Evaluation of Some of the Properties of Plasma Treated Wool Fabric

C. W. Kan, C. W. M. Yuen

Institute of Textiles and Clothing, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong, China

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ABSTRACT: Low temperature plasma (LTP) treatment was applied to wool fabric with the use of a nonpolymerizing gas, namely oxygen. Properties of the LTP-treated samples including low stress mechanical behavior, air permeability, and thermal characteristics were evaluated in this study. Kawabata evaluation system fabric (KES-F) was employed to determine the tensile, shearing, bending, and compression strength properties and surface roughness of the specimens. The changes in these properties are believed to be closely related to the interfiber and interyarn frictional force induced by the LTP. The decrease in the air permeability of the LTP-treated wool fabric was found to be probably because of the plasma action effect on increasing the fabric thickness and a change in fabric surface morphology, which was confirmed by scanning electron microscopy micrographs. The change in the thermal properties of the LTP-treated wool fabric was in good agreement with the earlier findings and can be attributed to the amount of air trapped between the yarns and fibers. This study suggested that the LTP treatment can influence the final properties of the wool fabric, and also provide information for developing LTP-treated wool fabric for industrial use. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 102: 5958–5964, 2006

Key words: etching; oxidation; fibers; surfaces; plasma treatment and textiles

INTRODUCTION

The low temperature plasma (LTP) technique is widely used for modifying textile materials, and it is regarded as an environmentally friendly process as no chemicals are used.^{1–3} LTP is an ionic gas whose compounds and characteristics are different from normal gas and this technique can then be used for modifying textile materials with different features such as change in hydrophilicity.³

The action of LTP is mainly interactive (such as oxidation and etching) on the surface of the textile material, hence the surface properties of the textile material together with those associated with the surface characteristics will also be changed. As for the application of LTP treatment in wool fiber, most of the discussions were focused on applying this technique to improve the surface wettability^{4–6} and shrink resistance.^{7–10} However, little discussion has been made on the mechanical and thermal properties and the air permeability related to fabric comfort. This article thus concerned mainly with the assessment for LTP modification of those properties of the wool fabric induced by LTP with a nonpolymerizing gas.

EXPERIMENTAL

Twill wool fabric (41 ends/cm, 31 tex; 36 picks/cm, 36 tex; 180 g/m²) with 100% purity were scoured with dichloromethane (A.R. Grade) for 4 h by Soxhlet extraction method. The solvent scoured wool fabrics were then rinsed twice with 98% ethanol (A.R. Grade) and washed twice with deionized water, respectively. The cleaned fabrics were dried in an oven at 50°C for 4 h. The fabrics were finally cut to the dimension of 20×20 cm² and conditioned at 25°C with relative humidity of 65% for 24 h before further evaluation.

A glow discharge generator (Showa, Japan) was used for the LTP treatment of the wool fabrics. The glow discharge apparatus was a radio-frequency etching system operating at 13.56 MHz and using an aluminum chamber with an internal diameter of 200 mm. The chamber diameter was 380 mm with a height of 180 mm. A nonpolymerizing gas, namely oxygen, with a flow of 20 cc/min was used. The discharge power and system pressure were set at 80 W and 10 Pa, respectively. The duration of LTP treatment was 5, 10, 20, and 30 min. Five specimens were prepared for each duration of treatment. After LTP treatment, the fabrics were conditioned at 25°C with relative humidity of 65% for 24 h before being evaluated.

The morphology of the wool fabrics was observed using a scanning electron microscope (SEM) (Lecia Stereoscan 440). The samples were gold-coated before conducting the SEM examination.

The Kawabata evaluation system fabric (KES-F) was used for measuring the low-stress mechanical

Correspondence to: C. W. Kan (tccwk@inet.polyu.edu.hk).

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Tensile energyWTEnergy in extending fabric to 500 gf/cm widthTensile resilienceRTPercentage energy recovery from tensile deformationExtensibilityEMTPercentage extension at the maximum applied load of 500 gf/cm specimen widthShear rigidityGAverage slope of the linear regions of the shear hysteresis curve to $\pm 2.5^{\circ}$ shear angleShear stress at 0.5° 2HGAverage width of the shear hysteresis loop at $\pm 0.5^{\circ}$ shear angle	gf.cm/cm ² % gf/cm degree
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	gf/cm
Shear stress at 5° 2HG5Average width of the shear hysteresis loop at $\pm 5^{\circ}$ shear angle	gf/cm
Bending rigidity B Average slope of the linear regions of the bending hysteresis curve to 1.5 cm ⁻¹	gf.cm ² /cm
Bending moment 2HB Average width of the bending hysteresis loop at 0.5 cm ⁻¹ curvature	gf.cm/cm
Fabric thickness at 0.5 gf/cm ² pressure T_0 Fabric thickness at 0.5 gf/cm ² pressure	mm
Fabric thickness at 50 gf/cm ² pressure T_m Fabric thickness at 50 gf/cm ² pressure	mm
Compressional energy WC Energy in compressing fabric under 50 gf/cm ²	$gf cm/cm^2$
Compressional resilience RC Percentage energy recovery from lateral compression deformation	%
Compressibility EMC Percentage reduction in fabric thickness resulting from an increase in lateral pressure from 0.5 to 50 gf/cm ²	%
Coefficient of friction MIU Coefficient of friction between the fabric surface and a standard contactor	_
Geometrical roughness SMD Variation in surface geometry of the fabric	micron

TABLE I The Tensile, Shearing, Bending, Compression, and Surface Properties Obtained From the Hysteresis Curves

properties of the LTP-treated fabrics which include the tensile, shearing, bending, compression, and surface properties. The parameters obtained for these hysteresis curves are defined in Table I and the configuration of the KES-F was shown in Figure 1.

The air permeability of the wool fabrics was obtained by the use of a KES-F8-AP-1 air permeability tester. The result of air permeability expressed as air resistance (R) was recorded in terms of kPa s/m in which a higher value of R indicates poorer air permeability of the fabric and *vice versa*. The insulation properties were studied by the use of a KES-F

Thermo Labo II. The heat loss per unit area under the condition of 10°C temperature difference was measured by the warm/cool feeling (q_{max}) in term of W/ cm². During the evaluation, the analytical tolerance was limited to 5%.

RESULTS AND DISCUSSION

Morphological study

SEM micrographs of untreated and LTP-treated wool fabrics are shown in Figures 2–5. Figure 2 shows the



Pure bending Tester

Figure 1 Schematic diagram of KES-F with low-stress mechanical properties tester.¹¹



Figure 2 SEM micrograph of untreated wool fiber.



Figure 3 SEM micrograph of 5 min LTP-treated wool fiber.

untreated wool fiber in which the escarpments are prominent and well defined. There is no evidence of separation between the neighboring cuticular cell, and the cleft lines are ill defined. The untreated wool fiber surface may be described as a smooth fiber surface. As shown in Figure 3, after 5 min of treatment with oxygen plasma, continuous cracks appear that are located parallel to the direction of the wool fiber axis, and the scale edges are slightly eroded and rounded. Figure 4 illustrates the appearance of wool fiber surface after 10 min of oxygen plasma treatment. The etching effect of plasma has created ridges on the surface and the scales are slightly rounded-off. In addition, continuous and deep cracks are clearly seen on the fiber surface. The cuticular escarpments were partially removed after 20 min of oxygen plasma treatment, but some of the escarpments were raised resulting in cleft lines along the scale edge. The cracks were



Figure 4 SEM micrograph of 10 min LTP-treated wool fiber.



Figure 5 SEM micrograph of 20 min LTP-treated wool fiber.

present along the fiber axis, as shown in Figure 5. It is also found that the scales still remained on the fiber surface. When compared, there were no significant changes in the appearance of the 30 min-treated wool fiber to that of 20-min treated wool fibers. As a result, the LTP treatment can increase the surface roughness, voids, and spaces in the wool fiber.

Tensile properties

The tensile properties composed of tensile energy (WT), tensile resilience (RT), and extensibility (EMT). The different tensile properties of the LTP-treated fabrics as measured by KES-F system are shown in Table II.

The tensile energy (WT) is defined as the energy required for extending the fabric, which reflects the ability of the fabric to withstand external stress during extension. Higher value of WT implicates a better tensile strength of the fabric. After the LTP treatment, it was noted that the WT increased steadily with the prolonged treatment time. However, the increment was not so great when compared with the untreated fabric.

In general, the tensile strength of the fabric depends on lot of factors such as fabric structure, yarn twist, and yarn count. As identical fabrics were used, the major factor affecting the WT of the fabric would be due to the fabric structure. However, the LTP treatment could not alter the fabric structure as it is only a surface treatment method causing an etching action resulting in adverse affect on surface roughness of the fibers.^{9,12} Such roughening effect might impart more contact points within the fibers microscopically and within yarns macroscopically.^{12,13} The increment in number of contact points would result in enhancing the intervarn and interfiber friction, where

		Plasma treatment time (min)				
KES-F properties	Symbol	0	5	10	20	30
Tensile	WT	11.61	12.72	12.75	12.79	12.84
	RT	64.07	59.59	59.39	59.15	58.97
	EMT	9.54	8.39	8.34	8.28	8.20
Shearing	G	0.71	1.30	1.32	1.35	1.38
		0.60	2.00	2.05	2.06	2.08
	2HG5	1.75	5.55	5.58	5.60	5.63
Bending	В	0.111	0.139	0.140	0.143	0.146
	2HB	0.038	0.083	0.087	0.089	0.095
Compression	T_{O}	0.659	0.709	0.712	0.716	0.718
·	T_m	0.531	0.557	0.559	0.563	0.568
	WC	0.08	0.08	0.08	0.09	0.09
	RC	46.63	19.21	19.16	19.08	19.02
	EMC	19.42	21.44	21.48	21.52	21.55
Surface	MIU	0.213	0.369	0.372	0.375	0.380
	SMD	3.88	4.20	4.32	4.43	4.53

TABLE II Low-stress Mechanical Properties of LTP-Treated Wool Fabric

a larger cohesive force would be developed during the application of tensile stress. The increases in value of WT were probably because of the larger cohesive force being developed during the extension period, so that a larger amount of energy was required for extending the fabrics.

Tensile resilience (RT) refers to the ability of fabric to recover after applying the tensile stress. The reduced fabric RT value indicates that the fabric becoming difficult to recover the original shape after removing the applied tensile stress. After the LTP treatment, the overall RT was decreased, which was further decreased with the prolonged treatment time. The reduction in value of RT after LTP treatment could be explained by the increment of cohesive force between fibers and yarns. When the extension load was removed, the frictional restraint would be created simultaneously by those increased cohesive force which would hinder the extended fabrics to recover their original position. The recovery ability of the extended fabric was finally lowered resulting in a reduced value of RT. It was therefore concluded that the LTP-treated fabrics had difficulty to recover the original shape after removing the applied tensile stress. However, with the prolonged treatment time from 5 to 30 min, it was found that decrease in RT value was not substantial.

Extensibility (EMT) is another interesting factor associated with the tensile properties of the fiber. It is the percentage of the extended length after applying a known tensile stress to the fabric when compared with the initial length. The greater the value of EMT, the larger will be the elongation of the fabric under a known applied stress. Under the influence of the LTP treatment, the fabrics showed a reduced EMT value and the reduction further enhanced with prolonged treatment time. This phenomenon could be because of the increasing interaction force between the fibers and yarns, which reduced the relative movement of the fibers and yarns during the extension period and also restricted the elongation of the fabrics. From the view point of shaping and sewing, the decrease in the fabric extensibility could adversely affect the tailorability. However, a high level of EMT could cause an excessive hygral expansion of the wool fabric leading to puckering problems in the tailored garment at various ambient relative humidity conditions. On the whole, the EMT of the LTP-treated fabric was still acceptable in the laying-up process.

Shearing properties

The shearing properties of the LTP-treated wool fabric were summarized in Table II. The shearing properties consisted of shear rigidity (*G*), shear stress at 0.5° (2HG), and at 5° (2HG5) shear angle, respectively. The shear rigidity (*G*) reflects the ability of the fabric to resist shear stress. After the LTP treatment, there was a significant increase in the value of *G* of the wool fabrics, but the increment was enhanced slightly with the prolonged treatment time.

The fabric recovery ability after applying the shearing stress can be reflected by the shear stress values at 0.5 and 5° shear angles. The greater values of the shear stress, the worse will be the recovery ability of the fabric. It was observed that after the LTP treatment, a significant increase in 2HG and 2HG values (more than 200% increased when compared with the untreated fabric) were obtained. However, the LTP treatment showed similar effect on the shear stress properties with the prolonged treatment time.

Shear is an important determinant of the handle and drape of fabrics. The shear rigidity reflects the subjective handle of the fabric, i.e., increasing the shearing rigidity will enhance the subjective stiffness of the fabric. After the LTP treatment, there was a very high increase in fabric shear rigidity and shear stress. The high shear rigidity indicated that draping and three-dimensional forming as required in tailoring for the LTP-treated fabric would be very difficult. On the other hand, the LTP-treated fabric exhibited a higher degree of inelasticity in shear as indicated by the extremely large values of shear stress. The shear rigidity of the fabric primarily depends on yarn interaction, i.e., an increase in yarn interactions will normally increase shear rigidity. The increased value of G of the LTP-treated fabrics implied that the intervarn friction in wool fabrics was increased after the LTP treatment. To overcome the rigid effect caused by the LTP treatment, finishing process such as softening should be applied to eliminate this deficiency.

Bending properties

The results of the bending properties of the LTPtreated fabrics were summarized in Table II. The bending properties have important effects on both the handle and tailoring performance of the fabric. In Table II, the bending properties of the LTP-treated fabric studied included the bending rigidity (B) and bending moment (2HB). The overall values of B of the LTP-treated fabrics increased as the duration of treatment time increased. However, further increment after 5 min of treatment time was not so much even at the longest treatment time, i.e., 30 min, in this study.

The LTP-treated fabrics had a dramatic increase in the values of 2HB, i.e., more than 115% increase. The bending moment (2HB) reflects the recovery ability of the fabric after bending. The smaller the values of the 2HB, the better will be the fabric bending recovery ability. In comparison, as the treatment time increases, the value of 2HB increased correspondingly.

The increase in the values of *B* and 2HB of the LTP-treated fabric will greatly reduce the fabric flexibility and elastic recovery from bending, which in turn affects the fabric tailoring, draping, and wear.

Compression properties

The results of compression properties of the LTPtreated fabric are shown in Table II, which includes the fabric thickness at 0.5 (T_0) and 50 (T_m) gf/cm² pressure, compressional energy (WC), compressional resilience (RC), and compressibility (EMC). It was obvious that, after the LTP treatment, the fabric thickness (T_0 and T_m) were increased and the degree of increment enhanced with the prolonged treatment time, but the results were quite similar. The increased fabric thickness reflected that the LTP treatment fabrics would be complete than the untreated fabric in fabric handle.

Generally speaking, the compressibility (EMC) indicates the change in the thickness of the LTP-treated fabrics and when the EMC value increases, the fabric handle will become complete. After the LTP treatment, the EMC values of the LTP-treated fabric with different treatment durations were increased to a similar extent.

On the other hand, the surface raising effect caused by the LTP treatment resulted in no significant change in the value of WC. The WC value implies the fluffy feeling of the fabric. When the values of WC are increased, the fabric will appear fluffier. In the present study, the WC values did not show significant change after the LTP treatment.

Another important property obtained from the compressional hysteresis curve is the compressional resilience (RC). This property can help to determine the recoverability of the fabric after the compression deformation. When the value is low, the retention ability of deformation after compression will be good. After the LTP treatment, there is a remarkably reduction in the fabric compressional resilience. Such a reduction in compressional resilience could be probably associated with the increased cohesive forces between the yarns because of the roughening effect imparted by the LTP treatment within yarns and hence blocked the recovery of the extended fabrics. As the treatment time prolonged, the values of RC decreased gradually, but not significant.

Surface properties

The results of the surface properties of the LTPtreated fabrics including the coefficient of friction (MIU) and geometrical roughness (SMD) of the fabric surface were summarized in Table II.

The MIU reflects the fabric smoothness, roughness, and crispness. After the LTP treatment, the values of MIU increased significantly and the increment was further enhanced with the prolonged treatment time. The increment in value of MIU indicated that the LTP-treated fabric surface became less smooth and rougher.

On the other hand, the SMD shows the even characteristics of the fabric surface. The greater the SMD value, the less even will be the fabric surface. LTP treatment in this study obviously increases the surface evenness of wool fabric, and the prolonged treatment time also played an important role to enhance the evenness of the wool fabric. It was evident that upon LTP reaction, the plasma species would bombard on the fabric surface resulting in etching effect, which could cause the changes in the evenness of the fabric surface, as shown in Figure 2, and hence altering the surface properties of the fabric.^{12,13}

Air permeability

In this study, air permeability of the LTP-treated fabrics expressed as air resistance (R) was investigated and the results were summarized in Table III. The LTP treatment increases the *R* value of the fabric with treatment time. The air permeability depends on the construction characteristics of the yarns and fibers in which a large proportion is occupied by air space. There are some factors affecting the air permeability of the fabric, e.g., the fabric structure, thickness and surface characteristics, etc. It is known that LTP treatment do not have influence on the fabric structure, therefore the change in *R* values is regarded as being closely related to the fabric thickness and surface characteristics. As discussed earlier, LTP treatment increases the fabric thickness and alters the surface morphology. It is possible to say that LTP treatment induces a certain degree of roughness^{9,12} as shown in Figures 3–5, on the fabric surface which increases the fabric thickness and changes the fabric surface characteristics. These changes act as a boundary to hinder the air flow through the fabric, thus resulting in a reduction of the air permeability of the fabrics.

Insulation properties

Table IV shows the insulation properties of fabric (expressed as warm/cool feeling, q_{max}) of the fabrics with the variation of treatment time. The value of the $q_{\rm max}$ indicates the heat loss per unit area under the condition of 10°C temperature difference. It reflects the instantaneous warm/cool feeling sensed when there is an initial contact of the fabric with the surface of the skin. A higher value of q_{max} denotes that there is a more rapid movement of heat from the skin to the fabric surface, which will provide a cooler feeling. It can be observed that the q_{max} value of the LTPtreated fabric show a reduction with prolonged treatment time. This implies that the LTP-treated fabric have a better warmth-retention property when compared with the untreated fabric. The insulation properties of a textile fabric depends a great extent on the air trapped within it. As mentioned earlier, the LTP treatment provides an etching effect on the fiber surface and such etching effect increases fabric surface roughness, voids, and space as shown in Figures 3-5,

TABLE III Air Permeability of LTP-treated Wool Fabric

Plasma treatment time (min)	Air permeability, <i>R</i> (kPa s/m)
0	6.79
5	7.7
10	7.75
20	7.82
30	7.9

TABLE IV Warm/Cool Feeling of LTP-treated Wool Fabric

Plasma treatment time (min)	Warm/cool feeling, q_{max} (W/cm ²)
0	0.155
5	0.128
10	0.124
20	0.120
30	0.113

which may increase the amount of the air trapped between the yarns and fibers.¹⁴ In addition, the air permeability results indicate that the LTP-treated fabrics have poorer air permeability, therefore, the air trapped inside the fabric will not escape easily. The air so trapped inside the fabric can act as a good insulation medium and help to prevent the heat loss of the fabrics.

CONCLUSIONS

The low-stress mechanical properties, air permeability, and insulation properties of LTP-treated wool fabrics had been investigated. It was revealed that the LTP treatment could influence not only the mechanical properties, but also affect the air permeability and thermal properties of the wool fabrics. The changes in the mechanical properties of the wool fabric could be explained by the intervarn and interfiber frictional force imparted by the LTP etching action. The change in the air permeability of the LTP-treated fabrics was found, which is probably because of the plasma action increasing the fabric thickness and changing the fabric surface morphology. In addition, the change in the insulation properties of the LTP-treated fabrics was in good agreement with the earlier findings and could be attributed to the amount of trapped air between the yarns. Although the LTP treatment showed significant influence on the properties of the wool fabric, the prolonged exposure time in the LTP treatment did not affect the mechanical properties, air permeability, and thermal properties of the LTP-treated wool fabrics. With the use of LTP treatment, the comfort properties of the wool fabric were changed to various extents in which some properties were improved and some were adversely affected. Therefore, it implied that careful selection of operation conditions should be considered for LTP treatment for industrial application. In concluding, the LTP treatment for modification of wool fabric has obviously high industrial application potential as it is an environmentally friendly dry process, which does not involve any of the solvents and reagents for the wet chemical process.

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