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Plasma-chemical surface functionalization of flexible substrates at atmospheric pressure

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Abstract

We demonstrated here fascicle surface functionalization of poly(tetrafluoroethylene) [PTFE] sheet through an atmospheric pressure plasma liquid deposition [APPLD] technique to design the interface for improvement of copper plated layer adhesion. We found that radiation with atmospheric pressure plasma to the PTFE sheet covered with thin liquid film, containing both an ion trapping polymer and copper ion (Cu^{2+}), simultaneously brought about the direct polymer grafting onto the PTFE surface and Cu^{2+} ion reduction to metallic Cu. The treated surface was characterized by water contact angle measurement, X-ray photoelectron spectroscopy, and atomic force microscopy.
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Keywords: Surface functionalization; Atmospheric pressure plasma; Poly(tetrafluoroethylene); Graft polymerization; Nanoclusters; Electroless deposition

1. Introduction

Plasma enhanced coating processes are well known as a route to well adhered, conformal, high performance coatings. These processes provide fascinate coatings without any complex equipments. Applications, however, have often been restricted to high value opportunities as plasma processes typically operate at reduced pressure with high capital cost and batch wise operation. Very recently, Badyal group at University of Durham (UK) and Dow corning have been developed new coating process. By combining atmospheric pressure glow discharge technology with a wet precursor delivery system, they developed Atmospheric Pressure Plasma Liquid Deposition (APPLD) [1,2]. The APPLD, operating at atmospheric pressure and ambient temperature, allows the use of a wide range of liquid precursors delivering higher coating rates onto flexible substrates, of which it can be both insulating and conducting. The precursors could be chosen from a range of polymer precursors such as acrylate and alkenes. Furthermore, for gas barrier applications, metal oxides could be produced from liquid organosilicon precursors. In this work, we demonstrated plasma-chemical surface functionalization of poly(tetrafluoroethylene) [PTFE] film using the APPLD approach to design an interface for high-frequency electronics, as a one of new APPLD coating approach.

Poly(tetrafluoroethylene) is one of ideal materials for high frequency electronics applications due to its fine thermal stability, chemical inertness and low dielectric constant, ca. 2.1 [3,4]. Due to its surface inertness, pristine PTFE substrate fails to satisfy the adhesive strength against to various metals and semiconductors that many of the industry require. Pre-treatment of the PTFE surface before metallization via electroless plating become key technology. A number of research groups demonstrated surface modification of PTFE surface for adhesion improvement through both physical and chemical approaches including ion beam processing, corona discharge, X-ray irradiation, plasma treatment, wet chemical etching, and graft polymerization [5–8]. Especially, ultraviolet (UV) light initialized copolymerization with a metal ion trapping polymer exhibited significant improvement of adhesive strength of electrolessly deposited copper (Cu) on PTFE surface [9–11]. However, graft copolymerization through these processes highly improved the adhesive strength, complex equipments and tedious processing steps were required. For instance, thermally or UV initiated grafting reactions generally operate in degassed solvents.

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Here we demonstrated simple step functionalization of PTFE surface by using APPLD approach when Cu^{2+} ion containing wet-coated poly(4-vinylpyridine) (P4VP) was used as a liquid precursor. Exposing with atmospheric pressure plasma to the PTFE sheet covered with thin wetted-P4VP film brought about the direct polymer grafting onto the PTFE surface. We also found Cu^{2+} ion was simultaneously reduced to metallic Cu(0) nanoclusters with high efficiency. This means that the APPLD approach requires no surface sensitization process with $SnCl_2$/$PdCl_2$ in conventional use for electroless plating. We believe that the APPLD approach containing both metal ion trapping layer as a precursor and metal ion will be very useful technique for pre-treatment of polymer surface functionalization before electroless plating with lower environmental burden characteristics.

2. Experimental section

2.1. Materials

Poly(tetrafluoroethylene) sheet with 1.0 mm thick was purchased from Nippon Valqua Industries Ltd. The PTFE sheets were cut into platelets with 20×20 mm^2 in area. The sheet was ultrasonically cleaned with acetone and pure water for 15 sec each. Poly(4-vinyl pyridine) (P4VP, Mw: 60 K) and copper (II) acetate monohydrate (CuAc) were purchased from Sigma-Aldrich corporation, and Wako Pure Chemical Industries, Ltd., respectively.

2.2. APPLD treatment

The process of surface functionalization by via APPLD treatment contains of three main steps: (a) plasma-chemical treatment of PTFE surface under atmospheric pressure helium (He) plasma with short period; (b) ultra-this film deposition from mixture solution coating containing both P4VP and CuAc (P4VP/CuAc) on the pretreated PTFE surface; (c) He plasma treatment of the P4VP/CuAc-coated PTFE surface.

He plasma pretreatment of the PTFE surface to hydrophilically modify the surface was performed in a self-produced chamber, as shown in Fig. 1. The glow discharge was produced

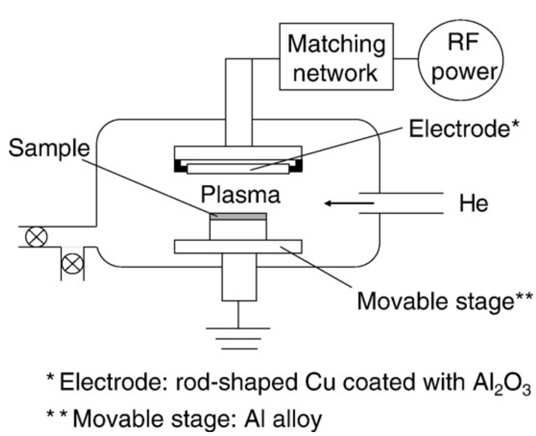

Fig. 1. Schematic illustration of experimental arrangement for the atmospheric pressure plasma treatment.

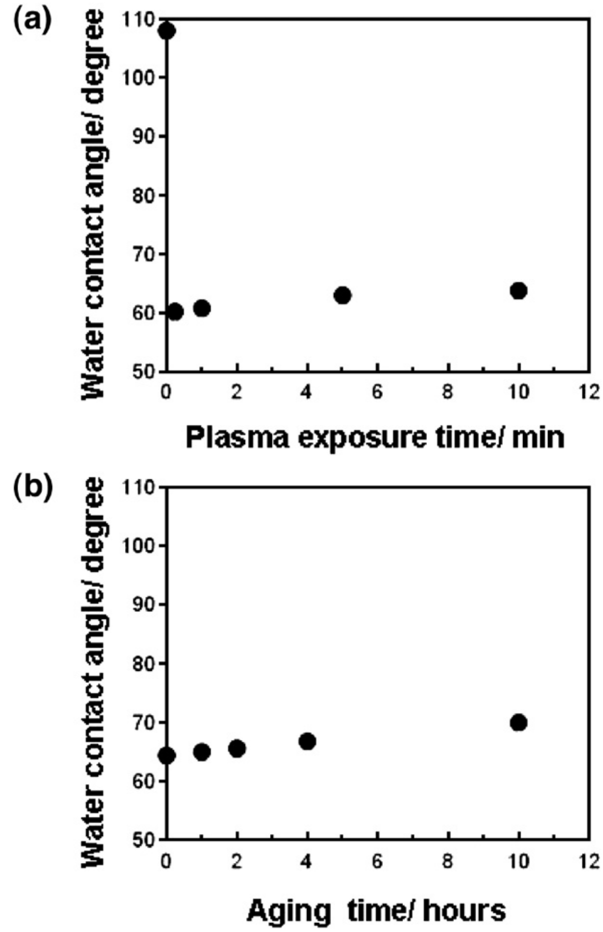

Fig. 2. Plasma-chemical surface hydrophilization of the PTFE sheet under atmospheric pressure. (a) The changes in the water contact angle as a function of the plasma exposure time. (b) The stability of the plasma-chemically modified PTFE surface hydrophilicity against aging time.

at an applied frequency of 13.56 MHz, an applying power of 15 W. The face-type electrodes consist of rod-shaped copper coated with thin alumina layer of 5 mm in diameter and aluminum plate of 500×20 mm^2 in area, respectively. The samples were exposed to a RF-generated atmospheric He plasma at designated time. The reactor was pumped down to 7.50×10^{-2} Torr prior to the introduction of He gas.

The P4VP/CuAc composite ultra-thin film was prepared on the treated PTFE sheet by spin-coating from a 300 μL of ethanol solution (3.920×10^{-2} M) at 2000 rpm for 20 s. The molar ratio of CuAc to the pyridine unit was controlled at 1:2. The films were heated at 80 °C for 10 min to remove residual solvent. The plasma-chemical P4VP grafting to the PTFE surface and reduction of Cu^{2+} into Cu(0) seeds was carried out in a manner similar to the hydrophilization.

2.3. Characterization

The water contact angle was measured using contact angle measurements [Dropmaster300, Kyowa interface science Co., Ltd, Japan]. Surface morphology was characterized by atomic

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