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Preparation of polytetrafluoroethylene by pulsed electron ablation: Deposition and wettability aspects

Redhouane Henda [a,*], Graeme Wilson [a], Joy Gray-Munro [b], Omar Alshekhli [a], Andrew M. McDonald [c]

[a] School of Engineering, Laurentian University, Sudbury, Canada
[b] Dept. Chemistry and Biochemistry, Laurentian University, Sudbury, Canada
[c] Dept. Earth Sciences, Laurentian University, Sudbury, Canada

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ABSTRACT

Polytetrafluoroethylene (PTFE) has been prepared by pulsed electron deposition technique on glass and silicon substrates. Deposition of the thin films has been carried out in the temperature range from room temperature to 300 °C, pressure range from 133.32×10^{-3} Pa to 799.93×10^{-3} Pa, and discharge voltages between 10 kV and 16 kV. Argon or nitrogen has been used as a background gas during the deposition of the films. Attenuated Total Reflection Fourier Transform Infrared spectroscopy shows absorption peaks in the films at 644 cm^{-1}, 1154 cm^{-1} and 1210 cm^{-1} consistent with those of PTFE target material. Atomic force microscopy and spectroscopic reflectometry reveal the clustered nature of the films and other morphological characteristics. Surface wettability of the films, expressed via the contact angle, has been measured via static angle goniometry. PTFE films increase the contact angle from about 32° (bare glass) and 43° (bare silicon) to up to 90° and 110° for PTFE-coated glass and silicon substrates, respectively. The contact angle decreases with an increase in both pressure and temperature, while it increases then decreases as the discharge voltage increases.
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1. Introduction

Polytetrafluoroethylene ('Teflon') possesses a combination of technologically attractive properties including good corrosion resistance, biological compatibility, low dielectric constant, low friction coefficient, high chemical and thermal stability, good mechanical strength, and excellent plasticity. This set of properties has attracted considerable attention to polytetrafluoroethylene (PTFE) for applications ranging from lubrication to molecular electronics [1–3]. PTFE thin films have been prepared by many techniques such as vacuum evaporation [4], radio frequency sputtering [5], plasma evaporation [6], ion beam sputtering [7], and pulsed laser deposition (PLD) [8–16]. Many problems exist with these techniques specifically in the area of controlling the structure/composition of the deposited films. Conventional wet processes such as spin-coating methods are not applicable to the formation of thin PTFE films as the latter exhibit poor solubility in all solvents and have low surface adhesion.

Pulsed electron deposition (PED) has been a promising alternative to PLD in thin film coating applications [17, 18]. PED is conceptually similar to PLD except that a short pulse (~100 ns) of energetic electrons replaces a short pulse of photons to ablate material from a target. Due to a short penetration depth (~1 μm) of the electron beam into the target, rapid non-equilibrium heating results, leading to the formation of a highly forward directed, stoichiometric plasma plume, which in turn facilitates stoichiometric preservation of the target composition in the deposited film under optimal process conditions. Compared to lasers with similar power densities (~10^8 W/cm^2), advantages of electron beam sources include higher electrical efficiency (30%), the ability to process materials that are transparent or highly reflective to laser light, and lower capital costs [18]. Nearly all solid-state materials with varying complexity can be deposited in thin film form with PED. The pressure domain for thin film deposition in PED is restricted relatively to PLD, due to the narrow working pressure range of the high voltage abnormal discharge. Recently, Chandra and Manoharan [19] have investigated the fabrication of PTFE films by PED under argon as the background gas. Thin films have been obtained under a constant gas pressure of 666.61×10^{-3} Pa, room temperature, and constant discharge voltage.

In this study, we report on the preparation of PTFE by channel-spark PED technique on glass and silicon substrates. PTFE thin films have been deposited under a wide range of process conditions, namely, background gas, temperature, pressure, and discharge voltage. The chemical composition and wetting properties of the deposited films are investigated.

2. Experimental details

The Polytetrafluoroethylene thin films were deposited in a pulsed electron ablation system, PEBS-20 model with pulse duration of 100 ns (Neocera, Inc.). A simplified schematic illustrating the main

* Corresponding author. Tel.: +1 705 675 1151; fax: +1 705 675 4862.
E-mail address: rhenda@laurentian.ca (R. Henda).

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features of the PED system is shown in Fig. 1. The pulsed electron beam source based on channel-spark discharge geometry consists of a trigger, a hollow cathode, and a dielectric capillary tube. A two-inch diameter PTFE rod (Small Parts, Inc.) was cut into ¼ — inch thick targets, and glass microscope slides (Labcraft) were cut into ½×½ inch^2 substrates. Both substrate and target were sonically cleaned at 50 °C prior to deposition. The substrate was further rinsed with acetone followed with methanol, while the target was cleaned in methanol. The electron pulse energy was varied from 10 to16 kV with fixed pulse frequency of 1 Hz and a constant number of 600 electron pulses throughout the experiments. Prior to film deposition experiments the deposition chamber was evacuated to 6.6×10^{-4} Pa before admitting the background gas into the chamber at a specific pressure for deposition. The gas pressure was varied from 133.32×10^{-3} Pa to 799.93×10^{-3} Pa. The distance between the target and electron gun ceramic tube tip was kept at 15 mm whereas the target to substrate distance was kept at 80 mm. Prior to deposition the target was pre-ablated by the electron beam over 1200 pulses at 1 Hz and 10 kV. The operating conditions of PED system were varied to determine their effect on the wettability of the film produced.

Subsequently, the deposited films were characterized using Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) spectroscopy. The spectra were collected using Hyperion infrared microscope (Bruker Optics) with a liquid nitrogen cooled mercury cadmium telluride detector and an ATR objective equipped with a germanium crystal. Each spectrum is the result of 100 scans collected at a resolution of 4 cm^{-1}. All spectra were collected with an air background and were corrected for CO_2 and H_2O with the atmospheric compensation function of the software. The PTFE film spectra are presented after subtraction of the spectrum for a bare glass substrate. Static angle goniometry (Model PG2, Fibro Systems AB, Sweden) was used to assess film hydrophobicity by measuring the contact angle between a drop of de-ionized water and the film surface under various process conditions. Single water drops were photographed using the goniometer built-in digital camera. Water droplets of volume around 4 μL were used in these measurements. The 2 μm×2 μm atomic force microscopy (AFM) images were taken with a Nanoscope IIIa scanning probe microscope (Veeco, USA) controller operated in tapping mode. Veeco TESPA tapping mode tips with resonant frequency and force constant of approximately 320 kHz and 42 N/m were used. The "E" scanner head with a maximum scan range of 25 μm was used with a scan rate of 0.5–1.0 Hz. The raw data were flattened using the instrument software and all image height scales were set to 25 nm. Film thickness and roughness have been obtained via reflectance spectroscopy in the visible range (M-Probe series, Semiconfsoft, USA), where experimental data were fitted by means of a modified Marquardt–Levenberg minimization using the reflectometer software.

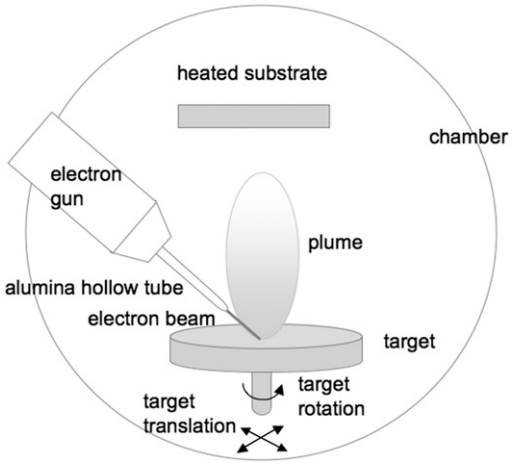

Fig. 1. Simplified schematic diagram of PED system.

3. Results and discussion

The chemical composition of the deposited thin films was characterized by ATR-FTIR. Fig. 2.a shows the FTIR spectrum of the PTFE target material. This spectrum has two strong vibrational bands at approximately 1154 cm^{-1} and 1210 cm^{-1} that are ascribable to the -CF_2- symmetric and asymmetric stretching vibrations of the PTFE molecule [20–22]. A third weaker peak assigned to the -CF_2- wagging of the PTFE molecule is also observed at approximately 644 cm^{-1}. Fig. 2.b shows the FTIR spectra for three different spots on PTFE films deposited on a glass substrate. The negative peaks at approximately 953 cm^{-1} and 776 cm^{-1} are due to the underlying substrate. The fact that the intensity of the substrate bands is negative upon subtraction of the substrate confirms the presence of a thin film at the surface. Upon comparison to the PTFE target material, the deposited film also has two absorption bands at approximately 1154 cm^{-1} and 1210 cm^{-1}. This confirms that PTFE is present on the glass surface and that, overall, its chemical structure is similar to PTFE target material. A new band was also observed at approximately 1259 cm^{-1}. This peak has previously been attributed to overlapping CF, CF_2 and CF_3 vibrational modes and may be due to the presence of cross-linked or unsaturated species [21, 22]. The intensity of these bands is very low which limits the detection of the -CF_2- wagging peak at 644 cm^{-1}, because it is by nature a weak absorber. This low intensity coupled with the presence of strong substrate bands indicates that the coating is much thinner than the overall sampling depth of the instrument (≈1 μm). The FTIR spectra for all three areas on the sample are similar, although the intensities do vary slightly. This confirms that PTFE is uniformly distributed across the surface with slight variations in thickness from spot to spot. Reflectance spectroscopy findings show films deposited on glass to have a uniform thickness within the range 351 ± 1 nm under various process conditions and background gas, except at 300° where the thickness is about 344 nm. Film roughness, defined as peak-to-valley distance, of 51 ± 1 nm and 60 ± 1 nm has been reported for films deposited under argon and nitrogen, respectively, except at 300°C (argon) where roughness is about 33 nm. No appreciable differences between film spectra have been noticed, an indication (at least qualitative) that deposited films have similar composition. Fig. 3 shows AFM topographic images of the deposited films on glass under various process conditions. The

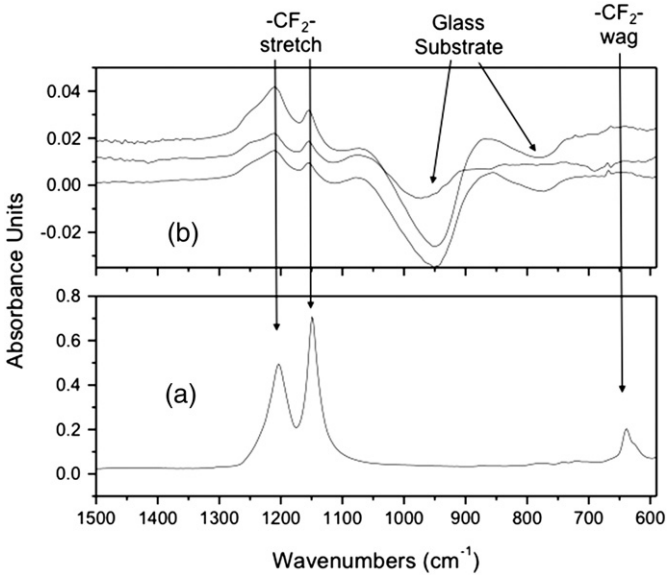

Fig. 2. ATR-FTIR spectra of (a) PTFE target material and (b) PTFE thin film (T = RT, P = 133.32×10^{-3} Pa) on glass substrate at three different positions on the film.

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