## Fabrication of A Superhydrophobic Surface with Silica Nanoparticles and Polytetrafluoroethylene

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#### ABSTRACT

In this paper we fabricate a superhydrophobic surface with Silica nanoparticles and Poly tetrafluoroethylene (PTFE) by using spin coating and microwave plasma treatment on the glass substrate. During the spin coating, the effect of rotation speed on superhydrophobicity is investigated and the optimum rotation speed with high contact angle is identified. It is found that the reaction of PTFE by microwave argon plasma yields a superhydrophobic surface with contact angle of 163° which is more than the contact angle of the surface without microwave plasma coating. The hydrophobicity of the surface is explained based on Wenzel and Cassie-Baxter regime. The characterization of the rough structure including SiO<sub>2</sub> nanoparticles aggregates coated by PTFE is carried out by Scanning Electron Microscopy (SEM), Contact Angle (CA) by contact angle goniometer with deionized water at room temperature.

Keywords: Superhydrophobic, Microwave plasma, Spin coating, Silicon dioxide

# Silika Nanoparçacik ve Politetraforoetilen Kullanarak Süperhidrofobik Yüzey Fabrikasyonu

## ÖZET

Bu çalışmada silika nanoparçacıkları ve Poly tetrafluoroethylene (PTFE) ile döndürmeli kaplama ve mikrodalga plazma kullanarak cam alttaş üzerine süperhidrofobik yüzey fabrikasyonu gerçekleştirilmiştir. Döndürmeli kaplama süresince süperhidrofobiklik üzerine döndürme hızı incelenmiş ve en büyük temas açısı için en iyi döndürme hızı belirlenmiştir. Argon ortamında mikrodalga plazmanın etkisiyle PTFE kaplı yüzeyin temas açısının 1630 ile mikrodalga plazma uygulanmadığı duruma göre daha yüksek olduğu tespit edilmiştir. Hibrofobiklik, Wenzel ve Cassie-Baxter durumuna uyduğu görülmüştür. Üzerine PTFE kaplı SiO2 nanoparcacıklar içeren yüzeyin karakterizasyonu Taramalı Elektron Mikroskopu (SEM) ile ve oda sıcaklığında de iyonize su kullanarak temas açısı (CA) ölçümleri yapılmıştır.

Anahtar Kelimeler: Süperhidrofobik, Mikrodalga plazma, Döndrürmeli Kaplama, Sisliyum dioksit

## 1. Introduction

Wettability of a surface is one of the important properties of a material which is

introduced by "hydrophobicity" term. A surface is hydrophobic when, the contact angel between surface and water is larger than 90°, while the contact angel is greater than 150° it is ultra-hydrophobic which is called superhydrophobic (Ramaratnam et al., 2008). On a superhydrophobic surface a droplet place as a circle on the surface and in some cases the surface will repel the droplet (Jung, 2009). Superhydrophobic surfaces have many applications in industry such as corrosion, self-cleaning materials, anti-icing, micro-fluidics, oxidation and other applications (Foroughi Mobarakeh, Jafari, & Farzaneh, 2011). Depending on the applications, there are various methods to create superhydrophobic surfaces like: including plasma treatment, lithography, sol-gel process, chemical vapor deposition, electrospinning, and colloidal assemblies (Gao, Yan, Chen, & Mee, 2011). Surface roughness and surface chemistry are two fabricating important factors in а superhydrophobic surface (Xue, Jia, Zhang & Ma, 2010). Hence, these surfaces prepared during a two-step process, surface roughness creation and coating with low surface tension materials (Momen & Farzaneh, 2012). There are some regimes that explain the factors. Wenzel regime that indicates the roughness and Cassie-Baxter model that indicates the low surface energy materials in superhydrophobic surfaces. Nanoparticles, especially Silica nanoparticles are used to create roughness on a surface (He et al., Silica nanoparticles 2012). can create hierarchical structure with nano and microscale roughness on a surface (Gao et al., 2011). Many polymers are hydrophobic, Fluorocarbons such as Teflon are materials with low surface energy, these materials are

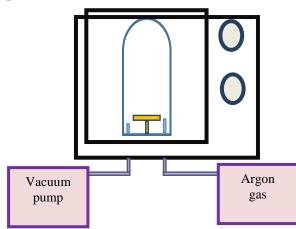
thermally and UV stable and can be used in waterproof clothing, concrete and paint with very low friction in water (Irzh, Ghindes, & Gedanken, 2011). In this study we prepare a hierarchical superhydrophobic surface including micro and nanostructure from SiO<sub>2</sub> and PTFE by of Plasma coating and without MW (Microwave) Plasma coating.

#### 2. Materials and Methods

PTFE spray which is for lubrication in industry application, Silica (SiO<sub>2</sub>) nanoparticles (fumed powder, Aldrich) with mix of two different average sizes (5 nm and 15 nm) are used in this study. Handmade spin coating and domestic microwave oven (1000W, Kenwood) including a drilled hole in its down part, a plasma chamber made from Pyrex, an argon cylinder, a vacuum pump is applied as a reaction system.

Glass substrates were cleaned during 3 cleaning process, first ultrasonically cleaned in acetone and distilled water, the dried and cleaned in piranha solution and at last cleaned by HF solution to remove oxides on the surface.

Silica suspension was prepared by adding 0.3 mg of SiO<sub>2</sub> nanoparticles in 10 ml toluene. Then the suspension was sonicated for 1 hour. The prepared suspension was coated on the clean dry glass substrates by spin coating in different rotation speed. The wettability of these coatings was investigated using water droplets by Kruss contact angle goniometer to measure the contact angle. Then prepared solution of PTFE in toluene (%w/v) was spin coated on rough glass slides deposited by silica nanoparticles. Then substrates divided into two groups, first group after spin coating process were placed in high vacuum system for 30 min and then dried in oven for 2 hours in 120°C, the second group after spin coating process were placed in microwave plasma chamber for plasma treatment, schematic illustration of the reaction system is shown at Figure 1, the vacuum pump start working the pressure inside the chamber down to  $1 \times 10^{-2}$  Torr, argon was pumped through the chamber for 15s, then the microwave activated in order to plasma treatment for about 5s.



**Figure 1.** Schematic illustration of the reaction system.

The treated glasses by microwave plasma placed in high vacuum for 30 min and then dried in oven the same as the first group.

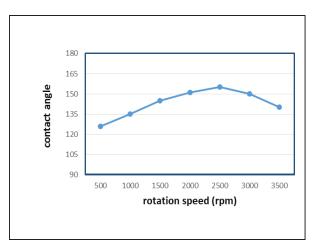
#### 3. Results and Discussion

We investigate the surface hydrophobicity of different rotation coatings according to the Table 1, the effect of spin coating rotation in surface roughness is depicted in Figure 2. The contact angle measurements show that the contact angle increases by increasing the rotation speeds. By increasing the speed, dispersion of SiO<sub>2</sub> nanoparticles, so the film thickness is decreases after 2500 rpm.

**Table 1.** Spin coating rotation speed effectin hydrophobicity.

Sample	SiO₂ (mg)/ (ml)	Rotation (rpm)	Contact Angle (°)
1	0.3	500	126
2	0.3	1000	135
3	0.3	1500	145
4	0.3	2000	151
5	0.3	2500	155
6	0.3	3000	150
7	0.3	3500	140

After spin coating with PTFE with concentration of 1 % w/v, contact in each rough surface increased because of coating by low surface energy PTFE. Plasma treatment of the same surfaces coated with SiO<sub>2</sub> nanoparticles showed about 4 degrees increase in contact angle. The water droplets easily roll off the surface coated in 2500 rpm and treated by Microwave Plasma coating. We find out that the hydrophobicity property



**Figure 2.** Contact angle relation with roughness in different rotation speeds

of the surfaces turns into superhydrophobicity and rolling property.

In surfaces coated in 2500 rpm and treated with Microwave Plasma, micro and nanoscale structure created because of the aggregation of silica nanoparticles. The aggregation in this rpm results in suitable roughness with good thickness. Superhydrophobicity enhanced by creating the micro and nano hierarchical structures. SEM images of the surfaces in Figure 3 show the surface hierarchical structure.

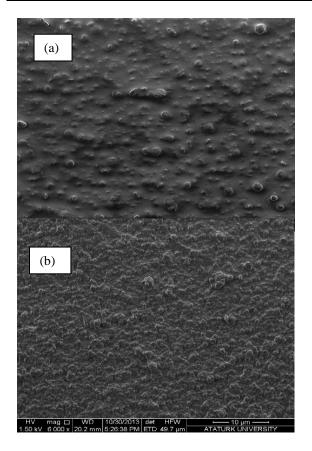
After spin coating by PTFE the contact angle increased about 2 degrees without microwave plasma treatment. After MW plasma treatment the contact angle increased about 4 degrees for some surfaces according to the Table 2.

The change in contact angle and change increasing in hydrophobicity property into superhydrophobicity and water repelling property after microwave plasma coating shows that the hydrophobicity before coating with PTFE follows the Wenzel regime which indicated just for roughness but after coating with PTFE and increase in hydrophobicity shows the change of Wenzel regime into Cassie-Baxter regime because of air trapped between roughness and reduce the adhesion between droplets and surface. It means that hierarchical structure of roughness is the main factor of change in Wenzel model into Cassie-Baxter model.

**Table 2.** Contact angle measurementsbefore and after microwave plasmatreatment.

Sample	Rotation (rpm)	Contact Angle (°) before MW plasma	Contact Angle (°) after MW plasma
1	500	127	128
2	1000	138	139
3	1500	146	150
4	2000	153	155
5	2500	159	163
6	3000	152	153
7	3500	143	147

The change in contact angle and hydrophobicity change increasing in property into superhydrophobicity and water repelling property after microwave plasma coating shows that the hydrophobicity before coating with PTFE follows the Wenzel regime which indicated just for roughness but after coating with PTFE and increase in hydrophobicity shows the change of Wenzel regime into Cassie-Baxter regime because of air trapped between roughness and reduce the adhesion between droplets and surface. It means that hierarchical structure of roughness is the main factor of change in Wenzel model into Cassie-Baxter model.



**Figure 3.** SEM image of 500 rpm rotation speed coating (a), SEM image of optimum 2500 rpm rotation speed coating (b)

## 4. Conclusion

This paper presents a simple method to fabricate a superhydrophobic coating on a glass surface. For 0.3 mg solution of silica nanoparticles the largest contact angle is observed when the rotation speed of coating is 2500 rpm while hierarchical structure is created and superhydrophobic property of this surface increased up to 159 after coating by PTFE and water repellency property is observed in this surface after microwave plasma treatment.

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