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A study on washing resistance of pp-HMDSO films deposited on wool fabrics for anti-pilling purposes

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ABSTRACT

In this work plasma polymerized coatings for anti-pilling purposes were deposited on knitted wool fabrics by means of a capacitively coupled RF discharge reactor using hexamethyldisiloxane as precursor. Their resistance to dry and wet cleaning was investigated and compared to that of a wet chemically deposited coating. Different gas mixtures and pre-treatment steps were tested to adjust the plasma process and to improve the film adhesion. An evaluation of the silica-like coatings behaviour to the washing stresses was performed by means of Fourier transform infrared and x-ray photoelectron spectroscopy. Anti-pilling performances of untreated, plasma-treated, and wet chemically treated wool fabrics were assessed.

1. Introduction

The textile industry is looking for applications which lead to products with additional properties in a cost-effective and eco-sustainable way. In this respect we have studied in a previous work [1], a method to solve an important issue in the textile field that is the fabrics tendency to pill. Abrasion from normal wear and cleaning causes the fibres to unravel and the loose ends to ball up on the fabric surface. This complex phenomenon is attributable to several factors comprising fibre, yarn and fabrics characteristics. To our knowledge no group of specific anti-pilling products exists and textile auxiliary producers mostly recommend chemical products that are primarily used for other purposes for anti-pilling finishes [2]. Moreover these products are usually applied on textile substrates in an aqueous bath or in foulard, using great amount of heated water and the production of polluting liquid effluents. In our study silicon containing thin films (Si:Ox:Cy:Hz) were deposited on knitted wool fabrics, by plasma-enhanced chemical vapor deposition (PECVD) in a low pressure plasma equipment, using hexamethyldisiloxane (HMDSO) as monomer and argon and oxygen as feed gases. The plasma treated samples compared with chemically treated and untreated samples, exhibited an improvement of about two grades as regards the standard pilling assessment. The results showed that this kind of treatments could represent an efficient technique to reduce pill formation on knitted wool fabrics and could also allow significant reductions of water, energy, and chemical consumption. Because of the poor resistance of these coatings to the washing stress, further investigations were required for an industrial application of the method. In general, due to the numerous ways a plasma interacts with a polymer surface, the gas type and plasma conditions must be adjusted to the polymer type in order to achieve a good adhesion of the deposited coatings. Glow discharges of non-polymerizing gases like argon or oxygen are usually used to prepare the substrate surface prior to the following deposition step. In particular argon is a commonly used inert gas for the pre-treatment of polymer surfaces and an Ar plasma can be applied to clean the surface before reactive gases are applied [3] to remove low molecular weight materials [3] and to improve the adhesive characteristics of polymers [4]. Oxygen and oxygen-containing plasma can at the same time etch the polymer surface and form oxygenated functional groups. The balance of these two processes depends on the operation parameters [3]. O_2 plasma pre-treatment can be carried out to roughen the surface resulting in an enhanced contact area at the beginning film growth [5]. Moreover, oxygen plays an important role in the deposition of the silicon-containing monomer in that the dilution of HMDSO with oxygen allows to increase the inorganic character of the coating [6].

The aim of this work was to study various plasma process operating conditions in order to guarantee durability of the anti-pilling coating on the wool surface to washing stress and dry cleaning. In particular, the number of plasma process steps and operational parameters including gas type were adjusted to improve adhesion of the thin film deposited. Morphologies of fibre surfaces were investigated using scanning electron microscopy (SEM). The pilling behaviour of untreated, plasma treated and wet chemically treated samples was assessed with modified

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Martindale abrasion testing method and washing resistance of plasma-deposited coatings and wet chemically-treated samples was evaluated by means of Fourier transform infrared (FT-IR) and x-ray photoelectron spectroscopy (XPS).

2. Materials and methods

2.1. Materials

The experiments were carried out on 100% knitted wool fabrics (292 g/m^2) and the dimensions of samples were 30 cm × 30 cm. The purities of both argon and oxygen were more than 99.99% (Siad S.p.A., Italy) and HMDSO [$(CH_3)_3$-Si-O-Si-(CH_3)] monomer was chemical reagent of grade 98% A.R.; (Sigma Aldrich, Germany).

Prior to the plasma treatments, the fabrics were previously scoured with petroleum ether (A.R. grade) for 3 h using Soxhlet extraction to remove sizing agents, followed by rinsing with deionized hot (T = 50 °C) and cool (room temperature) water for 1 h. Before and after the treatment, they were transported in a dry box to minimize humidity.

2.2. Plasma treatment

Plasma treatments were carried out in the lab scale low pressure plasma reactor of the Institute for Macromolecular Studies, National Research Council, Biella, Italy.

The discharge was powered by an RF (13.56 MHz) generator coupled to the capacitively-coupled electrodes by a fully tunable matching network. The sample was fixed to a roller below electrodes that was set at 4 rpm of working speed. This configuration allowed a homogeneous plasma deposition on all the substrate surface.

In the deposition step (Step 2) the pressure, the treatment time and the discharge power were kept constant at 2 Pa, 5 min, 40 W, respectively. The combination of different gas mixtures and number of steps used are shown in Table 1. The samples 1 and 2 were pre-treated with argon and oxygen plasma, respectively, while in the case of sample 3, the silicon-like film was directly deposited onto substrate. Ar and O_2 flux was fixed at 20 sccm while the HMDSO flux was 3 sccm.

2.3. Wet chemical anti-pilling treatment

Knitted wool fabrics were treated with an aqueous solution (pH adjusted to 5.5 with 2% o.w.f acetic acid) containing 2,5% o.w.f of aminomodified silicone emulsion (CT-80, Bilab, Italy) at 40 °C for 20 min. The material-to-liquor ratio was 1:50. The fabrics were dried at room temperature until constant weight. This treatment was used as a reference to evaluate the effectiveness and durability of plasma treatment in reducing pilling tendency.

2.4. SEM analysis

Morphological characterization of treated and untreated wool fabrics was carried out with a LEO 435VP scanning electron microscope from LEO Electron Microscopy Ltd.

Table 1
Plasma treatment operating conditions.

	Step 1	Step 2
Sample 1	**Ar 50 W 3 min 20 Pa**	**HMDSO/O_2/Ar**
Sample 2	**O_2 50 W 3 min 20 Pa**	**HMDSO/O_2/Ar**
Sample 3	**–**	**HMDSO/O_2/Ar**

2.5. Washing test

Washing resistance was investigated according to UNI EN ISO 26330. An Electrolux Wascator FOM71MP-Lab washing machine was used. Two 8A wash cycle at 30 ± 3 °C was used, with the total load of 2Kg for 15 min. The washing test was carried out on polymerized samples and wet chemically treated sample.

2.6. Dry cleaning

Dry cleaning was performed according to UNI EN ISO 105-X05 using an organic solvent (CCl_4) The test was carried out with a Linitest (URAI S.p.A) machinery at room temperature for 30 min. The dry cleaning test was carried out on polymerized samples and wet chemically treated sample.

2.7. Pilling behaviour

The determination of the propensity to pilling of knitted was assessed with modified Martindale method. Nu-Martindale Abrasion and Pilling Tester from James H. Heal & Co. Ltd was used. The samples were placed under standard laboratory conditions (65% R.H. and 20 °C) for 24 h before testing. The pilling tests were carried on untreated, plasma treated and wet chemical treated samples.

2.8. FT-IR analysis

Attenuated total reflectance FT-IR (ATR-FT-IR) spectroscopy measurements of untreated, plasma treated, wet chemical treated samples were carried out using an FT-IR Nexus 510 spectrometer (Thermo Nicolet) provided with a ATR accessory (Specac Ltd.) equipped with a Zn Se crystal with a 45° angle of incidence. Furthermore, FT-IR spectra of plasma treated and wet chemical treated samples were acquired after dry cleaning and washing test.

Data were collected from 400 cm^{-1} to 4000 cm^{-1} wavelength range with 100 scans and a spectral resolution of 4 cm^{-1}.

2.9. XPS analysis

The composition of elements of the deposited layer was studied by XPS using a monochromatic Al radiation at 1486.6 eV, VSW model TA10 and a hemispherical analyzer equipped with a single channel detector. XPS measurements were performed at a pressure of 1 × 10^{-6} Pa. The pass energy of the hemisphere analyzer was maintained at 187.8 eV for survey scan and 29.3 eV for high-resolution scan while the take off angle was fixed at 45°. Binding energies of XPS spectra were corrected by referencing the C_{1s} signal of adventitious hydrocarbon to 285 eV. XPS data fittings were carried out with PHI multipack™ software using the Gauss-Lorenz model and Shirley background.

3. Results and discussion

3.1. SEM analysis

Fig. 1 shows SEM micrographs of untreated and Sample 1–3 knitted wool fabrics. The thin inorganic coating of the treated samples was not visible and the morphology of the fibres appeared practically unchanged compared to the untreated wool sample. Samples 1 and 2, that were subjected to a preliminary plasma activation with Ar and O_2 gas respectively, showed an increased roughness due to the appearance of microcraters distributed all along the surface and to the re-deposition of etched material on the surface [7].