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Energy harvesting with peptide nanotube–graphene oxide flexible substrates prepared with electric field and wettability assisted self-assembly

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ABSTRACT

Piezoelectric diphenylalanine peptide nanotubes (PNTs) have recently been demonstrated in energy harvesting applications, typically based on vertically aligned PNTs that generate charge when pressed. In this work, we use a wettability difference and an applied electric field to align PNTs and PNT-based composites on flexible substrates. Open-circuit voltages and short-circuit currents exceeding 6 V and 60 nA, respectively, are achieved by bending the substrate, opening up the use of horizontally aligned PNTs as flexible energy harvesting substrates.

I. INTRODUCTION

Energy harvesting with nanogenerators can accelerate the development of self-powered systems for personal and structural health monitoring and Internet of Things applications.[1–4] Piezoelectric materials are particularly well-suited for harvesting energy from mechanical vibrations because of their inherent electromechanical coupling. Piezoelectric nanogenerators are commonly made using inorganic materials with favorable piezoelectric coefficients and electromechanical coupling factors, such as zinc oxide and lead zirconate titanate.[5–7] The search for sustainable, "green" materials has led to the use of organic materials, including peptide-based materials, in piezoelectric energy harvesting applications.[8–14] Diphenylalanine peptide nanotubes (PNTs) have been reported to have high mechanical strength and produce a strong shear piezoelectric response ($d_{15} = 46.6$ pm/V), comparable to conventional inorganic piezoelectric materials, making PNTs an attractive energy harvesting material.[12]

Nguyen *et al.* have shown that controlling both the alignment and the polarization of the PNTs is critical to the power generation.[8,14] The vertically aligned PNTs were grown from a seed layer on a substrate placed upside down and floating on a saturated diphenylalanine solution as the solution evaporated.[8,14] When the seed layer was prepared in the presence of an electric field (1 MV/m), the dipoles of the diphenylalanine peptides aligned with the field,[15] and the polarization of the seed layer and of the resulting PNTs was uniform.[8] Polarization alignment led to an enhanced power output when the vertically aligned PNTs were pressed (60 N force), achieving an open-circuit voltage of 1.4 V, a current of 39.2 nA, and a power density of 3.3 nW/cm^2.[8] A flexible nanogenerator was also realized by bending a substrate comprising an array of vertically aligned PNTs, producing an open-circuit voltage of 0.6 V and a current output of ~7 nA.[9] Recently, horizontally aligned and pressed PNTs have also been demonstrated for

energy harvesting.[12] Lee *et al.* used meniscus-driven self-assembly to align the PNTs and their polarization, achieving voltage, current, and power of up to 2.8 V, 37.4 nA, and 8.2 nW, respectively.[12]

Previously, the use of a wettability difference to aid the alignment of self-assembling PNTs was demonstrated.[16,17] In this work, building on previous reports of the effect of electric field on PNT alignment[18] and polarization control,[8,9,14] we investigate the

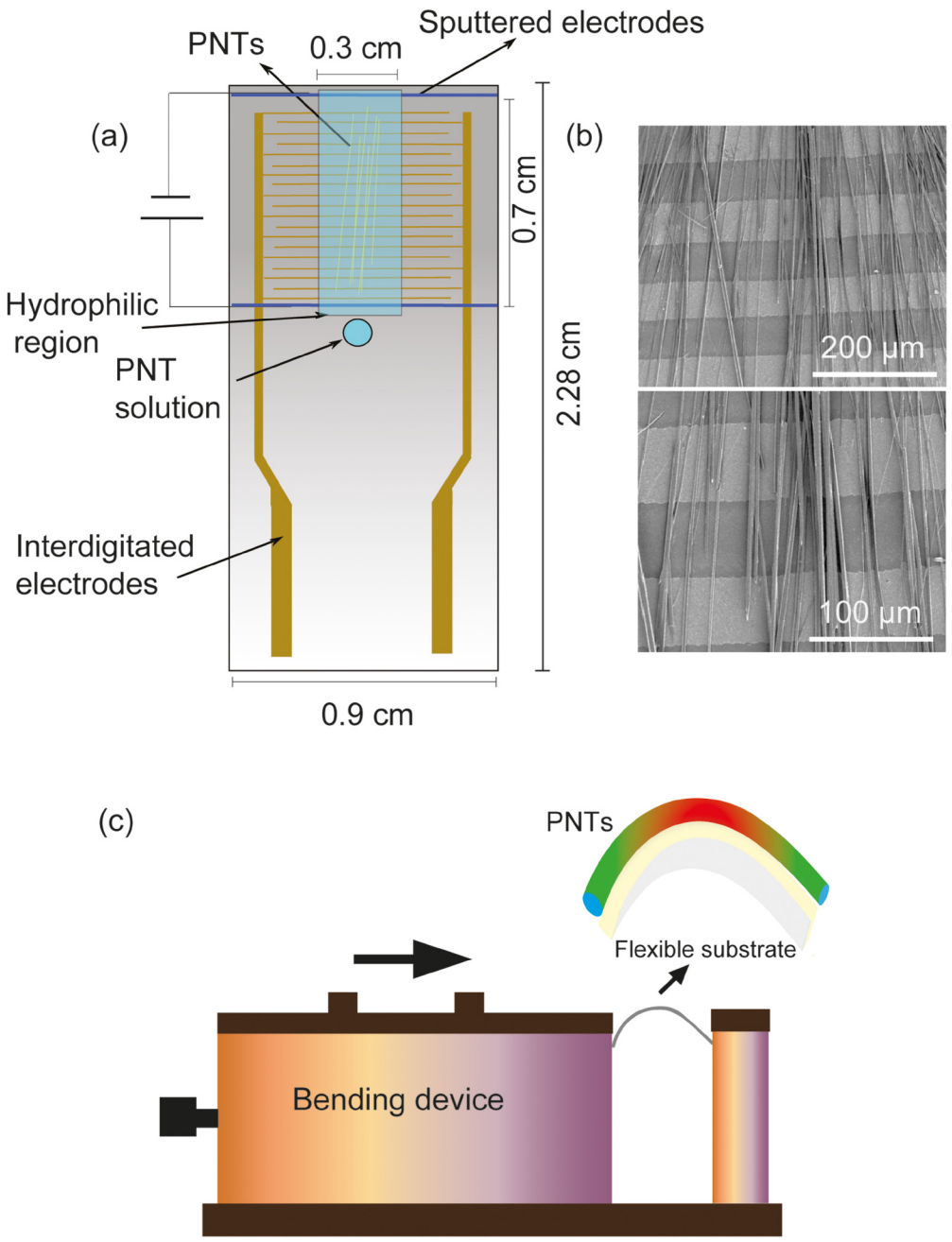

FIG. 1. (a) PNTs aligned across interdigitated electrodes under the influence of external voltage for one UV/ozone exposure opening. (b) SEM images of the aligned PNTs. (c) The bending stage used to flex the substrate. The substrates were flexed such that the aligned PNTs were bent, as indicated in the inset in (c), where the region of highest stress is indicated in red.

combination of UV/ozone exposure (to create a wettability difference) and an applied electric field for the creation of horizontally aligned polarization controlled PNTs on flexible substrates with interdigitated electrodes for energy harvesting, achieving open-circuit voltages as large as 4.0 ± 0.5 V and currents as high as 37.5 ± 3.8 nA. Furthermore, we investigate the effect of incorporating graphene oxide (GO) with the PNTs on the energy harvesting performance. Incorporation of GO has been shown to improve the stability of PNTs in solution[19] and thus may facilitate the use of PNTs in biomedical applications.[20] GO is also known to increase PNT conductivity,[21] facilitating an open-circuit voltage of 6.6 ± 0.5 V and a current output of 63.8 ± 1.4 nA, both significantly higher than for PNTs alone.

II. MATERIALS AND METHODS

A. Preparation of PNTs on flexible substrates

PNTs were prepared by dissolving L-diphenylalanine peptide (G-2925, Bachem) in 1,1,1,3,3,3-hexafluoro-2-propanol (Sigma-Aldrich) at an initial concentration of 100 mg/ml. The solution was further diluted in de-ionized water to a final concentration of 2 mg/ml for PNT self-assembly and kept in a fridge at 4 °C overnight.[15] Fresh stock solutions were prepared for each experiment.

Flexible plastic substrates (L 22.8 mm × W 7 mm × H 0.175 mm) with interdigitated gold electrodes (P-IDEAU100, Dropsens) having 100 μm electrodes and separations [see Fig. 1(a)] were first cleaned of surface contaminants by washing with de-ionized water and dried using nitrogen. In order to apply an electric field, the flexible substrate with interdigitated gold electrodes was modified by sputtering (Quorum, SC7620) two outer gold electrodes through two ∼0.1 cm openings, separated by ∼0.7 cm, in a mask made from Kapton tape. These outer electrodes were positioned parallel to and isolated from the interdigitated electrodes. Following deposition, the mask was removed.

To create hydrophilic regions, pieces of a Si wafer, cut using a diamond scribe and separated by 0.3 cm to create one or two 0.9 cm long openings orthogonal to the interdigitated electrodes and within the electrode region, were placed in contact with the

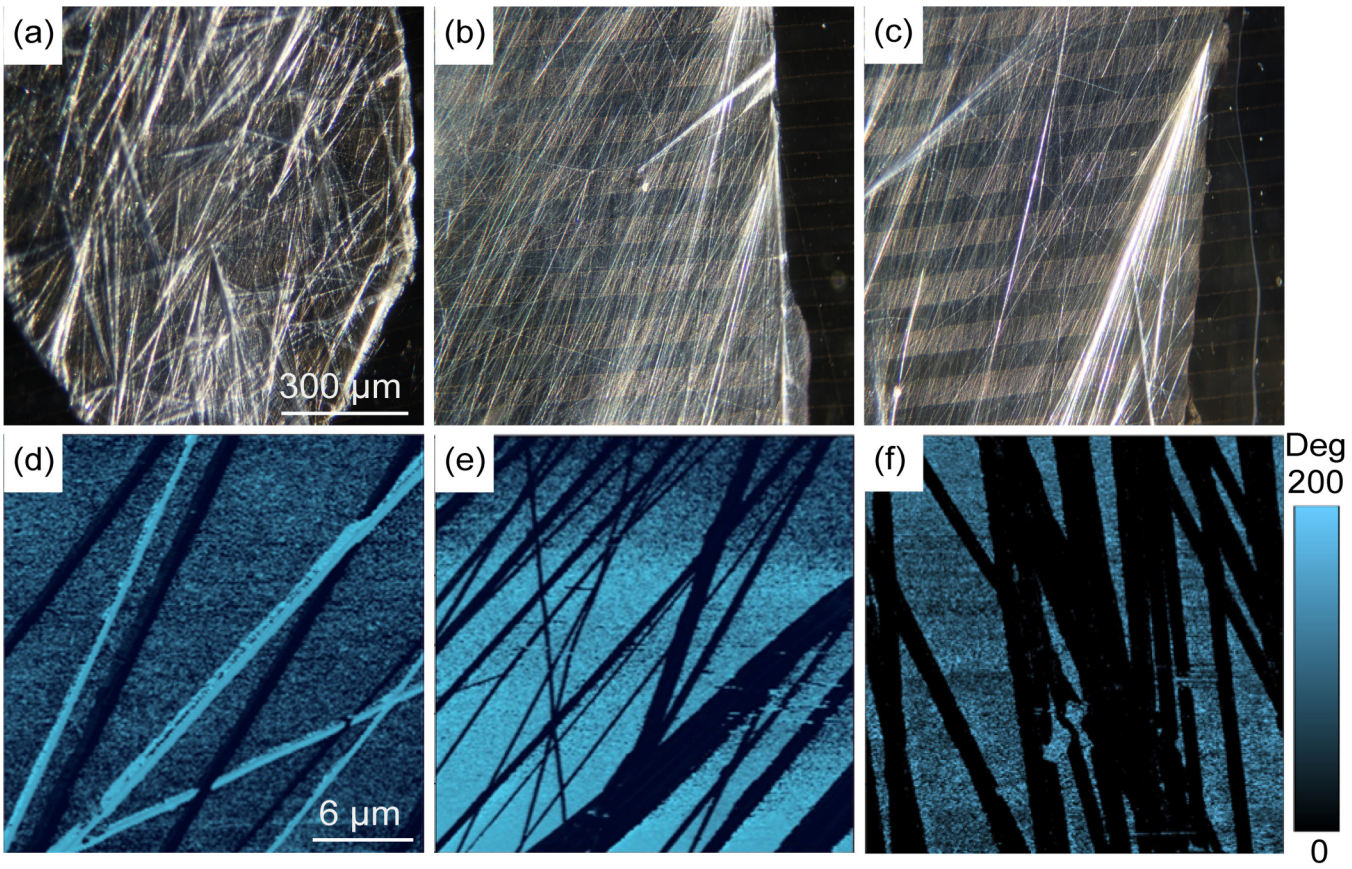

FIG. 2. (a)–(c) Optical and (d)–(f) lateral piezoresponse force microscopy (LPFM) phase images of the PNTs on interdigitated electrode substrates: without alignment [(a) and (d)], aligned using both electric field and UV/ozone [(b) and (e)], and aligned PNTs with GO using both electric field and UV/ozone [(c) and (f)].

flexible substrate during UV/ozone (ProCleaner, BioForce) exposure for 35 min, resulting in the growth of a hydrophilic oxide layer on the unmasked surface. A 3D-printed mask was used to make samples with three 0.2 cm × 0.9 cm openings. A contact angle measuring system (DSA10, Kruss) was used to measure the contact angle (typically ∼68° before exposure and ∼5° after).

Several types of PNT-based flexible substrates, both with and without GO, were prepared, as described below. PNTs were prepared with (UV/ozone)[21] and without (random) a UV/ozone-defined wettability difference, and also with and without an applied electric field and combinations thereof. For substrates where the electric field was not applied, the electrodes were not deposited.

GO was prepared by mixing single layer graphene oxide (SLGO, Cheap Tubes) in de-ionized water at a concentration of 1 or 2 mg/ml. PNT/GO solutions were prepared by adding 40 μl of the GO solution to 60 μl of 2 mg/ml of the PNT solution heated at 100 °C for 2 min.[21] The PNT/GO solution was then stirred for 3 min. 7.5 μl of the PNT or PNT/GO solutions (heated at 100 °C for ∼5 min) was then deposited on the flexible substrate (with or without UV/ozone patterning and with or without applied electric field) for one [see Fig. 1(a)], two, or three openings.

Prior to the application of an electric field, the outer electrodes were electrically isolated from the interdigitated electrodes using tweezers to cut the electrodes. While the PNT or PNT/GO solution was being placed on the flexible substrate, 5 V, chosen based on preliminary experiments and corresponding to an electric field of ∼7 V/cm, was applied (E3631A, Agilent) across the outer electrodes, which were contacted at the substrate edge using silver

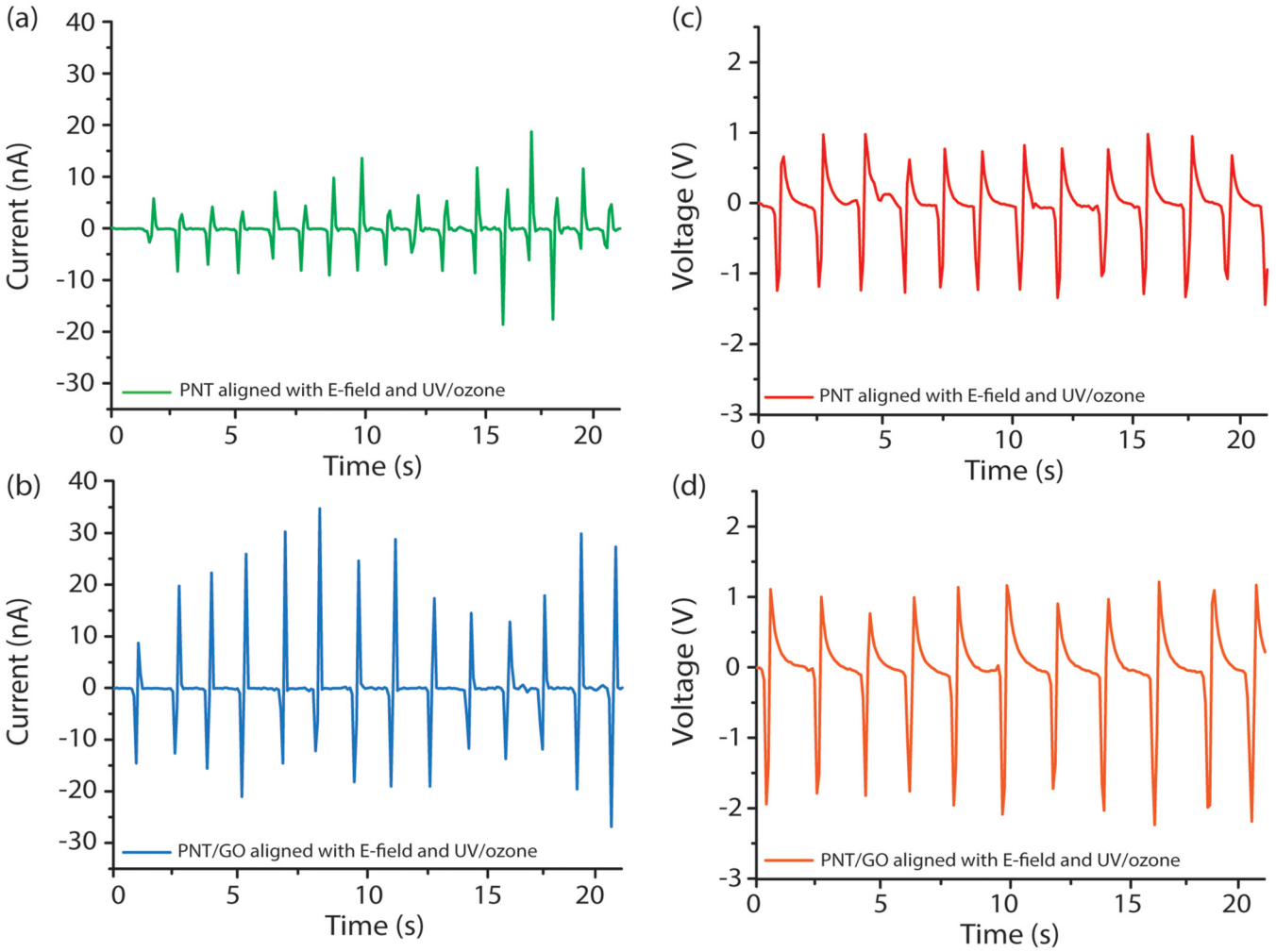

FIG. 3. (a) and (b) Short-circuit current output for PNTs prepared on a flexible substrate with interdigitated electrodes made with one opening and aligned with electric field and UV/ozone without and with 1 mg/ml GO, respectively. (c) and (d) Open-circuit voltage output for PNTs prepared on a flexible substrate with interdigitated electrodes made with one opening and aligned with electric field and UV/ozone without and with 1 mg/ml GO, respectively.

paint [Fig. 1(a)]. After the PNTs self-assembled, electrical connections to the interdigitated electrodes were restored using silver paint and the outer electrodes were electrically isolated using tweezers. The surface wettability difference induced by UV/ozone exposure along with the applied voltage helped align the PNTs across the interdigitated electrodes [Fig. 1(b)].

B. Piezoelectric measurements of PNTs

Piezoresponse force microcopy (PFM) was implemented using an Asylum Research MFP 3D AFM and n-type Si probes with Pt coating (HQ:CSC37/Pt, Mikromasch) with a nominal stiffness of 0.3 N/m and nominal resonance frequency of 20 kHz. A 10 kHz, 30 V AC voltage, which had been amplified using a voltage amplifier (F10A, FLC Electronics AB), was applied to the AFM probe and a lock-in amplifier (HF2LI, Zurich Instruments) was used to measure the in-plane piezoresponse amplitude and phase signals.

C. Measuring piezoelectric output during substrate bending

A Keithley 6514 electrometer with a LabVIEW interface was used to measure open-circuit voltage and short-circuit current. Average current and voltage values are determined from a total of 30 measurements, 10 measurements (peaks) from three samples. Electrical connections to the interdigitated gold electrodes were made using conductive silver paint. The substrates were bent manually using a bending stage [Fig. 1(c)] and the corresponding output was recorded. The stage was moved ∼0.5 cm and back at rates of ∼0.5–1 Hz. Bending the substrate produces both compressive and tensile strains, $\epsilon_x = \pm Y/\rho$, of equal magnitude on the top and bottom surfaces of the substrate, respectively, where Y is the distance from the neutral axis to the surface of the sample and ρ is the radius of curvature during bending.[22] The bending stage clamped 0.7 cm of the substrate. As a result, 1.58 cm of the substrate was subjected to bending and the edge of the flexed substrate typically had a ∼30° angle with respect to the unflexed substrate. Assuming the PNTs bend with the substrate, we estimate the bending stress ($\sigma_x = E\epsilon_x$, where E is Young's modulus) for a radius of curvature of ∼3.0 cm to be ∼2 GPa (assuming a PNT modulus of 19 GPa).[23]

III. RESULTS AND DISCUSSION

A wettability difference created through selective UV/ozone exposure has been reported previously to drive the physical alignment of PNTs from the hydrophobic to the hydrophilic region compared to their random self-assembled arrangement in the absence of a wettability difference.[15] The polarization of the PNTs, as determined from lateral PFM (LPFM) phase images, however, was not found to be uniform. As electric fields were successfully used to achieve uniform polarization (and PFM phase response) from vertically aligned PNTs,[8,9,14] the effect of the applied electric field on the alignment and polarization of horizontally aligned PNTs with and without GO was investigated.

Shown in Fig. 2 are representative images of PNTs prepared without UV/ozone or applied electric field [Fig. 2(a)], PNTs prepared with UV/ozone and applied electric field [Fig. 2(b)], and PNTs prepared with GO, UV/ozone, and electric field [Fig. 2(c)]. It can be seen that PNTs prepared with UV/ozone and electric field on flexible substrates with interdigitated electrodes are aligned even when PNTs are prepared with GO. The chemical force (arising from the wettability difference) and/or the electric force overcome the tendency of the PNTs to self-assemble in random directions, facilitating the fabrication of a more ordered array of PNTs.

The degree of alignment was assessed as the average peak full width at half maximum (FWHM) of the radial summation of the FFT of the optical images (from three images for each sample type), determined by Gaussian fit, as employed previously.[15] The FWHM decreases (and the alignment approves) from 39 ± 3° in the absence of UV/ozone and electric field to 8 ± 1° with both UV/ozone and electric field, and remains at 8 ± 1° when GO is also included. For comparison, the FWHM for PNTs prepared with UV/ozone only was determined to be 13 ± 2°, suggesting that the electric field may offer a slight enhancement in the PNT alignment; in the absence of UV/ozone, the FWHM for electric field alone was 14 ± 3°. Similar values were obtained with GO: 11 ± 1° for UV/ozone and 12 ± 3° for electric field alone. The average angles of the

TABLE I. Current and voltage output measurements of PNTs (2 mg/ml) on flexible substrates with interdigitated electrodes prepared using different conditions: effect of UV/ozone exposure, electric field application, number of openings, and the presence of GO at concentrations of 1 or 2 mg/ml.

				Current output (nA)		Voltage output (V)	
UV/ozone	Electric field	Openings	GO	Positive	Negative	Positive	Negative
…	…	…	…	0.2 ± 0.1	−0.8 ± 0.1	0.12 ± 0.03	−0.14 ± 0.03
✓	…	1	…	7.2 ± 0.7	−5.1 ± 0.6	0.8 ± 0.1	−1.3 ± 0.2
…	✓	1	…	5.1 ± 0.7	−5.5 ± 0.7	0.8 ± 0.1	−1.1 ± 0.2
✓	✓	1	…	18.9 ± 0.9	−16.1 ± 1.4	1.2 ± 0.2	−1.5 ± 0.3
✓	✓	2	…	23.5 ± 1.4	−26.3 ± 0.8	2.1 ± 0.2	−2.1 ± 0.2
✓	✓	3	…	32.4 ± 2.2	−37.5 ± 3.8	2.4 ± 0.7	−4.0 ± 0.5
✓	…	1	1 mg/ml	29.2 ± 1.8	−19.6 ± 0.9	1.3 ± 0.2	−1.8 ± 0.2
…	✓	1	1 mg/ml	25.8 ± 1.4	−16.8 ± 0.8	1.2 ± 0.1	−1.6 ± 0.1
✓	✓	1	1 mg/ml	39.2 ± 1.4	−25.7 ± 1.4	2.2 ± 0.2	−2.8 ± 0.5
✓	✓	3	2 mg/ml	41.3 ± 1.3	−63.8 ± 1.4	5.1 ± 0.8	−6.6 ± 0.5

PNTs with respect to the interdigitated electrodes were estimated using FibrilTool in ImageJ to be 71° for PNTs in Fig. 2(b) and 78° for PNTs with GO in Fig. 2(c).

Representative PFM phase images of PNTs prepared without alignment and with UV/ozone and electric field for PNTs and PNTs with GO are shown in Figs. 2(d)–2(f). The PFM phase images provide information on the polarization direction of the PNTs.[24] The LPFM phase response can be considered in-phase or out-of-phase with respect to the driving voltage and depends on the orientation of the PNT and thus the sign of the shear piezoelectric coefficient. For convenience, we assign the dark contrast on the PNTs as being in-phase. In the absence of UV/ozone and electric field, PNTs can be seen to deform in-phase and out-of-phase [dark and bright contrast in Fig. 2(d)]. Under the influence of UV/ozone and electric field, both PNTs and PNTs with GO appear to align with uniform polarization directions and deform in-phase with the applied voltage [dark contrast in Figs. 2(e) and 2(f)]. We note that these images were measured in less-dense regions of the samples where the PNTs are isolated (as typically shown in the literature[13]) and that it was not possible to map the polarization across a sample

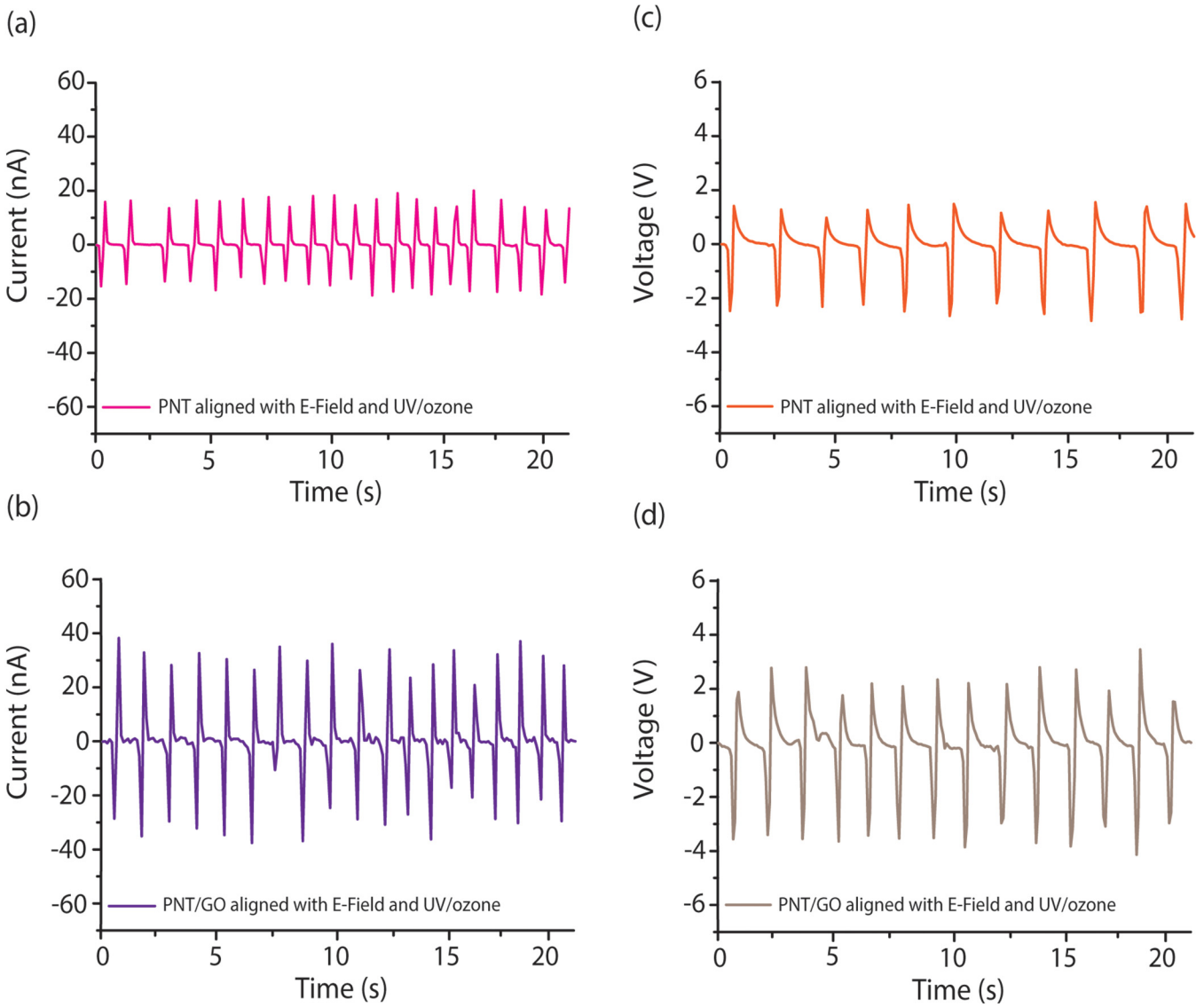

FIG. 4. (a) and (b) Short-circuit current output for PNTs prepared on a flexible substrate with interdigitated electrodes made with three openings and aligned with electric field and UV/ozone without and with 1 mg/ml GO, respectively. (c) and (d) Open-circuit voltage output for PNTs prepared on a flexible substrate with interdigitated electrodes made with three openings and aligned with electric field and UV/ozone without and with 1 mg/ml GO, respectively.

due to the high density of the PNTs, which makes contact mode PFM difficult to perform. Second harmonic generation[25,26] imaging might be used in future work to verify the polarization alignment. We suspect the electric field enhances the polarization ordering of horizontally aligned PNTs, as has been reported previously for vertically aligned PNTs,[8,27] and now investigate the influence of electric field and GO on the energy harvesting capabilities of aligned PNTs on flexible substrates.

Current and voltage outputs for PNTs prepared with applied electric field and UV/ozone exposure through one opening are shown in Fig. 3. Upon bending, negative current and voltage outputs are obtained, followed by positive outputs upon release, in agreement with Ref. 12. The average current output was -16.1 ± 1.4 nA and 18.9 ± 0.9 nA for the negative and positive current peaks, respectively, whereas the corresponding voltages were -1.5 ± 0.3 V and 1.2 ± 0.2 V. These values are roughly within a factor of two of those obtained in other studies for pressed vertically aligned PNTs (39.2 nA and 1.4 V),[8] flexed vertically aligned PNTs (~7 nA and 0.6 V),[9] and pressed horizontally aligned PNTs (37.4 nA and 2.8 V);[12] they are greater than values reported for flexed horizontally aligned PNTs (250 pA and 0.1 V).[12] Samples prepared with UV/ozone alone, electric field alone, and in the absence of UV/ozone and electric field are compared in Table I. Randomly oriented PNTs prepared in the absence of UV/ozone exposure and electric field application had a low response (greatest magnitudes of 0.8 nA and ~0.1 V). Both UV/ozone alone and electric field alone provided a modest response, with a slight advantage to UV/ozone alone (greatest magnitudes of 7.2 nA and 1.3 V compared to 5.5 nA and 1.1 V). Notably, currents for UV/ozone and electric field aligned PNTs are ~47 times higher than for randomly oriented PNTs, whereas voltages are ~28 times higher.

To investigate the influence of number of channels, coverage area, and volume of PNT solution (7.5 μl per channel), samples were prepared having one 0.3 cm × 0.9 cm opening (0.27 cm) vs two 0.3 cm × 0.9 cm openings (0.54 cm^2) or three 0.2 cm × 0.9 cm openings (0.54 cm^2). The current and voltage increased with increasing number of channels and volume, with the greatest magnitudes (37.5 ± 3.8 nA; 4.0 ± 0.5 V) recorded for three channels (Table I and Fig. 4).

To investigate the influence of including GO on the outputs, we first looked into devices with one channel. The addition of 1 mg/ml of GO when just one of UV/ozone or electric field was applied led to increased outputs—currents are notably ~3–5 times higher when GO is included. The outputs increased further when both UV/ozone and electric field were applied and were significantly higher when GO was included compared to without GO. Outputs for one 0.3 cm × 0.9 cm opening (0.27 cm^2) increased significantly for three 0.2 cm × 0.9 cm openings (0.54 cm^2) when the GO concentration increased to 2 mg/ml (Fig. 4). The negative outputs upon bending reached -63.8 ± 1.4 nA and -6.6 ± 0.5 V. Unfortunately, data for three channels and 1 mg/ml GO were not obtained in this study; further investigations should be performed to understand the impact of volume, area, and concentration on the outputs.

Inclusion of GO has been reported to affect the appearance and properties of PNTs aligned via a wettability difference.[21] We previously reported that the diameter of PNTs reduced when prepared with GO from ~4 μm to ~1 μm.[21] Moreover, for samples prepared on SiO_2, resistance decreased with increasing GO concentration from ~930 Ω for PNTs alone to 547 Ω and 466 Ω for GO concentrations of 1 mg/ml and 2 mg/ml, respectively.[21] We expect that the conductivity of the PNTs prepared on flexible interdigitated electrode substrates would similarly increase with the incorporation of GO. In fact, other studies have shown that energy harvesting outputs can be enhanced by increasing the conductivity through the incorporation of carbon nanomaterials.[28,29] In this work, as the average piezoelectric responses for PNTs with and without GO were found to be within 6% of each other, we conclude that the increased conductivity improved the current and voltage outputs.

IV. CONCLUSION AND OUTLOOK

Energy harvesting using graphene oxide (GO)-doped and undoped horizontally aligned piezoelectric diphenylalanine peptide nanotubes (PNTs) prepared on flexible substrates has been demonstrated. The largest magnitude outputs are achieved when the substrates are prepared using both UV/ozone to create a wettability difference that drives the physical alignment of the PNTs and an applied electric field that likely acts to align also the PNT polarity. In the presence of GO, as a result of increased conductivity, open-circuit voltages and short-circuit currents exceeding 6 V and 60 nA, respectively, are achieved, highlighting the potential of UV/ozone and electric field aligned PNT/GO nanocomposites for flexible energy harvesting applications. While in this work, L-diphenylalanine was used, it should be noted that the self-assembly and mechanical, structural, and piezoelectric properties of peptide structures formed from diphenylalanine and diphenylalanine derivatives can vary depending on chirality and aromaticity.[30–34] Thus, future work should explore the design and optimization of peptide structures for efficient energy harvesting from bending flexible devices.

ACKNOWLEDGMENTS

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DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

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