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(54) **PTC TRACING BELT AND MANUFACTURING METHOD THEREFOR.**

(57) The present invention provides a PTC tracing belt and a manufacturing method therefor. The manufacturing method includes: mixing a molten polymer with a surface coated and modified tetrapod-like zinc oxide whisker, and then performing blending; then co-extruding a mixture and two parallel core wires to coat conductive plastic layers between outer layers of the two core wires and the two core wires; and performing cooling 10 and curing formation to obtain the PTC tracing belt, where a mass ratio of the polymer to the surface coated and modified tetrapod-like zinc oxide whisker is 100 to (5-15). A preparation method for the surface coated and modified tetrapod-like zinc oxide whisker includes: depositing a metal film layer on a surface of a tetrapod-like zinc oxide whisker by means of a physical vapor deposition method.

PTC TRACING BELT AND MANUFACTURING METHOD THEREFOR

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BACKGROUND OF THE INVENTION

[0001] 1. Technical Field

[0002] The present invention relates to the technical field of polymer composites, in particular to a PTC tracing belt and a manufacturing method therefor.

[0003] 2. Description of Related Art

[0004] Conductive polymer composites with a positive temperature coefficient (PTC) effect of resistance may be obtained by blending conductor fillers and polymer matrix materials. These materials are widely applied in the technical fields of heaters, sensors, tracing belts, etc.

[0005] A self-regulating PTC tracing belt is a common application product using a modified material, in which a polymer PTC material has a conductor network connected and conducted at a room temperature inside; and at the room temperature, resistivity in the conductor network is low. Therefore, under the effect of a high-flux current, the PTC tracing belt will heat up rapidly, while heating will lead to volume expansion of a polymer matrix material in the polymer PTC material; and under the effect of expansion, the conducted conductor network will be gradually disconnected, so that a current flux decreases, and the heating is inhibited. In the prior art, carbon black or metal particles are usually used as a conductive filler. Such conductor filler has the effect of rapid reaction speed under a temperature effect, but has a small size itself, and has the problem of poor dispersity in the process that it and the polymer material are dispersed; while with the repeated formation of crystal forms in the expansion process of a polymer body, the poorly dispersed conductor particles will move in a matrix, and a huge difference in the conductor network at this time from an initial stage is eventually caused, which leads to poor performance repeatability of the material and low stability of a product.

[0006] At present, stability improvement in the polymeric PTC tracing belt is mainly based on dispersity improvement in the conductor filler. How to provide a PTC tracing belt with better stability and high performance repeatability has become one of the technical problems to be solved urgently.

BRIEF SUMMARY OF THE INVENTION

[0007] In view of this, the present invention proposes a PTC tracing belt and a manufacturing method therefor, aiming to improve the stability and the repeatability of the

PTC tracing belt.

[0008] The technical solution of the present invention is such implemented that: a manufacturing method for a PTC tracing belt of the present invention includes the following steps:

[0009] mixing a molten polymer with a surface coated and modified tetrapod-like zinc oxide whisker, and then performing blending; then co-extruding a mixture and two parallel core wires to coat conductive plastic layers between outer layers of the two core wires and the two core wires; and performing cooling and curing formation to obtain the PTC tracing belt, where a mass ratio of the polymer to the surface coated and modified tetrapod-like zinc oxide whisker is 100 to (5-15). A preparation method for the surface coated and modified tetrapod-like zinc oxide whisker includes: depositing a metal film layer on a surface of a tetrapod-like zinc oxide whisker by means of a physical vapor deposition method.

[0010] In some implementations, an additive, such as a stabilizer, a flame retardant, an antioxidant and a pigment may further be added to the conductive plastic layer.

[0011] In some implementations, the metal film layer deposited on the surface of the surface coated and modified tetrapod-like zinc oxide whisker is a copper film.

[0012] In some implementations, for the surface coated and modified tetrapod-like zinc oxide whisker, a mass ratio of a zinc oxide whisker to metal copper is 1 to (0.001-0.01).

[0013] In some implementations, the surface coated and modified tetrapod-like zinc oxide whisker has a diameter of 0.5-5 μm and a length of 10-50 μm.

[0014] In some implementations, the polymer is one of polyethylene, polypropylene and polyvinylidene fluoride.

[0015] In some implementations, before depositing the metal film layer on the surface of the tetrapod-like zinc oxide whisker, the manufacturing method further includes: washing the tetrapod-like zinc oxide whisker with hydrochloric acid.

[0016] In some implementations, after washing the tetrapod-like zinc oxide whisker with the hydrochloric acid, and before depositing the metal film layer on the surface of the tetrapod-like zinc oxide whisker, the manufacturing method further includes: washing the tetrapod-like zinc oxide whisker in a plasma atmosphere.

[0017] In some implementations, in a case of mixing a molten polymer with a surface coated and modified tetrapod-like zinc oxide whisker, and then performing

blending, the manufacturing method further includes: adding carbon black, wherein a mass ratio of the polymer to the carbon black is 100 to (2-5).

[0018] In another aspect, the present invention further provides a PTC tracing belt manufactured using the above method.

[0019] In some implementations, the PTC tracing belt is further coated with an insulating layer on an outer side of the conductive plastic layer.

[0020] In some implementations, the PTC tracing belt is further coated with a sheath layer on an outer side of the insulating layer.

[0021] In some implementations, the PTC tracing belt may further be provided with a shielding layer between the insulating layer and the sheath layer.

[0022] Compared with the prior art, the PTC tracing belt and the manufacturing method therefor of the present invention have the following beneficial effects:

[0023] In the present invention, the tetrapod-like zinc oxide whisker is used as the matrix for filling a conductor. By depositing the metal layer on the surface of the tetrapod-like zinc oxide whisker, the tetrapod-like zinc oxide whisker is electrically conductive; compared with the conventionally dispersed conductor particles, the dispersion stability of the tetrapod-like whisker in the polymer matrix is stronger; and even in the recycling use process of high cycle times, the relative position stability of the tetrapod-like whisker is good, which may avoid a stability decrease caused by a slip of the conductor filler. Secondly, the tetrapod-like zinc oxide whisker is overlapped well in the polymer matrix after dispersion; and at the room temperature, an initial current value of a tracing zone is large, and a heating response speed is rapid. Thirdly, in the process of heating, the metal film deposited on the surface may also have expansion cracks with the tetrapod-like zinc oxide whisker; and the cracks may also weaken the conduction effect of the conductor network, so that a temperature feedback effect of the PTC tracing belt is good.

DETAILED DESCRIPTION OF THE INVENTION

[0024] The technical solution in the implementations of the present invention is clearly and completely described below in conjunction with the implementations of the present invention and the accompanying drawings. It is obvious that the described implementations are merely a part rather than all of the implementations of the present invention. All other implementations obtained by those of ordinary skill in the art based on the implementations of the present invention without creative efforts shall fall within the protection scope of the present invention.

[0025] Preparation Example 1

[0026] A tetrapod-like zinc oxide whisker having a diameter of 0.5-5 μm and a length of 10-50 μm was added in a vacuum coating machine, keeping a degree of vacuum in the vacuum coating machine at 500 Pa; with argon as working gas and copper as an evaporation source, the copper was heated to 1200°C, and the copper source started to be evaporated, keeping a temperature of the tetrapod-like zinc oxide whisker at 40°C; during evaporation coating, the tetrapod-like zinc oxide whisker was stirred at a stirring speed of 5-30 rpm; and evaporation coating was stopped until a mass ratio of the tetrapod-like zinc oxide whisker to a copper coated film on its surface was 1 to 0.001, so as to obtain a surface coated and modified tetrapod-like zinc oxide whisker.

[0027] Preparation Example 2

[0028] On the basis of Preparation Example 1, other conditions were kept unchanged, but the mass ratio of the tetrapod-like zinc oxide whisker to the copper coated on the surface was 1 to 0.005.

[0029] Preparation Example 3

[0030] On the basis of Preparation Example 1, other conditions were kept unchanged, but the mass ratio of the tetrapod-like zinc oxide whisker to the copper coated on the surface was 1 to 0.01.

[0031] Comparative Preparation Example 1

[0032] On the basis of Preparation Example 1, other conditions were kept unchanged, but the mass ratio of the tetrapod-like zinc oxide whisker to the copper coated on the surface was 1 to 0.0005.

[0033] Comparative Preparation Example 2

[0034] On the basis of Preparation Example 1, other conditions were kept unchanged, but the mass ratio of the tetrapod-like zinc oxide whisker to the copper coated on the surface was 1 to 0.02.

[0035] Preparation Example 3

[0036] On the basis of Preparation Example 1, other conditions were kept unchanged; but the tetrapod-like zinc oxide whisker was washed with hydrochloric acid in advance, then dried to a constant weight, and then subjected to evaporation coating.

[0037] Preparation Example 4

[0038] On the basis of Preparation Example 1, other conditions were kept unchanged; but the tetrapod-like zinc oxide whisker was washed with hydrochloric acid in advance, then dried to a constant weight, then washed for 5 min in an oxygen plasma

atmosphere, and then subjected to evaporation coating.

[0039] Comparative Preparation Example 3

[0040] On the basis of Preparation Example 1, other conditions were kept unchanged; but aluminum was used as the evaporation source, and a heating temperature for the aluminum during evaporation coating was 400°C.

[0041] Comparative Preparation Example 4

[0042] On the basis of Preparation Example 1, other conditions were kept unchanged; but iron was used as the evaporation source, and a heating temperature for the iron during evaporation coating was 1100°C.

[0043] Comparative Preparation Example 5

[0044] On the basis of Preparation Example 1, other conditions were kept unchanged; but zinc was used as the evaporation source, and a heating temperature for the zinc during evaporation coating was 600°C.

[0045] Comparative Preparation Example 6

[0046] On the basis of Preparation Example 1, other conditions were kept unchanged; but the tetrapod-like zinc oxide whisker was replaced with a nanorod-like zinc oxide whisker having a diameter of 0.5-5 μm and a length of 10-50 μm.

[0047] Embodiment 1

[0048] 5 parts of the surface coated and modified tetrapod-like zinc oxide whisker prepared in Preparation Example 1 and 100 parts of high-density polyethylene were weight separately, mixed, and then added in a plastic refining machine, where for the plastic refining machine, a heating temperature of a feeding region was 150°C, a heating temperature of a middle section was 170°C, a temperature of an extrusion region was 190°C, and a temperature of a die head was 60°C; and after melt mixing by the plastic refining machine, a product was co-extruded with two parallel copper stranded wires at a die head part of an extruder, so as to obtain a PTC tracing belt coated with a conductive plastic layer on its surface.

[0049] Embodiment 2

[0050] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Preparation Example 1 was used in an amount of 10 parts by weight.

[0051] Embodiment 3

[0052] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in

Preparation Example 1 was used in an amount of 15 parts by weight.

[0053] Comparative example 1

[0054] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Preparation Example 1 was used in an amount of 1 part by weight.

[0055] Comparative Example 2

[0056] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Preparation Example 1 was used in an amount of 20 parts by weight.

[0057] Embodiment 4

[0058] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Preparation Example 2 was used.

[0059] Embodiment 5

[0060] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Preparation Example 3 was used.

[0061] Comparative Example 3

[0062] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Comparative Preparation Example 1 was used.

[0063] Comparative Example 4

[0064] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Comparative Preparation Example 2 was used.

[0065] Embodiment 6

[0066] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Preparation Example 3 was used.

[0067] Embodiment 7

[0068] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Preparation Example 4 was used.

[0069] Embodiment 8

[0070] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Comparative Preparation Example 3 was used.

[0071] Embodiment 9

[0072] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Comparative Preparation Example 4 was used.

[0073] Embodiment 10

[0074] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Comparative Preparation Example 5 was used.

[0075] Comparative Example 5

[0076] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Preparation Example 1 was replaced with an equal amount of carbon black.

[0077] Embodiment 11

[0078] On the basis of Embodiment 1, other conditions were kept unchanged, but 2 parts of carbon black was further added.

[0079] Embodiment 12

[0080] On the basis of Embodiment 1, other conditions were kept unchanged, but 3 parts of carbon black was further added.

[0081] Embodiment 13

[0082] On the basis of Embodiment 1, other conditions were kept unchanged, but 5 parts of carbon black was further added.

[0083] Comparative Example 6

[0084] On the basis of Embodiment 1, other conditions were kept unchanged, but the surface coated and modified tetrapod-like zinc oxide whisker prepared in Comparative Preparation Example 6 was used.

[0085] Performance detection

[0086] 1. Stability test:

[0087] The PTC tracing belt prepared in each of above Embodiments 1-10 and Comparative Examples 1-8 was placed in an oven for heating-cooling cycling treatment, where a highest temperature for heating was 140°C; a heating rate was 10°C/min; after the temperature was raised to 140°C, thermal insulation treatment was performed for 10 min; a

temperature for cooling was 30°C; and a cooling rate was 10°C/min. The resistivity at 30°C was measured before and after 10 cycles, 50 cycles and 100 cycles, respectively. Measurement results are shown in the following table:

Group	0 cycle (Ω·cm)	10 cycles (Ω·cm)	50 cycles (Ω·cm)	100 cycles (Ω·cm)
Embodiment 1	1.55	1.62	1.66	1.71
Embodiment 2	1.28	1.30	1.32	1.35
Embodiment 3	0.93	0.96	0.98	1.01
Embodiment 4	1.48	1.54	1.57	1.60
Embodiment 5	1.42	1.47	1.50	1.54
Embodiment 6	1.56	1.58	1.60	1.62
Embodiment 7	1.55	1.55	1.56	1.57
Embodiment 8	1.63	1.82	2.55	4.31
Embodiment 9	1.69	2.21	2.34	2.57
Embodiment 10	1.65	1.85	2.23	2.43
Embodiment 11	1.51	1.55	1.57	1.58
Embodiment 12	1.47	1.49	1.50	1.51
Embodiment 13	1.46	1.49	1.50	1.51

Group	0 cycle (Ω·cm)	10 cycles (Ω·cm)	50 cycles (Ω·cm)	100 cycles (Ω·cm)
Comparative Example 1	3.17	5.62	7.74	12.31
Comparative Example 2	0.02	0.02	0.02	0.02
Comparative Example 3	1.67	1.74	1.79	1.82
Comparative Example 4	1.38	1.40	1.41	1.41
Comparative Example 5	5.88	7.92	15.41	26.69
Comparative Example 6	3.72	3.74	6.51	6.62

[0088] It can be seen from data of the resistivity after the cycles in the above embodiments and comparative examples that the PTC tracing belt manufactured by the manufacturing method of the present invention has good repeatability. Compared with conventional use of the conductor filler such as the carbon black, if the tetrapod-like zinc oxide whisker coated with a metal film layer on its surface is used as the conductor filler, a prepared polymeric PTC conductive coating has good performance reproducibility. Compared with the conventional carbon black conductor filler, it can still maintain a normal temperature conductivity close to the initial state after 100 heating cycles. It can be found through experimental comparison that if the copper is used as a coating metal material, it has better stability and repeatability than other metals, which may be related to the conductivity and the self-healing ability of the copper. Next, the copper has higher stability than the aluminum and the iron, and is not easily oxidized.

[0089] The PTC tracing belt before and after the above stability test is electrically heated, and an upper temperature limit of its temperature self-regulating is tested. Following data are obtained:

Group	0 cycle (°C)	10 cycles (°C)	50 cycles (°C)	100 cycles (°C)
Embodiment 1	142	141	140	140
Embodiment 2	143	142	141	140
Embodiment 3	145	143	142	142
Embodiment 4	148	146	146	146
Embodiment 5	152	151	150	150
Embodiment 6	142	142	141	141
Embodiment 7	142	142	142	142
Embodiment 8	137	132	120	103
Embodiment 9	135	130	127	124
Embodiment 10	136	132	129	128
Embodiment 11	145	144	144	143
Embodiment 12	146	145	144	144
Embodiment 13	146	145	143	143

Data from comparative examples are as follows:

Group	0 cycle (°C)	10 cycles (°C)	50 cycles (°C)	100 cycles (°C)

Comparative Example 1	88	65	52	50
Comparative Example 2	-	-	-	-
Comparative Example 3	136	134	131	128
Comparative Example 4	142	142	141	141
Comparative Example 5	133	124	110	96
Comparative Example 6	52	51	43	40

[0090] It can be seen from the heating performance test that when a type and an amount of the whisker are within a preferred range of the present invention, and the preparation conditions of the whisker are within the preferred range, the obtained PTC tracing belt shows good heating stability; whereas if the amount of the whisker is too small or the amount of the surface coating is weakly low, or if the tetrapod-like zinc oxide whisker coated with the metal film layer is not used, the corresponding PTC tracing belt is weakened in heating performance sharply, and does not have good reproducibility at the same time. In the above Comparative Example 2, the tracing belt is overheated and subjected to wire burning after energization, and the temperature self-regulating effect cannot be achieved. Therefore, there is no data record.

The foregoing is only preferred exemplary implementations of the present invention and is not intended to be limiting of the present invention, and any modifications, equivalent substitutions, improvements and the like within the spirit and principles of the present invention are intended to be embraced by the protection range of the present invention.

Claims

1. A manufacturing method for a PTC tracing belt, comprising the following steps: mixing a molten polymer with a surface coated and modified tetrapod-like zinc oxide whisker, and then performing blending; then co-extruding a mixture and two parallel core wires to coat conductive plastic layers between outer layers of the two core wires and the two core wires; and performing cooling and curing formation to obtain the PTC tracing belt, where a mass ratio of the polymer to the surface coated and modified tetrapod-like zinc oxide whisker is 100 to (5-15). A preparation method for the surface coated and modified tetrapod-like zinc oxide whisker includes: depositing a metal film layer on a surface of a tetrapod-like zinc oxide whisker by means of a physical vapor deposition method.

2. The manufacturing method for the PTC tracing belt according to claim 1, wherein the metal film layer deposited on the surface of the surface coated and modified tetrapod-like zinc oxide whisker is a copper film.

3. The manufacturing method for the PTC tracing belt according to claim 2, wherein for the surface coated and modified tetrapod-like zinc oxide whisker, a mass ratio of a zinc oxide whisker to metal copper is 1 to (0.001-0.01).

4. The manufacturing method for the PTC tracing belt according to claim 1, wherein the surface coated and modified tetrapod-like zinc oxide whisker has a diameter of 0.5-5 μm and a length of 10-50 μm.

5. The manufacturing method for the PTC tracing belt according to claim 1, wherein the polymer is one of polyethylene, polypropylene and polyvinylidene fluoride.

6. The manufacturing method for the PTC tracing belt according to claim 1, before depositing the metal film layer on the surface of the tetrapod-like zinc oxide whisker, further comprising: washing the tetrapod-like zinc oxide whisker with hydrochloric acid.

7. The manufacturing method for the PTC tracing belt according to claim 6, after washing the tetrapod-like zinc oxide whisker with the hydrochloric acid, and before depositing the metal film layer on the surface of the tetrapod-like zinc oxide whisker, further comprising: washing the tetrapod-like zinc oxide whisker in a plasma atmosphere.

8. The manufacturing method for the PTC tracing belt according to claim 1, in a case of mixing a molten polymer with a surface coated and modified tetrapod-like zinc oxide whisker, and then performing blending, further comprising: adding carbon black, wherein a mass ratio of the polymer to the carbon black is 100 to (2-5).

9. A PTC tracing belt manufactured using the manufacturing method for the PTC tracing belt according to any one of claims 1 to 8.

Revendications

1. Un procédé de fabrication d'une bande chauffante PTC, caractérisé en ce qu'il comprend les étapes suivantes :

mélanger et compacter un polymère fondu avec des cristaux de zinc oxyde modifiés en forme de quatre aiguilles, puis coextruder avec deux fils conducteurs parallèles de manière à ce que la couche externe des deux fils soit enveloppée entre les deux fils par une couche de plastique conducteur, refroidir et solidifier pour obtenir la bande chauffante PTC ; le rapport de masse entre le polymère et les cristaux de zinc oxyde modifiés en forme de quatre aiguilles est de 100: (5-15) ; le procédé de préparation des cristaux de zinc oxyde modifiés en forme de quatre aiguilles comprend le dépôt d'une couche de film métallique sur la surface des cristaux de zinc oxyde modifiés en forme de quatre aiguilles par dépôt physique en phase vapeur.

2. Le procédé de fabrication de la bande chauffante PTC selon la revendication 1, caractérisé en ce que la couche de film métallique déposée sur la surface des cristaux de zinc oxyde modifiés en forme de quatre aiguilles est une couche de cuivre.

3. Le procédé de fabrication de la bande chauffante PTC selon la revendication 2, caractérisé en ce que le rapport de masse entre les cristaux de zinc oxyde et le cuivre dans les cristaux de zinc oxyde modifiés en forme de quatre aiguilles est de 1: (0,001-0,01).

4. Le procédé de fabrication de la bande chauffante PTC selon la revendication 1, caractérisé en ce que le diamètre des cristaux de zinc oxyde modifiés en forme de quatre aiguilles est de 0,5 à 5 μm et leur longueur est de 10 à 50 μm.

5. Le procédé de fabrication de la bande chauffante PTC selon la revendication 1, caractérisé en ce que le polymère est du polyéthylène, du polypropylène ou du polyfluorure de vinylidène.

6. Le procédé de fabrication de la bande chauffante PTC selon la revendication 1, caractérisé en ce que, avant le dépôt de la couche de film métallique sur la surface des cristaux de zinc oxyde modifiés en forme de quatre aiguilles, ceux-ci sont nettoyés à l'acide chlorhydrique.

7. Le procédé de fabrication de la bande chauffante PTC selon la revendication 6, caractérisé en ce qu'après le nettoyage des cristaux de zinc oxyde à l'acide chlorhydrique, avant le dépôt de la couche de film métallique sur la surface des cristaux de zinc oxyde modifiés en forme de quatre aiguilles, ceux-ci sont également nettoyés dans une atmosphère de plasma.

8. Le procédé de fabrication de la bande chauffante PTC selon la revendication 1, caractérisé en ce que lors du mélange et du compactage du polymère fondu avec les cristaux de zinc oxyde modifiés en forme de quatre aiguilles, on ajoute également du noir de carbone, le rapport de masse entre le polymère et le noir de carbone étant de 100: (2-5).

9. Une bande chauffante PTC obtenue par le procédé de fabrication de l'une quelconque des revendications 1 à 8.