

# INVESTIGATION OF THIN FILMS DEPOSITION INTO POROUS MATERIAL

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Although the direct contact of the treated material with the plasma is assumed by the plasma community as a necessary condition of successful plasma treatment, several references mention penetration of active species into the porous material. Hydrophylity enhancement has been observed even inside porous material. The aim of this study is experimental investigation of plasma. This work is aimed to experimental investigation of thin layers deposition on porous substrates.

The porous substrate was simulated with a specimen made from two glass wafers, on the margins of which two difference strips of varying thickness were placed. These strips define the thickness of the slot in the middle. After the deposition the substrate was decomposed and the film deposited inner walls of the glass wafers was investigated. Layers were deposited by method PECVD used RF plasma from gas  $C_2H_2$ . The film thickness was measured in dependence on the distance from the margin into the centre of the slab by optical profilometer. Penetration dept was tested in dependence on deposition conditions and geometric configuration of the substrate. Depending on deposition conditions, the film deposition was observed even on the whole substrate.

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## 1. INTRODUCTION

By deposition on porous material overall coverage is an important factor, i.e. penetration of active particles into single pores of the substrate.  $SiO_2$  and  $TiO_2$  layers were deposited on porous material (textile fabric) by method sol-gel. Resulting layer was only on surface of the fabric and material inside of binding points of fabric remained untreated [1]. This undesirable effect could be avoided by application of PECVD method. Owing to active particles motion in plasma discharge is achieved more effective penetration into porous substrates. This work is focused on investigation of above mentioned methods and processes of plasma treatment.

Penetration of plasma effects is subject of research at many institutions. Many of the works are focused on penetration of porous material plasma modification. Measurement of penetration is carried out by many various methods. Testing of penetration at powder materials is based on wettability change of modified material. Wettability of separate layers of modified material is measured [2-4]. Testing of plasma effects penetration into textile structures also uses wettability-based method [5]. Other approach involves chemistry detection of separate layers of treated fabric by ESCA method [6].

## 2. EXPERIMENTAL

The low-pressure PECVD device was RF planar reactor schematically shown in Fig. 1. The reactor consists of a high-voltage electrode and a vertically adjustable substrate holder. Distance between substrate and RF electrode are 60 mm. The volume of the vessel was about 125 l. The powered electrode was capacitive coupled to the RF generator via a matching unit. The applied power was up to 15 W with the frequency 13,56 MHz. The vessel was evacuated by pumping

system consisted of rotary oil pump and Root's pump. Ultimate pressure was below 1 Pa. Pressure during deposition was 2, 5, 8, 10 Pa. The pumping system was protected by a cold trap cooled by liquid nitrogen. The butterfly valve was used to reduce the pumping speed and control the pressure.  $C_2H_2$  was used as the working gas. The gas was introduced into the reactor through the tube positioned on the side of the reactor. The flow of the working gas was controlled by mass-flow meter. Mass flow of the working gas  $C_2H_2$  was 10 sccm.

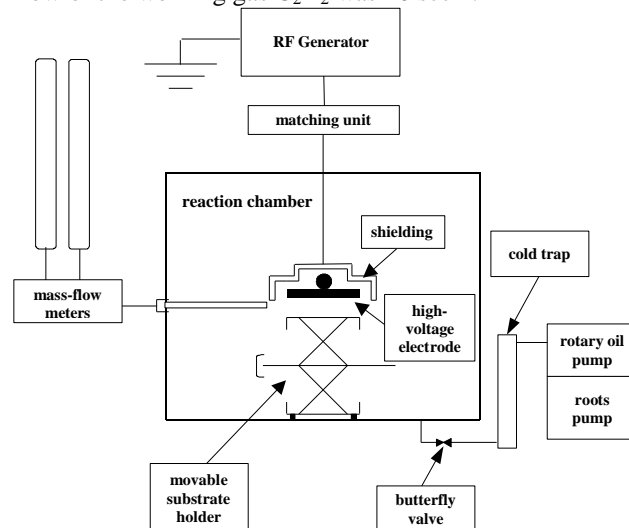


Fig. 1. Schematic diagram of the vacuum PECVD deposition device

The porous substrate was simulated with a specimen made from two glass wafers, on the margins of which two difference strips of varying thickness were placed. These strips define the thickness of the slot in the middle. Thickness of difference strips 0,8; 1,6; 6; 10 mm was used. Length of slot was 80 mm and width 8 mm. Fig. 3 shows the model substrate.

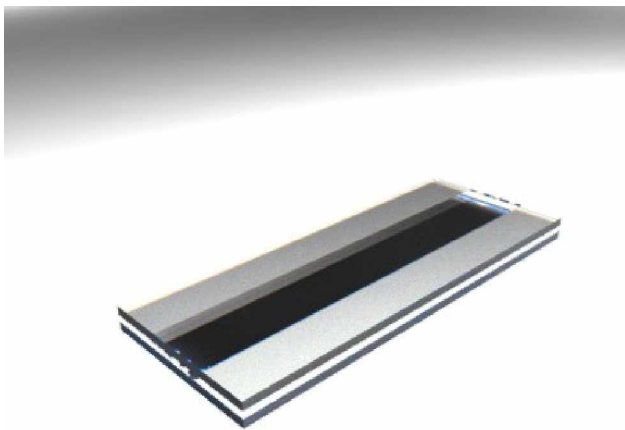


Fig. 2. Model substrate with defined slot

After the deposition the substrate was decomposed and the film deposited inner walls of the glass wafers was investigated. The resulting film thickness depending on a distance from the slot opening was measured by means of the optical profilometer. In addition to the model substrate, reference sample without slot was deposited. This reference sample was used for confrontation of deposition process with and without slot.

### 3. RESULTS AND DISCUSSION

This paper is focused on testing of penetration depth into defined slot. This slot was model for porous substrate. Penetration depth was tested in dependence on deposition conditions and geometric configuration of the substrate. Thin film was ascertained in the whole length of substrate.

Layers thickness at the opening of the slot was measured in tenths micrometers. Towards the centre of the substrate the layer thinned beneath measurable values.

The thickness was measured in defined intervals from opening of the slot by optical profilometer. For greater clarity and comparability is a relative thickness indicated. The relative thickness determines ratio of layer thickness measured in the slot and respective layer on plain sample without the slot.

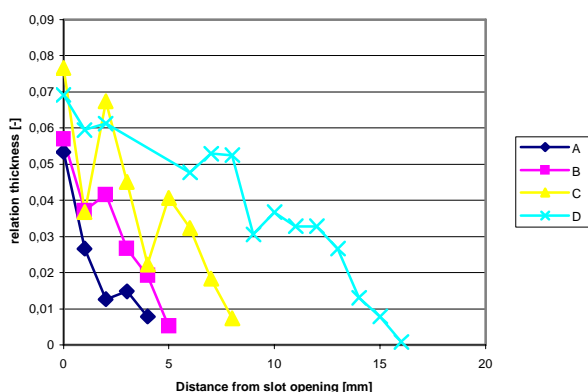


Fig. 3. Dependence of relative layer thickness on distance from the opening of the slot for different size of slot. Deposition pressure was 10 Pa

Fig 3 shows dependence of relative layer thickness on distance from the opening of the slot. Dependence on

geometric configuration of the substrate is shown also on the fig 3. Results for substrate with 0,8 mm slot high describes line A. Line E is for 10 mm slot high. According to presumption was highest penetration ascertained on samples with bigger slot.

Fig. 4 depicts influence of pressure during deposition process on active particle penetration. Highest penetration was ascertained on samples deposited at pressure 10 Pa. The layer was still measurable 14 mm from the slot opening. The lowest penetration showed the sample deposited at pressure 2 Pa. This sample's layer was measurable only 6 mm from the slot opening.

This result supports presumption based on mean free path of active particles. This mean free path lowers with increasing pressure. Higher pressure causes higher number of active particles collisions. Due of this effect, some active particles can be bounced perpendicular in the direction of the field activity between the electrode and the substrate.

These active particles could form the layer in the limited space of the slot.

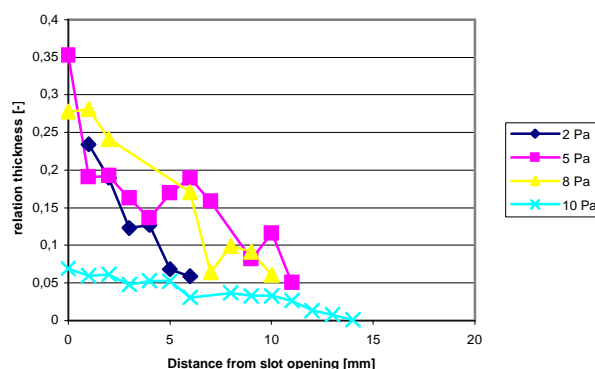


Fig.4. Dependence of relative layer thickness on distance from the opening of the slot for different deposition pressures. High of slot was 10 Pa

### CONCLUSIONS

Most suitable deposition conditions in relation to penetration of active particles were tested. Complex form substrate was simulated by glass substrate with the slot. The deposition pressure is a distinctive factor influencing the penetration into the limited space of the slot. Highest penetration was ascertained on samples deposited at pressure 10 Pa. The layer was still measurable 14 mm from the slot opening. The lowest penetration showed the sample deposited at pressure 2 Pa.

Mass of work gas and distance of substrate from electrode could be next factors, which influence the penetration. These parameters will be further tested.

### ACKNOWLEDGEMENT

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## **ИССЛЕДОВАНИЕ ОСАЖДЕНИЯ ТОНКИХ ПЛЕНОК ВНУТРИ ПОРИСТЫХ МАТЕРИАЛОВ**

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Хотя в плазменном сообществе существует мнение о том, что необходимым условием плазменной обработки является непосредственный контакт плазмы с обрабатываемым материалом, имеются сообщения о проникновении активных компонентов в пористые материалы. Усиление гидрофильности наблюдалось даже внутри пористого материала. Цель настоящего сообщения – экспериментальное изучение процесса осаждения тонких слоев на пористые подложки.

Пористая подложка моделировалась образцом из двух стеклянных пластинок, по краям которых помещались две полоски переменной толщины. Эти полоски определяли толщину зазора в центре. После осаждения подложка разбиралась и исследовалась пленка, осажденная на внутренних стенках стеклянных полосок. Осаждение осуществлялось методом PECVD с использованием ВЧ плазмы в C<sub>2</sub>H<sub>2</sub>. Толщина пленки измерялась профилометром при различных расстояниях от края к центру пластины. Находилась глубина проникновения в зависимости от условий осаждения и геометрической конфигурации подложки. При некоторых условиях осаждение пленки имело место даже на всей площади подложки.

## **ДОСЛІДЖЕННЯ ОСАДЖЕННЯ ТОНКИХ ПЛІВОК УСЕРЕДИНІ ПОРИСТИХ МАТЕРІАЛІВ**

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Хоча в плазмовому співтоваристві існує думка про те, що необхідною умовою плазмової обробки є безпосередній контакт плазми з оброблюваним матеріалом, маються повідомлення про проникнення активних компонентів у пористі матеріали. Посилення гідрофільності спостерігалось навіть усередині пористого матеріалу. Ціль даного повідомлення – експериментальне вивчення процесу осадження тонких шарів на пористі підкладки.

Пориста підкладка моделювалася зразком із двох скляних пластинок, по краях яких містилися дві смужки змінної товщини. Ці смужки визначали товщину зазору в центрі. Після осадження підкладка розбиралася і досліджувалася плівка, осаждена на внутрішніх стінках скляних смужок. Осадження здійснювалося методом PECVD з використанням ВЧ плазми в C<sub>2</sub>H<sub>2</sub>. Товщина плівки вимірялася профілометром при різних відстанях від краю до центра пластины. Знаходилася глибина проникнення в залежності від умов осадження і геометричної конфігурації підкладки. При деяких умовах осадження плівки мало місце навіть на всій площі підкладки.