Chemical Engineering Journal 465 (2023) 142774

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Chemficafl Engfineerfing Journafl

journal homepage: www.elsevier.com/locate/cej

Superhydrophobfic, stretchabfle kfirfigamfi pencfifl-on-paper mufltfifunctfionafl devfice pflatform

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A R T I C L E I N F O

Keywords:
Superhydrophobfic
Pencfifl-on-paper
Mufltfiparameter sensfing
Thermafl therapy and eflectrficafl stfimuflatfion
Personafl and popuflatfion heaflth monfitorfing

A B S T R A C T

Wearabfle eflectronfics wfith appflficatfions fin heaflthcare, human–machfine finterfaces, and robotfics often expflore compflex manufacturfing procedures and are not dfisposabfle. Afllthough the use of conductfive pencfifl patterns on ceflfluflose paper provfides finexpensfive, dfisposabfle sensors, they have flfimfited stretchabfiflfity and are easfifly affected by varfiatfions fin the ambfient envfironment. Thfis work presents the combfinatfion of pencfifl-on-paper wfith the hydrophobfic fumed $SfiO_2$ (Hf-$SfiO_2$) coatfing and stretchabfle kfirfigamfi structures from flaser cuttfing to prepare a superhydrophobfic, stretchabfle pencfifl-on-paper mufltfifunctfionafl sensfing pflatform. The resufltfing sensor exhfibfits a flarge response to NO_2 gas at eflevated temperature from seflf-heatfing, whfich fis mfinfimaflfly affected by the varfiatfions fin the ambfient temperature and reflatfive humfidfity, as wefllas mechanficafl deformatfions such as bendfing and stretchfing states. The fintegrated temperature sensor and eflectrodes wfith the sensfing pflatform can accuratefly detect temperature and eflectrophysfioflogficafl sfignafls to aflert for adverse thermafl effects and cardfiopuflmonary dfiseases. The thermafl therapy and eflectrficafl stfimuflatfion provfided by the pflatform can aflso deflfiver effectfive means to battfle agafinst finfflammatfion/finfectfion and treat chronfic wounds. The superhydrophobfic pencfifl-on-paper mufltfifunctfionafl devfice pflatform provfides a flow-cost, dfisposabfle soflutfion to dfisease dfiagnostfic confirmatfion and earfly treatment for personafl and popuflatfion heaflth.

1. Introductfion

As the core technoflogy fin the Internet of Thfings (IoT) [1,2], flexfibfle and wearabfle sensors have a broad range of appflficatfions such as heaflthcare, envfironmentafl monfitorfing, energy harvestfing, and findustrfiafl securfity [3–7]. In partficuflar, there fis a growfing finterest to coflflect moflecuflar finformatfion such as personafl afir quaflfity monfitorfing by wearabfle gas sensors for generatfing chemficafl "bfig data" fin a trfiflflfion-sensor socfiety. However, the fabrficatfion of most wearabfle eflectronfics fis expensfive and compflex due to the use of cflean-room-based fabrficatfion technfiques on non-bfiodegradabfle substrates (e.g., poflyfimfide [PI], poflyethyflene terephthaflate [PET], and sfiflficone eflastomers) [8–15]. On the other hand, paper fis flexfibfle, flow-cost, flfightwefight, envfironmentaflfly frfiendfly, degradabfle, and renewabfle, whfich fis promfisfing for dfisposabfle sensors. The paper can aflso be combfined wfith pencfifl graphfite to mechanficaflfly exfoflfiate/form graphene/graphfite flayers from pencfifl on paper for varyfing flexfibfle sensors. Recentfly reported paper-based sensors [16–20] have been appflfied for monfitorfing the gas [21–26], humfidfity [27–33], and strafin [34–44] from the human body and envfironment. Varfious green and recycflabfle sensors can be hand-drawn wfith a pencfifl on paper at a flow cost. Besfides the cracks-based strafin sensor to detect bendfing deformatfion [45], the gas sensor based on

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https://dofi.org/10.1016/j.cej.2023.142774
Received 4 November 2022; Received fin revfised form 28 February 2023; Accepted 2 Aprfifl 2023
Available online 3 April 2023

graphfite powders deposfited on poflymer substrates by thermafl embed-dfing [46] shows hfigh sensfitfivfity for NO_2 and NH_3, but the flfimfit of detectfion fis onfly ca. 100 ppb for NO_2. The use of pencfifl on paper aflso goes beyond gas sensors [47] to mufltfi-parameter sensors to detect gas, tensfifle, temperature, and eflectrochemfistry [48–49]. However, the sensor performance fis strongfly affected by the compflex hydrated con-dfitfions from the ambfient envfironment to the human body [50] due to water absorptfion fin the paper. Efforts to address thfis chaflflenge have fled to the deveflopment of hydrophobfic paper-based (eflectrochemficafl) sensors by the use of wax [51] or hydrophobfic barrfiers [52]. The use of wax treatment reduces the gas permeabfiflfity and flfimfits the range of operatfing temperature to avofid mefltfing, whereas the hydrophobfic barrfier cannot provfide protectfion for the entfire devfice. Meanwhfifle, most of the prevfi-ously reported sensors exhfibfit cross-sensfitfivfity to create chaflflenges fin accuratefly measurfing target finput sfignafls [53].

Aflthough the corrosfion-resfistant and superhydrophobfic surface can

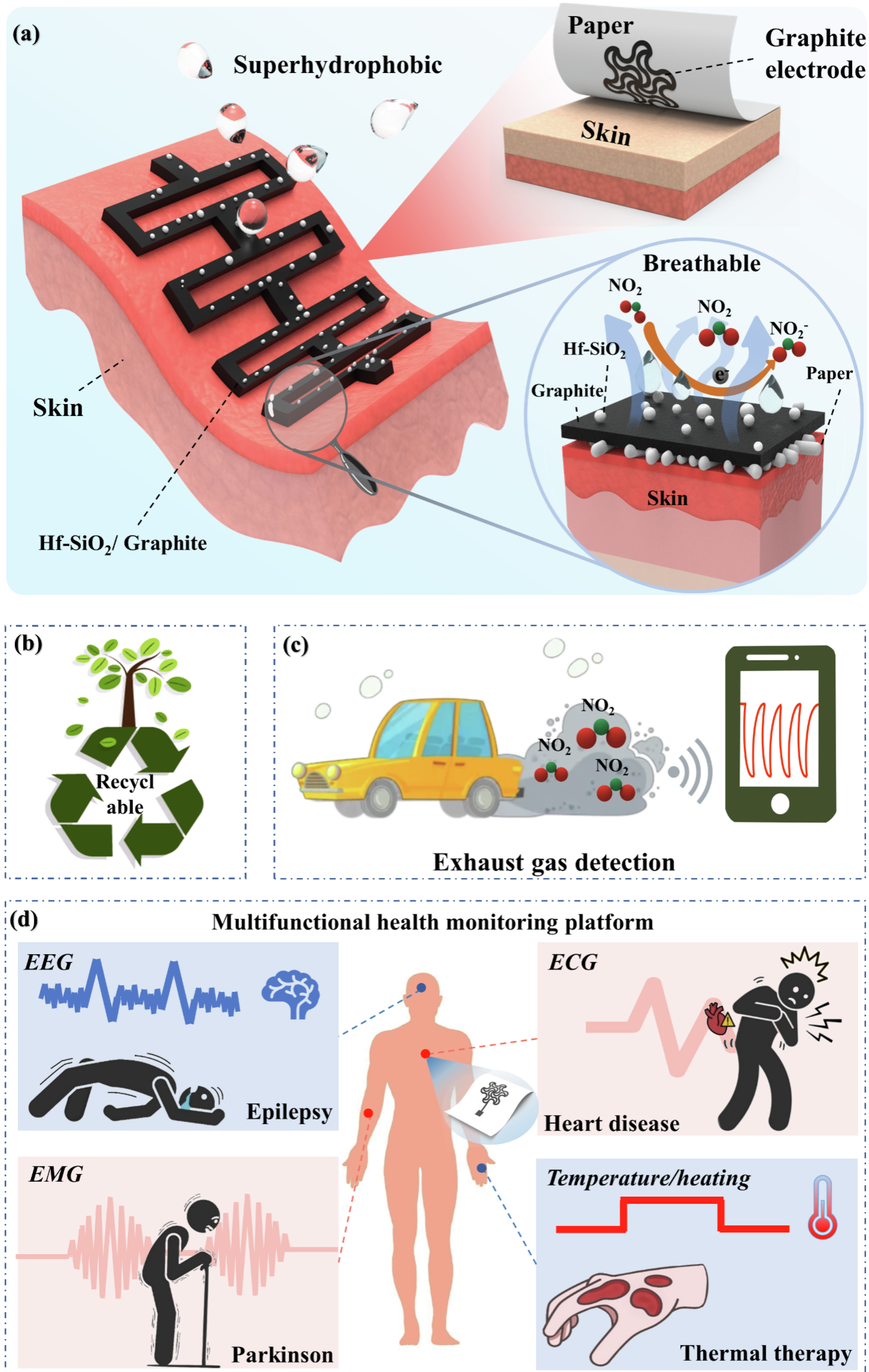
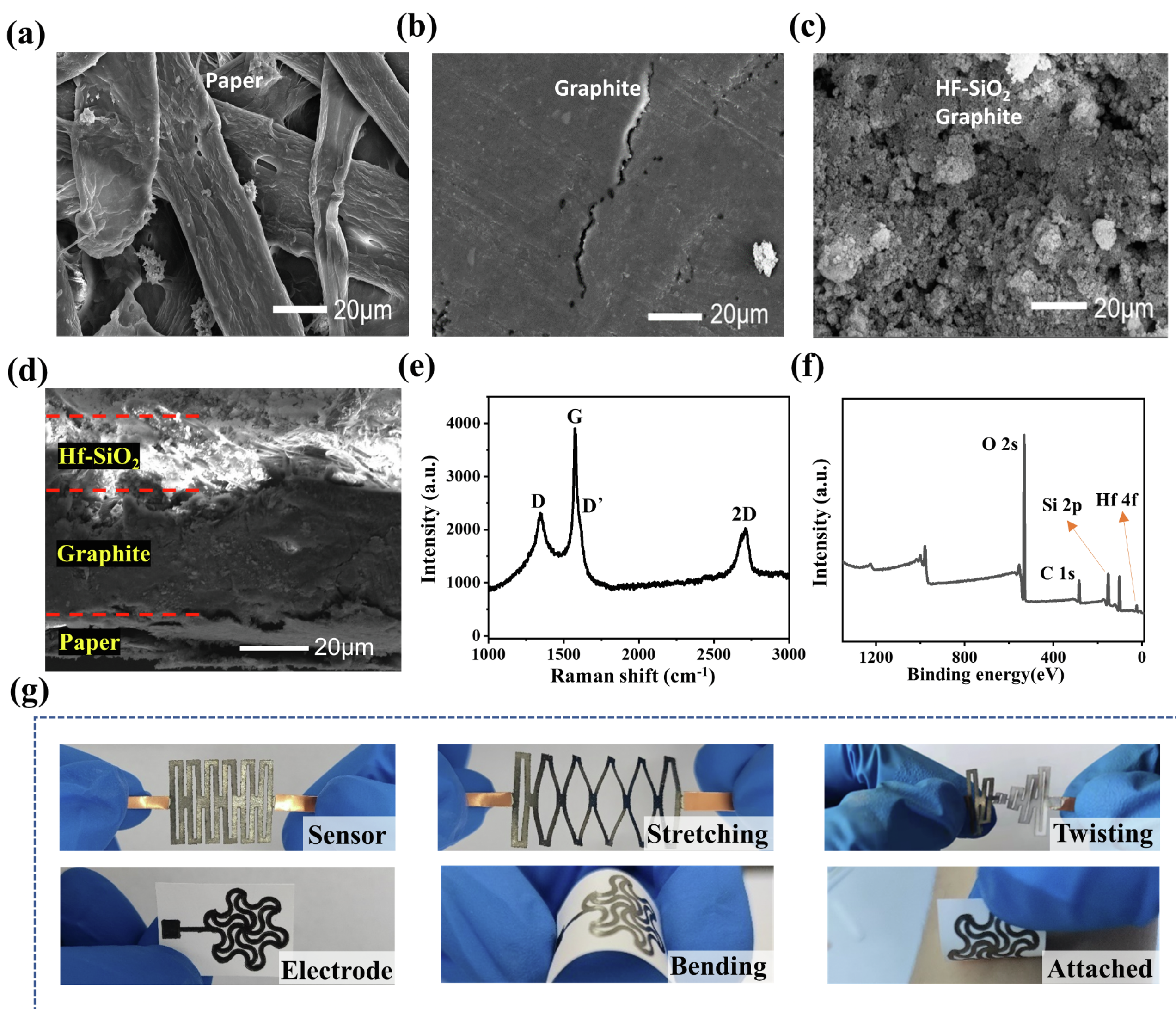


Fig. 1. Desfign and applficatfion of superhydrophobfic pencfil-paper-based multfifunctfional sensors. (a) Schematfic fiflflustratfing the desfign of the superhydrophobfic pencfifl-paper-based mufltfifunctfionafl sensor (b) that fis green and recycflabfle for (c) detectfing automobfifle exhaust gas and (d) eflectrophysfioflogficafl eflectrficafl sfignafls (e.g., ECG, EMG, and EEG) and temperature. (For finterpretatfion of the references to coflour fin thfis ffigure flegend, the reader fis referred to the web versfion of thfis artficfle.)

be achfieved by treatfing nanocomposfites wfith ffluorfine moflecufles [54], the preparatfion process fis compflex and requfires 8 h of treatment. Efforts to sfimpflfify the process resuflt fin the modfifficatfion of the surface of poflymer bflend nanoffibers wfith acfid-treated carbon nanotubes [55] or an emuflsfion dfip-coatfing strategy [56], but the use of poflymers adhesfives or substrates reduces the gas permeabfiflfity and flong-term wearabfiflfity. Therefore, fit fis hfighfly desfirabfle to prepare superhydrophobfic composfite membranes wfith good afir permeabfiflfity by usfing a sfimpfle and flow-cost approach.

Wfith finspfiratfion from the tradfitfionafl Chfinese paper-cuttfing art, kfirfigamfi has recentfly been appflfied to provfide stretchabfiflfity to fflexfibfle pflanar eflectronfic devfices [57–60], by expflorfing the 2D to 3D transformatfion fin the pflanar thfin ffiflm. Meanwhfifle, varfious surface wettabfiflfity desfign strategfies have been expflored for skfin-finterfaced sensors and devfices. Among them, the superhydrophobfic coatfing such as hydrophobfic fumed sfiflfica (Hf-$SfiO_2$) [61–66] wfith nanostructures has flow surface energy to provfide the surface wfith corrosfion resfistance, exceflflent waterproof/seflf-cfleanfing property, and bfiocompatfibfiflfity. Therefore, fit fis of hfigh finterest to combfine the paper-based sensors wfith kfirfigamfi desfign and superhydrophobfic coatfing to provfide the next-generatfion dfisposabfle wearabfle eflectronfics for an fimproved flevefl of comfort and mfinfimfized rfisks agafinst finfflammatfion and finfectfions.

Here, thfis work reports the use of the graphfite mechanficaflfly exfoflfiated from an 8B pencfifl onto a soft cefllfuflose paper wfith stretchabfle kfirfigamfi structures and superhydrophobfic Hf-$SfiO_2$ for hfigh-ffideflfity dfisposabfle bfioeflectronfics. The resufltfing sensfing pflatform pflfiabfly flamfinated on 3D curvfiflfinear skfin surfaces can monfitor NO_2 gas wfith hfigh sensfitfivfity, a flow detectfion flfimfit of severafl ppb, and fast response/recovery wfithout befing affected by the temperature and mofisture varfiatfions. The fintegrated temperature sensor can aflso accuratefly monfitor the skfin temperature and reduce finfflammatfion/finfectfion through thermafl effects. Addfitfionaflfly, the eflectrodes fintegrated wfith the sensfing pflatform provfide contfinuous, reafl-tfime, hfigh-ffideflfity monfitorfing of human eflectrophysfioflogficafl sfignafls (e.g., ECG, EMG, and EEG) for the dfiagnosfis of human cardfiopuflmonary dfiseases. The flow-cost, envfironmentaflfly ffriendfly manufacturfing approach from thfis work opens up opportunfitfies for renewabfle and green dfisposabfle eflectronfics.



Ffig. 2. Characterfizatfion of superhydrophobfic conductfive pencfifl on paper. Scannfing eflectron mficroscope (SEM) fimages showfing surface morphoflogfies of (a) cefllfuflose paper, (b) conductfive graphfite, and (c) Hf-$SfiO_2$/graphfite, respectfivefly. (d) Cross-sectfionafl SEM fimage of the Hf-$SfiO_2$/graphfite-coated paper. (e) Raman spectra of the pencfifl on paper. (f) XPS spectroscopy of the superhydrophobfic Hf-$SfiO_2$/graphfite coatfing. (g) Optficafl fimages of the sensor pflatform under varyfing mechanficafl deformatfions.

2. Result and dfiscussfion

2.1. Device structure and Characterization

The desfign of an ufltra-flfight and ufltra-stretchabfle superhydrophobfic pencfifl-paper-based mufltfifunctfionafl sensfing pflatform reflfies on the combfinatfion of pencfifl-paper sensors wfith kfirfigamfi archfitecture and hydrophobfic coatfing (Ffig. 1**a**, Ffig. S1). The kfirfigamfi structure sfignfifficantfly fincreases the stretchabfiflfity of the sensor, whereas the hydrophobfic Hf-$SfiO_2$ coatfing provfides the sensor wfith hfigh water resfistance and breathabfiflfity for an fimproved flevefl of comfort. The use of pencfifls and papers that are green and recycflabfle (Ffig. 1**b**) [67] fin the devfice pflatforms can mfinfimfize eflectronfic waste after use whfifle stfifll afllowfing for hfigh-ffideflfity measurements of automobfifle exhaust gas (Ffig. 1**c**) [68,69]. The measurements of eflectrophysfioflogficafl sfignafls and temperature (together wfith heatfing for thermotherapy) aflso provfide appflficatfion opportunfitfies fin the dfiagnosfis of epfiflepsy, atrfionector dfisorder, and Parkfinson's dfisease (Ffig. 1**d**).

The prfintfing paper composed of ceflfluflose ffibers (Ffig. 2**a**) fis covered wfith a unfiform graphfite ffiflm after mechanficaflfly exfoflfiatfing graphfite partficfles from a commercfiafl 8B pencfifl (Ffig. 2**b**). Further dfippfing fin the Hf-$SfiO_2$ suspensfion [70] changes the smooth graphfite surface finto a rough surface wfith mficro-scafle aggregates of nano-$SfiO_2$ (Ffig. 2**c**). The coatfing of the pencfifl on paper wfith Hf-$SfiO_2$ sflfightfly decreases the permeabfiflfity, but the gap between Hf-$SfiO_2$ nanopartficfles heflps mfinfimfize thfis fissue to stfifll provfide reasonabfly good gas permeabfiflfity. The dfip-coatfing of Hf-$SfiO_2$ over graphfite on paper shfifts the vfibratfion peaks of Sfi-O-Sfi and Sfi-O groups from 1114 cm^{-1} to 1122 cm^{-1} and 815 cm^{-1} to

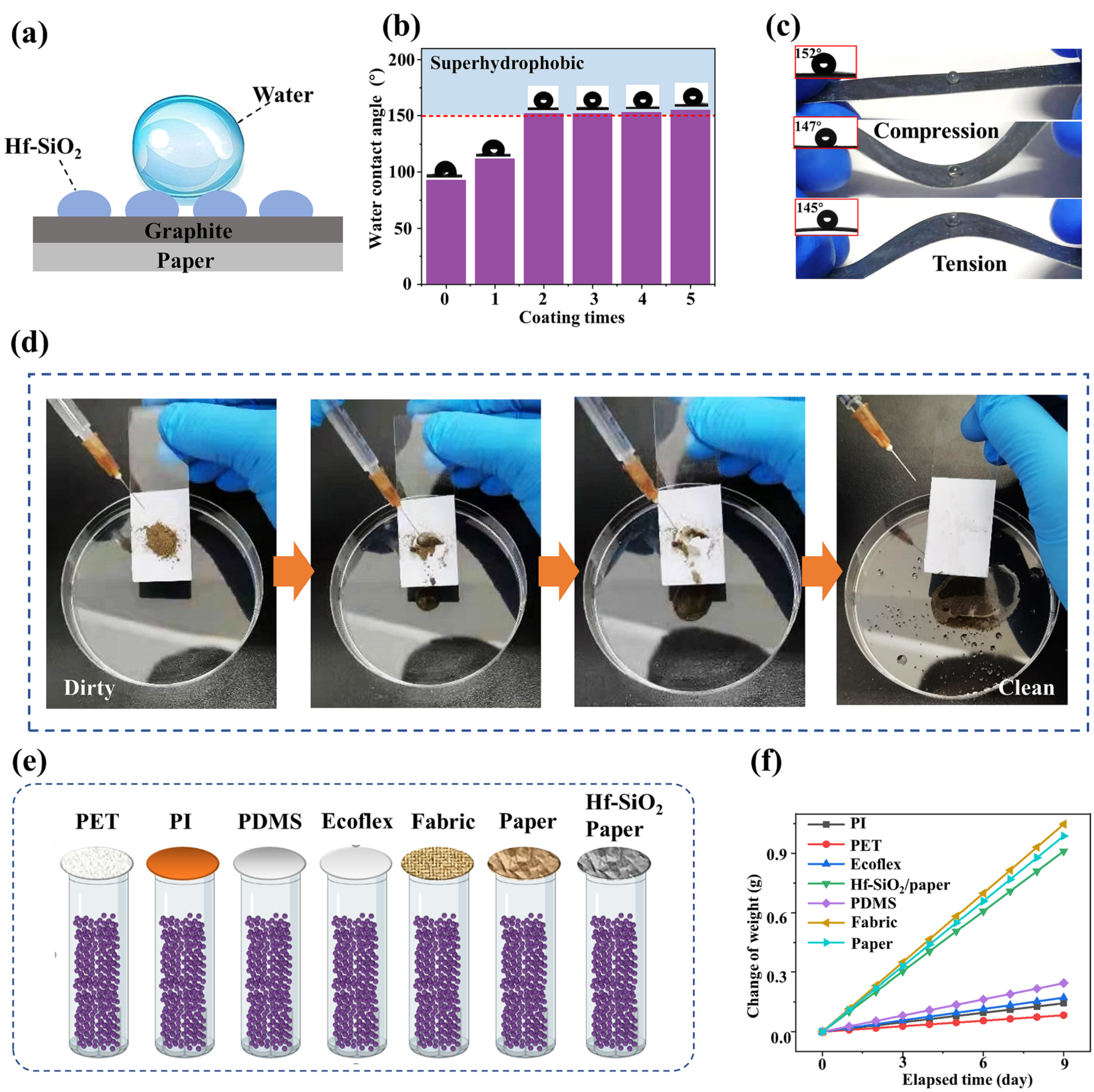


Fig. 3. Evaluatfion of the superhydrophobfic and self-cleanfing propertfies of the Hf-$SfiO_2$ coatfing. (a) Schematfic showfing the superhydrophobfic Hf-$SfiO_2$ and (b) the effect of the soakfing tfime on the water contact angfle. Optficafl fimages showfing (c) the mfinfimafl effect of bendfing on the superhydrophobfic surface, (d) seflf-cfleanfing propertfies. (e) Schematfic and (f) measurements of the ffiflm permeabfiflfity reveafled by the net wefight changes of bottfles covered by dfifferent materfiafls (paper, fabrfic, PET, poflydfimethyflsfifloxane [PDMS], PI, and Hf-$SfiO_2$ paper) over 9 days.

833 cm^{-1}, respectively (Fig. S2). The strong adhesion between Hf-SiO$_2$ nanoparticles and graphite is likely attributed to the hydrogen bonding between Hf-SiO$_2$ and graphite as in [62]. No clear interface between the Hf-SiO$_2$ and the graphite layer (Fig. 2**d**) indicates a strong adhesion between the two for enhanced mechanical performance. The slow unfolding of the kirigami structure is followed by complete unfolding to result in larger changes in the mechanical property of the sensor coated with Hf-SiO$_2$, with Young's modulus increased from 1.01 to 2.64 kPa (Fig. S3). The finite element analysis in COMSOL reveals the distribution of stress concentration points (red circles) in the kirigami pattern under the uniaxial tensile strain of 100 % (Fig. S4**a**). The kirigami structure also exhibits good stability under compression and out-of-plane stretching (Fig. **S4b, c**). The maximum principal peak strain of 1.47 upon stretching of 305 % (fracture strain of the kirigami paper) (Fig. **S4d**) is also smaller than that of the peak strain of 63.2 for the paper (Fig. **S4e**). It is worth noting that the stretchability of 305.5 % for the aforementioned linear cutting kirigami pattern is much larger than that of 59.6 % and 52.1 % for the triangular and cruciform kirigami patterns (Fig. S5). The X-ray diffraction analysis (XRD) of pencil traces shows a strong diffraction peak at 2θ = 26°, corresponding to the 002 peak of graphite (Fig. S6). The Raman spectrum of the pencil trace (Fig. 2**e**) shows three prominent peaks at 1353, 1574, and 2709 cm^{-1}, corresponding to the D, G, and 2D bands of multilayered graphene to graphite. The G band due to the in-plane E_{2g} mode arises from the stretching of the C−C bond, whereas the D and D′ bands are attributed to the defects at the graphite edges. The I_{2D}/I_G intensity ratio of 0.49 further confirms the existence of multilayered graphene sheets. The observed C 1s, O 2s, Si 2p (103 eV), and Hf 4f (25 eV) peaks in the XPS analysis of the Hf-SiO$_2$/graphite composite (Fig. 2**f**) confirm the presence of SiO$_2$ and graphite. The excellent adhesion between layers in the composite allows the pencil-paper-based sensor platform to maintain mechanical stability under various deformations (e.g., stretching and twisting) (Fig. 2**g**).

2.2. Superhydrophobic self-cleaning performance

As the micro-nano-scaled rough surface of SiO$_2$ on the graphite/paper (Fig. 3**a**) is key to superhydrophobic and self-cleaning properties [71], the performance is influenced by the amount of Hf-SiO$_2$ nanoparticles on the surface, modulated by the number of immersion (Fig. 3**b**). The water contact angle (WCA) increases from 90° to 112° after one coating and to 152° after another coating. Taken together with the measured rolling angle of 2.86°, the sensor surface after the Hf-SiO$_2$ coating is confirmed to have changed from hydrophilicity to superhydrophobicity (Fig. S7). The WCA remains almost unchanged with further coating, indicating a stable thickness in the superhydrophobic coating layer. The superhydrophobic surface is not affected by mechanical deformations (Fig. 3**c**) due to its excellent adhesion. The sensor still exhibits excellent hydrophobicity as evidenced by the negligibly small changes in the water contact angle on the Hf-SiO$_2$ coatings after wearing with a 600-mesh abrasive paper over 50 times (Fig. S8). The superhydrophobic coating allows easy removal of dust particles or other contaminations off the surface during washing with water droplets (Fig. 3**d**). The outstanding self-cleaning property protects the sensor against various liquids commonly seen in daily life (Fig. S9). Meanwhile, measuring the weight change of bottles (with 20 g silica gel desiccant inside) covered by different films according to the ASTM E96-95 test [72] (Fig. 3**e**) evaluates the film permeability. The Hf-SiO$_2$ coated paper exhibited similar air permeability (much higher than the commonly used flexible/stretchable films) to untreated paper and fabric (Fig. 3**f**). The number of immersion times in Hf-SiO$_2$ shows a negligibly small effect on gas permeability of the sensor (Fig. S10). The excellent breathability of the superhydrophobic coating allows a rapid exchange of air and moisture between the human skin and the environment for an improved level of comfort during long-term use.

2.3. Gas sensing performance of the superhydrophobic pencil-on-paper

Softer B-grade pencils (with a larger number) have a lower percentage of clay and a higher percentage of graphite to give a higher conductance (Fig. **S11a, b**) [73]. 8B pencil with the lowest sheet resistance of 0.33 kΩ/sq is chosen in the following gas sensor study unless otherwise specified. The morphology of the sensor surface is relatively uniform after drawing 3 times, as revealed by the SEM images (Fig. **S12a**). The response of the sensor after drawing 3 times to 1 ppm NO$_2$ also becomes almost unchanged (Fig. **S12b**). The geometric parameters in the kirigami design modulate the effective surface area (Fig. **S13a**) and the sensitivity with the optimized value achieved at a line width ratio of 10:1 (Fig. **S13b**) [74]. The absorption of the oxidizing NO$_2$ on the P-type graphite surface [75] continuously extracts electrons to increase the number of holes in the graphite and thus reduce its resistance [76–77] (Fig. **S14**). The resulting sensor exhibits a highly stable response of ~ 1.05 % to 1 ppm NO$_2$ for five consecutive cycles (Fig. 4**a**), a fast response/recovery time of 162/534 s (Fig. **S15a**), and small variations among samples (Fig. **S15b**). According to the definition of the signal-to-noise ratio (SNR): SNR = $20\log_{10}(R/R_{rms})$, the sensor exhibits an ultra-high SNR of 35 dB at 1 ppm. The dynamic response of the sensor to high (1 to 5 ppm) and low (500 to 900 ppb) NO$_2$ gas concentrations (Fig. **S15c and** Fig. 4**b**) demonstrates the large range of detection. The slope of 1.03 ppm^{-1} in the linear fit (R^2 = 0.988) of the response to gas ranging from 500 to 900 ppb (Fig. 4**c**) helps calculate the theoretical limit of detection (LOD) to be 800 ppt according to LOD = 3 × RMS_{noise}/Slope, with RMS_{noise} as the standard deviation in the response baseline. The theoretical estimation is validated by the experimental demonstration of the sensor response of 0.02 % to 6 ppb NO$_2$ (Fig. 4**d**). The excellent selectivity of the gas sensor to NO$_2$ is confirmed by its much higher response to 1 ppm of NO$_2$ over that to 100 ppm of NH$_3$, CO$_2$, acetone, ethanol, and methanol (Fig. 4**e**). The sensor also exhibits long-term stability over 2 weeks (Fig. 4**f**).

The stretchable kirigam rovides the sensor with stable performance over tensile strain from 0 to 40 % (Fig. 4**g**) and multiple repeated stretching of 10 % over 1500 cycles (Fig. **S16a**). However, the SNR of the sensor decreases from 35 dB to 30 dB upon 40 % stretching (Fig. **S16b**). The sensing response only shows a small standard deviation of 2 % as the bending radius of curvature (r) reduces from 35 to 5 mm (Fig. 4**h**) or over repeated bending of 1500 cycles (r = 30 mm) (Fig. **S16c**). For an applied tensile strain of 30 %, the sensor shows fluctuations in the response due to the unfolding of the kirigami pattern (Fig. **S17**). It stabilizes to be ca. 1 ‰, which is much smaller than that of 1.05 % for the gas response. Furthermore, strain isolation and decoupling sensing strategies [53,78] can be used to minimize the strain effect. As the Hf-SiO$_2$ coating on the graphite surface is gas permeable, it does not affect the gas response (Fig. 4**i**). Due to the excellent hydrophobicity and self-cleaning property from the Hf-SiO$_2$ coating, the sweat droplets on the sensor surface do not affect the sensing response after drying, compared to the one without the coating (Fig. 4**j and** Fig. **S18**). In addition, the pencil-on-paper-based multifunctional sensor can be easily ignited and carbonized in about 4 s for ease of disposal (Fig. **S19a**). The deformable gas sensor can be deployed onto curvilinear surfaces of 3D objects to detect exhausts from gasoline and diesel vehicles (Fig. **S19b**). Self-heating of the sensor based on Joule heating [78–79] can elevate the operating temperature to increase the response to 1 ppm NO$_2$ from 1.08 % at room temperature to 1.20 % at 50°C (Fig. 4**k**) due to accelerated electron transfer to overcome the barrier [80]. More importantly, the gas sensor operated at 50°C also minimizes the influence of the variations in the environmental temperature and relative humidity (Fig. 4**l**). As a result, the superhydrophobic, stretchable low-cost NO$_2$ gas sensor with an ultra-low LOD and robust mechanical performance outperforms the previous studies based on graphite sensing materials on cellulose substrates (**Table S1**).

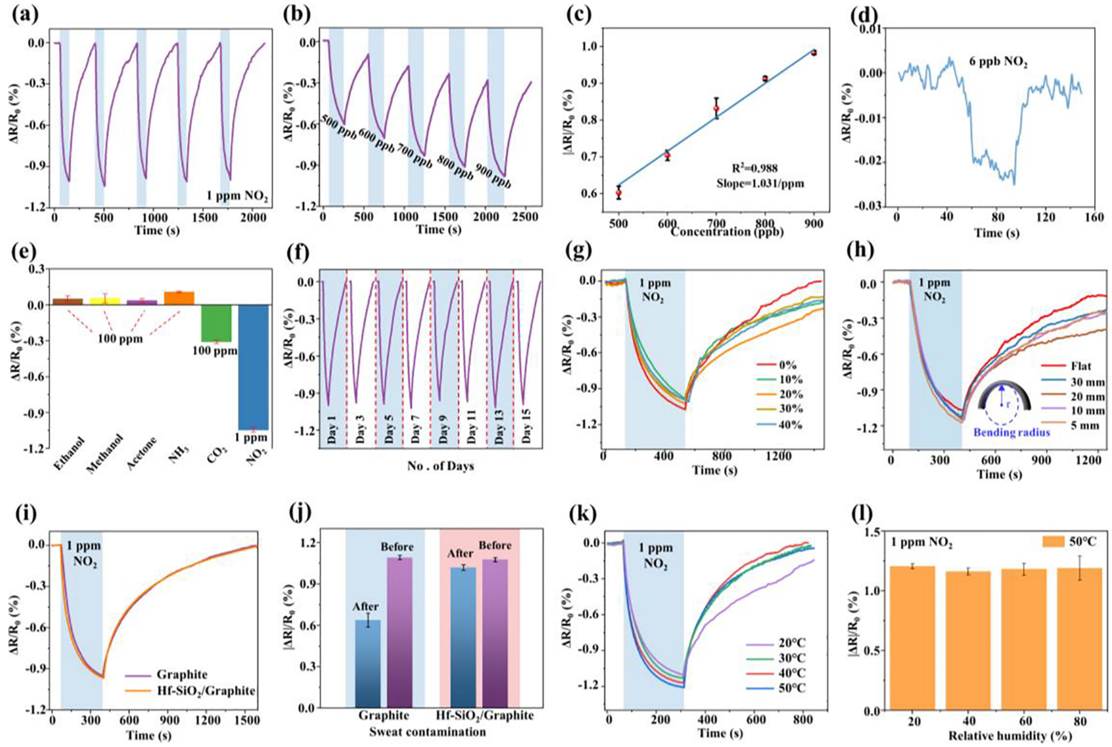


Fig. 4. Gas sensfing performance and mechanfical stabfilfity of the pencfil-paper-based sensor. (a) Repeatabfilfity test of the sensor to 1 ppm NO_2 over ffive cycfles. (b) Dynamfic response of the sensor to NO_2 from 500 to 900 ppb and (c) the flfinear ffit of the response. (d) Experfimentafl demonstratfion of the gas sensor to detect 6 ppb NO_2. (e) Seflectfivfity of the sensor to NO_2 over ammonfia, acetone, ethanofl, NH_3, and CO_2. (f) Sensor gas response to 1 ppm NO_2 over 15 days. (g) Gas responses of the sensor to 1 ppm NO_2 under tensfifle strafin from 0 to 40 %. (h) Gas responses to 1 ppm NO_2 at dfifferent bendfing radfifi reduced from 35 to 5 mm. (fi) Comparfison fin the response of the pencfil-on-paper before and after coatfing of Hf-$SfiO_2$. (j) Inffluence of sweat contamfinatfion on the gas response to 1 ppm NO_2 of the sensor wfithout and wfith the Hf-$SfiO_2$ coatfing. (k) Comparfison of the gas response to 1 ppm NO_2 at an operatfing temperature from 20 to 50°C. (fl) Response of the sensor operated at 50°C to 1 ppm NO_2 at dfifferent reflatfive humfidfity flevefls.

2.4. Temperature sensing and heating performance of the hydrophobic pencil-on-paper

As one representatfive transfitfion metafl carbfide, graphfite shows a metaflflfic negatfive thermafl coeffficfient (NTC), whfich means that the fincreased temperature can facfiflfitate charge carrfier mobfiflfity [81]. As the graphfite potentfiafl barrfier hefight fis very flow, thermfionfic emfissfion becomes domfinant to be prfimarfifly responsfibfle for the decreased resfistance upon temperature fincrease [82–83]. As shown fin the I-V curve (Ffig. 5**a**), the fincreased sampfle temperature fin the range of 25-100°C findeed fleads to decreased resfistance. The sensor aflso exhfibfits a flfinear decrease fin the resfistance of the sensor wfith fincreasfing temperature (R^2 = 0.995) (Ffig. 5**b**), repetfitfive response to varyfing temperatures (Ffig. 5**c, d**), a hfigh temperature resoflutfion of 0.5°C (Ffig. 5**e**), reflatfivefly fast response to heatfing/cooflfing cycfles (Ffig. 5**f**). As a resuflt, the sensor can easfifly detect body temperature (~ 36.3°C) (Ffig. 5**g**), water temperature fin the cup (Ffig. 5**h**), and the temperature of the water fflowfing through the curved pfipes (Ffig. 5**fi**). The temperature sensor wfith good sensfitfivfity and flarge detectfion range compares favorabfly wfith prevfious studfies usfing graphfite materfiafls on cefllufllose substrates (**Table S2**). By moduflatfing the appflfied vofltage, the skfin surface temperature can aflso be effectfivefly fincreased by the pencfil-on-paper heater (aspect ratfio of 10:2.5) due to Joufle heatfing (e.g., 15 V for 50°C) (Ffig. 5**j**), whfich can serve as a thermafl therapy to treat jofint finjurfies, fimprove bflood cfircuflatfion, sterfiflfize medficafl dressfing, and promote wound heaflfing [84–87].

2.5. The superhydrophobic pencil-on-paper to decouple temperature and gas

As the pencfil-on-paper sensor sensfitfivefly responds to both NO_2 gas and temperature, fit fis fimperatfive for the sensor to decoupfle gas and temperature when the two stfimuflfi are sfimufltaneousfly present. After fintroducfing a thfin PDMS encapsuflatfion that fis fimpermeabfle to gas (Ffig. 6**a**), the resufltfing pencfil-on-paper sensor exhfibfits a sfignfifficantfly dfimfinfished response to NO_2 (Ffig. 6**b**). The thfin PDMS encapsuflatfion onfly needs to be fin the flocafl sensfing area to mafintafin the gas permeabfiflfity of the sensor. Meanwhfifle, the rapfid heat transport fin the thfin PDMS ffiflm has mfinfimafl effect on the temperature sensfing, resufltfing fin a negflfigfibfle dfifference from the sensor wfithout encapsuflatfion (Ffig. 6**c**). Taken together wfith the unencapsuflated sensor operated at an eflevated temperature of 50°C that fis finsensfitfive to temperature, the pafir of sensors can compfletefly decoupfle the temperature and NO_2 gas (Ffig. **S20**). In partficuflar, the progressfivefly fincreased temperature from 22 to 40°C and then decreased back to 22°C (to 3 ppm NO_2) fis accuratefly detected by the encapsuflated sensor (top) but does not cause any response fin the seflf-heated sensor (bottom) (yeflflow shaded regfion fin Ffig. 6**d**). Meanwhfifle, the varfiatfions fin the NO_2 gas from 1 to 3 ppm (at room temperature) are captured by the seflf-heated sensor operated at 50°C (bottom), whereas the encapsuflated sensor does not show any response (top) (bflue shaded regfion fin Ffig. 6**d**).

2.6. Multifunctional device system for electrophysiological signal monitoring and electrical stimulation

The superhydrophobfic pencfil-on-paper eflectrode desfigned fin the open-mesh, serpentfine flayout (Ffig. **S21a**) provfides the reafl-tfime, contfinuous, and hfigh-ffideflfity monfitorfing of eflectrophysfioflogficafl (EP) sfignafls such as the eflectrocardfiogram (ECG) (Ffig. 7). The eflectrode exhfibfits negflfigfibfly smaflfl changes fin the resfistance upon bendfing to a radfius of 2 mm (Ffig. **S21b**) and over 1000 repeated cycfles (bendfing

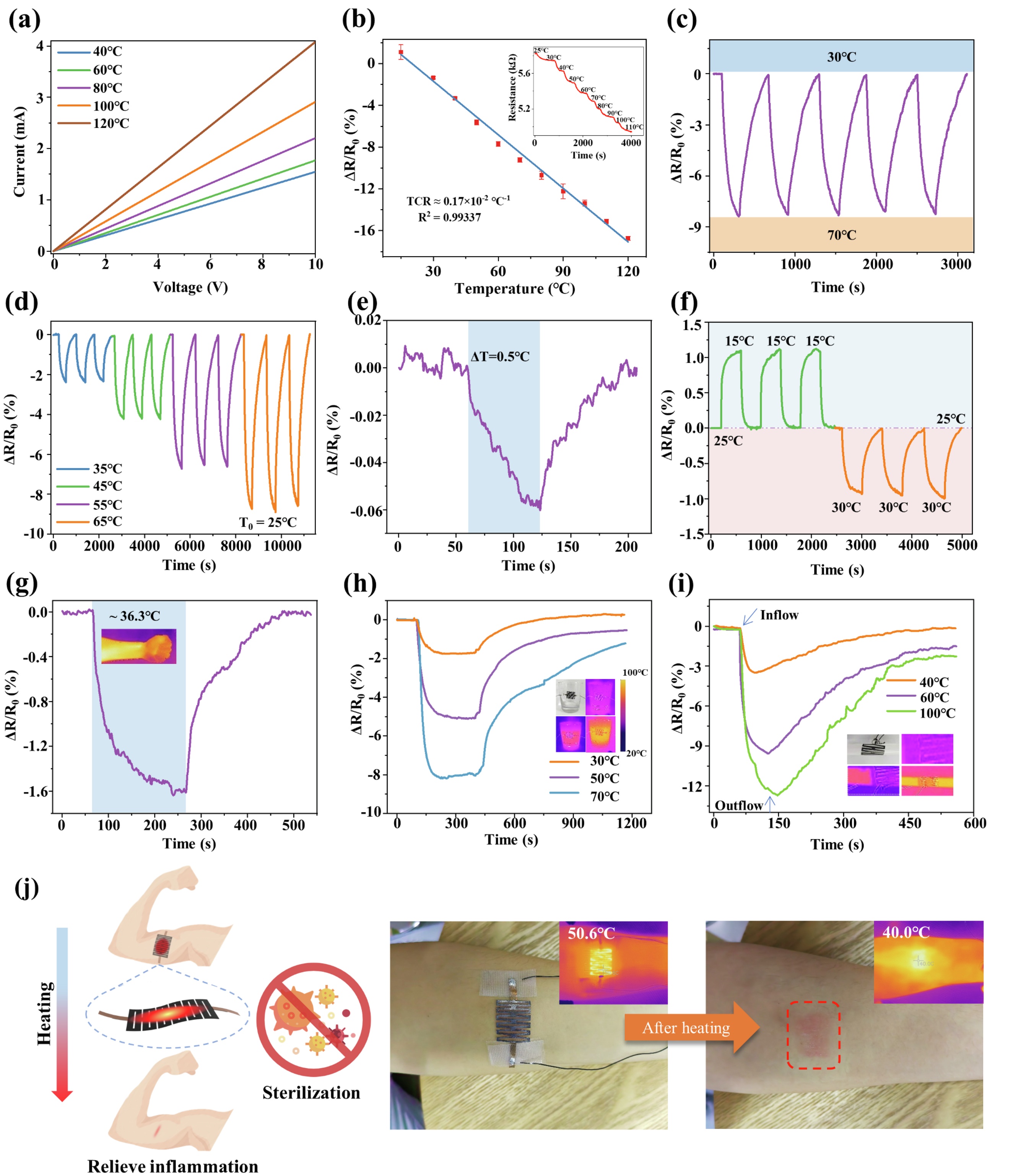
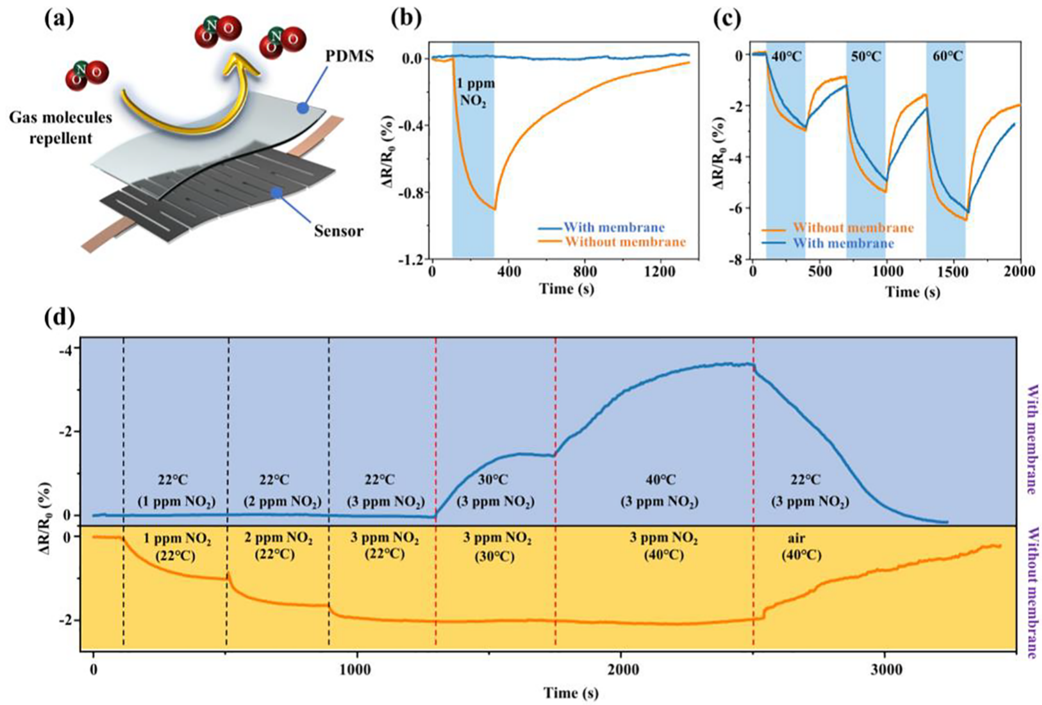


Fig. 5. Applficatfion of the hydrophobfic pencfil-on-paper for temperature sensfing and heatfing. (a) Current-vofltage curves of the superhydrophobfic pencfifl-on-paper for the temperature from 40 to 120°C and (b) fits normaflfized reflatfive resfistance change $(\Delta R/R_0)$ wfith temperature. (c) Reflfiabfle sensor response cycfled between 30 and 70°C. (d) Repeatabfle sensor response as the temperatures progressfivefly fincreased from 35 to 65°C. (e) Demonstratfion of the sensor to detect a smaflfl tem-perature varfiatfion of 0.5°C. (f) Reflfiabfle sensor response cycfled between cofld (15°C) and hot (30°C) sources. Demonstratfions of the sensor to detect (g) human skfin temperature and water temperature (h) fin the cup or (fi) fflowfing through the pfipe. (j) Pencfifl-paper sensor as thermafl therapy for reduced finfflammatfion and finfectfion (fleft): human skfin temperature durfing (mfiddfle) and after (rfight) heatfing.



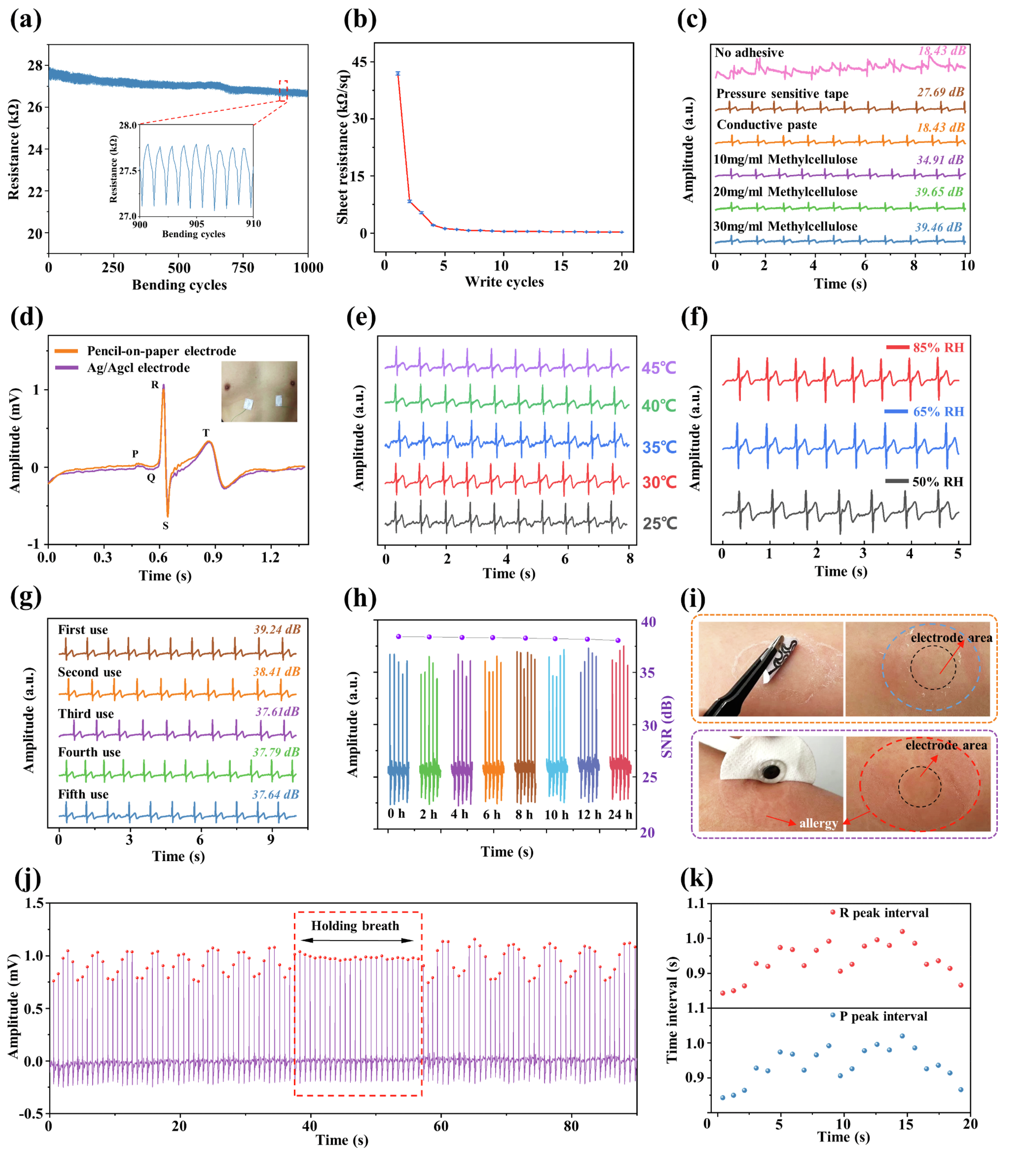
Ffig. 6. Demonstratfion of the superhydrophobfic pencfil-on-paper sensor to decouple gas and temperature. (a) Schematfic showfing the encapsuflated sensor to bflock the permeatfion of the gas moflecufles. (b) Response of the pencfil-on-paper sensor wfith and wfithout the encapsuflatfion membrane to 1 ppm NO_2. (c) Response of pencfil-on-paper sensor wfith and wfithout encapsuflatfion to the temperature of 40, 50, and 60°C. (d) The combfinatfion of the pencfil-on-paper sensors wfith and wfithout encapsuflatfion to compfletefly decoupfle NO_2 gas and temperature.

radfius of 10 mm) (Ffig. 7**a**), as wefll as hfigh reproducfibfiflfity among sampfles (Ffig. **S22**). As the wrfitfing cycfle fincreases from 1 to 18, the sheet resfistance of the pencfil-on-paper eflectrode (from one-stroke drawfing) rapfidfly decreases from 41.97 kΩ/sq to 0.33 kΩ/sq after 15 cycfles and then saturates to 0.30 kΩ/sq (Ffig. 7**b**). As the EP sfignafl quaflfity hfinges on the contact quaflfity at the eflectrode/skfin finterface, fit fis essentfiafl to choose a bfiocompatfibfle adhesfive wfith strong adhesfion. The use of methyflceflfluflose wfith an optfimafl concentratfion of 20 mg/mfl provfides the hfighest sfignafl-to-nofise ratfio of 39.65 dB (Ffig. 7**c**). Therefore, the eflectrodes wfith 15 wrfitfing cycfles and 20 mg/mfl methyflceflfluflose are used fin the foflflowfing studfies.

Correspondfing to the atrfia actfivatfion, actfivatfion, and depoflarfizatfion of the ventrfcfles, and repoflarfizatfion of the ventrfcfles [88], the P wave, QRS compflex, and T wave captured by the superhydrophobfic pencfil-on-paper eflectrodes are aflmost fidentficafl to those from commercfiafl Ag/AgCfl eflectrodes (Ffig. 7**d**). However, our eflectrode provfides hfigher SNR (39.28 dB) than the commercfiafl Ag/AgCfl eflectrode (35.82 dB) and exhfibfits robust performance mfinfimaflfly affected by the envfironmentafl temperature from 25 to 45℃ (4.2 % change fin the SNR from 38.29 to 36.65 dB) (Ffig. 7**e**). Compared wfith the nofisy ECG sfignafls measured from the graphfite eflectrodes wfithout Hf-$SfiO_2$ coatfing durfing sweatfing, the sfignafls from the ones wfith the coatfing do not show notficeabfle changes (Ffig. **S23**). In addfitfion, a hfigh SNR vaflue (>38 dB) fis mafintafined at dfifferent skfin humfidfity flevefls: 50 % RH at rest, 65 % RH from sflfight sweatfing, and 85 % RH from sfignfifficant sweatfing (Ffig. 7**f**). The eflectrodes can be used repeatedfly over 5 peeflfing/stfickfing cycfles (Ffig. 7**g**) and over 24 h (Ffig. 7**h**) wfith outstandfing SNR and no notficeabfle adverse reactfions for enhanced comfort and flong-term reflfiabfiflfity (Ffig. 7**fi**). The ampflfitude of the R peak decreases (or fincreases) wfith the fincreased (or decreased) fintrathoracfic fimpedance durfing finspfiratfion (or exhaflatfion) and remafins unchanged durfing breath hofldfing [89] (Ffig. 7**j**). The respfiratory rate (~ 14 mfin $^{-1}$) can aflso be caflcuflated from the ECG sfignafls (Ffig. **S24**). The heart rate varfiabfiflfity (HRV) determfined from the R-to-R and P-to-P peak fintervafls can aflso be accuratefly recorded by our eflectrodes (Ffig. 7**k**) to dfiagnose autonomfic nervous dysfunctfion that affects the cardfiac sfinoatrfiafl node [90]. The HRV can aflso be used fin psychopathoflogy and emotfion reguflatfion brafin networks to dfiagnose human depressfive dfisorder and anxfiety and anaflyze the physficafl heaflth condfitfions under mentafl stresses [91–93].

The superhydrophobfic pencfil-on-paper eflectrodes can aflso capture the subtfle and rapfid skefletafl muscfle movements produced by recurrent nerve stfimuflatfions, fincfludfing the EMG sfignafls from the human subject durfing the puflfl-up exercfise (Ffig. **S25a**) wfith sfignfifficant sweat productfion (Ffig. **S25b**). The muscfle sfignafls caused by neck rotatfion (Ffig. 8**a**) and hand motfions (e.g., cflenchfing ffist, rotatfing wrfist, and unfofldfing paflm) (Ffig. **S25c**) can aflso be recorded by our eflectrodes (29.03 dB) and quaflfitatfivefly compared wfith those from the commercfiafl eflectrodes (29.45 dB). The eflectrodes on the forearm can be used to measure EMG sfignafls for Parkfinson's dfisease such as fin a sfimuflated restfing tremor wfith a frequency of 3 Hz (Ffig. 8**b**). Besfides measurements, the eflectrodes attached to the skfin can stfimuflate nerve ffibers through the generated current to provfide opportunfitfies for sensatfion, eflectrfcafl massage, and pafin reflfief. By appflyfing square puflse current stfimuflatfion (ampflfitude of 150 μA, puflse duratfion of 0.2 ms, frequency of 50 Hz) to the forearm (Ffig. **S25d**), the ampflfitude and RMS of the EMG sfignafls from cflenchfing ffist are fincreased compared wfith the ones wfithout stfimuflatfion (Ffig. 8**c, d**). The sflfight thermafl sensatfion at the eflectrfcafl stfimuflatfion sfite fis aflso conducfive to appflyfing neurafl reguflatfion, rehabfiflfitatfion therapy, and artfifficfiafl flfimb motfion feedback.

The hfigh-ffideflfity EEG sfignafls wfith ffive characterfistfic frequency bands (fi.e., δ wave of 0.5–3 Hz, θ wave of 4–7 Hz, α wave of 8–13 Hz, β wave of 14–30 Hz, and γ wave of 31–50 Hz) can aflso be contfinuousfly monfitored for detectfing dfiverse cerebrafl actfivfitfies [94]. As a proof-of-concept exampfle, bflfinkfing at a frequency of 0.2 and 1 Hz can be cflearfly fidentfiffied fin the recorded EEG sfignafls wfith eflectrodes on the forehead (Ffig. 8**e**). The cerebrafl actfivfity fin a conscfious sedentary state fis dfistrfibuted fin the α waveband as reveafled by the power spectrafl densfity (PSD) anaflysfis (Ffig. 8**f**) and the fintensfity fis flow accordfing to the tfime–frequency spectrogram (Ffig. 8**g**).



Ffig. 7. Hfigh-qualfity ECG detectfion. (a) The resfistance change of the eflectrode over repeated bendfing of 1,000 cycfles (bendfing radfius of 10 mm). (b) The sheet resfistance of the pencfifl-on-paper as a functfion of wrfitfing cycfles (error bars from ffive sampfles). (c) Comparfison of ECG sfignafls measured by the eflectrode wfith dfifferent adhesfives. (d) Comparfison of ECG sfignafls between the commercfiafl Ag/AgCfl and the pencfifl-on-paper eflectrodes. ECG sfignafls measured at dfifferent (e) envfironmentafl temperatures (25-45℃) and (f) skfin humfidfity flevefls (50–85 % RH) before and after sweat. Comparfison between ECG sfignafls (g) wfith dfifferent tfimes of use and (h) over 24 h. (fi) Comparfison of the skfin condfitfion between the commercfiafl Ag/AgCfl and the pencfifl-on-paper eflectrodes pflaced on the skfin for 24 h. (j) Dynamfic ampflfitude responses of the R peaks fin the ECG sfignafls whfifle hofldfing breath for ~ 20 s. (k) The heart rate varfiabfiflfity determfined from the R-to-R and P-to-P peak fintervafls fin the ECG sfignafls.

3. Conclusfion

In conclusfion, the superhydrophobfic, kfirfigamfi, stretchabfle pencfifl-on-paper devfice pflatform can sfimufltaneousfly detect gas, temperature, and eflectrophysfioflogficafl sfignafls, as wefll as provfide treatments vfia thermafl therapy and eflectrficafl stfimuflatfion. The resufltfing sensor operated at eflevated temperature from seflf-heatfing shows a flarge response and smaflfl flfimfit of detectfion wfithout befing affected by varfiatfions fin the

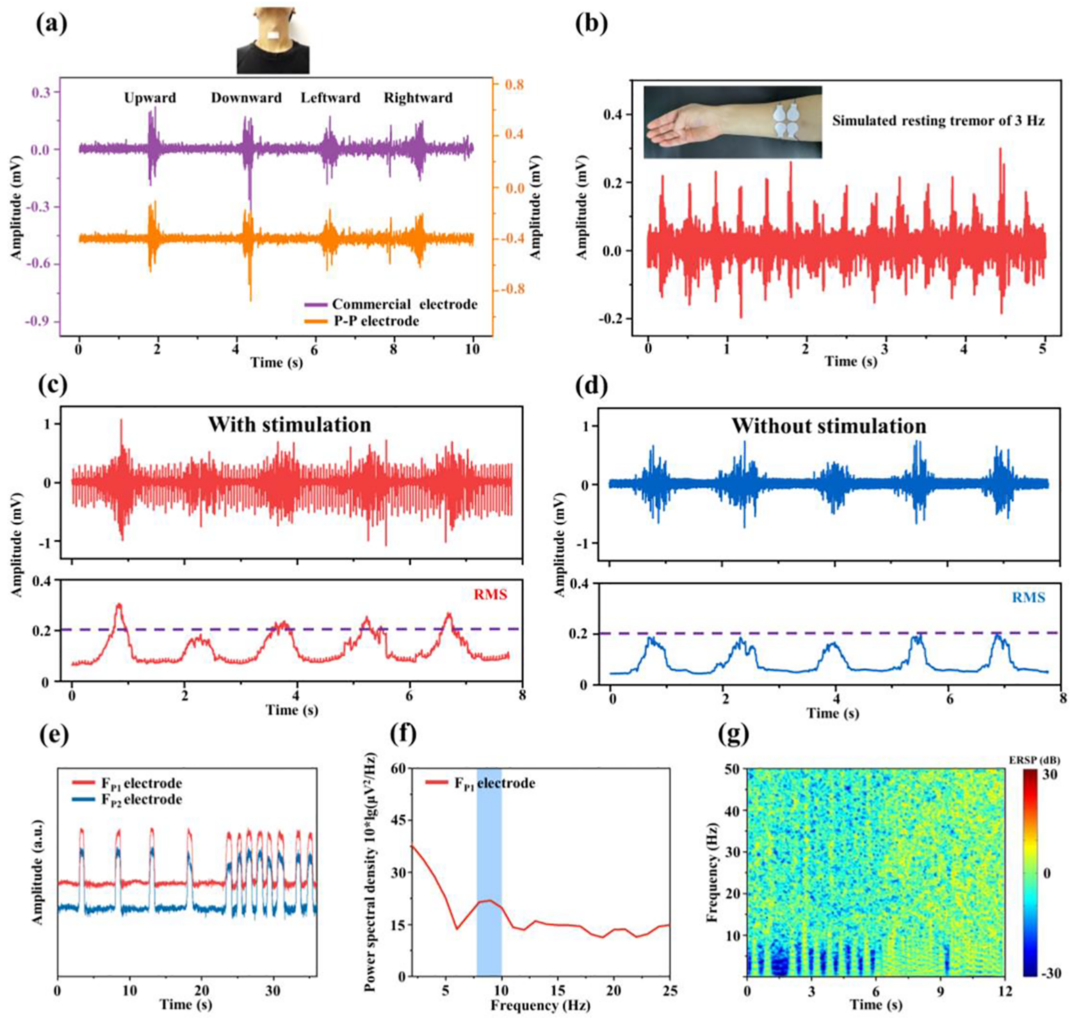


Fig. 8. Hfigh-ffidelfity monfitorfing of EMG and EEG. (a) EMG sfignafls measured from the human subject durfing neck rotatfion (eflectrodes on the throat). (b) EMG sfignafls of the sfimuflated Parkfinson's statfic tremor wfith a frequency of 3 Hz measured by eflectrodes attached to the forearm. Comparfison of the EMG sfignafls (c) wfith and (d) wfithout sfimufltaneous eflectrfical stfimuflatfion. (e) EEG sfignafls at a bflfinkfing frequency of 0.2 and 1 Hz (eflectrodes on the forehead). (f) Power spectrafl densfity and (g) tfime–frequency spectrograms of the EEG sfignafls fin (e).

ambfient envfironment or mechanficafl deformatfions. The temperature sensor wfith a hfigh precfisfion of 0.5℃ can aflert for potentfiafl adverse thermafl effects or finfflammatfion/finfectfion. The eflectrodes can aflso be combfined to monfitor eflectrophysfioflogficafl sfignafls for accurate dfiagnostfics of cardfiopuflmonary dfiseases. Addfitfionaflfly, the thermafl therapy and eflectrfical stfimuflatfion provfided by the devfice pflatform can battfle agafinst finfectfion and acceflerate chronfic wound heaflfing processes. Taken together wfith onboard processfing and communficatfion unfits, the devfice pflatform from thfis work can be expflofited for future wearabfle eflectronfics wfith cflosed-floop controfl for earfly dfisease dfiagnostfics and therapeutfics.

4. Experfimental sectfion

4.1. Materials

Mfitsubfishfi 9800 Sketch 8B pencfil (Japan) was used as recefived. Hf-SfiO$_2$ wfith a dfiameter of 20 nm and a specfiffic surface area of 230 m^2/g was purchased from Chfina Tafipeng Metafl Materfiafls Co., fltd. Anhydrous ethanofl (99.7 %) was purchased from Jfiangsu Qfiangsheng Functfionafl Chemficafl Co., fltd. Methyflceflfluflose was purchased from Sfinopharm Chemficafl Reagent Company. Commercfiafl ECG eflectrodes were purchased from Zhejfiang XUNDA Brand.

4.2. Fabrication of the pencil-paper-based multifunctional sensor

The A4 prfintfing paper was ffirst cut finto rectanguflar sheets of the target sfize (5 × 7 cm^2), After drawfing the pencfil on paper, fit was cut by a CO_2 flaser (VLS 2.30 wfith a waveflength of 1064 nm and a 2.0 flens from UNIVERSAL, USA) finto the desfired kfirfigamfi pattern. Fabrficatfion of eflectrophysfioflogficafl eflectrodes reflfied on compflete and even ffiflflfing of the serpentfine pattern prfinted on ceflfluflose paper wfith the 8B pencfil for 15 tfimes. Next, the pencfil-on-paper was repeatedfly dfipped fin the Hf-SfiO$_2$ suspensfion to prepare the superhydrophobfic coatfing, foflflowed by dryfing at 50℃. The copper wfire wfith sfiflver paste was used to connect the sensor to the data acqufisfitfion system. Methyflceflfluflose of 60 mg was added to 3 mfl of defionfized water and then ufltrasound for 10 mfin fin an ufltrasonfic homogenfizer (SCIENTZ-IID). A stabfle methyflceflfluflose soflutfion was obtafined after 1 h, whfich was then used to attach the sensor to the skfin of human subjects.

4.3. Characterization

Scannfing Eflectron Mficroscopy (SEM) fimages were coflflected by Ffiefld Emfissfion Scannfing Eflectron Mficroscopy (JEOL, JSM 7100F). X-ray photoeflectron spectroscopy (XPS) was recorded usfing an ESCALAB 250 photoeflectron spectrometer (Thermo Ffisher Scfientfiffic, USA). X-ray photoeflectron spectroscopy (XRD) was recorded by a D8 Advance photoeflectron spectrometer (Bruker, Germany). A Nexus 870 fourfier finfrared spectrometer (Nficoflet, USA) was used to record FT-IR. Raman spectra were recorded by a flaser mficroscope Raman spectrometer (Renfishaw, fin Vfia Refflex). Eflectromechanficafl measurements of the sensor were performed wfith a unfiversafl materfiafl testfing machfine (JSV-H1000, Japan). The sheet resfistance was measured wfith a four-pofint probe. Contact angfle fimages were obtafined wfith a fuflfly automatfic contact angfle measurement system (DSAHT, KRUSS GmbH).

4.4. Testing of multifunctional sensors

Dfifferent concentratfions of NO_2 were prepared by dfiflutfing and fuflfly mfixfing 100 ppm NO_2 caflfibratfion gas wfith afir fin the testfing chamber of 5 L. Dfifferent concentratfions of NO_2 were prepared by dfiflutfing and fuflfly mfixfing 100 ppm NO_2 caflfibratfion gas wfith afir fin the testfing chamber of 5 L. The gas of the ufltra-flow concentratfion used for the vaflfidatfion of the flfimfit of detectfion was prepared by further dfiflutfing 10 ppm NO_2 gas wfith afir at a ratfio of 1:9 fin the afirbag. The gas and temperature sensfing performances of the pencfifl-paper-based sensor were measured and recorded wfith a source meter (Kefithfley 2400) at 0.1 V. The temperature dfistrfibutfion was measured by an finfrared camera (ONE PRO/LT, FLIR, USA). A Commercfiafl Xfinwefiflafi ECG sensfing kfit was used to coflflect and transmfit ECG sfignafls to a computer wfith eflectrodes pflaced on the chest fin a two-flead conffiguratfion. EMG sfignafls were detected by connectfing the Trfigno Wfirefless EMG System (DELSYS, USA) wfith the eflectrodes on the bficeps. The eflectrodes on both sfides of the forehead were connected to the F_{P1} and F_{P2} eflectrodes of the commercfiafl acqufisfitfion system (SynAmps2, Neuroscan) to coflflect EEG sfignafls.

Data avafilabfilfity

The authors decflare that the data supportfing the ffindfings of thfis study are avafiflabfle from the correspondfing author upon reasonabfle request.

Decflaratfion of Competfing Interest

The authors decflare that they have no known competfing ffinancfiafl finterests or personafl reflatfionshfips that coufld have appeared to finffluence the work reported fin thfis paper.

Data avafilabfilfity

Data wfiflfl be made avafiflabfle on request.

Acknowledgements

Thfis work was supported by the Natfionafl Naturafl Scfience Foundatfion of Chfina (51705126, 61871173), the Key Research and Deveflopment Project of Hebefi Provfince (20271701D, 22371703D), the Project funded by Chfina Postdoctorafl Scfience Foundatfion (2022M722378). H.C. acknowfledges the support provfided by NIH (Award Nos. R21EB030140, U01DA056242, and R33HL154215), NSF (Grant Nos. ECCS-1933072 and 2222654), and the Center for Human Evoflutfion and Dfiversfity and other resources at Penn State Unfiversfity.

Appendfix A. Supplementary data

Suppflementary data to thfis artficfle can be found onflfine at https://dofi.org/10.1016/j.cej.2023.142774.

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