

## Dimensional Properties of Low Temperature Plasma and Silicone Treated Wool Fabric

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(Received February 10, 2001; Revised March 7, 2001; Accepted March 15, 2001)

**Abstract:** Three different silicone polymer systems, such as aminofunctional, epoxyfunctional, and hydrophilic epoxyfunctional silicone polymers, were applied onto plasma pretreated wool fabric to improve the dimensional properties. The results showed that the plasma pretreatment modified the cuticle surface of the wool fiber and increased the reactivity of wool fabric toward silicone polymers. Felting shrinkage of plasma and silicone treated wool fabric was decreased with different level depending on the applied polymer system. Fabric tear strength and hand were adversely affected by plasma treatment, but these properties were favorably restored on polymer application. Therefore, it has been concluded that the combination of plasma and silicone treatments can achieve the improved dimensional stability, and better performance properties of wool fabric. The surface smoothness appearances of treated fabrics were measured using a new evaluation system, which showed good correspondence with the results of KES-FB4 surface tester

**Keywords:** Silicone, Plasma, Wool, Dimensional stability, Hand

### Introduction

Low temperature plasma technology is being increasingly regarded as an environment-friendly alternative for wool chlorination in pretreatment processes[1,2]. This technology can modify the fiber surface, increasing surface wettability and improving shrink resistance[3-5]. Although the low temperature plasma treatment can improve the surface properties of wool fibers, an apparently harsher handle is imparted on the treated fiber[6]. Thus, in this study, we selected three types of silicone polymers that had been reported to provide dimensional stability as well as soft hand and applied them to low temperature plasma pretreated wool fabric[7]. Then, the changes in mechanical, dimensional and performance properties of the treated wool fabrics were investigated and surface smoothness appearances were examined with the application of plasma and silicone polymers.

### Experimental

#### Materials

A scoured and crabbed plain-weave worsted fabric (150 g/m<sup>2</sup>) with 62 ends/inch and 64 picks/inch was used for the experiments. The silicone softeners selected for testing included Dow Corning<sup>®</sup>108 emulsion as aminofunctional silicone softener, and Dow Corning<sup>®</sup>4592 and hydrophilic Dow Corning<sup>®</sup>193 as epoxyfunctional silicone softeners. Triton X-100 was used as wetting agent. All other chemicals were of reagent grade and used without further purification.

#### Treatments

After air-cleaning wool fabric, oxygen plasma treatment

was subsequently carried out in plasma equipment manufactured by Vacuum Science Co. as shown in Figure 1.

It consists of four main parts such as: high vacuum pump, gas-feeding part, radio frequency generator and reaction chamber. The frequency and maximum power of the radio frequency generator were 13.56 MHz and 600 W, respectively. To process the wool fabric, the reactor chamber, 380 mm diameter and 250 mm height in dimension, was firstly evacuated to below 0.1 torr, and then 99.9% pure oxygen gas was injected at 50 cc/min flow rate. The samples were then treated by plasma discharge in different levels of power of 50, 100, and 150 W for 60 seconds. Finally, the fabrics were conditioned prior to the pad-dry-cure process.

The wool fabric samples were then impregnated in an aqueous bath (bath ratio=1:10) containing silicone polymers (2% owf) for 2 minutes and padded for 80±3% through squeeze rollers under 1.5 kg/cm<sup>2</sup>. The padded samples were then dried at 100°C for 3 minutes followed by curing at 130°C for 3 minutes. Treated samples then rinsed with fresh water and air-dried. Samples were stored under standard conditions (21°C and 65%RH) before other properties were evaluated.

#### Measurements

KES-FB system was used to test the mechanical properties of fabric samples under standard condition and sixteen mechanical properties were obtained for each sample. Then, the fabric primary hand value for men's summer suit was evaluated. Felting dimensional shrinkage was measured according to AATCC test method 99-1993. The performance properties of treated samples were evaluated using standard procedures, including breaking strength (ASTM D-1682-64), and Elmendorf tear strength (ASTM D-1424-81). All the measurements were repeated for the five equally treated samples and averaged. Surface analysis was carried out using ESCA MK II (LVG Scientific Ltd.) by irradiating a

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sample with monoenergetic soft X-rays and analyzing the energy emitted by electrons.

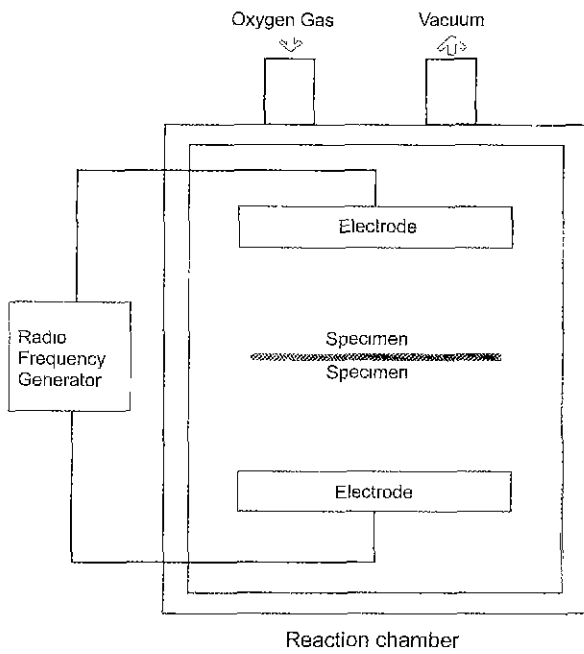


Figure 1. Parallel type of plasma treatment device.

## Results and Discussion

### Surface analysis

ESCA has been used for wool fiber by a number of researchers, who directed most of their interest and attention

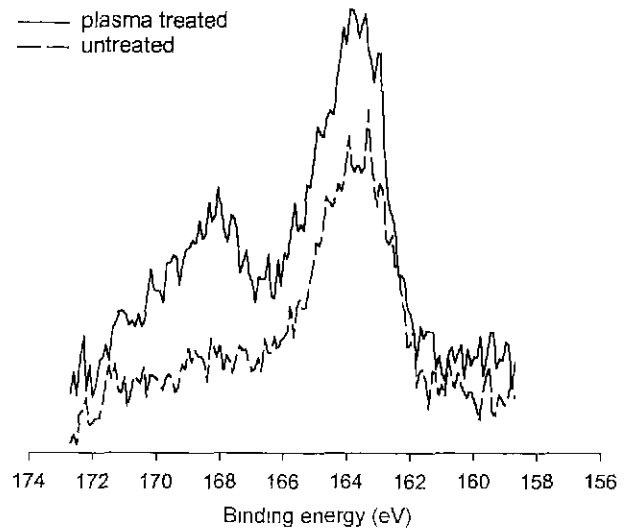


Figure 2. X-ray photoelectron spectra of  $S_{2p}$  using a narrow scan.

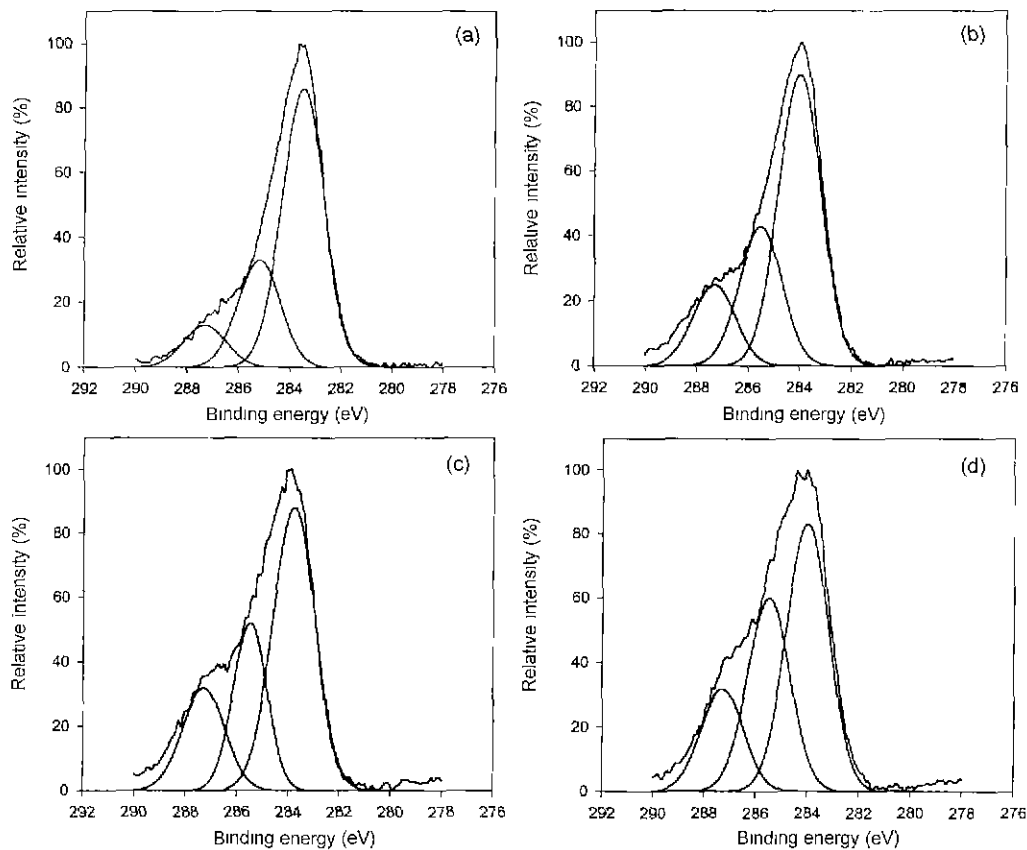


Figure 3. Curve fittings of  $C_{1s}$  spectra of wool fabric treated with plasma: (a) control, (b), (c), and (d); plasma treated at 50,100, and 150 W.

to the changes in the  $S_{2p}$  spectrum after various oxidative treatments[8]. Millard showed that the sulfur peak in untreated wool fiber occurred at a binding energy level of 164 eV, corresponding to cystine, while in wool exposed to an oxidizing plasma or corona discharge a second peak appeared at approximately 168 eV, corresponding to cysteic acid (R-SO<sub>3</sub>H) groups[9]. In Figure 2, therefore, the second peak of sulfur observed at 168 eV corresponds to cysteic acid groups induced by O<sub>2</sub> plasma treatment.

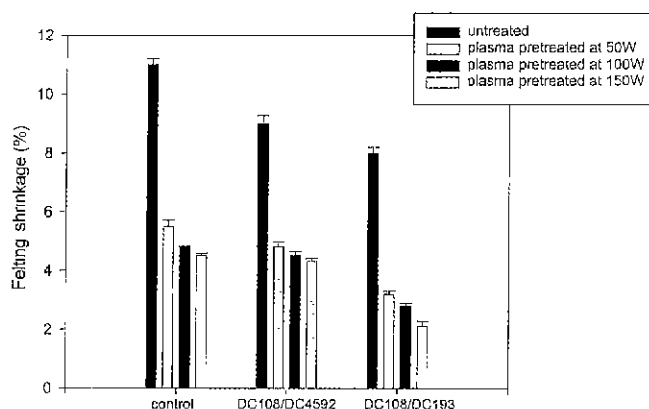
Peak deconvolutions of the C<sub>1s</sub> spectra of wool fabric are shown in Figure 3. Wave separation was divided into three components, assuming that peak 1 at 283.5 eV corresponds to -CH, peak 2 at 285.5 eV to -CO- and -CN-, and peak 3 at 287.3 eV to -COO- and -CON-[9]. It is clear from the Figure 3 that O<sub>2</sub> low-temperature plasma treatment of wool fiber increased peak 2 and peak 3 compositions considerably, and their relative intensities are summarized in Table 1. Thus the oxygen atoms induced on the wool surface might exist in the form of -C-O- and -COO-, which seems to play an important role in increasing the hydrophilicity of the wool surface.

### Felting shrinkage

Felting shrinkage of wool fabric is correlated with the frictional coefficient of the constituent wool fibers, and it is common knowledge that plasma treatments increase the dry and wet frictional coefficient in the scale and anti-scale directions[10]. However, the effect of the plasma process is

**Table 1.** Peak deconvolutions of C<sub>1s</sub> spectra of plasma treated wool fabric

Treatment	Relative peak area, %		
	-CH	-CO-	-COO-
Untreated	65.15	25.00	9.85
Plasma treated at 50 W	56.96	27.22	15.82
Plasma treated at 100 W	54.46	25.74	19.80
Plasma treated at 150 W	47.43	34.29	18.29



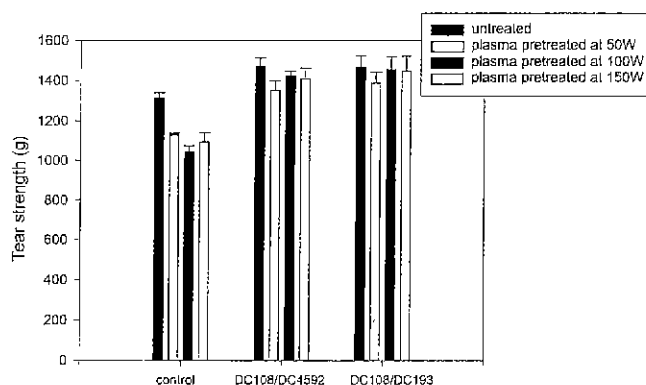
**Figure 4.** Felting shrinkage of plasma and silicone treated wool fabric in the warp direction.

attributed to several changes in the wool surface, such as the formation of new hydrophilic groups, partial removal of covalently bonded fatty acids belonging to the outermost surface of the fiber, and the etching effect[11,12]. Whereas the first two changes contribute mainly to increased wettability properties, the last change basically reduces the differential friction coefficients of the fibers and thus decreases the natural shrinkage tendency. We can see the decreased felting shrinkage by plasma treatment in Figure 4. The felting shrinkage is further decreased by post-application of silicone polymers. The extent of reduction in felting shrinkage depends significantly on the kind of polymer applied. The mixture of Dow Corning®108 and Dow Corning®193 exhibits a marked improvement in shrink resistance, whereas the mixture of Dow Corning®108 and Dow Corning®4592 exhibits lower improvement. As shown in Figures 2 and 3, concentration of anionic groups on the wool surface raised by plasma treatment will increase hydrophilicity of the wool surface. For this reason, the spreading and adhesion of hydrophilic silicone polymer on plasma pretreated wool fibers are relatively increased, therefore there is a further improvement in shrink resistance.

### Performance properties

It is well known that surface characteristics of fibers play an important role in the functional and aesthetic properties of fabrics, and many surface modifications by chemical treatments are able to improve the performance properties. In general, the function of silicone polymer is to maintain the mechanical properties of a garment as well as to impart a soft pleasing feel.

The tear strength of the wool fabric is reduced by plasma treatment, however, it has been improved with silicone polymer application as shown in Figure 5. That is, the silicone polymers restore the loss in tear strength resulting from the plasma modification. The increase in tear strength can be attributed to the formation of an elastic network on the fiber surface.



**Figure 5.** Tear strength of plasma and silicone treated wool fabric in the warp direction.

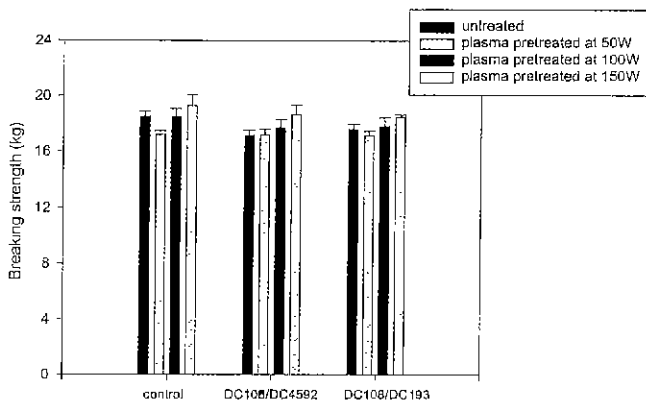


Figure 6. Breaking strength of plasma and silicone treated wool fabric in the warp direction.

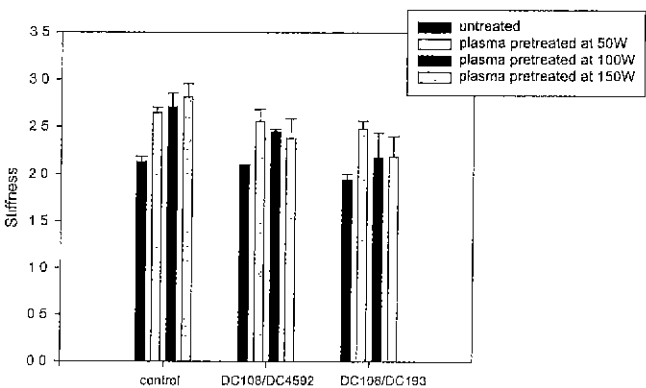


Figure 7. Stiffness of plasma and silicone treated wool fabric.

The relationship between the breaking strength and the plasma-silicone treatment is illustrated in Figure 6. It shows that there are slight changes in breaking strength due to the silicone post application.

As the result of surface roughening, almost all samples of plasma treated wool fabric exhibit more or less firm or harsh handle in Figure 7. however, the silicone post application significantly decreases the stiffness values. Clearly, the surface application of silicone polymer masks any adverse effect on fabric handle imparted by the plasma treatment.

**Smoothness appearance**

KES-FB4 surface tester measures objectively the friction coefficient and geometrical roughness of fabric surface along a given direction at a time. With this tester, we already observed the effects of plasma and silicone treatments on the surface properties of wool fabric. Roughness values measured by KES-FB4 surface tester showed the changed extent of roughness. however, could not give an unified topological parameter on surface roughness or smoothness of fabric. Therefore, we applied a new evaluation system to plasma and silicone treated samples and evaluated the effects of

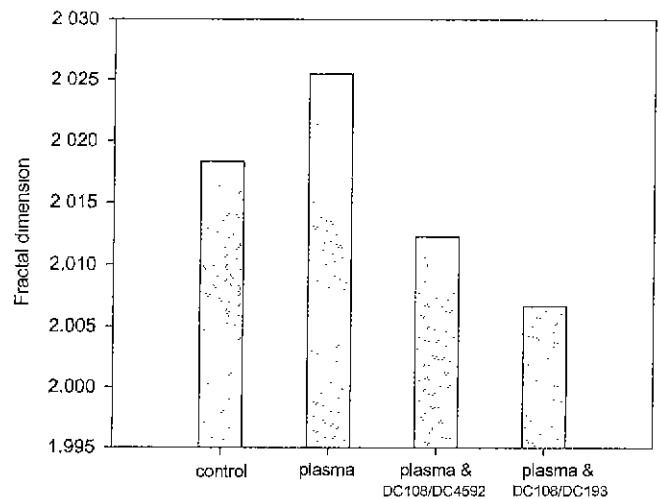


Figure 8. Fractal dimension of plasma and silicone treated wool fabric.

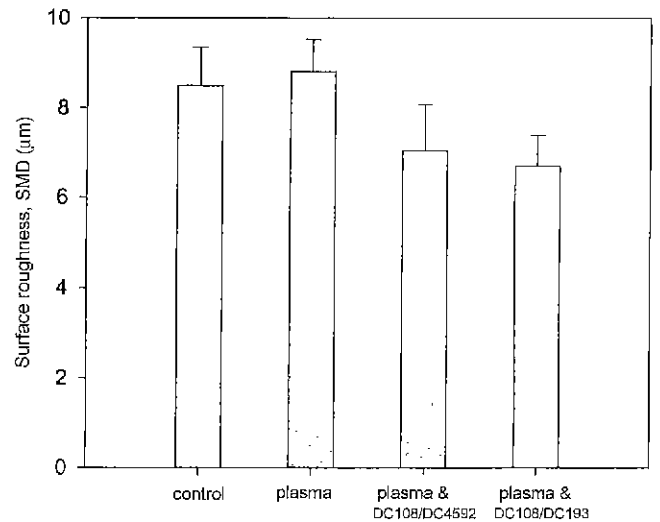


Figure 9. Surface roughness of plasma and silicone treated wool fabric

those treatments on the surface morphology of the samples. This system was developed using the stereovision algorithm and fractal geometry and includes the quantitative values such as fractal dimension to describe the random structures of fabric smoothness[13]. With this system, smoothness appearances of wool fabric plasma pretreated at 100 W and silicone treated were evaluated. Figure 8 shows well the influence of plasma and silicone treatment on the surface morphology by calculated fractal dimensions. Wool fabric treated with plasma has higher fractal dimension than untreated sample. which corresponds with the results of SMD values measured by KES-FB4 surface tester as shown in Figure 9.

On silicone post-treatment, fractal dimensions of samples

are decreased, however, there is no significant difference between the samples treated with two types of epoxy-functional silicone.

### Conclusion

O<sub>2</sub> low temperature plasma pretreatment affected the surface properties of the wool fiber physically as well as chemically. By ESCA spectra of narrow, high-energy resolution scans of the S<sub>2p</sub> level, an appearance of the second peak at 168 eV indicated that cysteic acid groups were induced in wool fiber by O<sub>2</sub> plasma treatments.

Felting shrink resistance of wool fabric was improved by plasma and silicone treatment. The extent of enhancement in felting shrink resistance for plasma and silicone treated wool was polymer dependent with relatively hydrophilic polymer system giving rise to a more significant improvement. We believe this effect is due to a greater extent of spreading and adhesion of the more hydrophilic polymers on the wool modified by plasma treatment as a result of its increased hydrophilic character. Plasma treatment on wool fabric caused a significant increase in the frictional coefficients of the fabric, which resulted in the apparently harsher handle, decreased tear strength and increased grab break strength of the fabric. With the silicone polymer post application, however, the fabric hand values were improved and the tear strength was increased, and the grab break strength was slightly decreased. The surface smoothness appearances

measured by a new evaluation system showed good correspondence with the results of KES-FB4 surface tester.

### References

1. C. W. Kan, K. Chan, and C. W. M. Yuen, *Textile Res. J.*, **68**, 814 (1998).
2. A. Hesse, H. Thomas, and H. Hocker, *Textile Res. J.*, **65**, 355 (1995).
3. K. S. Gregorski and A. E. Pavlath, *Textile Res. J.*, **50**, 42 (1980).
4. S. Tokini and T. Wakida, *J. Soc. Dyers Colour.*, **109**, 334 (1993).
5. J. Ryu, T. Wakida, and T. Takagishi, *Textile Res. J.*, **61**, 595 (1991).
6. I. M. Muchairah, M. T. Pailthorpe, and S. K. David, *Textile Res. J.*, **67**, 69 (1997).
7. T. J. Kang and M. S. Kim, *Textile Res. J.*, **71**, 295 (2001).
8. C. M. Carr, S. F. Ho, D. W. Wewie, E. D. Owen, and M. W. Roberts, *J. Textile Inst.*, **56**, 419 (1985).
9. M. M. Millard, *Anal. Chem.*, **44**, 828 (1972).
10. J. Ryu, T. Wakida, and T. Takagishi, *Textile Res. J.*, **61**, 595 (1991).
11. A. Hess, H. Thoma, and H. Hocker, *Textile Res. J.*, **65**, 355 (1995).
12. W. Rakowski, *J. Soc. Dyers Colour.*, **113**, 250 (1997).
13. T. J. Kang, D. H. Cho, and S. M. Kim, *Textile Res. J.*, **71**, 446 (2001).