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Sone et al.

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[54] **ACRYLIC FIBER PRODUCT HAVING ANIMAL HAIRY HAND**

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[51] **Int. Cl.^{2}**... **D02G 3/04;** D02G 3/36; D02G 3/40

[58] **Field of Search** .... 117/138.8 UA, 139.5 A. 117/141, 161 ZA; 106/287 SB; 161/175, 176; 57/140 BY

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[57] **ABSTRACT**

Acrylic fiber product made by mix-spinning animal hair fibers, nonshrinkable acrylic fibers having a silicone resin deposited thereon, and shrinkable acrylic fibers having a latent shrinkage at least 3% higher than that of the nonshrinkable acrylic fibers, and heat-treating the resultant article.

3 Claims, 2 Drawing Figures

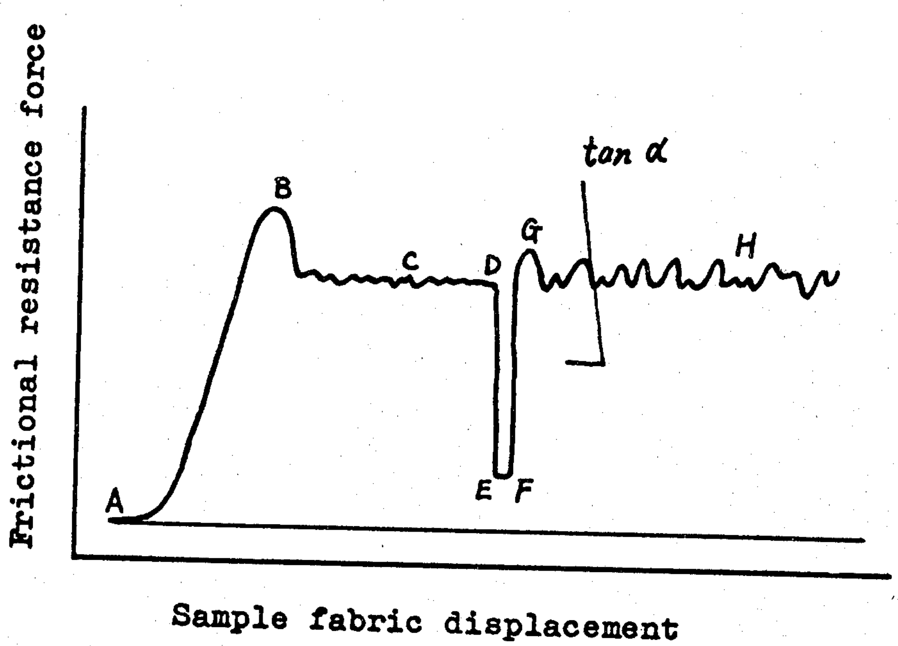

Fig. 1

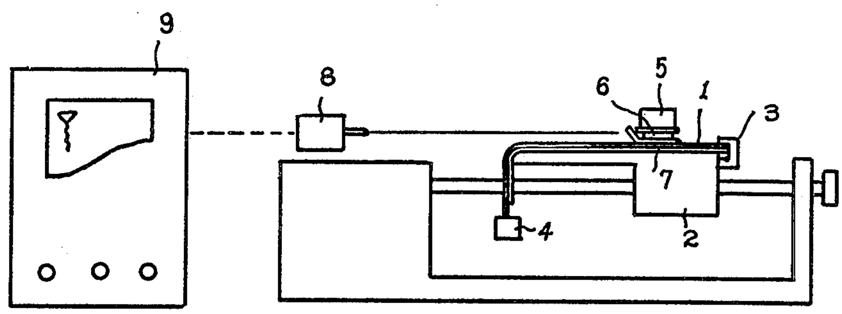
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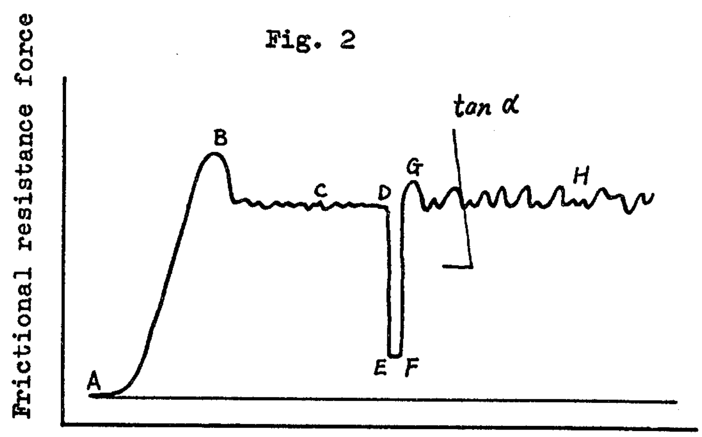
Fig. 2


Frictional resistance force
tan α
B
C
D
G
H
A
E
F
Sample fabric displacement

ACRYLIC FIBER PRODUCT HAVING ANIMAL HAIRY HAND

This invention relates to novel acrylic fiber products having an animal hair-like hand and more particularly to an acrylic fiber product having an animal hair-like hand produced by forming mix-spun yarns of such animal hair fibers as wool or cashmere, nonshrinkable acrylic fibers having a specific type of silicone resin deposited on their surfaces and highly shrinkable acrylic fibers having a latent shrinkage at least 3 % higher than said nonshrinkable acrylic fibers, and then heat-treating the yarns.

It is well known that a knit or woven fabric made of such animal hair fibers as wool, cashmere or Angora rabbit hairs has an excellent hand based on a peculiar slippery touch. However, there are recognized defects in a knit or woven fabric made of such animal hair fibers. For example, there has been recognized a defect that, due to a scaly structure on the fiber surface, by such treatment accompanied with a tumbling action as washing, a peculiar movement having a directivity is caused between the fibers forming the knit or woven fabric to shrink the fabric. In fact, there has been already attempted a method wherein a chemical solution is made to act on animal hair fibers or a knit or woven fabric made of said fibers so that the scaly structure of the fiber surface may be destroyed and an antishrinkability may be imparted to eliminate such defect. It is true that the antishrinkability of a knit or woven fabric made of animal hair fibers is improved by such chemical solution treatment. However, by such method, there have been caused such disadvantages that the hand or particularly the slippery touch of the final product is remarkably reduced and its commodity value is greatly reduced.

On the other hand, in order to eliminate such defects of the conventional technique and to improve the dimensional stability at the time of washing, there is generally used a method wherein a knit or woven fabric is formed by mix-spinning synthetic fibers having no scaly structure on the such surfaces as acrylic fibers with animal hair fibers. However, although in such mix-spun knit or woven fabric, some improved effect is seen in the dimensional stability in the tumbling action, the level of the slippery touch contributing directly to the maintenance of the animal hair-like hand is inherently low, and naturally the result of the sensory evaluation of said slippery touch is shown to be quite different from that of an animal hair fiber knit or woven fabric.

There is also known a method wherein, aside from an attempt to use the above mentioned animal hair fibers as materials for forming knit or woven fabrics, in order to give a slippery touch to synthetic fibers or yarns or knit or woven fabrics obtained from said fibers, a polyhydric alcohol type nonionic surface active agent or a specific cationic surface active agent or anionic surface active agent is deposited on synthetic fibers. However, although by the surface treatment with such surface active agent, some slippery touch is imparted to the synthetic fibers, the slippery touch caused by such surface active agent is recognized to have no durability to washing at all and quickly reduces the commodity value of said fiber products. It has been strongly desired in the industry to find a way to solve such defects.

Further, it is mentioned in Japanese Pat. Publication No. 27520/**1969** or 28733/**1970** to use a higher ester of acrylic acid or methacrylic acid or a vinyl ester of a

higher fatty acid as a fiber treating agent. However, the slippery touch given by such fiber treating agent is quite different from the slippery touch seen in animal hair fiber products and it is generally difficult to expect the effect of improving the hand of the final product with such fiber treating agent.

Further, it is mentioned in British Pat. No. 1,111,880 or Japanese Pat. Publication No. 26436/**1969** to improve the hand of acrylic fiber products by processing the acrylic fibers with a silicone resin. However, in the former, no definite knowledge on the silicone resin to be used as a fiber treating agent is disclosed and the hand of the acrylic fiber product improved by such silicone resin process is not a slippery touch but rather only an improvement in the softness or antipilling property. In the latter, since a mixture of a silicone resin and epoxy resin is used as a treating agent for acrylic fiber products, the hand of the final product will become rough and hard due to the bonding and hardening of the fibers and, as the slippery touch imparted by the silicone resin is barred by the epoxy resin, the commodity value of the final product will greatly reduce.

The present inventors have endeavored to find an industrial process of imparting a slippery touch, very similar to that of an animal hair fiber products and an antishrinkability to mix-spun yarns of acrylic fibers and animal hair fibers or to knit or woven fabrics obtained from said mix-spun yarns, which would totally eliminate such defects of the conventional technique. As a result, they have reached the present invention by discovering the fact that an acrylic fiber product remarkably improved in dimensional stability, even in such tumbling operation as in washing, and having a permanent slippery touch can be produced by depositing a specific type of silicone resin on the surfaces of acrylic fibers mix-spun with animal hair fibers and adjusting the monofilament fineness and latent shrinkage.

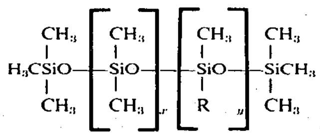
A main object of the present invention is to provide a novel acrylic fiber product having an animal hair-like hand.

Another main object of the present invention is to provide an industrial process in imparting an animal hair-like slippery touch and, dimensional stability during washing treatment to acrylic fiber products.

Another object of the present invention is to provide a novel acrylic fiber product wherein the structural characteristics of a silicone resin to be deposited on the surfaces of acrylic fibers forming yarns or knit or woven fabrics, and the monofilament fineness and latent shrinkage of said acrylic fibers, are maintained in a critical range favorable to the improvement of the slippery touch by mix spinning acrylic fibers with animal hair fibers.

Other objects of the present invention will become clear from the description of the following specification.

These objects of the present invention can be effectively attained by providing an acrylic fiber product made by forming yarns or knit or woven fabrics by mix-spinning animal hair fibers, nonshrinkable acrylic fibers having a monofilament fineness which is 50 to 100 % of the average monofilament fineness of said animal hair fibers and having had a silicone resin defined by the structural formula (1) deposited in an amount of 0.1 to 3.0 % based on the dry weight of the fibers on the surfaces thereof and highly shrinkable acrylic fibers whose latent shrinkage is higher by at least 3 % than that of said nonshrinkable acrylic fibers, and then heat-treating them.



(1)

(wherein R is $R'NH_2$, $R'NHR''$ or $R'NHR''_2$, R' is CH_2, n is 1 to 3, R'' is C_mH_{2m+}, m is 1 to 3, x and y are positive integers and the molecular weight of the silicone resin is less than 100,000).

The invention will be further explained below in detail by referring partly to the accompanying drawings wherein:

FIG. 1 is an explanatory view of a fabric friction measuring apparatus to be used to measure slippery touch; and

FIG. 2 is an orthogonal coordinate diagram exemplifying the relation between the frictional resistance force recorded by said measuring apparatus and the sample fabric displacement.

The animal hair fibers used in the present invention are generally such animal hair fibers as wool, mohair, alpaca, cheviot, cashmere or Angora rabbit hairs. The acrylic fibers are generally fibers consisting of an acrylonitrile polymer or of a copolymer of more than 70 % by weight acrylonitrile and other vinylic monomers.

In the process of depositing the silicone resin defined by the structural formula (1) on the surfaces of nonshrinkable acrylic fibers in working the present invention, the amount of said silicone resin deposited is not critical, and it is also possible to deposit said silicone resin on the surfaces of the fibers after having been heat-stretched, while in a swollen gel state containing less than 100 % by weight of water or after being heat-relaxed, mechanically crimped or dried. However, in order to strengthen the deposition of said silicone resin on the fibers to be treated and to give a permanent slippery touch, it is preferable to dip the fibers, while they are in a swollen gel state containing a fixed amount of water, in an emulsion prepared by dispersing said silicone resin in such emulsifier as a POE alkylphenyl phosphate, so that the silicone resin may be deposited in an amount of 0.1 to 3.0 % or more preferably 0.5 to 2.0 % based on the dry weight of the fibers on the surfaces of the fibers.

In working the present invention, in case the amount of silicone resin is less than 0.1 % the slippery touch of the nonshrinkable acrylic fibers will not sufficiently improve and no animal hair-like hand will be imparted to the final product. In case the amount of the silicone resin deposited on the surfaces of the fibers exceeds 3 % on the dry weight of the fibers, the monofilaments will stick to each other and will roll up on the taker-in roller of the carding machine in the spinning step. This of course is not desirable.

It is necessary that the monofilament fineness of the nonshrinkable acrylic fibers on which the silicone resin defined by the structural formula (1) is to be deposited should be maintained in a range of 50 to 100 % or more preferably 50 to 80 % of the monofilament fineness of the animal hair fibers to be mix-spun with said acrylic fibers. When such monofilament fineness range is selected for the nonshrinkable acrylic fibers, the bending hardness of said nonshrinkable acrylic fibers will become substantially equal to that of the animal hair fibers and, by the multiplied action of said acrylic fibers having had the silicone resin deposited on the surfaces to form the surface part of the yarn, the hand or particularly the slippery touch of the final fiber product will be remarkably improved.

On the other hand, the highly shrinkable acrylic fibers whose latent shrinkage is higher by at least 3 % than that of the above mentioned nonshrinkable acrylic fibers are generally fibers obtained by restretching acrylic fiber tows in an ordinary manner with a turbostapler or the like under the action of wet heat or dry heat. So long as they satisfy the above mentioned favorable range of the latent shrinkage, they may be monocomponent fibers obtained from an acrylonitrile homopolymer or may be composite fibers made by eccentrically bonding, in the axial direction of the fibers, two kinds of acrylonitrilic polymers different from each other in thermoshrinkability or gel-swellability. Further, the restriction on the upper limit of the latent shrinkage of the highly shrinkable acrylic fibers, is not as strict as on the lower limit. However, in order to impart a hand very similar to that of animal hair fibers and to improve the uniformity of the meshes, it is desirable to maintain the latent shrinkage below 15 %.

In the case of making mix-spun yarns of animal hair fibers and acrylic fibers, it is desirable to maintain the blend ratio of the animal hair fibers to 10 to 70 % or more preferably 20 to 50 % of the total weight of the mix-spun yarn. Further, in order to make it easy to develop a latent shrinkability, it is desirable to mix-spin the highly shrinkable acrylic fibers in an amount of at least 20 % or more preferably at least 30 % by weight. In order to maintain the bulkiness of the final product on a fixed level, it is recommended to maintain the upper limit of the blend ratio of said highly shrinkable acrylic fibers to less than 70 % or more preferably less than 50 %.

When the thus obtained mix-spun yarn is heat-treated, the latent shrinkability will develop, the highly shrinkable acrylic fibers will be positioned in the center part of the yarn and the loosely crimped animal hair fibers and acrylic fibers having had the silicone resin deposited on the surfaces will be arranged around them to remarkably improve the slippery touch of the final product. The present inventors have confirmed the fact that the slippery touch of a knit or woven fabric can be quantitatively determined by the frictional characteristic between knit or woven fabrics or particularly the stress reduction rate tan α at the time of the kinetic friction as measured with the cloth friction measuring apparatus exemplified in FIG. 1, and have disclosed that, though somewhat different depending on the construction or structure of the knit or woven fabric, when said tan α is in a range of 5 to 25 g./min., a slippery touch very similar to that of a knit or woven fabric made of animal hair fibers will be imparted to the acrylic fiber product. According to the results of experiments made by the present inventors, the frictional characteristic of a knit or woven fabric having no slippery touch shows a clear stick-slip wave form and its hand characteristic can not be indicated as the stress reduction rate tan α at the time of the kinetic friction. In order to impart an animal hair-like slippery touch to an acrylic fiber product by reducing tan α, it is necessary that the nonshrinkable acrylic fibers positioned on the outside of the yarn should have a frictional characteristic similar to that of animal hair fibers. In this sense, the amount of the silicone resin deposited on the nonshrinkable acrylic fibers, or the monofilament fineness of said fibers, has a very critical significance.

By the way, in the present invention, the stress reduction rate tan α at the time of the kinetic friction for quantitatively indicating the slippery touch, the hot water shrinkage, the average fineness, the amount of deposition of the silicone and the washing shrinkage are measured by the following methods.

1. Stress reduction rate tan α at the time of the kinetic friction:

It is determined by magnifying and measuring the kinetic frictional force between sample fabrics by using the fabric friction measuring apparatus shown in FIG. **1.** It shall be explained more particularly by also using FIG. **2.** A sample fabric **1** is mounted on a sample table **2** in a humidity adjusted atmosphere at 20°C. under a relative humidity of 65 %, is fixed at one end with a sample presser **3,** has a load **4** to 30 g. act on the other end and is thus kept tensioned. A slider **6** of an effective contact area of 3 cm.^{2} (2 cm. × 1.5 cm.) on which a compression load **5** of 450 g. is made to act is mounted on the sample fabric **1.** A sample fabric piece **7** is fixed to the lower surface of the slider **6.** The sample table **2** is thus moved at a constant velocity of 12 mm./min. and the frictional force produced between the sample fabrics is detected with a resistance wire strain meter **8** connected with the slider **6** and is recorded with a recorder **9.** In the case of the measurement, when the kinetic frictional force shows a constant state, the indicator of the recorder is shifted to zero point and then, as shown at the points F and G, the detection sensitivity is magnified to 5 to 10 times as high to magnify and measure the slight variation of the kinetic frictional force. The stress reduction rate tan α at the time of the kinetic friction means the gradient of the stress reducing part in which a slip occurs between the sample fabrics in the magnified measurement graph and can be indicated as a stress reduction rate per mm. of the displacement of the sample fabric. Therefore, it is apparent that the smaller the value of tan α shown in FIG. **2,** the larger the slippery touch.

2. Washing shrinkage:

Two sheets of sample fabric of a length of 50 cm. on one side are prepared, have a circle of a diameter of 20 cm. described in the center of each of them, are then put into a domestic washing machine (of a vortex type) together with 1 g./liter of Monogen Uni (detergent), are washed for 5 minutes while maintaining a liquor ratio of 50:1 and are then rinsed for 7 minutes. Then the sample fabrics are taken out and spread on a table without being dehydrated and are naturally dried. Then the diameters in the longitudinal direction and lateral direction of the circle described in each sample fabric are measured to determine the average values of the shrinkages in the longitudinal direction and lateral direction.

3. Silicone resin deposition rate:

Several-kinds of organic solvent phases in which the concentration of the same kind of silicone resin as the silicone resin to be deposited on the sample fibers is varied are first prepared and intensities of the infrared absorption at 800 cm.$^{-1}$ of these organic solvent phases are determined with the infrared spectro-photometer Model 521 manufactured by Perkin Elmer Co. Then a calibration line showing a relation between the amount of the silicone resin and the intensity of the infrared absorption of the groups, $Si—CH_3$ and $Si—(CH_3)_2$, at 800 cm.$^{-1}$ is determined.

Then the acrylic fibers to be tested are cut to a length of 0.1 to 0.3 mm., 3 mg. of them are mixed with 200 mg. of potassium bromide and the mixture is further mixed and ground in an ordinary manner and is then molded into tablets (sample A). Further, tablets of acrylic fibers having no silicone resin deposited on them are made in the same manner (sample B).

Then, the sample A is placed on the sample side and the sample B on the compensating side of the infrared spectro-photometer Model 521 and the intensity of the infrared absorption at 800 cm.$^{-1}$ is measured. The amount of the silicone resin deposited on the sample A is obtained from the intensity thus measured and the calibration line determined previously.

4. Hot water shrinkage:

A fiber bundle consisting of about 10 acrylic fibers and having a latent shrinkability is bonded at both ends, has the distance between both ends fixed to a fixed length (L_0), is treated in hot water at 100°C. for 15 minutes, is then cooled to below 60°C., is then taken out, is dried in a hot air dryer at 80°C. for 30 minutes and is left for more than 1 hour in a chamber in which the humidity is adjusted to a relative humidity of 65 % at 20°C. Then the length (L_1) of the shortest fiber in the fiber bundle is measured and the shortest acrylic fiber only is cut. Then the length (L_2) of the second shortest sample fiber is measured and said fiber is cut in the same manner. By repeating the same operation, the lengths of all the sample fibers forming the fiber bundle are measured. **5** of such fiber bundles are prepared, the lengths (L_1, L_2, . . . L_{50}) after hot water treatment of a total of **50** sample acrylic fibers are determined and the hot water shrinkages of the sample fibers are calculated by the formula:

$$\text{Hot water shrinkage } (\%) = \mathbf{100}\,(L_1 + L_1 = \ldots L_{50})/(\mathbf{50} \times L_o)$$

5. Average fineness (monofilament deniers):

According to the positive quantity fineness method A of JIS L-1074.

An example of the present invention is mentioned in the following but the present invention is not limited thereby.

In the example, the parts and percentages are all by weight unless otherwise specified.

EXAMPLE

A spinning solution, obtained by dissolving in an aqueous solution of sodium thiocyanate an acrylonitrile copolymer obtained by copolymerizing 9.8 % methyl acrylate and 0.2 % sodium methallylsulfonate with 90 % acrylonitrile, was wet-spun into cold water and was then water-washed and stretched in an ordinary manner to prepare a swollen gel fiber tow of a water content of 80 %. This fiber tow was dipped for 3 seconds in an emulsion prepared by emulsifying and dispersing 2 % silicone resin of the structural formula (1) wherein R is CH_2NH_2, 1 % POE (9) nonylphenyl phosphate and 0.2 % catalyst Sumitex Accelerator SX - 70A, produced by Sumitomo Chemical Company, Limited, was then squeezed so that the amount of the emulsion deposited becomes 80 % based on the dry weight of the fibers and was then treated for 15 minutes in an atmosphere having a dry bulb temperature of 125°C. and a wet bulb temperature of 60°C. so that the silicone resin becomes bonded with the treated acrylic fibers simultaneously with the collapsing of the void in fiber structure.

The fiber tow was then further treated for 8 minutes in compressed steam at 130°C. so that the fiber structure becomes relax, was then fed into a stuffer box to be crimped, had 0.33 % Nissan Unilube 50 MB-168, produced by Nippon Oils and Fats Co., as a spinning oil deposited on it, was dried and was then cut to unequal lengths of 6 to 140 mm. to make nonshrinkable acrylic fibers of a monofilament fineness of 2.5 deniers (acrylic fibers **1**). The rate of deposition of the silicone on these fibers was 0.97 %.

An acrylonitrile copolymer having the same composition as of the above mentioned nonshrinkable acrylic fibers was wet-spun in an ordinary manner. The obtained acrylic fiber tow of a monofilament fineness of 3 deniers was fed into a turbostapler, was secondarily stretched to 1.16 times as long at a hot plate temperature of 150°C., was then mechanically crimped and was cut to make highly shrinkable acrylic fiber staples having a latent hot water shrinkage of 12.8 % (acrylic fibers **2**).

40 % of nonshrinkable acrylic fibers (acrylic fibers **1**), 30 % of highly shrinkable acrylic fibers (acrylic fibers **2**) and 30 % of wool fibers of an average monofilament fineness of 3.6 deniers were mix-spun in an ordinary manner to make a two folded yarn of a yarn count of 52 (metric yarn count).

The obtained mix-spun yarn was dipped in a mixed dyeing solution of a cationic dye and acid dye and was skein-dyed in one stage in one bath in an ordinary manner so that, at the same time, the latent shrinkability might be developed and a bulkiness might be imparted. Two of such mix-spun yarns were plyed and were fed into a weft knitting machine of 14 G, were knitted into a plain knit fabric, were then stretched by 4 % in the longitudinal direction and were set by Hoffman-set (knit fabric **1**).

Then, as a control, a knit fabric of a plain knit structure (knit fabric **2**) was made under the same conditions as above mentioned except that nonshrinkable acrylic fibers having had only a cationic softening agent Zontes TA 460–15, produced by Matsumoto Oils and Fats Co., deposited on them without applying the silicone emulsion bath treatment recommended in the present invention were used instead of the above mentioned acrylic fibers **1**.

Further, a decomposed knit fabric of a sweater of a plain knit structure consisting of commerical wool fibers alone was prepared as another control sample (knit fabric **3**).

The stress reduction rate tan α at the time of the kinetic friction of each of these knit fabrics is shown in Table **1**. From these results, it will be understood that the knit fabric **1** satisfying all the conditions proposed in the present invention has a permanent slippery touch very similar to that of animal hair fibers. The tan α of the knit fabric after being washed was measured by using a sample whose washing shrinkage had been measured.

Table 1

	Stress reduction rate tan α at the time of the kinetic friction		Washing shrinkage
	Before being washed	After being washed	
Knit fabric 1	14.0 g./mm.	14.4 g./mm.	3.5 %
Knit fabric 2	16.7 g./mm.	over 100 g./mm. Stick-slip	3.0%
Knit fabric 3	13.2 g./mm.	—	15.0 %

Further, aside from Table 1, the slippery touch was sensorily evaluated. It was confirmed that the knit fabric **1** had a favorable slippery touch so similar to that of the knit fabric **3** as to be hardly distinguished from it. The knit fabric **1** was favorable also in such other sensorily evaluated elements as, for example, the bulkiness, stiffness and hardness and was recognized to have a remarkably improved commodity value.

By the way, other sample acrylic fibers for which the concentration of the silicone resin in the emulsion had been increased, the squeezing rate after the dipping treatment in the emulsion had been reduced and the amount of deposition of the silicone had been adjusted to 3.7 % rolled up frequently due to the sticking in the carding step in the spinning process and no satisfactory yarn could be made of them.

What we claim is:

1. An acrylic fiber product having an animal hair-like hand comprising a heat-treated yarn or knit or woven fabric containing mix-spun (1) animal hair fibers, (2) nonshrinkable acrylic fibers having a monofilament fineness 50 to 100 % of the average monofilament fineness of the animal hair fibers and having a silicone resin defined by the formula

$$H_3C\overset{CH_3}{\underset{CH_3}{Si}}O-\left[\overset{CH_3}{\underset{CH_3}{Si}}O\right]_x-\left[\overset{CH_3}{\underset{R}{Si}}O\right]_y-\overset{CH_3}{\underset{CH_3}{Si}}CH_3$$

wherein

R is $R'NH_2$, $R'NHR''$ or $R'NR''_2$, R' is CH_2, n is **1** to **3**, R'' is C_mH_{2m+1}, m is 1 to 3, x and y are positive integers and the molecular weight of the silicone resin is less than 100,000

deposited on the fiber surfaces in an amount of 0.1 to 3.0 % based on the dry weight of the fibers and (3) highly shrinkable acrylic fibers having a latent shrinkage at least 3 % higher than the latent shrinkage of the nonshrinkable acrylic fibers.

2. An acrylic fiber product as claimed in claim **1** wherein the amount of the animal hair fibers is 10 – 70 % by weight and the amount of the highly shrinkable acrylic fibers is at least 20 % by weight, based on the total weight of the fiber product.

3. An acrylic fiber product as claimed in claim **1** wherein the said product has a stress reduction rate, tan α, at the time of kinetic friction of 5 to 25 g./min.

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