

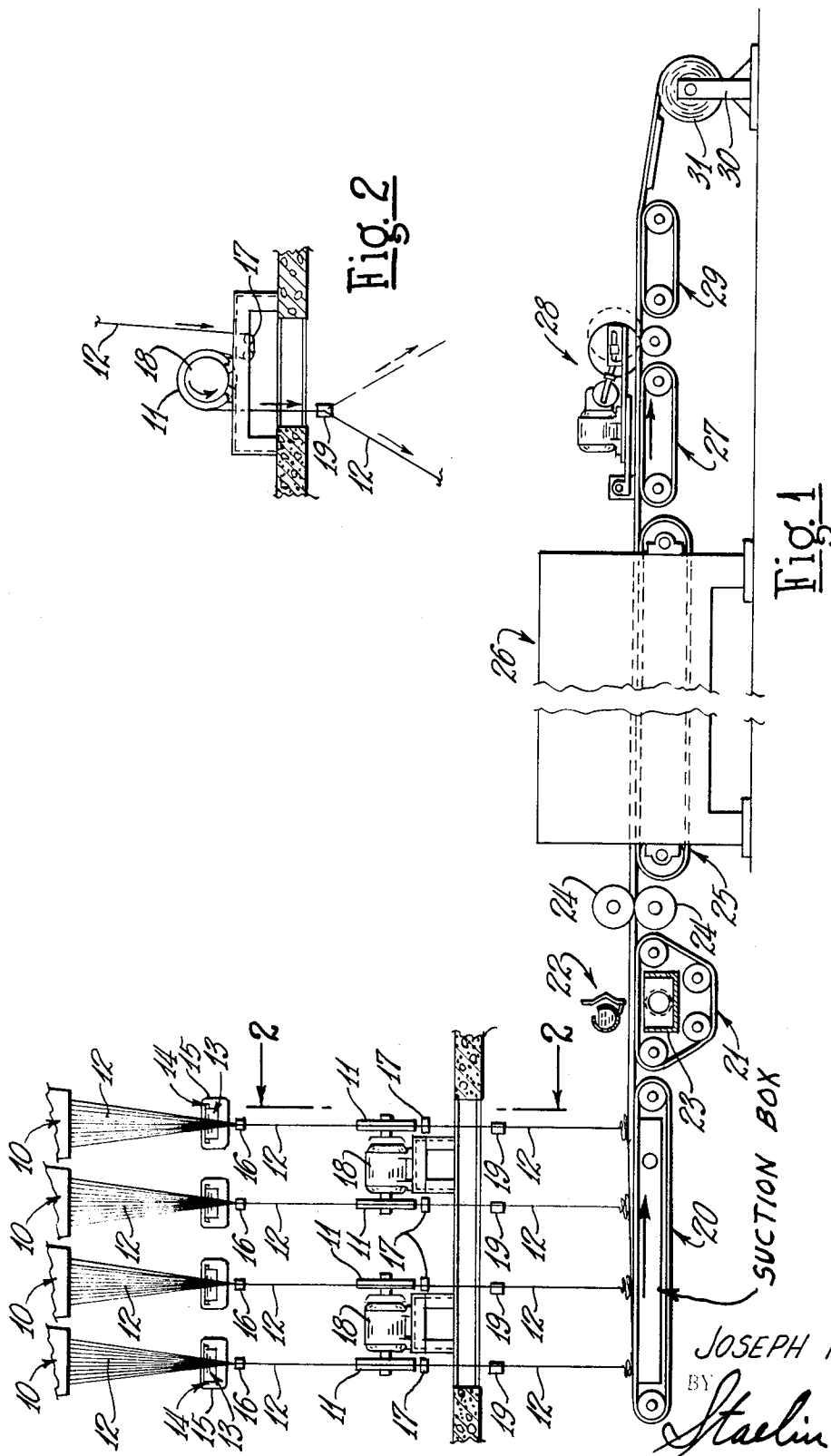
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3,676,095

PROCESS FOR PRODUCING AIR BLOWN GLASS FIBER STRAND MAT

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PROCESS FOR PRODUCING AIR BLOWN GLASS FIBER STRAND MAT

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8 Claims

ABSTRACT OF THE DISCLOSURE

A process of making a mat of glass fibers wherein a strand of glass fibers frictionally engages the surface of a revolving cylinder from which the strand is tangentially discharged onto a moving conveyor. The strand is caused to be randomly looped upon the conveyor by alternate blasts of air which laterally impinge upon the strand to throw the strand in a looped fashion from side to side across the conveyor. High uniform mat strengths are obtained by wetting the individual filaments of glass with a water solution of organosilane and a nonionic lubricant, the principal constituent of the molecules of which is a polyoxyethylene polyhydric alcohol condensate grouping.

CROSS-REFERENCES TO RELATED APPLICATION

The general type of glass fiber mat making machinery with which we are concerned is shown generally in U.S. Pat. No. 2,736,676 and application Ser. No. 530,518 filed Feb. 28, 1966, now Patent 3,599,848. The present application is a continuation application of parent co-pending application Ser. No. 693,351 filed Dec. 26, 1967, and now abandoned.

BACKGROUND OF THE INVENTION

Mats of glass fibers can be made by several different processes. In the process in which the present invention has particular advantages, molten streams of glass fibers are pulled into very fine filaments by a revolving drum over which the filaments are wrapped. The filaments are discharged tangentially downwardly from the revolving drum onto a moving conveyor which moves the mat that is laid down on the conveyor through subsequent operations where a binder is applied, and through an oven where the binder is cured. The filaments which are discharged onto the conveyor are thrown from side to side across the conveyor by the impingement of alternate lateral blasts of air. The lineal speed of the strand is many times the speed at which the strand traverses the conveyor, so that the strand assumes a looped condition on the conveyor. After the molten streams of glass are attenuated into filaments and before these filaments reach the revolving drum, they are coated with an aqueous lubricating material and are brought together in the form of a strand. This aqueous material is sometimes called a "size" and functions to prevent the fibers from breaking as they pass around guide surfaces and are pulled over the surface of the revolving drum. The size also must perform the function of preparing the surface of the fibers for proper wet-out by a binder material which is applied to the mat formed on the conveyor for the purpose of holding the fibers together.

Men working with apparatus which functions according to the process above described, have tried solutions of many materials including conventional starches, cationic lubricants, such as the reaction product of tetraethylene pentamine and stearic acid in a molar ratio of 1 to 1.8 etc. as a lubricant to protect the fibers from abrasion.

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Some of these sizes, while providing proper lubrication of the fibers, have prevented a proper random looping of the strand on the conveyor. Others have caused the fibers to wrap around the revolving drum to disrupt the process. The best of the materials that have been applied heretofore, has produced a mat whose strength crosswise of the mat was appreciably greater than the strength lengthwise of the mat. The reasons for the directional strength characteristics of the mat has not been known until the present invention.

A principal objective of the present invention has been the improvement of the above described process, be it mechanical, chemical or otherwise to achieve a high strength of the finished mat—which strength is generally the same measured crosswise of the mat and lengthwise of the mat.

SUMMARY OF THE INVENTION

According to the invention, it has been found that a mat of random swirls of glass fibers can be made, using lateral blasts of air in the equipment above described, to have high generally equal strengths in both the length and width dimensions of the mat, and provided the fibers are first wetted with an aqueous solution of a particular type of nonionic lubricant and an organosilane coupling agent. It has been found that the proper random distribution of the strand is achieved in the process above described without additional mechanical guiding action of the strand as it is laid down upon the conveyor to form the mat. Prior to the present invention, it was not known that this result was possible in a mat the fiber pattern of which is achieved with air jets alone. The solution that is applied to the fibers preferably comprise from approximately .25 percent to 2.0 percent of an organosilane coupling agent, and a nonionic lubricant of a type which will be indicated in an amount of from 1.0 percent to approximately 25 percent of the organosilane.

Using the coatings of the present invention in the process above indicated, it is found that the mat when wetted with the binder and cured, has high approximately equal strengths both lengthwise and crosswise of the mat. This high strength is attributed to a more efficient usage of the organosilane coupling agent. It appears that ether oxygen of the polyoxyethylene groups of an adduct of a polyhydric alcohol are capable of affecting a secondary bond with hydrolyzed organosilane molecules apparently through the OH groups attached to the silicon atoms. This appears to have the effect of holding the hydrolyzed organosilane molecules separated to prevent them from forming dimers, trimers and high siloxanes. Because the lubricant is nonionic, the molecules of hydrolyzed organosilane are attracted to the surface of the glass to which they attach in the monomeric state. This may occur in the presence of the water to an appreciable degree and will occur substantially completely upon drying. This increase in effectiveness of the coupling agent explains in part the higher over-all strength that is achieved with the present invention.

The fact that the mat has the same high strength both longitudinally and crosswise of the mat is believed attributed to an improved circular pattern of the strand forming the mat. It has been noticed that strand wetted with the coating solution of the present invention is a more firm strand in the finished mat than has been achieved heretofore. This is attributed to the polyhydric group which attracts water molecules to cause the size to have a high surface tension. There has also been noticed less mist being driven from the strand by the lateral jets which produce the lateral distribution of the strand across the conveyor. This is further evidence that the nonionic lubricant molecules not only hold a plurality of the silane

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molecules in spaced apart relationship, but that the lubricant molecules also help to hold water intact around the fibers. Because the strand does not separate into filaments while it is wet, the strand flexes in uniform swirls to provide more uniform fiber distribution in all directions. It has been noticed that the strand of the present invention also requires less air pressure for its traverse across the conveyer, and this is attributed to its greater retention of the water. The retention of more water gives the strand more weight and more inertia to carry it laterally across the conveyer. Improved directional uniformity of strength is therefore attributed to circular swirls whereas prior art materials produce a somewhat elliptical pattern with the major axis of the swirls being crosswise to the conveyer. The combination of the organosilane coupling agent and the polyoxyethylene polyhydric alcohol adduct therefore produces a combination of effects which include: the provision of an aqueous solution having high surface tension, to hold the fibers in a tight strand; good wetting of the fibers since a high percentage of the lubricant is hydrophilic; the provision of secondary forces for retention of the organosilane molecules in their monomeric condition; the retention of a large amount of water due to ether oxygen and/or OH groups; and the lack of interference with the attraction of the silane for the surface of the glass, since the lubricant is nonionic.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a side elevational view of apparatus capable of performing the process of the present invention;

FIG. 2 is a sectional view taken approximately on the line 2—2 of FIG. 1.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

FIGS. 1 and 2 of the drawings show apparatus which perform manipulative steps in which the present invention has particular advantages. In the apparatus shown in FIGS. 1 and 2, molten streams of glass which issue from small openings in the bottom of a molten glass tank 10 are attenuated into fibers by the pulling action of a revolving cylinder 11 over which the fibers pass. Before reaching the cylinder 11, the fibers 12 are drawn over an applicator 13 which coats the fibers with an aqueous solution. The applicator 13 shown in the drawing comprises an endless belt which passes over a revolving pulley 14 to form an arc over which the fibers are drawn. The endless belt extends down into a tank 15 of the coating solution so that a continuing supply of the coating solution is brought to the belt area contacted by the fibers. The coated or "sized" fibers are drawn over a V-shaped guide 16 which groups the fibers into a strand 12 following which they pass downwardly around the idler pulley 17 and then upwardly over the revolving cylinder 11. The revolving cylinder 11 is of the type known in Pat. 3,071,301, and is driven by a synchronous motor 18. The fibers engage approximately 180° of the cylinder surface to leave the cylinder on the opposite side from the idler roll 17 on a tangentially downward path. The strand leaving the revolving cylinder 11 passes between a pair of air jets 19, respective ones of which are positioned laterally on opposite sides of the strand. The air jets 19 are alternately supplied with air pressure which issues in a narrow stream several inches wide to blow the strand alternately from one side edge of the gathering conveyer 20 to the opposite side edge of the conveyer 20. The lineal velocity of the strand is many times the speed at which the air jets move the strand laterally, so that the strand forms randomly oriented loops while it is moving laterally from side to side across the conveyer. The conveyer 20 is formed of metal screen, and the looped strand that is formed on its top flight is compacted and held positioned by a flow of air downwardly through the top flight of the conveyer. This air flow is produced by a suitable exhaust fan, not shown.

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The mat that is formed on the endless conveyer 20 is transferred to another screen conveyer 21 where a water solution of a thermosetting binder is caused to impregnate the mat. The impregnation can be done in any manner which does not disturb the lay of the fibers, and as shown in the drawing, is performed by a stream of a binder solution which issues from a trough 22 which extends crosswise of the conveyer. The thin stream of binder solution which issues from the trough 22 flows downwardly through the mat and through the porous screen conveyer to a binder collection tray 23 from which the excess binder is pumped back into the trough 22.

The mat which leaves the conveyer 21 has fibers which are coated with a thin coating of the binder solution and which forms droplets in the area where one strand touches another strand. This mat is compressed by rolls 24 and is then transferred to another endless conveyer 25 which moves the mat through an oven 26 where the water in the mat is evaporated, and the binder is transformed into a thermoset condition. The binder in the droplets of water which previously bridge the strand at their crossover points now firmly holds the crossing strands together to give the mat strength in all directions. The mat is then transported by a conveyer 27 past a rotary cutter 28 which trims the edges. A conveyer 29 then moves the mat to a roll stand 30 where it is wound into a coiled package 31. See application Ser. No. 530,518 filed Feb. 28, 1966, now Pat. 3,599,848.

The apparatus above described has been used to produce mat using aqueous solutions of various size materials and binder materials. The following example is indicative of results achieved by a prior art size:

Example 1

A glass fiber mat was made according to the procedure above described using a fiber "size" or pretreatment material in the tank 15 of the following composition:

3,5 dimethyl-1 hexene 3-ol—5.25 grams (.525%)
Octylphenoxypoly (ethyleneoxy) ethanol—.26 gram (0.026%)
Water—994.49 grams.

The first mentioned material was purchased under the trade name "Surfynol 61," and the second material was purchased under the trade name "Igepal CA-630." These two materials were mixed together and then added to and thoroughly mixed with water at room temperature.

The mat that was laid down on the conveyer using the above pretreatment composition was then wet out by a binder solution having the following composition:

	Pounds solids
Starch (thickening agent).....	52.5 lbs..... 52.5
Bone glue (syrup).....	72 lbs..... 66.25
Dimer acid (softening agent).....	15 lbs..... 15
Phenol formaldehyde resin.....	117 lbs..... 57.5
Petroleum oil emulsion.....	15.5 lbs..... 10
Water.....	136 gallons.....

The binder composition was made by adding 120 gallons of water to a mixed tank, heating the same to 190° F., and thereafter adding the starch. The starch solution was cooled to 140° F. and the dimer acid and bone glue added. The material was thoroughly mixed and adjusted to a pH of 8.0 using ammonium hydroxide. The phenol formaldehyde resin was then added, and the pH adjusted to 8.0, following which the petroleum oil emulsion was added, and mixed for 10 minutes.

In the above composition the petroleum oil emulsion is used as a release agent for the conveyers, and the bone glue reacts with phenol formaldehyde to give a quick set resin. The dimer acid is a dimer of stearic acid sold under the trade name of "Empol 1022" by Emery Industries, Inc. The dimer is used to increase flexibility and toughness of the resin. The starch provides a thickening action for the composition and does so without inter-

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fering with the formation of the resin binder, since it is capable of forming methylene bridges with the phenolic material.

Using this binder material, the oven 25 was operated at approximately 400° F. The cylinders 11 were operated at a peripheral speed of 14,000 feet per minute and the strand produced comprised 402 filaments each having an average diameter of 0.00025 inch. The strands were caused to traverse the conveyer by alternate blasts of air supplied the nozzles and the strands were blown apart into individual filaments, i.e. fully dispersed. The finished mat was approximately .018 inch thick and weighed 2.0 pounds per 100 square feet. Test strips 3 inches wide and 14 inches long were cut from the mat made as above described and pulled apart lengthwise in a tensile testing machine. Ten strips were cut crosswise of the mat and ten strips were cut lengthwise of the mat. The ten strips which were cut lengthwise of the mat had an average breaking strength of 85 pounds, while the ten strips which were cut crosswise of the mat had an average breaking strength of 90 pounds. The piercing strength, however, is very low.

The following example demonstrates that a silane coupling agent helps to hold the strand together but causes the mat to have a strength lengthwise of the mat which is much smaller than crosswise of the mat.

Example 2

A glass fiber mat was made using the same conditions described in Example 1 above excepting that the fiber pretreatment composition had the following composition:

3,5 dimethyl-1 hexene 3-ol—400 grams (.53%)
Octylphenoxypoly (ethyleneoxy) ethanol—20 grams (.027%)
Gammaaminopropyl triethoxy silane—400 grams (.53%)
Water—20 gallons.

The mat so made when tested has an average breaking strength lengthwise of the mat of 37 pounds and an average breaking strength crosswise of the mat of 63.6 pounds.

The following test is not according to the invention and shows the effect of a nonionic lubricant.

Example 3

The process of Example 1 was repeated using a pretreatment of the following composition:

Lignin sulfonate—72 grams (.095%)
Gammaaminopropyl triethoxy silane—600 grams (.80%)
Water—20 gallons

The mats so made had an average breaking strength lengthwise of the mat of 52.4 pounds and an average breaking strength crosswise of the mat of 61.8 pounds.

The following example is not according to the invention and shows the effect of another nonionic lubricant.

Example 4

A mat was made in the same manner as described in Example 1 using the following pretreatment composition:

Dioctylsulfosuccinate—72 grams (.095%)
Gammaaminopropyl triethoxy silane—600 grams (.80%)
Water—20 gallons.

Mats made therefrom when tested in the same manner as described in Example 1 had an average breaking strength lengthwise of the mat of 60 pounds and an average breaking strength crosswise of the mat of 85.6 pounds.

The following example is not according to the invention and shows the effect of a nonionic lubricant.

Example 5

The process of Example 1 was repeated excepting that the following pretreatment composition was used:

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Octylphenylpolyethoxyethanol—72 grams (.095%)
Gammaaminopropyl triethoxy silane—600 grams (.80%)
Water—20 gallons.

The mat so made when tested had an average breaking strength lengthwise of the mat of 56.7 pounds and an average breaking strength crosswise of the mat of 81.5 pounds.

The following example is not according to the invention and shows the effect which nonionic lubricants by themselves have upon the strength of the bonded mat.

Example 6

The process of Example 1 was repeated using a pretreatment material of the following composition:

Ahco 185 AE¹—14 grams (.017%)
Ahco 185 AN²—6 grams (.007%)
Acetic acid—8.5 grams
Water—22 gallons

¹ A trademark of Arnold, Hoffman & Co. used to identify a pelargonic acid, tetraethylene and pentamine condensate solubilized with acetic acid.

² A trademark of Arnold, Hoffman & Co. used to identify a caprylic acid, tetraethylene and pentamine condensate solubilized with acetic acid.

The mat so formed when tested had an average breaking strength lengthwise of the mat of 42.4 pounds and had an average breaking strength crosswise of the mat of 58.3 pounds.

The following example is not according to the invention and demonstrates the effect which silanes or silicones have upon the strength of the bonded mat. The results show that in general silicones make the ratio of the strength widthwise of the mat relative to the strength lengthwise of the mat greater than is attributable to the nonionic lubricant itself.

Example 7

A glass fiber mat was made according to the procedure above described using a fiber "size" or pretreatment material in the tank 15 of the following composition:

Ahco 185 AE¹—14 grams (.017%)
Ahco 185 AN²—6 grams (.007%)
Gamma-methacryloxypropyl trimethoxy silane—84 grams (.10%)
Acetic acid—8.5 grams
Water—22 gallons

^{1, 2} Same as in Example 6.

The composition was prepared by adding the acetic acid to a mixture of both surfactants and diluting the same in approximately 1 gallon of water. Thereafter the silane was added and thoroughly mixed and this mixture was added to the remainder of the water. The strength lengthwise was 38 pounds and the strength across the width was 66 pounds.

The following example is according to the invention and shows that generally equal strengths are achieved lengthwise of the mat and widthwise of the mat by the unique cooperation of an aminosilane and a nonionic lubricant containing a polyethylene sorbitan grouping.

Example 8

The process of Example 1 was repeated using a pretreatment material having the following composition:

Polyoxyethylene sorbitan monostearate — 72 grams (.095%)
Gammaaminopropyl triethoxy silane — 600 grams (.793%)
Water—20 gallons

The mat made from this material when tested in the same manner described in Example 1 had an average breaking strength of 78.2 pounds lengthwise of the mat and had an average breaking strength of 81.4 pounds crosswise of the mat.

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As stated above, the generally equal strengths lengthwise of the mat and widthwise of the mat is obtained by a cooperation of the aminosilane and type of lubricant specified. This cooperation produces a generally high surface tension which holds the strand together and produces the other beneficial effects above described.

The following examples show how an improvement in surface tension is had by the present invention.

Example 9

The following material intended to duplicate that of Example 1 was prepared from the following ingredients:

3,5-dimethyl-1-hexene-3-ol—5.25 grams
Octylphenoxypoly (ethyleneoxy) ethanol—0.26 gram
Water—994.49 grams

The materials were added to the water at room temperature separately, in the order given and thoroughly mixed. This material had a surface tension of 31 dynes, and 3 grams of the solution gives 100 drops.

Example 10

A material corresponding to that of Example 8 was prepared from the following materials:

Gammaaminopropyl triethoxy silane—7.92 grams
Polyoxyethylene sorbitan monosterate—0.95 gram
Water—991.13 grams

The material is prepared by mixing the lubricant and the silane together, and then stirring the mixture into the water at room temperature until a generally clear solution is formed. This material has a surface tension of 45 dynes and 3 grams of the solution produced 60 drops.

Water has a surface tension of 72 dynes and the following example was made to show the effect which the gammaaminopropyl trimethoxy silane has upon surface tension.

Example 11

Gammaaminopropyl triethoxy silane—7.92 grams
Water—992.08 grams

The silane was added to the water at room temperature and thoroughly mixed until a generally clear solution was formed. This material had a surface tension of 65 dynes, showing that the silane has the effect when used in the quantities that are involved in the present invention, of reducing the surface tension.

The following test shows that the nonionic lubricants of the present invention by themselves have the effect of greatly reducing the surface tension of water.

Example 12

Polyoxyethylene sorbitan monosterate—0.95 grams
Water—999.05 grams

The lubricant was added to the water at room temperature until a clear solution was formed. This material had a surface tension of 37 dynes, and 3 grams of the solution gives 66 drops. It will be seen that both the silane by itself, and the nonionic lubricant by itself depresses the surface tension. Example 10, however, shows that when the materials are used together, the surface tension is greater than would be expected from the cumulative effects of each.

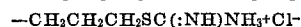
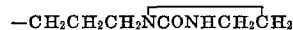
The following examples were made in the same manner as that of Example 10 excepting that an identical amount of other nonionic surface active agents having the polyoxyethylene polyhydric alcohol grouping of the present invention are used.

Nonionic surfactant	Surface tension (dynes)	Drops per 3 grams
Example:		
13..... Polyoxyethylene sorbitan hexaoleate....	45	64
14..... Polyoxyethylene sorbitan mannitan monoleate....	42	62
15..... Polyoxyethylene sorbitan tristearate....	43	69

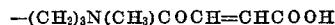
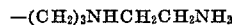
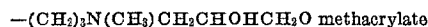
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The same surface tension test was made with a nonionic lubricant which did not contain the polyhydric alcohol grouping to show the effect which the polyhydric alcohol grouping has upon the surface tension. 95 grams of polyoxyethylene palmitate was substituted for the .95 grams of nonionic lubricant of Example 10. This material had a surface tension of 39 dynes and gave 64 drops per 3 gram sample.

The above tests show that a nonionic lubricant containing polyoxyethylene but which does not contain a polyhydric alcohol such as sorbitan, mannitan, etc. does not have the beneficial effect of the present invention upon the amino silane. Any type of tetrahydric alcohol radical containing 6 carbon atoms can be used as the intermediate portion of the lubricant molecule in cooperation with any type of aminosilane, as for example: gamma-aminopropyl trimethoxy silane, gammaaminopropyl triethoxysilane, n(trimethoxysilopropyl) ethylene diamine, n-(dimethoxymethylsiloisobutyl) ethylene diamine. The amino silanes preferably contain 3 hydrolyzable groups which may be halogens, alkoxy groups, etc. and further examples of these materials may contain the following radicals:



Lecithin



The undispersed strand of the present invention is produced with water solutions having from approximately .04 to approximately 2 percent of the silane, and from approximately .0005 to approximately 10 percent of the lubricant. The preferred materials comprise from approximately 0.25 percent to approximately 2 percent silane coupling agent, and from approximately 0.005 percent to approximately 0.5 percent of the lubricant.

The mat of the present invention produced as above described has particular advantages when it is used to reinforce asphaltic materials and/or organic resins to produce laminates. A particularly useful product is made when the mat is impregnated with asphaltic material and coated with a tack reducing particulate material to form a roofing material. The roofing material so formed has much greater strength, and is much more puncture resistant than is a roofing material made from the same weight of glass fibers, but which fibers are fully dispersed rather than being in the integral strand formed of the mat of the present invention. The integral strand mat of the present invention groups fibers at stress points to take concentrated loads such as occur when personnel walk across the roofing. Similarly, improved results are obtained when the integral strand mat of the present invention is used to reinforce organic resins, either thermoplastic resins or thermoset resins. Particularly useful composites are made by thermoset epoxy resins reinforced by the mat of the present invention, and another useful composite is obtained when a phenolic thermoset resin is reinforced by the integral strand mat of the present invention.

While the invention has been described in considerable detail, it is not intended to be limited to the particular embodiments shown and described, and it is my intention to cover hereby all novel adaptations, modifications, and arrangements thereof which come within the practice of those skilled in the art to which the invention relates.

I claim:

1. In the process of making mats of glass fibers wherein a strand of the fibers is oscillated laterally across a moving collection surface while the strand is fed to the collection surface at a rate that is greater than its rate of oscillation, the steps of: attenuating molten streams of glass into fibers, applying a water solution consisting es-

essentially of from approximately 0.04 to approximately 2% by weight of hydrolyzed aminosilane and from approximately 0.0005 to approximately 10% by weight of a nonionic lubricant composed principally of a polyoxyethylene ether condensate of a polyhydric alcohol having 4, 5 or 6 alcohol radicals and 6 carbons to the fibers, passing the fibers over a revolving wheel which pulls the fibers and delivers them downwardly in the form of a strand, causing the strand to be traversed back and forth across the moving foraminous collection surface at a rate that is a fraction of the rate of its delivery downwardly by the wheel puller, and depositing the strand upon the collection surface by air flow downwardly through the collection surface to form a mat, whereby a mat having substantially equal strength longitudinally and transversely of the mat is produced.

2. The process of claim 1 followed by the steps of: applying a water solution of a thermosetting binder forming material to the mat to wet the mat, draining excess solution from the mat, and curing the binder forming material at the crossover points of the fibers forming the mat.

3. The process of claim 1 wherein the ether is the ether of polyoxyethylene and sorbitan.

4. The process of claim 3 wherein the lubricant is an ester of a fatty acid and an ether of polyoxyethylene and sorbitan.

5. The process of claim 2 wherein the first applied solution is a water solution of gammaaminopropyl trialkoxysilane and polyoxyethylene sorbitan monostearate.

6. The process of claim 1 including the steps of: applying a binder to the mat, and curing the mat.

7. The process of claim 6 including impregnating the cured mat with an asphaltic material.

8. In the process of making mats of glass fibers wherein a strand of the fibers is oscillated laterally across a moving collection surface while the strand is fed to the collection surface at a rate that is greater than its rate of oscillation, the steps of: attenuating molten streams of glass into fibers, applying a water solution consisting essentially of from approximately 0.04 to approximately 2% by weight of hydrolyzed aminosilane and from approximately 0.0005 to approximately 10% by weight of a nonionic lubricant containing a polyoxyethylene ether condensate of a polyhydric alcohol having 4, 5 or 6 alcohol radicals and 6 carbons to the fibers, passing the fibers over a revolving wheel which pulls the fibers and delivers them downwardly in the form of a strand, causing the strand to be traversed back and forth across a foraminous moving collection surface at a rate that is a fraction of the rate delivered downwardly by the wheel puller, and depositing the strand upon the collection surface by air flow downwardly through the collection surface to form a mat having substantially equal strength longitudinally and transversely thereof.

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