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Water interaction with hydrogenated and oxidized detonation nanodiamonds – Microscopic and spectroscopic analyses

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1. Introduction

Detonation nanodiamonds (DNDs) represent a fascinating nanomaterial which is made by a detonation of oxygen-deficient explosives such as trinitrotoluene (TNT) and hexogen (RDX). Their size is typically 4–5 nm, however, by using nano-textured explosives one can even further reduce their typical size below 3 nm as recently shown [1,2]. Once the DNDs are extracted from detonation soot by various wetchemical purification treatments they can serve in many diverse applications such as polishing, electroplating, lubrication, catalysis, and drug delivery [3,4]. The surface properties of DNDs often play an important role due to the high surface-to-volume ratio and the possibility of adjusting the surface chemistry [5]. The most common, well-defined surface chemistry of DNDs is obtained by oxidation or hydrogenation of raw DND powders. Hydrogen termination of DNDs is typically achieved by a hydrogen plasma treatment [6,7] or by annealing in hydrogen gas at elevated temperature [8,9]. Both processes lead to the formation of carbon-hydrogen bonds on the DND surface and the formation of positive zeta potential in colloidal solution of hydrogenated H-DNDs [9,10]. Oxygen termination of DNDs is typically achieved by annealing in air at temperatures between

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ABSTRACT

Water interaction with surface modified nanodiamonds (NDs) is critical for many possible applications of NDs e.g. in biomedicine. Here we report on investigation of water interaction with hydrogenated and oxidized detonation nanodiamonds (H-DNDs, O-DNDs) by means of Fourier transform infrared spectroscopy (FTIR), thermal analysis, and Kelvin probe force microscopy (KPFM). Higher water content (4.4%) as well as weaker interaction of water with H-DNDs are identified by thermal analysis. It is explained by hydrophobicity of the H-DND surface, as revealed by the analysis of bending and stretching vibrations of surface water in FTIR spectra. On the other hand, hydrophilic nature of O-DNDs leads to lower water content (3.1%) but stronger interaction with the O-DND surface. This is evidenced by as high as 300 °C desorption temperature of the surface water from O-DNDs. KPFM analysis shows that the surface-bound water can have significant screening effect on contact potential difference (CPD) of nanodiamonds (up to 510 mV) as well as on the CPD difference between H-DNDs and O-DNDs (from 50 mV to 210 mV). Nevertheless, H-DNDs exhibit always lower work function.

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400–500 °C [11,12]. In contrast to H-DNDs the formation of functional groups containing oxygen on the surface of DNDs leads to a negative zeta potential of oxidized O-DNDs in colloidal solution [10,13]. It is known that hydrogenated or oxidized surface of bulk diamond (mono or polycrystalline) exhibits very different physicochemical properties such as contact angle, surface conductivity or work function. Whether hydrogenated and oxidized DNDs exhibit also such differences is still an open question [14] though some recent reports indicate a surface conductivity of H-DNDs [15] or difference in work function between H-DNDs and O-DNDs [10].

Geometric constraints of a solid surface as well as the presence of specific functional groups generally lead to structural changes of a surface-bound water layer compared to bulk water properties. In this respect, the water interaction with DNDs is fundamental for both the technology and applications. However it is still not fully understood and explanations of various phenomena are debated. For instance, the formation of nanophase water on DNDs was reported, however, no attention was paid to the surface chemistry of the studied DNDs [16]. Recent study showed that orientation of water molecules in the first solvation shell depends on the zeta potential of NDs [17]. Giant dielectric permittivity of DNDs was also attributed to adsorbed water (1%–4%) but actual mechanism required further investigation, being tentatively related to proton-releasing functional groups on DNDs [18].

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In this paper we provide experimental evidence of different water interactions with H-DNDs and O-DNDs by using Fourier transform infrared (FTIR) and thermal analysis (TA) techniques. We observe different water adsorption abilities and different water bindings on H-DND and O-DND surfaces and we deduce a corresponding structural model. By using Kelvin probe force microscopy (KPFM), we also show how the adsorbed water significantly and differently affects the contact potential difference (CPD) of H-DNDs and O-DNDs that are deposited on highly ordered pyrolytic graphite (HOPG).

2. Experimental

Detonation nanodiamonds were synthesized from TNT and RDX precursors in a detonation chamber. The DNDs were purified in HCl/ HNO_3 (1:3) and then rinsed with deionized water to pH 5 [12]. Oxidized O-DNDs were obtained by air annealing at 385 °C for 14 h. A part of already oxidized O-DNDs was subsequently hydrogenated by annealing under hydrogen atmosphere at 700 °C for 3 h. By this approach hydrogenated H-DNDs were obtained.

The specific surface area (SSA) of H-DNDs and O-DNDs were measured by using an ASAP 2020 system. Thermal desorption at 225 °C during 12 h was applied to the samples before measurements. SSA values of 368 m^2/g and 355 m^2/g were measured for H-DNDs and O-DNDs respectively.

FTIR was measured on a Bruker Tensor 27 apparatus equipped with a single attenuated total reflectance (ATR) accessory (PIKE MIRacle). The detector was a Digitect RT-DLATGS and the resolution was 4 cm⁻¹. The ATR crystal was a diamond (with a Zn/Se lens). The measurement was performed at the temperature of 20 °C. The H-DNDs and O-DNDs were measured in form of powders.

TGA was performed on a SII Seiko Instruments Exstar 6000 (TG/DTA 6200) and Differential Scanning Analyses (DSC) was performed using a TA instruments Q1000 apparatus. The experiments were carried out in alumina cups and gold crucibles for TGA and DSC respectively. Both analyses were conducted under a nitrogen flow (100 mL/min). An isothermal step at 25 °C during 2 h was maintained before heating, and then a ramp of 5 °C/min was applied to reach 500 °C.

For the KPFM experiments, colloidal suspensions from H-DND and O-DND powders were prepared at first by ultrasonication (ultrasonic bath 100 W) and ultracentrifugation (350,000 g, 30 min), and then used for electrophoretic deposition on highly ordered pyrolytic graphite (HOPG) [19] using positive zeta potential of the H-DNDs and negative zeta potential of the O-DNDs. The prepared samples were then left few days at ambient conditions to reach equilibrium with air humidity (typical RH ~50%). The samples were then introduced into a glovebox filled with dry nitrogen, ensuring inert and humidity-free atmosphere (O₂ and H₂O were kept below 0.1 ppm level) and analyzed by in-situ KPFM immediately. These KPFM data are denoted as "not annealed". In order to check the effect of adsorbed water on the CPD of H-DNDs and O-DNDs, an annealing sequence in the glovebox was performed by following steps: 50 °C, 100 °C, 150 °C, 200 °C, 250 °C, and 300 °C for 30 min, each step followed by immediate KPFM measurement. A FlexAFM with a C3000 controller (Nanosurf) was used for topography acquisition and a UHFLI lock-in amplifier (Zurich Instruments) served as an external Kelvin controller. The KPFM measurement was carried out in a single pass regime with amplitude (AM) modulation, i.e. both topography and CPD were acquired simultaneously using the first and second resonance of AFM cantilever [20]. For all reported data, we used a single Pt/Ir5-coated PPP-EFM cantilever (Nanosensors) with a resonance frequency of $f_1 = 76,950$ Hz. This fundamental resonance frequency was used for topography acquisition. The second resonance frequency of the cantilever ($f_2 = 481,498$ Hz) was used for KPFM. The free oscillation amplitude was 10 nm, setpoint 65%, scan size $2 \times 2 \,\mu$ m, scan speed 0.5 Hz, and AC oscillation voltage 1 V. The cantilever was grounded and both AC oscillation and DC compensation voltage were applied to the sample. Using this arrangement, lower CPD value corresponds to lower work function value however work function of cantilever would need to be determined to get absolute values. We used a compensation of the capacitive crosstalk to obtain accurate KPFM results [21].

3. Results

Fig. 1a shows full range FTIR spectra of DNDs after oxidation in air (O-DNDs, red spectrum) and subsequent hydrogenation (H-DNDs, blue spectrum). The FTIR spectra of both H-DNDs and O-DNDs were acquired several weeks after the respective treatments, and stored at ambient conditions meanwhile. Thus we suppose that equilibrium with respect to ambient humidity was reached. The common features in the spectra are related to O-H stretching (2500–3500 cm⁻¹) and bending (1625 cm⁻¹) vibrations from the adsorbed water [8,22].

In addition, the spectrum of O-DNDs exhibits a carbonyl peak (C=O stretching at 1800 cm⁻¹) indicating the formation of anhydrides and lactones by air-annealing which shifts the peak to higher wavenumbers than in carboxylic acid groups [23]. A broad absorption feature at 1000–1500 cm⁻¹ is related to a combination of many overlapping peaks, which besides C–O–C stretching may include also the O–H deformation and other groups [22]. The FTIR spectrum of H-DNDs shows the successful hydrogenation by the formation of C–H bonds (2800–3000 cm⁻¹) accompanied by complete disappearance of the carbonyl peak and suppression of the carbon–oxygen related features in the 1000–1500 cm⁻¹ region. The features in 2200–2400 cm⁻¹ region are not related to DNDs but arise from software subtraction of atmospheric CO₂ absorption.



Fig. 1. Full range FTIR spectra of H-DNDs (blue curve) and O-DNDs (red curve) equilibrated (eq.) in ambient air (a). Magnified region of 1500–1800 cm⁻¹ shows in detail the different shapes of the –OH bending peak of H-DNDs and O-DNDs (b). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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