Thermal Properties of Wool Fabrics Treated in Atmospheric Pressure Post-Discharge Plasma Equipment

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ABSTRACT

A plain-weave wool fabric has been treated in a roll-to-roll atmospheric pressure, post-discharge plasma equipment at three fabric speeds. The thermal properties of the treated fabric have been investigated by means of an Alambeta instrument. The thermal resistance and thermal diffusivity increased after the plasma treatment while the thermal absorptivity and volumetric heat capacity decreased. The longer the plasma-to-fabric exposure time, the more marked the change in the fabric's thermal properties. Since thermal conductivity was unaffected after the treatment, the thermal properties changed due to the increase in thickness that was observed after the plasma treatment. A validated model has been used to predict the human psychophysical perception of the fabric hand. As a consequence of the more voluminous structure, a softer and warmer hand has been predicted for the treated fabrics compared to the untreated ones.

Keywords: wool; atmospheric plasma; thermal properties; hand.

INTRODUCTION

Low temperature plasma (LTP) technologies are regarded as appealing tools for the pre-treatment and finishing of wool fabrics. Plasma processing does not require the use of water or chemicals, resulting in an economical and ecological process [1]. The ability of an LTP treatment to increase the wool dyeability [2-6] and shrink resistance [7-13] has been extensively studied in the last decades. Much research has been published concerning wool chemical, morphological and physical characterizations after a plasma treatment. Since plasma action is limited to the depth of some tens of nanometres below the fiber surface, much research regarding the change of the wool's superficial properties, in particular the surface chemistry and morphology, has been widely investigated by means of an X-ray photoelectron spectroscopy (XPS) [3, 4, 7, 10, 12-18] and scanning electron microscopy (SEM) [3, 5, 7, 9, 10, 12, 13, 16, 18-20] techniques.

Non-polymerizing plasma acts by removing the covalently bound fatty acid layer (F-layer) from the epicuticle, the very surface of wool fibers, and oxidizes the disulfide bridges of the exocuticle, the intermediate layer between the epicuticle and the underlying hydrophilic protein material [11, 17]. These phenomena change the wool's surface character from hydrophobic to hydrophilic. The SEM analysis visibly shows how different the effects of a plasma treatment can be, ranging from an unchanged morphology on a micro-scale [10, 12, 20] to a great deal of damage to the fiber structure with the formation of grooves and micro-craters, and scale erosion [3, 5, 16]. The removal of the surface material by plasma etching is responsible also for the change of low-stress mechanical properties [21-23] and friction properties [24], the former being bound to the hand performance and the latter to shrink proofing.

In this wide panorama of investigated properties, only a few studies focused on the thermal properties of plasma treated wool fabric. Kan and Yuen [22] measured the heat loss per unit area from fabrics exposed to oxygen low-pressure plasma (LPP), exposure times between 5 to 30 minutes, for the instantaneous warm/cool feeling sensed at the initial contact between the fabric and skin. An increase of heat retention was observed for the treated fabrics compared to the untreated ones. The authors explained that the behavior was caused by the etching effect increasing the fabric void degree, and thus the amount of air trapped between the yarns and fibers. Karahan, et al. [19], measured the thermal resistance and thermal conductivity of knitted wool fabrics treated in a glow-discharge plasma system operating under atmospheric conditions, using air and argon as process gases, with exposure times between 20 and 60 seconds and three power levels (50, 100 and 130 W). They observed an increase in thermal resistance and a decrease in thermal conductivity as exposure time and power was increased.

In this study, a comprehensive study of the thermal properties of a plain-weave worsted wool fabric treated in a roll-to-roll atmospheric pressure, post-discharge plasma equipment has been carried out. Post-discharge technology is supposed to be gentler than other plasma technologies because the sample is not located in the same place where the plasma is being generated; thus, the fabric is not in contact with the short-lived active species present in a freshly-generated plasma but only with the most stable and metastable non-ionic species [16]. Even though there have been dissenting opinions on this issue [21, 23], it can be reasonably stated that a gentle treatment is required for wool, since a harsh hand could be imparted by a strong plasma action. This harshness could be a possible major drawback of plasma treatment of wool.

Treatments at three fabric speeds have been carried out and the fabric thermal properties, such as thermal resistance, thermal absorptivity, thermal diffusivity, volumetric heat capacity and thermal conductivity, have been investigated using an Alambeta instrument. In order to verify if the plasma treatment under investigation affects the fabric hand, a validated model estimating the primary hand named *softness and warmth* [25] has been used to predict the hand performance of the treated and untreated fabrics. The model combines six selected low-stress mechanical properties, the fabric weight per unit area and thermal absorptivity.

MATERIAL AND METHODS

Textile materials were standard undyed worsted wool fabrics (ISO 105-F01, 125 ± 5 gm^{-2}). Strips of 400 mm were cut along the weft direction, dried in the oven at 60 °C for 90 minutes before being processed in the plasma equipment. The fabric has a 1/1 plain-weave structure. More fabric characteristics are reported in a previous study [20].

The atmospheric pressure plasma jet (APPJ) equipment, based on a post-discharge plasma jet generator, processes a 400 mm wide flat material by transferring it from roll to roll. The nitrogen stream (0.4 Nm^3/min) enters the plasma generator, is converted into plasma and then flows out through a 380x2 mm^2 exit slot. The generator, consisting of two cylindrical and coaxial electrodes, powered with a high voltage supply, was set to 4.0 kW. The internal cylinder supporting the fabric is perforated according to a pitch given by 7 alignments of 40 flared 4 mm drills in its upper surface. The jet of plasma impinges on the fabric and spreads over its surface. The penetration of plasma through the fabric is promoted by suction into the collector placed along the axis of the cylindrical geometry. The gas collected can be properly disposed of or recirculated into the plasma generator by applying a suitable blowing device.

Three experimental conditions were investigated, keeping the plasma generator parameters constant and changing the fabric speed (1, 3 and 6 m/min). After the plasma treatment, the samples were conditioned under standard laboratory conditions for at least 24 hours before testing.

Steady-state and transient-state thermal properties were measured with an Alambeta instrument (model T675, Zweigle, Germany), which simulates the heat flow taking place from human skin to a fabric during a short initial contact [26]. The temperature gradient between the lower non-heated measuring head and the upper heated measuring head was adjusted to 10 °C, while the clamping pressure of the upper measuring head exerted over the fabric was 250 Pa.

RESULTS AND DISCUSSION

Thermal conductivity (λ), thermal resistance (R_{ct}), volumetric heat capacity ($c\rho$), thermal diffusivity (α), thermal absorptivity (b) and fabric thickness at 250 Pa (h) with different fabric speeds are shown in *Table I*. Each measurement was repeated five times and the average values with the standard deviations are reported. The percentage variation between the property of the treated fabric and that of the untreated one is reported in brackets.

TABLE I. Thermal properties of the untreated and treated fabrics.

	Untreated	6 m/min	3 m/min	1 m/min
h [μm]	393 ±27	424 ±15 *(+8%)*	436 ±11 *(+11%)*	448 ±13 *(+14%)*
λ [$10^{-3}Wm^{-1}K^{-1}$]	43.0 ±3.3	42.2 ±0.9 *(-2%)*	42.8 ±1.2 *(-0.5%)*	42.8 ±1.5 *(-0.5%)*
R_{ct} [$10^{-3}m^2KW^{-1}$]	9.15 ±0.34	10.05 ±0.24 *(+10%)*	10.19 ±0.18 *(+11%)*	10.46 ±0.26 *(+14%)*
$c\rho$ [$10^3WsK^{-1}m^{-3}$]	573 ±52	490 ±38 *(-14%)*	480 ±46 *(-16%)*	447 ±19 *(-22%)*
α [$10^{-9}m^2s^{-1}$]	75.3 ±8.0	86.6 ±8.4 *(+15%)*	89.7 ±9.2 *(+19%)*	96.1 ±6.0 *(+28%)*
b [$Ws^{1/2}m^{-2}K^{-1}$]	156.9 ±10.4	143.6 ±4.9 *(-8%)*	143.2 ±5.6 *(-9%)*	138.2 ±3.4 *(-12%)*

It can be observed that fabric thickness increased in all experiments and the lower the fabric speed, i.e., the longer the exposure time, the larger the increase in thickness. This phenomenon has been already observed by Kan and Yuen [22]. Nevertheless, in their study, the exposure time had little effect on the fabric thickness, whose increase was around 8-9% regardless of the exposure time ranging between 5 and 30 minutes. Karahan, et al. [19], carried out tests at different exposure times, powers and gas types and the data showed that power was the most relevant parameter affecting the increase in thickness. A considerable increase in thickness was observed by Sun and Stylios after a 60-seconds oxygen low pressure plasma treatment [21]. They contributed this result to a roughening effect on the treated fiber surface that increases the space between the fibers and yarns.

Table II shows a summary of previous results with regard to the increase in thickness of the wool fabrics treated in the plasma equipment with this study's presented results.

TABLE II. Comparison between this work and literature works reporting fabric thickness increase after a plasma treatment.

	This work	Kan and Yuen [22]	Karahan et al. [19]	Sun and Stylios [21]
Fabric structure	1/1 plain	2/1 twill	knitted	twill
Mass per unit area [g m^{-2}]	125	180	251	237
Untreated fabric thickness [mm]	0.393	0.659	1.634	1.382
Plasma technology	Hybrid corona/dielectric barrier discharge	Glow-discharge	Glow-discharge	Glow-discharge
Plasma-to-fabric contact	remote	in situ	in situ	in situ
Pressure	atmospheric	under vacuum	atmospheric	under vacuum
Gas type	nitrogen	oxygen	air, argon	oxygen
Power [W]	4000	80	50, 100, 130	300
Exposure times [s]	4-12	300-1800	20-60	60
Fabric thickness increase	8-14%	8-9%	7-12%	15%

It is noteworthy to observe that different fabric structures, different plasma technologies and process parameters lead to the same increase in thickness. The observed increase in thickness could be ascribed to the electrostatic charge induced in the electric field of plasma [20], which may rearrange the yarn and the fiber structure in a more voluminous texture. This hypothesis was confirmed by the observation that power affects thickness more than exposure time or gas type, as shown by Karahan, et al. [19]: the higher the power and the higher the electric field intensity and the electrostatic charge. The ablation of the superficial fatty acid layer, which acts as a lubricant in yarn-to-yarn friction, opposed the recovery of the initial thickness even when the electric field is removed.

Alambeta calculates the thermal resistance (R_{ct}) from the measured heat flux through the fabric thickness (j_q) in steady conditions when a temperature gradient is set between the top and bottom side of the fabric according to the Fourier's law:

$$j_q = \frac{1}{R_{ct}}(T_{top} - T_{bottom}) = \frac{\lambda}{h}(T_{top} - T_{bottom}) \qquad (1)$$

As shown in *Table I*, thermal resistance increased to the same extent as the fabric thickness. This means that thermal conductivity, an indirect measure of the ratio of fabric thickness and thermal resistance, does not change. Thus, the thermal resistance increased due to a larger amount of trapped air, which is actually the most important factor governing the thermal insulation of textiles [27]. It is well known that a fabric is a heterogeneous material made of a solid phase and trapped air, whose thermal conductivity (0.025 $Wm^{-1}K^{-1}$ at 20 °C) is much lower than the solid phase conductivity. Thus, a heat flux occurs mostly through the solid phase rather than the air phase. Since the plasma treatment has not changed significantly, the thermal conductivity is unchanged in the amount of the solid phase of the treated sample, as confirmed by a negligible weight loss. On the other hand, Karahan et al. [19], measured a slight decrease in thermal conductivity of the treated samples. This could be due to a significant erosion of the superficial layer that affects the weight of the sample. This hypothesis is confirmed by the SEM images of the treated samples, in which scale rounding, micro cracks, recesses and tiny grooves appear. SEM images of the samples treated in this study are reported elsewhere [20] and no visible changes appear. Thus, the post-discharge treatment combined with a low exposure time assures a soft treatment of wool fabrics.

Fabric volumetric heat capacity decreased significantly after the treatment as shown in *Table I*. This result is due to a reduction of the fabric bulk density, which is again a consequence of the increase in thickness. Since the air volumetric heat capacity (1.2 kJ $K^{-1}m^{-3}$) is much lower than the solid phase one, more air trapped per volume unit leads to a lower overall heat capacity.

When the measuring head of Alambeta at temperature T_1 is brought into contact with a fabric at temperature T_0, with $T_1>T_0$, the temperature profile within the fabric thickness at any time is obtained from the solution of the differential equation:

$$\frac{\partial T}{\partial t} = \alpha \frac{\partial^2 T}{\partial x^2} \tag{2}$$

where α is the thermal diffusivity of the fabric, which is considered a pseudo-homogeneous solid. Thermal diffusivity is defined as the ratio between thermal conductivity and the volumetric heat capacity:

$$\alpha = \frac{\lambda}{c\rho} \tag{3}$$

For short contact times, the fabric can be considered as a flat semi-infinite body, that is, the heat flux involves a much thinner layer than the fabric thickness. Moreover, it is assumed that the fabric heat capacity is so low that the fabric surface temperature instantly adjusts its temperature to that of the measuring head when the two bodies are brought in contact.

Thus, the initial and boundary conditions for the above differential equation are the following:

$$\begin{aligned} t&=0 \quad T=T_0 \\ x&=0 \quad T=T_1 \\ x&=\infty \quad T=T_0 \end{aligned} \tag{4}$$

For these initial and boundary conditions, the solution of Eq. (2) is the following:

$$T - T_0 = erfc\left(\frac{x}{2\sqrt{\alpha t}}\right)(T_1 - T_0) \tag{5}$$

where *erfc* is the complementary Gaussian error function. The expansion series of *erfc* for $x \rightarrow 0$ truncated at the first order is:

$$erfc\left(\frac{x}{2\sqrt{\alpha t}}\right) \cong 1 - \frac{x}{\sqrt{\pi \alpha t}} \tag{6}$$

Thus, by substituting Eq. (6) in Eq. (5) and applying the Fourier's conductivity law, the heat flux at the top surface of the fabric (x=0) at any time will be:

$$j_q(x=0,t) = -\lambda\left(\frac{\partial T}{\partial x}\right)_{x=0} = \frac{\lambda}{\sqrt{\pi \alpha t}}(T_1 - T_0) = b\frac{T_1 - T_0}{\sqrt{\pi t}} \tag{7}$$

where *b*, named thermal absorptivity, is defined as:

$$b = \frac{\lambda}{\sqrt{\alpha}} \tag{8}$$

The higher the thermal absorptivity, the higher the skin-to-fabric heat flux when the fabric is brought in contact with a human body. Thus, fabrics with high thermal absorptivity values are initially cool to the touch, since the thermal receptor feels the extent of the heat flux from the skin.

As shown in *Table I*, fabric thermal diffusivity increases with the increasing plasma exposure time as a consequence of the trends of thermal conductivity and heat capacity. Thermal diffusivity plays an important role in the transient-state heat transfer, describing how fast heat propagates through a fabric. The treated fabrics showed a higher value of thermal diffusivity, meaning that they adjust their temperature to that of their surroundings more rapidly with increasing the plasma exposure time. On the other hand, the fabric thermal absorptivity decreased after the plasma treatment, meaning that the treated fabrics have a warmer hand than the untreated ones. Since the fabric structure is more voluminous after the plasma treatment, the skin-to-fabric contact points decrease and a lower heat flux occurs from the body to the fabric when the fabric is put in contact with the body.

The observation that the atmospheric plasma treatment made the wool fabric more voluminous introduces another very important aspect, which is the influence of this plasma process on the fabric hand characteristics. To investigate this matter, the information and experience gained in previous study, reported and discussed below, were used.

As discussed by Bishop [28] it is likely that the sensory responses to handling textiles should be related to measurable stimuli represented by their mechanical, surface, thermal, optical and aural properties. Thus, the complex sensation obtained through the active manipulation of a fabric by a human hand can be related to instrumentally-measured fabric properties through a model.

The responses of the fabric to low-stress bending, shearing and compression are among the most influencing parameters as far as fabric hand is concerned. Besides the mechanical behavior, the thermal component evidently contributes to the perception of touch by handling a fabric: specialized sensory receptors of the human skin detect temperature changes due to transient heat flow to or from the body surface, producing the thermal sensation of warmth or coolness [29]. Mazzuchetti, et al. [25], and Rombaldoni, et al. [30], investigated the possibility of using low-stress mechanical properties measured by the FAST (Fabric Assurance by Simple Testing, CSIRO, Australia) instruments and thermal absorptivity measured by the Alambeta equipment for predicting the human psychophysical perception of a specific hand for suit fabrics made from animal fibers (wool, mohair, cashmere and alpaca). In particular, the *softness and warmth* and *crispness and coolness* hand feel were related not only to the mechanical properties of the fabrics, but also to the perception of heat transfer. In the present study, in order to find whether the atmospheric pressure plasma treatment under investigation did change the fabric hand somehow, the model for predicting the *softness and warmth* hand was used [25], as it was demonstrated that the two hands were highly correlated: high *crispness and coolness* hand values corresponded to low *softness and warmth* hand values, and vice versa [30].

TABLE III. Objectively-determined fabric properties and predicted *softness and warmth* hand values of the treated and untreated values.

k = -51.898			**Untreated**	**6 m/min**		**3 m/min**		**1 m/min**	
***ith* parameter**		a_i	x_i	x_i	Δy	x_i	Δy	x_i	Δy
1	*T100* [mm]	-23.824	0.296	0.301	-0.173	0.305	-0.310	0.312	-0.545
2	*ST* [mm]	9.704	0.103	0.130	**+0.981**	0.138	**+1.233**	0.142	**+1.353**
3	*B* [μNm]	-5.656	4.31	4.39	-0.045	4.39	-0.045	4.74	-0.234
4	*E100* [%]	6.208	5.75	5.58	-0.081	5.37	-0.184	5.17	-0.287
5	*G* [Nm^{-1}]	-3.806	14.0	14.8	-0.092	14.8	-0.092	17.3	-0.350
6	*F* [mm^2]	-7.472	0.46	0.47	-0.070	0.44	0.144	0.45	0.071
7	*W* [gm^{-2}]	40.390	125	125	0.000	125	0.000	125	0.000
8	*b* [$Ws^{1/2}m^{-2}K^{-1}$]	-15.604	156.9	143.6	**+0.598**	143.2	**+0.621**	138.2	**+0.859**
	y (*softness and warmth* hand value)		**0.8**	**2.0**		**2.2**		**1.7**	

$$\Delta y(x_i) = a_i \log x_i - a_i \log x_{i,untreated} = a_i \log\left(\frac{x_i}{x_{i,untreated}}\right)$$

The model combines six low-stress mechanical parameters, the weight per unit area and thermal absorptivity. The mathematical form of the model is the Weber-Fechner law, a well-established psychophysical model to translate instrumentally measured fabric properties of a wide range of fabrics into corresponding hand parameters [28, 29]:

$$y = k + \sum_{i=1}^{8} a_i \log x_i \qquad (9)$$

where k and a_i are regression constants, x_i is the value of the i^{th} parameter of the model and y is the predicted value of the *softness and warmth* hand value, ranging from 0 to 10 with increasing the intensity of the tactile sensation. The higher the value of this index, the softer and warmer the fabric hand is expected to be.

The eight objectively-determined model parameters x_i are the following: thickness at 9.81 kPa *T100* (mm), surface thickness *ST* (mm), bending rigidity *B* (μNm), extensibility at 98.1 N/m *E100* (%), shear rigidity *G* (Nm^{-1}), formability *F* (mm^2), weight per unit area *W* (gm^{-2}), and thermal absorptivity *b* ($Ws^{1/2}m^{-2}K^{-1}$). For each fabric, the mechanical properties were measured by means of a FAST integrated set of instruments within a previous work [20], while thermal absorptivity has been measured in the present study. The x_i values, the regression constants (k and a_i) and the predicted *softness and warmth* hand values for the treated and untreated fabrics are shown in *Table III*.

The fabric under investigation is a close woven fabric, not a fluffy fabric as confirmed by the very low value of the *softness and warmth* hand of the untreated fabric itself. After the plasma treatment, the hand value has slightly increased, meaning that the wool fabric has become slightly softer and warmer. The column named *Δy* shows the contribution of each parameter to the change of the hand value of the treated fabrics with respect to the untreated one. It is worthwhile noting that the increase of surface thickness and the decrease of thermal absorptivity, after the plasma treatment, show the strongest influence on the change of the primary hand defined as *softness and warmth*. The surface thickness (*ST*) is obtained from the difference of the thicknesses measured at two different loads:

$$ST = T2 - T100 \quad (10)$$

where *T2* is the thickness at 0.196 kPa and *T100* is the thickness at 9.81 kPa.

The surface thickness is a measure of fabric compressibility and is bound with the softness feeling. After the plasma treatment, surface thickness increased between 26-38% due to the air entrapment in the fabric structure. The expected slight change in the hand value, due to the plasma treatment, is a very interesting result as it proves that it has been possible to modify the wool surface properties without damaging the fabric hand. In fact, the gentle treatment under investigation was effective for the modification of wool character, as shown in a previous study [20], having increased dramatically its hydrophilicity. The change of wool hydrophilicity was proven to be great enough to improve dyeability of wool fabric [31, 32].

CONCLUSIONS

The thermal properties of a wool fabric treated in atmospheric pressure, plasma equipment at different exposure times were investigated. The thermal conductivity was unaffected while the thermal resistance increased after the plasma treatment. The key factor for interpreting the change of the thermal properties is the increase in thickness observed after the plasma treatment. More air trapped in the fabric is responsible for the change of the fabric thermal properties. The use of a specific model for predicting the hand of fabrics made from animal fibers confirmed that the treated fabrics were slightly softer and warmer than the untreated ones. This outcome was mainly due to the increase of surface thickness and the decrease of thermal absorptivity after the plasma treatment.

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