STSM scientific report

Title project: Activation and functionalisation of nonwoven polypropylene by atmospheric pressure plasma
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COST STSM Reference Number: **COST-STSM-CM0601-05646**
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- **Purpose of the visit**

The scientific mission was focused on the detection of changes on the polypropylene surface after plasma activation on atmospheric pressure and functionalisation with different chemicals (silver nitrate solution, solution of gold nanoparticles and solution of AEM 5700).

- **Description of the work carried out during the visit**

The first aim of the visit was analyzing the surface of polypropylene (PP) nonwoven textile (50 g/m^2, Pegas Nonwovens, s.r.o.) untreated and treated with different plasma resources and by different chemicals to obtain changes on the surface of polypropylene.

For plasma activation were used two different types of plasma devices, diffuse coplanar surface discharge (DCSBD) and volume dielectric barrier discharge (DBD), Figure 1. Both discharges were operated at atmospheric pressure and room temperature. Idea was to see any difference between surfaces of nonwoven PP after different plasma treatment.

After plasma treatment samples were chemically treated by different solutions; silver-nitrate solution (0.01 mmol/dm^3), solution of gold nanoparticles (0.0625 mmol/dm3) and with solution of AEM 5700 (1%).

The surface morphology of prepared samples was investigated by scanning electron microscopy (SEM) using JEOL JSM 6460LV instrument and with atomic force microscopy (AFM) using tapping mode AFM Dimension V produced by Veeco.

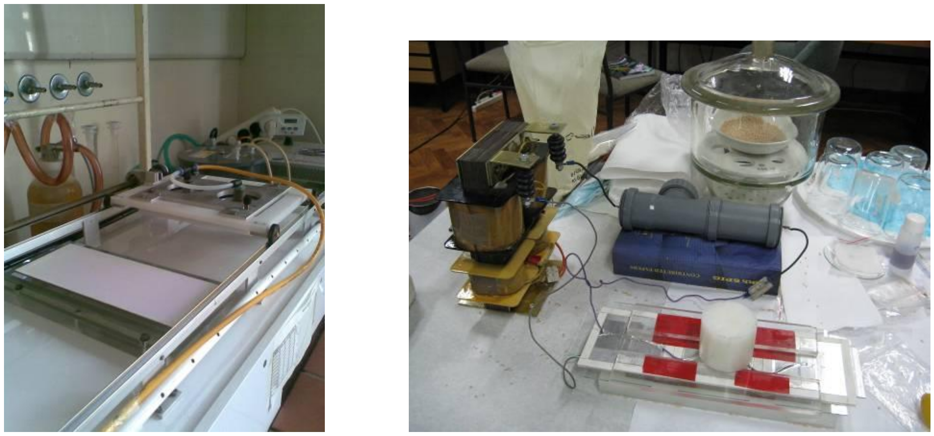

Figure 1 a) diffuse coplanar surface barrier discharge (DCSBD)
b) volume dielectric barrier discharge (DBD)

- **<u>Description of the main results obtained</u>**

As it shown in Fig. 2. and 3. the surface of PP is not clean and smooth, furthermore it is difficult to detect the potentially bonded particles.

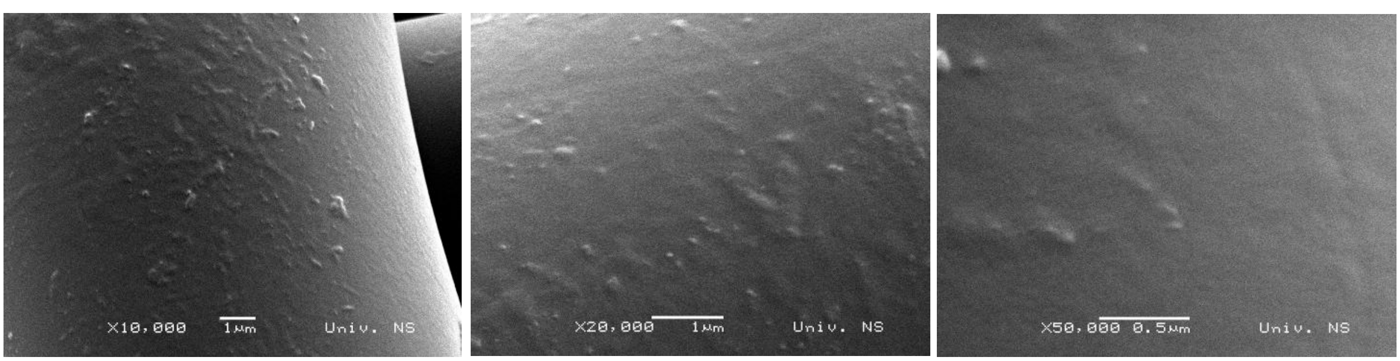

Figure 2. SEM images of nonwoven polypropylene

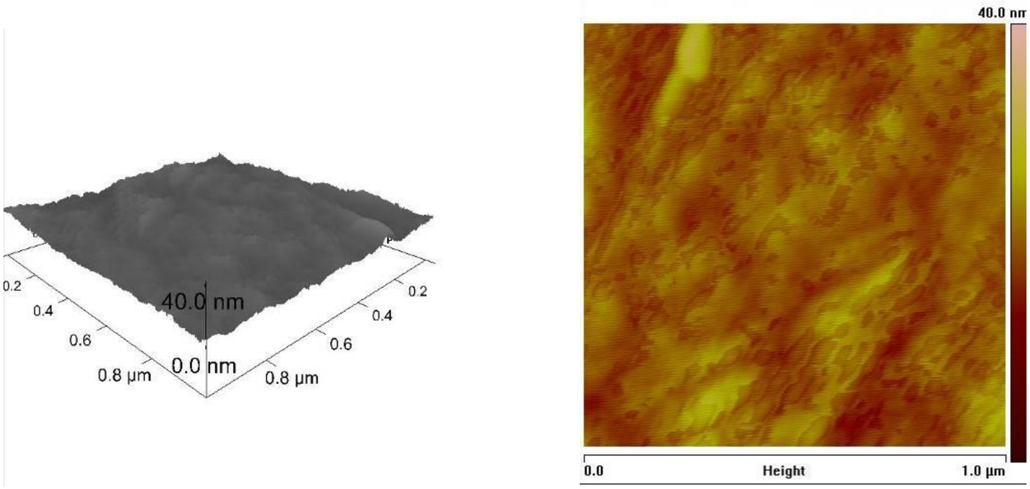

Figure 3. 3D and 2D AFM images of polypropylene

Silver

Samples of untreated polypropylene and plasma treated nonwoven PP by DCSBD and by volume DBD were immersed a 0.01 mmol/dm^3 solution of $AgNO_3$ for 4 hours and dried 24h at air.
PP surface is not clean and it is hard to detect silver after treatment with solution, even using SEM surface analysis was not enough to confirm silver on surface of PP because silver was in ionic station. SEM analysis confirmed a presence of particles on the surface (Fig. 4.a), however, back scattering SEM imaging (Fig.4.b) confirmed that particles are not PP.

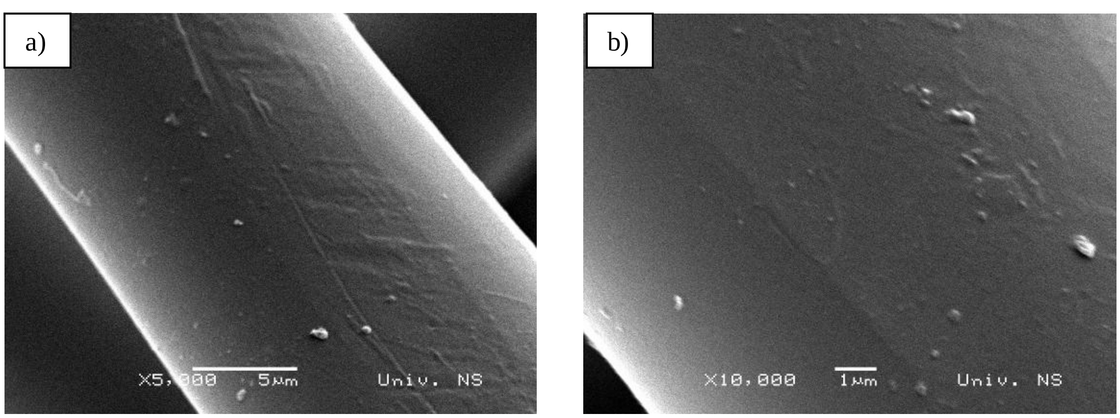

Figure 4. SEM images of treated polypropylene by silver-nitrate
a) SEM image
b) Back scattering image

On the surface of PP treated by volume DBD for 120 s and by DCSBD for 6s we can see similar particles like on untreated PP (Fig.5). Back scattering images (Fig. 5.b, d) also confirmed that particles are not PP. Also it is possible to notice changes in the roughness of the surface of PP caused by plasma treatment by DCSBD.

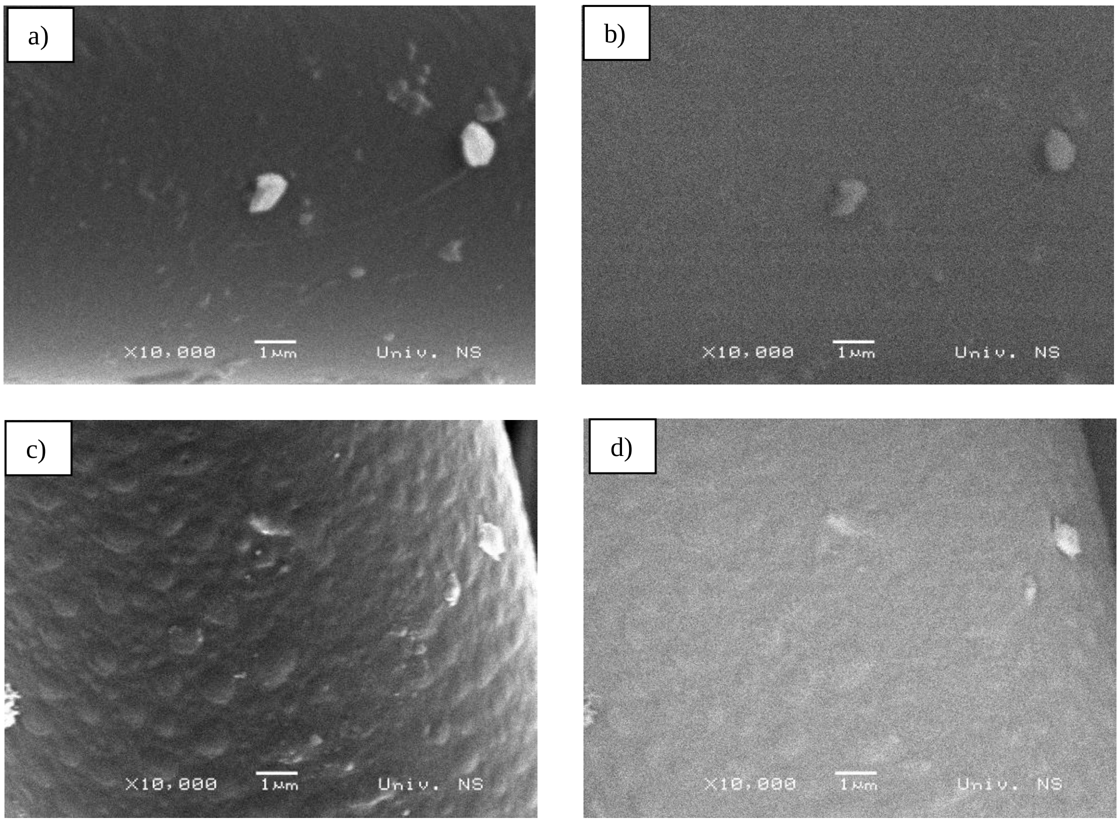

Figure 5. SEM images of plasma treated polypropylene after absorption of silver nitrate

a) SEM image of PP treated by volume DBD
b) Back scattering image of PP treated by DBD
c) SEM image of PP treated by DCSB
d) Back scattering image of PP treated by DCSBD

AFM images of polypropylene (Fig.6) confirm changes of the roughness of the surface of nonwoven polypropylene.

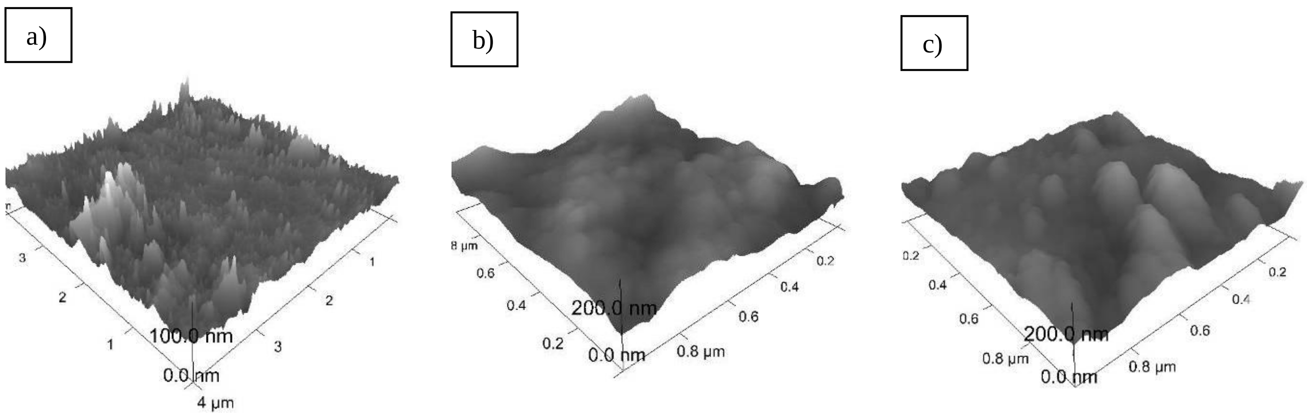

Figure 6. 3D AFM images of PP treated by plasma and silver-nitrate
a) Untreated PP
b) PP treated by DBD
c) PP treated by DCSBD

Nanogold

Samples of untreated polypropylene and plasma treated nonwoven PP by DCSBD and by volume DBD were immersed 10 minutes in the 0.0625 $mmol/dm^3$ solution of gold nanoparticles and dried at air for 24 hours.
According to UV - Vis spectra nanoparticles in solution of gold (Fig. 7) are spherical, isolated and small (up to 20 nm). Prepared solution was clean red color.

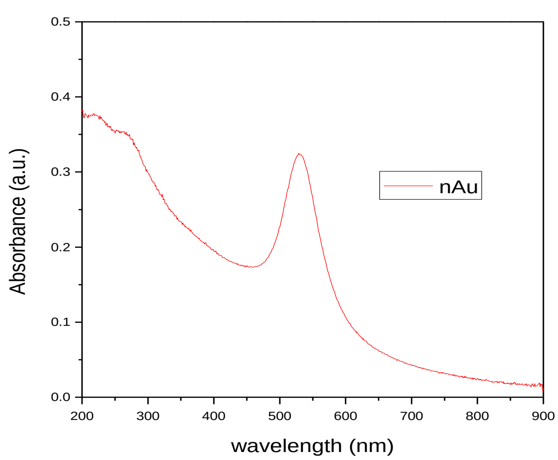

Figure 7. UV-Vis spectra of solution of gold nanoparticles

SEM (Fig.8.-10.) and AFM (Fig. 11) analysis confirmed a presence of nanoparticles on the surface of PP, and it was confirmed by back scattering SEM imaging (Fig.10.b, d)) that particles are not PP.
The smallest size of nanoparticles which was possible to measure by used SEM device was around 30nm (Fig. 10). After plasma treatment gold nanoparticles are more evenly distributed on the surface of PP (Fig 11.a, c).

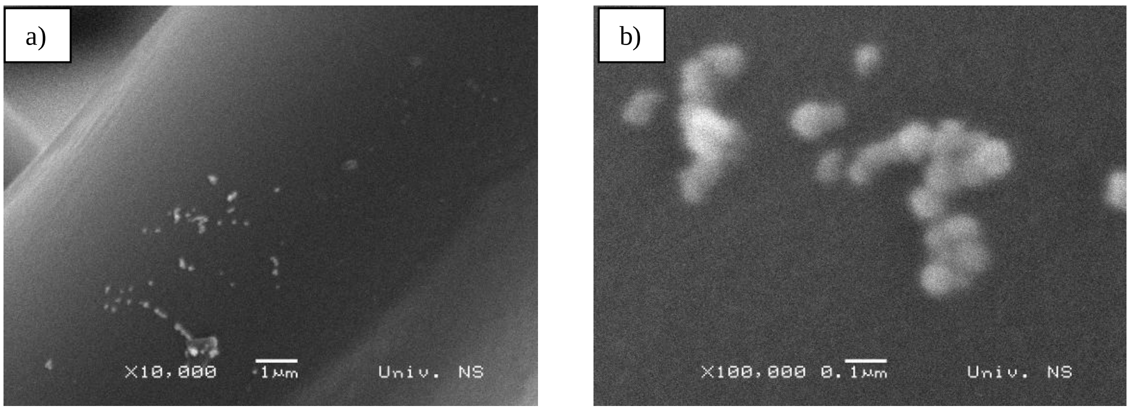

Figure 8. SEM images of nonwoven polypropylene after absorption of gold nanoparticles

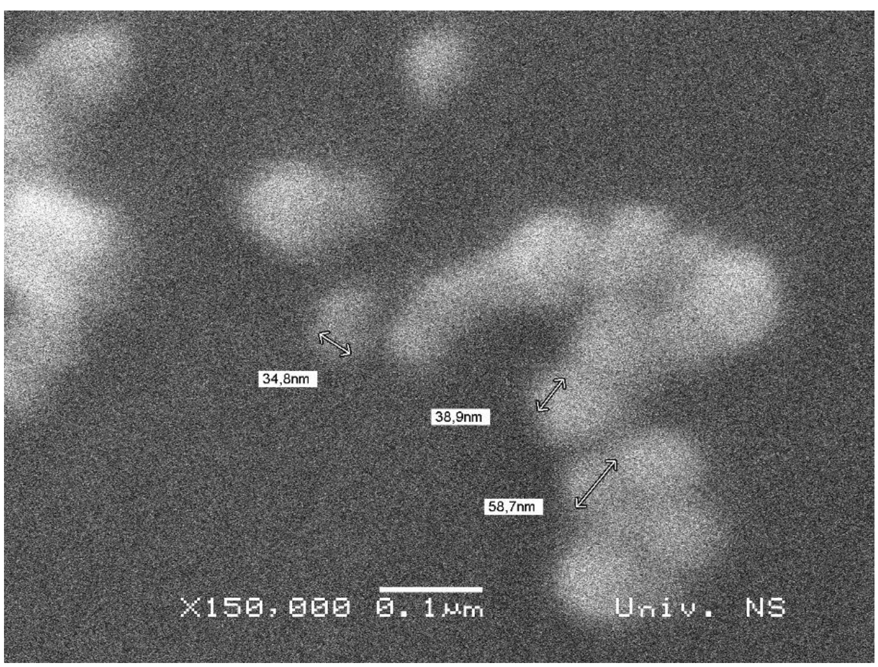

Figure 9. SEM image of nanogold particles on the surface of nonwoven polypropylene

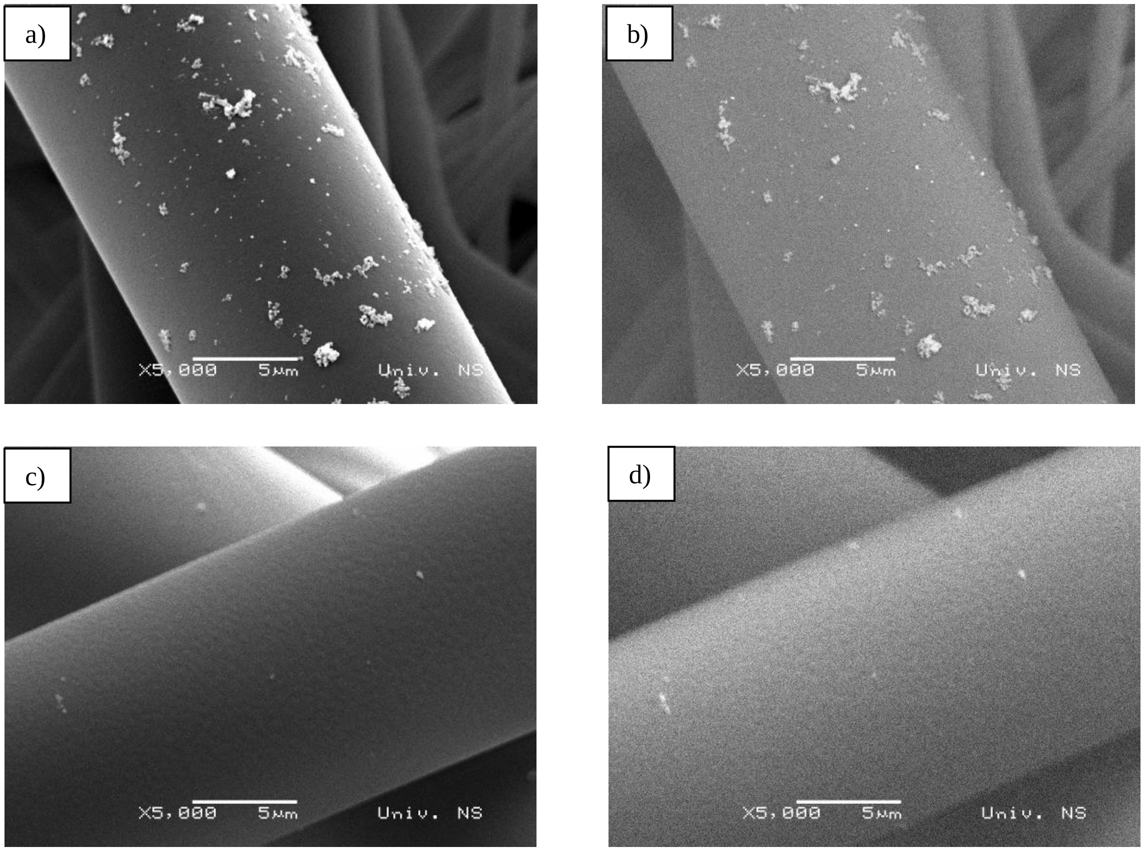

Figure 10. SEM images of plasma treated nonwoven polypropylene after absorption of gold nanoparticles

a) SEM image of PP treated by volume DBD
b) Back scattering image of PP treated by volume DBD
c) SEM image of PP treated by DCSBD
d) Back scattering image of PP treated by DCSBD

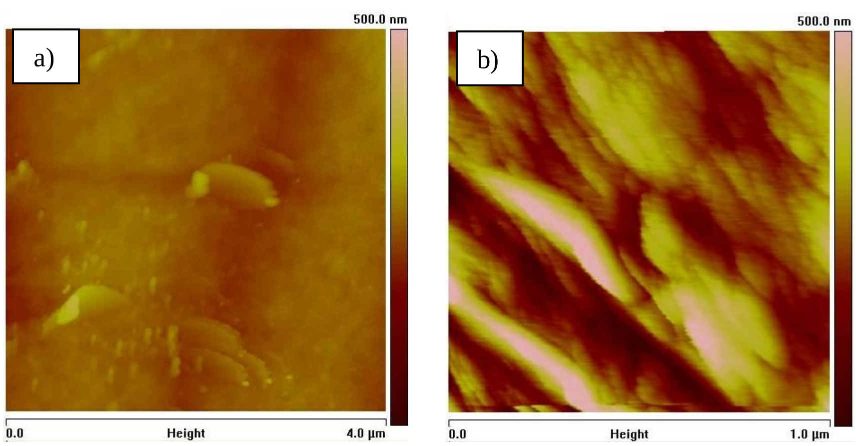

Figure 11. 2D AFM image of plasma treated polypropylene after absorption of gold nanoparticles

a) Plasma treatment by volume DBD
b) Plasma treatment by DCSBD

AEM 5700

Samples of untreated polypropylene and plasma treated nonwoven PP by DCSBD and by volume DBD were immersed in the 1% solution of AEM 5700 (3-trimeth-oxysilylpropyldimethyloctadecyl ammonium chloride) and dried at air 24 h. Samples were flushed with water and dried at air 24 hours before analyzing.

Fig. 12. and 13. show the fiber of PP fabric grafted with solution of AEM 5700. Here we can see in detail the AEM 5700 layer coating the fiber of PP fabric. AEM 5700 does not create a continuous film but covers the individual fibers.

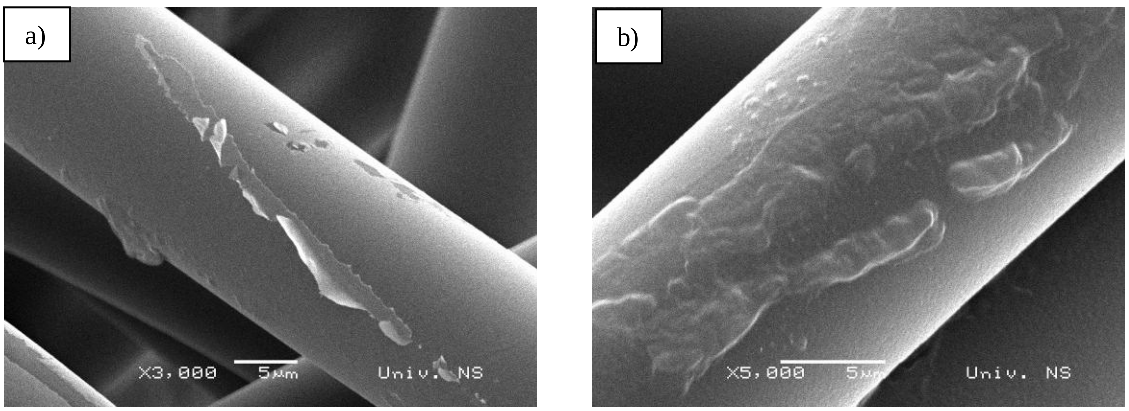

Figure 12. SEM images of polypropylene after absorption of AEM 5700

a) Untreated polypropylene
b) Treated polypropylene by DCSBD

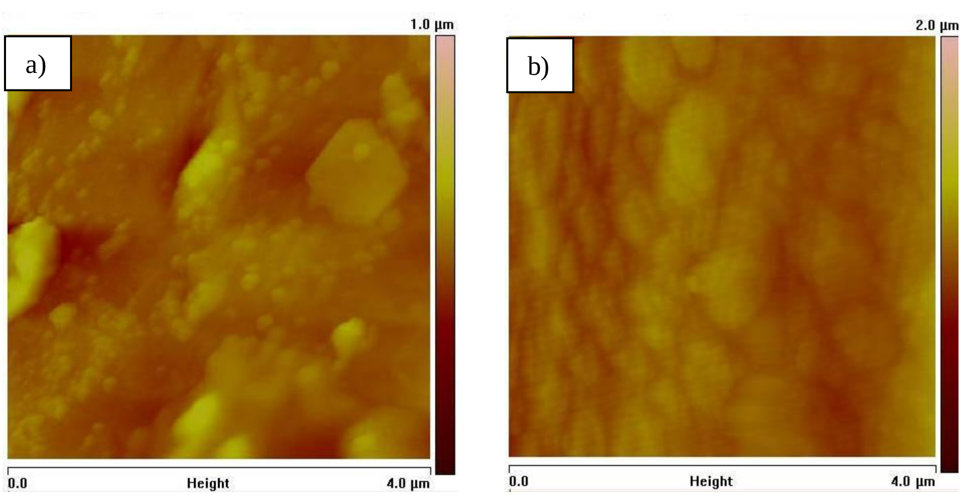

Figure 13. 2D AFM images of polypropylene after absorption of AEM 5700

a) Untreated polypropylene
b) Treated polypropylene by DCSBD

- **<u>Future collaboration with host institution (if applicable)</u>**

  Future visit at the host institute is planed in the second half of year 2010. Results obtained during this visit opened a lot new questions connected with effects of different plasma treatments on surface of polypropylene. Sending one DCSBD device to Belgrade for easier preparation of samples is also in plans. The main aim of these visits would be the study of plasma activation on atmospheric pressure and chemical functionalisation of polypropylene which lead us to find answers about structure changes of the surfaces after plasma treatment and influence of plasma treatment on bonding of chemicals on the plasma treated surfaces.

- **<u>Projected publications/articles resulting and to result from the STSM</u>**

  During this visit rich material was obtained for publishing min 2 papers in the journal of Plasma Processes and polymers.