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Original Research Article

Functionalization of insulating substrate and its hybridization with carbon fibers

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ABSTRACT: In this work, surface functionalization of an insulating glass substrate was performed, and it was hybridized with conducting carbon fiber materials, carbon coils. Glass substrate was carried out via different chemical treatments, and then an amine-terminated self-assembled monolayer was introduced on its surface. Carbon coils were also treated with nitric acid. These surface-modified carbon coils, glass substrates, and carbon coils immobilized on glass substrates were analyzed through different analytical tools. Finally, hybridization of carbon coils on glass substrates resulted only in functionalized glass (amine-terminated) surfaces via chemical bonding, while the unfunctionalized glass substrate did not. Thus, such a stable, recognized practice can apply to fabricate simple microarrays to bind carbon materials or biomolecules for further application.

KEYWORDS: insulating glass substrate; conducting carbon materials; hybridization; self-assembled monolayer; surface functionalization

1. Introduction

In the semiconductor industry, silicon and indium are widely used substrates due to their extraordinary properties. Indium Tin Oxide (ITO) has been used in device applications for a long time. ITO is an expensive material. It was used too much, and the continuation is still going on. At present, scientists are trying to find alternative materials for ITO and are trying to make artificial materials that have similarities with ITO and could be cheaper than existing ones. Glass substrate is commonly used in various fields, and it is cheaper than other existing substrates. The main properties of glass are its amorphous shape, variable density, variable textures depending on the components, malleability in the liquid state, pressure, and breakage resistance. Glass has several strong points concerning its optical properties of light transmission, good thermal properties, and capacity to be recycled: it can be produced in large and homogenous panes. Functionalization of glass surfaces might be useful to generate new types of substrates and simple microarrays to bind carbon nanomaterials or biomolecules for further application. Recently, an increasing number of such functional molecules have been synthesized in laboratories, for instance, artificial molecular motors[1,2] or electrical switches[3,4]. Such molecules have been used for the light-induced changing of the hydrophobicity[5], or for attaching proteins or DNA to surfaces for microarray synthesis and applications[6].

Carbon materials (carbon nanotubes, carbon coils, and graphenes), nanoparticles (metal oxide particles), and other materials (silane and porous materials, biomolecules), which can be incorporated as components in the fabrication of functional hybrid and composite systems (nanoarchitecture), are also matters of interest in the research community. Thus, prepared hybrids/composites are built into systems, devices, and sensors that are applicable to biomedical, energy, environmental, and photonic sciences.

However, such a study has not been found yet. So, this study is trying to introduce a technique that immobilizes one of the allotropes of carbon fibers, named carbon coils (CCs), on an insulating substrate (glass). In recent days, carbon materials (carbon nanotubes, graphene, carbon fibers, etc.) have attracted huge interest due to their potential applications[7–9]. Because they have incorporation properties with other materials (substrate, polymer, etc.)[10–12]. The formation of these materials into thin films, composites, or devices, which are highly desirable materials in research communities. It has been reported that CCs are an amorphous material and have super-elastic properties; elastic CCs are extended by 3–15 times and contracted to the original coil length[13]. Since CCs have shown a helical structure[14], when it has been touched, its inductance, capacitance, and resistance (L, C, R) parameters have been changed and become in their original position when it has been left to touch[15]. So, such a material is highly applicable in sensing devices; likewise, touch pad sensors, artificial skin, etc.

The chemical versatility of the silane chemistries adds synergistically to the desirable properties of glass, providing an accessible approach to efficiently derivatize the native hydroxyl groups with a wide variety of functional groups that can be used to create well-defined surface properties, which are suitable for reactive groups to immobilize carbon materials and other biological molecules.

In this research, an ordinary glass substrate has been used to bind CCs to its surface. After binding each other, it has observed a stable connection between them. Such a technique could be applicable to fabricating sensor devices, especially touch pad sensors, as well as an innovative substrate.

2. Experimental section

All required chemicals and CCs were used as received. The ordinary glass slide for the microscope, which was used as a substrate (PG). PG was cut down into small pieces, which were washed by acetone, and carried out for UV irradiation to clean the surface, then it was immersed into piranha solution (3:1) for up to 5 minutes. Then it was washed by deionized (DI) water and dipped into DI water for another 5 min and dried glass via blowing N_2 gas. It was expected that sufficient hydrophilic functional groups, especially hydroxyl groups, were introduced onto the glass surface (FG). Finally, FG was inserted into the solution mixture of 2% amino-propyl triethoxysilane (APTES) in toluene solvent in an airtight glass vessel overnight. The FG surface was rinsed with ethanol and later dried by N_2 gas. It was further supposed that amine-terminated self-assembled monolayers (SAMs) were introduced on the surface of FG (AG). CCs was functionalized (CCs-COOH) accordingly to our previous report[16]. Then, CCs was dispersed with chloroform in the presence of PCl_5 through ultrasonication (5 min), and then this solution (CCs-CO-Cl) was poured into the AG sample-containing glass vessel for up to 12 h. Then, the sample was rinsed with ethanol and dried via blowing N_2 gas. Finally, a CCs-deposited sample was obtained onto a glass surface (CG). Proceeding the same way, CCs were also immobilized onto a bare glass surface, and other reference samples were also prepared. The overall schematic procedure is represented in **Figure 1**.

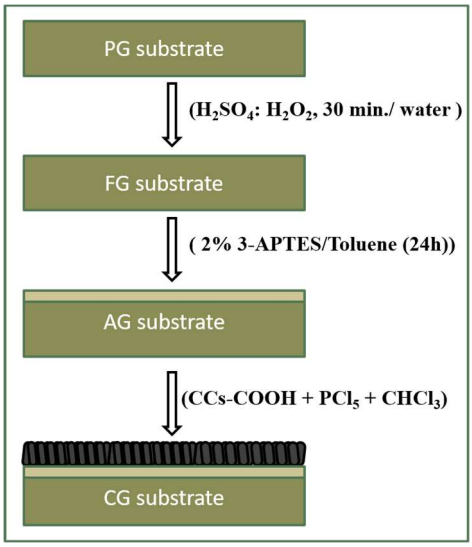

Figure 1. Schematic procedure of CCs immobilization onto glass surface.

Characterization

S Surface functionalization of glass surfaces was analyzed by water contact angle goniometry (CAM 100) and x-ray photoelectron spectroscopy (XPS) were obtained on a theta probe ESCA VG scientific (2002) using a monochromatic ALK source at a pressure of 2×10^{-9} mbar. The morphology of CCs on the glass surface and the binding of each other were analyzed by optical microscopy, JEOL JSM-6500F Scanning Electron Microscopy (SEM) at accelerating voltages of 0–15 kV, and XPS, respectively. The electrical conductivity of the CG specimen was measured by a DMM-85 conductmeter.

3. Findings and discussion

Glass surfaces are smooth, but their surfaces were distinguished by water contact angle measurement. Pristine glass surface (PG) was shown at nearly a 7° angle, while for surface treatment FG, it was shown as almost zero degrees, which means more hydrophilic groups were introduced after piranha treatment. However, after APTES deposition on FG, the water contact angle turned into 39°, which could be the successful introduction of an amine-terminated group onto the FG surface (AG) as shown in **Figure 2;** this value is close to the previously published report[16].

Figure 2. a) PG, **b)** FG, and **c)** AG samples.

These samples were further characterized by XPS. In PG and FG there is no trace of nitrogen in the N1s peak (**Figure 3a),** but in the AG sample, it has shown a strong N1s peak as shown in **Figure 3b**; this indicates that the amine-terminated group is successfully introducing on the glass surface because more than 8% of atomic nitrogen on the glass surface comes from the amine group[17,18]. The atomic percent of nitrogen is also remaining almost the same for the CG sample. It means CCs are bonded with the glass surface through amide linkage[16,19]; which is also expected, but the atomic percent of nitrogen content is found slightly increased after hybridization. This might have come from the CCs due to treatment of it

with nitric acid; in such a situation, attributed/unattributed nitrogen could be associated with CCs. In addition, C1s peak positions for FG, AG, and CG were also compared, as shown in **Figure 3**.

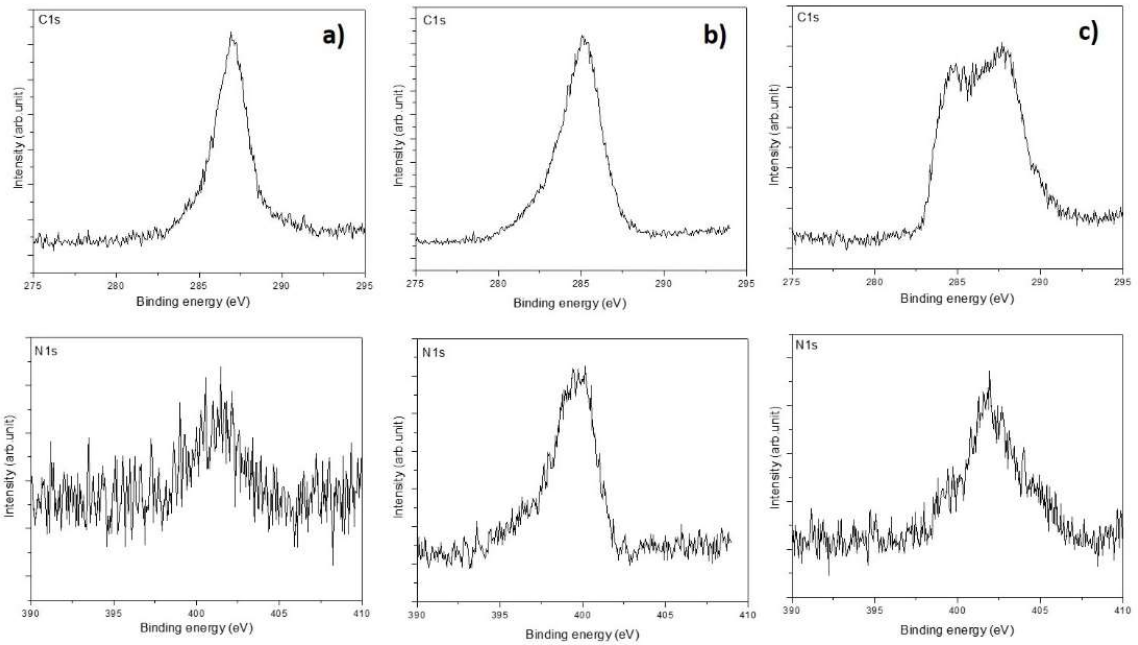

Figure 3. XPS spectra of C1s, and N1s; **a)** PG, **b)** AG, and **c)** CG samples.

The presence of carbon content on glass samples was found in increasing order; likewise, compared to FG, carbon content is found more in AG; it means more carbon content on the AG surface comes from the carbon chain linked with the amine group. Similarly, carbon content was found further in CG samples than AG. The increase of carbon content for this sample is due to the association of CCs, because CCs themselves are carbon-rich sources; CCs in pristine and acid-treated forms are shown in **Figure 4**.

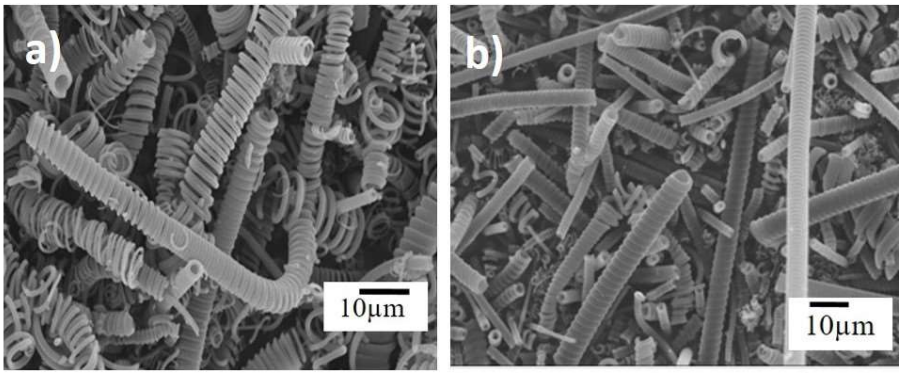

Figure 4. SEM images of pristine CCs **(a)**, and acid treated CCs **(b)**.

In addition, the C1s XPS peak for the CG sample is marginally split into two peaks; the reason might be after CCs hybridization with the amine-terminated glass sample, the amine group is converted into the amide group; i.e., the C1s peak position differs from the initial C1s peak position, which confirms that conducting carbon materials (CCs) and insulating glass substrates (glass) were chemically binding to each other[17,20] as shown in **Figure 3c**. The atomic percentages of C1s and N1s for those samples obtained by XPS were shown in **Table 1**.

Table 1. XPS elemental analysis for PG, AG, and CG samples.

Elements	PG	AG	CG
C1s	48.5	48.5	51.5
N1s	1.1	8.3	8.9

This hybridization test is further confirmed by optical microscope analysis as shown in **Figure 5**. It is clearly seen CCs on substrate surface.

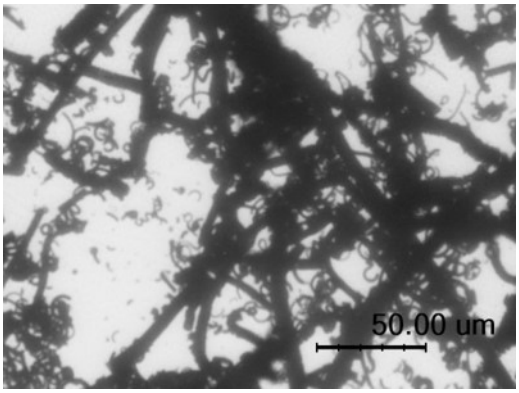

Figure 5. Optical image of CG sample.

This test is further confirmed during optical tests; different samples were checked, and CCs (CCs-CO-Cl) were immobilized onto PG, FG, AG, and AG without the acid chloride group CCs (CCs-COOH). Through the optical observation, it was found that CCs only immobilized onto the AG sample as shown in **Figure 6b**, which is due to strong binding between CCs-COCl and the $–NH_2$-Glass sample via amide linkage. In addition, without the acid chloride group, CCs were also partly obtained on the AG sample, due to possible electrostatic interaction between NH_2- group and the COOH group via weak hydrogen bonding. However, it was not found that CCs onto PG and pristine CCs onto AG samples, due to the unavailability of interacting reaction groups as shown in **Figure 6**.

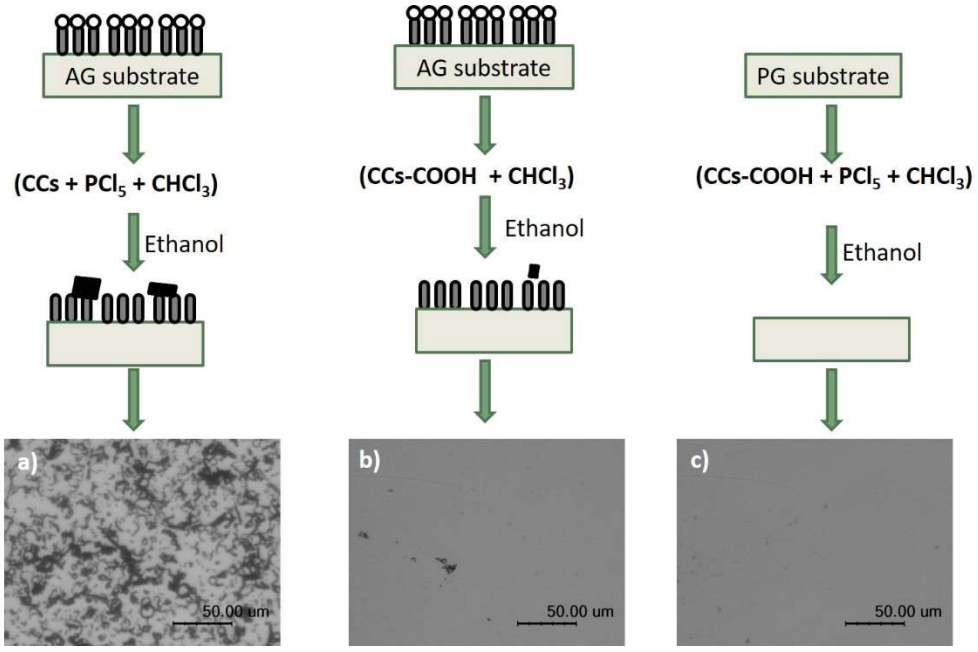

Figure 6. a) Acid chloride CCs onto AG substrate, **b)** Without acid chloride CCs onto AG substrate, and **c)** Acid chloride CCs onto PG substrate.

Since CCs is a conducting material having a helical structure with spring features. So, LCR parameters are varied in contact and non-contact mode. The conductance of CG was checked by a conductometer and found to have good conductance on the glass surface (the figure was not shown). Nevertheless, the conductance is not observed regularly, which could be due to a lack of uniform deposition on the glass surface (CG sample) and agrees with the optical image (**Figure 6b**). However, current practice could be fruitful to fabricate sensor devices, especially touchpad sensors. Simply, to generate microarrays by means of chemical photolithography, SAMs functionalized with each silane molecule on the glass surface have a vital role in fabricating the patterning layer[6,17]. Therefore, using in situ photolithographic CC synthesis, it can generate a simple microarray with the desired layer of CC sequences for its further application.

4. Conclusion

In this work, it has been shown that active amino silanes greatly increase the hydrolytic stability of glass surface functionalization, and these functionalizations are compatible with the in situ CCs combination of complex CCs arrays. CCs and glass substrates are chemically bonded and form a stable hybrid material. This technique could be fruitful for converting ordinary glass substrates into film/composite substrates to ensure a high surface area. Even after being bonded with glass, CCs have shown worthy conductivity. In addition, due to the variable parameters of CCs in contact and non-contact modes, this practice could be highly applicable to fabricating sensor devices and new types of substrates.

Conflict of interest

The author declares no conflict of interest.

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