



Contact angles of diiodomethane on silicon-doped diamond-like carbon coatings in electrolyte solutions

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ARTICLE INFO

Article history:

Received 14 March 2008

Accepted 25 June 2008

Available online 29 June 2008

Keywords:

Adhesion

Ion–solid interactions

Amorphous surfaces

Coatings

Wetting

Surface energy

ABSTRACT

The influence of surrounding electrolyte type and concentration on the contact angle of hydrophobic diiodomethane on silicon-doped diamond-like carbon (DLC) coatings was examined to provide insight into how the presence of electrolytes in the solution influences adhesion of hydrophobic material to doped DLC surfaces. There was a small but statistically significant increase of contact angle with increasing electrolyte concentration over the range from 0 to approximately 0.01 M, after which the contact angle was virtually unaffected by further increase in the concentration of electrolyte. It was shown that CaCl_2 has a stronger influence on the change of the contact angle than NaCl, and that an increase in Si content in the DLC coatings increased the change in the contact angle of diiodomethane for all types of electrolyte. These observations suggest that the adhesion to the Si-doped DLC surfaces is reduced by addition of the electrolytes to the surrounding solvent. This could be explained by increased ion adsorption on the DLC surface with increase in silicon doping, causing the surfaces to be more hydrophilic.

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1. Introduction

A full understanding of the fundamental principles that control adsorption and adhesion of particles to surfaces in electrolyte solutions would be of considerable value in a variety of applications such as anti-fouling filters, microfluidic devices, medical implants and surfaces involved in marine transportation and the oil industry [1]. In particular, an understanding of the influence of ionic strength of the media on the adsorption of proteins to surfaces could be utilised in the design of novel biocompatible materials. The dependence of the interactions between materials, as determined by interfacial surface energy, on the ionic strength of the media has been a subject of a number of investigations. However, there still remain unresolved issues about the underlying mechanisms, due to scarcity of experimental data.

Previous experimental measurements of the dependence of contact angle on the electrolyte type and concentration can be split into two groups: measurements involving one liquid, with electrolyte solution on the surface; and measurements with two liquids, when the contact angle of a liquid on the surface is measured in the environment of the electrolyte solution.

Using the single liquid method, measurements of dynamic contact angle for a range of salt solutions on fluorocarbon surfactant

monolayers deposited on mica have shown decreasing contact angles with increasing concentration of electrolyte. This could be explained in part by adsorption of electrolyte ions to the surface [2]. However, slight increases in the contact angles of aqueous solutions with increasing NaCl concentrations were observed on a PTFE surface [3]. This result was interpreted as a modification to the solvent structure by the electrolyte. Some studies have found no change in contact angle with changes in the ionic strength of the medium. For instance, one study found a constant contact angle of aqueous solutions of different electrolyte concentrations on paraffin wax surfaces and also suggested that the contact angle was independent of the type of electrolyte [4]. Self-assembled monolayers of hexadecanethiol on gold have shown no dependence of contact angle on different concentrations of NaCl [5]. Again, a constant contact angle was observed for various concentrations of different electrolytes on a range of polymers, including polystyrene, Teflon AF, and polyethylene-terephthalate [6]. Although indicative of the interactions between the surface, dissolved ions and the solvent, these measurements do not directly relate to the interactions of the surface with other materials when surrounded by electrolyte solution.

Using the two-liquid method, an investigation of hexadecane contact angles on gold-coated glass surfaces in solutions containing sodium dodecyl sulphate (SDS) and different concentrations of NaCl have revealed a considerable increase in the contact angle with increasing ionic strength of the medium, reaching a constant value at higher concentrations of electrolyte [7]. These results

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could reflect differences in the properties of the studied surfaces, and different influences of the ionic strengths on the adsorption and adhesion processes, depending on the materials, which are difficult to predict a priori. Detailed measurements of the influence of ionic strength on interactions between materials are therefore needed to help interpret the experimental data and to assist in further understanding of the forces governing adsorption and adhesion processes in each particular case.

The present work investigates the influence of ionic strength of electrolyte media on interactions between small drops of diiodomethane and diamond-like carbon (DLC) surfaces doped by different amounts of silicon by measuring the contact angle of diiodomethane. These measurements will give more information on the interactions between doped DLC surfaces and hydrophobic materials in different types and concentrations of electrolytes, which is related to the environment that would be encountered in many applications. The findings of the present study might provide information that could help to build a theoretical model of such interactions and assist in the future design of novel surfaces with tailored properties toward protein adhesion and adsorption.

2. Experimental

2.1. Materials

Si-doped diamond-like carbon films were prepared on 316L stainless steel tokens of 10 mm diameter and 1 mm thick, by a magnetron sputtering technique. The tokens were cleaned in an ultrasonic bath containing acetone for 10 min, rinsed in distilled water and dried before being placed in the coating chamber. The substrates were further cleaned by Ar⁺ bombardment prior to deposition. The silicon doping of the DLC films was achieved by introducing tetramethylsilane into the coating chamber and silicon content in the films was altered by changing its flow rate. The average Si content in the three types of coating used in the present study was $0.4 \pm 0.5\%$, $1.8 \pm 0.6\%$, and $4.5 \pm 1.9\%$ as determined by X-ray photoelectron spectroscopy (XPS). Electrolyte solutions with different concentrations were prepared using NaCl (over 99% purity, Aldrich) and CaCl₂·2H₂O hydrate (over 99% purity, Riedel-Haem) dissolved in distilled water in a volumetric flask.

2.2. Cleaning procedures

The samples were cleaned well. They were firstly sonicated for at least twenty minutes in a 2% solution of sodium dodecyl sulphate (Fisher Scientific) in distilled water, rinsed under tap water for five minutes and then sonicated twice for ten minutes in distilled water. They were then sonicated in acetone (99% purity) for at least fifteen minutes, rinsed thoroughly with distilled water and sonicated twice in distilled water for ten minutes. Finally, the samples were dried in an air flow from a hot air gun at about 50 °C.

2.3. Contact angle measurements

Measurements of contact angles of diiodomethane on silicon-doped diamond like surfaces using the sessile drop method were performed in electrolyte media with a DataPhysics dynamic contact angle/surface energy analyser (model OCA 20). Care was taken to ensure that the contact angles were measured inside the diiodomethane. The measurements were performed in a 20 mm square cuvette, 40 mm high, made of 2 mm thick polished borosilicate glass (Carl Stuart, Leek, UK) with a lid. The cuvette was placed on a constant temperature base, maintained at 26 °C by the circulation of temperature-controlled liquid. The Si-doped DLC samples were placed at the bottom of the cuvette filled with electrolyte and drops of diiodomethane about 5 µl in volume were applied to the

Table 1

Average values of diiodomethane contact angle θ (°) on different Si-doped diamond-like carbon surfaces in aqueous solutions with different concentrations of NaCl

Electrolyte concentration (M)	DLC surface		
	0.4% Si	1.8% Si	4.5% Si
0	146.1 ± 0.9	145.8 ± 0.8	145.4 ± 1.3
0.001	146.1 ± 1.0	147.4 ± 0.7	146.7 ± 1.0
0.002	148.2 ± 0.8	147.5 ± 0.8	147.5 ± 1.0
0.005	148.3 ± 0.7	149.0 ± 0.9	148.8 ± 0.9
0.01	148.5 ± 0.8	149.1 ± 0.8	148.7 ± 0.7
0.02	149.1 ± 0.6	149.2 ± 0.6	148.7 ± 1.1
0.05	148.9 ± 0.7	149.1 ± 0.8	148.5 ± 0.8
0.1	148.9 ± 0.7	148.8 ± 0.7	148.6 ± 0.8

Table 2

Average values of diiodomethane contact angle θ (°) on different Si-doped diamond-like carbon surfaces in aqueous solutions with different concentrations of CaCl₂

Electrolyte concentration (M)	DLC surface		
	0.4% Si	1.8% Si	4.5% Si
0	146.1 ± 0.9	145.8 ± 0.8	145.4 ± 1.3
0.001	147.8 ± 0.7	148.3 ± 0.7	148.8 ± 0.5
0.002	148.9 ± 0.9	149.3 ± 0.6	149.9 ± 0.9
0.005	149.5 ± 0.7	149.9 ± 0.7	150.1 ± 0.9
0.01	149.4 ± 0.6	149.8 ± 0.7	149.9 ± 0.7
0.02	149.3 ± 0.5	149.6 ± 0.5	149.6 ± 0.7
0.05	149.3 ± 0.6	149.5 ± 0.5	149.7 ± 0.6
0.1	149.2 ± 0.5	149.5 ± 0.5	149.7 ± 0.6

sample surface. The drops of diiodomethane, being denser than the electrolyte, attached to the surface. The projections of the drops viewed from two adjacent sides of the cuvette were recorded and the static contact angles (θ) were determined using the ellipse fitting method [8], which was available as part of the software suite of the contact angle measurement system.

Overall, 36 measurements of contact angle were obtained on each type of surface for each of eight electrolyte concentrations for each electrolyte, using three tokens for each type of surface and twelve repeated measurements. In addition, measurements using distilled water as a medium were performed for comparison. Table 1 lists the mean values of diiodomethane contact angles with corresponding standard deviations on the different Si-doped DLC surfaces with different sodium chloride concentrations and Table 2 gives values using calcium chloride solutions as the medium.

3. Results and discussion

For each surface coating the contact angle of diiodomethane initially increased slightly with the concentration of the electrolyte, then reached an approximately constant value for the higher concentrations. Although small, the observed increase was statistically significant at the 95% confidence level as indicated by pairwise *t*-tests comparing the average contact angle in distilled water with the highest values of the average contact angles in electrolyte solution, 0.02 M for sodium chloride and 0.005 M for calcium chloride. The tests were performed using STATA software. The conditions for applicability of the *t*-test, such as normality of distribution of measurements and equality of variances, were also checked. Small, but noticeable differences in the contact angle changes were observed for the two types of electrolyte. For the same concentration of the electrolyte the increase in the contact angle as compared to pure water was larger for CaCl₂ than for NaCl for all surfaces tested.

It has been observed previously that the surface tension of water increases linearly with the salt concentration [9]. It would be expected for the contact angle of diiodomethane to decrease linearly with increasing salt concentration if the surface tension

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