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Hydrophobic coating on glass surfaces via application of silicone oil and activated using a microwave atmospheric plasma jet

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ABSTRACT

In this study, a hydrophobic coating on glass surfaces was fabricated by application of a silicone oil lubricant and activated using a microwave atmospheric plasma jet. Optimization of the treatment was done by variation of the working gas flow rates, input microwave power and plasma treatment time, based on contact angle measurements. In comparison with the untreated glass (37.6°), results show that at best discharge conditions of 600 W microwave power, 5/0.5 LPM Ar/N_2 flow rate and 10 s treatment time, the plasma-treated glass obtained a water contact angle of 105.7°. Surface energy of the glass also decreased from 45.07 mN/m for the untreated to 27.97 mN/m after plasma treatment. Atomic force microscopy (AFM) and Fourier transform infrared (FTIR) spectroscopy results suggest that increased root-mean-square roughness and introduction of hydrophobic species may have been responsible for the hydrophobicity of the glass surface.

1. Introduction

Hydrophobic glass surfaces are widely used in different fields such as in microbiology, optics and microelectronics [1–3]. These surfaces are characterized by low surface energy ($\gamma < 35$ mN/m), and high contact angles ($\theta > 90°$) [3,4]. In order to fabricate hydrophobic surfaces, different approaches have been done including surface texturing [5], chemical treatment [6,7], solvent exchange method [8], spin coating technique [9], and plasma treatment [10–15].

Surface treatment of glass using atmospheric plasma has the advantage of modifying surface properties without alteration of the bulk material. In addition, it is fast, effective and relatively cheaper due to the removal of a vacuum system. Previous studies on atmospheric plasma treatment of glass surfaces employed silicone-based oils to form the hydrophobic coating. Silicones are composed of Si–O–Si, Si–O, and freely rotating methyl (CH_3) or phenyl (C_6H_5) groups, rendering them hydrophobic [16]. Three approaches were suggested for hydrophobization of glass including: (i) surface roughening [13], (ii) hydroxylation [3,14], and (iii) plasma-oil interaction [12–15]. For these approaches, dielectric barrier discharge [12–15], plasma torch [17], and radio frequency atmospheric plasmas [3,17] are more frequently used.

In terms of efficiency and cost effectiveness, microwave-induced atmospheric plasmas are one of the most attractive atmospheric plasma systems due to its lower power and gas flow rate requirements. However, no study has been found implementing this type of atmospheric plasma system for the hydrophobization of glass. As such, the aim of this study is to fabricate hydrophobic coatings on glass surfaces via application of a silicone oil lubricant, and activated using a microwave atmospheric plasma jet. A set of discharge parameters including working gas flow rates, input microwave power and treatment time was optimized based on contact angle measurements. Aging time in air of the hydrophobic glass coating was also determined.

2. Methodology

Glass slides (Sail Brand CAT. No. 7101) with dimension 76.2 ×25.4 × 1.0 mm were used in the experiments. A two-stage treatment process was done using the plasma device as discussed in the succeeding sections.

2.1. Microwave atmospheric plasma jet

The microwave atmospheric plasma jet (MAPJ) device is composed of a 2.45 GHz magnetron with up to 3 kW continuous wave output, the tuning system and the tapered waveguide as shown in Fig. 1. The tuning system consists of the three-stub tuner and a sliding short. Gases are injected through the gas nozzle located at the top of the tapered waveguide. Microwaves generated by the magnetron propagate through the waveguide assembly and ignites the plasma in a hollow cylindrical quartz discharge tube 20 mm in diameter inside the tapered waveguide. To prevent melting of the discharge tube due to

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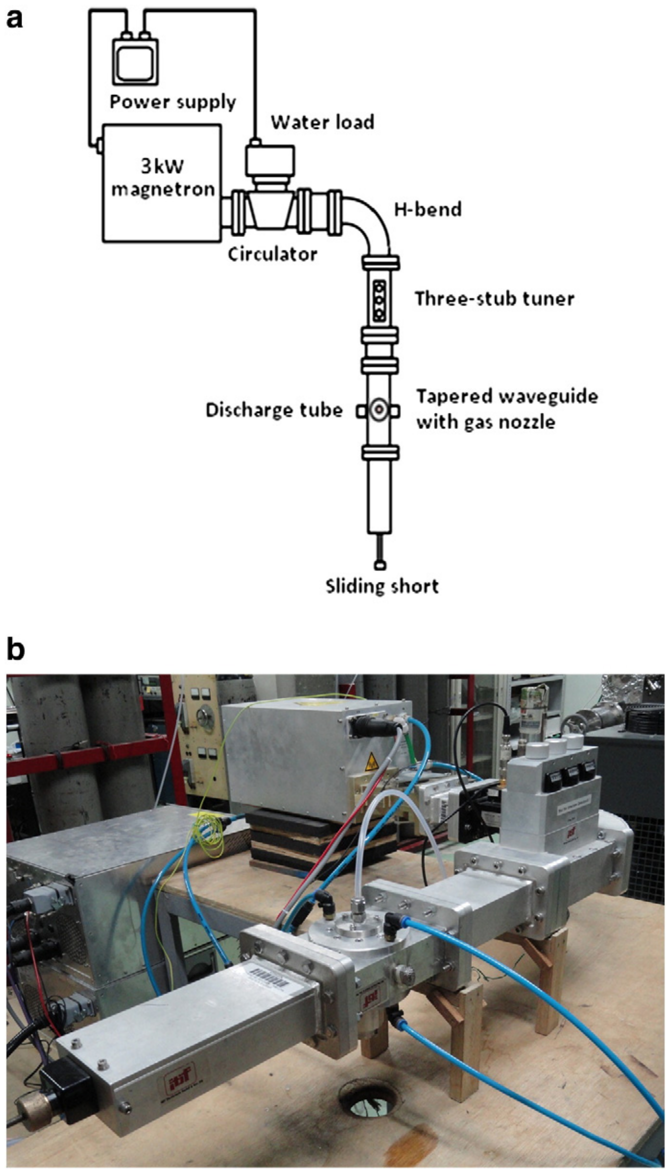

Fig. 1. (a) Schematic diagram and (b) actual image of the microwave atmospheric plasma jet.

plasma heating, two sets of gas ports were created: (a) a central port for the central gas, and (b) eight peripheral gas ports positioned uniformly around the central port for the "shroud" gas. The shroud gas acts as a barrier that protects the wall of the discharge tube from the plasma. This results to a stable plasma that is confined at the center of the discharge tube. In addition, the shroud gas also serves as a secondary gas that partially mixes with the working gas and undergoes ionization as well. A 5.9 kW recirculating chiller serves as the cooling system.

2.2. Plasma treatment

Glass slide samples were positioned 3 cm below the discharge tube. Initial plasma treatment was done at 600 W input microwave power and 5/0.5 LPM Ar/N_2 gas flow rate, for 10 s treatment time to increase its surface energy and remove any surface contaminations. It is noted that for all treatments, argon (Ar) was injected as the central gas, with nitrogen (N_2) serving as a shroud gas. Afterwards, the samples were subjected to further plasma treatment.

The hydrophobic coating was formed by application of one drop of silicone oil (3-in-one Professional Silicone Spray Lubricant No. 10041) using a 0.5 mL syringe on the glass surface, then subsequent activation using plasma treatment. Treatment was optimized using One-Factor-at-a-Time method wherein a baseline set of values is set and each factor/parameter was successively varied while other parameters were kept constant. For this study, the baseline values were 600 W microwave power, 5/0.5 LPM Ar/N_2 gas flow rates, and 5 s treatment time. The first parameter varied was input microwave power (300, 400, 500, 550, 600, 700, 800, 900 and 1000 W). Based on the highest average water contact angle (WCA), the best power was determined and used to vary the succeeding parameters which were N_2 gas flow rate (0.5, 1, 3, 5 and 7 LPM), treatment time (1, 3, 5, 8, 10, 12 and 15 s) and Ar gas flow rate (4, 5, 6, 7 and 8 LPM). Based on the highest average WCA, the best set of input microwave power, treatment time, and Ar/N_2 gas flow rates were determined. Aging time in air of the best sample was also investigated for a period of 50 days to determine the stability of the hydrophobic coating.

2.3. Characterizations

Changes in the wettability of the glass samples were determined via water contact angle measurements using Dino-Lite Digital Microscope Premier and DinoCapture 2.0 software. For surface energy calculations, deionized water (DI) and ethylene glycol (EG) were used as polar test liquids, whereas diiodomethane (DM) was used as the non-polar liquid. Test liquids were dropped onto the sample surface using a 0.5 mL syringe. The average of ten measurements was considered as the contact angle of the sample.

The chemical composition and surface roughness of the untreated and plasma-treated glass surfaces were analyzed using Fourier transform infrared spectroscopy (FTIR-ATR) and an NT-MDT Solver atomic force microscopy (AFM). AFM images were taken for a sample size of $50 \times 50\ \mu m^2$, and were corrected using Flatten Correction 2D option to remove the "tilt" effect.

2.4. Surface energy calculations

The Young's equation [Eq. (1)] describes the relation between the interfacial energies and the equilibrium contact angle (θ) formed by a given liquid drop (L) on top of a solid surface (S). The surface energies at the liquid–vapor, solid–vapor, and the solid–liquid interfaces are designated as γ_{LV}, γ_{SV}, and γ_{SL}, respectively. Since interactions with vapor are very low, γ_{LV}, is often considered as the surface energy of the liquid (γ_L), whereas, γ_{SV} is the surface energy of the solid surface (γ_S) [18,19]. In order to calculate the solid surface free energy γ_S, an estimate of γ_{SL} is done through Eq. (1).

$$\gamma_{LV}\cos\theta = \gamma_{SV} - \gamma_{SL} \tag{1}$$

Good, Van Oss and Chaudhury developed an equation expressing the total surface energy γ_{TOT} as the sum of the Lifshitz–van der Waals apolar component γ^{LW} and Lewis acid–base polar component γ^{AB}, as shown in Eq. (2). γ^{LW} considers the contributions from van der Waals forces originating from dipole–dipole (Keesom force), and dipole-induced dipole (Debye and London) interactions. On the other hand, γ^{AB} takes into account all acid–base interactions such as electron donor–electron acceptor interactions [19–21].

$$\gamma_{TOT} = \gamma^{LW} + \gamma^{AB} \tag{2}$$

The acid–base polar component γ^{AB} resulting from electron donor (γ^-) and electron acceptor (γ^+) interactions is given by Eq. (3). Using Eqs. (2) and (3), the surface energy for a solid can be expressed as in Eq. (4)

$$\gamma^{AB} = 2\sqrt{\gamma^+\gamma^-} \tag{3}$$