Cold Plasma Technology for Textile Products, Taking into Account their Specific Properties

Jo Verschuren

Abstract: **During this research a combination of plasma electrodes and sample holder was developed that enables the treatment of textiles as 3D porous structures. Experimental results show e.g. the influence of fabric structure on weight loss and of fibre processing chemicals on plasma induced hydrophylic properties. Spectroscopic methodology at STP is developed to assess the content and intensity of the plasma treatment. It is hoped that the research findings will be useful for both the vacuum and atmospheric plasma treatment of textile and other porous materials in industrial reality.**

I. Introduction

The research presented here works on three fields: (a) a critical literature study, (b) the development of a (vacuum) plasma reactor specifically designed for the study of the treatment of textiles in a plasma, and (c) the development of methods at STP for plasma treatment analysis. The PhD also considers practical consequences of using a plasma for treating textile product in industrial reality [1].

II. Critical Literature Study

Literature on plasma-for-textiles was critically scanned and following observations were made:

- treatment conditions are often not industrially feasible,
- typical textile properties are not considered, and
- essential treatment information is missing.

The experimental work developed during this PhD was initiated by these observations in literature and by the observation of plasma treatments in a reactor designed for CF_4 plasma cleaning of electronic components.

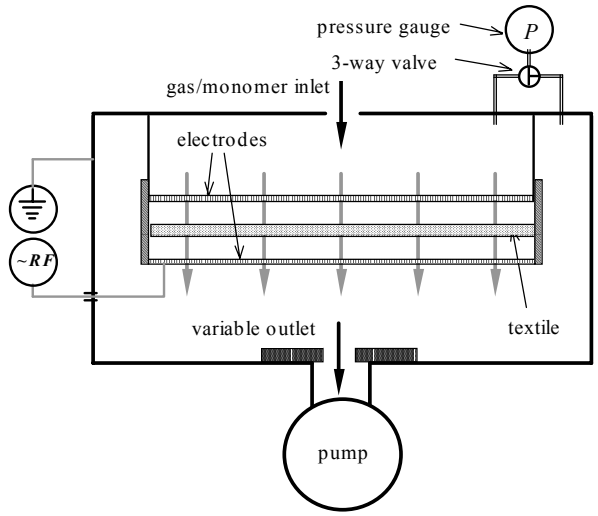

Fig. 1: Schematic view of developed reactor system

III. Reactor Design

The reactor design (Figure 1) considers the specific behaviour of a textile during all the steps of its plasma treatment. A sample (0.23 m square) is framed between a pair of electrodes. A homogeneous column of gas is introduced in the reactor at a linear velocity between 0 and 0.5 $m.s^{-1}$. During the functional treatment (5 - 600 s) the virtual average residence time of a species in the plasma (> 0,04 s) can be changed independently from the pressure (15 - 250 Pa) in the vacuum room. The gas is forced through the sample, enabling deep penetration into the textile of metastable plasma species. A 3-way valve enables the measurement of the pressure buildup across the sample. The pressure buildup - which depends on several parameters - can cause the plasma to become unstable.

Experiments show that also at reduced pressures (15 - 250 Pa) - with its high diffusion rates - the penetration of a gas into a textile is slowed down by the porous structure of the textile. As a consequence, a gas flow which is not forced through the textile is unable to homogeneously treat the complete fibre surface, as required in applications such as filtration. Penetration of the plasma treatment effect was demonstrated by weight loss depth profiling, in which a stack of 7 layers of a thin polyester textile structure was treated in a direct oxygen plasma (53 Pa, 47 $mW.cm^{-3}$). Results (Figure 2) show that plasma species, capable of chemical etching, can penetrate deeply into the textile structure.

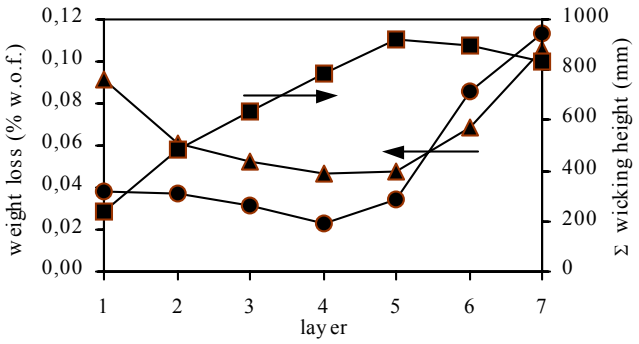

Fig. 2: Weight loss profile after a 5 minute O_2 plasma treatment (▲: open weave structure,●: nonwoven). Wicking test profile after a 20 second O_2 plasma treatment (■: nonwoven).

The profile is influenced by the thickness and structure of the textile; while a 0.9 mm thick stack of open weave fabrics results in near symmetrical treatment, a more closely structured 2.4 mm thick stack of nonwovens results in a higher etch effect at the side of the powered electrode. Similar effects were found after characterisation of the nonwoven structure by wicking tests, for short treatment durations (20 s).

The influence of fibre processing chemicals on plasma treatment results is illustrated by consecutive wicking tests of

J. Verschuren is with the Department of Textiles, Ghent University (UGent), Gent, Belgium. E-mail: jo.verschuren@UGent.be

a meta-aramid nonwoven treated in an oxygen plasma. The processing chemicals are plasma treated rather than the fibre polymer surface. Their easy removal in water at room temperature also removes most of the treatment effect. In figure 3 the wicking height, summed over a 10 minute period, is plotted against plasma treatment duration (O_2, 53 Pa, 47 $mW.cm^{-3}$). The treatment durations are limited with industrial productivity in mind (30-180 $m.min^{-1}$). The initial wicking properties are good, even for non-treated samples. A second wicking test - after drying - on the same samples shows a much reduced wicking. The dependence on treatment duration has increased. Mild washing of the sample after the second wicking test reduces the hydrophylic properties to negligible proportions. Washing of samples before plasma treatment requires longer treatment durations for a given effect, but the effect is more stable.

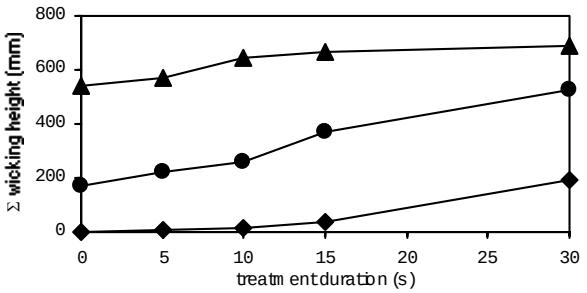

Fig. 3: Summed wicking height as function of treatment duration in an oxygen plasma. First (▲), second (●) and third (◆) wicking tests.

VI. SPECTROSCOPIC SAMPLE ANALYSIS

Apart from sample weight loss, and water wicking or absorption tests [2] an effort was made to develop spectroscopic methodolody at STP. This was done in view of their industrial applicability:

- they do not require the use of vacuum techniques, and
- the acquisition and interpretation of data from surface selective techniques is often problematic for textiles.

UV-VIS Diffuse Reflectance Spectroscopy (DRS) shows to have most potential in this area. Challenges are the small spectral changes (< 0,2 FR), as well as their interpretation.

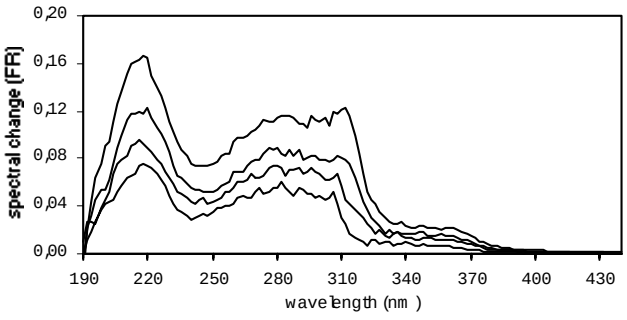

Fig. 4. UV diffuse reflectance difference spectra for plasma treated PET nonwoven (argon, 33 Pa, 28 $mW.cm^{-3}$, 2 (top), 5, 10 and 15 min. treatment duration).

The largest spectral change after a 15 minute argon plasma treatment is 0,15 FR (<5% change) (figure 4); results after an oxygen plasma are near-identical. The interpretation of the UV DRS data towards (physico)chemical reality necessitates comparison between results obtained via different techniques. XPS analysis shows - for polyester - a constant surface chemical constitution from 2 minutes plasma treatment onwards. This contradicts the spectral changes in figure 4, which increase with durations up to 15 minutes. A possible explanation is that the vacuum UV-light penetrates the fibres and modifies the polymer backbone in a way yet unexplained.

Principal Components Analysis (PCA) is used to extract absorption maxima from a set of overlapping peaks. The data are split into a sets of scores and loadings, whose recombination results in the original data. The score plots for PC1 and PC2 are given in figure 5, showing a linear behaviour with plasma treatment duration.

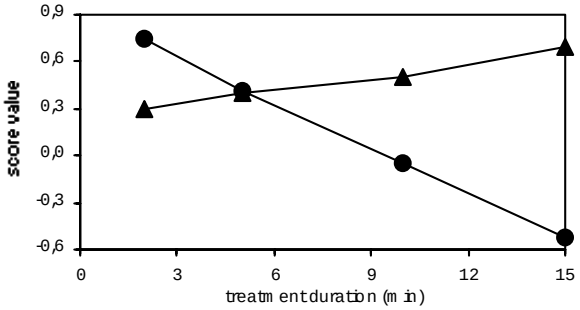

Fig. 5. Score plots of PC1 (▲) and PC2 (●).

The loadings plots for PC1 and PC2 are given in figure 6, showing a peak at 314 nm, also visible in polyester foil treated in an atmospheric plasma[3].

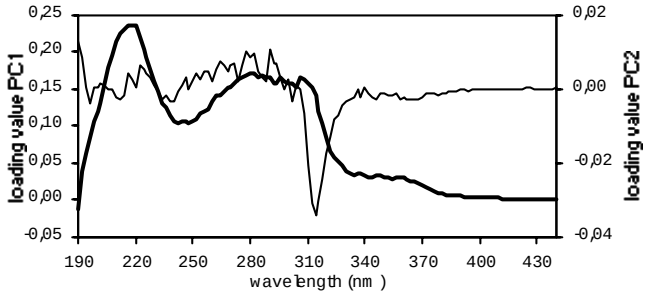

Fig. 6. Loadings plot for PC1 (thick) and PC2 (thin).

Correlation matrices as well as spectral simulation[4] assist the interpretation of spectral changes and more in particular their correlation at different wavelengths.

ACKNOWLEDGEMENTS

The author greatly acknowledges the support from Bijzonder Onderzoeksfonds (UGent).

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