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ScienceDirect
Surface & Coatings Technology 202 (2008) 1437–1442
SURFACE & COATINGS TECHNOLOGY
www.elsevier.com/locate/surfcoat

Electrodeposition and characterization of silane thin films from 3-(aminopropyl)triethoxysilane

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Received 30 March 2007; accepted in revised form 25 June 2007
Available online 29 June 2007

Abstract

3-(aminopropyl)triethoxysilane based films have been electrodeposited directly on polycrystalline gold and gold (111) electrodes in aqueous 3-(aminopropyl)triethoxysilane based electrolyte and in tetrahydrofurane based electrolyte. These films were characterized by means of IR-ATR and X-ray photoelectron spectroscopies. The film morphology was investigated by scanning tunneling microscopy while the film growth was observed by ellipsometry measurements. The vibrationnal and X-ray photoelectron analysis suggest that the chemical composition of the electrodeposited films either in liquid tetrahydrofurane or in liquid 3-(aminopropyl)triethoxysilane is identical. The resulting coating thickness is different for the same biasing time in the two liquid media. The gold surface is coated irreversibly by an amino terminated film of great interest for sensor applications which was used as the functionalized part of a surface plasmon resonance biosensor to monitor α-lactalbumin graft.
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Keywords: Electrodeposition; 3-(aminopropyl)triethoxysilane; Scanning tunneling microscopy; Ellipsometry; X-ray photoelectron spectroscopy; Surface plasmon resonance

1. Introduction

As an alternative to sol–gel method, there were some reports of the electrochemical reduction of silanes yielding linear Si backbone polymers [1,2]. Recently, the electrodeposition of trimethoxysilane (TMOS) on negative biased conducting electrode surfaces to form thin silane films was also reported [3]. The deposition process of TMOS in aqueous solution is induced by the electrochemical reduction of oxygen to hydroxyl ions which acts as the catalyst for the hydrolysis and condensation of TMOS on the surface electrode.

A few years ago, we have discovered a new way to electrosynthesize thin linear polypropylenimine films on gold electrodes by anodic oxidation of pure 1,3-diaminopropane [4–6]. Our aim is to diversify the way to graft amine based molecule on conducting surfaces and if possible using a molecule which plays at the same time the part of solvent and monomer (to electropolymerize). This is the reason why we used 3-(aminopropyl)triethoxysilane (3-APTES) monomer, a liquid in standard conditions, which contains both amino and silano groups. In this connection, we transposed the work made with TMOS with 3-APTES so that the gold electrode presents amino terminated groups on its surface and makes it possible to graft biological species.

Effectively, two recent papers reported the electrodeposition of 3-APTES on gold or on recordable gold-type compact disks but first modified with a monolayer of alkanedithiol [7,8]. Here

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doi:10.1016/j.surfcoat.2007.06.038

we used the electrochemical generation of the condensation catalyst [9,10] and applied it to 3-APTES.

2. Experimental

All products were from Sigma-Aldrich (France) and ultra pure water (Milli-Q, Millipore) was used when needed. Bovine α-lactalbumin was solubilized at 2 mg/mL in distilled water and stored at −20 °C. A standard three-electrode electrochemical cell setup was used and all potentials are quoted vs. a silver wire reference electrode (SRE).

Several gold substrates have been used in this study as working electrodes. 5 MHz polished gold coated AT cut quartz (Maxtek, USA) was used (in shear mode) and mounted on a probe for electrochemical quartz crystal microbalance (EQCM) experiments (1.37 cm^2 surface area). For scanning tunnel microscopy (STM) imaging, gold coated mica surface (Molecular Imaging, USA) with 1500 of Au (111) was used. 50 nm thickness gold on glass (10×12 mm^2 surface area) was used for surface plasmon resonance (SPR) measurements (Au Kit, Biacore).

Functional group analyses were carried out by means of the Omnisampler IR-ATR module plugged to the FT-IR spectrometer (Nexus 470; ThermoElectron, France).

Ellipsometric measurements were performed *via* an UVISEL (Horiba, France) phase-modulated spectroscopic ellipsometer. The incident angle was set at 70° and the light spot was 1 mm in diameter. The measurements were performed in the spectral range 250–850 nm, with sampling steps of 5 nm.

A Picoscan system (Molecular Imaging, USA) equipped with a 10×10 mm^2 scanner (1 nA/V sensitivity) was used for STM measurements using Pt–Ir tips prepared by electrochemical etching [11].

X-ray photoelectron spectroscopy (XPS) measurements were conducted on a ThermoElectron VG spectrometer equipped with a monochromatized Mg Kα X-ray source (1486.6 eV photons) using pass energies of 150 and 20 eV for survey and detailed scans, respectively. C1s peak of binding energy (284.6 eV) was used as the reference.

SPR experiments were conducted on a Biacore 3000 (Biacore International AB, Uppsala, Sweden) using 3-APTES electrodeposited on bare gold sensor chip. After electrodeposition, the sensor chip was inserted in the Biacore device and a continuous flow of 10 mM Hepes, 0.15 M NaCl, 3 mM EDTA, 0.005% surfactant P20, pH=7.4 (HBS-EP), over the sensor surface at 5 μL/min, was maintained. All the following experiments were done at 25 °C. First, the surface was rinsed with water and activated by injecting 300 μL of glutaraldehyde 1%. Then, after washing with water, 300 μL α-lactalbumin at 2 mg/mL were injected on the surface.

3. Results and discussion

3.1. Electrochemical modifications

Initially we wished to use pure 3-APTES as solvent for electrolyte synthesis. But due to its low dielectric constant, it

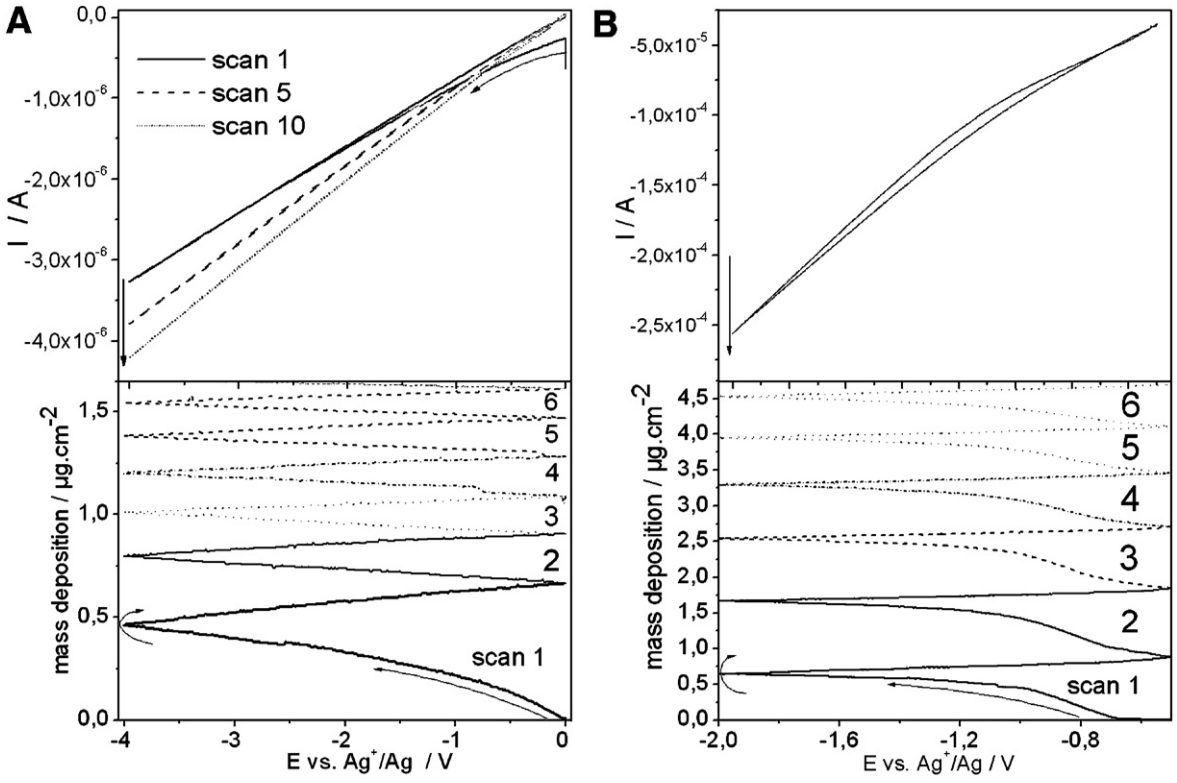

Fig. 1. CV on polycrystalline gold (1.37 cm^2 surface area), (A) of 3-APTES containing 1 mM of $N(C_4H_9)_4PF_6$ plus water (10^{-3} M) in the range [0 V, −4 V], (B) of 3-APTES (10^{-2} M) plus water (10^{-3} M) in THF in the range [0 V, −2 V] at 20 mV/S and the simultaneous mass deposition as a function of the potential applied to a 5 MHz gold coated AT cut quartz crystal.

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