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DEVELOPING HIGH-TEMPERATURE WATER-REPELLENT GLASS FIBER CLOTHS THROUGH ATOMIC LAYER DEPOSITION

Mohammad Reza Shaeri[1]*, Dongsheng Guan[2], Fangyu Cao[1], Chris Yingchun Yuan[3]

[1]Advanced Cooling Technologies, Inc., 1046 New Holland Avenue, Lancaster, PA 17601, USA
[2]University of Wisconsin-Milwaukee, 3200 N Cramer Street, Milwaukee, WI 53211, USA
[3]Case Western Reserve University, 10900 Euclid Avenue, Cleveland, OH 44106, USA

ABSTRACT

Atomic layer deposition (ALD) was used to develop hydrophobic and water-repellent glass fiber cloths for operation in high temperatures up to 200°C. The cloths were deposited with highly conformal, pinhole-free, and uniform Al_2O_3 films with a thickness at 200 nm. The characteristics of the deposited films were studied using a scanning electron microscope. While the uncoated cloth was hydrophilic with an equilibrium contact angle around 40°, the ALD-coated cloth became hydrophobic with an equilibrium contact angle around 125°. Such a hydrophobicity resulted in an excellent water repellant characteristic, as such, while non-coated cloth absorbed the water immediately, the ALD-coated cloth showed a resistance against water transport into the nanoscale-coated fiber cloth for over 10 hours. This characteristic was maintained in the ALD-coated cloth after thermal cycling processes by heating the coated cloth up to 200°C for one hour. The present study would open a new research direction to leverage ALD for developing water-repellent glass fiber cloths in high-temperature applications, such as those for developing high-temperature wire insulations.

KEY WORDS: Atomic layer deposition, Glass fiber cloth, Hydrophobic, Water-repellent, Thermal cycling.

1. INTRODUCTION

Atomic layer deposition (ALD) is a cyclic process in which a binary reaction $a + b \rightarrow c + d$ is split into two self-limiting surface reactions by injection of individual gaseous precursors a and b, alternately, into the reactor with a purge between them. Each ALD cycle includes four steps, as: (i) forming a new layer on the substrate due to the surface reactions at the first precursor exposure, (ii) purging the reactor by an inert gas, (iii) deposition of the desired film due to the surface reactions between the second precursor and the adsorbed species on the substrate, and (iv) purging the reactor by an inert gas. The purge gas is also used as the carrier gas to transport the precursors to the substrate. Purging the reactor is a crucial step in preventing interactions between the two precursors since their gaseous reactions adversely affect the uniformity of the deposited films. The self-limiting characteristic of surface reactions results in excellent advantages such as highly conformal, pinhole-free, and uniform nanoscale films, as well as growth rates proportional only to the number of ALD cycles. In fact, since the deposition rate is the same among all the ALD cycles in a given ALD process, the desired film thickness is controlled precisely only by appropriately repeating the number of ALD cycles [1-5]. While the semiconductor processing has been widely recognized as the main application of ALD, the significant advantages achieved by ALD have resulted in substantial attentions to leverage this unrivalled nanotechnology in other applications. In this study, ALD is used to develop hydrophobic and water-repellent glass fiber cloths, which have a potential application in wire insulations, in order to prevent the water transport into the conductor and, in turn, avoid a short circuit.

*Corresponding Author: mohammadreza.shaeri@1-act.com

2. RESULTS

To deposit a metal oxide film by an ALD process, one precursor is used as the metal source and another as the oxidant. In this study, Al_2O_3 films were deposited using trimethylaluminium ($Al(CH_3)_3$, TMA) and H_2O as the metal and oxidation precursors, respectively. Nitrogen was used as both the purge and carrier gases. The deposition process was conducted in a Savannah 100 ALD system. Two typical self-limiting chemical reactions are involved in the deposition process and can be described by [6]:

$$AlOH^* + Al(CH_3)_3 \longrightarrow AlOAl(CH_3)_2^* + CH_4 \qquad \text{(i)}$$
$$AlCH_3^* + H_2O \longrightarrow AlOH^* + CH_4 \qquad \text{(ii)}$$

where the asterisks stand for the surface species, and the remaining species are gases. The overall reaction is:

$$2Al(CH_3)_3 + 3\,H_2O \longrightarrow Al_2O_3 + 6CH_4 \qquad \text{(iii)}$$

Al_2O_3 films were deposited on commercially available glass fiber cloths in two different geometries: one type of cloth was flat, and another type was sleeving (cylindrical), as illustrated in Fig. 1. The ALD-coated zone for the flat cloth was a square with an edge length of 9 cm, and for the sleeving fiber cloth, was a length of 9 cm. The entire outer surface of the sleeving cloth, as well as both sides of flat cloth, were deposited by Al_2O_3. When ALD began, vapors of TMA and H_2O were alternately carried by continuous nitrogen gas at a flow rate of 20 sccm into a sealed chamber where the temperature was 200°C to heat up the glass fiber cloth. The program sequence was: (i) dose H_2O for 0.015 s to produce a hydroxyl functionalized surface of cloth, (ii) purge the reactor for 8 s, (iii) dose TMA for 0.015 s for chemical reactions, and (iv) purge the reactor for 8 s. The growth rate of Al_2O_3 was 0.1 nm/cycle; therefore, the sequence was repeated 2000 times to obtain amorphous alumina coatings with a thickness of 200 nm. Fig. 1 illustrates the scanning electron microscopy (SEM) images of both uncoated and ALD-coated sleeving cloths.

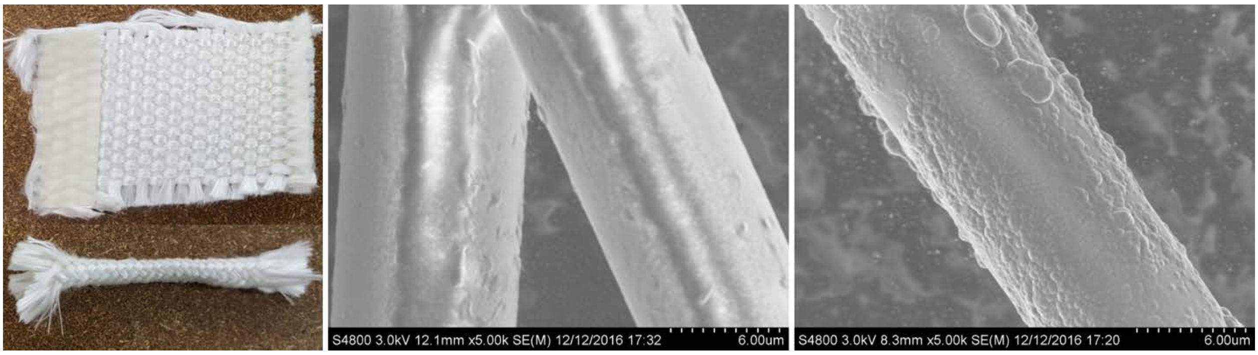

Fig. 1. [Left]: flat and sleeving glass fiber cloths at top and bottom of the picture, respectively. [Middle]: SEM image of uncoated cloth. [Right]: SEM image of ALD-coated cloth with 200 nm Al_2O_3 films.

Fig. 2 illustrates the water droplet formation on both uncoated and ALD-coated cloths. The yellow tape around the ALD-coated cloth was put only to avoid the fiber cloth becoming frayed during the experiments. Basically, for the equilibrium contact angles (θ) smaller and larger than 90°, the surface is called hydrophilic, and hydrophobic, respectively. If $\theta \geq 150°$, the surface is superhydrophobic and has a substantial water repellent characteristics [7]. Based on Fig. 2, while the uncoated cloth is hydrophilic with $\theta \approx 40°$, the ALD-coated cloth with Al_2O_3 deposited films became hydrophobic with $\theta \approx 125°$ that is very close to a superhydrophobic surface. Such a hydrophobicity potentially results in a highly water resistant cloth. To assess this characteristic, wicking tests were performed by putting the samples in a dye water and tracking the dye water transport across the cloths at different times, as illustrated in Fig. 3. While the dye water immediately penetrated into the uncoated cloth in less than 8 s, the ALD-coated cloth showed a durable resistance against water penetration for around 10 hours. The excellent water-repellent characteristic of the ALD-coated cloth indicates the pinhole-free deposited Al_2O_3 films that filled all the pores on the surface of the cloth, otherwise dye water penetrated into the cloth.

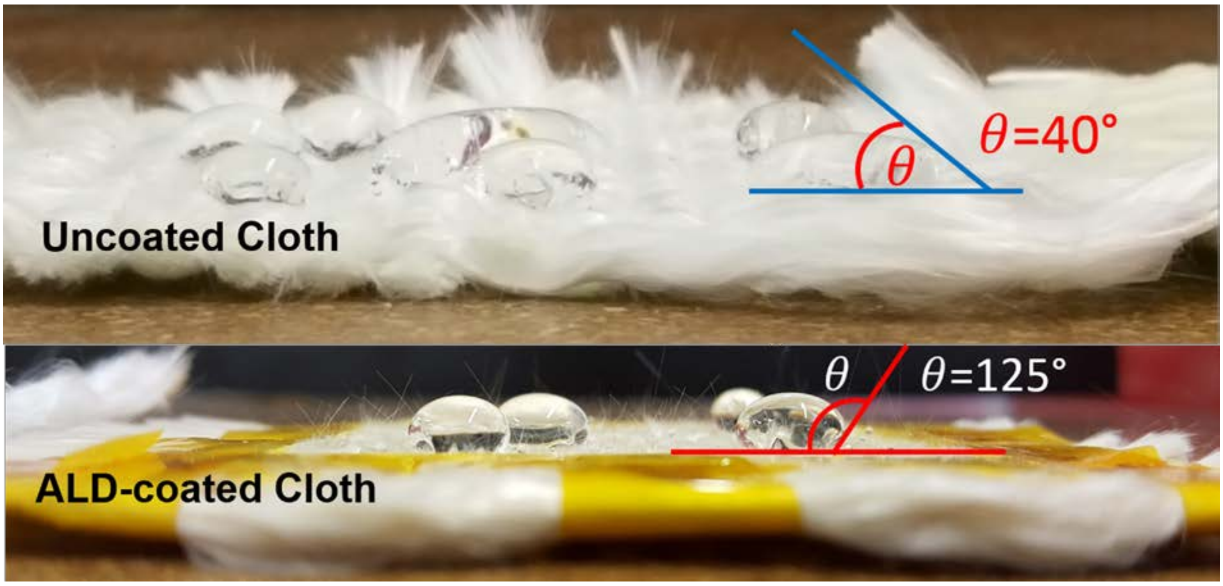

Fig. 2. Water droplet formation on the uncoated and ALD-coated fiber cloths.

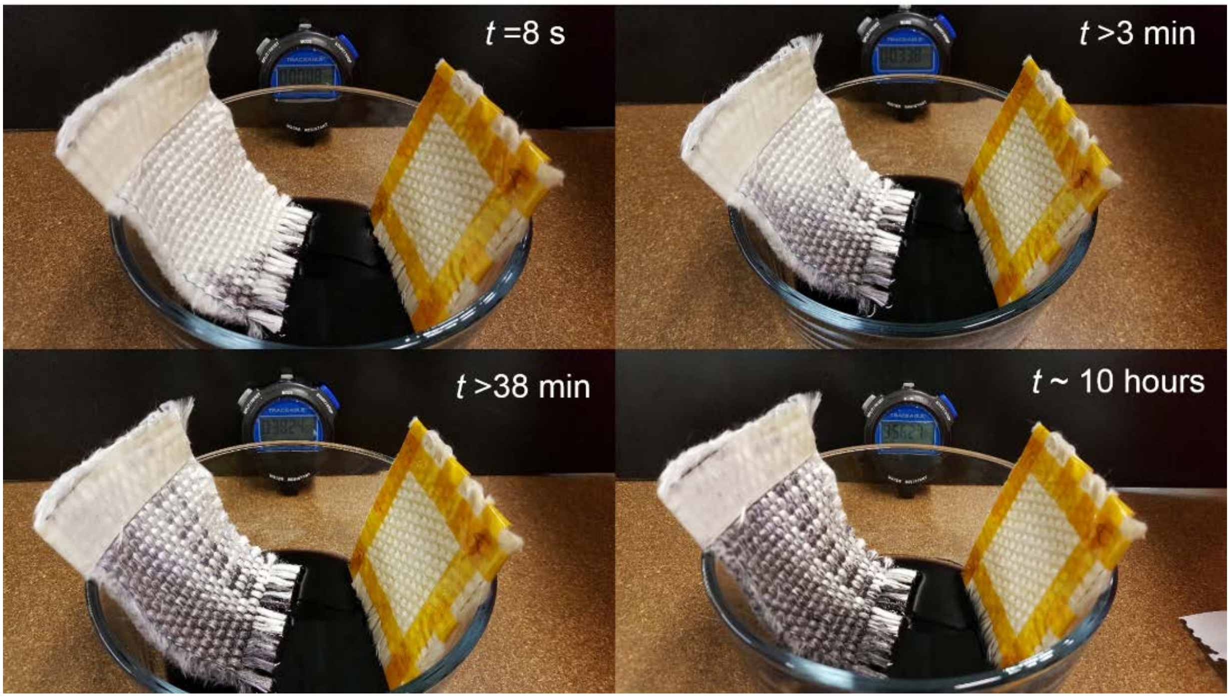

Fig. 3. Wicking test using a dye water at different times. Times (indicated by *t*) are shown at the top right of individual picture. The cloths at the left and right sides of the bowl correspond to the uncoated and ALD-coated cloths, respectively.

To evaluate the endurance of the characteristics of the coating in high temperatures, which is very important in wire insulation applications, wicking tests were performed after thermal shock, as well. For this purpose, the ALD-coated cloth was heated up to 200°C for one hour in an oven, it was then quickly cooled down to room temperature. Fig. 4 illustrates the wicking test after thermal shock. The excellent and durable water-repellent characteristic of ALD-coated cloth after a thermal shock is clear since dye water did not penetrate into the ALD-coated cloth after a 7.5-hour wicking test. Note that since the ALD-coated cloth was heated up in the oven, the yellow tape from the edge of the cloth (observed in Figs. 2 and 3) decomposed.

Fig. 4. Wicking test after heating the ALD-coated fiber cloth up to 200°C for one hour. The wicking test was performed for over 7.5 hours.

3. CONCLUSIONS

Durable hydrophobic and water-repellent glass fiber cloths for operations in high temperatures up to 200°C were developed through ALD of Al_2O_3 on fiber cloths. The uniform and pinhole-free deposited films inside irregular pores of the cloth were demonstrated through wicking tests. The present study is the first attempt to introduce ALD as a promising nanoscale coating technology to develop hydrophobic and water-repellent fiber cloths for high temperature applications, like those in wire insulations.

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