



Regular Article

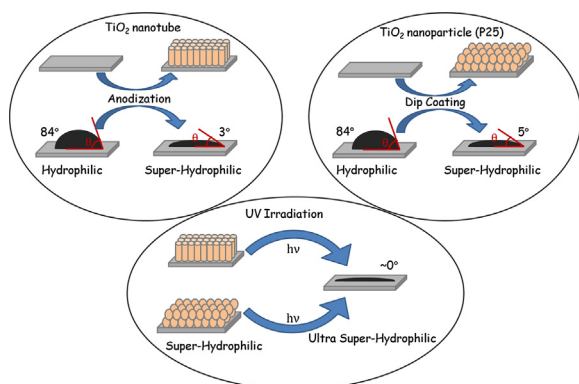
Fabrication of zero contact angle ultra-super hydrophilic surfaces



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GRAPHICAL ABSTRACT



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ABSTRACT

Zero contact angle surfaces have been created with the combined effect of nanostructure and UV illumination. The contact angle of titanium surface has been optimized to $3.25^\circ \pm 1^\circ$ with nanotubular structures through electrochemical surface modification. The porosity and surface energy of tubular TiO_2 layer play critical role over the surface wettability and the hydrophilicity of the surface. The surface free energy has been enhanced from 23.72 mJ/m^2 (bare titanium surface) to 87.11 mJ/m^2 (nanotubular surface). Similar surface with TiO_2 nanoparticles coating shows superhydrophilicity with contact angle up to $5.63^\circ \pm 0.95^\circ$. This implies liquid imbibition and surface curvature play a crucial role in surface hydrophilicity. The contact angle has been further reduced to $0^\circ \pm 0.86^\circ$ by illuminating the surface with UV radiation. Results shows that by tuning the nanotube morphology, highly porous surfaces can be fabricated to reduce contact angle and enhance wettability. This study provides an insight into the inter-relationship between surface structural factors and ultra-superhydrophilic surfaces which can help to optimize thermal hydraulic and self cleaning surfaces.

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1. Introduction

Ultra wetting surfaces have appealed much interest due to its wide range of applications in solar energy conversion, heat transfer, gas sensors and anti-fogging coatings. Surface morphology,

surface free energy, porosity, capillary wicking and roughness are considered to be the factors governing wettability [1–6]. Surface modification in the micro/nano scale range paves a way for tuning the wettability towards ultra high wetting. Fabrication of nanotubes and nanoparticle coating over the surface enhances the surface area which in turn enhances the spreading of liquid droplet. Due to wide range of material properties like thermal stability, corrosion resistance, non-toxicity make TiO_2 a potential candidate for

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solar cells, storage devices, water splitting, water purification, bio-implants, etc [7–11]. Due to overwhelming concerns over the energy savings, fabrication of ultra wetting surfaces has gathered momentum and TiO₂ has been identified as the potential candidate for such applications [12–17]. Hence, in recent years, huge intellectual investment has been ensued for fabrication of TiO₂ nanostructures like nanorods, nanowires and nanotubes. These structures are fabricated using various methods such as hydrothermal method, potentiostatic anodization, vapor deposition and sputtering techniques [18–20]. These results motivate the research for fabrication of super-hydrophilic surface with TiO₂ nanostructures.

Hamlekhan et al. [21] reported surface wettability enhancement with respect to increase in surface roughness. Conversely, Anitha et al. [22] and Minagar et al. [23] reported a decrease in wettability with increase in surface roughness. From the above discussion it is evident that, roughness factor alone does not ensure super-hydrophilic behavior. The mechanism of the effect of roughness in influencing the wettability needs to be quantified. Apart from surface roughness, nanoscale modified surfaces possess high surface energy leading to increased water adhesion. Sorkin et al. [24] reported an increase in surface energy from 44.8 mJ/m² to 71.5 mJ/m² for Ti foil and TiO₂ nanotube surface. Due to this increase in surface energy, contact angle has been decreased from 52° to 11° resembling enhanced wettability. Similar increase in surface energy and wettability upon nanotube fabrication has been observed and reported by Das et al. [25] and Minagar et al. [23]. TiO₂ samples exposed to plasma for longer time results in surface energy increase exhibiting superhydrophilicity [26]. Fang et al. [27] proposed a correlation of surface energy and hydrophobicity/hydrophilicity of nanoparticles and concluded that metal oxides are most hydrophilic in nature due to its high surface free energy [28–30].

In the present study, surface energy has been increased by fabricating highly porous TiO₂ nanotube surface. The increase in surface energy is confirmed with the help of Zisman and Owens-Wendt theory [31,32]. Surface energy and contact angle of Ti foil reads 23.72 mJ/m² and 86.49° with a deviation of ±1.2°, whereas for TiO₂ nanotube surface it is 87.11 mJ/m² and 3.25° with a deviation of ±1°. Photo-induced superhydrophilicity on TiO₂ nanoparticle coated and nanotube fabricated surface has been studied and achieved CA of 0°. The reversible wettability behavior was also confirmed by storing the samples in dark for prolonged hours. The influence of crystal phases in enhancing wettability has been studied by annealing the samples at different temperatures. Surface wettability enhancement with respect to nanotube morphology has also been investigated. From the present work, it is evident that nanotubes and P25 nanoparticle has higher surface energy leading to hydrophilic behavior. The mechanism of liquid filling inside pores of tubes and gaps in between nanoparticles enhances the spreading ability exhibiting lower droplet curvature leading to lesser contact angle values is illustrated in Fig. 1.

2. Experiment

2.1. Anodization

Titanium foil of thickness 2 mm were purchased from Sigma Aldrich (99.99%) and cut in the dimension of 1 cm × 2 cm for anodization. Before anodization the samples were polished with emery paper and sonicated with water and acetone for 5 min. The samples were anodized in ethylene glycol electrolyte containing 0.5 wt% NH₄F. Anodization was done at 40 V for 3 h and annealing temperature has been varied from 250 °C to 850 °C with heating and cooling rates of 10 °C min⁻¹. Anodization was carried out in room temperature with 10 V, 20 V, 30 V and 40 V for 3 h. The anodized samples were annealed at 450 °C in the air atmosphere for 3 h with same heating and cooling rate.

2.2. P25 nanoparticle coating

Aeroxide P25 TiO₂ nanoparticle is dissolved in acetic acid and grinded with mortar and pestle. The mixture is then added to 1 ml of DI water and mixed in grinder followed by adding ethanol of 1 ml and repeating the step for 15 times. The mixture is transferred to ethanol solution of 100 ml and stirred for 1 min. Add terpeneol of 20 g to the stirring mixture; add ethyl cellulose solution in the ratio of 1:10 by weight of TiO₂ solution. Stir final mixture for 3 min and evaporate the ethanol solution by heating it at 80 °C for 20 min. The prepared solution has been used for manual dip coating onto Ti foil of thickness 2 mm.

2.3. Evaluation

The crystal phases of TiO₂ nanotubular film was evaluated by X-ray diffractometer (Bruker AXS D8 Advance) with Cu K α radiation and the 2 θ was scanned in the range of 10–90°. Raman Spectroscopy (Renishaw inVia Raman microscope) with Ar ion laser source of wavelength 785 nm has been used to quantify the crystal phases. The surface topography of nanotubular films were observed using FESEM. Contact angles were measured using sessile droplet method; the experiment has been carried out at room temperature with the help of commercial optical tensiometer (Thetalite-TL 101) with experimental error of ±2°. The tensiometer is equipped with a light source to capture the image and video of spreading liquid using a CCD camera. The experimental apparatus uses the Young's Laplace fit for the determination of contact angle. After the evaluation of contact angle, the samples were irradiated with UV lamp (11 W) to study the influence of UV irradiation in wettability of TiO₂ nanotubular thin film. The titanium nanotube arrays (TNTA) sample exhibiting WCA 10° has been subjected to UV irradiation for an hour and its influence in enhancing wettability has been studied. The reversible wettability switching has been studied by storing the UV irradiated samples under dark conditions for long hours. In general, water is capable of adhering with high surface free energy substrates. Surface free energy of nanotubular

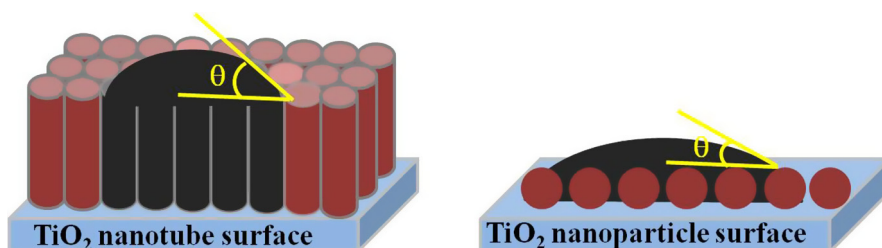


Fig. 1. Wetting mechanism in nanotube and nanoparticle coated surface.

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