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Nano Thin Aluminium Coating as Superhydrophobic Surface: Fabrication by RF/DC Magnetron Sputtering and its Characterisation

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Abstract: *The hierarchical structured surface of the lotus (Nelumbo nucifera) leaf provides a model for the development of bio mimetic self-cleaning surfaces. On these water-repellent surfaces, water droplets move easily at a low inclination of the leaf and collect dirt particles adhering to the leaf surface. These surfaces are called as super hydrophobic surfaces. Inspired by this "lotus-leaf effect", lot of techniques have been developed to create super-hydrophobic surfaces. Here, we report the development of super hydrophobic surfaces on a glass substrate by coating a thin layer (thickness ~2 nm) of Aluminium by RF/DC magnetron sputtering. The super hydrophobic phenomena is further enhanced by coating a layer of metallic stearates (Zinc stearate, Copper stearate), nano particles (copper oxide) suspended in a polymer matrix like polyvinyl chloride in tetrahydrofuran. Nano structured aluminium after sputter deposition has been explored by AFM, SEM, XRD studies. AFM data and SEM image reveals the existence of nano particles in the order of 5 – 10 nm. Hydrophobic nature of the surfaces is confirmed from the contact angle measurement made from Goniometric studies. Step by step enhancement in contact angle is noted at various stages. Over a plain glass substrate, water shows a contact angle of 28^o but after coating with a nano Aluminium layer of 2 nm thickness, the contact angle rises to 98^o. Modification of surfaces (nano roughness) have been performed by coating a thin layer of metallic stearate in polymer matrix and nano particles impregnated polymer suspension containing metallic stearate using spin coater. Such modified surfaces show improved contact angles (above 137^o).*

Keywords: *Superhydrophobic surface; Nano aluminium film; metallic stearate; Copper oxide nano particles; Sputtering; contact angle.*

1. INTRODUCTION

The design and fabrication of micro and nano structured surfaces have received considerable interest in recent year [1]. Since it possess a very important property called super hydrophobic. Superhydrophobic surfaces are also considered as self cleaning surfaces [2]. Inspired by the phenomenon observed in some plant leaves and insects [3, 4, 5] superhydrophobic surfaces whose contact angles (CA) with water are higher the 150° [6] have significant application towards corrosion prevention. Since water is mainly responsible for corrosion problems on most of materials especially on steel structures. These materials which mimic this superhydrophobic property on coating may suppress the role of corrosion.

Therefore the aim of scientists is to create a coating with a surface morphology that mimics morphology of the lotus leaf. In order to fabricate such superhydrophobic surface, the physical parameter utilised to assess its water repellent phenomena is its CA towards water. Generally, for fabrication of superhydrophobic surface or film, it is necessary to create a rough surface on hydrophobic surface [7] (CA>90°) and to lower the surface energy by chemical modification such as coating with fluoroalkylsilanes [8-13] or thiols. Many researches modified the substrates surface by variety of techniques like electrochemical process, wet etching [14], casting polymer solution on surfaces, nanocasting, lithography technique [15], etc., Also plasma techniques have been used to modify the surface chemistry of substrates.

In this present study, superhydrophobic nature on substrates by developing nano coating using RF-DC sputtering technique. Further, step by step enhancement in contact angle is achieved by coating different materials like metallic stearate, nano particles along with polymer matrix over nano film coated surface.

2. EXPERIMENTAL

2.1 MATERIALS AND METHODS

2.1.1 Synthesis of Copper oxide

Copper oxide nano particles were prepared by polyol method [16]. After isolating the copper metal nanoparticles it was subjected to atmospheric oxidation in order to get the oxide particles.

2.1.2 Fabrication of Superhydrophobic film

Step 1: Nano aluminium thin film has been developed on glass substrate by sputtering technique.
Step 2: Coating of PVC thin film over nano aluminium coating by spin coater
Step 3: PVC in THF & metallic stearate has been mixed in 1.5:1 ratio. A thin film of this combination is coated over aluminium layer and dried.
Step 4: PVC in THF, metallic stearate and copper oxide nanoparticle in 1.5:1:1 ratio is mixed well in an ultrasonicator (probe type) sonics. The thin film is developed over nano aluminium coating in spin coater at a speed of 1800 rpm.

2.2 CHARACTERISATION TECHNIQUES

2.2.1 FTIR analysis
FTIR Spectra for Copper stearate, Zinc stearate, etc., was recorded in solid mode using a modern type Bruker optic GmbH-Alpha spectrometer, Germany.

2.2.2 Sputtering technique
Nano aluminium film was coated over glass substrate using planar magnetron RF-DC sputtering unit, Model-12'' MSPT.

2.2.3 XRD
The crystallographic structure, chemical composition and particle size for nano aluminium thin film and copper oxide nanoparticle was recorded using X-ray diffraction technique.

2.2.4 Surface morphology
Surface morphology was analysed using Atomic force microscopy in non contact mode using XE 70, SPM, Park system of scan area of 10µm.

2.3.5 Goniometer
Contact angle measurements of water drops on various thin films were made with a Rame-Hart contact angle goniometer supplied by M/s GBX, France

3. RESULTS & DISCUSSION

3.1 FTIR SPECTRA
Metallic stearates like Copper stearate, Aluminium stearate, Zinc stearate etc., used in this work was prepared by employing literature methods [17]. The obtained Zinc stearate has been confirmed from their IR spectra [Fig.1.b]. Pure stearic acid shows a characteristic absorption band at 3460 cm^{-1}[Fig.1.a] attributable to presence of carboxylic acid -OH group frequency. The disappearance of this band is noted in the metallic stearate [17] confirms the formation of metallic soap.

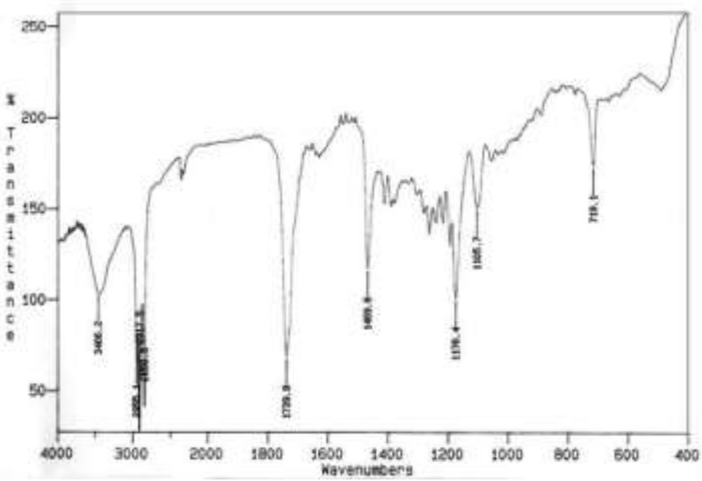

Fig. 1.a. IR spectra of Stearic acid

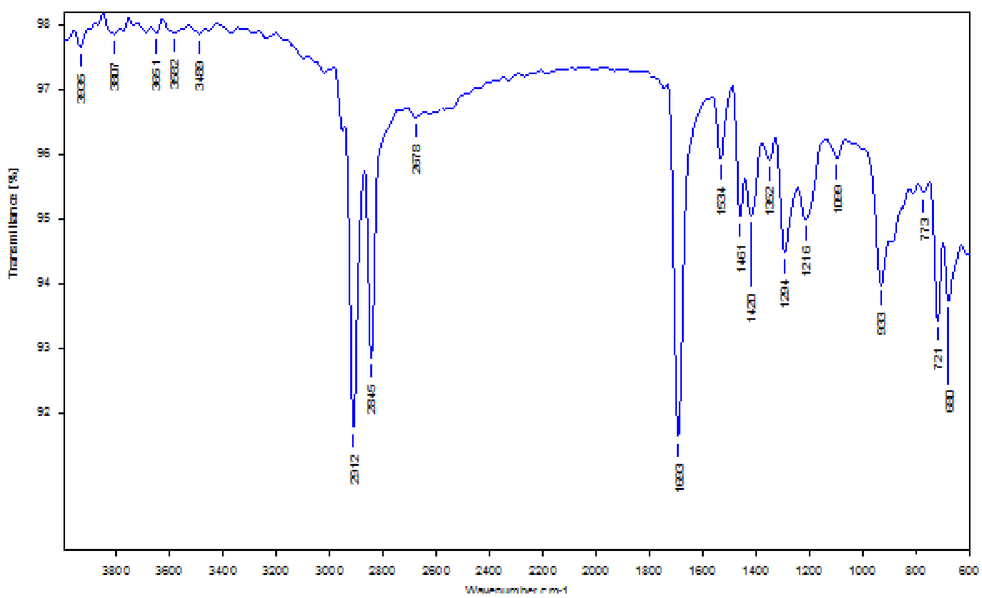

Fig.1.b.IR spectra of Zinc stearate

3.2 XRD ANALYSIS

3.2.1 XRD of Nano Aluminium film

Nano thin Aluminium film (thickness is about 2nm) was coated over glass substrate by using RF-DC magnetron sputtering. The structural features of this film are explored from XRD data [18][19]. XRD of aluminium film is shows in fig.2.a. The broad absorption is due to the dominance of glass matrix [20] over the nano coating. And also from the line profile data (FWHM), the particle size has been evaluated using Scherrer equation & found in the range of 3nm.

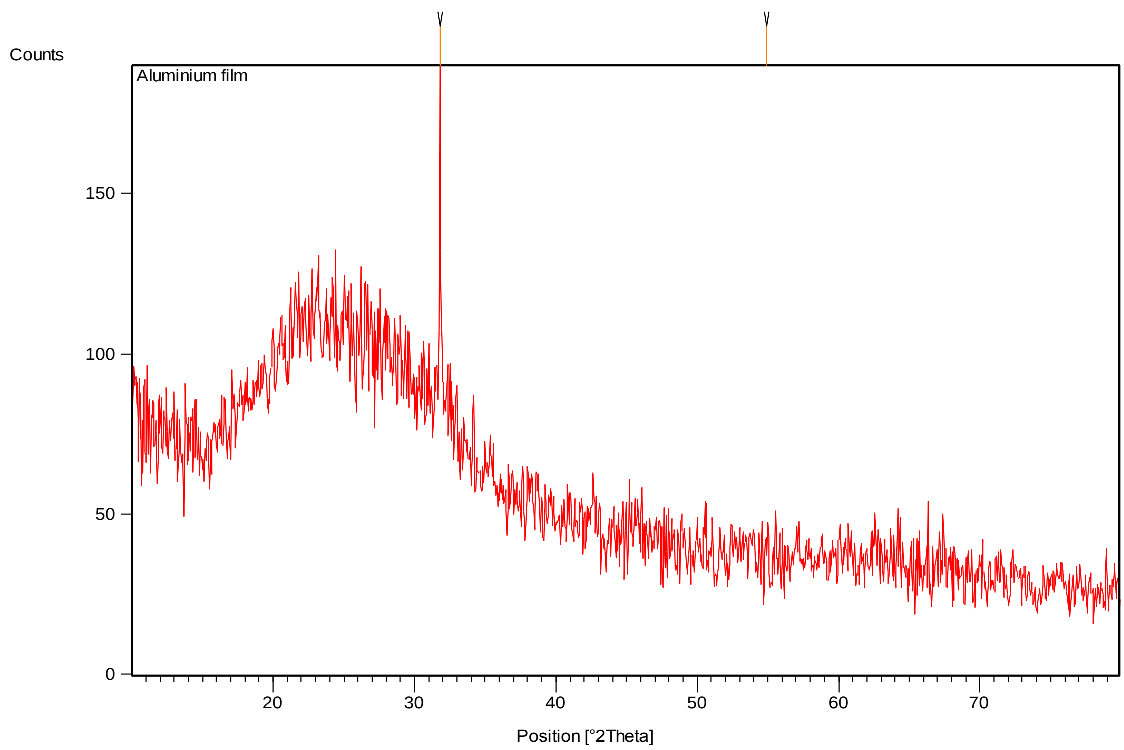

Fig. 2.a. XRD pattern of nano Aluminium thin film

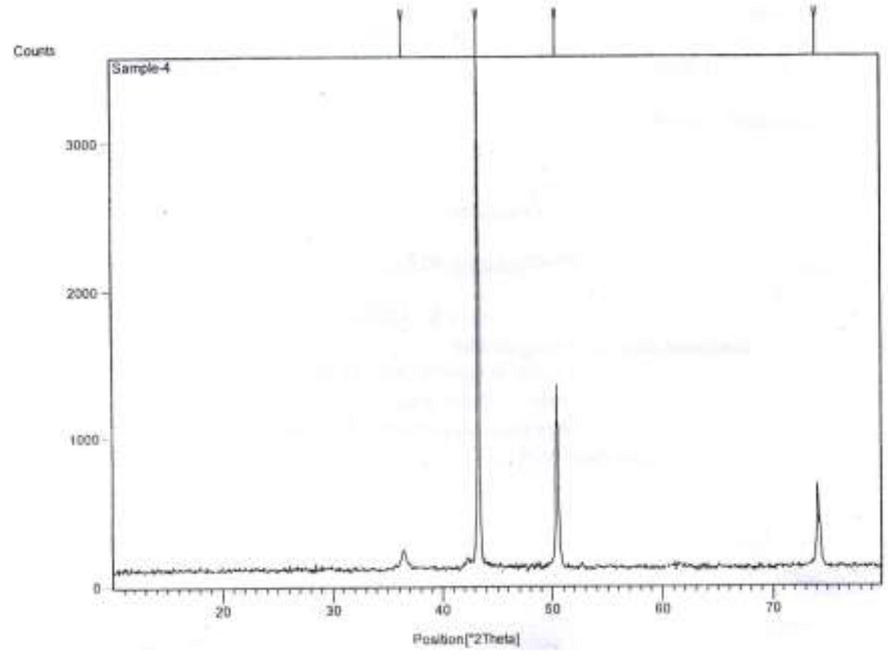

Fig. 2.b. XRD pattern of Copper oxide nano particles

3.2.2 XRD of Copper oxide

Copper oxide nano particles used in this work has been synthesised by polyol method [16] followed by atmospheric oxidation. The XRD of CuO nano particle is shown in the fig.2.b. The observed XRD pattern is well in accordance with the published results [16]. Particle size of the nano particle is also calculated by using Scherrer equation and it is in the range of 36nm.

3.3 SURFACE MORPHOLOGY HYDROPHOBIC COATING (AFM IMAGE)

Super hydrophobic character developed on because of the glass substrate is because of the nano (2nm thick) Aluminium film by RF-DC magnetron sputtering. The nano textured structure is well explored by imaging the surface through the AFM image [Fig.3.b]. AFM image of the Aluminium film (2D) is given in Fig.3.a. From this image and the line drawn horizontally at 2.2µm, the particle size distribution existing along the line found is uniform. It is also evidenced from the histogram represented in Fig.3.c. The line profile reveals the particle size present in the film is around 2-5nm which is represented as Fig.3.d.

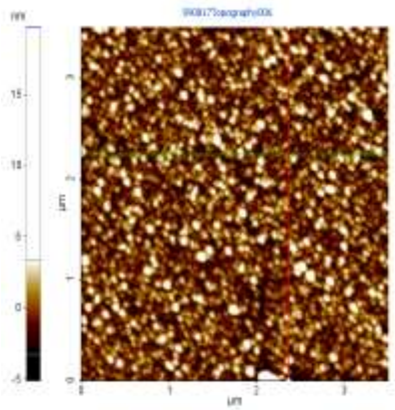

Fig. 3.a. 2D Image of the nano aluminium film

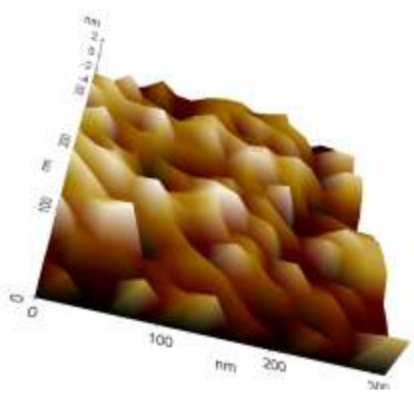

Fig.3.b. 3D Image of nano aluminium film

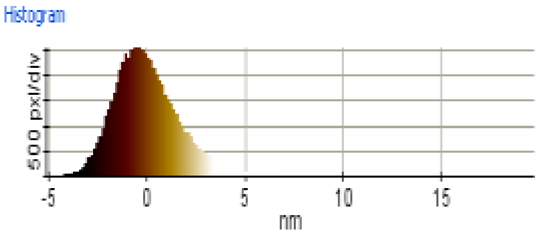

Fig.3.c. Histogram

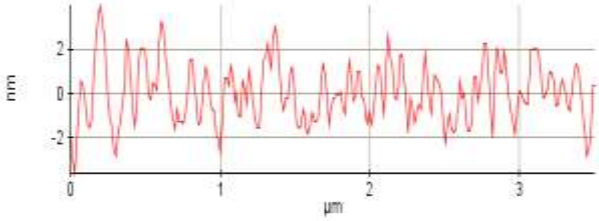

Fig.3.d. Line profile

Figure 3: Surface morphology of nano aluminium thin film performed in AFM-NC mode.

3.5 CONTACT ANGLE MEASUREMENTS

Hydrophobic nature developed on glass substrate is assessed based on their contact angle with reference to water measured using goniometer.

Normally, the glass substrate is hydrophilic since it contains alumina, silicate, etc., constituents. The base surface shows a poor contact angle, i.e., 27.4° on placing a 6µl of water. Fig.4.a. represents this situation. The interaction between water molecules and the hydrophilic constituents present in the glass substrate is prevented by introducing a barrier in the form of nano aluminium film. The contact angle measurement over thin nano aluminium film shows a contact angle of 96° [Fig-4.b] supports the inhibition of hydrophilic groups present in the glass.

Enhancement in the contact angle is further evidenced by developing different layers over Aluminium layer in a stepwise manner. On Aluminium film, polyvinylchloride thin film in THF is coated as first layer and the contact angle found is 101.6° [Fig-4.c.]. Metallic stearate along with PVC is coated on nano aluminium coating and the contact angle found in this film is 133.6° [Fig-4.d]. Among the other coatings developed so far, nano particle (Copper oxide) containing suspension (which also contains PVC & metallic stearate) produces a thin film with higher contact angle 137.5° [Fig.4.e]. All these data reveals the fact that superhydrophobic character has been enhanced.

Fig.4.a. Contact of water with glass substrate. CA= 27.4°

Fig.4.b. Contact of water with nano aluminium film. CA= 96.4°

Fig.4.c. Contact of water over a thin film of PVC coated on Al film. CA= 101.6°

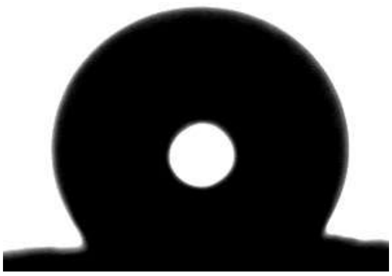

Fig.4.d. Contact of water over a thin film of PVC containing metallic stearate coated on Al film.CA= 133.6°

Fig.4.e. Contact of water over a thin film of PVC containing metallic stearate & nano copper oxide on Al film. CA= 137.5°

Figure 4: Contact angle measurements of different thin layers over nano aluminium film.

4. CONCLUSION

A simple path to prepare a surface with superhydrophobic properties has been achieved in which the nano aluminium films were deposited on glass substrate using a direct current magnetron sputtering system. On Aluminium thin film, different layers containing different materials have been fabricated by spin coater. Contact angle measurements on these layers shows step by step enhancement in contact angle from 27° to 137°. Based on these results, superhydrophobic surfaces on cemented wall and different substrate can be developed by nano formulated paints with these compositions and thereby self cleaning action can be achieved.

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