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A request for correction of the originally filed description and claims has been filed pursuant to Rule 88 EPC. A decision on the request will be taken during the proceedings before the Examining Division (Guidelines for Examination in the EPO, A-V, 2.2).

54 **Improvements in and relating to synthetic down.**

57 The present invention relates to a method of forming a thermal insulating material which method comprises
forming a fibre assemblage from 70 to 95 percent by weight of synthetic polymeric microfibrils having a diameter of from 3 to 12 microns; and
from 5 to 30 percent by weight of synthetic polymeric macrofibrils having a diameter not less than 12 microns,
shaping the assemblage so formed,
and effecting bonding between at least some of the fibres at their contact points such that the density of the resultant structure is within the range 3 to 16 kg/m³, the bonding being effected without significant loss to thermal insulating properties compared with the unbonded assemblage.

EP 0 279 677 A2

Description

IMPROVEMENTS IN AND RELATING TO SYNTHETIC DOWN

This invention relates to synthetic down and has particular reference to light-weight thermal insulation systems which can be achieved by the use of fine fibres in low density assemblies.

United States Patent Specification No. 4,588,635 describes and claims a synthetic fibre batt thermal insulator material which comprises a blend of

(a) 80 to 95 weight percent of spun and drawn crimped, staple synthetic polymeric microfibrils having a diameter of from 3 to 12 microns; and

(b) 5 to 20 weight percent of synthetic polymeric staple macrofibrils having a diameter of from more than 12 up to 50 microns, said batt having the following characteristics:

(i) a radiation parameter defined as the intercept on the ordinate axis at zero density of a plot of $K_c P_f$ against P_f less than 0.173 (W/m-K) (kg/m³).

(ii) a density P_f from 3.2 to 9.6 kg/m³ and an apparent thermal conductivity K_c measured by the plate to plate method according to ASTM C518 with heat flow down of less than 0.072 W/m-K. ²

This material approaches, and in some cases exceeds the thermal insulating properties of natural down.

From a mechanical standpoint, it is a matter of experience that extremely fine fibres suffer from deficiencies of rigidity and strength that make them difficult to produce, manipulate and use. Recovery properties of such a synthetic insulator material are enhanced at larger fibre diameters, but an increase in the large fibre component will seriously reduce the thermal insulating properties overall.

The problems associated with mechanical stability of fine fibre assemblies are exacerbated in the wet condition since surface tension forces associated with the presence of capillary water are considerably greater than those due to gravitational forces or other normal use loading and they have a much more deleterious effect on the structure.

According to the present invention there is provided a synthetic fibre thermal insulator material in the form of a cohesive fibre structure, which structure comprises an assemblage of: (a) from 70 to 95 weight percent of synthetic polymeric microfibrils having a diameter of from 3 to 12 microns; and

(b) from 5 to 30 weight percent of synthetic polymeric macrofibrils having a diameter of 12 to 50 microns,

characterised in that at least some of the fibres are bonded at their contact points, the bonding being such that the density of the resultant structure is within the range 3 to 16 kg/m³, the bonding being effected without significant loss of thermal insulating properties of the structure compared with the unbonded assemblage.

The invention also includes a method of forming a thermal insulating material which method comprises forming a fibre assemblage comprising

(a) from 70 to 95 percent by weight of synthetic polymeric microfibrils having a diameter of from 3 to 12 microns; and

(b) from 5 to 30 percent by weight of synthetic polymeric macrofibrils having a diameter not less than 12 microns,

(c) shaping the assemblage so formed, and effecting bonding between at least some of the fibres at their contact points such that the density of the resultant structure is within the range 3 to 16 kg/m³, and

(d) effecting bonding without significant loss of thermal insulating properties compared with the unbonded assemblage.

It is preferred that the resultant fibre assemblage has a radiation parameter defined as the intercept on the ordinate axis at zero density of a plot of $K_c P_f$ against P_f less than 0.173 (W/m-K) (kg/m³) and a density P_f from 3.2 to 9.6 kg/m³ and an apparent thermal conductivity K_c measured by the plate to plate method according to ASTM C518 with a heat flow down of less than 0.072 W/m-K.

Microfibrils and macrofibrils for use in the present invention may be manufactured from polyester, nylon, rayon, acetate, acrylic, modacrylic, polyolefins, spandex, polyaramids, polyimides, fluorocarbons, polybenzimidazoles, polyvinylalcohols, polydiacetylenes, polyetherketones, polyimidazoles and phenylene sulphide polymers such as those commercially available under the trade name RYTON.

In general it is preferred that the microfibrils are drawn following extrusion to impart tensile modulus of at least 63 grams per d.tex.

The bonding may be effected between at least some of the macrofibrils to form a supporting structure for the microfibrils, or may be between both macrofibrils and some of the microfibrils at their various contact points.

The macrofibrils may be selected from the same material and may be either the same as the microfibrils or different.

In one advantageous embodiment of the invention microfibrils are formed from polyethylene terephthalate and the macrofibrils are selected from polyethylene terephthalate or a polyaramid, such for example, as that commercially available under the Trade Mark "Kevlar".

The macrofibrils can be monofibrils, i.e. fibres having a substantially uniform structure or may be multi-component fibres having a moiety to facilitate microfibre to microfibre bonding. The microfibre may be a fibre mixture in which at least 10% by weight comprises macrofibrils of a lower melting point thermoplastic material to assist the microfibre to microfibre bonding. In a further embodiment of the invention the

macrofibres may be a fibre mixture comprising multi-component macrofibres and a monocomponent macrofibre capable of bonding one with the other.

In another embodiment of the present invention the macro component fibre may be a mix or blend of macrofibres having different properties for example, a macro fibre mix may comprise two or more different fibres such as a polyester fibre to give the desired bonding and a "Kevlar" fibre to give stiffness. The proportion of stiffening fibre to bonding fibre may be varied to provide different properties subject to the requirement that the proportion of bondable fibres is sufficient for the macrofibre structure to provide an open support for the microfibres as hereinafter described.

Some materials, such for example as polyphenylene sulphide fibres, aromatic polyamides of the type commercially available under the trade name "APYIEL", and polyimide fibres such as those manufactured by Lenzing AG of Austria, exhibit flame retardant properties or are non-flammable. Such materials can, therefore, confer improved flame or fire resistant properties on manufactured products containing the materials in accordance with the present invention. Methods of manufacturing such fibres are well known, see for example, United States Patent Specification No. 4,148,103.

Useful two component fibres include type TJ04S2, a side-by-side polyester/polyester material and type TJ04C2, a sheath/core polyester/polyester material, both available from Messrs. Teijin Ltd., of Japan.

The bonding in the structures in accordance with the invention is preferably, principally between the fibres of the macrofibre component at their contact points. The purpose of the macrofibre to macrofibre bonding is to form a supporting structure for the microfibre component, said supporting structure contributing significantly to the mechanical properties of the insulating material. By bonding the macrofibres, in accordance with the invention the macrofibres maintain an open bonded fibre structure within which the microfibres can be accommodated.

Any means of bonding between the macrofibres may be employed such, for example, as by the addition of solid, gaseous or liquid bonding agents whether thermoplastic or thermosetting or by the provision of autologous bonds in which the fibres are caused to bond directly through the action of an intermediary chemical or physical agent.

The method of bonding is not critical, subject only to the requirement that the bonding should be carried out under conditions such that the macrofibre component, does not lose its structural integrity. It will be appreciated by one skilled in the art that any bonding will affect the thermal properties adversely; the bonding step needs, therefore, to be conducted to maintain the physical properties and dimensions of the fibre components and the assemblage as much as possible.

The thermal insulating properties of the bonded assemblage are preferably substantially the same as, or not significantly less than, thermal insulating properties of a similar unbonded assemblage.

In a particular embodiment of the present invention bonding within the structure may be effected by heating the assemblage of fibres for a time and at a temperature sufficient to cause the fibres to bond. Such heating period may be at a temperature of from about 125° C (257° F) to 225° C (437° F) for a period of the order of 1 minute to 10 minutes and preferably at a temperature of from about 140° C (284° F) to 200° C (392° F) for a period of about 3 to 7 minutes; these periods are, of course, dependent upon the material the macrofibre component.

The microfibres and optionally also the macrofibres constituting the assemblage of the invention may be crimped to assist in the production of low density intimate blend or assemblage of the two components. Crimping techniques are well known in the art, but the average crimp number for both microfibres and macrofibres is preferably within the range of 3 to 8 crimps/cm (8 to 20 crimps per inch). The presence of crimp further assists re-establishment of loft in the fibre assembly after compression or wetting.

In a preferred embodiment the microfibres may have a tensile modulus of from 36 to 81 gm/dtex (40 to 90 grams/denier). This relatively high tensile modulus contributes to a high bending modulus in the material of the invention and assists with the mechanical performance of the material in accordance with the invention.

In another embodiment of the present invention, lubricants may be included in one or both components of the assemblage. Typical lubricants are aqueous solutions of organopolysiloxanes, emulsions of polytetrafluoroethylene and non-ionic surfactants. Such lubricants may be applied to the fibres by spray or dip techniques well known in the art.

The assemblage of macrofibres and microfibres may be a batt consisting of plied card-laps although other fibrous forms such as air-laid webs are equally suitable. Webs and batts in which some fibres are oriented in the through-the-thickness direction as well as in the primary sheet plane are of distinct advantage from a mechanical performance standpoint. Webs of continuous filaments whether spun, bonded or otherwise produced may be used.

In another embodiment of the invention, the assemblage may be in the form of clusters or balls. Such clusters can be prepared by hand or through the use of commercially available machinery such as automatic dicing, tumbling or ball-rolling machinery. Batts or clusters in accordance with the invention may achieve densities comparable to the densities of natural down, i.e. of the order of less than 16 kg/m³ (1.0 lb/cu ft) and typically about 8 kg/m³ (0.5 lb/cu ft).

In cluster form, the insulator material of this invention surprisingly provides extremely good recovery from compressional loading. Furthermore, since it is compatible with current down processing equipment, it represents a viable synthetic down replacement material both from a performance and a processing standpoint.

Thermal insulating material in accordance with the present invention in the form of clusters tends to enjoy a more random orientation of the fibres. Thus, providing greater compressional recovery and more uniform properties. These clusters furthermore enjoy the advantage of being capable of being handled in established down handling and filling machinery. Such clusters may be made by shaping the fibre assemblage using "cotton ball" rolling machine. Typical machines suitable for this purpose are manufactured by Bodolay/Pratt Division of the Package Machinery Co., of Florida, USA, and by Internationale Verbandstoff-Fabrik of Switzerland.

Following is a description by way of example only of methods of carrying the invention into effect.

In the following examples where reported the following tests were employed:-

Density: The volume of each insulator sample was determined by fixing two planar sample dimensions and then measuring thickness at 0.014 kPa (0.002 lb/in²) pressure. The mass of each sample divided by the volume thus obtained is the basis for density values reported herein.

Thickness was measured at 0.014 kPa (0.002 lb/in²). Apparent thermal conductivity was measured in accord with the plate/sample/plate method described by ASTM Method C518.

Radiation Parameter, C was calculated from the expression:

$$C = k_c P_F - k_a P_F$$

where k_c = apparent thermal conductivity of the material,

P_F = density of the material, and

k_a = the thermal conductivity of still air,

$$= 0.025 \text{ W/m-K (0.175 Btu-in/hr-ft}^2\text{-}^\circ\text{F)}.$$

Compressional Strain: Strain at 34.4 kPa (5lb/in²) , which was the maximum strain in the compressional recovery test sequence, was recorded for each test.

Compressional Recovery and Work of Compression and Recovery:

Section 4.3.2 of Military Specification MIL-B-41826E describes a compressional-recovery test technique for fibrous batting that was adapted for this work. The essential difference between the Military Specification method and the one employed is the lower pressure at which initial thickness and recovered-to-thickness were measured. The measuring pressure in the specification is 0.07 kPa (0.01 lb/in²) whereas kPa (0.02 lb/in²) was used in this work.

Water Absorption Capacity: ASTM Method D1117 provided the starting point for development of the water absorption-capacity and absorption-time test used. However, wetted-sample weighings were made at frequent intervals during the first six hours of immersion and another weighing was made after twenty-four hours (Method D1117 requires only one wetted sample weighing). A unique sample-holder and a repeatable technique for draining excess water prior to each weighing were adopted after some initial experimentation.

Drying Time: After each absorption capacity test, weighings were made at one-half hour intervals as the sample air-dried on a wire rack in a 21°C (70°F), 65% r.h. atmosphere.

Batt Cohesiveness: A 5.1 cm (2 inch) thick, 14.5 cm (5.7 inch) diameter circular test-specimen was cut from each batt. Each specimen was gripped so that it could be pulled apart in the direction perpendicular to the batt plane, i.e. tensile tested in the through-the-thickness direction. Results were recorded in terms of tensile strain at the time of initial batt separation and expressed as extension ratios, which are defined as the ratio of the batt thickness at separation or disruption to the original batt thickness under zero applied load.

Cluster Cohesiveness: Individual clusters weighing 60 mg., and having diameters of 3.05 to 3.15 cm (1.20 to 1.25 inches) were mounted in light-weight. spring-action jaws in a tensile test machine. The jaw faces were lined with rubber and measured 0.64 x 0.64 cm (0.25 x 0.25 inches); they were spaced to provide an initial separation (gauge length) of 1.91 cm (0.75 inch). The maximum force attained as each cluster was drawn apart and fully separated was recorded.

The down used throughout the examples was actually a down/feathers mixture, 80/20 by weight, per MIL-F-43097G, Type II, Class I. This mixture is commonly and commercially referred to as "down" and is referred to as "down" herein.

COMPARATIVE EXAMPLE 1

Consistent with U.S. Patent No. 4,588,635 a quantity of spun and drawn 3.05 cm (1.2 inch) long microfibrils having a diameter of 7.5 microns was provided. The fibres were lubricated with a silicone finish. The spun-and-drawn microfibrils were polyester and were drawn to achieve a relatively high tensile modulus 54-81 gm/dtex (60-90 grams/denier), which contributed significantly to a high bending modulus. After drawing they were crimped, cut into staple, and thoroughly opened, or separated, in a card. The average crimp frequency was 5.5/cm (14/in), and the average crimp amplitude was 0.10/cm (0.04/in). Loft and compressional characteristics were improved further through the blending with 10 percent by weight of macrofibrils of the same polyester (polyethylene terephthalate) having a diameter of 25.5 microns. The macrofibrils were lubricated with a silicone finish and were characterised in part by a staple length of 5.6 cm (2.2 in), an average crimp frequency of 3.4/cm (8.5/in), and a crimp amplitude (average) of 0.15 cm (0.06 in). The blend was carded into a batt. The physical properties of the batt are shown in Table I below.

COMPARATIVE EXAMPLE 2

The procedure of Comparative Example 1 was repeated with the exception that the macrofibre used therein was replaced with 20 percent by weight of uncrimped poly(p-phenylene terephthalamide) fibres having a diameter of 12 microns, a length of 7.6 (3.0 in), and a silicone lubricant finish. The physical characteristics of the material formed are given in Table I below.

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EXAMPLE 1

A quantity of 0.55 dtex (0.5 denier) 7.5 micron diameter polyester microfibre that had been spun, drawn, cut to a staple length of 3.0 cm (1.2 in) and crimped was first opened in a wire-clothed carding machine. The opened fibre was then scoured, dried and treated with a silicone finish that imparts lubricity and water repellency. The microfibre was then combined and uniformly blended with a 4.4 dtex, 5.1 cm (4 denier, 2 in) long polyester binder fibre of the side-by-side type (Type TJ04S2, available from Teijin). Blending was achieved by subjecting the mixed fibre stock to several passes through a carding machine. The mixture ratio was 90/10, microfibre/binder macrofibre, by weight. After the mixed fibres had been uniformly blended and opened, card laps (output webs from the carding machine) were plied to form batts. The final processing step was oven exposure of the batts at 160°C (320°F) for 5 minutes to obtain thermoplastic bonds between microfibres and binder macrofibres and between binder macrofibres. These bonds ensured that each batt was a cohesive, non-separable fibrous assembly.

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The prepared batts were evaluated in accord with the test procedures described above and the results are set forth in Table I below.

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EXAMPLE 2

A quantity of 0.55 dtex (0.5 denier) 7.5 micron diameter polyester microfibre that had been spun, drawn, cut to a staple length of 3.0 cm (1.2 in), and crimped was first opened in a wire-clothed carding machine. The opened fibre was then scoured, dried and treated with a silicone finish that imparts lubricity and water repellency. The microfibre was then combined and uniformly blended with 4.4 dtex, 5.1 cm (4 denier, 2 in) long, polyester binder fibre of the side-by-side type (TJ0452, available from Teijin). Blending was achieved by subjecting the mixed fibre stock to several passes through a carding machine. The mixture ratio was 90/10, microfibre/binder macrofibre, by weight. After the mixed fibres had been uniformly blended and opened, the card lap (output of the carding machine) was separated into clusters. These clusters were made more or less spherical in shape with an average diameter of 1.91 cm (0.75 in), and an average weight of 15 mg. Cluster formation was achieved in the laboratory through hand manipulation, although at least two commercial processes for transforming carded fibres into clusters or balls are known. The final processing step was oven exposure of the down-like clusters to a temperature of 160°C (320°F) for 5 minutes to obtain thermoplastic bonds between microfibres and binder macrofibres and between binder macrofibres. These bonds made each individual cluster a cohesive, non-separable unit.

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The prepared clusters were evaluated in accord with the test procedures described above and the results are set forth in Table I below.

EXAMPLE 3

A quantity of 0.55 dtex (0.5 denier) 7.5 micron diameter polyester microfibre that had been spun, drawn, cut to a staple length of 3.0 cm (1.2 in), and crimped was first opened in a wire-clothed carding machine. The opened fibre was then scoured, dried and treated with a silicone finish that imparts lubricity and water repellency. The microfibre was then combined and uniformly blended with 4.4 dtex, 5.1 cm (4 denier, 2 in) long, polyester binder fibre of the side-by-side type (Type TJ04S2, available from Teijin). Blending was achieved by subjecting the mixed fibre stock to several passes through a carding machine. The mixture ratio was 85/15, microfibre/binder macrofibre by weight. After the mixed fibres had been uniformly blended and opened, card laps (output webs from the carding machine) were plied to form batts. The final processing step was oven exposure of the batts at 160°C (320°F) for 5 minutes to obtain thermoplastic bonds between microfibres and binder macrofibres and between binder macrofibres. These bonds ensured that each batt was a cohesive, non-separable fibrous assembly.

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The prepared batts were evaluated in accord with the test procedures described above and the results are set forth in Table I below.

The insulator produced in this example was used to manufacture jackets, sleeping bags and quilts. All were found to have and maintain thermal insulating performance equivalent to or better than those using down as the insulator.

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EXAMPLE 4

A quantity of 0.55 dtex (0.5 denier), 7.5 diameter polyester microfibre that had been spun, drawn cut to a staple length of 3.0 cm (1.2 in), and crimped was first opened in a wire-clothed carding machine. The opened fibre was then scoured, dried and treated with a silicone finish that imparts lubricity and water repellency. The microfibre was then combined and uniformly blended with 4.4 dtex, 5.1 cm (4 denier, 2 in) long, polyester binder fibre of the side-by-side type (Type TJ04S2, available from Teijin). Blending was achieved by subjecting the mixed fibre stock to several passes through a carding machine. The mixture ratio was 85/15 microfibre/binder macrofibre, by weight. After the mixed fibres had been uniformly blended and opened, the

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card lap (output of the carding machine) was separated into clusters. These clusters were more or less spherical in shape with an average diameter of 1.91 cm (0.75 in) and an average weight of 15 mg. Cluster formation was achieved in laboratory through hand manipulation, although at least two commercial processes for transforming carded fibres into clusters or batts are known. The final processing step was oven exposure of the down-like clusters to a temperature of 160° C (320° F) for 5 minutes to obtain thermoplastic bonds between microfibrils and binder macrofibrils and between binder macrofibrils. These bonds made each individual cluster a cohesive, non-separable unit.

The prepared clusters were evaluated in accord with the test procedures described above and the results are set forth in Table I below.

EXAMPLE 5

A quantity of 0.55 dtex (0.5 denier), 7.5 micron diameter polyester microfibre that had been spun, drawn, cut to a staple length of 3.0 cm (1.2 in), and crimped was first opened in a wire-clothed carding machine. The opened fibre was then scoured, dried, and treated with a silicone finish that imparts lubricity and water repellency. The microfibre was then combined and uniformly blended with 4.4 dtex, 5.1 cm (4 denier, 2 in) long, polyester binder fibre of the side-by-side type (Type TJO452, available from Teijin). Blending was achieved by subjecting the mixed fibre stock to several passes through a carding machine. After the mixed fibres had been uniformly blended and opened, card laps (output webs from the carding machine) were plied to form batts. The final processing step was oven exposure of the batts at 160° C (320° F) for 5 minutes to obtain thermoplastic bonds between microfibrils and binder macrofibrils and between binder macrofibrils. These bonds ensured that each batt was a cohesive, non-separable fibrous assembly.

The prepared batts were evaluated in accord with the test procedures described above and the results are set forth in Table I below.

EXAMPLE 6

A quantity of 0.55 dtex (0.5 denier) 7.5 micron diameter polyester microfibre that had been spun, drawn, cut to a staple length of 3.0 cm (1.2 in), and crimped was first opened in a wire-clothed carding machine. The opened fibre was then scoured, dried and treated with a silicone finish that imparts lubricity and water repellency. The microfibre was then combed and uniformly blended with 4.4 dtex, 5.1 cm (4 denier, 2 in) long, polyester binder fibre of the side-by-side type (Type TJ04S2, available from Teijin). Blending was achieved by subjecting the mixed fibre stock to several passes through a carding machine. The mixture ratio was 80/20, microfibre/binder macrofibre, by weight. After the mixed fibres had been uniformly blended and opened, the card lap (output of the carding machine) was separated into clusters. These clusters were more or less spherical in shape with an average diameter of 1.91 cm (0.75 in) and an average weight of 15 mg. Cluster formation was achieved in the laboratory through hand manipulation.

The final processing step was oven exposure of the down-like clusters to a temperature of 160° C (320° F) for 5 minutes to obtain thermoplastic bonds between microfibrils and binder macrofibrils and between binder macrofibrils. These bonds made each individual cluster a cohesive, non-separable unit.

The prepared clusters were evaluated in accord with the test procedures described above and the results are set forth in the following table:

TABLE I
(Imperial Units in Brackets)

	Down	Comparative Example 1	Comparative Example 2	Example 1 (Batt, 90/10)
Apparent thermal conductivity W/m-K	0.040	0.040	0.039	0.039
(Btu-m/hr-ft ² °F)	(0.280)	(0.281)	(0.271)	(0.269)
Thermal cond. test density kg/m ³	7.21	7.53	7.69	8.01
(lb/ft ³)	(0.45)	(0.47)	(0.48)	(0.50)
Radiation parameter C (W/m-K) (kg/m ³) (10 ⁻²)	10.8	11.5	10.6	10.8
(Btu-in/hr-ft ² °F) (lb/ft ³) (10 ⁻²)	(4.7)	(5.0)	(4.6)	(4.7)
Minimum density kg/m ³	3.85	4.01	4.01	6.89
(lb/ft ³)	(0.24)	(0.25)	(0.25)	(0.43)
Comp. strain at 34.4 kPa (%) ^b }	95	96	92	97
(5lb/in ² (%)) }				
Comp. recovery from 34.4 kPa (%) ^b }	102	112	112	83
(5lb/in ² (%)) }				
Work to compress to 34.4 kPa (N-m)	0.55	0.39	0.40	0.36
(lb-in)	(4.91)	(3.49)	(3.57)	(3.21)
Resilience ^c	0.53	0.62	0.60	0.59
<u>Wetting during Immersion</u>				
Water absorption after 20 min. (x dw) ^d	1.16	2.16	1.41	1.09
Density after 20-min wetting (kg/m ³)	7.69	8.02	8.17	8.49
(lb/ft ³)	(0.48)	(0.50)	(0.51)	(0.53)
Water absorption after 6 hour (x dw)	3.75	5.15	3.44	1.42
Density after 6 hour wetting kg/m ³	56.91	15.07	16.35	11.86
(lb/ft ³)	(3.55)	(0.94)	(1.02)	(0.74)

TABLE I (Cont/d)

	Example 2 Clusters 90/10	Example 3 Batt 85/15	Example 4 Clusters 85/15	Example 5 Batt 80/20	Example 6 Clusters 80/20
Apparent thermal conductivity (W/m-K)	0.038	0.042	0.039	0.042	0.041
(Btu-in/hr-ft ² -°F)	(0.264)	(0.291)	(0.268)	(0.291)	(0.286)
Thermal cond. test density (kg/m ³)	8.02	8.02	8.02	8.02	8.02
(lb/ft ³)	(0.50)	(0.50)	(0.50)	(0.50)	(0.50)
Radiation parameter C (W/m-K) (kg/m ³) (10 ⁻²)	10.2	13.4	10.6	13.4	12.9
(Btu-in/hr-ft ² -°F) (lb/ft ³) (10 ⁻²)	(4.4)	(5.8)	(4.6)	(5.8)	(5.6)
Minimum density (kg/m ³)	4.17	7.37	4.17	6.25	3.85
(lb/ft ³)	(0.26)	(0.46)	(0.26)	(0.39)	(0.24)
Comp. strain at 34.4 kPa (%) ^b }	95	96	95	96	95
(5lb/in ²) %					
Comp. recovery from 34.4 kPa (%) ^b }	130	81	135	87	132
(5lb/in ²) %					
Work to compress to 34.4 kPa (N-m)	0.54	0.35	0.54	0.34	0.52
(lb-in)	(4.75)	(3.13)	(4.76)	(3.01)	(4.56)
Resilience	0.44	0.58	0.43	0.61	0.46
<u>Wetting during Immersion</u>					
Water absorption after 20 min. (x dw) ^d	1.61	1.04	1.14	1.08	1.06
Density after 20 min wetting (kg/m ³)	6.89	8.17	5.13	8.02	4.49
(lb/ft ³)	(0.43)	(0.51)	(0.32)	(0.50)	(0.28)
Water absorption after 6 hr (x dw)	2.96	1.75	2.03	1.39	1.41
Density after 6 hr wetting (kg/m ³)	14.43	15.39	10.26	10.90	6.89
(lb/ft ³)	(0.90)	(0.96)	(0.64)	(0.68)	(0.43)

TABLE 1 (cont/d)

	<u>Down</u>	<u>Comparative Example 1</u>	<u>Comparative Example 2</u>	<u>Example 1 Batt 90/10</u>		
<u>Drying after 24 hrs.</u>					5	
<u>Water Immersion</u>						
Weight after 30 min drying (x dw)	3.88	4.83	3.29	1.27	10	
Density after 30 min drying (kg/m ³)	83.37	15.23	14.43	9.94		
(lb/ft ³)	(5.20)	(0.95)	(0.90)	(0.62)	15	
Weight after 6 hr drying (x dw)	2.45	1.68	1.01	1.0		
Density after 6 hr drying (kg/m ³)	51.30	6.57	7.05	7.85	20	
(lb/ft ³)	(3.20)	(0.41)	(0.44)	(0.49)	25	
	<u>Example 2 Clusters 90/10</u>	<u>Example 3 Batt 85/15</u>	<u>Example 4 Clusters 85/15</u>	<u>Example 5 Batt 80/20</u>	<u>Example 6 Clusters 80/20</u>	30
Weight after 30 min drying (x dw)	2.79	1.53	1.87	1.27	1.35	
Density after 30 min drying (kg/m ³)	12.98	13.65	8.49	9.94	6.57	35
(lb/ft ³)	(0.81)	(0.84)	(0.53)	(0.62)	(0.41)	
Weight after 6 hr drying (x dw)	1.92	1.0	1.0	1.0	1.0	40
Density after 6 hr drying (kg/m ³)	8.82	7.85	4.49	7.37	4.33	45
(lb/ft ³)	(0.55)	(0.49)	(0.28)	(0.46)	(0.27)	
a. Heat flow down: 5.23 cm specimen thickness						50
b. Gauge length: 5.1 cm (2.00 inches) density at 5.1 cm (2.00 inches) thickness was 8.02 kg/m ³ (0.50) lb/ft ³ .						
c. Resilience equals: work-of-recovery divided by work-to-compress.						55
d. x dw times dry weight.						

It can be seen from the above Table I that the insulating efficiency of each of Examples 1 through 6 of the invention, as characterised by apparent-thermal-conductivity data and radiation parameter values, closely

approximates that of the down/feathers mixture and of Comparative Examples 1 and 2. The insulating value of material produced in accord with the invention, as exemplified by Example 2, is further illustrated in Figure 1, in which the apparent thermal conductivity/density diagrams for down/feathers and the synthetic clusters of Example 2 are seen to be nearly coincident. It can be seen from Table I that the mechanical performance of Examples 1 through 6, as characterised by minimum density, compressional strain, compressional recovery, work to compress, and resilience, compares favourably in most instances to the mechanical performance of the down feathers mixture and Comparative Examples 1 and 2.

Differences do exist, however, among values for two important mechanical performance indicators those of minimum density (loft) and compressional recovery. The minimum density and compressional recovery values for the batts of Examples 1, 3 and 5 indicate inferior performance compared to down/feathers and Comparative Examples 1 and 2, while the compressional recovery values for the cluster forms of Examples 2, 4 and 6 indicate significant performance improvement over down/feathers. The minimum density (loft) values for the cluster forms are virtually equal to those of down/feathers and non-bonded Comparative Examples 1 and 2. This mechanical performance advantage of the synthetic clusters is a direct consequence of difference in fibre orientation. An aggregation of clusters like those of Examples 2, 4 and 6 (and as would be employed in a typical insulator application) constitutes a collection of fibres of random orientation. This is in distinct contrast to the ordered fibre orientation of the batt form. A large fraction of the fibres that comprise each batt lie more or less parallel to the plane of the batt, contributing relatively little to its loftiness and compressional elasticity. In the cluster form, the random fibre alignment provides some fibres that are perpendicular to, or nearly perpendicular to, the insulator plane. These fibres are, in effect, structural columns. They improve the loftiness of the assembly and, through elastic bending and/or buckling, greatly enhance the compressional recovery of the insulator.

Further examination of Table I makes clear the considerable improvement in performance during and following water exposure that further distinguishes Examples 1 through 6 in comparison to the down/feathers mixture. Density values for Examples 1 through 6 at the "6 hr. wetting", "3 min. drying", and "6 hr. drying" intervals in the wetting/drying cycle are much lower than those for down/feathers, indicating that Examples 1 through 6 retain loft while wet and, most probably, insulating value to a far greater degree than does down. Resistance-to-wetting and resistance to loss-of-loft while wet are inherent advantages of the fibre combination described herein. The hydrophobic nature of polyester and the microporous structure of the insulators are assumed to contribute to these desirable characteristics.

Several further comparative examples were prepared for the purpose of documenting the insulator stability and cohesiveness that was manifest through examination and handling of Examples 1 through 6, above. These comparative examples were as follows:

COMPARATIVE EXAMPLE 3

The procedure of Example 1 was repeated to produce another batt having a fibre mixture ratio of 90/10, microfibre/binder macrofibre by weight. However, the final processing step described for Example 1, oven exposure was omitted to provide a non-bonded batt for comparative purposes.

COMPARATIVE EXAMPLE 4

The procedure of Example 5 was repeated to produce another batt having a fibre mixture ratio of 80/20, microfibre/binder macrofibre by weight. However, the final processing step described for Example 5, oven exposure, was omitted to provide a non-bonded batt for comparative purposes.

COMPARATIVE EXAMPLE 5

The basic procedure of Example 4 was repeated to produce another collection of clusters having a fibre mixture ratio of 85/15 microfibre/binder macrofibre, by weight, with the exception that the final oven exposure step was omitted. The clusters produced differed from those of Example 4 in that their average diameter was 3.0 cm (1.2 in), their average weight was 60 mg, and they were not bonded.

An additional example of the subject invention was also prepared to further facilitate documentation of the stability and cohesiveness of insulating media made according to the invention. This example was as follows:

EXAMPLE 7

The basic procedure of Example 4 was repeated to produce another collection of clusters having a fibre mixture ratio of 85/15, microfibre/binder macrofibre by weight. The clusters produced differed from those of Example 4 only in size and weight. The clusters of this example, like those of Comparative Example 5, had an average diameter of 3.0 cm (1.2 in), and an average weight of 60 mg. The clusters of the present example were, however, subjected to oven exposure at 160° C (320° F) for 5 minutes to obtain thermoplastic bonds between microfibrils and binder macrofibrils and between binder macrofibrils.

Insulating batts of Examples 1 and 5 of the subject invention and Comparative Examples 3 and 4 were evaluated, the batt cohesiveness test previously herein described being used, and the results are set forth in the following table.

TABLE II

Extension Ratios Measured at the Point
of Initial Batt Separation
in Through the Thickness Tensile Tests

	<u>Extension Ratios</u>	
Comparative Example 3; 90/10; non-bonded	3:1	10
Example 1; 90/10; bonded	12:1	15
Comparative Example 4; 80/20; non-bonded	3:1	20
Example 5 80/20; bonded	16:1	25

It will be understood from the above descriptions of the examples and comparative examples (1) that the batts of Example 1 and Comparative Example 3 are alike in terms of types of fibres and proportional quantities of fibres that they contain and (2) that they differ in that only the batt of Example 1 has been subjected to oven exposure to achieve fibre-to-fibre bonding. Similarly, the batts of Example 5 and Comparative Example 4 are alike in basic composition but differ in that only Example 5 contains fibre-to-fibre bonds.

The important effect of fibre-to-fibre bonding upon the cohesiveness of batts of the subject invention, specifically upon that of Examples 1 and 5, is shown by the high extension ratios measured at the point of initial batt separation and set forth in Table II. The high extension ratios of these embodiments are in direct contrast to the low ratios measured for Comparative Examples 3 and 4 (also set forth in Table II).

In corresponding fashion, the importance of fibre-to-fibre bonds to the cohesiveness and integrity of individual clusters is exemplified through comparison of the average separation force measured for clusters of Example 7 with the average force measured for those of Comparative Example 5, as set forth in the following table:

TABLE III

Tensile Force Required to Pull Apart Clusters

	<u>Average Force (gms)</u>	
Comparative Example 5; 85/15; non-bonded	3	45
Example 7; 85/15; bonded	41	50

The results shown above represent a surprisingly high
13.7 x increase in average cluster separation force.

EXAMPLES 8 to 13

Bonded structures were produced in the manner described in Example 1 using a mix of macrofibres. In each example the microfibrils are a 0.55 dtex (0.5 denier) polyester fibre. The macrofibres were a blend of a 4 denier polyester binder fibre as described in Example 1 with a 1.5 dtex (1.4 denier) stiffening fibre of "Kevlar 49".

The results are set out in Table IV. The percentage given of constituents at the head of each example column are percent by weight;

the first figure is the percent by weight of microfibrils (polyester), the second figure is the percent by weight of polyester macrofibre, and the third figure is the percent by weight of "Kevlar" stiffening fibre. Thus, 80/10/10

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has the composition:

0.55 dtex (0.5 denier) polyester microfibre 80 percent by wt.

4.4 dtex (4 denier) polyester macrofibre 10 percent by wt.

1.5 dtex (1.4 denier) "Kevlar 49" stiffening fibre 10 percent by wt.

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TABLE IV (Imperial Units in brackets)

	Example 8 Batt 80/10/10	Example 9 Clusters 80/10/10	Example 10 Batt 75/15/10	Example 11 Clusters 75/15/10	Example 12 Batt 70/20/10	Example 13 Clusters 70/20/10
Apparent thermal conductivity (W/m-K)	0.041	--	0.043	--	0.044	--
(Btu-in/hr-ft ² -°F)	(0.283)		(0.296)		(0.303)	
Thermal cond. test density (kg/m ³)	8.02	8.02	8.02	8.02	8.02	8.02
(lb/ft ³)	(0.50)	(0.50)	(0.50)	(0.50)	(0.50)	(0.50)
Radiation parameter C (W/m-K) (kg/m ³)						
(10 ⁻²)	12.5	--	13.8	--	14.8	--
(Btu-in/hr-ft ² - F)						
(lb/ft ³) 10 ⁻²	(5.4)		(6.0)		(6.4)	
Minimum density (kg/m ³)	6.57	4.01	7.05	4.17	6.09	3.85
(lb/ft ³)	(0.41)	(0.25)	(0.44)	(0.26)	(0.38)	(0.24)
Comp. strain at 34.4 kPa (%) ^b	96	95	95	95	95	95
(51b/ft ² /m)%						
Comp. recovery from 34.4 kPa kPa (%) ^b	86	125	87	120	89	117
(51b/in ²)%						
Work to compress to 34.4 kPa ² (N-m)	0.41	0.40	0.38	0.44	0.38	0.52
(lb/in)	(3.60)	(3.57)	(3.34)	(3.86)	(3.41)	(4.56)
Resilience	0.66	0.76	0.58	0.56	0.58	0.50

- Heat flow down: 5.23 cm (2.06 inch) specimen thickness.
- Gauge length: 5.1 cm (2.00 inches) density at 2.00 inch thickness was 8.02 kg/m³ (0.50 lb/ft³).
- Resilience equals: work-of-recovery divided by work-to-compress.
- x dw: time dry-weight.

Claims

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1. A method of forming a thermal insulating material which method comprises forming a fibre assemblage from 70 to 95 percent by weight of synthetic polymeric microfibrils having a diameter of from 3 to 12 microns; and from 5 to 30 percent by weight of synthetic polymeric macrofibrils having a diameter not less than 12 microns,

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shaping the assemblage so formed, and effecting bonding between at least some of the fibres at their contact points such that the density of the resultant structure is within the range 3 to 16 kg/m³, the bonding being effected without significant loss to thermal insulating properties compared with the unbonded assemblage.

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2. A method as claimed in claim 1 characterised in that bonding is effected by the application of heat, by autologous bonding or by bonding agents.

3. A method as claimed in claim 1 or claim 2 characterised in that the shaping of the assemblage is carried out using a carding machine and an automatic ball-rolling machine ("cotton-ball machine") or a carding machine and a lapper for plying card laps into a batt.

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4. A method as claimed in any preceding claim characterised in that the bonding is effected between at least some of said macrofibrils to form a supporting structure for said microfibrils.

5. A method as claimed in any preceding claim characterised in that the macrofibre and/or the microfibre is selected from one or more of polyester, nylon, rayon, acetate, acrylic, modacrylic, polyolefins, spandex, polyaramids, polyimides, fluorocarbons, polybenzimidazoles, polyvinylalcohols, polydiacetylenes, polyetherketones, polyimidazoles and phenylene sulphide polymers.

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6. A method as claimed in any preceding claim characterised in that the macrofibre is selected from one or more of:

(i) multi-component fibres having a moiety to facilitate macrofibre to macrofibre bonding;

(ii) a fibre mixture in which at least 10% by weight of the macrofibrils comprise macrofibrils of a low melting point material; and

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(iii) a fibre mixture comprising multi-component macrofibrils and single component macrofibrils capable of bonding one with the other.

7. A method as claimed in claim 6 characterised in that multi-component macrofibrils are selected from two component fibres in a side-by-side construction or in a sheath/core construction.

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8. A method as claimed in any preceding claim characterised in that said bonded material has a radiation parameter defined as the intercept on the ordinate axis at zero density of a plot of $K_c P_F$ against P_F less than 0.173 (W/m-K)(kg/cm³) and a density P_F from 3.2 to 16 kg/cm³ and an apparent thermal conductivity K_c measured by the plate-to-plate method according to ASTM C518 with heat flow down of less than 0.072 W/m-K.

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9. A method as claimed in any preceding claim characterised in that at least one of the fibrous components is treated with a finish having water repellent and/or lubricant properties.

10. A method as claimed in any preceding claim characterised in that the microfibrils and/or the macrofibrils are crimped prior to bonding.

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11. A method as claimed in any preceding claim characterised by the inclusion of a proportion of stiffening fibre within the fibre structure.

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