

# Properties of Wool Fibers Treated Under High-Temperature Water and Steam

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**ABSTRACT:** In this study, we investigated the hygroscopicity and the extension properties of wool fibers treated under high-temperature steam and water. The scale structure and the crystallinity of the treated wool fibers were analyzed with scanning electron microscopy and X-ray diffraction analysis. The experimental results show that the hygroscopicity of the wool fibers treated under high-temperature steam was higher than that of the wool fibers treated under

high-temperature water. The strength, the breaking elongation, and the breaking work of the treated wool fibers decreased obviously compared with the untreated wool fibers. The scale surface became rough and the crystallization index decreased for the treated wool fibers. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 109: 852–858, 2008

**Key word:** wool; fibers; crystallinity; tensile property

## INTRODUCTION

Wool is a natural protein fiber that has been widely used as a high-quality textile material. In recent years, some wool fibers have been developed for new applications, such as biotechnological and biomedical products.<sup>1–4</sup> Keratins were extracted from wool by incubation with a mixture of sodium dodecyl sulfate (SDS), urea, and 2-mercaptoethanol at 60°C for 5 h; then, wool keratin sponges were made to form useful scaffolds for long-term and high-density cell cultivation.<sup>4</sup> Xu<sup>5</sup> reported the preparation of wool powder from wool fibers under chemical conditions and with apparatus so that the powder could be used for chemical fiber modification. Yang<sup>6</sup> reported the extracting protein method from wool waste to make new protein fiber. The solution of wool keratin obtained from the reduction-C method was used to coat fabrics to improve the setting of the fabrics.<sup>7</sup> So, it is important to investigate new application fields for wool fibers.

The picture of the structure of wool fiber, which in effect is a hydrophobic fiber, is very complex. Epicuticle surrounds every separate cell of the epidermis and is an integral part. It has strong hydrophobic properties, significant resistance to chemical action, and an enzymatic attack. The epicuticle material is a protein containing about 12% cystine. These hydrophobic features are supported by a layer of exocuticle containing about 35% cystine, formed

from an amorphous protein. The cuticle is submerged in an intercellular binding agent, which adheres to the cortex layer (ca. 90% of the fiber mass). The cortex layer cells are able to form disordered regions (matrix) and ordered fibrillar structures built from protofibrils. Wool fibers have not only high strength but also high breaking intensity and high elongation, so it is very difficult for wool to lose its virtues with no chemical agent action.

Wool fibers swell in boiling water or in hot steam. Because there is a chemical action between wool and water that results in the peptide bond hydrolysis of protein molecules, the mechanical properties of the fibers change.<sup>8</sup> The application of Raman spectroscopy facilitates the testing of the local changes in the conformation of the amino acidic residues of the polypeptide chains of wool fiber keratin, with their changes resulting from reactions with the environment. In this study, Raman spectroscopy was used to investigate the effect of different temperatures of water and steam on wool virtues. Some studied results on the characterization of the wool under high-temperature water and steam are reported.

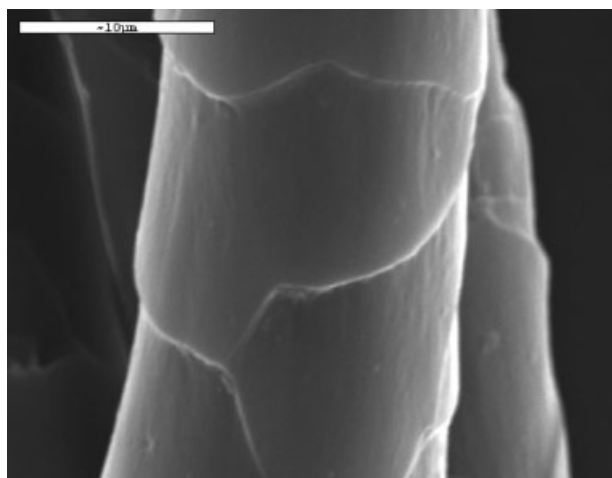
## EXPERIMENTAL

### Wool treatment conditions

Unprocessed Australian Merino wool fibers (diameter = 22 μm) were used. The fibers were cleaned by Soxhlet extraction in dichloromethane for 2.5 h to remove wool wax, dried, and then rinsed thoroughly with water.

Scoured wool fibers were treated under high-temperature steam and water with a RY-12000 high-tem-

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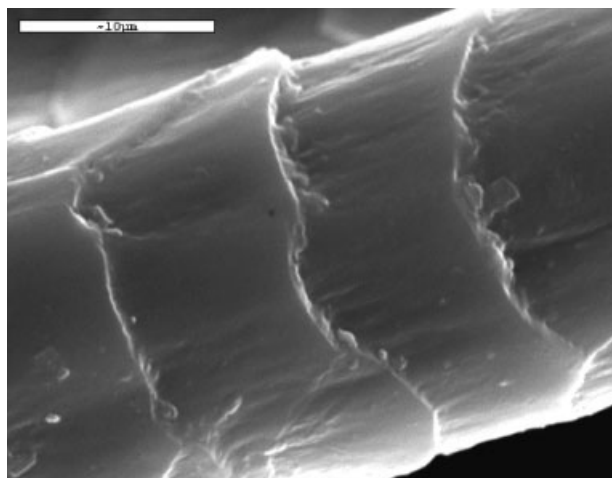
**Figure 1** Scale of the untreated wool fiber.

perature dyeing machine (Shanghai Hongda Chemical Co., Ltd., China). The water temperature increasing step was 3°C/min. The treatment temperatures were 100, 110, 120, and 130°C, and the treatment times were 1, 3, and 5 h.

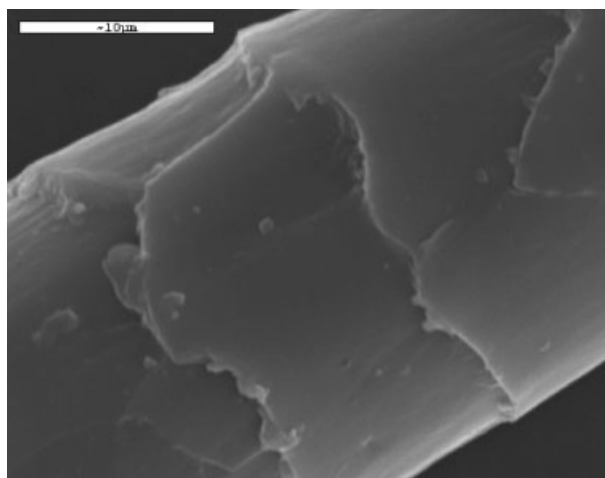
#### Hygroscopicity test

We put the treated wool fibers and untreated wool fibers (scoured wool fibers) in an atmospheric room for 24 h and then weighed them [ $W_1$  (g)]. Then, the treated wool fibers and untreated wool fibers were dried in an oven at 105°C until a constant weight [ $W_2$  (g)] was reached. The hygroscopicity of the wool fibers was calculated as follows:

$$\text{Hygroscopicity of wool fibers (\%)} = [(W_1 - W_2)/W_2] \times 100\%$$



**Figure 2** Scale of the wool fiber treated under 100°C steam for 3 h.



**Figure 3** Scale of the wool fiber treated under 100°C water for 3 h.

#### Tensile properties

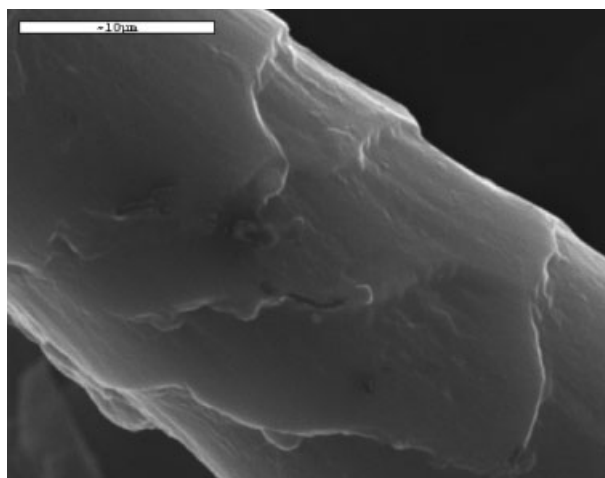
The single-fiber tensile properties of the wool fibers were tested with a YG001 single-fiber tensile tester (Taicang Textile Apparatus Co., China). The test length of fiber was 10 mm, and the tensile speed was 10 mm/min. For each result, 100 wool fibers were tested.

#### Scale structure

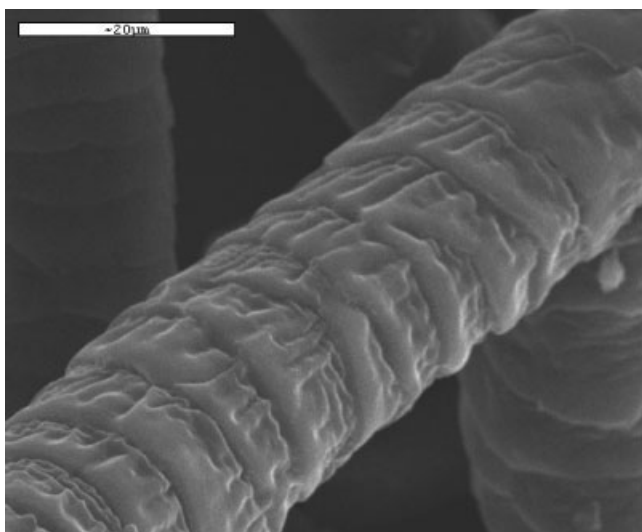
The scale structures of the treated wool fibers and untreated wool fibers were observed with a JSM-5800 scanning electronic microscope (Electronic Co., Ltd., Japan). The fibers were coated with gold, and then, the testing was done.

#### Crystallinity test

The crystallinity of the wool fibers was tested with a D/MAX-2400 X-ray diffraction analyzer (Rigaku Co.,



**Figure 4** Scale of the wool fiber treated under 130°C steam for 3 h.



**Figure 5** Scale of the wool fiber treated under 130°C water for 3 h.

Ltd., Japan). The test conditions were as follows: voltage = 46 kV, current = 100 mA, Cu K $\alpha$  radiation,  $\lambda = 1.54050 \text{ \AA}$ , scanning scope  $2\theta = 3\text{--}40^\circ$ , step =  $0.02^\circ$ , and scanning speed =  $4^\circ/\text{min}$ .

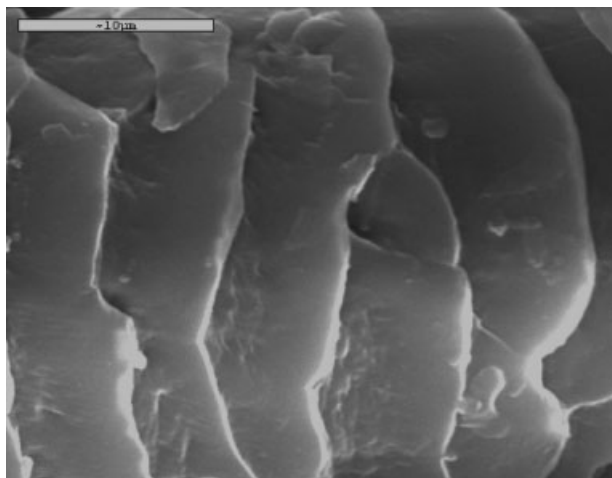
#### Raman spectrophotometry test

The Raman spectrophotometry of the wool fibers was tested with a Renishaw laser microscope Raman spectrum tester (Invia Co., UK) with a 632.81-nm argon ion laser source; the power was 20.5 MW. The scanning time was 20 s. The microscope amplification was 50 $\times$ .

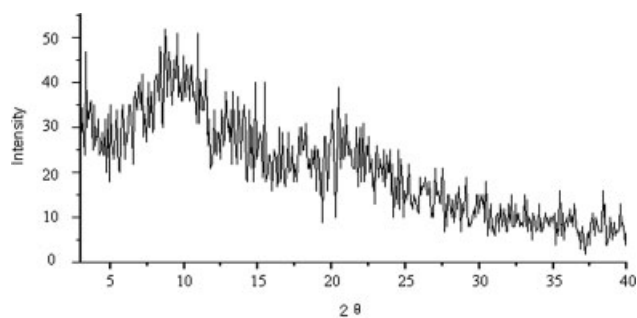
## RESULTS AND DISCUSSION

### Scale structure

Figure 1 shows the scale structure of the untreated wool fiber. Figures 2 and 3 show the scales of the



**Figure 6** Scale of the wool fiber treated under 130°C water for 5 h.



**Figure 7** X-ray diffraction of the untreated wool.

wool fibers treated under 100°C steam for 3 h and 100°C water for 3 h, respectively. Figures 4–6 show the scales of the wool fibers treated under 130°C steam for 3 h, 130°C water for 3 h, and 130°C water for 5 h, respectively.

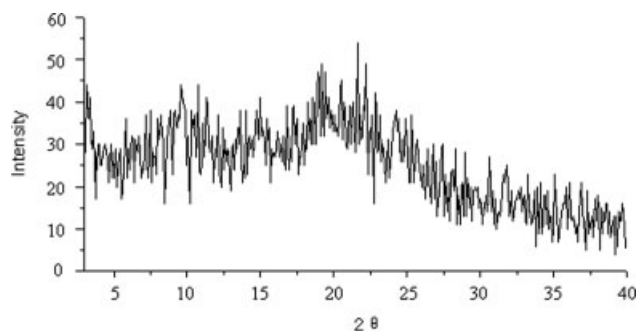
As shown in Figures 2 and 4, the scale surface became less smooth for the wool treated under steam. Figure 5 shows that the scale of the wool fiber lost its original structure, and the scale density along the fiber length increased compared with the untreated wool fiber. The scale of the treated wool fiber lost its luster compared with the untreated wool fiber. Figure 6 shows that the scales of the wool were seriously damaged and the scale density was increased.

### Crystallinity

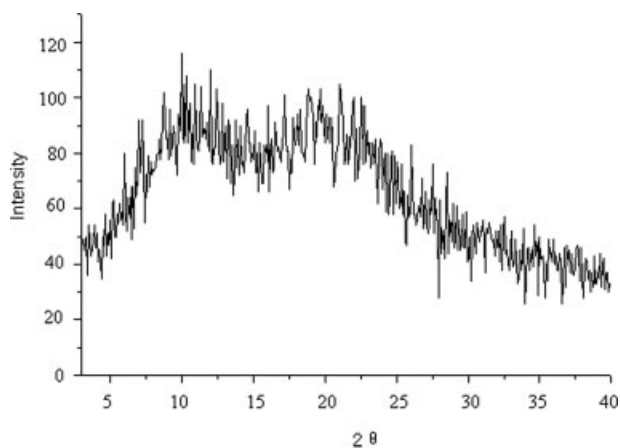
Figures 7–9 show the X-ray diffraction results for the untreated wool fibers and the wool fibers treated under 120°C water for 5 h and 130°C water for 5 h. According to ref. <sup>9</sup>, the crystallization index (CI) of wool fibers is calculated by the following equation:

$$CI (\%) = (I_{9^\circ} - I_{14^\circ})/I_{9^\circ} \quad (1)$$

where  $I_{9^\circ}$  is the intensity at  $2\theta = 9^\circ$  and  $I_{14^\circ}$  is the intensity at  $2\theta = 14^\circ$ .



**Figure 8** X-ray diffraction of the wool treated under 120°C water for 5 h.



**Figure 9** X-ray diffraction of the wool treated under 130°C water for 5 h.

On the basis of eq. (1), the CI results of the treated wool fibers and untreated wool fibers are listed in Table I.

As shown by the results in Table I, the CI and relative CI values of the treated wool fibers were less than those of the untreated wool fibers. This was because high-temperature water treatment changed the inner structure of the wool fibers.

**Hygroscopicity of the wool fibers**

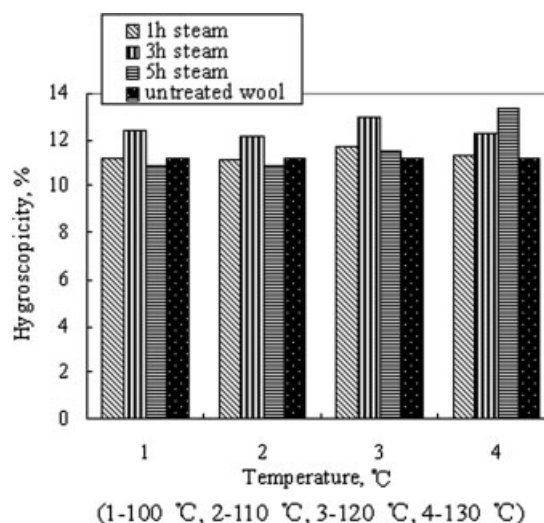
Hygroscopicity shows the water content in wool fibers. A high hygroscopicity shows that there is more water in the wool fibers under atmospheric conditions. A low hygroscopicity shows a water decrease in the wool fibers under atmospheric conditions.

As shown by the results in Figure 10, the hygroscopicity of the wool fibers treated under high-temperature steam for 1 h changed less compared with that of the untreated wool fibers. However, the hygroscopicity of the wool fibers treated under steam for 3 h was higher than that of the untreated wool fibers. The hygroscopicity of the wool fibers treated under 130°C steam for 5 h increased obviously.

As shown by the results in Figure 11, the hygroscopicity changed less for the wool fibers treated

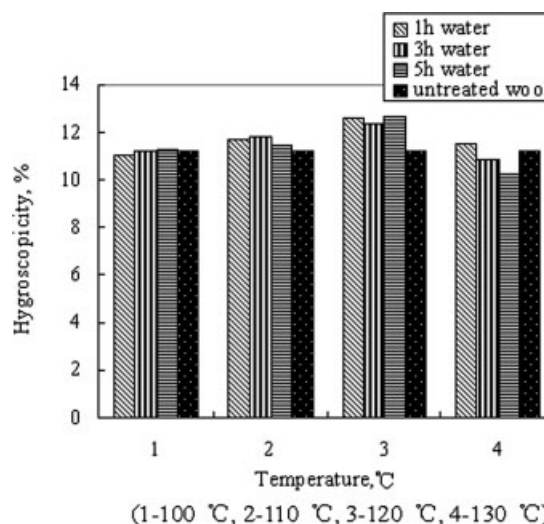
**TABLE I**  
CI and Relative CI Values of the Wool Fibers

Sample	$I_9^\circ$	$I_{14}^\circ$	CI	Relative CI (%)
Untreated wool fibers	360	181	0.50	100
Wool fibers treated under 120°C water for 5 h	308	223	0.28	56
Wool fibers treated under 130°C water for 5 h	781	623	0.20	40



**Figure 10** Hygroscopicity of the wool treated under high-temperature steam.

under 100 and 110°C water for 1, 3, and 5 h compared with that of the untreated wool fibers. The hygroscopicity of the wool fibers treated under 120°C water increased, but the hygroscopicity of the wool fibers treated under 130°C water decreased. There were two reasons for the hygroscopicity increase.<sup>10</sup> First, the scale of the treated wool fibers was damaged, which resulted in a decrease in water-repelling compositions and an increase in hydrophilic groups on the scale surface. Second, the CI of treated wool fibers decreased, and the hydrophilic properties increased. The scale structure was seriously damaged; however, some hydrophilic groups were lost. So, the hygroscopicity of the wool fibers treated under 130°C water for 5 h decreased.



**Figure 11** Hygroscopicity of the water-treated wool fiber.

**TABLE II**  
Tensile Properties of the Wool Treated Under Steam for Various Times

Treatment conditions		Breaking strength		Breaking elongation		Breaking work	
Temperature (°C)	Time (h)	Mean (cN)	CV (%)	Mean (%)	CV (%)	Mean (μJ)	CV (%)
100	1	6.84	36.90	44.03	26.12	22.59	44.72
100	3	6.68	30.93	39.89	31.51	20.30	46.90
100	5	6.64	34.64	41.31	33.03	20.76	52.90
110	1	6.89	32.84	35.87	41.51	19.74	58.15
110	3	6.96	36.71	38.29	38.92	20.91	56.44
110	5	5.38	35.17	40.59	39.49	17.74	59.49
120	1	5.44	29.53	40.73	45.83	17.98	53.44
120	3	5.25	35.12	32.25	65.12	14.70	82.88
120	5	3.53	38.43	19.52	80.44	5.85	135.22
130	1	5.28	45.45	30.58	55.09	14.35	78.59
130	3	3.70	34.51	10.86	43.46	2.29	86.11
Untreated wool fibers		7.35	30.60	44.40	22.18	24.25	36.92

CV, coefficient of variation.

### Tensile properties

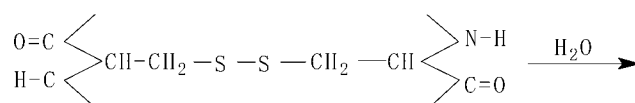
Tables II and III show the tensile properties of the wool fibers treated under high-temperature water and steam, respectively.

As shown in Table II, the strength, breaking elongation, and breaking work of the wool fibers treated under high-temperature steam were lower than those of the untreated wool fibers. When the steam temperature was 130°C, the tensile properties showed a obvious decrease, and with increasing treatment time, the strength, breaking elongation, and breaking work decreased. For the wool fibers treated under 130°C steam for 5 h and 120 and 130°C water, the length of all fibers were shorter than 10 mm; the tensile properties were not tested.

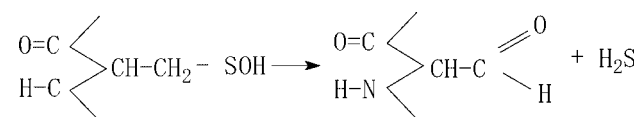
As shown in Table III, the strength, breaking elongation, and breaking work of the wool fibers treated under high-temperature water were lower those that of the untreated wool fibers. The tensile properties of the wool fibers treated under 110°C seriously decreased.

If one compares the results in Tables II and III, it is shown that the strength, breaking elongation, and breaking work of the wool fibers treated under high-temperature water were lower than those of the wool fibers treated under high-temperature steam.

Therefore, the wool fibers treated under high-temperature water had obvious damage. Because the wool fibers were treated in boiling water for a long time, the —S—S— bond of cystine in the wool protein was broken:



—CH<sub>2</sub>—SOH group released H<sub>2</sub>S:



The wool fibers were treated with 110, 120, and 130°C steam for 5 and 10 min in a RY-12000 high-temperature dyeing machine; the breaking strength and breaking elongation of the treated wool fibers are listed in Table IV.

**TABLE III**  
Tensile Properties of the Wool Treated Under Water for Various Times

Treatment conditions		Breaking strength		Breaking elongation		Breaking work	
Temperature (°C)	Time (h)	Mean (cN)	CV (%)	Mean (%)	CV (%)	Mean (μJ)	CV (%)
100	1	6.65	26.82	37.52	35.13	19.01	46.20
100	3	7.38	35.41	41.69	30.98	23.32	51.47
100	5	5.71	31.96	45.55	26.91	19.51	43.10
110	1	5.63	34.49	28.24	59.72	13.48	81.66
110	3	4.88	29.12	24.97	76.80	10.93	94.15
110	5	4.60	37.46	23.10	70.20	9.38	96.86
Untreated wool fibers		7.35	30.60	44.40	22.18	24.25	36.92

CV, coefficient of variation.

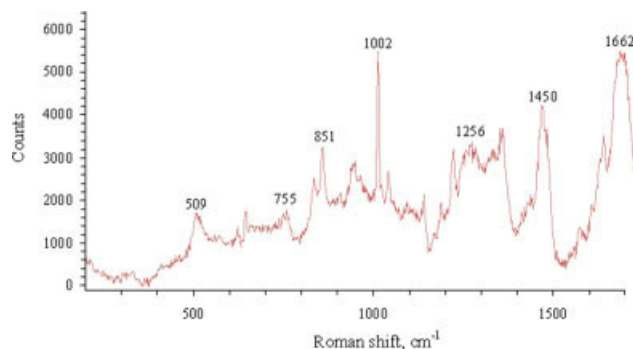
**TABLE IV**  
**Tensile Properties of Wool Treated Under Steam for a Short Time**

Treatment conditions		Breaking strength		Breaking elongation	
Temperature (°C)	Time (min)	Mean (cN)	CV (%)	Mean (%)	CV (%)
110	5	6.83	30.64	48.86	25.54
110	10	6.89	30.06	47.40	31.74
120	5	7.26	29.77	49.44	28.38
120	10	7.12	33.03	41.21	28.47
130	5	6.73	32.99	41.42	29.40
130	10	6.38	36.22	40.20	37.41
Untreated wool fibers		7.35	30.60	44.40	22.18

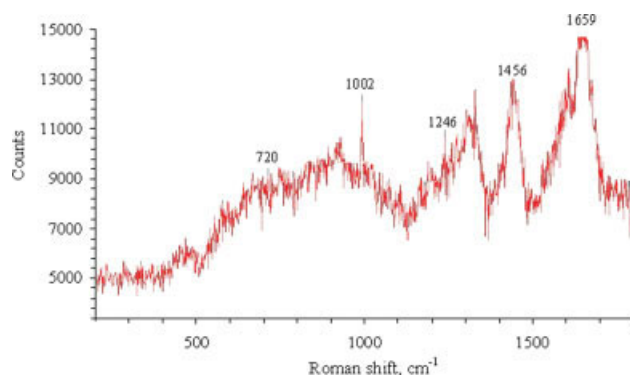
CV, coefficient of variation.

When the results in Tables IV and II are compared, the breaking strength and breaking elongation of the wool fibers treated under 110°C steam for various times had less difference. When the steam temperature was 120 or 130°C, the breaking strength and breaking elongation of the treated wool fibers seriously decreased with increasing treatment time.

Figures 12–14 show the Raman results of the untreated wool and treated wool under 120°C water for 3 h and 100°C steam for 3 h, respectively. There



**Figure 12** Raman spectrophotometry of the untreated wool. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

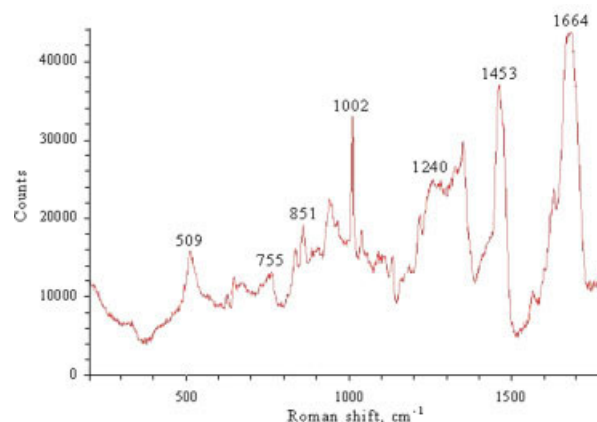


**Figure 13** Raman spectrophotometry of the wool treated under 120°C water for 3 h. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

was not a serious peak at 510 cm<sup>-1</sup>, as shown in Figure 13, which shows that the —S—S— bond of the cystine of wool was damaged. So, the breaking strength and the breaking elongation of the wool treated under 120°C water for 3 h was lower than that of the untreated wool and the wool treated under 100°C steam for 3 h.

In boiling water, the action between wool fibers and water is strong. However, in steam, the density of the water medium is thin, so the action between wool fibers and steam is relatively weak. The —S—S— bond of the cystine of wool is broken in boiling water but not under steam conditions.

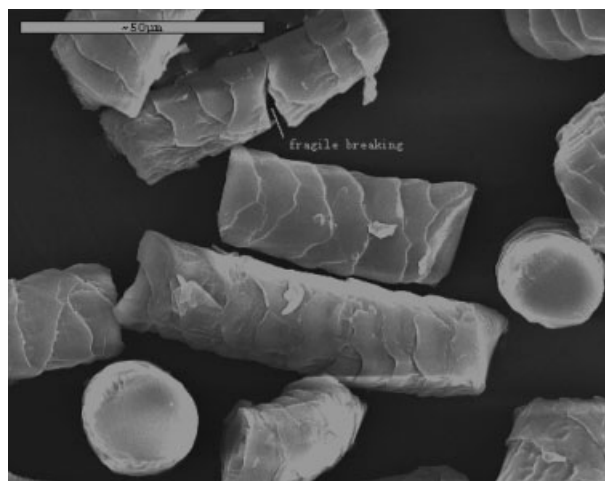
After being ground for 3 min with a mortar, the wool fibers treated under 130°C water for 3 and 5 h were broken and changed into very short fibers (as shown in Figs. 15 and 16). Moreover, the wool fiber breakage was fragile, and the broken wool fibers showed round cross sections (see Fig. 17). The other treated wool fibers and untreated wool fibers could not be ground into short fibers. So, the structure of the wool fibers treated under 130°C water for 3 and 5 h were seriously damaged.



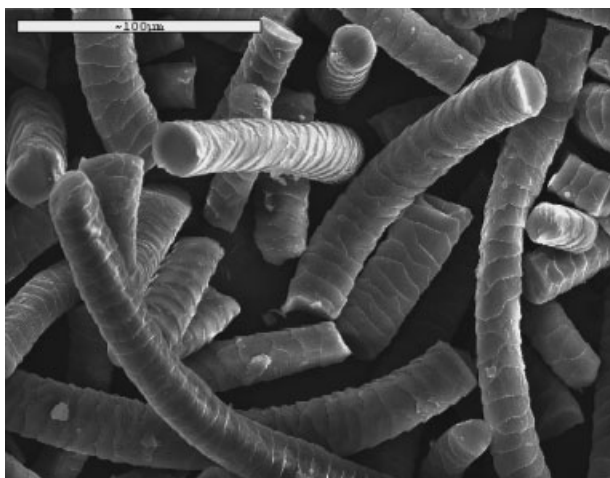
**Figure 14** Raman spectrophotometry of the wool treated under 100°C steam for 3 h. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



**Figure 15** Structure of the wool treated under 130°C water for 3 h.



**Figure 17** Wool fiber breaking state after grinding.



**Figure 16** Structure of the wool treated under 130°C water for 5 h

### CONCLUSIONS

The hygroscopicity of the wool fibers treated under high-temperature steam and water increased, but the hygroscopicity of the wool fibers treated under 130°C water for 3 and 5 h decreased. The scale of the wool treated under high-temperature water were seriously damaged compared to the wool fibers treated under high-temperature steam. The strength,

breaking elongation, and breaking work of the treated wool fibers were lower than those of the untreated wool fibers. The tensile properties of the wool fibers treated under high-temperature water obviously decreased. The CI of the treated wool fibers was lower than that of the untreated wool fibers. The nontextile uses for wool fibers have been investigated by some researchers. On the basis of the high hygroscopicity of wool treated under high-temperature water and steam in this study, wool treated can be used as a water-absorbing material.

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