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Author(s)	Umehara, Ryo; Shibata, Yutaka; Miyamoto, Takeaki; Ito, Hiraku; Onogi, Yoshihiko; Inagaki, Hirosi
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Crimp Formation and Stabilization of Wool Fibers by Draft and Immediate Relaxation

Ryo Umehara*, Yutaka Shibata*, Takeaki Miyamoto**,

Hiraku Ito***, Yoshihiko Onogi**** and Hiroshi Inagaki****

Recently we succeeded in constructing a machine for artificial crimping, called IWS SUPERCRIMPING. This processing consists of two stages, i.e. crimp formation by draft-relaxation and stabilization of the resultant crimp by chemical modification. It is demonstrated that the present draft-relaxation process is suitable particularly for wool fibers having a bilateral structure, such as Merino and Shropshire.

KEY WORDS: Wool/ Crimp formation/ Crimp stability/ Draft-relaxation process/ Reduction/ Bilateral structure

INTRODUCTION

An important inherent property of the wool fiber is the presence of natural crimp based on the bilateral structure. The crimp shape of the fiber mass and the volume occupied by this crimped mass bring about a very significant effect on the aesthetic properties of the products. In order to improve yarn performance and to increase the efficiency of wool textile industry, various crimping processes have been commercially introduced[1~5]. However, most of these processes are based on the so-called stuffing box principle, and the stability of extra crimps thus formed has never been satisfactory. In addition, loss in tensile strength has been a disadvantage.

Since 1975, one of the authors (R.U.) has been concerned with development of an artificial crimping process. This consists of two stages, i.e. crimp formation by draft-relaxation and stabilization of the crimp by chemical modification of disulfide bonds present in wool. Recently we established conditions for the optimum crimping and succeeded in constructing a crimping machine, called IWS SUPERCRIMPING[5~7]. At present, the new process with this machine has been widely introduced into the Japanese wool industry to produce bulky hand knitting yarns, woollen and worsted fabrics, machine knitting yarns and futons etc.

This report deals with results on the performance of crimped wool fibers prepared by using the IWS supercrimping machine. Treatment for the supercrimping

* 梅原　亮, 柴田　豊 : Present address: IWS Ichinomiya Technical Centre, Mabiki, Yamato-cho, Ichinomiya, Aichi 491, Japan

** 宮本　武明 : Laboratory of Polymer Separation and Characterization, Institute for Chemical Research, Kyoto University, Uji, Kyoto 611, Japan

*** 伊藤　啓 : Present address: Saint Agnes' Junior College, 5-81-1 Nampeidai, Takatsuki, Osaka 569, Japan

**** 小野木禎彦, 稲垣　博 : Present address: Mukogawa Women's University, Nishinomiya, Hyogo 663, Japan

was carried out under industrial conditions. The structural changes of the fibers, and the change in tensile strength and cystine content induced by the chemical modification were also investigated to examine the damage of the crimped wool fibers.

EXPERIMENTAL

Wool Samples

The wools used in the present work were Australian Merino and Shropshire, New Zealand Luster (NZ Luster) and Mohair. The characteristics of these wool fibers are listed in Table 1.

Table 1. Fiber diameter and length of wool used

Sample	Cortex Structure	Diameter (μm)	Length (mm)
Aust. Merino	Bilateral	21.50± 4.71	65.8
Aust. Shropshire	Bilateral	32.90± 9.25	82.9
NZ Luster	Non-bilateral	37.77±10.25	84.1
Mohair	Non-bilateral	37.87±11.17	80.2

Crimp Stability Test

The length of a sliver under free tension (A) and that of the sliver extended up to crimp removal (B) were measured. The sliver was then immersed in water at 20°C for 60 min, and squeezed with filter papers and air-dried. After letting it stand overnight, the length of the sliver under free tension (C) was measured again. The crimp stability has been expressed as Stability (%) = (B−C)/(B−A) ×100. While, the crimp contraction (%) is given as Contraction (%) = (B−A)/B×100, where A and B are those as defined above.

The Aachen Felt Ball Test[9]

The Aachen three-dimensional shaking machine was used for producing felt balls. 50 ml of distilled water was added to 1 g of each treated wool sample in a 100 ml screw top cylindrical container, and the container was shaken at 150 rpm for 60 min at 20°C. The balls were dried under standard conditions (20°C, 65% RH), and the diameter of each ball was determined as a mean value of three perpendicular diameters measured by a travelling microscope. For each sample, three balls were prepared.

Tensile Strength Measurements[10]

Wet tensile strength measurements were made on single fibers using the Instron tensile tester. Samples with a testing length, 10 mm were wetted by treatment with an aqueous solution containing 0.5% Tergitol TMN at 20°C for 1 min before the test. The sample mounted on the tensile tester was placed in water at 20°C for 10 min and stretched at a rate of 20 mm/min to the rupture.

Determination of thiol contents

The thiol content (SH-content) was determined by using the method of S-car-

boxymethylation: Wool samples were S-carboxymethylated with iodoacetic acid according to the method of O'Donnel and Thompson[11], followed by acid hydrolysis ucing 6N HCl at 110°C for 24 h. The content of S-carboxymethylcysteine in the hydrolysate was estimated using a Hitachi KLA-5 amino acid analyzer.

X-ray diffraction and Scanning Electron Microscopy

The X-ray diffraction photograph was taken by a Rigakudenki model RU-3H X-ray diffractometer with a flat-plate camera, using a Ni-filtered copper $K\alpha$ beam. The wool fiber was coated with a thin layer of gold by using ion coater Eiko IB-3, and then examined in a Hitachi S-310 scanning electron microscope (SEM).

CRIMPING PROCESS

An artificial crimping machine, called IWS SUPERCRIMPING machine[8], has a production capacity of 60 kg/h. The outline of this machine is as follows.

1) 8 ends of card or top sliver (25 g/m) are fed to a rotary gill at the rate of 20 m/min (draft ratio = 1/3.85) to prepare an even sliver web having 200 mm width and 3 mm thickness.

2) After the fiber opening with the gill, the sliver web is immersed in an aqueous solution containing 2.6% w/v sodium metabisulfite and 0.5% w/v Tergitol TMN wetting agent for 10 sec at pH 4.5, squeezed at 100% pick-up and stored for 5 min in a J-box. (Sample I)

3) Wool sliver is re-squeezed at 50% pick-up, and drafted at 30% using a ratch length equal to the mean fiber length of the sliver to be crimped (roller drafting machine) and immediately relaxed (formation of helical crimps).

4) Then, steam-setting takes place at 95°C for 5 min by overfeeding to the sliver between the two conveyor belts. (Sample II)

5) The sliver is further kept in hot water (85°C) for 80 sec to impart the permanent chemical set to the fibers and rinsed with warm water (40°C) for 80 sec.

6) Finally, the crimped sliver is dried with an air-suction-drum-drier at 85°C for 2 min and then cooled with air cool. (Sample III)

RESULTS AND DISCUSSION

Performance of Supercrimped Wool Fibers

Figure 1 shows the comparison of supercrimped wool sliver with untreated sliver. The supercrimped wool sliver was prepared by 30% drafting in an aqueous solution (pH 4.5) containing 0.5% w/v Tergitol TMN wetting agent. The number of crimps per cm, the crimp contraction, and the stability of extra crimp formed are summarized in Table 2.

Table 3 shows the result on the felting of untreated and supercrimped wool fibers by the Aachen felt ball test. From these Tables, one can see that the values of crimps/cm and crimp contraction as well as the felting resistance were remarkably improved by the supercrimping treatment.

To be noted is that the stability of extra crimp thus formed was excellent, i.e., 98 and 92% for Australian Merino and Shropshire, respectively, showing that the

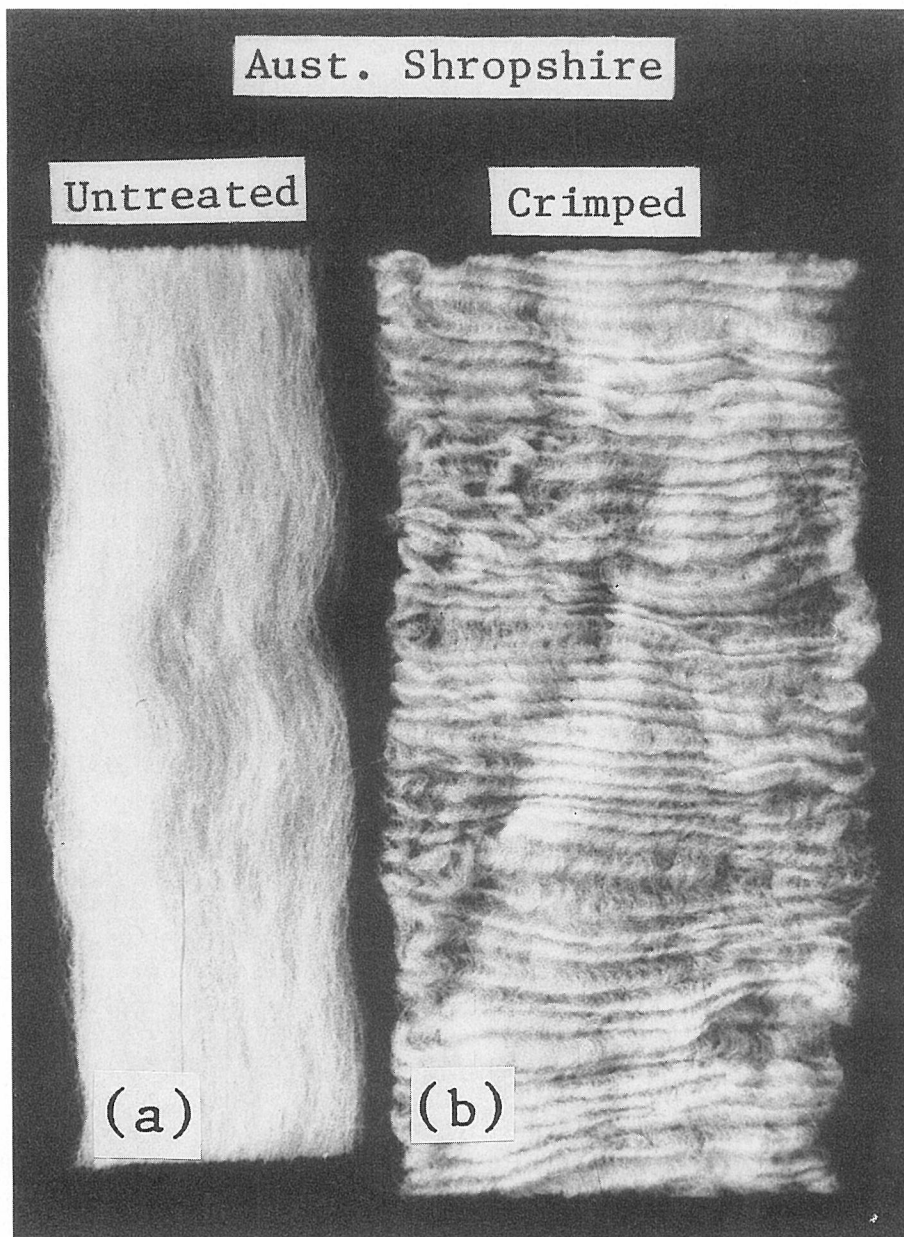R. Umehara, Y. Shibata, T. Miyamoto, H. Ito, Y. Onogi and H. Inagaki

Fig. 1 Wool sliver (a) before and (b) after supercrimping treatment

present draft-relaxation process is particularly suitable for wool fibers having a bilateral structure. In the case of NZ Luster and Mohair having no bilateral structure, however, the crimp stability was not satisfactory. These results indicate that the formation of additional crimp is due to the differential stress relaxation in the ortho and para cortices of the wool fibers. It is clear that the present treatment conditions should be modified to some extent in order to impart stable

Table 2. Comparison of crimps/cm, crimp contraction and stability
between untreated and supercrimped wool fibers

Sample		Crimps/cm	Contraction (%) Fiber	Sliver	Stability of Extra Crimps (Sliver) (%)
Merino	Untreated	5.77	20	—	—
	Crimped	7.80	38	50	98
Shropshire	Untreated	1.31	15	—	—
	Crimped	1.70	32	47	92
NZ Luster	Untreated	0.40	5	—	—
	Crimped	0.73	24	46	79
Mohair	Untreated	—	3	—	—
	Crimped	—	24	44	62

Table 3. Comparison of felt ball density between
untreated and crimped wool fibers

Sample		Density[a] Shaking Time (min) 15	60
Merino	Untreated	1.00	1.56
	Crimped	0.87	1.48
Shropshire	Untreated	1.00	1.60
	Crimped	0.80	1.39
NZ Luster	Untreated	1.00	1.55
	Crimped	0.93	1.39
Mohair	Untreated	1.00	2.00
	Crimped	1.00	1.36

a) The value is expressed in terms of a ratio to
untreated wool fibers (15 min shaking time).

Table 4. Comparison of mechanical properties between untreated
and supercrimped wool fibers

Sample		Young's Modulus (kg/mm²) Mean	Breaking Strength (kg/mm²) Mean	Extension of Breaking (%) Mean
Shropshire	Untreated	129.5	14.7	60.4
	Crimped	122.5	13.9	60.4
NZ Luster	Untreated	174.6	17.9	70.9
	Crimped	157.1	16.1	67.8

crimp to wool fibers having no bilateral structure.

Table 4 shows the comparison of tensile properties between untreated and supercrimped fibers. It can be seen that the reduction in Young's modulus, breaking strength and extension at breaking point for Shropshire were 5.4, 5.4 and 0 %, respectively, while, those for NZ Luster were 10.0, 10.0 and 4.4%, respectively.

Such differences between Shropshire and NZ Luster are considered to be due to the difference in the structure of cortex. As already mentioned, NZ Luster has no bilateral structure and is larger in the fiber diameter, coaser and more rigid than Australian Merino and Shropshire.

Effects of Chemical Modification on Crimp Stability

Table 5 shows the comparison of crimps/cm, crimp contraction and stability of supercrimped fibers with those of the fibers treated without chemical modification by bisulfite. A positive effect of bisulfite treatment on the permanent setting of the extra crimp is obvious.

In order to elucidate the degree of reduction-oxidation reaction by bisulfite, the thiol content of the samples in three different stage, described in the section of the crimping process, i.e., Sample I, II and III, was examined by amino acid analysis. The results on Shropshire wool fibers are shown in Table 6. The total amount of cysteine and half cystine contained in original Shropshire wool fibers was determined by amino acid analysis and found to be approximately 830 ± 20 μmol/g wool. The results indicate that only 5% of total cystine contained in original wool fibers was converted to cysteine by bisulfite treatment and that 50% of cysteine converted was re-oxidized to cystine during subsequent treatments. By this experiment it also follows that after-treatment with water plays an important role in the chemical setting of the extra crimps formed.

To be discussed here is why the excellent stability of extra crimps can be

Table 5. Effect of chemical modification by bisulfite on crimps/cm, crimp contraction and stability of extra crimps

Sample	Crimps/cm	Crimp Contraction (%) Fiber	Crimp Contraction (%) Sliver	Stability of Extra Crimps (%)	Remarks
Shropshire A	1.31	15	—	—	a)
B	1.70	32	47	92	b)
C	1.46	17	20	60	c)

a) Untreated
b) Normal crimping treatment
c) Treated under the same condition as sample B except for no bisulfite treatment

Table 6. Change of SH content with crimping treatment

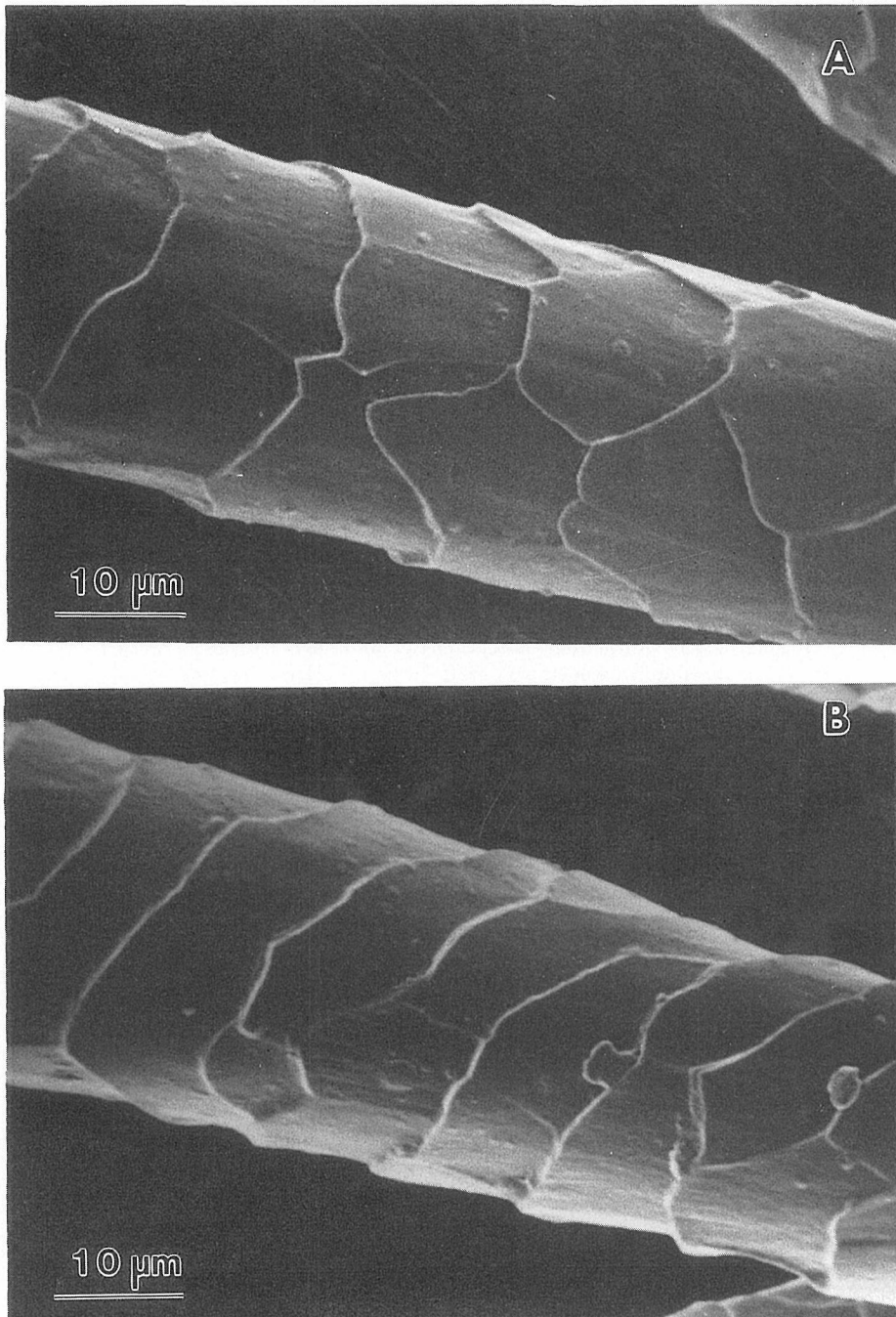| Sample[a]	SH Content (μm/g wool)
Untreated	10.5 ± 0.9
I	50.2 ± 3.5
II	39.1 ± 1.8
III[b]	28.2 ± 1.0

a) Shropshire wool fibers
b) Final products

Fig. 2. Scanning electron micrographs of untreated (A) and crimped
Shropshire wool fiber (B).

attained by such a small chemical modification. This may be interpreted by a following topochemical consideration. That is, the chemical modification by bisulfite will occur mainly at the cuticle present in the outside of the fiber. In such a case, the degree of recrosslinking, which is required to stabilize the crimps formed, does not necessarily need to be so high. In fact, the cuticle is known to be rich in cystine. To confirm this consideration, the same crimping treatment was made
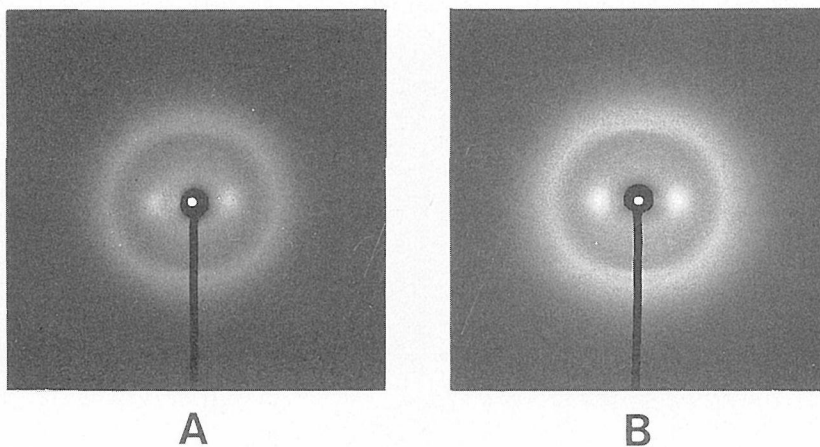

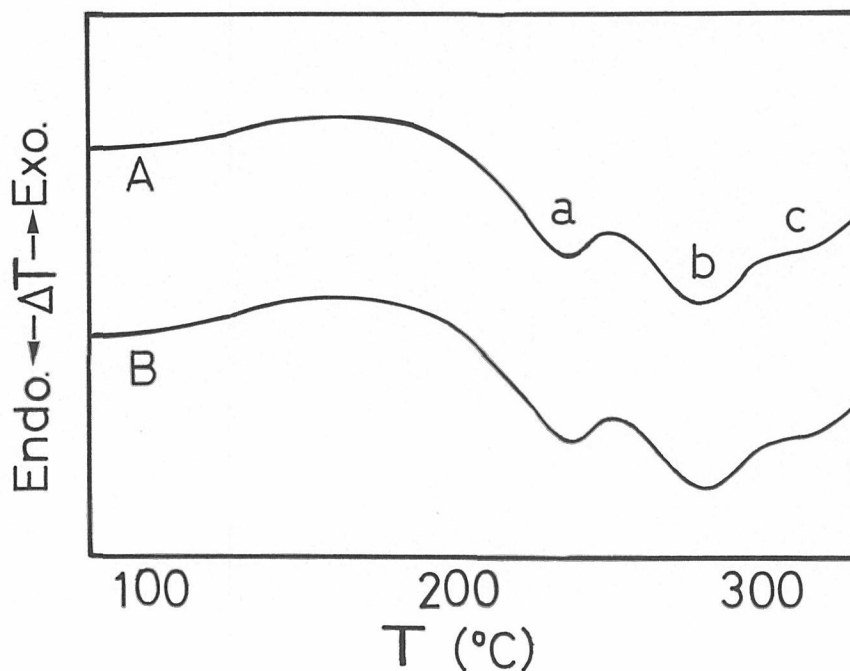Fig. 3. X-ray diffraction photographs of untreated (A) and crimped Shropshire wool fibers (B).

Fig. 4. DTA curves of Merino wool fibers in vacuo at a heating rate of 20°C/min. (A): Untreated, (B): Supercrimped

for decuticled wool fibers (Australian crossbred, 28 μm in fiber diameter). It was found that the stability of extra crimps in the decuticled wool fibers was very low, i.e. 70% as compared with 98% and 92% in normal Merino and Shropshire wool fibers, respectively. (see Table 2)

Damage of Cuticle and Fiber Structure

Figure 2 shows a scanning electron micrograph of a supercrimped wool fiber as compared with that of untreated wool fiber. Note that the crimp is rendered to the fibers without any serious damage to the cuticle. Figure 3 shows the X-ray diffraction photographs of Shropshire wool fibers. It is obvious that all the samples give almost the same X-ray diffraction pattern characteristic of coiled-coil α-helix, showing that no structural change of α-keratin to β-keratin occurred during the crimping treatment. Similar results were also obtained from differential thermal analysis (DTA). Figure 4 shows the DTA curves of Merino wool fibers in vacuo at a heating rate of 20°C/min. Three endotherms in a range of 200~300°C are observed in the DTA curve of native wool fibers (A). The peaks a, b and c have been assigned to the melting of the α-helix structure, the melting of the β-structure and the pyrolysis of keratin proteins, respectively[12]. It can be seen that the DTA curve (B) of the supercrimped fibers is almost the same as that of untreated fibers.

Crimp Stability of Wool Fibers Having no Bilateral Structure

As already mentioned, the crimping conditions employed were not effective for the wool fibers having no bilateral structure. Finally, we attempted to impart more stable crimps to this type of wool. It was found that the fiber performance and the crimp stability could be improved by prolonging the time of bisulfite treatment. The results on NZ Luster are shown in Table 7. Sample H was supercrimped wool obtained by conventional 5 min immersion. Sample J was obtained by treating with bisulfite for 12 h instead of 5 min immersion. The other treatment conditions were same for both the samples. Amino acid analysis showed that the SH-content in the sample corresponding to the sample I in Table 6 was 141.4 μmol/g wool as compared with 50.2 μmol/g wool in sample I.

Table 7. Crimp stability of wool fibers having no bilateral structure

Sample		Crimps/cm	Contraction (%) Fiber	Sliver	Stability of Extra Crimp (Sliver) (%)	Remarks
NZ Luster	G	0.40	5	—	—	a)
	H	0.73	24	46	79	b)
	J	1.05	28	49	90	c)

a) Untreated
b) Conventional crimping condition
c) 12 h bisulfite treatment instead of 5 min immersion

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Processing Co. Ltd. for co-operation to construct the IWS SUPERCRIMPING machine.

REFERENCES

1) J.L. Barach and L.W. Rainard, *Text. Res. J.*, **20**, 308 (1950).
2) D.A Ross, *Appl. Polym. Symp.*, **18**, 1455 (1971).
3) L. Hirst, J.M.D. Pitts and R. Umehara, Proc. 5th Int. Wool Text. Res. Conf., Aachen, Sept. Vol. IV, p. 157 (1975).
4) J.A. Iredale and J. Myers, Proc., 6th Int. Wool Text. Res. Conf., Pretoria, Aug/Sept., Vol. III, p. 313 (1980).
5) R. Umehara and F. Bekku, Jpn. patent 852611
6) R. Umehara and F. Bekku, G.B. patent 1385699
7) R. Umehara and F. Bekku, U.S. patent 3849848
8) R.Umehara et al., Unpublished results.
9) I.W.T.O. Test. Method 20~69.
10) JIS L-1081-1977.
11) I.J. O'Donnell and E.O.P. Thompson, *Austr. J. Biol. Sci.*, **17**, 973 (1964).
12) H. Sakabe, T. Miyamoto and H. Inagaki, *Sen-i Gakkaishi*, **38**, 157 (1982).