Preparation and Characteristics of Hydrophobic Wettability Function Micro-Nano Structures Surface Fabricated by Femtosecond Laser

Sayed Abdul Moqim Hussaini 1\* (MSc.) Mohammad Nasim Wafa 2 (MSc)

Physics Department Mathematics Department

Education Faculty, Ghazni University Education Faculty, Ghor University

Ghazni, Afghanistan Ghor, Afghanistan

Email: [sayed\_moqim@yahoo.com](mailto:sayed_moqim@yahoo.com) **(\*Correspondence)** Email: [nasim\_wafa58@yahoo.com](mailto:nasim_wafa58@yahoo.com)

***Abstract*— Various micro/nanostructures can be prepared directly on the metal surface by using a femtosecond laser. These structures provide the metal surface with good wettability, for example, hydrophilic or hydrophobic function. Based on these functional characteristics, they find important applications in fields such as aerospace, architecture, medicine, and solar energy. In this paper, we use the femtosecond laser pulse on the stainless-steel surface and scan with a pitch smaller than the size of the focused beam size. After homogeneous laser irradiation on the surface, columnar two-scale micro/nanocomposite structures have been prepared. The results of this paper show that the contact angle of the micro /nano-structured surface can reach up to 148.6o after silanization. This discovery is of significant importance for the selection of the base materials for the preparation of metal wettability surface by micro/nanostructures.**

*Keywords—* **Femtosecond laser, Stainless Steel Micro/Nanostructures, Hydrophobicity, Hydrophilicity.**

# Introduction

In the last century one of the greatest inventions of humankind is the laser and the laser has significantly changed most of the aspects of our daily life. The manufacture of increasingly large optical electric fields is because of lasers, which have specified a substitution of new high-intensity optical regimes [1]. Femtosecond (1fs = 10-15 s) laser is a pulsed laser with the pulse width from 1 to 1000 femtoseconds and was developed in the last ’80s [1]-[2]. The femtosecond lasers are a revolution in technology and have thrilling potential for numerous applications and have fetched about important progress in the studying of light-matter interaction [1]-[3]-[5]. Ever since the original reports on femtosecond laser micromachining in the mid ’90s [6]-[7], revolutions in laser technology composed with photonic material design have empowered the observation of several kinds of new physical and chemical phenomena and encouraged micro and nanostructures, and hence, made probable for researchers and technologists to govern and manipulate light in an infrequent and exciting way. The intense high peak intensities in the effort of femtosecond laser pulses have proposed the probability for a diversity of new applications ranging from precise scalpels for delicate life science [8]. In order to drive sources for table-top particle accelerators [9]. In current years femtosecond laser micromachining has appeared as a new practice for fabrication micro and nanostructure as of its applicability to almost all kinds of materials in an easy one-step process that is scalable [10]. Femtosecond laser material processing has been demonstrated as a very effective means for micro/nano surface modification of solid materials due to less debris contamination, reproducibility, and minimal heat-affected zone. When irradiating a metal surface with femtosecond laser pulses, ripples, quasi-periodic nanostructures, or self-organized structures can be directly formed in a simple, one-step process [11]-[12]. This technique is referred to as femtosecond, laser-induced periodic surface structures (FLIPS).

Femtosecond laser microfabrication is emerging as a hot tool for controlling thewettability of solid surfaces [13]. Wettability plays a very important role in the creature’s survival and our daily life [14]-[15]. Recently, much attention has been paid to fabricate special wettable surfaces due to their significance in basic researches, practical applications, and bionics [16]-[17]. It is well known that the metal surface has medium wettability: it has neither good hydrophilic function nor hydrophobic ability, which makes the metal does not show good application value in this respect. In the past research work, pure copper [18] and aluminum materials [19] have been used to carry out experiments. The experimental results show that the wettability of the prepared samples will change spontaneously from hydrophilicity to hydrophobicity with the time when they are placed in the air. The special functions of many plants and animals in nature are closely related to the micro and nanostructures on their surfaces. In the development of new functional surface materials, these special micro and nanostructures provide important inspiration for researchers. It has broad application prospects and is expected to solve the problems of energy shortage and environmental pollution. Among many surface functions, wetting functional surface has not unique self-cleaning function but also has great potential application in anti-water, anti-fog, anti-icing [20]-[21], anti-corrosion [22], anti-biofouling[21]-[23] and other aspects, which can greatly improve the application value of materials and expand their scope of use. Therefore, more and more scientists and engineers begin to pay attention to and study functional wetting surfaces. This research has the flowing sections; section 2 contains a literature review, section 3 experimental and methodology, section 4 discussion and result in analysis, and section 5 has a conclusion and future works.

# II. Literature Review

A web application directly accessed and interpreted by clients’ browsers on the internet. The inspiration for the expansion of materials with superhydrophobic properties is for use in real-world applications. Superhydrophobic surfaces have attracted increasing interest in the last 20 years and the reason for this is their unique water repellency, self -cleaning property and their significance in numerous applications which includes self-cleaning windows, roof tiles, textiles, solar panels, and applications demanding anti-biofouling and reduction of drag in fluid (Micro/Nano scale) [24]. Some of its applications in agriculture were also discussed, such as biomedical applications [25], anti-sticking of snow for antennas and windows [27], stain resistant textiles [26], and many others. Corrosion resistance is one of the key applications for superhydrophobic surface is for metals and alloys, and remains the most important goal in this review. The following section consists that how superhydrophobic coatings are used to advance the performance of numerous applications by the surface modification. In a physical environment, the surfaces get polluted very easily and often and in order to clean them it requires lot of effort as well as time and money. Thus, the creation of substrates that are self-cleaning and that have a low-adhesion to toxins which has been a hot research topic for so many years. When a water droplet rolls off a superhydrophobic surface, it contains dust and pollutants. This is not the case for normal surfaces, where the dust remains. Self-cleaning ability depends on superhydrophobic or superhydrophilic functions. In superhydrophobic surface bucket, water droplets are easy to roll and fall under the influence of external factors. In the process of rolling, they will take away the tiny mud or dust and other dirt along the way. For example, the lotus leaf surface introduced above is a classic example of self-cleaning ability. The self-cleaning principle of superhydrophilic surface is different from that of superhydrophobic surface. It has self-cleaning ability because water droplets are easy to spread out on the surface, forming a layer of water sense between pollutants and superhydrophilic surface, thus reducing the yellow-adhering force of pollutants. Sea dyes are very easy to follow the water film when exposed to wind and other external effects. Slip, and eventually achieve self-cleaning effect. This self-cleaning functional surface is widely used in glass and building walls. Supothina and Nimittrakoolchai [28] examined the superhydrophobic film with the properties of self-cleaning and anti-adhesion using red powder and dust. The coating superhydrophobic was accomplished by the deposition of a polyelectrolyte film on substrate glass.

III. Experimental Device and Method

The experimental device for fabricating the nanostructure of the femtosecond laser is shown in Fig.1, which includes femtosecond laser, electronic switch shutter, neutral absorption attenuator, and focusing lens. The focusing lens is placed on a one-dimensional translation table (z-axis), which can move forward and backward along the direction of laser incidence to control the focusing position D (the distance between lens and target surface). The samples used in the experiment are stainless steel (SS) with dimension (30mm\*30mm) and thickness 1mm. Before and after irradiation of the experiment, the samples were cleaned with ultrasonic for three minutes in deionized water to remove impurities such as dust, oil, and dirt. The sample is fixed on a three-dimensional translation table perpendicular to the direction of laser incidence. The x-axis of the two-dimensional translation table is along the horizontal direction. Its velocity is defined as the scanning speed v, and the y-axis is along the vertical direction. The distance moved when the next scan is completed after each scan is called the scanning distanced. In the experiment, the femtosecond laser pulse is perpendicular to the surface of the stainless-steel target, and the (incidence angle is 0o) to the target. The attenuator is used to control the output laser energy of the femtosecond laser amplification system. The scanning speed v, y, is adjusted by computer controlling x-axis translation table. The scanning distance d is controlled by the y-axis platform, and the focusing position D is controlled by the z-axis translation table. The whole experiment was carried out at a standard atmospheric pressure with a relative humidity of 30% and the indoor ambient temperature of 22oC.



Figure 1: The schematic diagram of the experimental device

## Characterization of the surface wetting characteristics of the micro-nanostructures was performed using a contact angle measuring instrument (as shown in Figure 2).



Figure 2: Contact angle measuring instrument for wetting characterizes testing.

**3.1 Characterization of Wettability Surface**

The spreading ability of water on a solid surface is also the appearance of the nature of the solid itself [29]. The static apparent contact angle (Fig.3) is also used to describe the difference between the advancing contact angle and the receding contact angle (i.e., the contact angle hysteresis) to describe the influence state of the water droplet on the solid surface. When the liquid is in contact with the solid, the contact angle is the angle between the tangential line between the liquid and the gas and the solid-liquid from the intersection of the solid, liquid, and gas three phases; this angle can be used to describe the three interfaces. The state in which the tension is relatively balanced, the smaller the value, the easier the liquid to infiltrate the solid surface, and the degree of influence of the reaction liquid to the solid surface. Usually, a solid surface with a contact angle <90° is called a hydrophilic surface, and when θ<5°, a solid surface is called a super-hydrophilic surface [30, 31]; A structural surface with θ > 90° is generally referred to as a hydrophobic surface. In particular, for a contact angle of 150°, it is called a superhydrophobic surface (or such a surface has superhydrophobic properties).

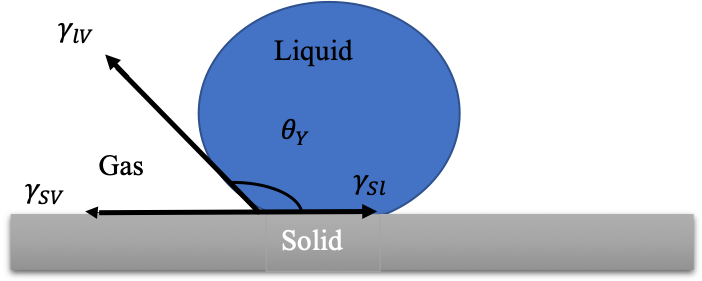


Figure.3. Contact angle diagram

As the research progressed, it was found that the static contact angle of the droplet on some solid surfaces is greater than 150°, but when the solid is erected or reversed, the liquid will still adhere to its surface, and the liquid will not fall off, such as attached to the water droplets on the red rose petals are not easy to roll off [32]. In real life, the dynamic properties of liquids are more difficult in applications such as self-cleaning of windshields and solid surfaces. On top of this understanding, people realized that using the apparent contact angle to adjust the superhydrophobic surface is too narrow, it only considers the static angle singly, and it is more precise to consider the dynamic aspect to define the superhydrophobic surface.

* 1. **Various surface wettability Approaches:**

In 1805, to study the wetting phenomenon of solid surfaces, Thomas Young was formulated for the first time in his report on the premise that the solid surface is smooth (solid composition is uniform, influence smooth and flat), Young equation [29], the equation provides a theoretical basis for the interpretation of the wettability phenomenon of solid structures, placing a foundation for the subsequent development of the field. He believes that when the droplets on the solid surface are balanced, the surface tension between the gas, liquid, and solid interfaces is balanced. From the equilibrium condition of mechanics, Young obtained the equilibrium equation of the droplet in the horizontal direction:

where is the equilibrium contact angle of water droplets on the solid surface in the model he sets, (Intrinsic Angle), 、、 are solid-gas, solid-liquid, liquid-gas interfacial tension.

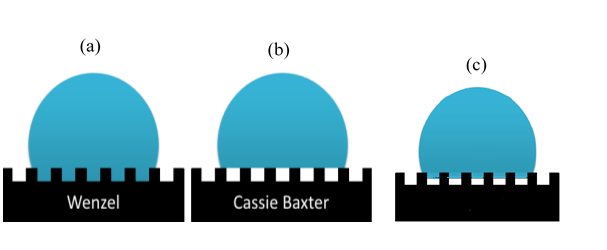
The relationship between roughness and wettability was defined already in 1936 by Wenzel, who stated that adding rough surface­ will increase the wettability caused by the chemistry of the surface [33]. Wenzel considered that the rough structure of the defective solid surface increased the real surface area, which made the actual solid-liquid contact area larger than the apparent geometric contact area. Accordingly, Wenzel proposed the concept of "roughness factor" to modify Young's equation. The contact angle of the ideal solid surface is different from that of the rough solid surface because the rough structure of the surface changes the influence of gas-solid interfacial tension and liquid-solid interfacial tension on the energy of the system. When the liquid is in equilibrium on the rough solid surface, assuming that the droplet fills the groove on the rough surface at any time (Fig.4, (a)), the real contact area of the liquid-solid will be larger than the apparent contact area. In the equilibrium state of constant pressure and temperature, according to the change of free energy and the principle of zero virtual work, Wenzel obtained the following equation:

Fig.4 (a) Wenzel model: (b) Cassie-Baxter model: (c) transition state model

in the above formula is the equilibrium (apparent) contact angle, called Wenzel contact angle; r is the roughness factor of solid surface, which is the ratio between the real contact area of solid liquid on rough surface and the apparent (geometric) contact area, and the real contact area is always larger than the apparent contact area, so the roughness factor is greater than 1(r > 1).C ssie and Baxter in 1944 [34] based on the Wenzel model, assuming a solid surface with uneven microstructure and a uniform distribution of components per unit area, and that the droplets are in a "composite contact" with the solid surface. That is, the air trapped inside the groove prevents the droplet from completely entering the groove, causing the droplet to not completely contact the solid surface, as shown in Fig.4, (b). This causes only the contact between the droplet and the solid to become part of the droplet to contact the rough solid, while the other part is in contact with the air inside the groove. Under the condition of constant temperature and pressure, the droplets are analyzed by thermodynamics. The Cassie-Baxter equation is also obtained according to the change of free energy of the system and the principle of zero virtual work.

Equation (3) shows that the area ratio of solids in the composite interface plays a decisive role in the apparent contact angle, which can reduce the influence of the chemical composition of solids on the contact angle. When is infinitely close to 0, will be infinitely close to - 1, is close to 180o, reaching the state of super-sulfur water, when is infinitely close to 1, that is,is infinitely close to the intrinsic contact angle . When the contact mode of droplets on solid surface conforms to Cassie model, it can be considered that droplets "suspended" on the micro-rough structure of solid surface need to overcome a smaller barrier to roll, and the rolling angle and contact angle lag are relatively small at this time.

V. **RESULTS AND DISCUSSION**

At high energy density, the grooved micro-nanostructures can be written directly by femtosecond laser, while at relatively low laser energy density, the "columnar" micro-nanostructures can be formed by the accumulation of the number of irradiated pulses. Femtosecond laser microstructures were fabricated using the devices and methods shown in section 3. The difference is that the scanning spacing is set in a range smaller than the size of the focusing spot, to obtain uniform irradiation to form self-assembled microstructures. The surface morphology of stainless steel was observed and analyzed by scanning electron microscopy (SEM) with incident different laser energy(E), scanning speed V=1 mm/s, scanning spacing d=0.04 mm, and focusing position 17mm, femtosecond laser. As shown in Fig.5, with the increase of energy, micron pit roughness appears randomly on the surface (Fig. 5 (a)), then micron bumps gradually form, and its size increases (Fig. 5 (b) - (i).



**Figure 5:** SEM images of samples fabricated micro and nanostructure on Stainless Steel surface, with scanning speed V=1 mm/s , scanning spacing d=0.04 mm, and focusing position 17mm, at incident different laser energy (E): (a) 0.4mJ, (b) 0.6mJ, (c) 0.8mJ, (d) 1.0mJ, (e) 1.2mJ, (f) 1.4mJ, (g) 1.6mJ, (h) 1.8mJ and (i) 2.0mJ, and their corresponding contact angles.

Fig.6, shows after the samples were placed in the air for one week, the samples were tested for wettability, and it was found that the wettability a little change significantly; after one month, there was still a little change subsequently, the sample was washed with ultrasonic in similar literature, and naturally dried at room temperature, and the hydrophilic properties of the surface of the sample were not changed, and the phenomenon of transition from wettability to hydrophobic properties as described in the literature was not observed.



Fig 6. A sample is in the air contact angle a little changing (a) first day, (b) one week, and (c) one month.

* 1. Surface wettability of micro-nano structure after silanization
     1. Surface silanization process

Rinse the sample with ultrasonic in deionized water to clean away the impurities inhaled during the air. Mix the perfluorodecyltriethoxysilane with absolute ethanol and shake it in a beaker for one hour to make a mass fraction. It is a 1% solution of perfluorodecyltriethoxysilane in ethanol, followed by silanization of the sample to reduce the surface energy of the sample. The sample is placed in the prepared solution for four hours, and finally, the soaked sample is placed bake in an oven at 100oC for 1 hour and cool in the oven.

4.1.1 Characterization of Surface Wettability after Silanization

The wettability of the samples without laser processing before and after silanization to reduce the surface energy is shown in Fig.7. The contact angle without laser processing is 57.3o and after laser processing, before silanization is 6.8o, and after silanization, the contact angle increases to 126.1o.The wettability of the stainless steel (SS) surface has changed significantly due to silanization treatment.



**Figure 7:** Contact angle without laser processing (a) and change of contact angle before silanization (b) and after silanization (c) of the original surface of SS metal, (laser energy 0.4mJ, scanning speed 1mm/s, scanning distance 0.04 mm).

Fig.8. shows the SEM images of bulk samples fabricated at different energy. The illustration at the upper right corner shows the contact angles measured after their silanization reduces the surface energy. With the increase of energy, the contact angles have different degrees, in our paper when E= 2.0mJ, the best contact angle is148.6o. After silanization, the surface of microstructures fabricated in femtosecond laser changed from hydrophilic to hydrophobic. This shows that after surface energy is repaired, femtosecond laser fabricated micro and nanostructures make the surface hydrophobic.



Fig.8. SEM image of sample fabricated Contact Angle after silanization on stainless steel surface with scanning speed 1mm/s, and scanning spacing 0.04mm, focusing positions 17mm at different energy (a) 0.4mJ, (b) 0.6mJ, (c) 0.8mJ, (d) 1.0mJ, (e) 1.2mJ, (f) 1.4mJ, (g) 1.6mJ, (h) 1.8mJ and (i) 2.0mJ.

Through the experimental results, we found that the columnar microstructure has the most obvious improvement in high hydrophobicity (Fig.8, (i)). The formation of the columnar microstructure is closely related to the laser irradiation. In terms of hydrophobic properties, the 9 samples showed better contact angles, about 148.6o, approaching superhydrophobic properties. It also shows that the optimized columnar surface morphology is more conducive to the improvement of hydrophobicity.

# VI. CONCLUSION

In this paper, the preparation of two typical micro and nanostructures were fabricated on the metal surface by femtosecond laser, and the hydrophilicity and hydrophobicity of the surface were studied. The following research results were obtained. In the aspect of hydrophobicity, femtosecond laser column micro and nanostructures were fabricated on the stainless-steel surface. The contact angle measurements showed that the results of femtosecond laser fabrication of superhydrophobic metal surfaces were different from those reported in the literature. We found that the fabricated microstructural surfaces were placed in air for a long time (7-30 days) without hydrophilic orientation natural transformation of hydrophobic properties. The hydrophobic function of the surface can be obtained by silanization of the prepared micro-and nanostructure surface (the best contact angle is 148.6o). This indicates that the preparation of metal microstructures superhydrophobic surface by femtosecond laser directly (no low-energy materials are added after structure formation) requires a certain base material premise. This discovery has certain application significance for the development of preparation technology of wettability microstructures.

Due to the limited research time of the paper, the research on the mechanism of the superhydrophobic micro-nano surface can be directly prepared for femtosecond laser. The stability of surface function is also the focus of future research. With the deepening of the research on the formation mechanism of surface wetting characteristics, it is believed that this technology will play an important role in the field of solar self-cleaning surface and aviation anti-icing.

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