

# Penetration of plasma effects into textile structures

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## Abstract

The penetration of plasma into textile materials turns out to be a crucial point for plasma modification of fabrics. Throughout the process where plasma treatment can be applied approximately 1–100 mbar is evaluated to be the optimum. This is due to the correlation between characteristic geometrical distances in fabrics and the mean free path of modifying particles in the gas phase as well as the energy transfer from activated plasma particles to surrounding inactive gas particles and to surface sites of textile fibres. Theoretical calculations and experimental results for the hydrophilisation of fabrics prove this behaviour. © 2001 Elsevier Science B.V. All rights reserved.

*Keywords:* Plasma modification; Textile fabrics; Hydrophilisation

## 1. Introduction

The pretreatment and finishing of textile fabrics by plasma technologies is increasingly replacing wet chemical applications. Plasma treatment modifies the uppermost atomic layers of a material surface and leaves the bulk characteristics unaffected. The possible aims of this are the affection of wettability, adhesion, or reflectivity, etc. (e.g. [1–10]).

In fabrics there is a difference between the visible surface and actual inner surface to be modified in contrast to foils or plates. A fabric consists of a complex structure of single fibres and threads that are distributed over the fabric thickness. The entire thickness of fabrics may range up to several millimetres. To ensure the plasma has an effect on the surface of all single fibres within the entire fabric the modifying particles must move through the textile structure in an acceptable time, keeping their modifying ability.

This paper investigates this non-trivial problem in the case of hydrophilisation of cotton fabrics in an oxygen plasma taking the gas pressure as the most important process parameter.

## 2. Theoretical background

Plasmachemical conversion of the feed gas produces chemically active particles (e.g. O radicals) that are able to modify textile surface molecules via chemical reactions after impinging on the surface. The radicals generated inside the plasma region must be given the opportunity to move to the reaction place at the textile fibre surface. Thereby the path of radicals between the locations of generation and reaction is limited on the one hand by the distance between single fibres, and on the other hand by the gas density, i.e. by the mean distance between gas particles. Assuming radicals react or recombine after several impacts with gas particles and at surface sites on fibres there is a relationship between penetration depth of the plasma effect inside the textile structure and process pressure as well as the textile structure itself. Reaction between active plasma particles and the surface of textile fibres deactivates the reaction site and enables further penetration of new active particles even if they hit the deactivated surface site again.

Considering the activation state of interacting partners three body collisions must be taken into account that result in non-activated particles as the final state of interaction. A small accommodation coefficient of

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energy, i.e. small probability of energy transfer in case of collision, leads to an increase of the mean free path between reacting collisions. As a result of chemical reaction between particles from gas phase and surface sites, and therefore the deactivation of modified surface sites, active particles penetrate more and more into the textile structure.

To estimate the mean free path of gas particles typical distances in textile structures have to be calculated. A cotton fabric consists of single fibres that form threads (warp and weft) in a weave. For example the mean distance of single fibres in a thread of a light cotton woven reaches approximately  $10\ \mu\text{m}$ . The mean distance between threads amounts to approximately  $100\ \mu\text{m}$ . Expanding the subject to very tight and very loose woven materials, typical distances of single fibres range from  $1$  to  $10\ \mu\text{m}$  and of threads from  $0.1$  to  $1\ \text{mm}$  (Fig. 1).

A comparison of these typical distances in fabrics with the mean free path of particles in the gas phase is necessary. For an estimation of the mean free path  $\Lambda$  of a radical in the gas phase it is assumed that the mean free path is comparable to that of a neutral gas at room temperature. Fig. 2 shows the mean free path of air, oxygen and hydrogen vs. pressure [11].

In the classical low pressure region ( $p < 1\ \text{mbar}$ ) the mean free path in the gas phase exceeds the typical distances in textile material. The radical impacts are only with the textile surface and the losses due to gas phase collisions are reduced to a minimum so that the modification is done very efficiently. On the other hand, the very low pressure causes a relatively low radical concentration per volume unit.

In the pressure range of  $p > 100\ \text{mbar}$  (especially at atmospheric pressure) the mean free path in the gas phase is much lower than textile distances. Most of the collisions are collisions with other gas particles reducing the lifetime of the radicals in a way, that they do not reach the reaction sites inside the voluminous fabric.

From these estimations a process pressure between  $1$

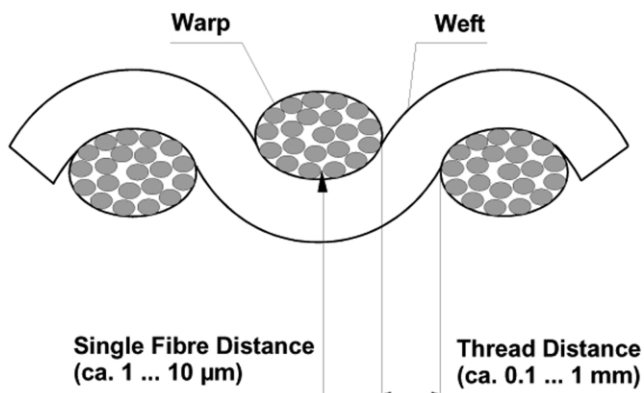


Fig. 1. Typical geometrical distances in a textile fabric.

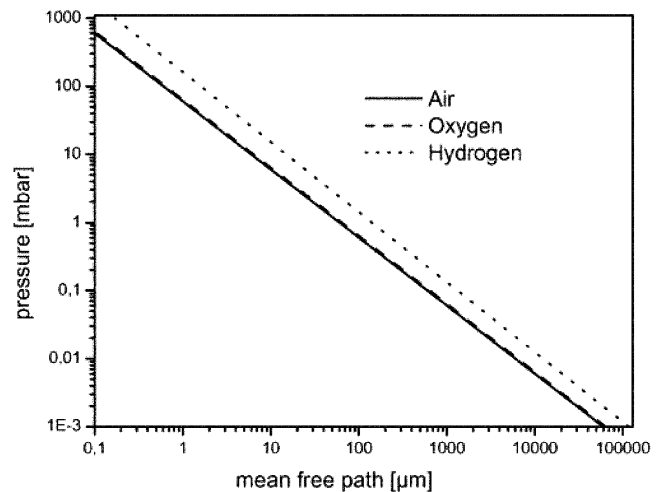


Fig. 2. Mean free path as a function of pressure for several gases.

and  $100\ \text{mbar}$  turns out to be an optimum value for the plasma modification of voluminous textile fabrics throughout the entire fabric thickness.

### 3. Experimental and results

#### 3.1. Low pressure

The plasma treatment experiments at low and medium pressure were carried out at a woven cotton fabric ( $115\ \text{g}/\text{m}^2$ ) to hydrophilise the textile material. For the study of the penetration depth of the plasma effect several fabric layers were put together tightly in order to simulate a total fabric thickness of approximately  $1\ \text{mm}$ . The hydrophilisation effect was measured by a suction test. A capillary is filled with a test liquid (coloured water) and positioned onto the according surface of the fabric layer to be checked. The liquid is adsorbed by the fabric to form a coloured circle. The diameter of the circle (hydrophilisation diameter) that

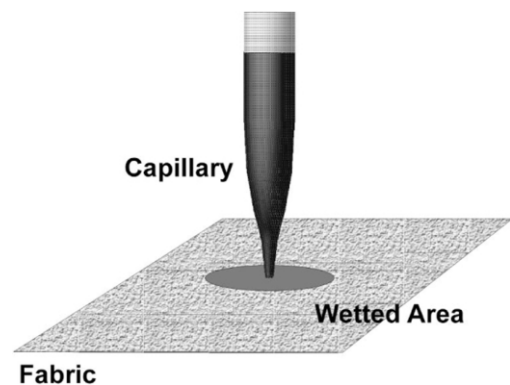


Fig. 3. Measurement method for hydrophilisation effect. A capillary is set onto the fabric surface. Depending on hydrophilic characteristics the textile material sucks a test liquid and a wetted area is formed that can be measured.

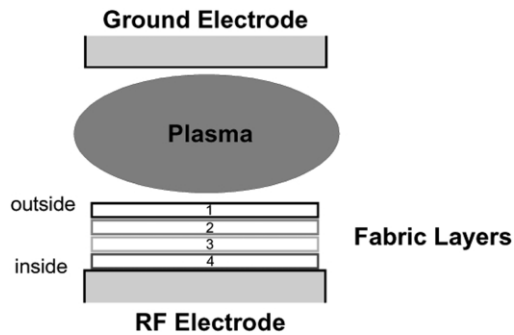


Fig. 4. Electrode arrangement and fabric layers for low and medium pressure plasma treatment.

is formed after an exposure time of 20 s is a measure of the hydrophilisation effect (Fig. 3).

The fabric layers were positioned onto the RF electrode of an asymmetric diode like system in order to have the samples right in front of the glow light as the main source of radicals (Fig. 4).

Low pressure plasma treatment was carried out at the following process parameters:

- electrode distance  $d = 4$  cm;
- frequency  $f = 20$  kHz;
- feed gas  $Q = 50$  sccm  $O_2$ ;
- vessel volume  $V = 40$  l;
- power density  $P/A = 0.64$  W/cm<sup>2</sup>;
- treatment time  $t = 50$ –800 s; and
- pressure  $p = 0.6$ –8 mbar.

### 3.2. Time dependency

With increasing plasma treatment time the hydrophilisation effect penetrates deeper into the fabric. An example is given in Fig. 5 for a process pressure of 0.6 mbar. After a certain time (approx. 700 s) the hydrophilisation is complete, even for the most inner fabric layer. Hydrophilisation was measured on both front and back sides of each fabric layer. Obviously, the plasma effect overcomes the distance within the fabric layer faster than the distance between two layers. This is due to an insufficient contact of the layers each to the other and it demonstrates the dependency of penetration on particle path in the gas phase and the textile structure.

It can be assumed that the radicals hitting the surface of the fibres react with cotton molecules hydrophilising the material. After hydrophilisation is completed in the upper regions the radicals are reflected from these surfaces keeping their hydrophilisation ability and they can move to deeper zones in the fabric pushing the hydrophilisation front forward.

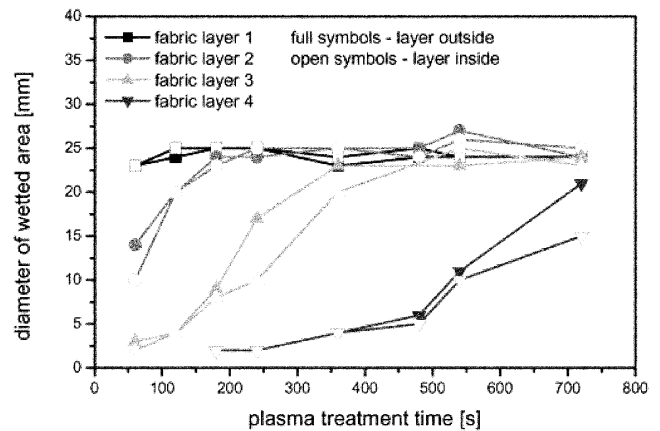


Fig. 5. Time dependency of the penetration of hydrophilising plasma effect expressed by the diameter of wetted area (layer numbers as indicated in Fig. 4).

### 3.3. Pressure dependency

Pressure increase results in a remarkable increase of depth and velocity of the penetrating hydrophilisation front. At a given treatment time of  $t = 180$  s a continuous improvement of hydrophilicity can be shown within the studied pressure range of up to 8 mbar (Fig. 6). This might be due to an increasing concentration of oxygen radicals per volume unit. Obviously, the pressure is still low enough to ensure that the mean free path between gas particles lies above the typical distances between the fibres in the fabric so that gas phase recombination does not play the main role.

Although theoretical estimations in Section 1 demand further increases of process pressure to approximately 100 mbar, it should be noted that the pressure in the experiments is limited to 8 mbar because of the applied electrode system. For pressure ranges above this value (rough vacuum range) a special electrode

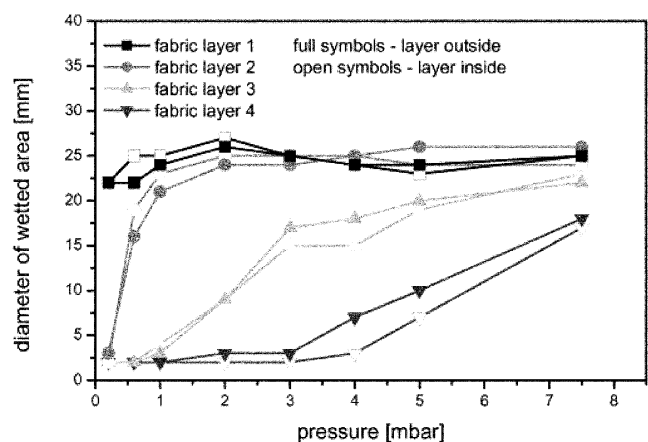


Fig. 6. Process pressure dependency of the penetration of hydrophilising plasma effect expressed by the diameter of wetted area (layer numbers as indicated in Fig. 4).

design must be developed to ensure a complete plasma coverage of the electrodes. Additionally, emphasis must be given to efficient cooling systems of electrodes. Sophisticated gas feeding is needed as well. With increasing pressure the gas transport mechanism turns from diffusion to convection or at least a mixture of both. Due to the small number of applications in the rough vacuum range there is a deficiency of according solutions. Only in recent months the first attempts have been undertaken [e.g. [12]].

### 3.4. Atmospheric pressure

Plasma treatment at normal pressure is involved in the studies as the upper limit of the pressure scale because of its technical advantages, especially when applying atmospheric air. Commonly Corona or other spark discharges are applied. The spark trace leads through the textile material along the space between fibres or more likely threads.

Our atmospheric pressure experiments were carried out applying a special type of a spark discharge (so-called '3D Plasmatreater' of Ahlbrandt). The discharge is blown outside of a nozzle by an air flow reaching the fabric surface. The fabric layers are positioned in the same way as for the low and medium pressure experiments. The plasma jet is directed perpendicularly onto the fabric layers in a way that the outer layer is in complete contact with the plasma (Fig. 7).

Besides increasing temperature and strong decomposition of the fabric (etching) a hydrophilisation effect on the outer plasma facing fabric layer is observed. Despite increasing treatment time and other supporting steps (e.g. increasing gas flow, forced penetration by a pressure difference across fabric layers) a penetration of the effect to the backside of the upper layer or to layers inside the stock does not occur (Fig. 8).

## 4. Conclusions

Plasma modification of voluminous fabrics over the

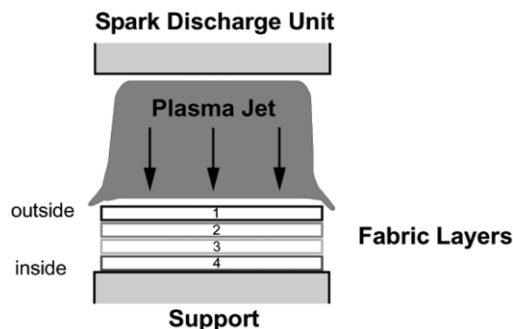


Fig. 7. Arrangement for plasma treatment at atmospheric pressure.

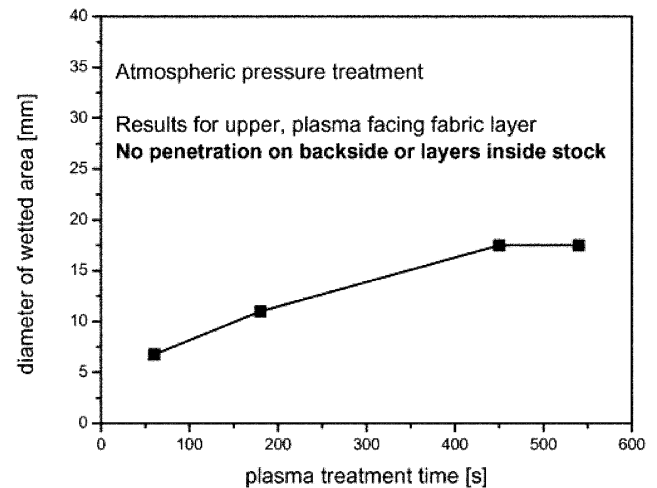


Fig. 8. Hydrophilisation effect of upper fabric layer (layer no. 1 in Fig. 7) at plasma facing side expressed by the diameter of wetted area (hydrophilic test).

entire thickness has its own specifics. Due to fabric structure and collision characteristics the pressure turns out to be a crucial process parameter for optimal treatment. The applied pressure value must be matched to the characteristic structure of the textile material to be plasma treated. The pressure range from approximately 1 to 100 mbar is applicable to most of the existing textile structures and leads to an optimal plasma effect throughout the complete textile structure.

Besides etching and burning of the fabric at normal pressure, no penetration of the plasma effect into the textile structure is observed. This behaviour is traced back to the pressure far exceeding the optimum value and thus favouring deactivating collisions in the gas phase.

A continuous plasma treatment of voluminous fabric lengths at such pressure values demands new concepts and electrode design for application. Electrodes should have reduced surfaces and be temperature-controlled. Additionally, a special gas feeding system leading the fresh gas into the reaction zone might be an advantage. The first suggestions have already been made [12].

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## References

- [1] H.U. Poll, S. Schreiter, *Melliand* 6 (1998) 466.
- [2] B. Bechter, S. Berndt, R. Greger, W. Oppermann, *Techn. Textilien* 42 (1) (1999) 14.
- [3] T. Okuno, T. Yasuda, H. Yasuda, *Text. Res. J.* 62 (8) (1992) 474.

- [4] S. Ruppert, T. Bahners, E. Schollmeyer, *Text. Praxis Int.* 49 (1994) 614.
- [5] T.L. Warol, R.R. Benerito, *Text. Res. J.* 52 (1982) 256.
- [6] T. Wakida, S. Tokino, *Indian J. Fibre Text. Res.* 21 (1,5) (1996) 69.
- [7] A.I. Maksimov, B.L. Gorberg, W.A. Titov, *Textilnaja Chimija* 2 (1993) 101.
- [8] W.G. Pitt et al., *J. Appl. Polym. Sci.* 48 (1993) 845.
- [9] L.S. Penn, T.J. Byerley, T.K. Liao, *J. Adhesion* 23 (1987) 163.
- [10] B.Z. Jang, *Compos. Sci. Technol.* 44 (1992) 333.
- [11] W. Jitschin, *Vakuum-Lexikon*, Wiley VCH (1999) 17-2.
- [12] Deutsche Patentanmeldung 199 60 401.0-52.