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(54) Title: A PROCESS FOR PREPARING A DISENTANGLED UHMWPE PRODUCT

(57) Abstract: A process for preparing a disentangled ultra-high molecular weight polyethylene product having a density not less than 1 g/cc is provided. The polyethylene product is prepared in the form of a sheet or a block by solid state compaction by a low flow process such as compression molding exhibiting a density not less than 1 g/cc. The product formed can further be hot stretched below the melting range of the polymeric material, to form films, tapes or fibers. The stretched films, tapes and fibers obtained have a high crystallization temperature and superior thermal properties with high strength and modulus, suitable for ballistic applications.

A PROCESS FOR PREPARING A DISENTANGLED UHMWPE PRODUCT

FIELD

A process for preparing a disentangled ultra-high molecular weight polyethylene product is provided herein. The product has density not less than 1g/cc. The present disclosure further relates to the use of articles obtained from the polyethylene product for ballistic applications.

DEFINITIONS OF TERMS USED IN THE SPECIFICATION

The term "disentangled ultra-high molecular weight polyethylene" used in the context of the present disclosure refers to a homo-polymer or copolymer of ethylene having molar mass in the range of 2M to 15M, wherein the polyethylene chains have low entanglement or are completely disentangled.

BACKGROUND

Ultra-High Molecular Weight Polyethylene (UHMWPE) is a linear homopolymer produced by the polymerization of ethylene with a Ziegler-Natta catalyst. This class of linear polyethylene is termed as "ultra-high" because its average molecular weight is 10-100 times greater than the regular polyethylene especially high density polyethylene (HDPE). Ultra-high molecular weight polyethylene (UHMWPE) in the form of fiber, solution or powder is used to prepare ballistic resistant articles because of its high resistance to impact. However, due to its processing difficulty the full potential of UHMWPE is not explored. For example, UHMWPE has a melt viscosity in the region of 10^{10} Pa.s and therefore cannot be processed by shear methods, as shear methods have limitation of processing polymers having melt viscosity in the range of 10^4 to 10^6 Pa.s.

Low flow processes such as sintering are known for processing UHMWPE. In a typical sintering process, UHMWPE powder is heated above its melting temperature i.e., 180-220° C and fused by compacting at high pressure i.e., 3-5 MPa to obtain a densely packed structure.

In another type of sintering process which is known as direct compression molding (DCM) process, polymer particles are compacted and fused in two steps. UHMWPE powder is initially cold compacted into a green preform and then transferred to an oven where it is heated above the melting temperature of UHMWPE and pressurized to obtain a fused block.

The drawback associated with these types of sintering processes is that pressure cannot be applied uniformly thereby limiting these techniques to prepare dimensionally symmetrical products.

Prior arts have suggested various methods for processing UHMWPE to obtain polyethylene product for ballistic applications.

For instance, UHMWPE is dissolved in a suitable solvent such as decalin or paraffin in order to disentangle the molecular network followed by removing the solvent to obtain UHMWPE in the form of a film or a tape. This method is difficult to execute and is expensive as it requires capital expenditure and high amount of solvent. Further, the solvent content in the resulting UHMWPE product cannot be reduced below 0.1 %.

To overcome the disadvantages associated with the method involving the use of solvent, some of the prior arts suggest solvent free processes. For instance, in one prior art plurality of strips of UHMWPE are aligned parallely adjacent to each other and compressed to obtain bilayered UHMWPE.

In another prior art, the blocks obtained by pressing UHMWPE at a temperature of 100° C to 130° C and at a pressure of 20 kg/cm^2 are skived to obtain film or tape. The film or tape thus obtained is drawn at different temperatures varying from 136° C to 153° C in four stages. However, the polyethylene product obtained by this process exhibit density value less than 1 g/cc, making them less suitable choice for ballistic applications.

A process for preparing polyethylene films having density not less than 1 g/cc is disclosed in one of the prior arts. However, the polyethylene obtained by this method is unsuitable for ballistic applications as it degrades in water or sunlight due to the presence of various fillers.

In view of the above, the inventors of the present invention envisage a simple and cost effective *in-situ* process for preparing a polyethylene product from disentangled ultrahigh molecular weight polyethylene powder which on subjecting to low flow and high compacting process gives a product having density not less than 1 g/cc and exhibit enhanced crystallinity and thermo-mechanical properties for ballistic applications.

OBJECTS

Some of the objects of the present disclosure, which at least one embodiment herein satisfies, are as follows:

An object of the present disclosure is to provide a process for the preparation of a disentangled ultra-high molecular weight polyethylene product having density not less than 1 g/cc.

Another object of the present disclosure is to provide a process for the preparation of a polyethylene product having enhanced crystallinity and thermo-mechanical properties.

Still another object of the present disclosure is to provide a disentangled ultra-high molecular weight polyethylene product.

Yet another object of the present disclosure is to prepare articles from a disentangled ultra-high molecular weight polyethylene product which are suitable for ballistic applications.

Other objects and advantages of the present disclosure will be more apparent from the following description, which are not intended to limit the scope of the present disclosure.

SUMMARY

A process for preparing a disentangled ultra-high molecular weight polyethylene product is provided. This product has a density not less than 1 g/cc and enhanced crystallinity and thermo-mechanical properties.

A positive mold of a compression molding machine is first preheated, followed by introducing disentangled ultra-high molecular weight polyethylene material into the preheated mold. The preheated material in contact with the preheated mold is then brought to room temperature. The material is then maintained at a predetermined temperature and then pressure is increased in a stepwise manner on the mold for different intervals of time to compact and fuse the polymeric material. The product, obtained at a temperature above room temperature, is allowed to be cooled to obtain a compacted and fused product which is in the form of a block or a sheet, having a density not less than 1 g/cc.

The polyethylene product obtained in the form of sheet may further be stretched below its melt temperature in an environmental chamber, into highly oriented tapes, films and fibers having high thermal and mechanical properties.

The disentangled UHMWPE material used in the process can be a homopolymer or a co-polymer, with additives or without any additives.

The disentangled UHMWPE product was characterized by measuring their physical properties (density, thickness), thermal properties (melting and crystallization temperatures, degradation temperatures) and mechanical properties (tensile strength and modulus). The product in the form of sheet, block, film, tape or fibers may be used for ballistic or any other suitable application.

BRIEF DESCRIPTION OF THE ACCOMPANYING DRAWINGS

The disclosure will now be explained in relation to the non-limiting accompanying drawing, in which:

Figure 1 illustrates DSC thermograms showing multiple melting peaks of stretched fibers obtained by stretching polyethylene product obtained in Example 4.

DETAILED DESCRIPTION

A process for preparing a disentangled ultra-high molecular weight polyethylene product is provided. This product has a density not less than 1 g/cc and enhanced crystallinity and thermo-mechanical properties.

The process for the preparation of the polyethylene product is described hereinafter.

Initially, a positive mold having a cavity and a top plate is heated inside a compression molding machine at a temperature ranging from 80° C to 120° C, particularly at 100° C. The cavity of the heated mold is then filled with disentangled UHMWPE. The preheated material in contact with the preheated mold is then brought back to room temperature. The compaction of disentangled UHMWPE material is carried out below the melt temperature in a solid state at a temperature ranging from 23° C to below the melting range of the material, preferably below 130° C and at a pressure ranging from 10 kg/cm^2 to 400 kg/cm^2 in a compressing molding machine for a time period ranging from 0.5 hour to 10 hours to obtain a compacted and a highly fused disentangled UHMWPE product without grain boundaries. The product, if obtained at a temperature above room temperature, is allowed to be cooled for a time period in the range of 10 min to 12 hours, to obtain a compacted and fused product which is in the form of a block or a sheet, having a density not less than 1 g/cc. The mold is then depressurized to recover the product. During the experimentation the inventors found that the desired results could be achieved only if the compacting is carried out at a temperature below melting temperature of the disentangled UHMWPE by varying pressure and temperature in a step wise manner.

The pressure applied for compacting disentangled UHMWPE may be gradually increased step wise at fixed temperature for different intervals of time. In an exemplary embodiment of the present disclosure the method of compacting may be carried out at a temperature of 125° C and applying a pressure in the following manner:

- 10 kg/cm^2 for 5 minutes,
- 50 kg/cm^2 for 5 minutes,
- 100 kg/cm^2 for 10 minutes,
- 150 kg/cm^2 for 20 minutes, and
- 180 kg/cm^2 for 60 minutes.

The total time period of the compaction or application of pressure is in the range of 0.5 hours to 10 hours.

The mold with compacted disentangled UHMWPE is then cooled to 23° C to obtain a polyethylene product which is in the form of a block or a sheet, having a density not less than 1 g/cc. The cooling is carried out by circulating water or air or chilled water. To achieve the desired quality of the resulting polyethylene product the cooling is carried out for a time period ranging from 10 minutes to 12 hours. In an exemplary embodiment, the cooling step includes gradually cooling up to 80° C followed by sudden cooling to a temperature in the range of 20 to 30° C. After cooling the compacted material, the mold is then depressurized to recover the product.

The disentangled UHMWPE product obtained in form of the sheet may further be converted into a shape selected from the group consisting of tape, film and fiber by way of stretching. In an embodiment, the stretching is carried out in a first stage at a temperature T_1 and a second stage at a temperature T_2, wherein T1 and T2 are in the range of 20° C to below the melt temperature of said product and the relation between T_1 and T_2 is one selected from the group consisting of $T_1 = T_2$ and $T_1 \neq T_2$. In an exemplary embodiment, the temperatures T_1 and T_2 are equal to 125° C.

In another embodiment, the temperatures T_1 and T_2 are 125°C and 137°C respectively. Due to stretching beyond a certain limit, the orthorhombic crystalline state of the polyethylene product gets converted into hexagonal state resulting into the lowering of the density to less than 1 g/cc. Therefore, the stretching may be varied depending on the end application of the resulting tape, film or sheet or fiber.

Depending upon the end application of the resulting polyethylene product, the molecular weight of the disentangled UHMWPE used for the preparation of the polyethylene product may be varied between 2 M and 15M, preferably in the range of 2M and 13M and more preferably in the range of 2M and 12.3 M. The average particle size of the disentangled UHMWPE may range from 25 to 800 microns, preferably from 50 to 600 microns, more preferably from 200 to 450 microns. Further, the disentangled UHMWPE used for the purpose has very low bulk density and high crystallinity.

The disentangled UHMWPE used is a homopolymer; however, a co-polymer or a combination can also be used.

In an embodiment of the present disclosure, the disentangled UHMWPE is a co-polymer of ethylene with an alpha-olefin of C_3 to C_8.

Though the disentangled UHMWPE is essentially devoid of any additives, primary and/or secondary antioxidants may be uniformly mixed with the disentangled UHMWPE before processing to prevent thermo-oxidative degradation. In addition to the antioxidants, other additives such as curing agents, plasticizers, modifiers, stabilizers, surfactants, binders and the like may also be uniformly mixed with disentangled UHMWPE before subjecting it to processing. The disentangled UHMWPE can be in any form which includes but is not limited to a granule and a powder.

It was observed, without binding to the theory, that when the disentangled polyethylene is processed under the conditions of the present disclosure a very high level of compaction is attained and the crystallites in the resulting polyethylene product are initially predominantly orthorhombic crystalline phase which later transformed into a hexagonal one during stretching into tape or fibre as observed by increase in heat of fusion values and fusion (melting) peak temperature through differential scanning calorimetry. The initial predominant orthorhombic nature of the crystallites has a significant effect on the density of the polyethylene product. Thus, the density of the polyethylene product is dependent on the extent of formation of orthorhombic crystallites in the polyethylene product which in turn is dependent on the processing parameters. In other words, the density of polyethylene product is dependent on the processing parameters, catalytic residue in the polymer powder and is independent of other factors such as molecular weight, average particle size and bulk density of the disentangled UHMWPE. In an exemplary embodiment of the present disclosure the density of the polyethylene product obtained by using disentangled UHMWPE having molecular weight of 3.53M is substantially equal to that of the density of the polyethylene product obtained by using disentangled UHMWPE having molecular weight of 9.4M.

The polyethylene product obtained by the process of the present disclosure is characterized for physical properties such as density and thickness, thermal properties such as melting temperature, crystallization temperature and degradation temperatures and mechanical properties such as tensile strength and tensile modulus.

In an embodiment, the characterization observed was that the melting temperature of the stretched products is higher than that of the powder form of the disentangled UHMWPE. The melting temperature for powder form of the disentangled UHMWPE is 140°C whereas the melting temperature of the resulting sheet, film and fiber obtained from the powder form of disentangled UHMWPE is 143° C, 144° C and 160°C respectively. This means that the molecules in the polymer get highly compacted during molding process and get highly oriented during stretching process and attain crystal perfection state indicating the transformation from the orthorhombic crystalline state into the hexagonal crystalline state. It is due to the transformation from one state to another state the density values for stretched products decrease.

The stretched products like tape, films and fibres showed efficient orientation as indicated by multiple splitting of melting endotherm (146° C, 154° C and 159° C) (**Figure 1**) with very high melting temperature of 160° C, crystallization temperature of 121°C to 140°C, high crystallinity and tensile modulus of 155 GPa. The orientation of the molecules in the stretched product is dependent on the number of stretching, type of stretching and temperature applied for stretching.

In another aspect of the present disclosure, there is provided a polyethylene product characterized by at least one of the following properties:

- having density not less than 1 g/cc at 23° C;
- having 2θ values around 22 and 24 at 23° C;
- having crystalline structure;
- melting temperature the range of 140° C to 160° C;
- crystallization temperature the range of 117° C to 140° C;
- degradation temperature (@ 50% weight loss) is in the range of 471° C to 478° C;
- tensile strength in the range of 1.01 GPa to 1.66 GPa;
- tensile modulus in the range of 74 GPa to 97 GPa;
- Heat of fusion from 190 to 230 J/g;
- DSC crystallinity from 64 to 77 %; and
- X – ray crystallinity from 90 to 94%.

In yet another aspect of the present disclosure, the polyethylene product may be used for preparing articles for ballistic or any other suitable application.

The present disclosure is further described in light of the following non-limiting examples which are set forth for illustration purpose only and not to be construed for limiting the scope of the disclosure.

Examples

Example 1: Processing of Disentangled UHMWPE having molecular weight of 3.53M

A positive mold was preheated to 100° C for 10 min by keeping it inside a compression molding machine. After heating, the mold was taken out of the compression molding machine and allowed to be cooled to room temperature. The mold cavity was then filled with 10 g of disentangled UHMWPE powder having molecular weight of 3.53M, bulk density of 0.054 g/cc and average particle size of 227 microns. The mold was then closed by placing top plate and placed in the compression molding machine which was maintained at a temperature of 125° C. Compression molding was carried out using the following molding conditions: Pressure 10kg/cm^2 for 5 min, 50 kg/cm^2 for 5 min, 100 kg/cm^2 for 5 min, 150 kg/cm^2 for 5 min, 180 kg/cm^2 for 70 min. After the compaction, the temperature of the mold was brought down to 80° C by natural cooling followed by quenching the mold in water kept at room temperature. The mold was depressurized to obtain a molded disentangled ultra-high molecular weight polyethylene product. The molded polyethylene product (DPE Sheet – 1) showed a density of 1.00170 g/cc and was seen sinking in water. The physical and thermal properties are depicted in **Tables 1, 2 and 4**.

Example 2: Processing of Disentangled UHMWPE having molecular weight of 4.9M

Solid state processing of disentangled UHMWPE having molecular weight of 4.9M was carried out in a similar manner as Example 1, The temperature, however, was maintained at 128° C and the pressure at the last stage was maintained at 175 kg/cm^2 and the time period for which this pressure was maintained was 90 min. The mold was cooled to room temperature by circulating water at room temperature. The polyethylene product (DPE Sheet – 2) obtained showed a density of 1.02778 g/cc. The sample was seen sinking in water. The physical and thermal properties are depicted in **Tables 1 – 4**.

Example 3: Processing of Disentangled UHMWPE having molecular weight of 12.1M

Solid state processing of disentangled UHMWPE having molecular weight of 12.1M was carried out in a similar manner as Example 1. The pressure of 180 kg/cm^2 at the last step was, however, maintained for a time period of 110 minutes. The mold was cooled to room temperature by circulating water at room temperature and depressurized. The polyethylene product (DPE Sheet–3) obtained showed a density of 1.03035 g/cc and was seen sinking in water. The physical and thermal properties are depicted in **Tables 1, 2 and 4**.

Example 4: Processing of Disentangled UHMWPE having molecular weight of 12.3M

Solid state processing of disentangled UHMWPE having molecular weight of 12.3M was carried out in a similar manner as Example 1 at a temperature of 125° C. The pressure of 175 kg/cm^2 at the last step was, however, maintained for a time period of 100 minutes. The mold was cooled by natural cooling by air for 12 hours followed by depressurizing the mold. The polyethylene product (DPE Sheet-4) obtained showed a density of 1.00210 g/cc and was seen sinking in water. The physical and thermal properties are depicted in **Tables 1- 4** and **Figure 1**.

Example 5: Processing of Disentangled UHMWPE having molecular weight of 5.4M at high pressure and room temperature

10 g of disentangled UHMWPE having molecular weight of 5.4M was compression molded under the conditions of 372 kg/cm^2 pressure, 23° C temperature into pellets having 10.3 mm diameter and 1.5 – 6 mm thickness using a mold pressing and tool providing positive pressure for 10 min. The mold was then depressurized to obtain the resultant molded product which was found to have the characteristics of sinking in water i.e. indicating a density of >1 g/cc.

Example 6: Hot stretching of the polyethylene product

The polyethylene products obtained from disentangled UHMWPE having different molecular weights (3.53M to 12.3M) were hot stretched in two stages with T_1 = 90° C and T_2 = 137° C in an environmental chamber of Universal Testing Machine. The stretching was carried out using strips cut from the round molded sheets obtained by compression molding as in Example 1. The stretching speed was kept as 20mm/min. The samples were stretched up to 150 times. The mechanical and thermal properties are given in Tables 2 and 4.

Comparative Example 1: Processing of entangled UHMWPE having molecular weight of 3.5M

10 g of entangled UHMWPE having 3.5M molecular weight was compression molded at a temperature of 150° C with a process cycle of 30 min to obtain polyethylene product by applying the following pressure condition. Pressure used is 10 kg/cm^2 for 5 min, 50 kg/cm^2 for 5 min, 100 kg/cm^2 for 10 min and 175 kg/cm^2 for 10 min. The polyethylene product (UHM Sheet – 1) showed a density of 0.96059 g/cc.

The physical and thermal properties of the obtained sheets from Examples 1-6 and Comparative Example 1 are compared between in Tables 1 – 4.

Table 1: Physical characteristics of polyethylene products

Polyethylene products obtained in	MW (M)	#Al / Ti Ratio	Bulk density (g/cc)	Density (g/cc) @ 23° C	Percent Crystallinity	
					DSC	X - Ray
Example – 1	3.53	641	0.059	1.0017	64	90
Example – 2	4.90	863	0.064	1.02778	65	93
Example – 3	12.10	919	0.062	1.03035	68	94
Example – 4	12.30	948	0.060	1.00210	77	90
Comparative Example – 1	3.5	-	-	0.96051	54	85

#: By incineration & gravimetric analysis

Table 2: Thermal properties of polyethylene products

Polyethylene products obtained in		Melting Temperatures (Tm)					Crystallization Temperature (Tc)
		1st heating (Tm_1)			2nd heating (Tm_2)		
	MW (M)	Tm_1 (°C)	ΔH (J/g)	C[*] (%)	Tm_2 (°C)	ΔH (J/g)	Tc (°C)
Example -1 / DPE Sheet -1	3.53	142	188	64	132	82	117
Example 2 / DPE Sheet -2	4.9	142	190	65	133	76	118
Sheet -2 stretched for 2 times @ 125°C into fiber	4.9	[#]147 / 154	189	65	133	68	[#]123 / 140
Example 3 / DPE Sheet -3	12.1	140	219	75	132	76	117
Sheet – 3 stretched for 2 times @ 125°C into fiber	12.1	[#]146,154, 159	225	77	134	61	[#]124, 140
Example 4 / DPE Sheet -4	12.3	142	224	77	132	76	119
Sheet -4 stretched 2 times @ 137°C into fiber	12.3	[#]145, 153, 159	230	79	133	65	[#]126, 140
Comparative Example - 1	3.5	141	159	54	131	92	119

[#] Multiple peaks

[*] based on 100 % crystalline PE having heat of fusion value of 293 J/g.

Table 3: Thermo-gravimetric analysis of Polyethylene products

Polyethylene products obtained in	MW (M)	Decomposition Temperatures Weight Loss (%)			Degradation Temp @ 50% Weight loss
		400° C	500° C	600° C	
Example 2 / DPE Sheet -2	4.9	3.6	94.5	94.6	471
Example 4 / DPE Sheet -4	12.3	2.2	97.2	97.3	478
Comparative Example 1	3.5	92	98.8	100	434

Table 4: Mechanical properties of stretched Disentangled polyethylene tape

Disentangled polyethylene tape obtained from polyethylene product of	MW (M)	Tensile Modulus$^+$ (GPa)		Tensile Strength$^+$ (GPa)	
		125° C (Two stage)	137° C (Two stage)	125° C (Two stage)	137° C (Two Stage)
Example 1 /DPE Sheet - 1	3.53	73.85	48.37	1.01	1.15
Example 2 /DPE Sheet - 2	4.9	82.33	37.69	1.17	1.03
Example -3 /DPE Sheet - 3	12.1	53.24	54.78	1.10	1.22
Example – 4 /DPE Sheet - 4	12.3	96.99	58.26	1.66	1.51
Comparative Example 1$^@$	3.5	0.64	0.32	0.188	0.17

$^+$ Two stage stretch at different temperatures @ 125° C and 137° C
@Sheets made at 150° C.

Evaluation of processed and stretched polyethylene products was carried out in the following manner:-

Density

Density of disentangled ultra-high molecular weight polyethylene sheets, stretched tape, films / block was measured as per ASTM D-792 using Mettler Toledo unit @ 23° C in butyl acetate.

X – Ray Analysis

X – Ray analysis of polymer powder, sheets and stretched products were carried out by using Kα radiation in the range of 2 - 80 2θ to measure their % crystalline & amorphous content on D8 Advance Powder Diffractometer (M/s Bruker Axes, Germany)

Nucleating efficiency

Disentangled UHMWPE powder, polyethylene products and stretched products of varying molecular weights were subjected to nucleating efficiency test using

Differential Scanning Calorimeter (Q2000 MDSC from M/s. TA instruments). The polymers were heated from ambient to 200° C in N_2 atmosphere with a heating rate of 10° C / min, held at 200° C and cooled to room temperature at the same rate. Melting temperature (T_m), Crystallization temperature (T_c), Heat of fusion during melting (ΔHTm) and cooling (ΔHTc) were recorded. The efficiency of various polyethylene products was evaluated by measuring their Tm, Tc and ΔT and provided in Table 2.

Thermo-gravimetric analysis

Thermo-gravimetric analysis (TGA) of Disentangled UHMWPE powder, polyethylene product and stretched products was carried out from ambient to 600° C with heating rate of 10° C / min under nitrogen atmosphere using TGA500 (M/s TA Instruments, USA). DUHMWPE molded products showed higher thermal stability as given in Table 3.

Tensile Properties

Tensile properties of the Disentangled UHMWPE samples were measured on universal testing machine - UTM 3366 (M/s Intron Ltd) as per ASTM D-882. The test temperature was maintained as 23° C. The initial distance between the grips (gauge length) was maintained as 15mm. The rate of grip separation was maintained as 50 mm/min. Pneumatic grips with serrated jaw were employed for the test.

Throughout this specification the word “comprise”, or variations such as “comprises” or “comprising”, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the disclosure to achieve one or more of the desired objects or results.

Any discussion of documents, acts, materials, devices, articles or the like that has been included in this specification is solely for the purpose of providing a context for the disclosure. It is not to be taken as an admission that any or all of these matters form a part of the prior art base or were common general knowledge in the field relevant to the disclosure as it existed anywhere before the priority date of this application.

The numerical values mentioned for the various physical parameters, dimensions or quantities are only approximations and it is envisaged that the values higher/lower than the numerical values assigned to the parameters, dimensions or quantities fall within the scope of the disclosure, unless there is a statement in the specification specific to the contrary.

While considerable emphasis has been placed herein on the specific features of the preferred embodiment, it will be appreciated that many additional features can be added and that many changes can be made in the preferred embodiment without departing from the principles of the disclosure. These and other changes in the preferred embodiment of the disclosure will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

CLAIMS:

1. A process for preparing a disentangled ultra-high molecular weight polyethylene product having a density not less than 1 g/cc, said process comprising the following steps:
   (a) compressing in a mold, disentangled ultra-high molecular weight polyethylene material, at a predetermined pressure and a predetermined temperature, said predetermined pressure being attained by applying pressure in a stepwise manner and maintaining the pressure at each step for a predetermined time period; and
   (b) optionally cooling the compacted material in the mold and depressurizing said mold to obtain a disentangled ultra-high molecular weight polyethylene product having density not less than 1 g/cc.

2. The process as claimed in claim 1, wherein the pressure is applied in the range of 10 kg/cm^2 to 400 kg/cm^2 and the temperature is configured to be above 20° C and below the melt temperature of said material.

3. The process as claimed in claim 1, wherein the process includes increasing the temperature to above 80° C during compression and gradually cooling up to 80° C and sudden cooling to a temperature in the range of 20 to 30° C.

4. The process as claimed in claim 3, wherein the cooling is carried out by at least one fluid selected from the group consisting of water and air.

5. The process as claimed in claim 1, wherein the total time period of step (a) is in the range of 0.5 hours to 10 hours.

6. A process for converting the form of said disentangled ultra-high molecular weight polyethylene product obtained from the process as claimed in claim 1, to a

form selected from the group consisting of tape, film and fiber, said process for converting comprises a step of stretching said product.

7. The process as claimed in claim 6, wherein said process of stretching is carried out in a first stage at a temperature T_1 and a second stage at a temperature T_2, wherein T_1 and T_2 are in the range of 20° C to below the melt temperature of said product and the relation between T_1 and T_2 is one selected from the group consisting of $T_1 = T_2$ and $T_1 \neq T_2$.

8. The process as claimed in claim 7, wherein T_1 and T_2 are equal to 125° C.

9. The process as claimed in claim 1, wherein the molecular weight of said disentangled ultra-high molecular weight polyethylene material is in the range of 2M to 15M and the average particle size of said material is in the range of 25 microns to 800 microns.

10. The process as claimed in claim 1, wherein said disentangled ultra-high molecular weight polyethylene material is selected from the group consisting of disentangled ultra-high molecular weight polyethylene homopolymer, disentangled ultra-high molecular weight polyethylene copolymer and combinations thereof.

11. The process as claimed in claim 10, wherein said copolymer is a copolymer of ethylene with a C_3-C_8 alpha-olefin.

12. A disentangled ultra-high molecular weight polyethylene product, prepared by the process as claimed in claim 1, said product characterized by at least one of the following properties:

    - density not less than 1 g/cc at 23° C;
    - 2θ values around 22 and 24 at 23° C;
    - a melting temperature in the range of 140° C to 160° C;
    - a crystallization temperature in the range of 117° C to 140° C;

- a degradation temperature (at 50% weight loss) in the range of 471° C to 478° C;
- tensile strength in the range of 1.01 GPa to 1.66 GPa;
- tensile modulus in the range of 74 GPa to 97 GPa;
- Heat of fusion in the range of 190 J/g to 230 J/g;
- DSC crystallinity in the range of 64 to 77%; and
- X – ray crystallinity in the range of 90 to 94%.

13. The product as claimed in claim 12, wherein said product is selected from a group consisting of disentangled ultra-high molecular weight polyethylene homopolymer product, disentangled ultra-high molecular weight polyethylene copolymer product and combinations thereof.

14. A stretched disentangled ultra-high molecular weight polyethylene product in the form of one selected from the group consisting of tape, film and fiber, prepared by the process as claimed in claim 6.

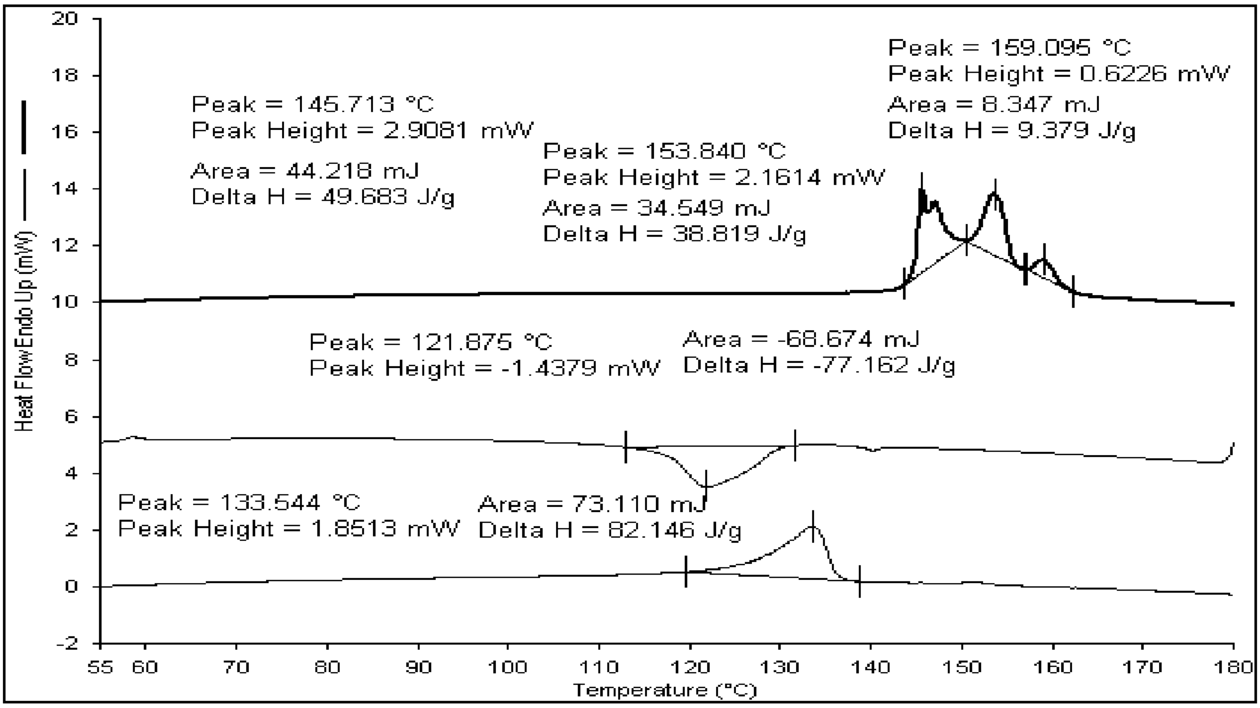
Heat Flow Endo Up (mW)
Temperature (°C)
20
18
16
14
12
10
8
6
4
2
0
-2
55
60
70
80
90
100
110
120
130
140
150
160
170
180
Peak = 145.713 °C
Peak Height = 2.9081 mW
Area = 44.218 mJ
Delta H = 49.683 J/g
Peak = 153.840 °C
Peak Height = 2.1614 mW
Area = 34.549 mJ
Delta H = 38.819 J/g
Peak = 159.095 °C
Peak Height = 0.6226 mW
Area = 8.347 mJ
Delta H = 9.379 J/g
Peak = 121.875 °C
Peak Height = -1.4379 mW
Area = -68.674 mJ
Delta H = -77.162 J/g
Peak = 133.544 °C
Peak Height = 1.8513 mW
Area = 73.110 mJ
Delta H = 82.146 J/g

FIGURE 1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2015/051845

A. CLASSIFICATION OF SUBJECT MATTER
C08F10/00,B29C55/00 Version=2014.01

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B29C, C08F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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

STN, IPO Internal Database

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 2009007045 A1 (TEIJIN ARAMID BVET AL), 15 Jan.,2009 (2009-01-15) Whole document	1-14
Y	RASTOGI, Sanjay et al, "Unprecedented High-Modulus High-Strength Tapes and Films of Ultrahigh Molecular Weight Polyethylene via Solvent-Free Route", Macromolecules, 2011, 44 (14), pp 5558-5568 (2011), DOI: 10.1021/ma200667m Whole document	1-14

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search 28-05-2015	Date of mailing of the international search report 28-05-2015
Name and mailing address of the ISA/ Indian Patent Office Plot No.32, Sector 14,Dwarka,New Delhi-110075 Facsimile No.	Authorized officer Ragini Telephone No. +91-1125300200

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/IB2015/051845

Citation	Pub.Date	Family	Pub.Date
WO 2009007045 A1	15-01-2009	EP 2014445 A1	14-01-2009
		US 2010144224 A1	10-06-2010
		RU 2010104433 A	20-08-2011
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