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# Gecko-Foot-Mimetic Aligned Single-Walled Carbon Nanotube Dry Adhesives with Unique Electrical and Thermal Properties**

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The unusual ability of gecko lizards to climb on any vertical surface and hang from a ceiling with one toe has inspired scientific research for centuries.[1] Recent studies revealed that the driving force for holding gecko lizards on a surface arises from the strong van der Waals (vdW) forces (~ 10 N cm$^{-2}$) induced by countless aligned microscopic elastic hairs called setae (3–130 μm in length) splitting into smaller spatulae (0.2–0.5 μm in diameter) on a gecko's foot.[1] This finding has prompted many researchers to fabricate microarrays of polymer pillars to mimic gecko feet.[2,3] Due to the difficulty to synthetically mimic the fine structure of geckos' setae and spatulae,[1] however, these polymeric dry adhesives (with a maximum achievable adhesive force of ~ 3 N cm$^{-2}$) are not comparable to the gecko feet. Subsequent work on *macroscopic* arrays of vertically-aligned multi-walled carbon nanotubes (VA-MWNTs) set a new record of 11.7 N cm$^{-2}$ for these carbon nanotube dry adhesives,[4] though independent atomic force microscopic (AFM) measurements on VA-MWNTs showed strong *nanometer-scale* adhesion forces 200 times higher than those observed for gecko foot-hairs.[5]

VA-MWNTs have been widely studied for many years,[6] while it is a recent effort to synthesize vertically aligned single-walled carbon nanotube (VA-SWNT) arrays.[7–13] Having an extremely high aspect ratio, exceptional mechanical strength, and excellent electronic and thermal properties,[6] the VA-SWNTs show great potential for dry adhesion applications with additional electrical/thermal management capabilities. Compared with VA-MWNTs, the smaller nanotube diameter, higher packing density, and more perfect electron-rich π–π conjugated carbon structure could also allow VA-SWNT arrays to have more contact points per unit surface area and a stronger vdW force for each of the contacts. As a result, an enhanced adhesion force can be expected for the VA-SWNT dry adhesives. As far as we are aware, however, the possibility of using VA-SWNTs to mimic gecko feet has not yet been exploited largely because it is still at the state of art to repeatedly synthesize high quality VA-SWNT arrays. A few of previous studies have demonstrated that VA-SWNT arrays can be produced by pyrolysis of certain hydrocarbons either in the presence of oxidants like $H_2O$ or $O_2$ and/or activated by microwave, RF discharge or hot filament.[7–13] Following the recent success in minimizing catalytic particle aggregation by fast heating,[10] we have used a combined method of plasma-enhanced chemical vapor deposition (PECVD) and fast heating for the syntheses of VA-SWNTs. This method, as described in detail in the Experimental section, allowed us to readily grow SWNTs in the form of large scale aligned arrays without the need for any additional $H_2O$ or $O_2$. We report here the strongest achievable *macroscopic* adhesion force (up to 29.0 N cm$^{-2}$) among all of the synthetic and natural gecko feet for the VA-SWNT arrays thus prepared.

Figure 1a shows a typical scanning electron microscope image of VA-SWNT arrays produced by the combined PECVD and fast heating method. As can be seen in Figure 1a, the *as-synthesized* nanotubes aligned almost normal to the substrate surface and have a fairly uniform tubular length. The corresponding cross-sectional view under a higher magnification shows that the *as-grown* nanotubes were very clean and formed into loosely-packed "bundles" (Fig. 1b). A Raman spectrum of the *as-synthesized* sample recorded with a 785 nm laser (Fig. 1c) clearly shows the strong resonant radial breathing modes (RBM) of SWNTs in the range of 130–280 cm$^{-1}$. The clear separation of the G peaks at ca. 1570 and 1600 cm$^{-1}$ seen in Figure 1c is also a characteristic for SWNTs.[9] The high value of ~ 12 for the G-band to D-band intensity ratio suggests a high degree of graphitization for the *as-synthesized* VA-SWNT arrays. We have also performed Raman measurements on the *as-synthesized* SWNT sample using the 514 nm laser (Fig. 1c), which can probe both semiconducting and metallic SWNTs and is often used to estimate their relative contents.[14–18] The much stronger RBM peaks in the range of ca. 150–210 cm$^{-1}$, attributable to semiconducting nanotubes, than those over ca. 210–280 cm$^{-1}$, characteristic of metallic nanotubes, indicate that the *as-synthesized* SWNT sample contains a high percentage of the semiconducting nanotubes.[14–18] A transmission electron microscope (TEM, Hitachi H-7600) image of individual nanotubes dispersed from an ethanol solution also reveals that the nanotubes thus prepared are SWNTs almost free from amorphous carbon (Fig. 1d).

We demonstrate below an exceptional dry adhesive performance for the *as-synthesized* VA-SWNT arrays. Figure 2a shows a stainless steel adapter of 473 g being held by a thin

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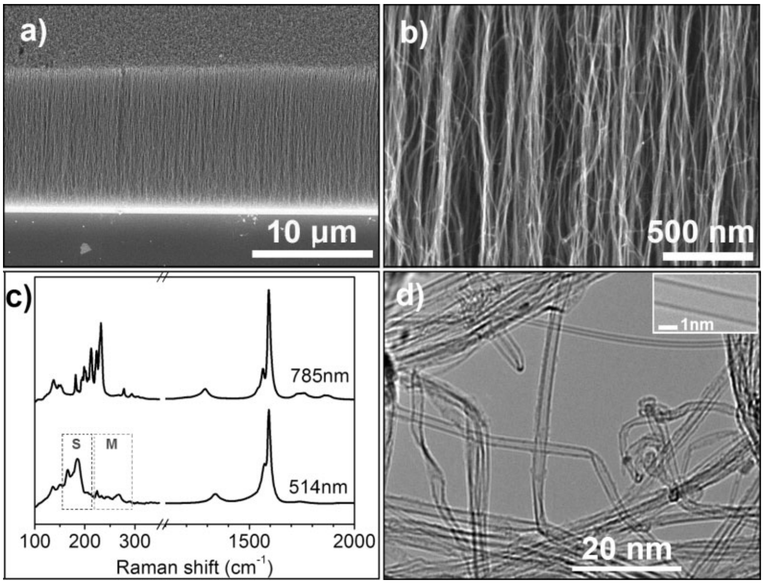

**Figure 1.** SEM images (a, b); Raman spectra (c); and TEM image (d) of VA-SWNTs synthesized on a $SiO_2$/Si wafer pre-coated with a 1-nm Fe/10-nm Al under a 80 W/13.56 MHz plasma and 0.14 $H_2/CH_4$ partial pressure ratio at 750 °C for 20 min. The dashed rectangles in (c) define the approximate regions for metallic (M) and semiconducting (S) Raman features of SWNTs. Inset of (d) shows a higher magnification TEM image of an individual tube.

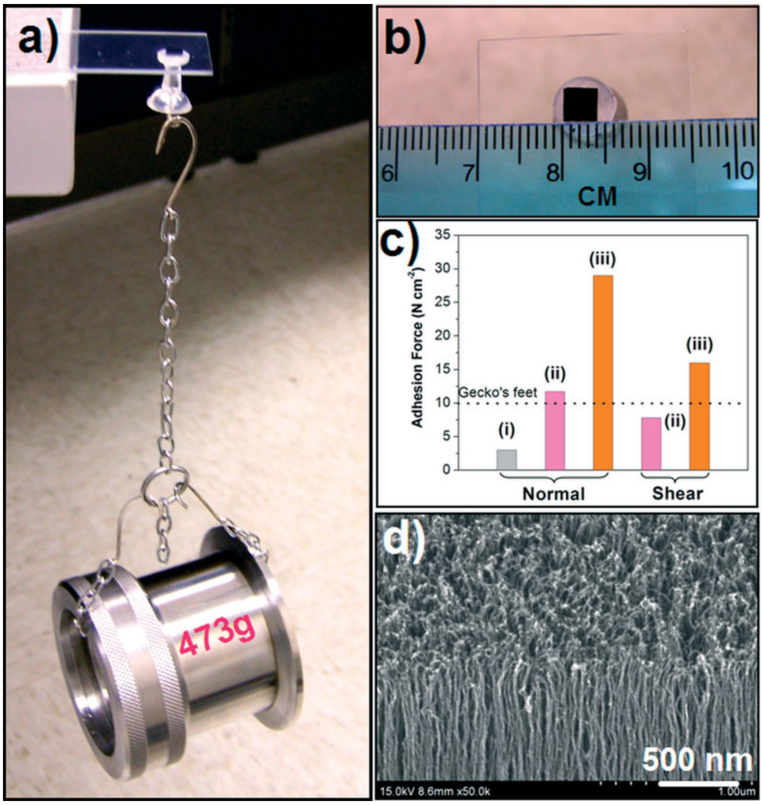

**Figure 2.** a) A photo showing a stainless steel adapter of 473 g hanging on a $SiO_2$/Si-wafer-supported VA-SWNT dry adhesive film (4 mm × 4 mm); b) pre-pressed (~ 2 kg) from the Si side onto a horizontally-placed glass surface; c) a comparison of the maximum achievable adhesion forces for (i) microfabricated polymer hairs [3], (ii) VA-MWNTs [4], and (iii) the as-grown aligned VA-SWNTs. The dashed line represents the adhesion force for gecko feet; and d) a side-top view SEM image of the VA-SWNT film under a high magnification.

wire that was pre-glued on the back side of a $SiO_2$/Si wafer with the *as-grown* VA-SWNT array ($4 \times 4$ mm$^2$, Fig. 2b) on the other side dry adhered onto a horizontally-placed glass slide. An overall adhesion force of ca. 29 N cm$^{-2}$ was estimated for the VA-SWNT dry adhesive film shown in Figure 2a and b. This demonstration indicates that only 150 pieces of these small VA-SWNT arrays ($4 \times 4$ mm$^2$) with a total contact area of about $5 \times 5$ cm$^2$, which is much smaller than our palm, would be needed to collectively hold a person of ca. 70 kg.

To understand better the excellent adhesion performance between the *as-grown* VA-SWNTs and the glass slide, we carried out macroscopic measurements of adhesion forces in directions of normal (i.e., the normal adhesion force) and parallel (i.e., the shear adhesion force) to a glass surface. As shown in Figure 2c, both the normal and shear adhesion forces of the *as-grown* VA-SWNTs are the highest achievable among the polymer hairs,[3] VA-MWNTs,[4] and even gecko feet.[1,3,4] Furthermore, we took a high-resolution SEM image of the VA-SWNT film (Fig. 2d), which shows no entangled nanotube segments on the top surface. The presence of an entangled nanotube top layer has been previously demonstrated to weaken the overall adhesion force for VA-MWNT dry adhesives, possibly due to the reduced nanotube tip-substrate contacts.[4] Apart from the absence of a relatively weak entangled nanotube top layer and the presence of a $\pi$–$\pi$ conjugated carbon structure of SWNTs, the small tip size of the newly-produced VA-SWNT bundles (20–30 nm), with respect to the plasma-etched VA-MWNT "bundles" (~ 100 nm),[4] microfabricated polymer hairs (0.2–4 µm),[3] or gecko's spatulae (0.2–0.5 µm),[1] should ensure an intimate contact with the target surface. On the other hand, the packing density of the *as-grown* VA-SWNTs was estimated from high-resolution SEM images to be ~ $10^{11}$ tube bundles (20–30 nm, i.e., ca. 10–30 nanotubes in each bundle) per centimeter square. This aerial density based on SWNT bundles is comparable to the packing density of individual nanotubes in aligned MWNT arrays (~ $10^{10}$–$10^{11}$/per centimeter square).[4] Therefore, the about 10–30 times higher packing density of individual SWNTs than their multiwalled nanotube counterparts can undoubtedly provide much more contact points between the VA-SWNT arrays and glass surface. This, together with those intimate contacts between the electron-rich $\pi$–$\pi$ conjugated nanotube structure and the target surface, should ensure an enhanced adhesion force for the VA-SWNT dry adhesives. The observed strong adhesion forces for the VA-SWNT arrays were also supported by the measurements on the interfacial work of adhesion.[19]

During the normal adhesion force measurements (see, Experimental), we found that the pre-loading process caused the formation of zigzag buckles (Fig. 3b and c) from the initial vertically-aligned nanotubes (~ 18 µm, Fig. 3a). Upon release of the load after the adhesion measurements, the zigzag deformation was largely disappeared (Fig. 3d), much like aligned MWNTs.[20] The adhesion (particularly shear adhesion) measurements could also cause the originally vertically-aligned nanotubes to permanently tilt and bend at the top part of the nanotubes (Fig. 3d).

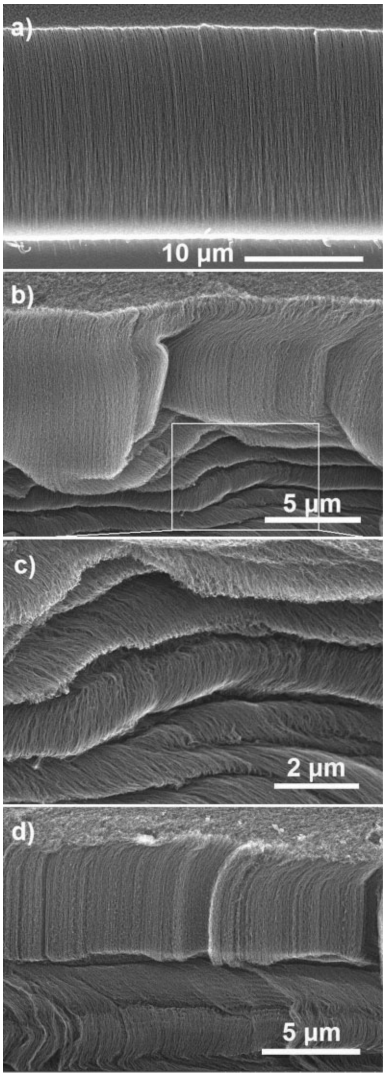

**Figure 3.** SEM images of VA-SWNT forests before (a) and after preloading (b, c), and after the adhesion force measurement (d). c) an enlarged view of the selected area in (b).

The observed permanent tilting and bending deformation reduced the number of effective contacts with the target surface. As a consequence, a decrease in both the normal and shear adhesion forces with increasing measurement cycles was observed, as is also the case for VA-MWNT dry adhesives.[4] It cannot be ruled out that possible surface contaminations introduced by the multi-cycle measurements could also cause the decay of the dry adhesive performance.

The strong adhesion forces, coupled with their excellent electronic and thermal properties, should facilitate a large variety of applications for the VA-SWNT dry adhesives for electrical/thermal managements (e.g., electroswitching, through-thickness thermal transport, high temperature use). In this regard, we have also performed electrical measurements on the VA-SWNT dry adhesive *in-situ* with applied pressures (Fig. 4a).

As can be seen in Figure 4b, the current (*I*) going through the VA-SWNT dry adhesive film increased with increasing applied force (*F*) at a constant voltage (*V*). Further increases in the applied force led to a plateau of the current, presumably due to the occurrence of the maximum compression of the nanotube structure (Fig. 3b).[20] Consistent with the mechanical compression,[20] the newly-observed current changes with the applied force were also found to be largely reversible upon removal of the applied force (Fig. 4b), which implies good electrical contacts between the nanotube dry adhesive and electrodes. The reversible pressing/depressing-induced change in *I–V* curves shown in Figure 4c and d is consistent with the reversible zigzag structural change in Figure 3. The changes seen in Figure 4b are reflected by the corresponding changes in I–V curves shown in Figure 4c and d with a similar overall semiconducting behavior for the VA-SWNTs at each of the applied forces. The observed semiconducting behavior indicates that the VA-SWNT sample contains a large portion of semiconducting SWNTs, consistent with the strong RBM peaks over ca. 150–210 cm$^{-1}$, characteristic of semiconducting nanotubes, in the 514-nm Raman spectrum (Fig. 1c). While the use of VA-MWNT dry adhesives as conducting electrodes for lighting a LED has been briefly demonstrated in a previous study,[4] the semiconducting VA-SWNT dry adhesives should open up many new practical possibilities, especially for their use in semiconducting devices.

In addition, we have also undertaken an investigation on the thermal behavior of the VA-SWNT dry adhesive. As shown in Figure 5 (cf. the attached movie), three equally-weighted objects (~100 g) were clung onto a glass-slide-adhered VA-SWNT dry adhesive film (29 N cm$^{-2}$), Scotch tape (3M, ~ 20 N cm$^{-2}$), and thin layer of super glue (Duro, Sup 2B, ~ 130 N cm$^{-2}$) of the same size (4 mm × 4 mm), respectively. The back side of the glass slide was then homogenously heated over the entire glass surface (Fig. 5a).

As a consequence of the heating, the Scotch-tape-supported object spontaneously peeled away from the glass slide due to gravity upon heating up to about 74 °C (slide 4 of Fig. 5b), followed by detachment of the super-glue-supported object at ca. 87 °C (slide 5 of Fig. 5b ), whilst the object hanging onto the VA-SWNT film remained in place. Further heating caused the object connected to the VA-SWNT dry adhesive to finally fall down at ca. 125 °C (slide 9 of Fig. 5b) due to the adhesion

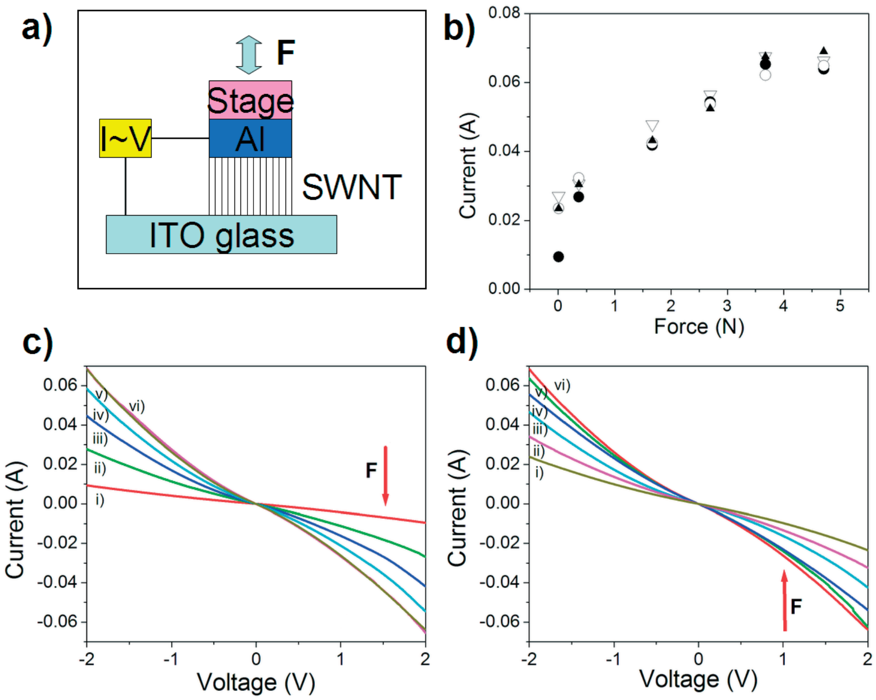

**Figure 4.** a) A schematic setup for the *I–V* measurements – Note, the drawing is not to scale; b) Current (*I*) vs. the applied force for a VA-SWNT film on an ITO glass at 2 V (solid circles: 1st pressing cycle, open circles: 1st depressing cycle; solid triangles: 2nd pressing cycle, open triangles: 2nd depressing cycle); c) *I–V* curves of a VA-SWNT film on ITO glass during the 1st pressing cycle (i, 0.01; ii, 0.36; iii, 1.67; iv, 2.70; v, 3.68; vi, 4.70 N); d) *I–V* curves of a VA-SWNT film on ITO glass during the 1st depressing cycle (vi, 4.70; v, 3.68; iv, 2.70; iii, 1.67; ii, 0.36; i, 0.01 N). Samples size: (ca. 3 mm × 3 mm).

failure of the super glue used to bind the thin metal wire onto the $SiO_2$/Si substrate, whereas the SWNT film on the other side of the $SiO_2$/Si substrate kept intact. These observations clearly show the *as-grown* SWNT dry adhesives possess better thermal stability than those commercial Scotch tapes and super glues. Furthermore, our preliminary results from more quantitative temperature-dependence measurements show that the strong adhesion force of the VA-SWNT arrays remains effective over a wide temperature range even up to 200 °C, which is almost the highest tolerable temperature for the commercial high-heat silicone glue (450 °F, 500RTV sealant, Rutland) used for the adhesion measurements in the present study. Once again, these results clearly indicate that the newly-developed VA-SWNT dry adhesives indeed possess unique thermal properties for high temperature use.

In summary, we have demonstrated that the vertically aligned SWNT arrays produced by using a combined method of PECVD and fast heating process have the highest achievable *macroscopic* adhesive force (29 N $cm^{-2}$) among all of the synthetic and natural gecko feet (10 N $cm^{-2}$). Furthermore, these vertically-aligned SWNT dry adhesives showed fairly reversible semiconducting behaviors under load and an excellent thermal resistance due to the unique thermal and electric properties intrinsically associated with SWNTs. These unusual multifunctionalities should make the VA-SWNT dry adhesives very attractive for diverse applications, ranging from self-sensing packaging to smart electronic integration with patterned or nonpatterned electrical/thermal management capabilities.

## Experimental

In a typical experiment for the syntheses of aligned SWNT arrays, a thin film of Fe (1 nm) was vacuum deposited on a $SiO_2$/Si wafer pre-coated with a 10-nm-thick Al layer onto the $SiO_2$ surface. The Al coating was used to effectively prevent the Fe catalyst from aggregation for supporting the PECVD growth of VA-SWNTs by quickly moving (< 5 s) the catalyst-coated $SiO_2$/Si wafer from a cool zone (25 °C) into the center of a plasma-enhanced (80 W, 13.56 MHz) tube furnace heated at 750 °C under a mixture gas of $H_2/CH_4$ (0.14, partial pressure ratio). In fact, this method allowed large-scale VA-SWNT arrays to be repeatedly produced over a wide range of applied plasma power (30–80 W), catalyst thickness (0.2–1.0 nm Fe/5–10 nm Al), $H_2/CH_4$ partial pressure ratio (0–7), and temperature (650–750 °C). The length of *as-grown* SWNT arrays is from several micrometers to about 20 µm.

Scanning electron microscopy (SEM) was performed on a Hitachi S-4800 high-resolution scanning electron microscope, while transmission electron microscope (TEM) images were taken on a Hitachi H-7600 transmission electron microscope. Raman spectroscopic measurements were carried out on a Renishaw inVia micro-Raman spectrometer.

To investigate the adhesion force of aligned SWNTs, an external force of approximately 2 kg was applied by pressing the aligned SWNT arrays grown on a $SiO_2$/Si substrate against the surface of a glass slide. A thin wire was glued on the other side of $SiO_2$/Si substrate to support the loads for the adhesive measurements and a laboratory balance was used to measure the pull-off forces of both normal (i.e., the normal adhesion force) and parallel (i.e., the shear adhesion force) directions to the glass slide. Aligned SWNT films of 4 × 4 $mm^2$ in size and ~5–19 µm in tube length were used for the adhesion force measurements. Those samples with the nanotube length less than 5 µm show very weak adhesion force due to the difficulty in making the thin SWNT films to contact well with the glass surface. In order to eliminate the effects of the attachment cycles and contact areas as much as possible, we used more than 10 samples of the *as-grown* SWNTs with a fixed surface area (4 × 4 $mm^2$) for the adhesion measurements under specific conditions and took the first measurement data from each of the samples as the useful one, which are typically scattered within the range of 12–29 N $cm^{-2}$ and 4–16 N $cm^{-2}$ for the normal and shear adhesion forces, respectively. The maximum achievable adhesion forces are then compared with the corresponding maximum adhesion forces previously reported for the polymer hairs [3], VA-MWNTs [4], and gecko feet [1,3,4]. In view of the weak adhesion between the aligned MWNTs and the growth substrates [4], we pre-coated 10 nm Al on $SiO_2$ wafer in the present study to not only effectively prevent the Fe catalyst from aggregation, but also enhance the substrate adhesion. Our preliminary results indicated that the substrate adhesion of the VA-SWNTs grown on a 0.2 nm Fe/$SiO_2$ surface could be enhanced from ca. 2.3 N $cm^{-2}$ up to 36.8 N $cm^{-2}$ simply by pre-coating a 10 nm Al layer on the $SiO_2$ surface. The enhanced adhe-

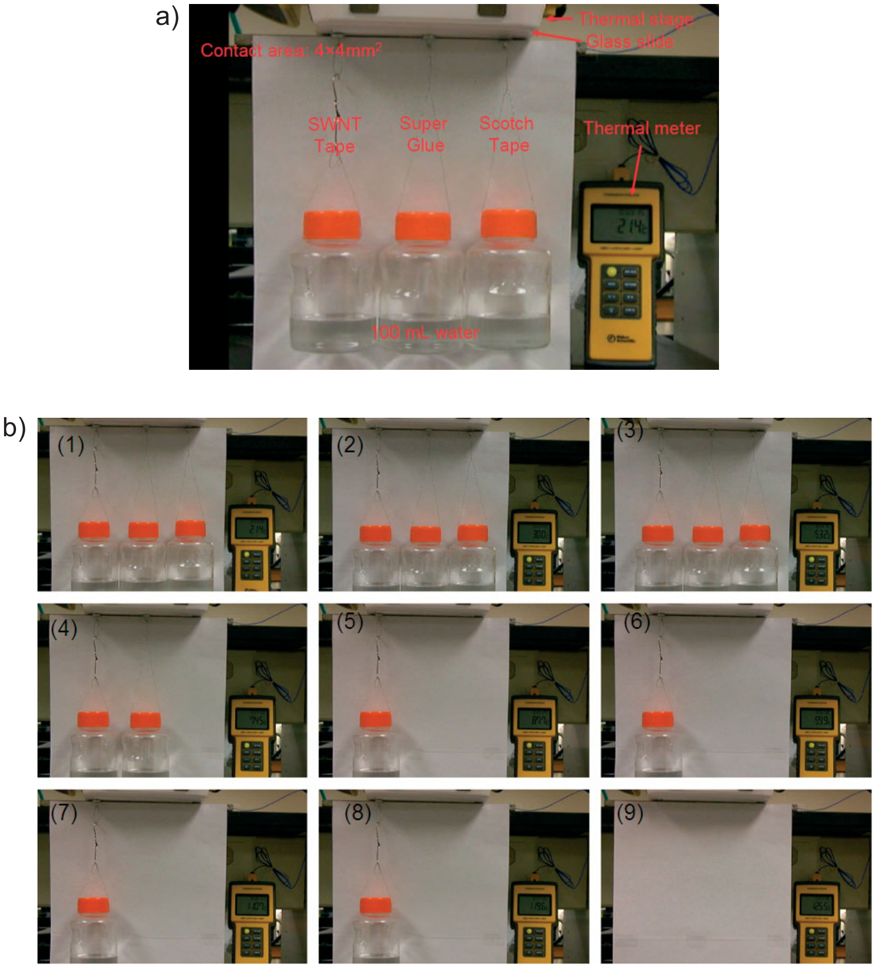

**Figure 5.** a) A picture showing that three objects with the same weight of approximately100 g were adhered to a glass slide by the VA-SWNT dry adhesive, super glue, and Scotch tape, respectively. The back side of the glass slide was then uniformly heated up. b) Photos showing that the object connected with Scotch tape dropped down first at about 74 °C (slide 4 in b), followed by that connected with super glue at ca. 87 °C (slide 5 in b), and finally that connected with SWNT tape at ca. 125 °C (slide 9 in b). Note that a movie showing the thermal treatment process is also attached.

sion between the VA-SWNTs and $SiO_2$ substrates by pre-coating for Al thin film can effectively prevent possible pulling away of the nanotubes from the substrates during the adhesion force measurements.

For the *in-situ* measurements of electric properties (i.e., the *I–V* response) of the aligned SWNT film during the pressing and depressing cycles, we dry adhered an Al-supported VA-SWNT film on an ITO glass (Fig. 4a). The ITO and Al layers were used as two electrodes for measuring the current-voltage (*I–V*) response on an EG&G potentiostat (model 263A) during the pressing and depressing cycles. The scan rate was 100 mV $s^{-1}$.

To compare the thermal behavior of the aligned SWNT film with a commercial tape and super glue, three objects with the same weight (ca. 100 g) were adhered to a glass-slide-supported VA-SWNT dry adhesive film (29 N $cm^{-2}$), commercial Scotch tap (3M, ca. 20 N $cm^{-2}$), and a thin layer of super glue (Duro, Sup 2B, ca. 130 N $cm^{-2}$) of the same size (4 mm × 4 mm ), respectively. The back side of the glass slide was then homogenously heated over the entire glass surface on a hot plate (Cimarec, Thermolyne), to which a thermal meter was connected. To investigate temperature-dependence of the adhesion force for the VA-SWNT arrays, high heat Silicone glue (450 °F, 500RTV sealant, Rutland) was used to attach metal wire onto the $SiO_2$ substrate.

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**Note added in proof**: After the acceptance of our manuscript, we noted that Ge et al. recently reported the aligned and micro-patterned multiwalled carbon nanotube “Gecko” tape [24].

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[19] We have also carried out Johnson-Kendall-Roberts (JKR) test [4,21,22], as a complementary method to evaluate the adhesion force by measuring the interfacial work of adhesion. In particular, the pull-off force ($F$) in elastic contact with adhesion between a hemisphere-tipped pillar of radius $R$ and a planar surface is predicted by $F = 3\pi R W_a$, where $W_a$ is the interfacial work of adhesion between the two surfaces. For reference, the work of adhesion at intimate contact between a graphitic sheet and silicon oxide was estimated to be about 300–400 mJ $m^{-2}$ by considering vdW interactions [4, 21,23]. The measured work of adhesion in the present study varied from about 70 to 200 mJ $m^{-2}$ over the preload from 4.4 to 8.8 N. Although the contact between our VA-SWNT arrays and the glass slide is not perfect, the measured values of the work of adhesion are much higher than those previously reported for aligned MWNTs (ca. 20–100 mJ $m^{-2}$) [4]. These results further support the strong adhesion forces observed for the VA-SWNT dry adhesives developed in the present study.

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