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Prentice

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[54] **NONWOVEN MAT BATTERY SEPARATORS**

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[*] Notice: The portion of the term of this patent subsequent to Nov. 28, 1989, has been disclaimed.

[22] Filed: **Oct. 17, 1972**

[21] Appl. No.: **298,317**

Related U.S. Application Data

[63] Continuation-in-part of Ser. Nos. 865,089, Oct. 9, 1969, Pat. No. 3,704,198, and Ser. No. 233,826, March 10, 1972.

[52] **U.S. Cl.** **136/146;** 136/148

[51] **Int. Cl.** **H01m 3/02**

[58] **Field of Search** 136/148, 146; 156/306; 161/150

[56] **References Cited**

UNITED STATES PATENTS

3,121,658	2/1964	Orsino et al.	136/148
3,276,944	10/1966	Levy	156/306
3,354,247	11/1967	Zehender et al.	136/148
3,650,866	3/1972	Prentice	156/306
3,704,198	11/1972	Prentice	161/150
3,715,251	2/1973	Prentice	156/306

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[57] **ABSTRACT**

The strip tensile strength of nonwoven mat battery separators of polypropylene fibers having a diameter from about 1 to about 10 microns is increased, for example, to strengths greater than 4,000 m, by fuse-bonding, as by calendering or point-bonding, at least a portion of the fibers of the mat at temperatures within the range from about 250°F to about 325°F, preferably, from about 280°F to about 315°F, while the mat is subjected to pressure sufficient to prevent shrinkage of the fibers in the mat.

4 Claims, 4 Drawing Figures

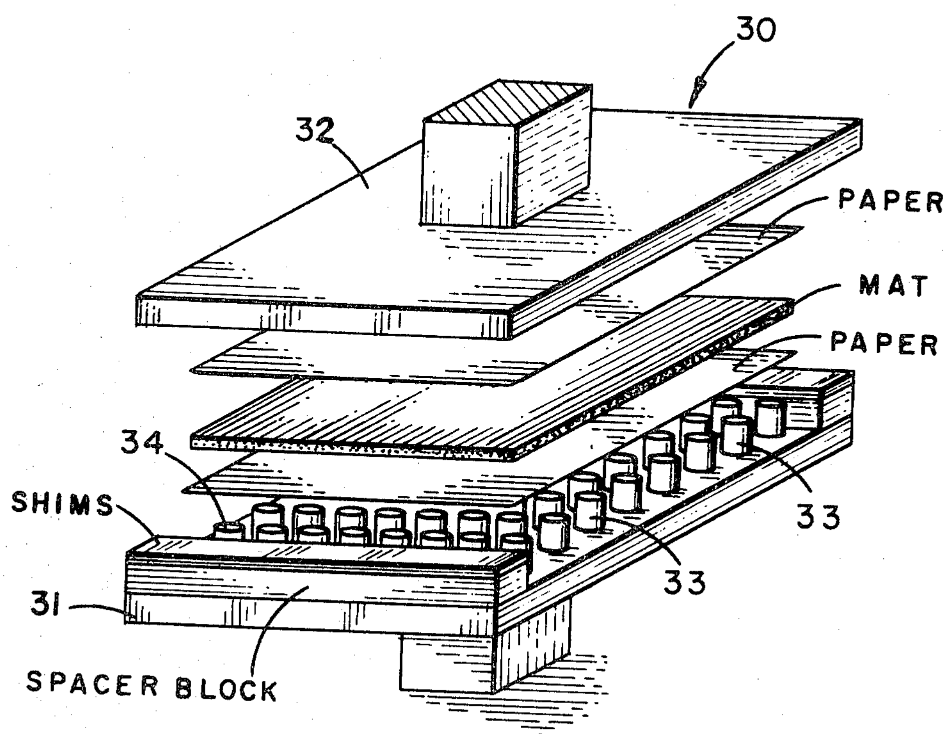
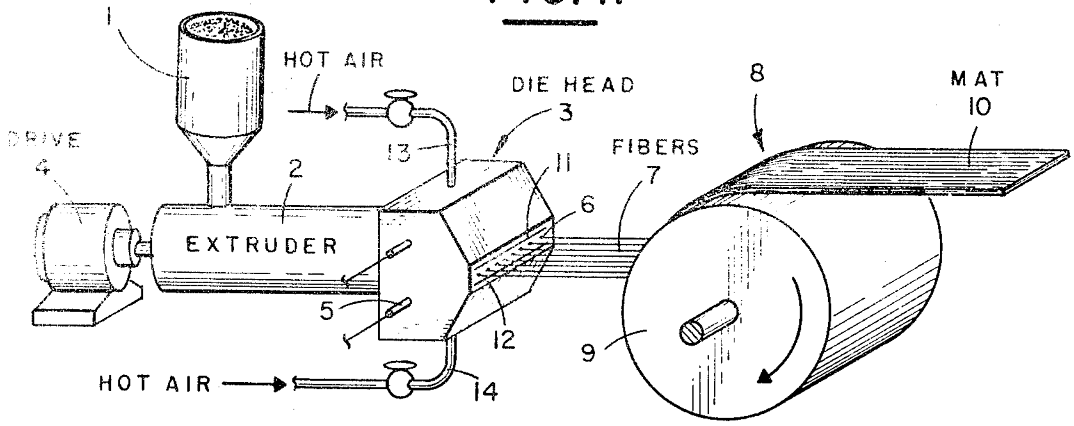


FIG. 1.



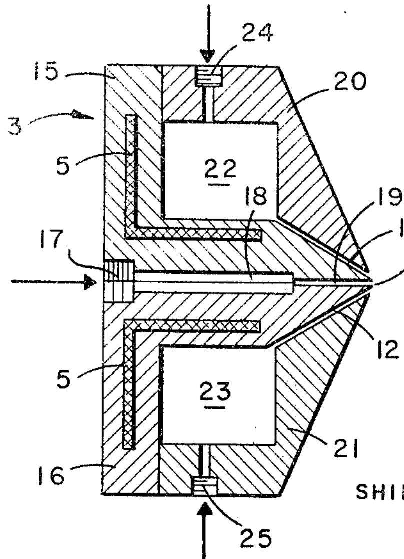


FIG. 2.

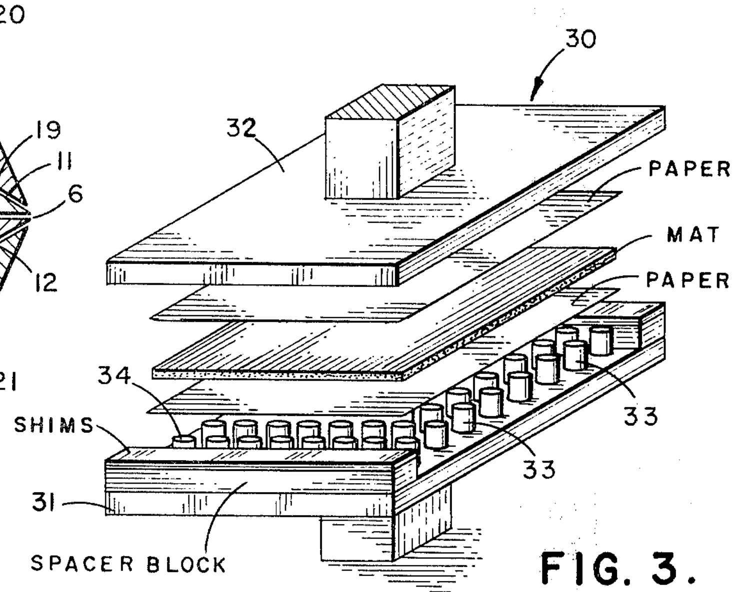


FIG. 3.

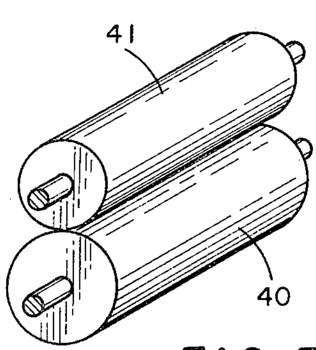


FIG. 3A.

NONWOVEN MAT BATTERY SEPARATORS

RELATED APPLICATIONS

This application is a continuation in part of my co-pending applications Ser. No. 865,089 filed Oct. 9, **1969** now U.S. Pat. No. 3,704,198 and its Division Ser. No. 233,826, filed Mar. 10, 1972.

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention is directed to nonwoven mat battery separators of polypropylene fibers having a diameter from about 1 to about 10 microns and to a method for treating such mats so that they have high strip tensile strength.

2. Prior Art

U.S. Pat. No. 3,276,944. Wente, Van A., "Super-Fine Thermoplastics," *Industrial and Engineering Chemistry*, Vol. 48, No. 8 (1956), pp. 1342–1346.

SUMMARY OF THE INVENTION

In this invention, the strip tensile strength of nonwoven mat battery separators of polypropylene fibers having diameters from about 1 to about 10 microns is increased by fuse-bonding, as by point-bonding or by calendering, at least a portion of the fibers of the mat at a temperature within the range from about 250°F to about 325°F, while maintaining pressure on the nonwoven mats sufficient to prevent shrinkage of the fibers while they are exposed to the fuse-bonding temperatures. The nonwoven mats so treated have fusion-bonds throughout their thickness, and may have strip tensile strengths greater than 4,000 m, preferably, greater than 5,000 or even 6,000 m. The battery separators are assembled in batteries between the positive and negative plates.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a schematic perspective view of an overall melt blowing process;

FIG. **2** is a detailed view in longitudinal cross section of a die which may be used in the melt blowing process;

FIG. **3** is a schematic view of a press used to increase the strip tensile strength of a nonwoven mat of polypropylene fibers of diameters from about 1 to about 10 microns; and

FIG. 3A is a schematic view of calender rolls which may be used instead of the press of FIG. **3.**

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring to FIG. **1** of the drawings, a polypropylene resin is introduced into a pellet hopper **1** of an extruder **2.** The resin is forced through the extruder **2** into a die head **3** by a drive **4.** The die head **3** may contain heating plates **5** which may control the temperature in the die head **3.** The polypropylene resin is then forced out of a row of die openings **6** in the die head **3** into a gas stream which attenuates the resin into fibers **7** which are collected on a moving collecting device **8** such as a drum **9** to form a continuous mat **10.** The gas stream which attenuates the polypropylene is supplied through gas jets **11** and **12** respectively, which are seen more clearly in FIG. **2.** The gas slots **11** and **12** are supplied with a hot gas, preferably air, by gas lines **13** and **14** respectively.

The process may be further understood by considering the details of the die head **3** which is more fully set forth in FIG. **2.** The die head **3** is made up of an upper die plate **15** and a lower die plate **16.** The polypropylene resin is introduced in the back of the die plates **15** and **16** through an inlet **17** as it is forced through the extruder **2** into the back of the die head **3.** The polypropylene then goes into a chamber **18** between the upper and lower die plates **15** and **16** respectively. The facing of the die plate **16** may have milled grooves **19** which terminate in the die openings **6.** It is understood, of course, that the milled grooves may be in the lower die plate **16,** in the upper die plate **15,** or that grooves may be milled in both plates **15** and **16.** Still further, if a single plate is used in place of the upper and lower die plates, the grooves may be drilled to produce the die openings **6.** An upper gas cover plate **20** and a lower gas cover plate **21** are connected to the upper die plate and lower die plate **15** and **16** respectively to provide an upper air chamber **22** and a lower air chamber **23** which terminate in the gas slots **11** and **12** respectively. The hot gas is supplied through inlet **24** in upper gas cover plate **20** and inlet **25** in lower gas cover plate **21.** Suitable baffling means (not shown) may be provided in both the upper air chamber **22** and lower air chamber **23** to provide a uniform flow of air through the gas slots **11** and **12** respectively. The die head **3** may contain heating means **5** for heating both the polypropylene and air in the die head **3.**

The characteristics of the nonwoven polypropylene mats produced by the melt blowing process will vary considerably depending upon the particular process conditions used. The characteristics of the nonwoven polypropylene mats are affected in large part by the air flow rates used relative to the rate of resin throughput in the melt blowing process and by the distance of the take-up device from the die openings in the die head. In the melt blowing process, when the air rates are high and the pounds of air to pounds of polypropylene extruded through the die head are high (suitably greater than about 140, desirably 150, and preferably in excess of 200 lbs.air/lbs.of polymer for polypropylene) the polypropylene fibers in the nonwoven mat are such that generally the mat has a high zero span tensile strength. The fibers at the higher air rates appear to be drawn, and are much finer fibers as evidenced by their small diameters, which range from 1 to 10 microns.

One of the other process variables in the melt blowing process which materially affects the characteristics of the nonwoven polypropylene mat is the distance of the take-up device from the die openings and the die head. When the collecting device is between 1 to 6 inches from the die openings, there is evidence of considerable self-bonding of the fibers as they are laid down in the nonwoven mat. At distances greater than 6 inches between the die openings and collector device, some self-bonding still occurs but the amount of self-bonding decreases with distance. The term "self-bonding" herein means thermal bonding of one fiber to another in the melt blowing process as the nonwoven thermoplastic polymer mats are formed. The strip tensile strength of the polypropylene mats are produced by the melt blowing process is greatest in those mats having the highest degree of self-bonding; but as the self-bonding decreases, the mat is comprised essentially of entangled fibers and the strip tensile strength materially decreases.

Another factor in the strip tensile strength of the melt blown mat is the tensile strength of the fiber itself. The tensile strength of the fibers in the melt blown nonwoven mat are found by measuring the zero span tensile strengths of the mats, a measurement which utilizes the same general procedure employed to obtain the strip tensile strength of the mats. The procedure for obtaining strip tensile strength and zero span tensile strength are those in ASTM procedure D-828-60, with the exception that to measure the strip tensile strength the clamps are set apart at a distance of two inches, using an elongation rate of 250%/min., but to measure the zero span tensile strength, the clamps are not separated by any distance. In both instances, the results are reported in meters, the unit resulting when the force necessary to break the mat, measured in grams, is divided by the width of the sample, measured in meters, all of which is divided by the basis weight of the sample in terms of gm/m^2. The process variables used in the melt blowing process to obtain mats of high zero span tensile strength result in low tear resistances in those mats, as measured by a standard Elmendorf tear strength tester in accordance with ASTM procedure D-689-62.

The strip tensile strength of melt blown nonwoven mats of polypropylene fibers are generally lower than desirable for load bearing applications, even though the zero span tensile strengths of the mats may be quite high. The principal object of the present invention is to make the strip tensile strength of a nonwoven mat approach, and even more desirably, attain or exceed the zero span tensile strength of that mat.

In accordance with this invention, the strip tensile strength of nonwoven mats of polypropylene fibers having diameters between about 1–10 microns is increased by fuse-bonding the nonwoven mat at temperatures within the range from about 250°F. to about 325°F., preferably, from about 280°F. to about 315°F., while compressing the mat sufficiently to prevent shrinkage of the fibers in the mat.

The fuse-bonding process may be accomplished by applying a spaced pattern of heat to the mat to melt discrete portions of the mat and to fuse a portion of the fibers in the mat. Herein, this is termed "point-bonding". Alternatively, the fuse-bonding may be effected by a general fusion of the fibers by applying an unbroken pattern of heat to the mat, such as by calendering. In the fuse-bonding process, application of sufficient pressure to the nonwoven mat to prevent shrinkage of the fibers in the mat is indicated by a lack of difference in the size of the mat before and after fuse-bonding and by a lack of decrease in the zero span tensile strength of the nonwoven mat.

Referring to FIG. 3, a heated press 30 may be used in the fuse-bonding process to increase the strip tensile strengths of the melt blown nonwoven mats. The press 30 comprises a plate 31 and a plate 32. The plate 31 has a plurality of spaced apart projections 33 which terminate in flat lands 34 that are spaced in a design or pattern. The spacing of the projections 33, the total surface of the flat lands 34 and the pattern of the projections 33 of the plate 31 will partially determine the degree to which the fibers of the nonwoven mats are point-bonded. The spacing of the projections 33 and the pattern may vary greatly although suitable patterns are rectilinear or diagonal grids. Suitable spacing for the projections 33 is between about 1/16 inch and ¼ inch. The depth to which the projections 33 will penetrate the nonwoven sheet may be controlled by the use of spacers or shims placed between plate 31 and plate 32. The plate 32 may have a flat surface which would come in contact with the lands 34 of the projections 33 of plate 31 or the plate 32 may also have projections which are aligned with the projections of plate 31 so that the lands of the projections of plate 32 would contact the lands 34 of the projections of plate 31. To obtain higher increases in the strip tensile strengths of the nonwoven mats, the deeper penetration of the projections 33 into the nonwoven mats is desired. Each of the plates 31 and 32 may be heated independently so that the temperatures of the plates are not necessarily the same.

In the fuse-bonding process of the nonwoven mats, it is convenient to use spacer sheets between the nonwoven mats and the plates 31 and 32 of the press to prevent the nonwoven mat from sticking to the plates either during or subsequent to the fuse-bonding operation. Suitable spacer sheet material which may be employed include insulating materials such as tissue paper, ordinary paper and the like or heat conducting materials such as aluminum foil and the like. The conducting spacer sheets are preferred since the additional heat increases the fusing of the fibers in the fuse-bonding process.

For the continuous production of the nonwoven mats of improved higher strip tensile strengths, it is advantageous to carry out the fuse-bonding process by calendering the nonwoven mats under heating conditions which effect fiber-to-fiber fusion under pressure sufficient to prevent shrinkage of the fibers of the nonwoven mat. Referring to FIG. 3A, calender rolls 40 and 41 are shown which may be employed instead of the press 30. The calender roll 40 may have projections which terminate in flat lands which may be patterned in a wide variety of forms, such as a diagonal grid (not shown). In the calendering operation to carry out the fuse-bonding, spacer sheets may also be used to prevent the nonwoven mats from sticking to the calender rolls.

The fusion-bonded nonwoven mat of polypropylene fibers suitably has a thickness within the range of from about 1 to about 25 mils, preferably less than about 10 mils. Basis weight may vary from about 10 gm/m^2 to 300 or more gm/m^2. Depending on the degree and manner in which fusion-bonding is accomplished, the fusion-bonds which occur throughout the thickness of the nonwoven mat are discrete points in an essentially spaced pattern along its length and breadth or else occur continuously along its length and breadth in an essentially unbroken pattern. The strip tensile strength of the fusion-bonded mat is high, greater than about 4,000 m, desirably greater than 5,000 m, and preferably in excess of 6,000 m. The tear resistance of the fusion-bonded mat is low, generally less than 200 dm^2, rarely if ever as great as 300 dm^2.

The fusion-bonded mats, by virtue of their high strength, which may exceed the zero span tensile strength of the untreated mat, are highly useful as fabrics for load bearing applications, such as tapes, reinforcing liners, carpet backing and the like. A special application involves their use as components in laminates to which they contribute high strip tensile strength. Another special application of the fusion-bonded mats is as battery separators. The mats are positioned between and separate the positive and negative plates in a battery.

The fusion-bonded nonwoven mats of the present invention and the fusion-bonding process are further illustrated by specific examples hereinafter following. These examples utilize nonwoven mats of melt blown polypropylene fibers produced by the melt blowing process illustrated in FIGS. 1 and 2 of the drawings with the specific operating conditions as set forth in Table I. The specific nonwoven mat characteristics are also set forth in Table I as to basis weight, zero span tensile strength and the degree of uniformity of the mat in terms of the zero span tensile strength by the ratio of the cross direction to machine direction (CD/MD). The fiber diameter of the mats ranged between about 1 to about 10 microns, usually between about 1 to 5 microns.

TABLE I

MAT NOS.	A	B	C	D
Melt Blowing Conditions				
Polypropylene MFR	0.6	0.6	0.6	0.6
Extruder Temp. (°F)	640	653	702	720
Die Temp. (°F)	600	630	643	642
Polymer Rate $\left(\frac{gm}{min}\right)$	8.2	8.0	8.0	8.0
Apparent Viscosity(poise)	—	177	307	251
Air Flow(lb/min)	4.2	3.85	3.78	4.24
$\frac{\text{Lb. air}}{\text{Lb. polymer}}$	233	218	214	240
Screen Distance(in.)	4	6	6	6
Mat Characteristics				
Basis weight $\left(\frac{gm}{m^2}\right)$	50	58	58	66
Zero Span Tensile(m)	5490	4580	6440	5340
CD/MD	0.75	0.57	0.62	0.76

TABLE I – Continued

MAT NOS.	E	F	G	H
Melt Blowing Conditions				
Polypropylene MFR	0.6	0.6	3.5	3.5
Extruder Temp.(°F)	691	690	647	650
Die Temp.(°F)	631	632	609	602
Polymer Rate $\left(\frac{gm}{min}\right)$	8.4	8.4	7.0	7.0
Apparent Viscosity(poise)	97	154	84	70
Air Flow(lb/min.)	4.32	4.15	3.35	3.36
$\frac{\text{Lb. air}}{\text{Lb. polymer}}$	233	224	217	218
Screen Distance(in.)	6	12	4	4
Mat Characteristics				
Basis weight $\left(\frac{gm}{m^2}\right)$	51	50	131	70
Zero Span Tensile(m)	4820	4730	4930	5383
CD/MD	0.78	0.82	0.70	0.69

EXAMPLE 1

This example illustrates how the strip tensile strength of a nonwoven mat is increased by fuse-bonding the mat.

Samples of nonwoven mat A, made under melt blowing conditions which produce fibers that have a diameter from about 1 to 10 microns (see Table I), were calendered once by passing them through heated calendering rolls at temperatures ranging from 200°F. to 320°F. under a roller pressure of 700 pounds per linear inch at a line speed of 20 feet/minute, using a three-roll calender. The results are tabulated in Table II.

TABLE II

EXAMPLE 1									
Calender Roll Temperatures									
Upper (°F)	70	200	225	250	275	287	300	310	320
Lower (°F)	70	147	165	185	205	206	220	230	240
Fuse-Bonded Mat Properties									
Strip Tensile (m)	2346	2250	2150	2600	3100	—	4900	5600	5600
Tear Resistance (dm²)	—	—	—	—	—	—	44	22	29
Zero Span Tensile (m)	5490	—	—	—	—	6560	—	—	7130

As shown by Table II, calender roll temperatures of 250°F. or greater were effective in increasing the strip tensile strength of nonwoven mat A. The lack of decrease, indeed, even an increase, of zero span tensile strength at the highest calender roll temperature as well as at an intermediate temperature shows that sufficient pressure was applied to prevent shrinkage of the fibers of the mat.

That the point-bonding technique is effective to increase the strip tensile strength of other nonwoven mats produced in the same manner as nonwoven mat A of Example 1 is illustrated by Examples 2–8.

EXAMPLES 2–8

Nonwoven mats B-H, made under melt blowing conditions which produce fibers that have a diameter between about 1 to 10 microns (see Table I), were point-bonded at elevated temperatures to fuse-bond the fibers of the mat. The fuse-bonding was accomplished utilizing a press wherein the temperatures of the plates of the press are separately controlled. The temperatures of the plates are set forth in Table II, the upper plate being a smooth surfaced plate and the lower plate having projections arranged in a spaced pattern. The fuse-bonding was carried out for 10 seconds, with the mats being closed to a spacing of 0.005 inches between the smooth-surfaced upper plate and the lands of the projections on the lower plate. Except for Example 6, two sheets of tissue paper were used between the nonwoven mats and the lower plate, and one sheet of foil was used between the nonwoven mats and the upper plate. Example 6 had two sheets of foil between it and the lower plate and two sheets of foil between it and the upper plate. The point-bonded nonwoven mats, as shown in Table III, exhibited high strip tensile strengths. The strip tensile strengths were greatly increased from the strip tensile strength of the untreated mat.

TABLE III

EXAMPLE NOS.	2	3	4	5	6	7	8
Mats	B	C	D	E	F	G	H
Press Plate Temperatures							
Upper (°F.)	280	290	280	275	260	275	280
Lower (°F.)	308	295	298	280	285	285	290
Fuse-Bonded Mat Properties							
Strip Tensile (m)	4849	5093	4975	4220	4180	4960	5720
Tear Resistance (dm^2)	108	52	113	127	52	70	65

Having fully and particularly described the fusion-bonded nonwoven sheet and the processes involved in this invention and having set out the best modes thereof, it will be appreciated that alterations and changes may be made by those skilled in the art which are nevertheless within the spirit and scope of the invention, as defined by the appended claims.

I claim:

1. In a battery, a positive plate, a negative plate and a nonwoven mat of polypropylene fibers, said nonwoven mat positioned between and separating said positive and said negative plates and said nonwoven mat having polypropylene fibers having diameters from about 1 to about 10 microns, said mat having fusion-bonds throughout its thickness, the strip tensile strength of the mat being greater than about 4,000 m and the tear resistance of the mat being no greater than about 3,000dm^2.

2. The battery of claim **1** wherein said fusion-bonds of said nonwoven mat occur in an essentially spaced pattern in said mat.

3. The battery of claim **2** wherein said fusion-bonds of said nonwoven mat occur continuously in an essentially unbroken pattern in said mat.

4. The battery of claim **3** wherein said mat has a thickness within the range of from about 1 to about 25 mils.

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