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# 2,318,560

# UNITED STATES PATENT OFFICE

### 2,318,560

### ASBESTOS YARN

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#### 7 Claims. (Cl. 117-126)

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This invention relates to asbestos yarn, and more particularly to increasing the strength thereof in order that it may be woven or in order to increase its utility for varied purposes.

Asbestos yarn has very little tensile strength and it has a high degree of elongation when subjected to tensile stress. For these reasons it is very difficult and in fact very nearly impossible to weave asbestos yarn which does not contain some other fibers or optionally fine metal wires 10 to give it strength. Previously it has been customary to mix about 10% of cotton fibers with asbestos fibers in spinning the yarn and then weaving the cloth or the like from this yarn. This is not only troublesome and expensive but 15 the resulting product containing the cellulosic fibers is not as heat resistant as if it contained only the asbestos fibers.

An object of the present invention is to increase the strength of asbestos yarn.

Another object of this invention is to provide an asbestos yarn containing no other fibers and one which can be woven without including therein any other reinforcing materials such as wire.

These and other objects are attained by impregnating asbestos yarn with a thermosetting resin which may be polymerized under alkaline conditions to the infusible and insoluble stage.

The following examples in which the proportions are in parts by weight are given by way 30 of illustration and not in limitation.

#### Example 1

Asbestos yarn is impregnated by passing the yarn through a melamine-formaldehyde resin syrup (Resin "A") diluted with ethanol in the ratio of 25 parts of the syrup to about 59 parts of ethanol. The excess resin, if any, may be removed by squeezing and then the yarn is dried at room temperature for about 1/2-2 hours. The dried yarn is placed in an oven at 75-80° C. for about 55 minutes to polymerize and insolubilize the resin. The impregnated yarn contains about 31% of resin solids (including about 6.7% of volatile material) based upon the total weight of 45 yarn and resin. In determining the volatile material a sample is weighed, heated at about 150° C. for 25 minutes and the loss in weight expressed in per cent of the weight of the original sample is the value given as the volatile mate- 50 rial. The impregnated asbestos yarn has an average breaking strength of about 8.6 pounds as compared to an average breaking strength of about 1.8 pounds for the untreated asbestos yarn. only about 3.6% as compared to about 7.2% for the untreated yarn. These values are much better than those obtained with asbestos yarn including about 10% of cotton fibers which have a breaking strength of about 2.7 pounds and an 60 5.1%.

elongation of about 8.2%. Thus, the yarn produced according to this example is suitable for weaving into various types of textile materials or it may be used in the production of twisted cords, braids, etc. Products prepared from my high 5 strength yarn have much higher strength than those produced from the ordinary asbestos yarn reinforced with cotton fibers, it being virtually impossible to compare such finished products with those produced from asbestos yarn which is not reinforced since the latter is practically unobtainable.

#### Example 2

Asbestos yarn is impregnated with a melamine-formaldehyde resin syrup (resin "A") diluted with ethanol in the weight ratio of about 25:40, dried and polymerized in the same manner as described in Example 1. The impregnated yarn contains about 43% of resin solids includ-20 ing 6.4% of volatile material. The average breaking strength of the impregnated yarn is about 8 pounds and the average elongation about 3.6%.

#### Example 3

Asbestos yarn is impregnated with a melamineformaldehyde resin syrup (resin "A") diluted with ethanol in the weight ratio of about 5:4, the excess resin, if any, removed, the impregnated yarn dried at room temperature and polymerized at 75-80° C. for about 55 minutes. The

impregnated yarn contains about 55% of resin solids including about 9% of volatile material. The average breaking strength of the impreg-35 nated yarn is about 10.8 pounds and the average elongation about 5.2%.

#### Example 4

Example 1 is repeated substituting a melam-40 ine-modified dicyandiamide-formaldehyde resin syrup (resin "B") for the melamine-formaldehyde syrup used in the former example. The impregnated material after polymerization contains about 40% of resin solids including 6.5%of volatile material and it has an average breaking strength of about 9.8 pounds and an average elongation of about 4.1%.

#### Example 5

Example 2 is repeated substituting a melamine - modified dicyandiamide - formaldehyde resin syrup (resin "B") for the melamine-formaldehyde resin syrup employed in Example 1. The treated yarn has an average elongation of 55 The impregnated yarn after polymerization of the resin contains about 37% of resin solids including about 5.7% of volatile material. The yarn has an average breaking strength of about 9.6 pounds and an average elongation of about

A melamine-modified dicyandiamide-formaldehyde resin syrup (Resin "B") is substituted for the melamine-formaldehyde resin syrup used in Example 3, the procedure of the latter example being otherwise followed. The impregnated yarn after polymerization contains about 50% of resin solids including about 7.5% of volatile material, has an average breaking strength of about 7.1 pounds and an average elongation of about 6.5%.

#### Example 7

Asbestos yarn is impregnated with a phenolformaldehyde resin syrup (resin "C") diluted with ethanol in a weight ratio of about 40:110. 15 The excess resin, if any, is squeezed out of the yarn, the impregnated material dried at room temperature for  $\frac{1}{2}$ -2 hours and polymerized in an oven at about 95° C. for approximately 75 minutes. The impregnated yarn after polymeriza- 20 tion of the resin contains about 34% of resin solids together with about 3% of volatile material. The average breaking strength of this impregnated yarn is about 12.7 pounds and the average elongation about 3.2%.

#### Example 8

Example 7 is repeated except that the phenolformaldehyde resin syrup is diluted with ethanol in a weight ratio of about 1:5. The impregnated 30 yarn after polymerization of the resin contains about 22.5% of resin solids including about 2.1% of volatile material, it has an average breaking strength of about 9.1 pounds and an average elongation of about 4.3%.

#### Example 9

The procedure of Example 7 is followed substituting as an impregnating medium a phenolformaldehyde resin syrup (resin "C") di- 40 luted with ethanol in a weight ratio of about 1:2. The impregnated yarn after polymerization of the resin contains about 47% of resin solids including about 3.7% of volatile material. This yarn has an average breaking strength of about 10.5 pounds and an average elongation of about 3%.

The values given in the above examples for the tensile strength and the elongation at the breaking point are obtained by testing the yarn on a 50 Scott "IP-3 inclined plane tensile testing machine."

Resin "A" may be prepared by reacting melamine and formaldehyde in any suitable manner. The particular resin designated as resin "A" used 55 in the preceding examples may be prepared by the following procedure in which the parts are by weight. About 2.4 parts of a 10% solution of triethanolamine are added to 240 parts of formalin (an aqueous solution containing 37% of 60 formaldehyde by weight). 126 parts of mel-amine are added to the formaldehyde and the mixture is reacted at about 65° C. for approximately 70 minutes, thereby producing a resin syrup which may be diluted with ethanol in ac-65 cordance with Examples 1-3.

Resin "B" may be prepared by reacting dicyandiamide and a minor proportion of melamine with an aldehyde such as formaldehyde to produce a clear syrup as described in my co- 70 pending applications Serial Nos. 311,935, 328,741 and 331,161. Resin "B" may also be prepared as described in my copending application Serial No. 351,915. Briefly the process involved in the latter application comprises refluxing a slightly 75 when phenol-formaldehyde resins are used as

acid mixture containing melamine and formaldehyde in the ratio of more than six mols of formaldehyde per mol of melamine and reacting the hydrophilic sol thus produced with dicyandiamide and formaldehyde with or without additional melamine.

Resin "C" may be prepared by reacting phenol and formaldehyde, preferably by the so-called "one step" procedure. The particular resin used 10 in Examples 7-9 may be prepared by refluxing for about 10 minutes 94 parts by weight of phenol, 110 parts by weight of formalin (an aqueous solution containing 37% formaldehyde by weight) and 3.6 parts by weight of a 25% aqueous solution of ammonia. After the refluxing operation the mixture is distilled under vacuum until the residue amounts to about 130 parts by weight, thereby producing a highly viscous syrup which may be diluted with ethanol as indicated in Examples 7-9.

Any other thermosetting resin which may be cured under alkaline conditions may be used in the same general manner as described above. Since urea-formaldehyde resins are hardened under acid conditions rather than alkaline con-25 ditions they are not suitable for use with asbestos inasmuch as the latter renders an acid catalyst ineffective. The asbestos generally used in the production of yarns, threads and cloth, etc., is the mineral "chrysotile" having the formula, H4Mg3Si2O9 or 3MgO.2SiO2.2H2O with a ratio of alkali to acid 3:2. This asbestos will neutralize an acid catalyst and therefore cannot be used with the urea-formaldehyde type of resinous materials which cure under acid conditions. Ex-35 amples of suitable resins which cure under alkaline conditions are: various aminotriazine-aldehyde resins such as melamine-formaldehyde resins, dicyandiamide-formaldehyde resins, meldicyandiamide-formaldehyde amine-modified resins, the phenol-aldehyde resins such as phenol-formaldehyde resins, cresol-formaldehyde resins, etc. The resins suitable for the purposes of this invention may be modified with such proportions of other amino plastic materials including urea and thiourea as will not affect the alkaline curing properties of the resin. The amino plastic resins may also be modified with any of the phenols. Furthermore, mixtures of any of the compatible resins which may be cured under alkaline conditions may be employed.

The amino plastic resins may be utilized not only in aqueous solutions but in solutions in organic solvents, particularly alcohols. Furthermore, the amino plastic resins may be alkylated with (i. e., reacted with) any suitable alcohol. e. g., methyl alcohol, ethyl alcohol, propyl alcohol, butyl alcohol, the amyl alcohols, cyclohexanol, benzyl alcohol, the monoethyl ether of ethylene glycol, the monoethyl ether of diethylene glycol, other monoalkyl and monoaryl ethers of ethylene glycol and diethylene glycol, 2-ethyl hexanol, n-octyl alcohol, lauryl alcohol, etc. The water miscible alkylated amino plastic resins, e.g., methylated melamine-formaldehyde resins, may be applied to the asbestos yarn in the form of aqueous solutions.

Any suitable alkaline or potentially alkaline curing catalyst may be used but ordinarily the alkalinity of the fiber will be sufficient to effect the polymerization of the resin.

Somewhat greater difficulty is encountered in obtaining uniform and reproducible results compared to the results obtained when resins such as melamine-formaldehyde resins are used. Furthermore, phenol-formaldehyde resins are not as flame resistant as melamine-formaldehyde resins and accordingly are not as well 5 suited for use with asbestos since in most instances the utility to which asbestos is put is one wherein resistance to high temperatures and inflammability is not only desirable but a requirement. Accordingly, for most purposes where 10 heat resistance and/or inflammability are necessary in the ultimate product, it is preferable that melamine-formaldehyde resins be used.

As it will be apparent from the preceding examples it appears that the optimum resin content of the impregnated yarn is about 30-40% of resin solids inasmuch as the strength of the resulting product begins to fall off when the resin solids content reaches about 50%. It appears that it is desirable to have at least about 2025% of resin solids in the impregnated asbestos yarn in order to obtain optimum strength but smaller proportions may be used if somewhat lower strengths are sufficient and if economic factors restrict the amount of resin which may 25

The asbestos thread which has been impregnated with a suitable resin syrup may be dried at ordinary room temperatures or by subjecting it to a temperature of about 50-100° C. for 30 a period of about 1/2-4 hours. During the drying operation the resin will be polymerized to some extent even at room temperature and may be substantially completely insolubilized if the temperature is near the high end of the range 35 specified above. In some instances it may be advantageous to subject the impregnated asbestos thread to a preliminary drying operation, for example, at room temperatures and then to an elevated temperature such as 75-80° C. in 40 order to polymerize the resin to the desired degree. For most purposes it is desirable that the resin be polymerized to the insoluble stage. If the yarn is to be used in the manufacture of molding compounds the resin should be only par- 45 tially polymerized. Where a minimum quantity of resin in the finished product is required or is advantageous, the resin may be only partially polymerized and then after the yarn is woven, part or all of the soluble resin may be removed 50 by washing with suitable solvent materials, e. g., water, alcohol, etc. The resin remaining in the yarn may then be polymerized further by subjecting it to elevated temperature.

The impregnating of the asbestos yarn may 55 take place immediately following the spinning thereof. Thus, before being wound on spools the asbestos thread may be passed through a bath of resin syrup, optionally passed through squeeze rolls and then dried either by means of hot rollers or by merely carrying the impregnated yarn through a heated drying compartment. The drying compartment or the rolls may be heated to progressively increasing temperatures in order that the polymerization of the resin may be effected or the temperature may be adjusted to such a point as to simultaneously dry and polymerize the resin.

The asbestos yarn used in the preceding examples is essentially a pure asbestos fiber, the 70asbestos content being about 98-99%, the balance being minor proportions of inorganic impurities. As indicated heretofore, the primary purpose of my invention is to strenthen asbestos yarn in such a manner that it does not require 75

any reinforcing materials. However, my invention is not restricted to pure asbestos yarn since in many instances the ultimate product does not necessarily have to have extremely high heat resistance or an extremely high degree of inflammability in which case minor proportions of other fibers may be included. Similarly, in those applications where high heat resistance and inflammability are required, it may be desirable to reinforce my impregnated asbestos yarn with fine wires or the like in order to produce a product having exceptionally high strength.

The resin syrups used for impregnating the asbestos yarn may contain suitable wetting agents in order to get better impregnation and the syrup may also contain compatible textile sizing materials and compatible textile softeners.

My impregnated asbestos yarn may be used not only for weaving into cloth but it may also be used for the production of molding compositions in accordance with my application Serial No. 378,740, entitled "Resinous molding compositions," filed February 13, 1941, and as to which the present application is a continuation-inpart.

Materials woven from my asbestos yarn are useful as electrical insulation, in the fabrication of flame proof clothing, and other apparel such as gloves, mittens, etc., as well as in fabrication of fire proof curtains such as for use in threaters. All of these articles have increased strength and do not stretch as much as those produced from ordinary untreated asbestos yarn.

Obviously many modifications and variations in the processes and compositions described above may be made without departing from the spirit and scope of the variation as defined in the appended claims.

I claim:

1. Asbestos yarn impregnated with a thermosetting resin which may be polymerized under alkaline conditions, said impregnated yarn having high mechanical strength.

2. Asbestos yarn impregnated with a polymerized melamine-formaldehyde resin, said impregnated yarn having high mechanical strength.

3. Asbestos yarn impregnated with a polymerized melamine-modified dicyandiamide-formaldehyde resin, said impregnated yarn having high mechanical strength.

4. Asbestos yarn impregnated with a polymerized phenol-formaldehyde resin, said impregnated yarn having high mechanical strength.

5. In a process of increasing the mechanical strength of asbestos yarn, the step which includes impregnating asbestos yarn with a thermosetting resin which may be polymerized under alkaline conditions.

6. A process of increasing the mechanical strength of asbestos yarn which includes impregnating asbestos yarn with a thermosetting resin syrup which may be polymerized under alkaline conditions, drying the impregnated yarn and polymerizing the resin.

7. A process for increasing the mechanical strength of asbestos yarn which includes impregnating yarn essentially composed of asbestos fibers with a melamine-formaldehyde resin syrup, drying the yarn and polymerizing the resin.

KURT E. RIPPER.

# CERTIFICATE OF CORRECTION.

May 4, 1943.

Patent No. 2,318,560.

## KURT E. RIPPER.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 3, second column, line 32, for "threaters" read --theaters--; line 38, for "variation" read --invention--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 8th day of June, A. D. 1943.

Henry Van Arsdale, Acting Commissioner of Patents.

(Seal)