the mixing apparatus to form a molten mixture;

(C) transporting the molten mixture from the mixing apparatus through a die;

(D) forming the molten mixture into a polymeric sheet; and

(E) attaching metal foil to at least one side of the sheet to form a laminate,

steps (A) to (E) being conducted sequentially in a single continuous procedure.

[0018] In the present invention, the molten conductive polymer must pass through the apparatus as a continuous stream, but the duration of the process can be short or long. For example, when the conductive polymer is first prepared in an internal batch mixer and is then added as a molten mixture to the conveying means, the process may last for only a few minutes while the batch is processed.

[0019] Referring now to the drawing, this illustrates a single screw extruder **1**, a passageway **2**, a first gear pump **3**, an extrusion head **4** having a nozzle orifice **41**, and a second gear pump **5** having an exit **51**. Pressure control system **8** monitors the pressure at junction of passageway **2** and first gear pump **3**, and controls the operation of the second gear pump **5** in response to that pressure. Pellets of conductive polymer are fed from hopper **7** into extruder **1**, which melts the conductive polymer and conveys it as a continuous stream under pressure through passageway **2** to the first gear pump **3**. The first gear pump forces the molten conductive polymer through the extrusion orifice **41**. The extrudate is calendered into a sheet by rollers **6**. If the pressure observed by control system **8** exceeds a first preselected level, the control system switches on the second gear pump **5** so that molten conductive polymer is removed from the passageway **2** and discharged through exit **51**. When the pressure observed by the control system **8** has dropped to a second selected level, it switches off the second gear pump **5**.

Claims

1. Apparatus for producing a melt-extruded article composed of a polymer, the apparatus comprising

(1) conveying means for conveying a molten polymer under pressure (1);
(2) a passageway (2);
(3) a first gear pump (3); and
(4) an extrusion orifice (41);
characterized in that the molten polymer is a conductive polymer and the apparatus further comprises
(5) a polymer relief means which is a second gear pump (5),

the components (1), (2), (3) and (4) being arranged so that molten conductive polymer conveyed by the conveying means passes in a continuous stream sequentially through the conveying means, the passageway, the first gear pump and the extrusion orifice; and
the polymer relief means being associated with the first gear pump so that, if the pressure in the first gear pump exceeds a selected level, the polymer relief means removes molten conductive polymer from the continuous stream thereof in the passageway until the pressure in the first gear pump falls below a second selected level, thus allowing a substantially constant volume of molten conductive polymer to be delivered to the extrusion orifice.

2. Apparatus according to claim 1 wherein the ratio of the maximum capacity of the first gear pump (3) to the maximum capacity of the second gear pump (5) is 5:1 to 50:1.

3. Apparatus according to claim 1 or 2 wherein the conveying means comprises a single screw extruder (1) or a twin screw extruder.

4. Apparatus according to claim I or 2 wherein the conveying means comprises a reciprocating single screw extruder.

5. Apparatus according to claim 1 or 2 wherein the conveying means is a non-reciprocating screw extruder having an I/d ratio of 8:1 to 15:1.

6. Apparatus according to any one of claims 1 to 5 wherein the extrusion orifice is a nozzle die (41).

7. Apparatus according to any one of claims 1 to 6 which includes a control system (8) which continuously monitors the pressure exerted on the conductive polymer in the first gear pump and operates the second gear pump in response to that pressure.

8. A method of making an article composed of a polymer, the method comprising the steps of

(A) conveying a continuous stream of a molten polymer under pressure to a first gear pump (3);
(B) operating the first gear pump so that the molten polymer conveyed to the first gear pump in step (A) passes through the first gear pump to an extrusion orifice (41) and is extruded through the orifice; and
(C) when the pressure within the first gear pump exceeds a selected level, removing molten polymer from the continuous stream thereof,

characterized in that

(i) the molten polymer is a conductive polymer that (1) comprises a particulate conductive filler dispersed in a polymer component, and (2) prior to becoming molten the conductive polymer is in the form of (a) a solid prepared by a previous melt-mixing process, or (b) a dry-blend of the filler and the polymer component; and

(ii) the molten conductive polymer is removed by means of a second gear pump (5) so that a substantially constant volume of the molten conductive polymer is delivered to the extrusion orifice.

9. A method according to claim 8 wherein the conductive polymer

(i) comprises a polymer having a melting point T_m°C;
(ii) has a viscosity of 2 to 15 K.Pa.sec at a shear rate of 50 sec^{-1} and at a temperature of 1.5 times T_m; and

(iii) is extruded at a temperature of not more than 1.5 times T_m.

10. A method according to claim 9 wherein the molten conductive polymer has a viscosity of 8 to 11 K.Pa. sec. at a shear rate of 50 sec^{-1} and at a temperature of 1.5 times T_m.

11. A method according to any one of claims 8 to 10 wherein the conductive polymer contains at least 35% by volume of at least one particulate filler.

12. A method according to any one of claims 8 to 10 wherein the conductive polymer exhibits PTC behavior and contains at least 40% by volume of carbon black.

13. A method according to any one of claims 8 to 12 wherein the extrusion orifice is a nozzle die (41), and the extrudate is calendered into a sheet.

14. A method according to any one of claims 8 to 13 wherein the extrusion orifice is a sheet die having a gap of at most 0.13 mm (0.005 in).

Patentansprüche

1. Vorrichtung zum Herstellen eines schmelzextrudierten Gegenstands, der aus einem Polymer besteht, wobei die Vorrichtung folgendes aufweist:

(1) eine Fördereinrichtung (1) zum Fördern eines geschmolzenen Polymers unter Druck;
(2) einen Durchgang (2);
(3) eine erste Zahnradpumpe (3); und
(4) eine Extrusionsöffnung (41);
dadurch gekennzeichnet,
daß das geschmolzene Polymer ein leitfähiges Polymer ist und daß die Vorrichtung ferner folgendes aufweist:
(5) eine Polymerentlastungseinrichtung, die eine zweite Zahnradpumpe (5) ist,

wobei die Komponenten (1), (2), (3) und (4) so angeordnet sind, daß von der Fördereinrichtung gefördertes geschmolzenes leitfähiges Polymer in einem kontinuierlichen Strom sequentiell durch die Fördereinrichtung, den Durchgang, die erste Zahnradpumpe und die Extrusionsöffnung strömt; und wobei die Polymerentlastungseinrichtung der ersten Zahnradpumpe zugeordnet ist, so daß dann, wenn der Druck in der ersten Zahnradpumpe einen ausgewählten Pegel überschreitet, die Polymerentlastungseinrichtung geschmolzenes leitfähiges Polymer von dem kontinuierlichen Strom davon in dem Durchgang entfernt, bis der Druck in der ersten Zahnradpumpe unter einen zweiten ausgewählten Pegel sinkt, so daß es ermöglicht wird, daß ein im wesentlichen konstantes Volumen von geschmolzenem leitfähigem Polymer zu der Extrusionsöffnung gefördert wird.

2. Vorrichtung nach Anspruch 1,
wobei das Verhältnis der maximalen Kapazität der ersten Zahnradpumpe (3) zu der maximalen Kapazität der zweiten Zahnradpumpe (5) 5:1 bis 50:1 beträgt.

3. Vorrichtung nach Anspruch 1 oder 2,
wobei die Fördereinrichtung einen Einschnecken-Extruder (1) oder einen Doppelschnecken-Extruder aufweist.

4. Vorrichtung nach Anspruch 1 oder 2,
wobei die Fördereinrichtung einen hin- und hergehenden Einschnecken-Extruder aufweist.

5. Vorrichtung nach Anspruch 1 oder 2,
wobei die Fördereinrichtung ein nicht hin- und hergehender Schneckenextruder mit einem 1/d-Verhältnis von 8:1 bis 15:1 ist.

6. Vorrichtung nach einem der Ansprüche 1 bis 5,
wobei die Extrusionsöffnung ein Düsenmundstück (41) ist.

7. Vorrichtung nach einem der Ansprüche 1 bis 6,
die ein Steuerungssystem (8) aufweist, das den auf das leitfähige Polymer in der ersten Zahnradpumpe aufgebrachten Druck kontinuierlich überwacht und die zweite Zahnradpumpe in Abhängigkeit von diesem Druck betätigt.

8. Verfahren zum Herstellen eines Gegenstands, der aus einem Polymer besteht, wobei das Verfahren die folgenden Schritte aufweist:

(A) Fördern eines kontinuierlichen Stroms eines geschmolzenen Polymers unter Druck zu einer ersten Zahnradpumpe (3);
(B) Betätigen der ersten Zahnradpumpe, so daß das in Schritt (A) zu der ersten Zahnradpumpe geförderte geschmolzene Polymer durch die erste Zahnradpumpe hindurch zu einer Extrusionsöffnung (41) strömt und durch die Öffnung extrudiert wird; und,
(C) wenn der Druck in der ersten Zahnradpumpe einen ausgewählten Pegel überschreitet, Entfernen von geschmolzenem Polymer von dem kontinuierlichen Strom davon,

dadurch gekennzeichnet, daß

(i) das geschmolzene Polymer ein leitfähiges Polymer ist, das (1) einen teilchenförmigen leit-

fähigen Füllstoff aufweist, der in einer Polymerkomponente verteilt ist, und wobei (2) das leitfähige Polymer, bevor es geschmolzen wird, in Form von (a) einem Feststoff, der durch einen vorhergehenden Schmelzemischvorgang hergestellt worden ist, oder (b) einer Trockenmischung aus dem Füllstoff und der Polymerkomponente vorliegt; und
(ii) das geschmolzene leitfähige Polymer mit Hilfe einer zweiten Zahnradpumpe (5) entfernt wird, so daß ein im wesentlichen konstantes Volumen des geschmolzenen leitfähigen Polymers zu der Extrusionsöffnung gefördert wird.

9. Verfahren nach Anspruch 8, wobei das leitfähige Polymer

(i) ein Polymer mit einem Schmelzpunkt T_m °C aufweist;
(ii) bei einer Scherrate von 50 s^{-1} und einer Temperatur, die das 1,5fache von T_m ist, eine Viskosität von 2 bis 15 kPa • s hat; und
(iii) bei einer Temperatur von nicht mehr als dem 1,5fachen von T_m extrudiert wird.

10. Verfahren nach Anspruch 9, wobei das geschmolzene leitfähige Polymer bei einer Scherrate von 50 s^{-1} und einer Temperatur, die das 1,5fache von T_m ist, eine Viskosität von 8 bis 11 kPa • s hat.

11. Verfahren nach einem der Ansprüche 8 bis 10, wobei das leitfähige Polymer mindestens 35 Vol.-% von mindestens einem teilchenförmigen Füllstoff enthält.

12. Verfahren nach einem der Ansprüche 8 bis 10, wobei das leitfähige Polymer ein PTC-Verhalten zeigt und mindestens 40 Vol.-% Ruß enthält.

13. Verfahren nach einem der Ansprüche 8 bis 12, wobei die Extrusionsöffnung ein Düsenmundstück (41) ist und das Extrudat zu einem Flächenkörper kalandriert wird.

14. Verfahren nach einem der Ansprüche 8 bis 13, wobei die Extrusionsöffnung eine Breitschlitzdüse mit einem Spalt von höchstens 0,13 mm (0,005 in) ist.

Revendications

1. Appareil pour produire un article extrudé à l'état fondu composé d'un polymère, l'appareil comprenant

(1) un moyen de transport pour transporter un polymère à l'état fondu sous pression (1);
(2) un passage (2);
(3) une première pompe à engrenages (3); et
(4) un orifice d'extrusion (41);
caractérisé en ce que le polymère à l'état fondu est un polymère conducteur et l'appareil comprend en outre
(5) un moyen de décharge de polymère qui est une seconde pompe à engrenages (5),

les organes (1), (2), (3) et (4) étant organisés de façon que le polymère conducteur à l'état fondu transporté par le moyen de transport passe séquentiellement, en un écoulement continu, à travers le moyen de transport, le passage, la première pompe à engrenages et l'orifice d'extrusion; et
le moyen de décharge de polymère étant associé à la première pompe à engrenages de façon que, si la pression dans la première pompe dépasse un niveau sélectionné, le moyen de décharge de polymère retire du polymère conducteur à l'état fondu de son écoulement continu dans le passage, jusqu'à ce que la pression dans la première pompe à engrenages tombe au-dessous d'un second niveau sélectionné, ce qui permet de fournir à l'orifice d'extrusion un volume pratiquement constant de polymère conducteur à l'état fondu.

2. Appareil selon la revendication 1, dans lequel le rapport entre la capacité maximale entre la première pompe à engrenages (3) et la capacité maximale de la seconde pompe à engrenages (5) est de 5:1 à 50:1.

3. Appareil selon la revendication 1 ou 2, dans lequel le moyen de transport comprend une extrudeuse à une seule vis (1) ou une extrudeuse à deux vis.

4. Appareil selon la revendication 1 ou 2, dans lequel le moyen de transport comprend une extrudeuse à une seule vis à mouvement alternatif.

5. Appareil selon la revendication 1 ou 2, dans lequel le moyen de transport est une extrudeuse à une seule vis à mouvement non alternatif ayant un rapport 1/d de 8:1 à 15:1.

6. Appareil selon l'une quelconque des revendications 1 à 5, dans lequel l'orifice d'extrusion est une filière de buse (41).

7. Appareil selon l'une quelconque des revendications 1 à 6 comprenant un système de commande (8) qui contrôle continuellement la pression exercée sur le polymère conducteur dans la première pompe à engrenages et actionne la seconde pompe à engrenages en réponse à cette pression.

8. Procédé de fabrication d'un article composé d'un polymère, le procédé comprenant les étapes suivantes :

(A) on transporte vers une première pompe à engrenages (3) un écoulement continu d'un polymère à l'état fondu sous pression;
(B) on actionne la première pompe à engrenages de façon que le polymère à l'état fondu transporté vers la première pompe à engrenages à l'étape

(A) traverse la première pompe à engrenages vers un orifice d'extrusion (41) et soit extrudé à travers l'orifice; et
(C) lorsque la pression à l'intérieur de la première pompe à engrenages dépasse un niveau sélectionné, on retire du polymère à l'état fondu de l'écoulement continu de celui-ci,

caractérisé en ce que

(i) le polymère à l'état fondu est un polymère conducteur qui (1) comprend une charge conductrice sous forme de particules dispersée dans un composant consistant en polymère, et (2) avant de prendre l'état fondu, le polymère conducteur est sous la forme de (a) un solide préparé par un processus de fusion-mélange antérieur, ou (b) un mélange à sec de la charge et du composant consistant en polymère, et
(ii) le polymère conducteur à l'état fondu est retiré au moyen d'une seconde pompe à engrenages (5), de façon qu'un volume pratiquement constant du polymère conducteur à l'état fondu soit fourni à l'orifice d'extrusion.

9. Un procédé selon la revendication 8, dans lequel le polymère conducteur

(i) comprend un polymère ayant un point de fusion de T_m°C;
(ii) a une viscosité de 2 à 15 kPa.s à une vitesse de cisaillement de 50 s^{-1} et à une température de 1,5 fois T_m; et
(iii) est extrudé à une température ne dépassant pas 1,5 fois T_m.

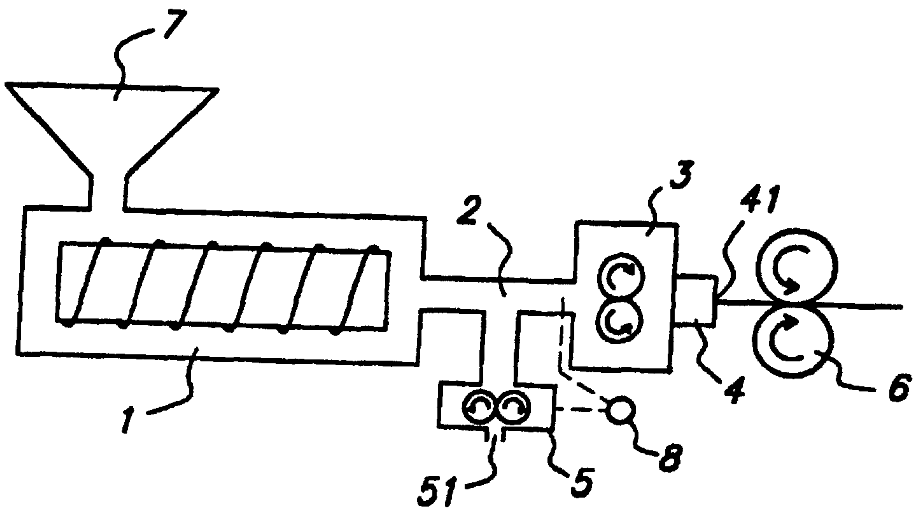
10. Procédé selon la revendication 9, dans lequel le polymère conducteur à l'état fondu a une viscosité de 8 à 11 kPa.s à une vitesse de cisaillement de 50 s^{-1} et à une température de 1,5 fois T_m.

11. Procédé selon l'une quelconque des revendications 8 à 10, dans lequel le polymère conducteur contient au moins 35% en volume d'au moins une charge sous forme de particules.

12. Procédé selon l'une quelconque des revendications 8 à 10, dans lequel le polymère conducteur présente un comportement à coefficient de température positif (CTP) et contient au moins 40% en volume de noir de carbone.

13. Procédé selon l'une quelconque des revendications 8 à 12, dans lequel l'orifice d'extrusion est une filière de buse (41), et l'extrudat est calandré pour donner une feuille.

14. Procédé selon l'une quelconque des revendications 8 à 13, dans lequel l'orifice d'extrusion est une filière de formation de feuille ayant un espace d'au plus 0,13 mm (0,005 pouce).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