

Damage-free dry transfer method using stress engineering for high-performance flexible two- and three-dimensional electronics

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Supplementary Note 1. Limitations of previously reported transfer printing methods

The challenges associated with various thin-film transfer methods include the requirement of expensive laser setups and transparent substrates in the laser lift-off method ¹, the presence of a residual thick substrate hindering the fabrication of ultrathin flexible electronics and the requirement of an additional post-etching process in the controlled thermal spalling method ²⁻⁴, the unwanted atomic diffusion and thermal damage that can occur during annealing in the thermal stress-assisted transfer printing method ⁵. Additionally, other unwanted side-effects may occur from high-temperature annealing ^{5,6}, especially in the air environment ⁶⁻⁸. Therefore, a new process for facile dry transfer of thin films should be developed for the fabrication, and also toward the commercialization, of high-performance soft electronic devices.

Supplementary Note 2. Generation of tensile stress in film formation and deformation

When atoms are vaporized under vacuum conditions and deposited on a substrate, residual tensile stress can be generated within the deposited film. This stress depends on deposition conditions such as energy of the deposited atoms, and interaction between the deposited atoms and the substrate (Extended Data Fig. 2a) ^{9, 10}. Additionally, a large lattice constant mismatch in a bilayer structure (Extended Data Fig. 2b) ¹¹ and external strain induced by mechanical bending deformation (Extended Data Fig. 2c) ^{12, 13} can amplify the internal stress gradient and increase the overall stress level. Ultimately, if the tensile stress and its gradient become large while the adhesion between the deposited film and the substrate is low, the deposited film becomes delaminated from the substrate.

Supplementary Note 3. Numerical simulation of the thin film

To validate this stress-enhancement strategy, we conducted numerical simulations to estimate the stress evolution induced by bending deformation in the single-layer and bilayer Pt thin films (Supplementary Fig. 1). The sample conditions for the simulation is depicted in Supplementary Fig. 1a. The single-layer structure consists of a 1000 nm thick Pt layer on a 725 μm thick Si wafer covered with a 300 nm thick SiO_2 . The Pt thin film has an initial stress of 800 MPa (Supplementary Fig. 1a, i). On the contrary, the bilayer structure comprises two consecutive layers of Pt, each 500 nm thick (Supplementary Fig. 1a, ii). The first and second layers have initial stress of -200 MPa and 800 MPa, respectively. The stress is assumed to be identical along the thickness direction of the film. The simulation was conducted to model a case in which the edge of the sample (Pt on wafer), sized 65 mm in length and 10 mm in width, undergoes upward-bending by a magnitude of 2.5 mm (Supplementary Fig. 1b). The Pt thin film and the wafer were modeled using linear hexahedral solid elements with reduced integration (C3D8R), with 20 and 30 elements in the thickness direction, respectively (Supplementary Fig. 1c). A cylinder with a diameter of 10 mm used for bending was modeled using discrete rigid elements (R3D4). The linear elasticity was used to represent the behavior of the layers and substrate. The Young's moduli of Pt and Si are 154 GPa and 140 GPa, respectively. The Poisson's ratios of Pt and Si are 0.389 and 0.265, respectively (These figures were referenced from the documents on the websites "Platinum-Properties and Applications"; (www.azom.com/properties.aspx?ArticleID=601) and "A Background to Silicon and its Applications"; (www.azom.com/properties.aspx?ArticleID=599)). The coordinate system was chosen so that the x, y, and z axes correspond to the longitudinal, thickness,

and width directions, respectively (Supplementary Fig. 1d). In order to simplify the condition, a quarter of the specimen excluding the gripped regions (measuring $30 \times 5 \text{ mm}^2$) was modeled, accounting for symmetry while disregarding the presence of SiO_2 in the wafer.

At the edge of the specimen on the grip side, the displacement was constrained while permitting the rotation around the z axis. The prescribed initial stresses were applied to the single and bilayer structures. The initial stress was assumed to be identical along the thickness direction of the film. Subsequently, these imposed initial stresses were released to achieve force equilibrium before bending. To simulate bending, the rigid cylinder, initially positioned just beneath the specimen, was displaced by 2.5 mm in the y-direction.

The distribution of normal stress in the x-direction along the y-direction is presented in Supplementary Fig. 1e for both the single and bilayer Pt film structures. Specifically, the normal stress distribution along the y-direction (thickness-direction) at the center of the specimen is plotted in Fig. 1d,1e.

Supplementary Note 4. Theoretical analysis of the thin film

1) Analytical model of the single layer film

The details of the stress distribution between the film and the substrate can be very complex. Nonetheless, some significant generalizations can be made for theoretical analysis. Extended Data Fig. 3a illustrates an idealized representation of the stress distribution across the film and substrate ¹⁰. This distribution of stress is determined by normal forces and bending moments across the entire cross-section, both resulting in a net sum of zero ¹⁴ (due to the absence of slippage between the film and the substrate). If the stress of the film is expressed as a linear function and the thickness of the substrate and film is known, the expression for the substrate can also be derived.

2) Analytical model of the bilayer film

In the case of bilayer deposition, it refers to a situation where the second layer with different conditions is deposited after the first layer. In this case, the changes can be calculated by considering the existing substrate and the first layer together as a new substrate ¹⁵. By adding the changes that occur when depositing the second layer to the situation where the 1st layer is deposited, the final expression for the substrate and the first layer can be obtained (Extended Data Fig. 3b).

3) Modeling of the cantilever deflection

In the case of the cantilever, when the thin film starts to detach from the substrate, it separates completely, leaving only the tip on one side. In such cases, the higher the tensile stress gradient in the z-direction, the more the film lifts upward. The deflection can be

calculated using the following equation ¹⁶:

$$\delta = \frac{ML^2}{2EI}$$

where M is the bending moment, L is the film length, $\bar{E}$ is the in-plane Young's modulus, and I is the area moment of inertia (Extended Data Fig. 3c, iv). To determine the bending moment, the average stress value of the film is obtained through force balance (Extended Data Fig. 3c, ii), and this value is then used to calculate the bending moment of the film that has detached from the substrate (Extended Data Fig. 3c, iii). We observed that as the stress difference or gradient between the top and bottom layer increases, the deflection of the cantilever also increases (Extended Data Fig. 3d).

4) Analysis of bending stress

When an external mechanical force is applied, it induces additional stress, resulting in the bending of both the substrate and the film. By incorporating the effect of bending into the distribution of residual stress, the resulting stress distribution can be determined (Extended Data Fig. 3e). When the residual stress remains constant at a tensile stress of 800 MPa, we can calculate the change in the energy release rate and the stress depending on the bending height of the wafer (Extended Data Fig. 4c). Additionally, variation in the energy release rate can be obtained when the residual stress ranges from -200 to 800 MPa, under constant bending height (Extended Data Fig. 4d).

5) Strain energy release rate of thin film

The residual stress can be divided into two components: average stress and gradient ¹⁷. The average stress primarily determines the in-plane strain, while the gradient determines

the bending moment and resulting curvature. Since strain is determined by stress, the strain energy release rate can also be considered separately. Therefore, the total strain energy release rate can be expressed using the following equation (Extended Data Fig. 4)¹⁸.

$$\frac{\sigma_{avg}^2 h}{2E} + \frac{M^2}{2EIw}$$

where σ_{avg} is average stress, h is the film thickness, and w the width of the film. The former represents the in-plane energy release rate, while the latter represents the bending energy release rate. If the total strain energy exceeds the interfacial toughness (or fracture toughness), the residual stress can act as a driving force for delamination to occur¹⁹.

Supplementary Note 5. Explanation of the generation of stress and its gradient from a crystallographic perspective

The generation of stress and its gradient in the bilayer can also be explained from a crystallographic perspective. First, the Pt thin films were prepared with the conditions that induce moderate compressive and strong tensile stresses. Using X-ray diffraction (XRD)- $\sin^2\psi$ technique, we measured the d-spacing values of the Pt(111) planes by varying the angle (ψ) between the surface normal and the bisector of incident and diffracted beams (Supplementary Fig. 2a), and obtained the linear relationship between $\sin^2\psi$ vs. d-spacing (Fig. 2e, left). According to the measurement principle, the angle (ψ) also represents the orientation of a crystal plane relative to the film surface²⁰. Also, lower $\sin^2\psi$ values indicating crystal planes horizontal with respect the film surface, and higher values indicating predominantly perpendicular planes. Specifically, $\sin^2\psi = 0$ corresponds to the Pt(111) planes horizontal to the film surface (Supplementary Fig. 2b), with decreased d-spacing compared to the stress-free condition (Fig. 2e, left). Conversely, $\sin^2\psi = 0.9$ corresponds to a crystal plane tilted at an angle of 76° with respect to the sample surface (Supplementary Fig. 2c), exhibiting increased d-spacing compared to the normal condition (Fig. 2e, left).

Supplementary Note 6. Estimation of stress gradient for Pt thin films

To estimate the stress gradient for different film structures, we measured the deflection in cantilever-shaped Pt thin films ¹⁶. The Pt thin film is patterned into a cantilever shape, and the underlying sacrificial layer is etched away (Supplementary Fig. 3a). Consequently, the edge of the cantilever pattern rises up due to the tensile stress and its gradient. The stress gradient ($\nabla\sigma$) can be calculated by using the following equation ¹⁶:

$$\nabla\sigma = \frac{2\bar{E}\delta}{L^2} \quad (1)$$

where $\bar{E}$ represents the in-plane Young's modulus of the deposited material (in this case, Pt film), and δ and L are the deflection and length of the cantilever (Supplementary Fig. 3b). The single-layer Pt film was deposited at 10 mTorr (Supplementary Fig. 3c). To further enhance the stress gradient, bilayer Pt films were formed by sequentially depositing a weak tensile or a compressive layer (deposited at 7 or 5 mTorr), followed by a tensile layer (deposited at 10 mTorr) (Supplementary Fig. 3d,3e).

Supplementary Note 7. Transfer printing of other metal thin films

We have explained the concept of transfer printing by utilizing tensile stress and stress gradient within thin films. Levering the concept, we also demonstrate transfer printing of other metal thin films onto a flexible substrate (Supplementary Fig. 4). The Ni thin film exhibits intrinsically high tensile stress (Fig. 2b), facilitating its pick-up from the wafer and subsequent printing onto a flexible substrate (Supplementary Fig. 4a). However, in the case of the Cu thin film, its deposition via thermal evaporation results in a low level of tensile stress within the Cu thin film (Fig. 2b). This low tensile stress alone is insufficient to overcome the adhesion between the deposited Cu thin film and the underlying SiO₂ surface (Supplementary Fig. 4b). Instead, the formation of a bilayer structure with different stress levels enables successful transfer printing (Supplementary Fig. 4c).

Supplementary Note 8. Calculation of residual stress of thin film using X-ray diffraction (XRD)- $\sin^2\psi$ technique

First, the diffraction angle (2θ) is measured while varying the angle (ψ) between the surface normal and the bisector of incident and diffracted beams (Supplementary Fig. 2a). Then, the stress level is derived from the relationship between $\sin^2\psi$ and d-spacing. To demonstrate the bending of the deposited film, wafers with a Pt layer are attached to the surface of convexly-curved and concavely-curved stages using a glue (Supplementary Fig. 5). These stages simulate outward (tensile force) and inward (compressive force) bending situations, respectively. The relationship between d-spacing and $\sin^2\psi$ for each case is shown in Fig. 2h. On the concavely-curved stage, the slope of the plot decreases, while it increases on the convexly-curved stage. A positive slope indicates tensile stress²¹, and a higher slope implies the stronger tensile stress. Finally, the residual stress (Fig. 2i) can be calculated using the following equation^{22, 23}:

$$\sigma = \frac{E}{1+\nu} \frac{m}{d_0} \quad (2)$$

where E is Young's modulus of the deposited material (i.e., Pt film: 154 GPa), ν is the Poisson ratio of 0.389, m is the slope ($\sin^2\psi$ vs. d-spacing), and d_0 is the strain free inter-planar spacing. This experimental analysis confirms that bending deformations enhance or reduce film stresses, consistent with numerical simulations (Fig. 1d,1e).

Supplementary Note 9. Transfer of 2D Pt thin films and their transformation into 3D architectures

By using the proposed dry transfer printing method, various patterns of 2D Pt films can be transferred onto flexible substrates in either 2D or 3D forms. For the bilayer, the first Pt layer is deposited at a pressure of 5 mTorr (weak compressive condition), followed by the second layer at 10 mTorr (strong tensile condition), each with a thickness of 500 nm. Initially, the Pt film is patterned as "THIN FILM" using photolithography and etching steps (Fig. 3a, i). Then, the patterned thin film is retrieved using a 20:1 PDMS stamp (Fig. 3a, ii) and subsequently transferred onto a receiver substrate with a patterned ultraviolet (UV) curable adhesion layer (Norland Optical Adhesive NOA 73) (Fig. 3a, iii). Upon removal of the stamp, the film adheres to the receiver substrate, while the non-adhered portion pops up, resulting in a 3D structure with lifted letters (Fig. 3a, iv).

To showcase the versatility of our 3D transfer printing process further, we demonstrate transfer of Pt thin films with various shapes onto substrates with different patterns of the adhesion layer (Fig. 3b-e). Our approach allows for the fabrication of diverse 3D architectures depending on how the adhesion layer is patterned. In Fig. 3b, a pattern composed of three blades is transferred to the prepared substrates. Different 3D structures are formed depending on whether the adhesion layer is present on the edges of the blades (Fig. 3b, i) or in the middle (Fig. 3b, ii). When the adhesion layer is present at the center in the circular pattern, a structure with the lifted ends of the blades is created (Fig. 3b, iii). Figure 3c shows the transfer of a pattern consisting of four circles merged with thin lines. Depending on whether the adhesion layer is located on the straight line (Fig. 3c, i) or diagonal line (Fig. 3c, ii), different 3D structures are formed. When the

adhesion layer is shaped like a square, a structure with lifted outer parts of the four circles is created (Fig. 3c, iii). In Fig. 3d, a pattern in the shape of a square is transferred. The film appears to be rolled in a direction perpendicular to the adhesion layer, whether it forms a rectangular, triangular, or diagonal line shape (Fig. 3d, i to iii). As shown in Fig. 3e, a pattern in the shape of an elongated parallelogram is transferred. When the adhesion layer occupies half of the pattern, the other half is lifted (Fig. 3e, i). In the case of the triangular shape, a helical structure is observed (Fig. 3e, ii). For the small parallelogram shape, it is rolled along the longer side (Fig. 3e, iii).

Supplementary Note 10. Demonstration of the simultaneous and continuous measurement of ion concentration and temperature

To demonstrate the simultaneous and continuous measurement of ion concentration and temperature using the integrated sensor array, we prepared solutions with varying KCl concentrations and temperatures (Fig. 4i). Both ion concentration and temperature were monitored by sequentially flowing these solutions onto the integrated sensor at a rate of 400 mL s^{-1} (Fig. 4j). The ion concentration sensor exhibits an immediate response to changes, whereas the temperature sensor shows a slight delay in resistance variation, particularly when the temperature changes significantly (Stage I and III). Additionally, the values measured by the ion concentration sensor seem to be influenced by temperature variations (Stage II $\rightarrow$ III and Stage IV $\rightarrow$ V). Therefore, for practical applications, it may be needed to integrate an additional compensation circuit.

Supplementary Note 11. Reasons behind the selection of a thin-film battery as a model system

For dry transfer printing of thin films, metals such as Ni, Cu, and Mo have been suggested as a releasing layer owing to their weak adhesion to the substrate ^{7,8}. However, it is only available when the film is processed at low temperature or in a reducing atmosphere because most of these materials are easily oxidized in contact with oxygen at high temperature, making the transfer process difficult. This is a critical limitation in fabricating various functional thin films that require high temperature heat treatment and oxidation processes. In contrast, Pt is highly resistive to oxidation even at high temperature under ambient or oxygen atmosphere. Thus, the current transfer process based on stress engineering enables the dry transfer of functional thin films that have undergone high-temperature heat treatment and oxidation processes. To demonstrate these advantages, a thin-film battery is selected as a model system, because it requires high-temperature processes under ambient or oxygen atmosphere to form a high-performance metal oxide cathode on a metal thin-film current collector.

Supplementary Table 1. Categorized comparison of advantages and disadvantages among different transfer printing methods.

	Sacrificial layer etching 1, 5, 24-27	Interfacial delamination in water 6, 28-30	Laser lift-off 1, 31-33	Controlled spalling 2-4	Thermal stress transfer 5	This work
Dry/wet	Wet	Wet	Dry	Dry	Dry	Dry
Special caution in experiments	Etching solution (HF ^{5, 24-27} & KOH ²⁴)	N/A	Laser beam	N/A	N/A	N/A
Device damage	Highly reactive etchants, uncontrollable fluidic motion	N/A	Requires precise control during laser ablation	Risk of damage during fracture propagation	Risk of damage due to thermal stress, atomic diffusion	N/A
Time consumption	Long (Si membrane: etching in HF for 6-12h) ²⁶	Relatively short (depending on water immersion)	Depending on laser equipment - 2 m/s ³² - 1.0-1.5 mm/s ³³	Δ (when heating and cooling involved)	Short (3s heating & 3s cooling) ⁵	Short * (No pretreatment/post-treatment)
Special equipment/ substrate	Expensive wafer (for instance, Si on insulator wafer)	N/A: general semiconductor manufacturing process	Expensive laser equipment & transparent substrate	N/A	N/A	N/A
Unit device dimension	Micron-sized device array (unit device: 250 μm $\times$ 250 μm) ²⁷	Wafer scale (4" wafer) ⁶	Wafer scale (2" GaN wafer) ³¹	Wafer scale (2" GaN wafer) ⁴	Wafer scale (scale up to a few submeters) ⁵	Wafer scale (6 cm $\times$ 3 cm)
High-temperature annealing	O (Si and sapphire wafers)	X (Oxidation of Ni)	O (Sapphire wafers)	X Under polymer decomposition temperature	X Atomic diffusion at 363 °C for Si-Au	O (Si/SiO ₂ wafer)

* As the bilayer serves as a material component of the fabricated devices, the formation time of the bilayer was not factored into the transfer printing process time but was considered as the material preparation time.

Supplementary Table 2. Adhesion force of Pt thin films with single or bilayer configurations prepared at different conditions.

Sample #		Argon pressure (mTorr)	Residual stress ² (GPa)
S1 ¹		5	-0.386
S2		7	0.128
S3		10	0.764
B1	1st layer	7	0.128
	2nd layer	10	0.764
B2	1st layer	5	-0.386
	2nd layer	10	0.764

¹S and B denote “single layer” and “bilayer”, respectively.

²Residual stress when the film is deposited on a Si/SiO₂ wafer.

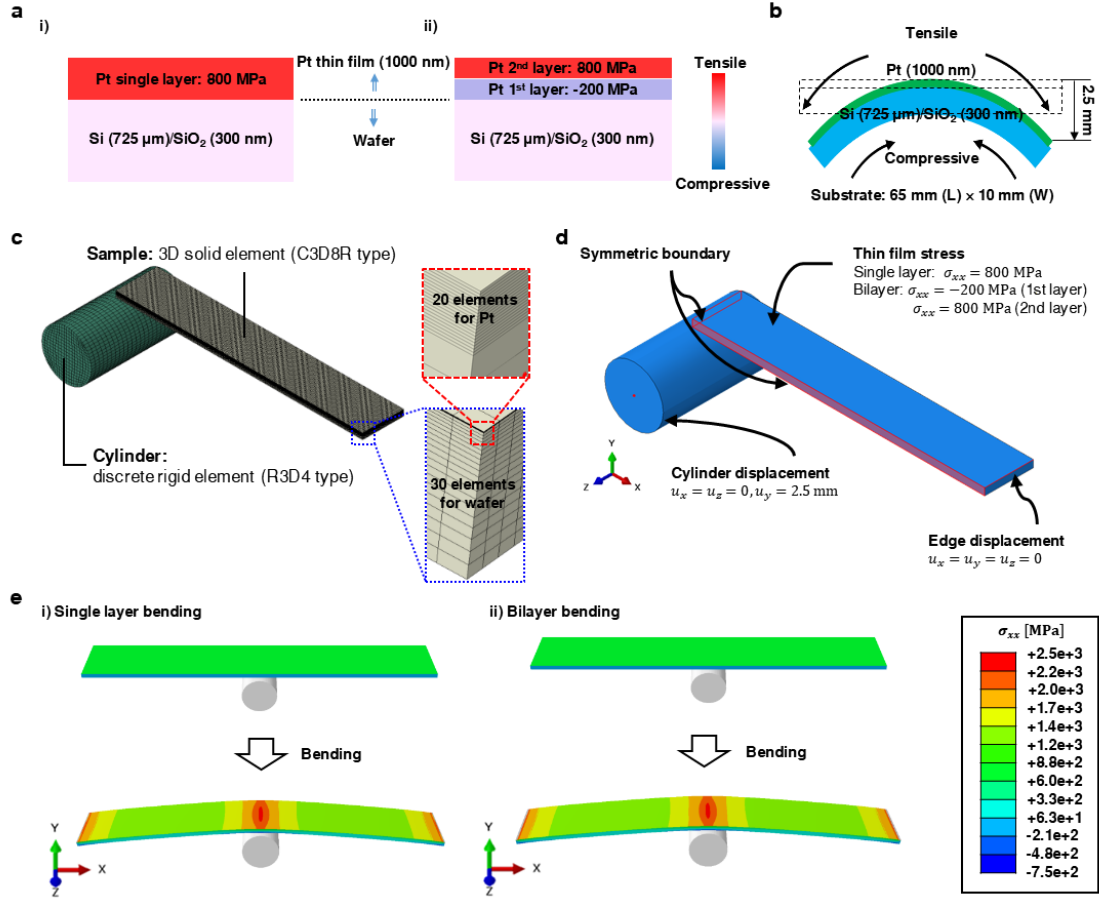


Fig. S1. Numerical simulation of Pt thin film. **a**, Single and bilayer of Pt thin films on the Si/SiO₂ wafer. **b**, Schematic illustration of the mechanical bending deformation. **c,d**, Mesh model (c) and boundary conditions (d) for finite element model simulation. **e**, Stress distribution of the single and bilayer before and after bending deformation.

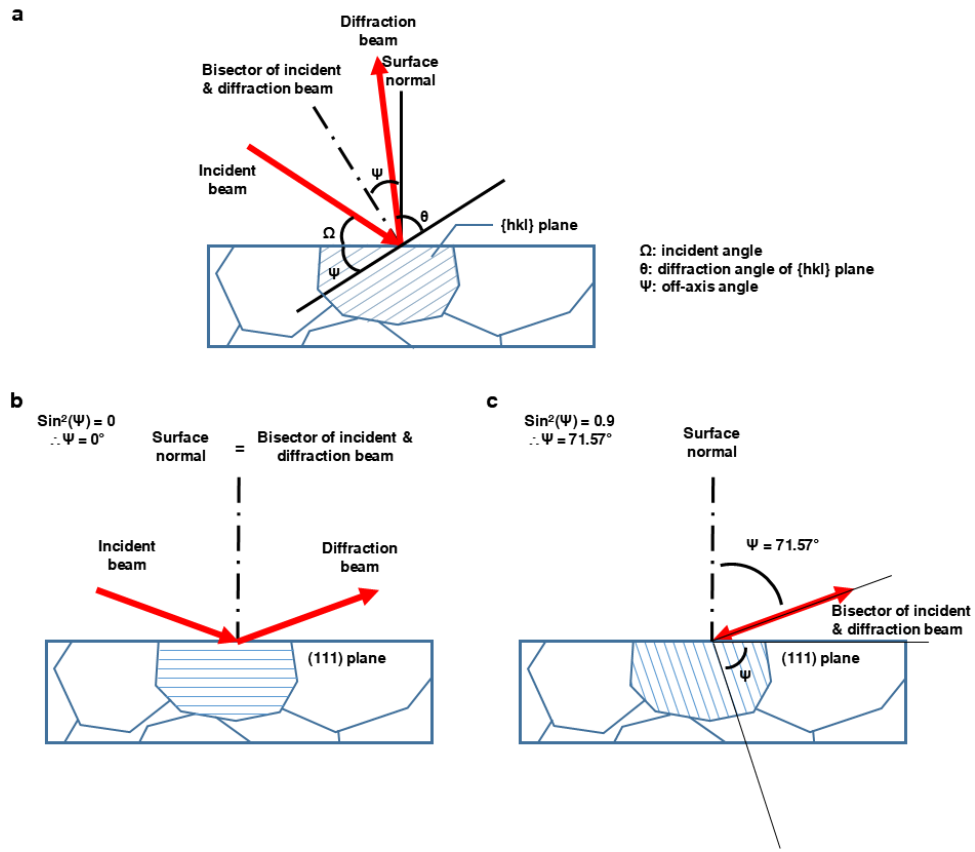


Fig. S2. Observation of crystal orientation of Pt(111) plane using X-ray diffraction (XRD) analysis. a, Schematic illustrations of fundamentals of X-ray diffraction (XRD) analysis. **b,c,** Crystal orientation of Pt(111) plane at $\sin^2\psi = 0$ (b) and 0.9 (c).

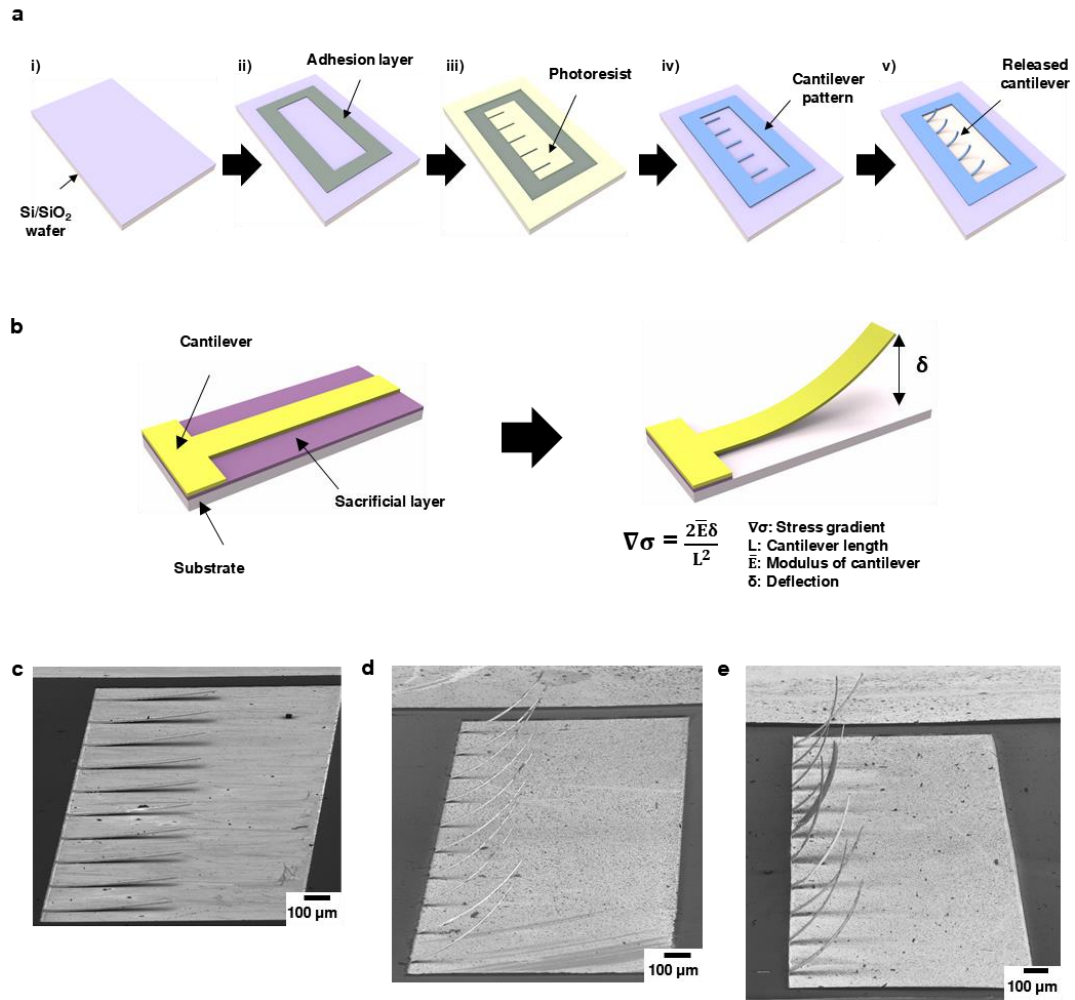


Fig. S3. An array of cantilevers for the measurement of stress-gradient in the deposited film. a, processing steps for preparing a cantilever. (i) Si/SiO₂ wafer. (ii) Patterning of adhesion layer. (iii) Patterning of photoresist to define cantilever patterns. (iv) Deposition of metal thin film and lift-off to define the cantilever patterns. (v) Etching the underneath supporting layer (SiO₂ layer). **b,** Calculation of the stress gradient through cantilever deflection. **c-e** SEM images of the cantilevers. Cantilevers with single layer condition deposited at 10 mTorr (c). Cantilevers with bilayer condition deposited at 7 and 10 mTorr, subsequently (d). Cantilevers with bilayer condition deposited at 5 and 10 mTorr, subsequently (e).

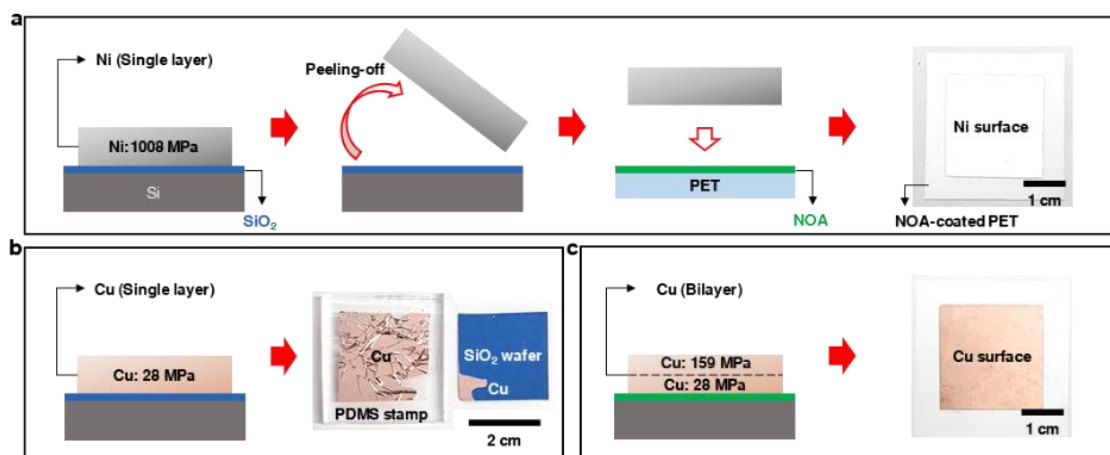


Fig. S4. Transfer printing of different metals onto a flexible substrate. a, Transfer printing process of a single layer of the Ni thin film onto a PET substrate, along with a picture of the transferred Ni thin film. **b**, Pick-up of a single layer of the Cu thin film from a Si/SiO₂ wafer with a PDMS stamp. **c**, Transfer printing of the Cu thin film utilizing a bilayer structure.

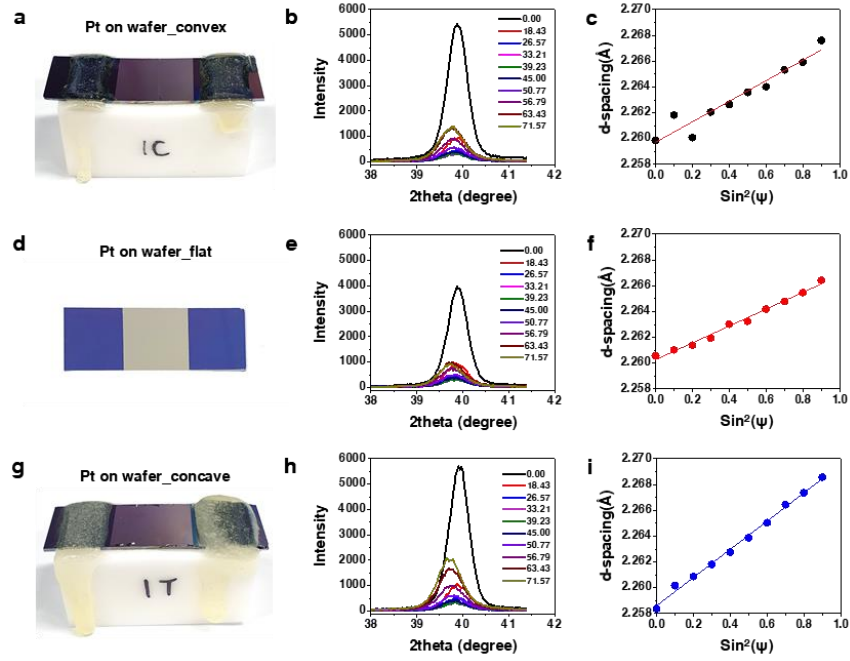


Fig. S5. Measurement of residual stress using XRD analysis. **a**, Picture of Pt thin film on wafer (Si/SiO₂) to the convex-curved stage. **b**, XRD peaks corresponding to Pt(111) at different ψ values. **c**, Variations in d-spacing corresponding to $\sin^2\psi$. **d**, Picture of Pt/wafer. **e**, XRD peaks at different ψ values. **f**, Variations in d-spacing corresponding to $\sin^2\psi$. **g**, Picture of Pt/wafer attached to the concave-curved stage. **h**, XRD peaks at different ψ values. **i**, Variations in d-spacing corresponding to $\sin^2\psi$.

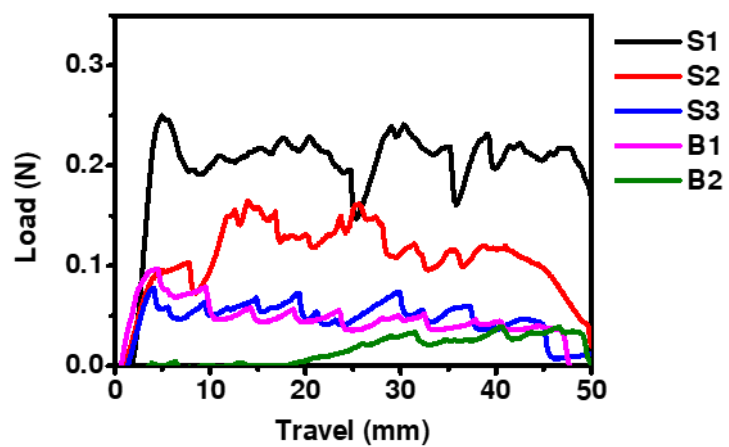


Fig. S6. Adhesion force of Pt thin films with single or bilayer configurations prepared at different deposition conditions.

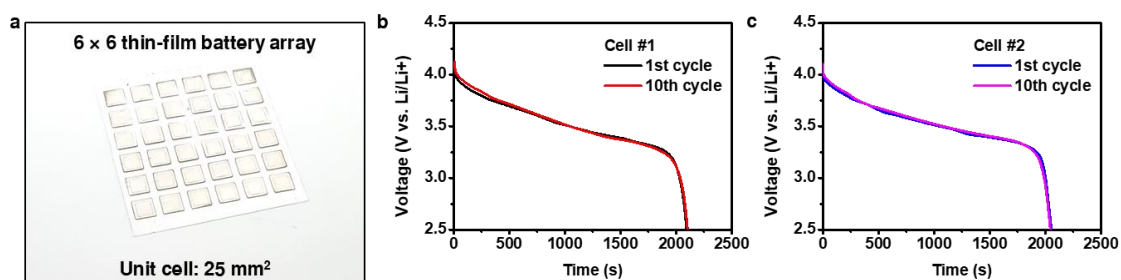


Fig. S7. Fabrication of high density thin-film batteries. **a**, Picture of a flexible 6 × 6 thin-film battery array fabricated by transferring high-temperature-annealed Pt/LCO films onto the PET substrate. **b and c**, Discharge curves of two arbitrarily selected samples at the first and tenth cycles.

Supplementary Movie 1. Retrieval of the patterned Pt bilayer thin film onto the PDMS stamp.

Supplementary Movie 2. Delamination of the deposited Pt thin film when tensile stress is generated inside the film.

Supplementary Movie 3. Delamination of the Pt bilayer thin film.

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