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Antisolvent-induced multiscale β -glycine for preparing flexible piezoelectric films

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ABSTRACT

Based on the antisolvent-induced crystallization method of β -glycine, we successfully achieved the controlled preparation of β -glycine crystals ranging from submicron to centimeter scale using antisolvent methods. Importantly, we developed one-step in situ inducing strategy to form β -glycine composite films. Based on the solubility differences of polymers in water and antisolvents, β -glycine/polymer films were classified into three categories with distinct properties. In particular, β -glycine/PCL films demonstrated high effective piezoelectric coefficient (10.3 pm/V) and superior stability in crystal form. The piezoelectric nanogenerator (PENG) fabricated using β -glycine/PCL films exhibited excellent electrical output performance under mechanical pressure. Furthermore, under ultrasound stimulation, the PENG generated outputs of about 620 and 274 mV in vitro and in vivo, respectively. The device also exhibited good biocompatibility, indicating promising applications in implantable electrical stimulation therapeutic devices.

1. Introduction

Piezoelectric materials enable conversions between mechanical energy and electrical energy [1–4], due to their piezoelectric and inverse piezoelectric effects, and can therefore be widely used in energy harvesters, sensors, and actuators [5–10]. In recent years, the rapid advancement of wearable and implantable electronics has posed increasing challenges for conventional rigid piezoelectric materials to achieve mechanical compatibility with human tissues [11,12]. The exploration of flexible piezoelectric materials for wearable health monitoring, human-machine interaction, and self-powered sensing systems has become a cutting-edge research focus [13,14]. Meanwhile, biocompatibility and biodegradability have emerged as key

requirements for the next generation of medical electronics. Researchers have been developing a range of biodegradable flexible piezoelectric materials, such as poly(L-lactic acid) (PLLA), chitosan, and nanocellulose [15–17]. However, the variety and low piezoelectric performance of materials severely restrict their practical applications in biodegradable electronics.

Amino acids are a type of simple and accessible piezoelectric biomaterials. Among the 20 natural amino acids, 17 have piezoelectric properties on measurement or theoretical prediction [11]. Glycine, the simplest chiral amino acid, exhibits the high piezoelectric coefficient in β and γ phase [18]. γ -Glycine has demonstrated broad application prospects due to excellent out-of-plane piezoelectric coefficients of ~ 10 pC/N [19–21]. β -Glycine has extremely high shear piezoelectric

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coefficients (d_{16}) of $\sim 178 \text{ pmV}^{-1}$, also showing great potential in degradable flexible piezoelectric electronics, while its thermodynamically instability leads to easy converting to α -glycine or γ -glycine in ambient conditions, posing challenges to its application [11,22,23]. In previous study, stable β -glycine was prepared at nanoconfined environments (such as micro-nano-scale channels, or electrostatic spraying) [24,25] or on high crystallinity material surface (such as (111) Pt/SiO₂/Si substrate surfaces) [26]. These special preparation conditions not only require expensive equipment and complex processes, but also difficult to achieve large-scale preparation.

Antisolvent-induced crystallization gains much attention in the field of crystal growth due to rapid nucleation, controllable size and morphology, simple process, and easy scale-up preparation [27–30]. The addition of antisolvent can significantly reduce the solubility of the target solute, thereby inducing rapid precipitation and crystallization. This characteristic enables the antisolvent method to play a crucial role in pharmaceuticals, ultrafine powder synthesis, and the advanced functional materials fabrication. For example, in the pharmaceutical field, the antisolvent method can be employed in drug crystallization processes to effectively improve product purity and yield, while meeting the requirements of industrial-scale production in terms of efficiency and cost. Using water-miscible organic solvents such as methanol (MeOH), ethanol (EtOH), and dimethylformamide (DMF) as antisolvents, β -glycine crystals can be rapidly crystallized from glycine aqueous solutions [31]. Nevertheless, the β -glycine crystals with inherent hardness and brittleness cannot be directly fabricated into flexible piezoelectric devices. A common method to fabricate flexible piezoelectric glycine devices is combining piezoelectric glycine with flexible polymers [24,32,33]. For instance, Nguyen et al. prepared glycine crystals firstly, then mixed them with PCL solution, and finally fabricated the piezoelectric film through electrospinning. The actuator constructed from this film generated a high ultrasonic output pressure of 334 kPa under 0.15 V, demonstrating their potential application in implantable device [25]. However, its multi-step preparation process not only increases experiment complexity but also elevates the probability of phase transitions during β -glycine transfer. A simple method for preparing stable β -glycine composite films is of great significance.

In this study, based on the antisolvent-induced crystallization method of β -glycine, we developed one-step in situ inducing strategy to form β -glycine composite films. we not only investigated the morphology, size, and proportion of β -glycine crystals prepared in various water-antisolvent solutions, but also compared the basic properties of the three types of β -glycine/polymer piezoelectric films in detail by one-step in situ inducing strategy, and selected the β -glycine/PCL piezoelectric film with better stability to prepare a flexible PENG with good force-electric output performance under mechanical pressure and ultrasonic stimulation.

2. Results and discussion

2.1. Preparation and performances of multi-scale β -glycine across antisolvents

We investigated the size, morphology, and crystal form of β -glycine prepared in more than ten water-antisolvent systems. Among these antisolvents, DMF has a strong solubility for organic matter, and can even dissolve some polyesters with better stability (non-hygroscopicity). In the β -glycine/polycaprolactone (PCL) composite film prepared in the water-DMF system, PCL can maintain stable performance even in water. After being wrapped by PCL, β -glycine can be well isolated from water and is expected achieving longer stability in piezoelectricity. Therefore, we selected DMF as a representative to investigate the performance of glycine and its composites.

When 1 ml of 10% (w/v) glycine aqueous solution was slowly dripped to 20 ml of DMF, white glycine powder rapidly precipitated (Fig. S1). This is because water is miscible with DMF, but glycine is

insoluble in DMF. Glycine precipitates out due to its low solubility in the water-DMF mixed solution and rapid supersaturation. X-ray diffraction (XRD) analysis confirmed that the precipitated white powder is β -glycine (Fig. 1a). Characteristic diffraction peaks at 2θ values of 17.9, 18.9, 20.3, 23.7, 28.4, 31.0, 33.8, and 36.6 correspond to the (001), (100), (-101), (110), (020), (101), (-102), and (-112) crystal face of β -glycine, respectively. Scanning electron microscopy (SEM) analysis revealed that β -glycine is irregularly particles, size ranging from a few microns to over ten microns (Fig. 1b), which different from the needle-shaped β -glycine reported previous studies [23]. Interestingly, micrometer-needle-shaped β -glycine were observed by evaporating supernatant of the turbid solution (Fig. 1c). These observations suggested DMF induces the formation of β -glycine with different morphologies. Specifically, the fast-growing crystalline β -glycine is in irregular granular shape, while the slow-growing glycine is in needle shape (Fig. 1d). To further investigate the effect of DMF concentration on glycine crystallization, glycine crystals were obtained by evaporating mixtures with different volume ratio of H₂O to DMF (5:0, 5:3, 3:5, 1:5, 1:1000) at 40°C (Fig. S3a, e). Metallographic microscopy and XRD results showed that crystals grown in pure water without DMF formed flaky α -glycine with a width of hundreds of microns (Fig. 1e-I, Fig. 1f). With the addition of DMF (the volume ratio of H₂O:DMF = 5:3), glycine transitioned from the α phase to the β phase, accompanied with a change in crystal shape from flakes to needles with a length of more than 1 μm (Fig. 1e-II, Fig. 1f). As DMF content further increased (the volume ratio of H₂O:DMF = 3:5), needle-like with reduced length and granular β -glycine formed (Fig. 1e-III, Fig. 1f). When the volume ratio of H₂O:DMF reached to 1:5, the size of glycine further reduced (Fig. 1e-IV, Fig. 1f). When the DMF content greatly increased (the volume ratio of H₂O:DMF = 1:1000), β -glycine mainly formed submicron particles (Fig. 1e-V, Fig. 1f). In the water-DMF solution, different DMF contents all induced β -crystals, because thermodynamically unstable β -glycine priority nucleates compared to the stable α and γ phase. Meanwhile, due to DMF changed the rate of crystal growth rate, the DMF content greatly affected morphology and size of β -glycine crystals. Specifically, a small amount of DMF induced a small amount of β -glycine nucleation, but due to the influence of a large amount of water, it is easy to form large-sized needle-shaped β -glycine crystals. On the contrary, a large amount of DMF quickly changed the supersaturation of the glycine solution, resulting in a large number of β -crystal nuclei formation in a short time, and then forming a larger number of small size granules β -glycine crystals.

We also investigated other amide solvents such as N, N-dimethylacetamide (DMAC) and N, N-dimethylacrylamide (DMFAA), similar to DMF, as antisolvents to induce the growth of glycine crystals. XRD and crystal phase microscopy results showed that the induced glycine was β phase (Fig. S4a). In addition, same results were observed in alcohols (methanol, ethanol, isopropyl alcohol (IPA), polyethylene glycol (PEG)), acetonitrile (ACN) and 1-methyl-2-pyrrolidinone (NMP) as antisolvents (Fig. S4b, c). However, acetone (Acton) and tetrahydrofuran (THF) failed to induce β -phase formation, and the obtained glycine crystals were α phase (Fig. S4d).

The piezoelectric performances of the β -glycine crystals were evaluated using the dual AC resonance tracking (DART) technique based on piezo-response force microscopy (PFM). Resonance measurement was conducted at different voltages to confirm the piezoelectric response (Fig. S5). Results indicated that the resonance peak intensity increased with increasing applied voltage, which confirmed the intrinsic piezoelectricity of β -glycine. Amplitude and phase mapping further indicated that β -glycine exhibits strong responsiveness to applied voltage (Fig. 2a, b). The resonant frequency and quality factor (Fig. S6a, b) were measured to correct the β -glycine piezoelectric signal after resonance amplification (Fig. S6c, d). The amplitude under the applied driving voltage was fitted using the Simple Harmonic Oscillator (SHO) model (Fig. S6e, f), and the effective piezoelectric coefficient of β -glycine was calculated through linear fitting of the voltage-amplitude relationship. The results revealed a linear increase in amplitude with applied AC

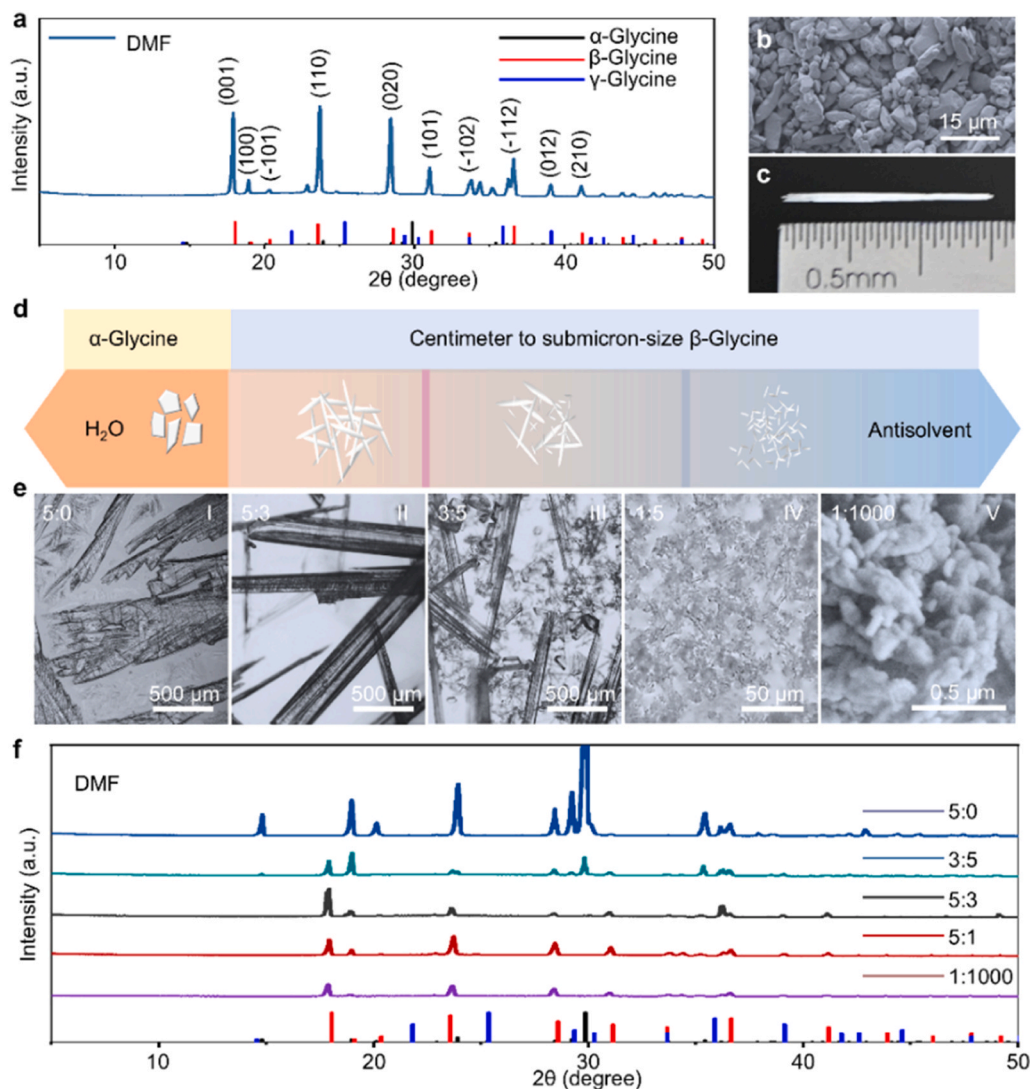


Fig. 1. Characterization of multiscale β -glycine. (a) XRD, (b) SEM image of β -glycine particle and (c) photograph of micrometer-needle-shaped β -glycine induced by DMF. (d) Schematic diagram of multiscale β -glycine induced by mixed solvents. (e) Metallographic microscope, SEM and (f) XRD images of β -glycine induced by different volume ratio of H_2O :DMF (5:0, 5:3, 3:5, 1:5, and 1:1000).

voltage, yielding an effective piezoelectric coefficient of approximately 9.4 pm/V based on the slope (Fig. 2c). The results of ferroelectric hysteresis loop measurements further demonstrated the ferroelectricity of the β -glycine crystal (Fig. 2d). The effective piezoelectric coefficient derived from the amplitude hysteresis loop in the DC off state was measured as 9.9 pm/V. This value was consistent with the linear fitting result, confirming the reliability of the measured piezoelectric coefficient.

2.2. One-step preparation of β -glycine/polymer piezoelectric films across antisolvents

Here, we proposed a simple one-step method (antisolvent method) to prepare flexible β -glycine/polymer composite piezoelectric film during directly mixing glycine aqueous, antisolvent and soluble polymer. One of the advantages of anti-solvent method is that the induced glycine crystal in different composite film is all β phase, avoiding the different glycine crystal forms induced by polymers [16,17]. According to the solubility characteristics of polymers in water-antisolvent solutions, the polymers can be divided into three types: hydrophilic polymers (only soluble in water), hydrophobic polymers (only soluble in antisolvent) and amphiphilic polymers (soluble in water, antisolvent and

water-antisolvent solution). Three water-soluble composite films of glycine/PVA, glycine/MC, and glycine/PAA were prepared in water-DMF solvent (antisolvent method) and in pure water solvent (co-dissolution-evaporation method), respectively (Fig. 3a-c). All the three polymers are soluble in water, DMF, or water-DMF solvent. Based on the co-dissolution-evaporation method, glycine exhibited different crystal forms in the three composite films. Specifically, the glycine crystals in glycine/PVA, glycine/MC, and glycine/PAA films were γ phase with piezoelectricity (Fig. 3a), α phase without piezoelectricity (Fig. 3b), and amorphous phase (Fig. 3c), respectively. In comparison, the glycine in the three composite films of glycine/PVA, glycine/MC, and glycine/PAA prepared by the antisolvent method was all β phase (Fig. 3a-c).

Furthermore, according to the solubility, the polymers can be divided into three types: hydrophilic polymers (only soluble in water), hydrophobic polymers (only soluble in antisolvent) and amphiphilic polymers (soluble in water, antisolvent and water-antisolvent). In the three types of glycine/polymer piezoelectric composite films prepared by antisolvent method (in water-antisolvent solvents), glycine was all mainly β crystal phase (Fig. 3d-g), including: glycine/PCL (hydrophobic polymers) in water-DMF, DMAc, or DMAA; Glycine/Gelatin (hydrophilic polymer) in water-MeOH, EtOH, IPA, DMF, DMAc, DMAA, NMP,

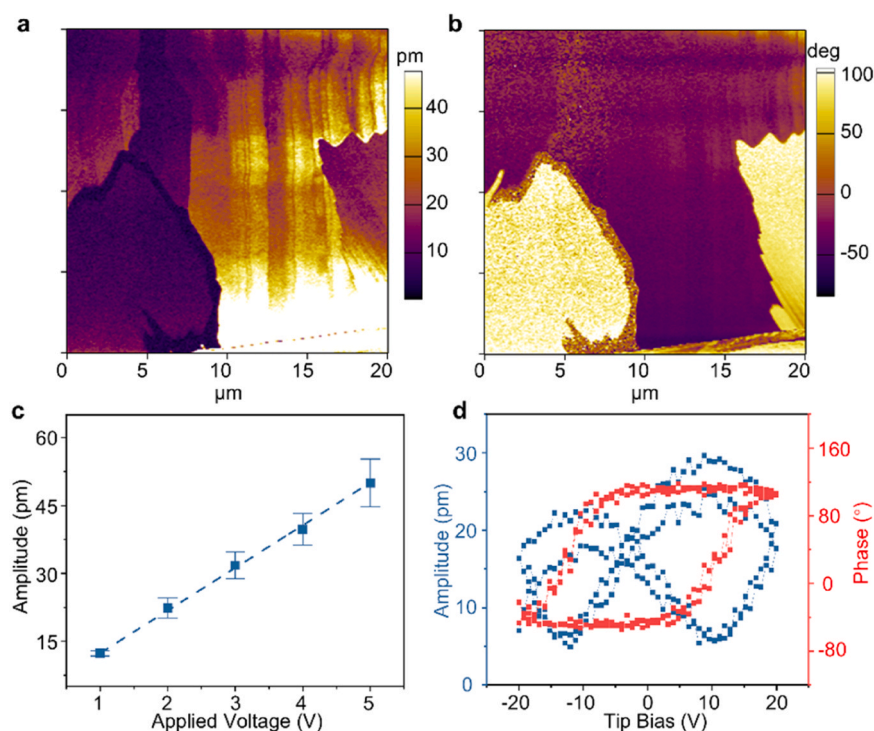


Fig. 2. Piezoelectric performance of β -glycine induced by DMF as antisolvent. (a) Amplitude and (b) phase mapping of β -glycine (the volume ratio of $\text{H}_2\text{O}:\text{DMF} = 5:3$). (c) Linear dependence of PFM amplitudes on the applied AC voltage. ($n = 5$ independent scan amplitude mapping on a glycine crystal. Data is presented as mean $\pm$ standard deviation.). (d) Ferroelectric amplitude and phase hysteresis loops with the DC off.

or CAN; glycine/PEO (amphiphilic polymers) in water-MeOH, EtOH, IPA, DMF, DMAC, DMAA, NMP, or CAN. For the preparation of the three types glycine/polymer films, the volume ratio of water to antisolvent is different, as detailed in the experimental section.

For the three types of β -glycine/polymer piezoelectric composite films, the XRD characteristic peak intensity showed a positive correlation with glycine concentration (Fig. S7a-c). Moreover, the volume ratio of water to DMF variations did not alter the β -glycine crystal form in the composite films (Fig. S7d-f), but it changed performance of the composite films and the dispersion of β -glycine due to the solubility variations of the polymer in the mixed solution. Specifically, excessive water ratio caused PCL precipitating, destroyed the integrity of the PCL-matrix film (Fig. S8a, 9a, b). Similarly, although DMF did not cause gelatin precipitation in aqueous solution (Fig. S8b), Glycine/Gelatin film was also severely defective (Fig. S9c, d). In comparison, the amphiphilic polymer PEO was soluble water-DMF with any volume ratios (Fig. S8c), enabling the intact β -glycine/PEO composite films (Fig. S9e, f). However, the crystal shapes in the β -glycine/PEO composite film were different. Specifically, glycine crystals were needle-shaped at high water volume ratios (Fig. S9e) and granular at high DMF volume ratios (Fig. S9f), consistent with the crystal formation observed without PEO during preparing (Fig. 1d).

The stability of piezoelectric film is the prerequisite for the long-term operation of piezoelectric devices. Among three types of β -glycine/polymer piezoelectric composite films, water absorption properties of hydrophilic polymer (gelatin) and amphiphilic polymers (PEO) (Fig. S10a, b) and the exposure of glycine crystals on the film surface (Fig. S11) cause β -glycine easily dissolving or undergoing crystal phase transformation in aqueous solutions or humid air, losing its piezoelectricity. In comparison, PCL is a hydrophobic polymer without dissolving or swelling in water or humid air, β -glycine wrapped by PCL well isolated from moisture, thus achieving longer-term stability of the crystal form (Fig. S10). Long-term stability studies showed that β -glycine/PCL films remained stable for 66 days without conversion to the α phase under environmental conditions (Fig. 4a), while the pure β -glycine

crystals transform into α phase after 21 days (Fig. S12). This stability was attributed to the hydrophobicity of the PCL film and its dense molecular network structure, which tightly encapsulates the β -glycine powder (Fig. 4b, c). Cross-sectional SEM images confirmed the dense network structure of PCL (Fig. 4d) and showed that β -glycine is embedded well in this structure (Fig. 4e). In addition, gelatin has a low elastic fracture strength and limited elongation at break, leading to brittle failure upon exceeding its elastic limit. PEO also has a low elastic limit but exhibits good flexibility, undergoing creep rather than immediate fracture. In contrast, PCL demonstrates higher elastic strength and superior flexibility, resulting in improved overall mechanical properties (Fig. S13). A PFM probe was utilized to scan the cross section of the β -glycine/PCL composite film to characterize its piezoelectric properties. The amplitude and phase mapping proved that the film has good electrical responsiveness (Fig. 4f, g). Additionally, the single-point butterfly curve confirmed the film's good ferroelectric properties. The effective piezoelectric coefficient of the composite film calculated by amplitude hysteresis loop was 10.3 pm/V, consistent with the (9.9 pm/V) effective piezoelectric coefficient of β -glycine crystal with same method (Fig. 2c, d).

2.3. Output performance of PENG based on β -glycine/PCL film

A β -glycine/PCL film based piezoelectric nanogenerator (PENG) was prepared to evaluate its device-level output performance. Under an 8 N force, the open-circuit voltage (V_{OC}), short-circuit-current (I_{SC}), and transferred charge (Q) of the PENG were measured as 1.8 V, 79.8 nA, and 1.7 nC, respectively (Fig. 5a-c). The reverse connection test yielded an opposite V_{OC} , confirming the authenticity of the piezoelectric output (Fig. 5d). In addition, effects of β -glycine content and the applied force on the output performance of the PENG were investigated. The results showed that V_{OC} positively correlated with increased β -glycine content. At a glycine to polymer mass ratio of 0.20–0.40, the V_{OC} reached 1.9 V (Fig. 5e). The V_{OC} increased with the applied impact force. When a 20 N force was applied, the V_{OC} reached 6.9 V (Fig. 5f), ranking among the

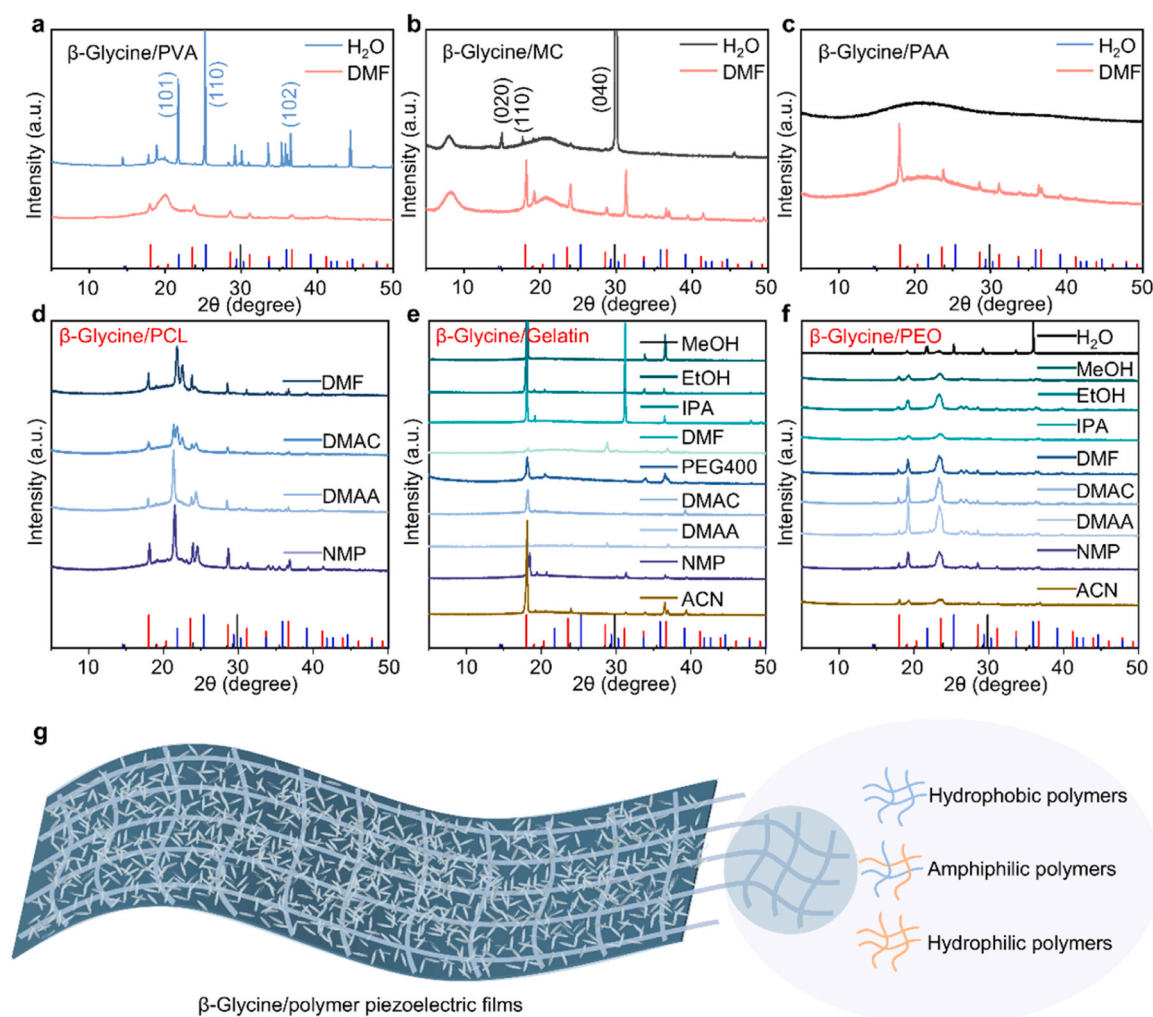


Fig. 3. XRD characteristics of piezoelectric composite films. XRD patterns of (a) glycine/PVA, (b) glycine/MC, (c) glycine/PAA composite films prepared by co-dissolution-evaporation and one-step antisolvent methods, respectively. XRD patterns of (d) β-glycine/PCL, (e) β-glycine/Gelatin and (f) β-glycine/PEO films prepared in different water-antisolvents. (g) Schematic diagram of three piezoelectric composite membranes.

best performances reported for glycine-based biodegradable piezoelectric materials (Fig. S14, Table S1) [20,32,34–38]. Although short-term fatigue testing showed that the VOC remained stable after 20000 s of continuous operation under a pulsed 4 N impact force (Fig. 5g), an obvious VOC decline was observed on the third day during continuous operation 7 days (Fig. S15). Although the crystal form of β-glycine remained unchanged, the decline was attributed to the structural damage of the PENG, including the structural damage of electrodes, piezoelectric films, encapsulation, or connection, etc. For the β-glycine/PCL film, potential stress relaxation or interfacial debonding can also induced the damage, due to PCL being a relatively soft polymer, subjecting it to several days of continuous impact force will induce changes at the microscopic level, leading to a change in the orientation of the embedded β-glycine crystals under continuous force, which we consider one of the possible reasons for the reduced piezoelectric performance in long-term stability evaluation. In our opinion, this issue is common in PCL-based piezoelectric films reported in other literature. Hence, optimizing polymer performance and device structure is an essential content for improving the long-term stability of piezoelectricity. Leveraging the favorable mechanical and electrical properties of the β-glycine/PCL-based PENG, we fabricated a finger bending angle sensor (Fig. 5h). When the finger joints were bent, the output voltage showed a linear relationship with the bending angle, indicating that the sensor can effectively monitor the bending angle of fingers (Fig. 5i). In addition, we

compared the impedance matching of PENGs fabricated from different composite films (Glycine/PCL 1:19, Glycine/PEO 19:1, Glycine/Gelatin 12:8) (Fig. S16). The output current, voltage, and power density of the PENGs exhibit similar trends: voltage increases with resistance, whereas current decreases. The power density of all three PENGs peaks at a load resistance of 16 MΩ, with corresponding values of 30.32, 28.93, and 28.38 nW/cm². Among these, the Glycine/PCL-based device exhibits the highest power generation performance. Compared with conventional piezoelectric devices, the low power density of the β-glycine/PCL-based PENG stems from two factors. First, β-glycine possesses a lower longitudinal piezoelectric coefficient than piezoelectric ceramics or polyvinylidene fluoride, and the efficiency of converting its shear piezoelectric coefficients into the film's d_{33} is also limited, leading to low d_{33} values of all the reported β-glycine-based PENGs. Second, the β-glycine has relatively random orientation within the PCL film prepared via antisolvent-induced method. Nonetheless, this method is methodologically innovative, and the film exhibits viable flexibility, stable crystal form, and capability for force-to-electric sensing of simple motions, thus deserving further study.

In addition to direct mechanical impact force, ultrasound is also an ideal driving source for PENG. The PENG generated an output voltage of approximately 620 mV driven by ultrasound (1 MHz, 2 W/cm², duty cycle 50%) (Fig. 6a). A low-level output signal was observed even when the ultrasound was turned off, it's due to contact-induced noise between

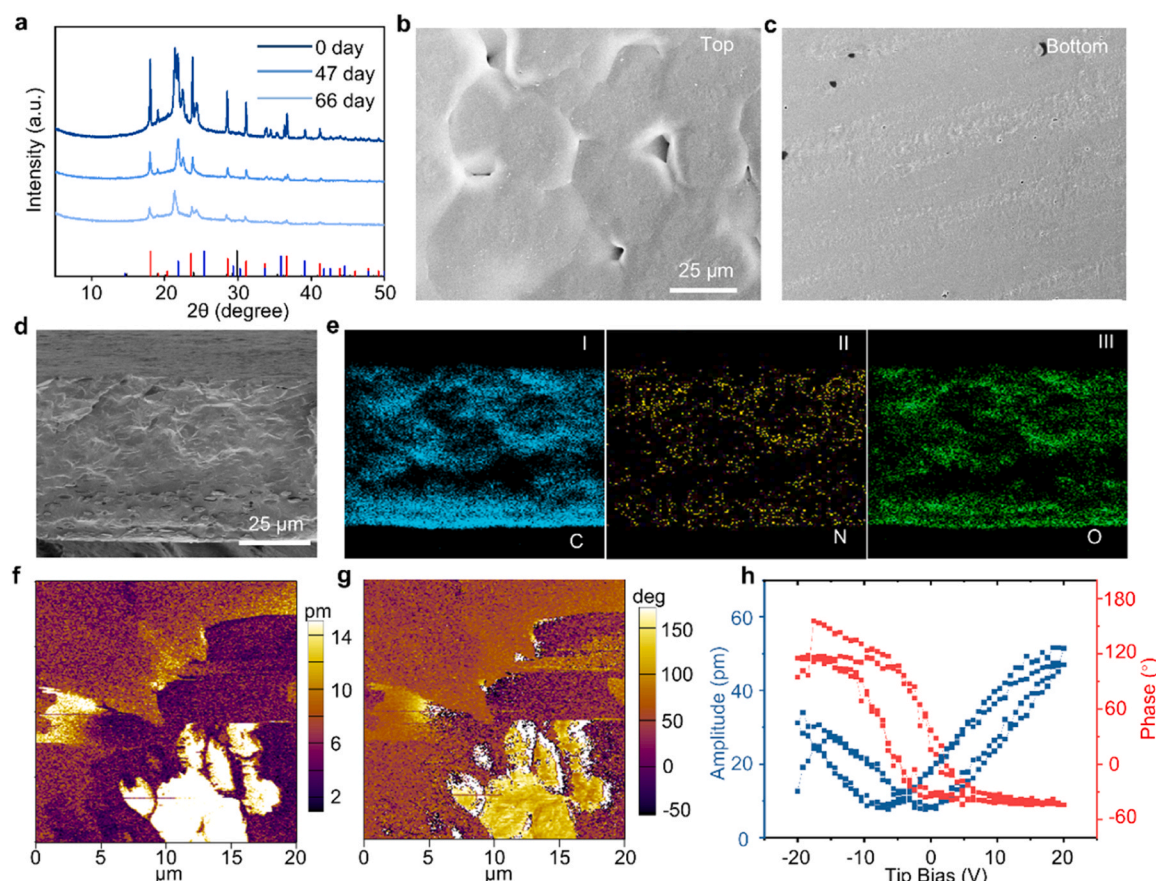


Fig. 4. Characterization of β -glycine/PCL film (the volume ratio of $\text{H}_2\text{O}:\text{DMF} = 1:20$). (a) XRD of long-term stability of β -glycine/PCL film. (b) Top surface and (c) bottom surface SEM images of β -glycine/PCL film. (d) Cross-sectional SEM image and (e) EDS mapping of β -glycine/PCL film. (f) Amplitude and (g) phase mapping of β -glycine/PCL, (h) Ferroelectric amplitude and phase hysteresis loops with the DC field off.

the ultrasound probe and the device. The baseline (ultrasound probe not contacting the device), outputs from ultrasound-driven non-piezoelectric PCL based device, and β -glycine/PCL based PENG were measured, respectively (Fig. 6b). Results indicated that the baseline output was nearly 0. Applying ultrasound to PCL based device produced an output similar to noise from the ultrasound probe contacting the device, while the output of PENG was higher than PCL based device, corroborating previous findings and confirming the authenticity of piezoelectric output. Furthermore, the materials utilized in the PENG exhibited excellent biocompatibility (Fig. 6d, e, S17). As shown in Fig. S17, the skin and subcutaneous tissues of the implantation group (30 days) exhibited normal morphology compared with the sham control group, with no evident inflammatory cell infiltration, necrosis, or fibrotic capsule formation. Consequently, the PENG was implanted into the subcutaneous abdominal tissue of rats to evaluate its *in vivo* output performance under ultrasound. The results demonstrated that the implanted PENG generated an output voltage of about 274 mV under ultrasound. Although the present output performance is insufficient to satisfy the electrical stimulation requirements of implantable devices, the device may serve as a promising implantable energy-harvesting platform for electrical stimulation-assisted tissue repair after further performance optimization.

3. Conclusions

In summary, this study developed one-step *in situ* inducing strategy to form β -glycine composite films based on the antisolvent-induced crystallization method of β -glycine. Optimizing the induce conditions, multi-scale piezoelectric β -glycine crystals from centimeter to

submicron in size were prepared. This method also provides a feasible approach for the preparation of polymer-encapsulated piezoelectric glycine microspheres. The β -glycine crystals showed excellent piezoelectricity, with effective piezoelectric coefficient ~ 9.9 pm/V measured by amplitude hysteresis loop. In addition, according to the solubility difference of polymers in water and antisolvents, three types of β -glycine/polymer films showed differences in performance and stability. Among them, β -glycine/PCL (hydrophobic polymer) film could maintain the longer crystal stability of β -glycine (66 days). The PENG based on β -glycine/PCL film showed good electrical output performance under mechanical pressure. In addition, the PENG generated about 620 and 274 mV output *in vitro* and *in vivo* under ultrasound, respectively, and with its good biocompatibility, which revealed conspicuous application prospects in implantable electrical stimulation therapeutic after further performance optimization.

4. Experimental section

4.1. Materials

Glycine (For electrophoresis, $\geq 99\%$), PEO (Mv ~ 100000), PVA (1799), MC (400 mPa), MeOH (AR, 99.5%), DMAC (DMAC, AR, 99%), PEG (Mv ~ 400), CAN (AR, 99%), PAA (Mv ~ 450000) and gelatin (glue strength ~ 250 g Bloom) were purchased from Aladdin. PCL (Mv ~ 80000) were purchased from Innochem. DMF (AR, 99.5%), DMAA (98%), NMP (AR, 99%), IPA (99.5%), EtOH (99.7%) were purchased from Macklin. Phosphate buffered saline (PBS, 1X), high glucose Dulbecco's modified Eagle medium (DMEM), sodium pentobarbital, and cell viability/cytotoxicity detection and cell counting kit-8 (CCK-8) assays

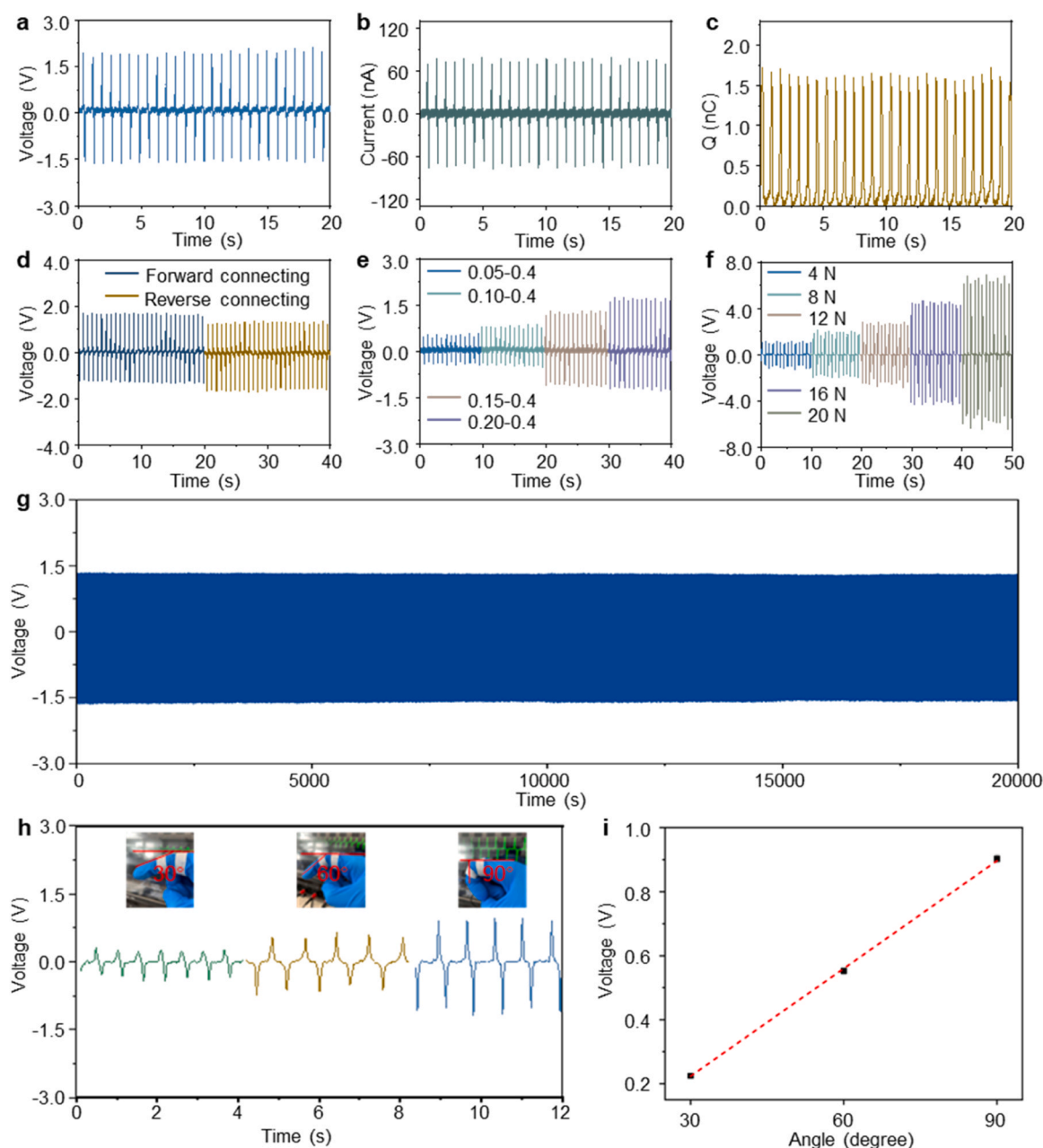


Fig. 5. Output performance of PENG based on β -glycine/PCL film under mechanical force. (a) Open-circuit voltage (V_{OC}), (b) short-circuit current (I_{SC}), and (c) charge (Q) of PENG under a pulsed 8 N force. (d) V_{OC} of PENG from forward and reverse connections. (e) V_{OC} of PENG based on β -glycine/PCL film at different glycine to PCL mass ratios, including 0.05–0.4, 0.10–0.4, 0.15–0.4, 0.20–0.4 (glycine–PCL). (f) Variation in V_{OC} with different forces. (g) Fatigue testing of PENG subjected to a pulsed 4 N force. (h) Finger joint bending angles sensor based on PENG. (i) Voltage vs. bending angle fit graph from (h).

were purchased from Solarbio.

4.2. Preparation of β -glycine crystal and β -glycine/polymer films

In this experiment, Deionized water was used as the primary solvent, while various solutions (DMF, DMAC, DMAA, MeOH, EtOH, IPA, NMP, ACN) served as antisolvents. A 10% (w/v) glycine aqueous was prepared by dissolving 2 g of glycine powder in 20 ml of deionized water. β -glycine microcrystals were synthesized by gradually adding 1 ml of 10% glycine aqueous into 20 ml of antisolvent (the volume ratio of H_2O :DMF = 5:3). To ensure uniform dispersion of β -glycine microcrystals, the mixture was stirred continuously during the addition (dropwise addition rate: one drop per five seconds; stirring rate: 500 rpm). Similarly, volume ratios of H_2O :DMF = 5:1, 5:3, 3:5, 1:5, 1:1000 were prepared by

using above method. The mixture was poured into a 6.5 cm diameter glass dish and dried at 40 °C on a heating stage for 24 h to form β -glycine crystals. In the main text and [supporting information](#), the Glycine/Polymer x:y means the Glycine/Polymer film prepared with a H_2O :DMF volume ratio of x:y; the Glycine/Polymer x-y is defined as the mass ratio of glycine to polymer.

To prepare the β -glycine/polymer films, 0.4 g polymer was dissolved in 20 ml antisolvents to form a uniform polymer solution. Subsequently, 1 ml of glycine solution with different concentrations was added dropwise to the polymer solution, resulting in a turbid mixture. Finally, the turbid mixture was transferred into a 6.5 cm diameter polytetrafluoroethylene (PTFE) dish and dried on a hot plate at 40 °C for 48 h to yield a composite film.

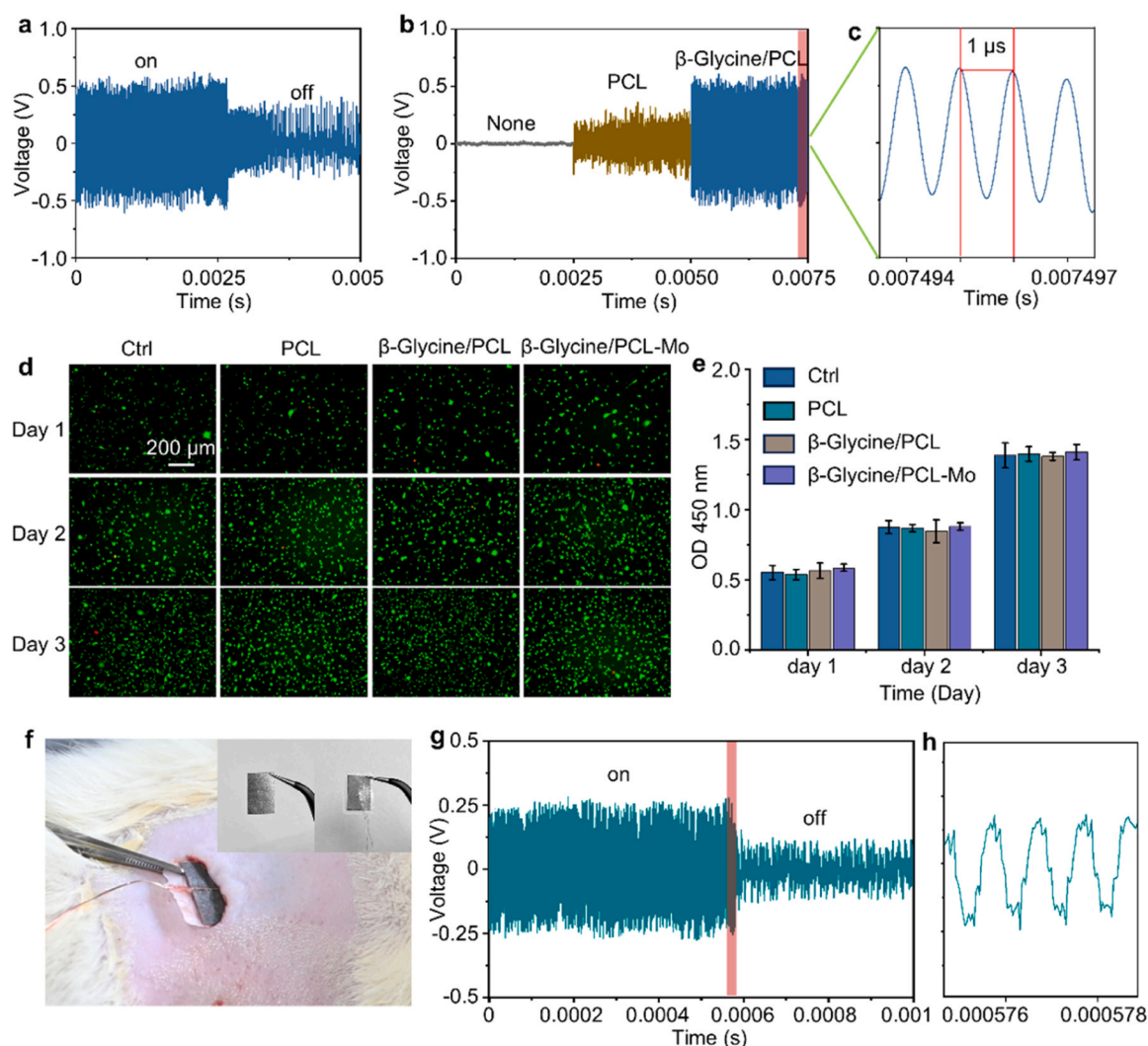


Fig. 6. Output performance of PENG under ultrasound. (a) V_{OC} of PENG based on β -glycine/PCL film with ultrasound on and off. (b) V_{OC} generated from baseline, PCL film-based device, β -glycine/PCL-based PENG, respectively. (c) The enlarged V_{OC} from (b). (d) Calcein-AM staining photographs and (e) cell viability of L929 cells in coculture with PCL, β -glycine/PCL, and β -glycine/PCL-Mo for 1, 2 and 3 days (green: live cells, red: died cells) ($n = 3$ independent samples. Data is presented as mean $\pm$ standard deviation). (f) Photographs and (g) V_{OC} of PENG implanted in rat under ultrasound stimulation. (h) Enlarged V_{OC} from (g).

4.3. Characterization of β -glycine crystal and β -glycine/polymer films

The morphology of β -glycine crystal and β -glycine/polymer composite films were characterized by scanning electron microscopy (SEM, SU8020) and metallographic microscopy (ECLIPSE, Nikon), and related elemental information were obtained energy dispersive spectroscopy (EDS) (SU8020). X-ray diffraction (XRD) patterns of glycine and polymer composite films were acquired using an X'Pert3 Powder with Cu K α radiation. The amplitude, phase mapping and hysteresis loops were conducted by a piezoelectric force microscopy (PFM, MFP-3D-SA-DV).

4.4. Preparation and characterization of PENGs based on β -glycine/PCL films

Molybdenum (Mo) was deposited on both sides of a β -glycine/PCL films as electrodes by using a magnetron sputter (635, Denton Discovery), followed by cutting the sample into 1 cm $\times$ 1.5 cm. Two lead copper (Cu) wires were connected to the Mo electrodes. Using heat-sealing technology, the Mo-deposited film was packaged by PCL. A digital oscilloscope (Teledyne LeCroy, HDO6104) and an electrometer (Keithley, 6517B) were employed to characterize the electrical output of the β -glycine/PCL films-based PENG. We obtained signed informed

consent from all human participants before the commencement of the experiments, and the wearable experiments were approved by the Committee on Ethics of Beijing Institute of Nanoenergy and Nanosystems (2025026LZ).

The ultrasonic (1 MHz, 2 W/cm², duty ratio: 50%) produced from an ultrasonic therapy device was used to driving PENG, and the output signal generated by PENG was measured using an electrometer and an oscilloscope.

The PENG was sterilized by ultraviolet light before operation. The PENG was implanted into the subcutaneous tissue of abdomen of a Sprague Dawley (SD) rat (male, 150 g - 200 g). Measuring signal output from PENG in vivo using the same method as in vitro. All animal experiments were conducted under a protocol approved by the Committee on Ethics of the Beijing Institute of Nanoenergy and Nanosystems (2025027LZ).

4.5. Cell viability

PCL, β -glycine/PCL, and β -glycine/PCL-Mo were soaked in DMEM culture medium for 3 days, which was filtered to obtain the supernatant culture media. L929 cells (5×10^3 cells per well, 100 μ L per well) were cultured with the above media and incubated in a humid atmosphere

containing 5% CO₂ at 37°C for 1, 2, and 3 days. Cell viability/cytotoxicity detection and CCK-8 assays were carried out to evaluate the biocompatibility of these materials. The optical density value at a wavelength of 450 nm was measured with a multimode microplate reader (Thermo Fisher Multiskan FC, Milton Freewater, OR, USA). Immunofluorescence images were acquired with a Leica confocal fluorescence microscope (LEICA TCS SP8).

CRediT authorship contribution statement

Hongyu Meng: Writing – review & editing, Writing – original draft, Visualization, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Meng Xiao:** Methodology. **Laipan Zhu:** Methodology. **Xi Cui:** Methodology. **Juwei Yang:** Methodology. **Di Wu:** Software. **Zhou Li:** Writing – review & editing, Supervision, Resources, Project administration, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Yudong Hou:** Supervision, Methodology. **Xiongxin Luo:** Methodology. **Zhi Luo:** Formal analysis. **Cong Li:** Methodology. **Wenlong Xu:** Methodology. **Yuan Bai:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Dongjie Jiang:** Validation, Formal analysis. **Qiao Yu:** Methodology.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.nanoen.2026.112075](https://doi.org/10.1016/j.nanoen.2026.112075).

Data availability

Data will be made available on request.

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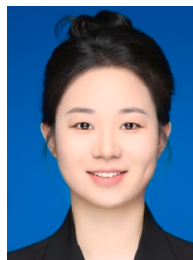
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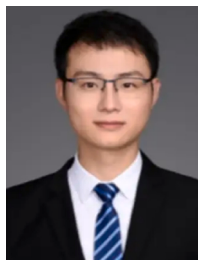
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