

Research Article

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Effect of Durable Superhydrophobic FS/PS Using DCTES on Carbon Steel

Rafik Abbas, Ahmed Hefnawy, Wael I El-Dessouky*, Asmaa El-Halag, Wagih A Sadik and Abdel Ghaffar M El-Demerdash

Department of Materials Science, Institute of Graduate Studies and Research, Alexandria University, 163, Horreya Av., El-Shatby 21526, P.O. Box: 832, Alexandria, Egypt

Abstract

Durable superhydrophobic surface on metal substrates was fabricated using very applicable method. The fabrication method aimed to decrease surface free energy and increase surface roughness at same time the process have been accomplished via the addition of silica nano particles with Dodecyltrimethoxysilane (DCTES) bonded to the surface of the silica particles. Adhesive polystyrene was added to improve bond between substrate and functionalized silica. The method is cheap, promising to be applied in factories heat exchangers to reduce fouling and corrosion on the large industrial scale. Scanning electron microscopy (SEM) was used for surface morphology analysis, showing the roughness produced by surface treatment. The wettability of the micro-nano silica film varied from hydrophilicity (water contact angle 88°) to superhydrophobicity (water contact angle 165.2°), while sliding contact angles dramatically decreased (<4°) by adding Functionalized silica and/or adhesive polymer. Roughness increased with silica increment which improves the wettability. The coatings were electrochemically characterized by electrochemical impedance spectroscopy (EIS) and Tafel polarization curves; it was found that both systems had good performance against corrosion in 3.5% sodium chloride solution.

Keywords: Superhydrophobicity; Metal surface; Contact angle; Silane; Corrosion resistance

Introduction

Recently, the solid surface with unusual wettability possessing both superhydrophobic and superoleophilic property has attracted great interest of research because of its enriched practical applications, including as a self-cleaning surface [1], as marine coatings [2-5], and as separation films for water and oil [6-8]. Superhydrophobic surfaces are usually fabricated via two approaches: creating rough structures on intrinsically hydrophobic substrates, or chemically modifying rough surfaces with low surface free energy materials [9-12]. So far, many methods have been used for fabricating the superhydrophobic surface [13-16]. All of these different methods for fabricating the superhydrophobic surfaces are almost based on the two main methods, which are to create the rough structure on the hydrophobic surface and to modify a rough surface by materials with low surface free energy. Therefore, the roughness is crucial for the preparation of the superhydrophobic surface [17].

It is well known that the wettability of solid surface could be characterized by contact angel (CA) of water droplets. If the CA of a surface is lower than 90°, the surface is described as a hydrophilic surface. In contrast, if that of a surface is higher than 90°, the surface is described as hydrophobic surface for smooth surfaces; CA can be represented by Young's equation. For rough surfaces, the wetting state can be described by the Wenzel [18,19] and Cassie-Baxter models [20]. The Wenzel model assumes that the liquid is in intimate contact everywhere with the rough surface, and completely fill any surface structures. The roughness factor in the Wenzel equation can enhance the natural state of material, and gives hydrophilic surfaces a lower contact angle and hydrophobic surfaces a larger contact angle. The Cassie-Baxter model introduces another new wetting state, in which the liquid allows very low friction during droplet movement, which introduces a low sliding angle. Both the Cassie-Baxter and Wenzel states can cause high static contact angles, but only the Cassie-Baxter state can lead to very low sliding angle.

Superhydrophobic materials have emerged as a new method to improve the corrosion performance of metals. Materials with such superhydrophobicity property possess the capacity as physical barrier to prevent corrosion electrolyte from penetrating onto metal substrate. Superhydrophobic films have been successfully prepared on lots of metals and their alloys, such as aluminum [21-23], magnesium [24,25], copper [26,27] and zinc [28,29] for corrosion protection.

Experimental Section

Materials

Steel alloy substrates with 3 mm thickness were supplied by The Egyptian Company for Copper Works, Alexandria, Egypt. Silicon dioxide nanopowders (99.5%; 15 nm) were purchased from MKNANO, Canada. Dodecyltrimethoxysilane (DCTES) was purchased from Zhejiang Feidian Chemical Company Ltd., China. Tetrahydrofuran (THF) was purchased from Central Drug House Ltd. polystyrene PS E251D (M wt. 6000 g/mole) purchased from Styrenex Company, Egypt. Ethanol 95% was purchased from Elnasr pharmaceutical Chemical Company, Alexandria, Egypt. All reagents were used as received without further purification.

Preparation of hierarchical superhydrophobic coatings

All substrate specimens were prepared with dimensions of 60 mm \times 25 mm \times 0.3 mm. The substrates cleaned by ethanol followed by acetone and dried in air. Functionalized silica nanoparticles (FS) are Silicon dioxide nanopowders treated with silane coupling gents. Three grams of DCTES, with long alkyl chain, were added dropwise to the hydrolysis ratio of ethanol to distilled water (ethanol ETOH: distilled water DW) 95: 5 after vigorously stirred for approximately 15

*Corresponding author: Wael I El-Dessouky, Research Assistant, Materials Science Department, Institute of Graduate Studies and Research, Alexandria University, 163, Horreya Av., El-Shatby 21526, P.O. Box: 832, Alexandria, Egypt, Tel: +20 122 382 9 252; E-mail: Wael200374@yahoo.com

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min at room temperature. Then, the solution was agitated for 3 hours [30,31]. Different concentrations of silica nanoparticles (i.e., 1, 1.5, 2 and 2.5 wt.%) were prepared by adding 1, 1.5, 2 and 2.5 g of silica to 100 ml ethanol and DW. Silicon dioxide nanopowders with previous percentage were added to the solution and agitated for extra 3 hours. After agitation the solution was kept in closed flask for 2 days at room conditions PS with specific amounts was added to different samples of the functionalized silica (FS) solutions. The spraying distance was 25 cm then the substrate was held in room temperature for 24 hours. The final coating solution was sprayed with nitrogen gas by a spray gun nozzle at the spray rate of 5 cm³ per minute during the spray deposition. The substrate temperature was maintained at room temperature for 24 hours.

Sample characterization

Water contact angle (WCA) and sliding angle are considered the main values in superhydrophobicity classification. CA measurements were performed using Gaosuo USB digital microscope. All the angles were determined by averaging values measured at five points on different locations on each sample surface. Surface morphology of samples was examined using scanning electron microscope (SEM, JSM-6390A). Samples were coated with a thin layer of gold using a sputtering machine prior to examination with SEM. Surface roughness mathematical value (R_a) was examined using Mituoyo SJ-201. Substrate surface was tested in ten different locations were the average value was supplied as surface roughness.

Electrochemical measurements

The corrosion behavior of the blank steel alloy with different superhydrophobic surfaces were investigated by electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization in NaCl solution (3.5 wt.%) using electrochemical system (GAMRY PCI4G750 instruments) at room temperature a typical-three electrode was used in electrochemical testing with reference electrode (saturated calomel electrode), a platinum wire as the counter electrode (auxiliary electrode) and the sample with an exposed area of 1.0 cm² as the working electrode as demonstrated. EIS were performed in the frequency range between 10 MHz and 100 kHz. Polarization curves (Tafel plots) were acquired at a scanning rate of 1 mV/s from -0.25 to 0.25 V. The corrosion current density (i_{corr}) for the specimens was determined by extrapolating the anodic and cathodic Tafel slopes. All EIS spectra were analyzed in Nyquist representation.

Results and Discussion

FTIR

Coating layer Characterized by using FTIR to check the formation of the FS where FTIR spectra of different silanol solution were recorded in the range of 400-4000 cm⁻¹ as shown in Figure 1. Figure 1a and 1b shows spectra of pure and hydrolyzed DCTES. By comparing the two spectra it was found that the strong peak which appeared at about 3389 cm⁻¹ is due to the presence of large amount of silanol OH group, Figure 1 shows FTIR spectrum of the hydrolyzed DCTES with SNP to



form FS the strong and broad peak which appeared at 3362 cm⁻¹ was attributed to the presence of large number of OH groups on the surface of the silica nanoparticles (SNP). From the resulted FTIR, the expected mechanism of formation of FS is as in Figure 2.

Surface morphology

Figure 3 shows SEM images under different Functionalized silica (FS) content sprayed on steel substrates. Figure 3a shows the surface morphology of the film prepared with 1 wt.% FS content in solutions. It's clear that the irregular forms of silica nano particles on substrate surface morphology also clarify the coating layer rough surface. A high-magnification SEM image the coating surface (Figure 3b) shows that the



Figure 2: The proposed mechanism of formation of the superhydrophobic solution of FS.



Figure 3: SEM images coated steel alloy surface with FS at different silica concentration (a and b) 1 wt.%; (c) 2.5 wt.% and (d) the corresponding high magnification.

particles morphology includes hierarchical micro/nano-structures. Figure 3c demonstrates the coating layer with higher functionalized nano silica of 2.5 wt.% in the sprayed solutions it shows that the particles become more uniform, smaller, more intensive and connected with each other as the silica percentage increased. And from the higher magnification SEM (Figure 3b and 3d), It's clear that the irregular forms of silica nano improve surface roughness which improve air entrapment in the microstructure, Where air layer can effectively prevent water-surface direct contact [32-34].

Figure 4 shows SEM images of Functionalized silica coating layer with PS as adhesive polymer. Figure 4a shows the surface morphology of the film prepared with 2.5 wt.% FS and 3 wt.% PS that increases the bond between FS particles causing silica particles to coagulate with each other. A high-magnification SEM image of the steel surface shows the PS and the FS particles in position (1) and position (2), respectively (Figure 4b) shows highly assembled and durable pattern where adhesives polymer form bridge-like creates strong bond increasing durability and bonding between silica particles.

The wetting behavior of the prepared surface

The surface wettability of the obtained superhydrophobic surface was studied by CA measurements. Figure 5 displays the profiles of CA of the obtained superhydrophobic surface as function of silica content on FS/polymer. It can be seen from Figure 5, without any polymer addition at 1 wt.% FS the CA is 156° and increased to 162° at 2.5 wt.% for steel this result demonstrates that silica achieve high roughness. As is reported, the superhydrophobic property can be achieved by an especial surface chemistry or surface geometrical microstructure [32]. This wettability can be well explained by Wenzel's equation [33]:

$$\cos\theta = r\cos\theta_0 \tag{1}$$

Where θ represents the apparent contact angle of water on the



Figure 4: SEM images coated surface by FS with PS at different concentration (a) 2.5 wt.% silica with 3% PS.



actual surface, θ_{\circ} is the equilibrium contact angle on a smooth flat surface and r is defined as the surface roughness ratio. According to eqn. (1), with increasing the roughness, the wettability of hydrophobic surfaces decreases. Introducing of PS as adhesive polymer as for low 1 wt.% PS CA was 148.6° and for higher 3 wt.% PS became 139.3° at low FS (1 wt.%). Increasing FS with PS show same improvement. For instance, at 1 wt.% PS with 2.5 wt.% FS has wettability of 156.8°. Increasing to 2 wt.% PS with FS 1 wt.% CA became 146° and with 2.5 wt.% FS CA was 154.3°. Further PS increase (3 wt.%) with 2.5 wt.% FS CA became 152.6°.

As shown in figure optimization should be done to attain superhydrophobicity and good stability which achieved at 2 wt.% FS and 2 wt.% PS.

Surface roughness

Surface roughness (Ra) is very important criteria in superhydrophobicity evaluation. Where, the hydrophobicity phenomena depend on surface tension and surface roughness. It is obvious that the surface roughness increases the hydrophobicity which directly proportional to the roughness of the coated layer. Figure 6 depicts the variation of surface roughness of coated steel surfaces with FS with DCTES. The surface roughness 310 nm for steel on as-received bare steel surface was found to increase to 670 nm, and 1700 nm at low content (1 wt.%) and high content (2.5 wt.%) of FS, respectively. This phenomenon is due to the unique effect of the hierarchical structure of sprayed rough silica on steel surface which revealed by the SEM images. By introducing the adhesive polymer, the roughness reduces as result of polymer layer bonding with FS due to the polymer layer bonding with FS particles and fills the vacant gabs between silica particles at 1 wt.% FS with 1 wt.% PS the Ra was 500 nm. Increasing adhesive PS to 2 wt.% the roughness dropped to 430 nm and 400 nm with 3 wt.% PS. Higher silica content 1.5 wt.% in coating layer shows higher roughness (1056 nm) but introducing PS resulting in roughness decrease to 960 nm with 1 wt.% PS. Further increase in PS concentration (2 wt.%), the Ra became 894 nm and for highest PS ratio (3 wt.%) the roughness became 850 nm. Moreover, higher silica content (2 wt.%) the roughness become 1200 nm with 1 wt.% PS. With increasing the PS content to 2 wt.%, the Ra decreases to 1165 nm and 1087 nm for 3 wt.% PS. Further increase in the FS, the roughness become 1150 nm for 1 wt.% PS, 1105 nm with 2 wt.% PS and the highest PS 3 wt.% give 1076 nm.

Corrosion resistance performance

Electrochemical impedance spectroscopy (EIS): Figure 7 depicts the Nyquist plots and Bode plots recorded for the bare substrate and resulting superhydrophobic surface in neutral 3.5 wt.% NaCl





solutions. Figure 7a shows the EIS results in the form of Nyquist plots, in which the imaginary impedance (Z_{im}) is plotted against the real impedance $(Z_{r_{r}})$. The results show quite different capacitive loops that can be attributed to the charge transfer resistance of the corrosion process (R_{et}). Steel substrate, the capacitive loops of the prepared superhydrophobic surface have a widespread trend attributed to a protective surface film of Si coating. It is well known that the diameter of the capacitive loop related to R_{et} in the Nyquist plots represents the impedance of the samples. The value of R_t for the superhydrophobic surface (5.05 k Ω) is much larger than that of the untreated substrate surface (0.091 k Ω), which indicates the superhydrophobic film has largely improved the corrosion properties. The same previous data is also displayed using Bode plot show the relation between the frequency (log f) with the equivalent impedance (log z) shown in Figure 7b. For better understanding the mechanisms of the corrosive processes, which occur at the surface of the samples studied, we have applied the fitting of the experimental impedance spectra using the appropriate equivalent electric circuits. The equivalent circuits are shown in Figure 8 and the fitted parameters are summarized in Table 1 for steel substrate. The equivalent circuit representing the electrochemical behavior of the untreated surface shows only one time constant. In this circuit, R_{rt} the charge transfer resistance, C_{al} the double layer capacitance, R_s the solution resistance. In the case of the silica modified superhydrophobic surface, the equivalent circuit model should have two time constants in the corresponding impedance spectra. The $R_{cl} || C_{dl}$ elements in Figure 8b showed the impedance with the interface reaction between the silica modified superhydrophobic film and the copper substrate. C_c would normally be assigned to the capacitance of the intact coating (a function of factors such as film thickness and defect structure). Its value is much smaller than a typical double layer capacitance. The resistance R_{po} (pore resistance) could be attributed to the resistance of ion conducting paths that develop in the coating (governed mainly by pore dimensions). Additionally, the inhibition efficiency (IE) of the coated modified silica was used to evaluate the corrosion protection performance of superhydrophobic surface, which can be calculated by the following equation [34]:

$$IE = \frac{R_{ct} - R_{cto}}{R_{ct}} \times 100$$

Where R_{cto} is charge transfer resistance of copper alloy covered with and without superhydrophobic coatings, respectively. Inhibition efficiency of superhydrophobic coatings increases up to 99% and showing excellent inhibition effect with increasing silica content closely related to their wettability and larger contact angle.

Potentiodynamic polarization: To investigate the instantaneous

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Specimen	Rct (kΩcm)	Cdl (µf)	n1	Rpo (kΩcm)	Cc (µf)	n2	Ru (Ω)	IE%
Steel substrate	0.091	124	0.656	-	-	-	2.5	-
1% Silica	4.09	2.91	0.276	0.186	0.113	0.721	0.198	97.7
1.5% Silica	4.13	0.981	0.686	0.21	0.317	0.102	2.18	97.49
2% Silica	5.05	3.84	0.429	0.375	0.27	0.624	9.25	99.19



Table 1: Electrochemical model impedance parameters derived from EIS for steel substrate.

Fe substrate	i _{corr} (µA cm⁻²)	E _{corr} (mV)	Corrosion rate (mpy)
0.%Si		-0.75	
1% Si	1.94	-0.76	0.592
1.5% Si	1.13	-0.713	0.445
2% Si	0.124	-0.665	0.238

 Table 2: The results of potentiodynamic polarization test of the steel alloy and after spraying of superhydrophobic coatings 3.5 wt% NaCl solution.



Figure 10: SEM of Aluminum in (a and b) shows the substrate after corrosion

test while (c and d) shows steel substrate after corrosion tests.

method, the corrosion current density (i_{corr}) and the corrosion rate (mpy) of the different sample can be got from Tafel curve (Table 2). The corrosion current density (i_{corr}) of the prepared superhydrophobic surface (2 wt.% Si) is 0.124 μ A/cm², while untreated steel alloy is 15.8 μ A/cm² and the corrosion rate of the sample after silica coating (0.238 mpy) high significant decreases compared with the untreated aluminum (5.83 mpy). From results obtained by potentiodynamic polarization and electrochemical impedance spectroscopy, it can be concluded that substrate is easily penetrated by the Cl⁻ in the seawater. So it cannot improve the corrosion resistance obviously. However, once the surface was chemical modified by silica/silane, the specimen can show excellent anticorrosion properties because of the superhydrophobicity phenomena. Figure 10 shows SEM after corrosion tests for aluminum and Steel respectively.

Conclusions

Superhydrophobic layer was fabricated on different metal surfaces with a contact angle around 165° and a sliding angle around 4° is successfully constructed on various metal substrates via applicable method. The fabrication process was cheap and easy to be applied on different metals, promising industrial applications for fabricating superhydrophobic surfaces on various metallic materials Functionalized silica nanoparticles (FS) was successfully used to





corrosion rate, the potentiodynamic polarization curves (Tafel) is employed. Figure 9 shows the potentiodynamic polarization curves of untreated aluminum alloy and prepared superhydrophobic surface in 3.5 wt% NaCl solution using the Tafel extrapolation method. The results of potentiodynamic polarization test are summarized in Table 2. The result clearly shows that the corrosion potential ($\rm E_{corr}$) positively increases from -0.76 V of the untreated aluminum alloy to -0.665 V of the sample of prepared superhydrophobic surface. This suggests that the film of silica/silane mainly retards the dissolution of aluminum between the interface of the aluminum surface and seawater According to the potentiodynamic polarization curves (Tafel) linear extrapolation Citation: Abbas R, Hefnawy A, El-Dessouky WI, El-Halag A, Sadik WA, et al. (2018) Effect of Durable Superhydrophobic FS/PS Using DCTES on Carbon Steel. J Material Sci Eng 7: 408. doi: 10.4172/2169-0022.1000408

construct superhydrophobic surface with high contact angle 165.2° and low sliding angle less than 4° with SNP content of 2.5 wt.%. The cooperation of rough surface with low surface energy plays an important role in the formation of effective superhydrophobicity. The results of the potentiodynamic polarization curves (Tafel) show that the low corrosion current densities and the results of the electrochemical impedance spectroscopy (EIS) show that the high impedance of the asprepared superhydrophobic surface. It is expected that such a technique may open a new approach for the large-scale industrial fabrication of superhydrophobic surfaces with the SNP on steel or other general conductive engineering materials.

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