Journal of Visualized Experiments

Fabrication method of polymer-based piezoelectric vibration energy harvester with 3D meshed-core structure
--Manuscript Draft--

Article Type:	Invited Methods Article - JoVE Produced Video
Manuscript Number:	JoVE59067R2
Full Title:	Fabrication method of polymer-based piezoelectric vibration energy harvester with 3D meshed-core structure
Keywords:	Vibration energy harvesting, PVDF, SU-8, 3D photolithography, Polymer device, Mesh structure, Low frequency
Corresponding Author:	Takaaki Suzuki Gunma Daigaku Kiryu-shi, JAPAN
Corresponding Author's Institution:	Gunma Daigaku
Corresponding Author E-Mail:	suzuki.taka@gunma-u.ac.jp
Order of Authors:	Takaaki Suzuki
	Takuya Tsukamoto
	Yohei Umino
	Kotaro Hashikura
	Sachie Shiomi
	Kou Yamada
Additional Information:	
Question	**Response**
Please indicate whether this article will be Standard Access or Open Access.	Standard Access (US$2,400)
Please indicate the **city, state/province, and country** where this article will be **filmed**. Please do not use abbreviations.	Kiryu, Gunma, Japan

1   **TITLE:**
2   A Polymer-based Piezoelectric Vibration Energy Harvester with a 3D Meshed-Core Structure
3
4   **AUTHORS AND AFFILIATIONS:**
5   Takuya Tsukamoto[1], Yohei Umino[1], Kotaro Hashikura[1], Sachie Shiomi[1], Kou Yamada[1], Takaaki
6   Suzuki[1,2]
7
8   [1]Gunma University, Gunma, Japan
9   [2]JST PRESTO, Saitama, Japan
10
11 **Corresponding Author:**
12 Takaaki Suzuki
13 suzuki.taka@gunma-u.ac.jp
14
15 **Email Addresses of Co-authors:**
16 Takuya Tsukamoto   (t13305020@gunma-u.ac.jp)
17 Yohei Umino   (t13302013@gunma-u.ac.jp)
18 Kotaro Hashikura   (k-hashikura@gunma-u.ac.jp)
19 Sachie Shiomi (sshiomi@gunma-u.ac.jp)
20 Kou Yamada   (yamada@gunma-u.ac.jp)
21

22 **KEYWORDS:**
23 vibration energy harvesting, PVDF, SU-8, 3D photolithography, polymer device, mesh structure,
24 low-frequency
25

26 **SUMMARY:**
27 In this study, we fabricated a flexible 3D mesh structure and applied it to the elastic layer of a
28 bimorph cantilever-type vibration energy harvester for the purpose of lowering resonance
29 frequency and increasing output power.
30

31 **ABSTRACT:**
32 In this study, we fabricated a flexible 3D mesh structure with periodic voids by using a 3D
33 lithography method and applying it to a vibration energy harvester to lower resonance frequency
34 and increase output power. The fabrication process is mainly divided into two parts: three-
35 dimensional photolithography for processing a 3D mesh structure, and a bonding process of
36 piezoelectric films and the mesh structure. With the fabricated flexible mesh structure, we
37 achieved the reduction of resonance frequency and improvement of output power,
38 simultaneously. From the results of the vibration tests, the meshed-core-type vibration energy
39 harvester (VEH) exhibited 42.6% higher output voltage than the solid-core-type VEH. In addition,
40 the meshed-core-type VEH yielded 18.7 Hz of resonance frequency, 15.8% lower than the solid-
41 core-type VEH, and 24.6 μW of output power, 68.5% higher than the solid-core-type VEH. The
42 advantage of the proposed method is that a complex and flexible structure with voids in three
43 dimensions can be relatively easily fabricated in a short time by the inclined exposure method.
44 As it is possible to lower the resonance frequency of the VEH by the mesh structure, use in low-

45 frequency applications, such as wearable devices and house appliances, can be expected in the
46 future.
47
48 **INTRODUCTION:**
49 In recent years, VEHs have drawn much attention as an electric power supply of sensor nodes for
50 implementing wireless sensor networks and Internet of Things (IoT) applications[1-8]. Among
51 several types of energy conversion in VEHs, piezoelectric-type conversion presents high output
52 voltage. This type of conversion is also suitable for miniaturization because of its high affinity
53 with micromachining technology. Because of these attractive features, many piezoelectric VEHs
54 have been developed using piezoelectric ceramic materials and organic polymer materials[9-13].
55
56 In ceramic VEHs, cantilever-type VEHs using high-performance piezoelectric material PZT (lead
57 titanate zirconate) are widely reported[14-18], and the VEHs often use resonance to obtain high-
58 efficiency power generation. In general, as the resonance frequency increases with the
59 miniaturization of the device size, it is difficult to achieve miniaturization and low-resonance
60 frequency simultaneously. Thus, although PZT has high-power-generation performance, it is
61 difficult to develop small-sized PZT-based devices that work in a low-frequency band without
62 special processing, such as nanoribbon assemblies[19-20], because PZT is a high-rigidity material.
63 Unfortunately, our surrounding vibrations such as household appliances, human motion,
64 buildings, and bridges are mainly at low frequencies, less than 30 Hz[21-23]. Therefore, VEHs with
65 its high-power-generation efficiency at low frequencies and small size are ideal for the low-
66 frequency applications.
67
68 The easiest way to lower the resonance frequency is to increase the mass weight of the tip of the
69 cantilever. As attaching a high-density material to the tip is all that is required, the fabrication is
70 simple and easy. However, the heavier the mass is, the more fragile the device becomes. Another
71 way of lowering the frequency is to lengthen the cantilever[24,25]. In the method, the distance from
72 the fixed end to the free end is extended by a two-dimensional meandered shape. The silicon
73 substrate is etched using a semiconductor manufacturing technique to fabricate a meandered
74 structure. Although the method is effective for lowering resonance frequency, the area of the
75 piezoelectric material decreases and, thus, the obtainable output power decreases. In addition,
76 there is a disadvantage that the vicinity of the fixed end is fragile. Regarding some polymer
77 devices, such as the low-frequency VEH, flexible piezoelectric polymer PVDF is often used. As
78 PVDF is usually coated by a spin-coating method and the film is thin, the resonance frequency
79 can be reduced because of the low rigidity[26,27]. Although the film thickness is controllable in the
80 range of sub-micron to several microns, the attainable output power is small because of the thin
81 thickness. Therefore, even if the frequency can be reduced, we cannot obtain sufficient power
82 generation, and so, practical application is difficult.
83
84 Here, we propose a bimorph-type piezoelectric cantilever (consisting of two layers of
85 piezoelectric layers and one layer of elastic layer) with two flexible piezoelectric polymer sheets,
86 which have already been subjected to stretching treatment for improvement of piezoelectric
87 characteristics. Furthermore, we adopt a flexible 3D mesh structure in the elastic layer of the
88 bimorph cantilever to reduce the resonance frequency and improve the power simultaneously.

89 We fabricate the 3D mesh structure by utilizing the backside inclined exposure method[28,29]
90 because it is possible to fabricate fine patterns with high precision in a short time. Although 3D
91 printing is also a candidate to fabricate 3D mesh structure, the throughput is low, and the 3D
92 printer is inferior to photolithography in machining accuracy[30,31]. Therefore, in this study, the
93 backside inclined exposure method is adopted as the method for micromachining the 3D mesh
94 structure.
95
96 **PROTOCOL:**
97
98 **1. Fabrication of the 3D mesh structure**
99
100 1.1. Cleaning of the glass substrate
101
102 1.1.1. Prepare 30 mm x 40 mm glass substrates.
103
104 1.1.2. Prepare the piranha solution by pouring 150 mL of sulfuric acid (concentration: 96%) into
105 the glass beaker. Then gently add 50 mL of hydrogen peroxide solution (concentration: 30%).
106 Ensure that the volume ratio of sulfuric acid:hydrogen peroxide water is 3:1.
107
108 NOTE: Wear protective glasses and clothing for safety while pouring the solutions.
109
110 1.1.3. Set a glass substrate in a Teflon jig for cleaning. Then immerse it in piranha solution for 1
111 min.
112
113 1.1.4. After a 1 min immersion in piranha solution, rinse the washed glass substrate 2-3 times
114 with pure water (overflow 2-3 times).
115
116 1.1.5. Remove the water drops on the glass substrate with air blow.
117
118 1.2. Patterning of the Cr mask pattern for backside exposure on a glass substrate
119
120 1.2.1. Set the glass substrate in a chamber of a RF (Radio-Frequency) magnetron sputtering
121 machine. Set the RF power to 250 W, the flow rate of Ar gas to 5.07×10^{-2} m^3/s, the chamber
122 pressure to 0.5 Pa, and the sputtering time to 11 min. Then form 100-200 nm of chromium film
123 on the glass substrate by RF magnetron sputtering.
124
125 NOTE: The thickness is controlled by the sputtering time, taking into account the sputtering rate
126 condition.
127
128 1.2.2. Set the substrate on a fixing stage in a spin-coater chamber. Drop a positive photoresist
129 S1813 on the chromium film and coat the 1-2 μm thin film by spin coating at 4,000 rpm for 30 s.
130
131 1.2.3. Bake the photoresist-coated substrate at 115 °C for 1 min on a hot plate to dry the resist.
132

133 1.2.4. Contact a photomask and a photoresist-coated substrate. Expose UV light vertically to the
134 photomask. Ensure that the exposure dose is 80 mJ/cm^2, and the wavelength is 405 nm. Use the
135 photomask shown in **Figure 1**.

137 1.2.5. Prepare two 500 mL beakers. Then pour 150 mL of TMAH (Tetramethylammonium
138 hydroxide: 2.38%, solvent: water) solution into one beaker and pour 150 mL of chromium etchant
139 (Ammonium Cerium(IV) Nitrate: 16%, nitric acid: 8%) into the other beaker.

141 1.2.6. Immerse the substrate in 150 mL of TMAH solution and develop the photoresist for 30 s to
142 1 min.

144 1.2.7. Rinse the substrate with pure water.

146 1.2.8. Immerse the substrate in the 150 mL of chromium etching solution and etch chromium for
147 about 1 to 2 min.

149 1.2.9. Rinse the substrate with pure water and remove water droplets with air blow.

151 1.2.10. Prepare piranha solution by pouring 150 mL of sulfuric acid (concentration: 96%) into the
152 glass beaker. Then gently add 50 mL of hydrogen peroxide solution (concentration: 30%). Ensure
153 that the volume ratio of sulfuric acid:hydrogen peroxide water is 3:1.

155 NOTE: Wear protective glasses and clothing for safety while pouring the solutions. Piranha
156 solution will lose activity after a while, so prepare each time.

158 1.2.11. Place a glass substrate on a Teflon jig for cleaning. Then, immerse it in the piranha solution
159 for 15-30 s to remove the photoresist.

161 1.3. Preparation for SU-8 coating

163 1.3.1. Set the substrate on the fixing stage in the spin-coater chamber. Drop approximately 1 mL
164 of acrylic resin solution (concentration: 10%, solvent: toluene) on the chromium pattern side of
165 the substrate to release a fabricated structure as a sacrificial layer. Then, form a thin film by spin
166 coating at 2,000 rpm for 30 s.

168 1.3.2. Bake at 100 °C for 10 min.

170 1.4. SU-8 spray coating

172 1.4.1. Launch the spray coater and pour acetone solution into the syringe for cleaning.

174 1.4.2. Clean and remove residues inside the spray nozzle by spraying acetone solution.

176 NOTE: If the cleaning is insufficient, it leads to clogging at the time of spraying. Repeat this step

177   twice to carefully clean.

178

179   1.4.3. Set the substrate on an attached plate in a spray coater.

180

181   1.4.4. Cover the substrate with an edge cover to prevent edge bead.

182

183   1.4.5. Pour the negative photoresist SU-8 3005 into the syringe.

184

185   1.4.6. Set the nozzle diameter to 5 mm, the nozzle movement speed to 120 mm/s, the
186   atomization pressure to 150 kPa, the fluid pressure to 60 kPa, the distance between the nozzle
187   and substrate to 40 mm, the pitch distance to 3 mm, and the interval time for each layer to 45 s.
188   Spray SU-8 multilayers on the substrate. Repeat the coating 10 times in the same way.

189

190   1.4.7. Leave the substrate to stand for 5 min after coating 10 times.

191

192   NOTE: During the standing time, the SU-8 film is uniformly flattened, and the air bubbles mixed
193   during spray coating are released.

194

195   1.4.8. Bake on a hot plate at 95 °C for 60 min.

196

197   1.4.9. Measure the thickness of 10 layers by micrometer. Then, calculate the thickness per layer.

198

199   1.4.10. Determine the remaining number of repetitions for spray coating from the calculated film
200   thickness per layer. Then spray the multilayer to form a thick film to achieve the target film
201   thickness. In this research, 40 layers are applied for a 200 μm thickness.

202

203   1.4.11. Let the substrate stand for 5 min after the multilayer spray coating.

204

205   1.4.12. Bake on a hot plate at 95 °C for 240 min.

206

207   1.4.13. Leave the SU-8 coated substrate on a hot plate for 60 min and then cool it slowly to room
208   temperature.

209

210   1.5. 3D mesh structure forming

211

212   1.5.1. Place the substrate on an angle adjustment table by flipping the substrate over (i.e., the
213   SU-8 film is facing down) as shown in **Figure 2**.

214

215   1.5.2. Fix the edge of the substrate with tape.

216

217   1.5.3. Tilt the angle of the adjustment table to 45°.

218

219   NOTE: 0° means the substrate is in the horizontal state. The angle at this time is determined by
220   Snell's law, calculated from the refractive index of the photoresist, the refractive index of air. By

221 irradiating at an incident angle of 45°, a mesh structure with a structure angle of 64° is fabricated.
222

223 1.5.4. Place the angle adjustment table under the UV light source.
224

225 1.5.5. Apply UV light vertically to the substrate at an exposure dose of 150 mJ/cm^2 and a
226 wavelength of 365 nm. After the exposure, return the angle of adjustment table to 0° and tilt it
227 to 45° in the opposite direction. Apply UV light vertically in the same way.
228

229 NOTE: Illustrations are shown in **Figure 3a,b**.
230

231 1.5.6. Place the substrate on a hot plate and set the temperature to 95 °C for PEB (post-exposure
232 bake). Bake the substrate for 8 min after the temperature becomes 95 °C.
233

234 1.5.7. Turn off the power of the hot plate. Wait until the temperature of the hot plate drops to
235 approximately 40 °C.
236

237 1.5.8. Pour 150 mL of SU-8 developer into a 500 mL glass beaker. Set the substrate in a Teflon jig
238 for developing.
239

240 1.5.9. Pour 150 mL of isopropanol (IPA) into another 500 mL glass beaker.
241

242 1.5.10. Develop for approximately 20 to 30 min. Ensure that if the developing time is not enough,
243 it leads to insufficient opening of the mesh voids.
244

245 1.5.11. Immerse the substrate with jig in IPA and rinse for 2 min.
246

247 NOTE: If the surface of SU-8 is apparently white and muddy, it indicates that development is
248 insufficient. In that case, repeat development and rinsing again. After complete development, a
249 mesh structure is formed, as shown in **Figure 3c**.
250

251 1.6. Structure release from the glass substrate
252

253 1.6.1. Pour 150 mL of toluene solution into a 500-mL glass beaker. Cover the beaker with
254 aluminum foil because toluene is easy to evaporate at room temperature.
255

256 1.6.2. Immerse the substrate in toluene solution for approximately 3-4 h. Ensure that the
257 sacrificial layer of acrylic resin is etched, and the SU-8 structure with the mesh structure is
258 released from the substrate, as shown in **Figure 3d**.
259

260 1.6.3. Blow air to the substrate and remove moisture. Store it in a desiccator until it is used in
261 step 4.3.
262

263 **2. Preparation of piezoelectric film**
264

265 2.1. Prepare a PVDF sheet. Also, prepare a cutter knife with a stainless steel blade and cutting
266 mat.
267
268 2.2. Cut out the PVDF sheet to the device shape with a 360 mm^2 sheet (10 mm x 30 mm for
269 cantilever and 6 mm x 10 mm for electrical connection), as shown in **Figure 3a**.
270
271 2.3. Place the cut PVDF films on a Petri dish with a cellulose wiper. Store them in a desiccator.
272
273 **3. Preparation of substrate for bonding mesh structure and piezoelectric film**
274
275 3.1. Pour 10 mL of the main agent of PDMS and 1 mL of curing agent into a centrifuge tube (i.e.,
276 the approximate volume ratio is 10:1).
277
278 3.2. Set the centrifuge tube in a planetary stirring and defoaming machine and mix both solutions
279 for 1 min.
280
281 3.3. Prepare two 30 mm x 40 mm glass substrates.
282
283 3.4. Set the glass substrate on a fixing stage in the spin-coater chamber. Drop PDMS solution onto
284 the glass substrate. Then, form the PDMS film by spin coating at 4,000 rpm, as shown in **Figure**
285 **3e**.
286
287 3.5. Bake on a hot plate at 100 °C for 60 min to dry the PDMS film.
288
289 3.6. Turn off the power of the hot plate. Wait until the temperature of the hot plate drops to
290 approximately 40 °C.
291
292 **4. Fabrication of bimorph vibration energy harvester**
293
294 4.1. Place the cut PVDF films one by one onto two different PDMS substrates, as shown in **Figure**
295 **3f**. Ensure that just by placing PVDF films on the surface of PDMS, they adhere to each other. If
296 wrinkles are seen on the PVDF films, extend them with a roller.
297
298 NOTE: These two PVDF films are called **PVDF flm1** and **PVDF flm2**, and the two PDMS substrates
299 are **PDMS sbs1** and **PDMS sbs2**, for the sake of clarity.
300
301 4.2. Drop SU-8 3005 onto the **PVDF flm1** placed on **PDMS sbs1**. Then, form the SU-8 thin film by
302 spin coating at 4,000 rpm as shown in **Figure 3g.**
303
304 NOTE: This SU-8 thin film becomes an adhesion layer between the mesh structure and the **PVDF**
305 **flm1**. The place where the SU-8 3005 was not dropped is used for wiring to acquire electric power.
306
307 4.3. Place the SU-8 mesh structure on the **PVDF flm1** and bond them as shown in **Figure 3h.**
308

319
320 **REPRESENTATIVE RESULTS:**
321 We fabricated a bimorph-type VEH composed of two layers of PVDF films and an intermediate
322 layer composed of an SU-8 mesh structure, as shown in **Figure 4**. The electrodes of the upper and
323 lower PVDF are connected in series to obtain output voltage. The optical image and the two SEM
324 images are elastic layers with a mesh structure. According to the images, the elastic layer
325 processed by the backside inclined exposure appears to have fine 3D mesh patterns without
326 development failure.
327
328 **Figure 5** shows the results of vibration tests. In the vibration tests, two VEHs—one with a meshed
329 core and the other with a solid-core structure—as the elastic layer are evaluated to verify the
330 validity of meshed-core-type VEH. The VEHs are set on a vibration shaker and excited with a
331 vibration acceleration of 1.96 m/s^2 (0.2 G). Both the meshed-core-type and solid-core-type VEHs
332 showed sinusoidal output synchronized with a sinusoidal input. The meshed-core-type VEH
333 exhibited a 42.6% higher output voltage than the solid-core type VEH. **Figure 5b** shows the
334 frequency response of the maximum output power. The meshed-core-type VEH exhibited a
335 resonance frequency of 18.7 Hz, which is 15.8% lower than the solid-core-type VEH, and an
336 output power of 24.6 µW, which is 68.5% higher than the solid-core-type VEH.
337
338 **FIGURE AND TABLE LEGENDS:**
339 **Figure 1: Photomask layout for photolithography to fabricate elastic layer with a 3D meshed-**
340 **core structure**. The photomask has two parts. One is the area for clamping, and the other
341 contains the line and space patterns for mesh-structure patterning.
342
343 **Figure 2: Set-up for inclined exposure.** UV light is exposed vertically to the inclined substrate
344 with a Cr pattern placed on angle adjustment table.
345
346 **Figure 3: Schematic of a proposed piezoelectric vibration energy harvester with a 3D meshed-**
347 **core structure and the fabrication process of the harvester.** The fabrication process can be
348 divided into 3 sections: (**a**)-(**d**) represent the fabrication process of the 3D mesh structure, (**e**)-(**g**)
349 represent the preparation of the PVDF film on a glass substrate, and (**h**)-(**j**) represent the bonding
350 process to form a bimorph cantilever. (These figures are published under gold Open Access,
351 Creative Commons license and have been modified from [21].)
352

Figure 4: (a) Photograph of the fabricated bimorph meshed-core vibration energy harvester, (b) cross-sectional optical image of the 3D meshed-core structure, (c) and (d) SEM images of SU-8 meshed-core elastic layer. (These figures are published under gold Open Access, Creative Commons license and have been modified from [21].)

Figure 5: (a) Sinusoidal output voltage of load resistance under each resonance condition (meshed-core 18.7 Hz, solid-core 22.2 Hz) and (b) Maximum output power as a function of vibration frequency under optimum load resistance (meshed-core 17 MΩ, solid-core 13 MΩ) and 0.2 G acceleration. (These figures are published under gold Open Access, Creative Commons license and have been modified from [21].)

DISCUSSION:

The successful fabrication of the 3D mesh structure and the proposed bimorph VEH described above is based on four critical and distinctive steps.

The first critical step is processing using backside inclined exposure. In principle, it is possible to fabricate a mesh structure by inclined exposure from the upper surface using the contact lithography technique. However, backside exposure presents a more accurate processing precision than contact lithography, and defects during development are less likely to occur[28,29]. This is because the gap between the photomask and the photoresist could arise due to the waviness of the photoresist surface. Hence, light diffraction occurs and processing precision is lowered because of the gap. Therefore, in this study, we fabricated a mesh structure using the backside inclined exposure method. In addition, the measured value of the structural angle of the fabricated mesh structure is about 65°, with just a 1% error as compared with the designed value of 64°. From the result, we conclude that it is appropriate to apply the backside inclined exposure method to fabricate the mesh structure.

The second critical step is the development process of SU-8. If a developing defect occurs, the mesh structure loses inherent flexibility. To develop the thick SU-8 film, typically 10-15 min is used. However, this developing time is insufficient for the development of a 3D mesh structure. The 3D mesh structure differs from the 2D pattern fabricated by photolithography because it has many internal voids inside the membrane. If the developing time is short, development does not progress to the interior of the mesh structure, causing patterning failure. That is why, it is necessary to apply a relatively long development time, 20-30 min[32]. If finer patterns are required, even longer developing time may be necessary. However, at that time, we have to consider the swelling caused by long development time[33].

Next, the method to exploit a PDMS-formed substrate in the bonding process of PVDF film and SU-8 mesh structure is unique. It makes spin coating possible and, as a result, PVDF and SU-8 can be easily adhered using a spin-coated SU-8 thin adhesive layer. PVDF and SU-8 can be bonded, even by using a commercially available instant glue. However, the adhesive material hardens after the adhesive is solidified. Moreover, it is difficult to form a thin film with the instant glue. If the thickness of the instant glue is larger, it will increase the rigidity of the entire device. An increase in rigidity leads to an increase in the resonance frequency (i.e., it prevents lowering the

397 resonance frequency, which is the main purpose of this study). On the other hand, using the SU-
398 8 thin film formed by spin coating as an adhesion layer does not greatly affect the increase in
399 rigidity because the formed SU-8 film is thin. In addition, as the mesh structure is made of SU-8,
400 it is possible to increase the adhesive strength by using the same material for the adhesion layer.
401 That is why the SU-8 adhesion has enough adhesive strength to bond an SU-8 mesh structure and
402 PVDF films. Furthermore, from the aspect of reproducibility of the device, it would be useful to
403 use the SU-8 thin film as an adhesion layer, as a constant film thickness can be realized by spin
404 coating film formation.
405
406 Fourth, the coating method of SU-8 is distinctive. We have selected a spray multilayer coating
407 method for the SU-8 thick film. Although it is possible to form a thick film by spin coating, large
408 surface waviness occurs, and it is difficult to coat the film uniformly[34]. On the other hand, using
409 the spray multi-coating method reduces the waviness and suppresses the error of film thickness
410 in the substrate[34]. Particularly, attention needs to be given to large waviness because when the
411 thickness of the 3D mesh structure becomes nonuniform, the vibration characteristics and
412 rigidity of the device is changed by the partially increased or decreased thickness.
413
414 In principle, as photolithography uses UV light, the fabricable shapes are limited. It is true that
415 we can fabricate complex structures such as a 3D mesh structure by using inclined exposure.
416 However, arbitrary shapes such as a three-dimensional structure with a curved shape in the film
417 thickness direction are difficult to form[35,36]. The 3D printing can produce arbitrary three-
418 dimensional shapes, and the design is flexible. However, the throughput of the fabrication is low,
419 and the processing precision and mass production are inferior to photolithography. Thus, it is not
420 suitable for fabricating structures with fine patterns in a short time. In addition, processing 3D
421 CAD data is necessary, and it takes time to create the 3D model. On the other hand, in the case
422 of photolithography, especially in the inclined exposure method, the CAD data necessary for the
423 photomask is two-dimensional, and the design is relatively easy. For example, the oriented design
424 for a 3D mesh structure is just the 2D line and space patterns, as shown in **Figure 3**. Considering
425 these facts, in this research, we exploited the 3D lithography technique to develop a flexible 3D
426 mesh structure.
427
428 In this study, we fabricated a flexible 3D mesh structure and applied it to the elastic layer of a
429 bimorph cantilever type VEH for the purpose of lowering resonance frequency and increasing
430 output power. Since the proposed method is useful in lowering resonance frequency, it will be
431 useful for vibration energy harvester targeted for low-frequency application such as wearable
432 devices, monitoring sensors for public buildings and bridge, house appliances, etc. Further
433 improvement of output power would be expected by combining the trapezoidal shape, triangle
434 shape, and thickness optimization which is previously proposed in other papers[37-39].
435

441
442 **DISCLOSURES:**
443 We have nothing to disclose.
444
445 **REFERENCES:**
446 1. Karim, F., Zeadally, S. Energy harvesting in wireless sensor networks : A comprehensive review.
447 *Renewable and Sustainable Energy Reviews*. **55**, 1041-1054 (2016).
448 2. Wei, C., Jing, X. A comprehensive review on vibration energy harvesting: Modelling and
449 realization. *Renewable and Sustainable Energy Reviews*. **74**, 1-18 (2017).
450 3. Priya, S. et al. A Review on Piezoelectric Energy Harvesting: Materials, Methods, and Circuits.
451 *Energy Harvesting and Systems*. **4** (1), 3–39 (2017).
452 4. Arroyo, E., Badel, A., Formosa, F., Wu, Y., Qiu, J. Comparison of electromagnetic and
453 piezoelectric vibration energy harvesters: Model and experiments. *Sensors and Actuators, A:*
454 *Physical*. **183**, 148-156 (2012).
455 5. Inoue, S. et al. A Fluidic Vibrational Energy Harvester for Implantable Medical Device
456 Applications. *IEEJ Transactions on Sensors and Micromachines*. **137** (6), 152-158 (2017).
457 6. Sano, C., Mitsuya, H., Ono, S., Miwa, K., Toshiyoshi, H., Fujita, H. Triboelectric energy harvesting
458 with surface-charge-fixed polymer based on ionic liquid. *Science and Technology of Advanced*
459 *Materials*. **19** (1), 317-323 (2018).
460 7. Tsutsumino, T., Suzuki, Y., Kasagi, N., Sakane, Y. Seismic Power Generator Using High-
461 Performance Polymer Electret. *Int. Conf. MEMS'06*. 98-101 (2006).
462 8. Arakawa, Y., Suzuki, Y., Kasagi, N. Micro Seismic Power Generator Using Electret Polymer Film.
463 *The Fourth International Workshop on Micro and Nanotechnology for Power Generation and*
464 *Energy Conversion Applications Power MEMS 2004*. 37-38 (2004).
465 9. Kim, S.G., Priya, S., Kanno, I. Piezoelectric MEMS for energy harvesting. *MRS Bulletin*. **37** (11),
466 1039-1050 (2012).
467 10. Rocha, J.G., Gonçalves, L.M., Rocha, P.F., Silva, M.P., Lanceros-Méndez, S. Energy harvesting
468 from piezoelectric materials fully integrated in footwear. *IEEE Transactions on Industrial*
469 *Electronics*. **57** (3), 813-819 (2010).
470 11. Chen, D., Chen, K., Brown, K., Hang, A., Zhang, J.X.J. Liquid-phase tuning of porous PVDF-TrFE
471 film on flexible substrate for energy harvesting. *Applied Physics Letters*. **110**, 153902 (2017).
472 12. Kim, H.S., Kim, J.H., Kim, J. A review of piezoelectric energy harvesting based on vibration.
473 *International Journal of Precision Engineering and Manufacturing*. **12** (6), 1129-1141, (2011).
474 13. Aktakka, E.E., Peterson, R.L., Najafi, K. Thinned-PZT on SOI process and design optimization
475 for piezoelectric inertial energy harvesting. *Transducers'11*. 1649-1652 (2011).
476 14. Xu, R. et al. Screen printed PZT/PZT thick film bimorph MEMS cantilever device for vibration
477 energy harvesting. *Sensors and Actuators, A: Physical*. **188**, 383-388 (2012).
478 15. Shen, D. et al. Micromachined PZT cantilever based on SOI structure for low frequency
479 vibration energy harvesting. *Sensors and Actuators, A: Physical*. **154** (1) 103-108, (2009).
480 16. Fang, H. Bin *et al.* Fabrication and performance of MEMS-based piezoelectric power
481 generator for vibration energy harvesting. *Microelectronics Journal*. **37** (11) 1280-1284, (2006).
482 17. Lefeuvre, E., Badel, A., Richard, C., Petit, L., Guyomar, D. A comparison between several
483 vibration-powered piezoelectric generators for standalone systems. *Sensors and Actuators, A:*
484 *Physical*. **126** (2), 405-416 (2006).

485 18. Ishida, K. et al. Insole pedometer with piezoelectric energy harvester and 2 v organic circuits.
486 *IEEE Journal of Solid-State Circuits*. **48** (1), 255-264 (2013).
487 19. Qi, Y., Kim, J., Nguyen, T.D., Lisko, B., Purohit, P.K., Mcalpine, M.C. Enhanced Piezoelectricity
488 and Stretchability in Energy Harvesting Devices Fabricated from Buckled PZT Ribbons. Nano
489 Letters. **11** (3), 1331-1336 (2011).
490 20. Dagdeviren, C. et al. Conformal piezoelectric systems for clinical and experimental
491 characterization of soft tissue biomechanics. Nature Materials. **14** (7), 728–736 (2015).
492 21. Tsukamoto, T., Umino, Y., Shiomi, S., Yamada, K., Suzuki, T. Bimorph piezoelectric vibration
493 energy harvester with flexible 3D meshed-core structure for low frequency vibration. *Science and*
494 *Technology of Advanced Material*. **19** (1) 660-668 (2018).
495 22. Bayrashev, A., Parker, A., Robbins, W.P., Ziaie, B. Low frequency wireless powering of
496 microsystems using piezoelectric-magnetostrictive laminate composites. *TRANSDUCERS 2003 -*
497 *12th International Conference on Solid-State Sensors, Actuators and Microsystems, Digest of*
498 *Technical Papers*. **2**, 1707-1710 (2003).
499 23. Yildirim, T., Ghayesh, M.H., Li, W., Alici, G. A review on performance enhancement techniques
500 for ambient vibration energy harvesters. *Renewable and Sustainable Energy Reviews*. **71**, 435-
501 449 (2017).
502 24. Karami, M.A., Inman, D.J. Electromechanical modeling of the low-frequency zigzag micro-
503 energy harvester. *Journal of Intelligent Material Systems and Structures*. **22** (3), 271-282, (2011).
504 25. Liu, H., Lee, C., Kobayashi, T., Tay, C.J., Quan, C. Piezoelectric MEMS-based wideband energy
505 harvesting systems using a frequency-up-conversion cantilever stopper. *Sensors and Actuators,*
506 *A: Physical*. **186**, 242-248 (2012).
507 26. Ramadan, K.S., Sameoto, D., Evoy, S. A review of piezoelectric polymers as functional
508 materials for electromechanical transducers. *Smart Materials and Structures*. **23** (3), 033001
509 (2014).
510 27. Sharma, T., Je, S.S., Gill, B., Zhang, J.X.J. Patterning piezoelectric thin film PVDF-TrFE based
511 pressure sensor for catheter application. *Sensors and Actuators, A: Physical*. **177**, 87-92 (2012).
512 28. Lee, J.B., Choi, K.H., Yoo, K. Innovative SU-8 lithography techniques and their applications.
513 *Micromachines*. **6** (1), 1-18 (2015).
514 29. Kim, K. et al. A tapered hollow metallic microneedle array using backside exposure of SU-8.
515 *Journal of Micromechanics and Microengineering*. **14** (4), 597-603 (2004).
516 30. Vaezi, M., Seitz, H., Yang, S. A review on 3D micro-additive manufacturing technologies.
517 *International Journal of Advanced Manufacturing Technology*. **67** (5-8), 1721-1754 (2013).
518 31. Gates, B.D., Xu, Q., Stewart, M., Ryan, D., Willson, C.G., Whitesides, G.M. New approaches to
519 nanofabrication: Molding, printing, and other techniques. *Chemical Reviews*. **105** (4), 1171-1196
520 (2005).
521 32. Zhang, J., Tan, K.L., Gong, H.Q. Characterization of the polymerization of SU-8 photoresist and
522 its applications in micro-electro-mechanical systems (MEMS). *Polymer Testing*. **20** (6), 693-701
523 (2001).
524 33. Chuang, Y.J., Tseng, F.G., Lin, W.K. Reduction of diffraction effect of UV exposure on SU-8
525 negative thick photoresist by air gap elimination. *Microsystem Technologies*. **8** (4-5), 308-313
526 (2002).
527 34. Akamatsu, M., Terao, K., Takao, H., Shimokawa, F., Oohira, F., Suzuki, T. Improvement of
528 coating uniformity for thick photoresist using a partial spray coat. *The 7th Annual IEEE*

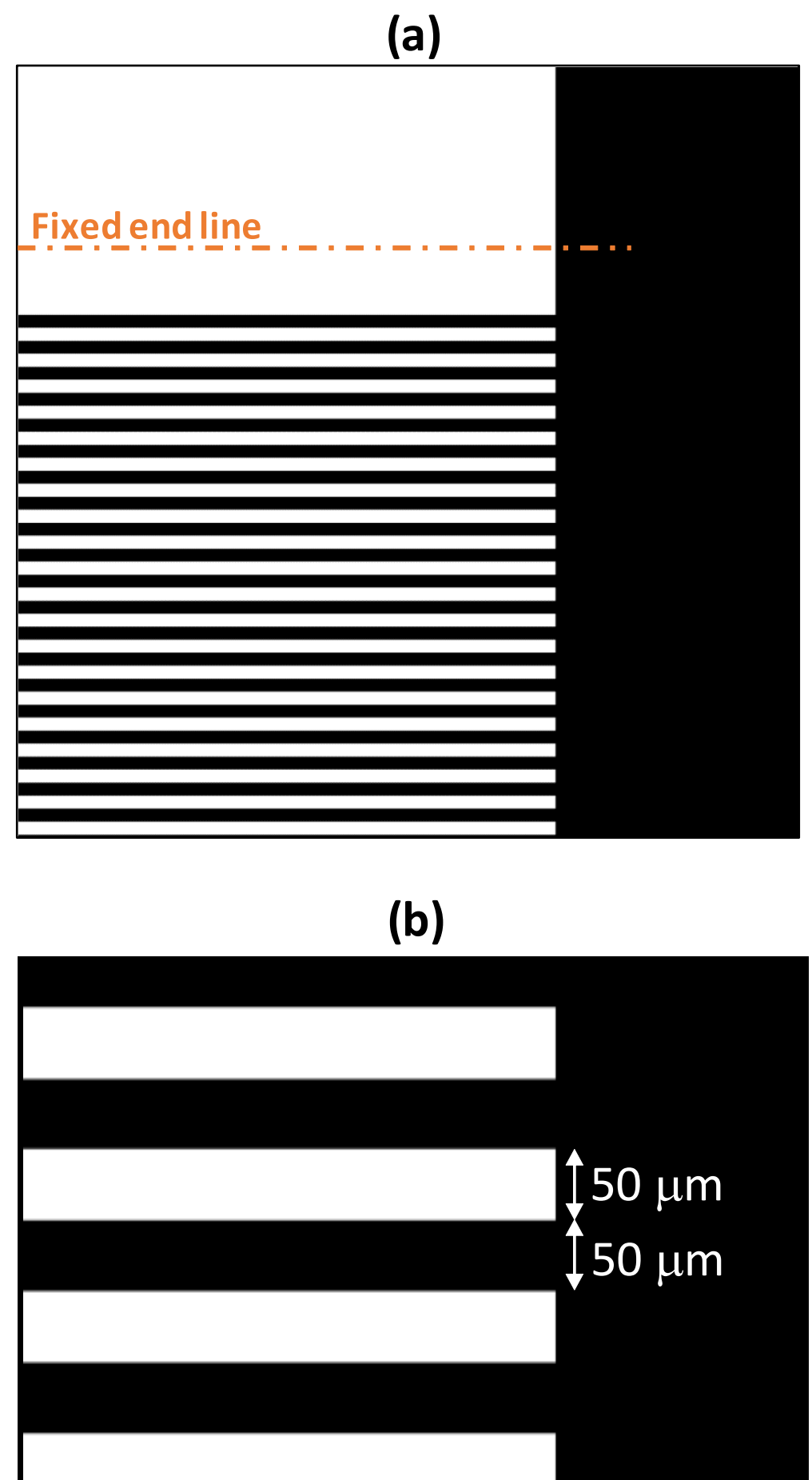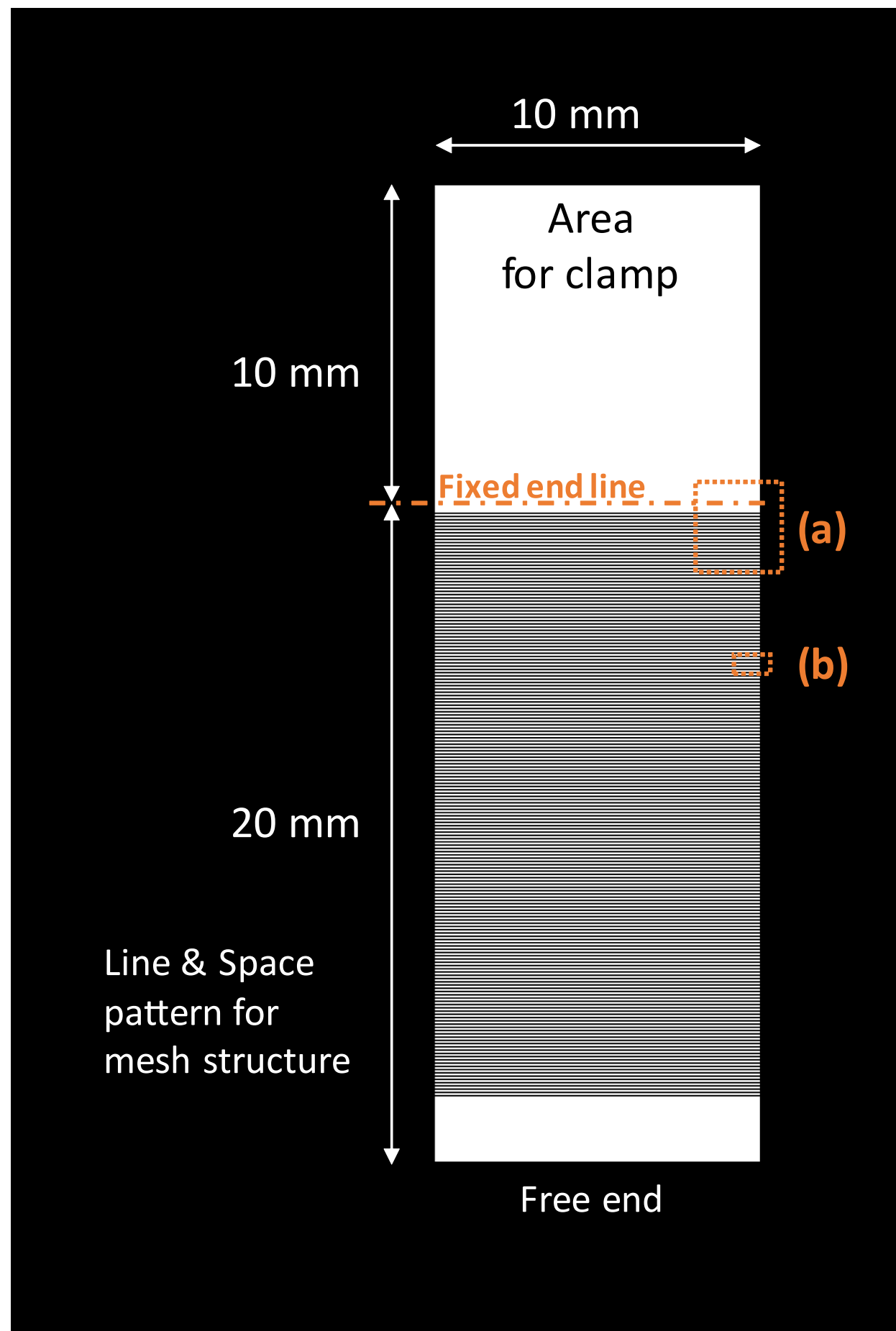*International Conference on Nano/Micro Engineered and Molecular Systems (IEEE-NEMS2012).* W3P-33 (2012).

35. Ingrole, A., Hao, A., Liang, R. Design and modeling of auxetic and hybrid honeycomb structures for in-plane property enhancement. *Materials and Design*. **117**, 72-83 (2017).
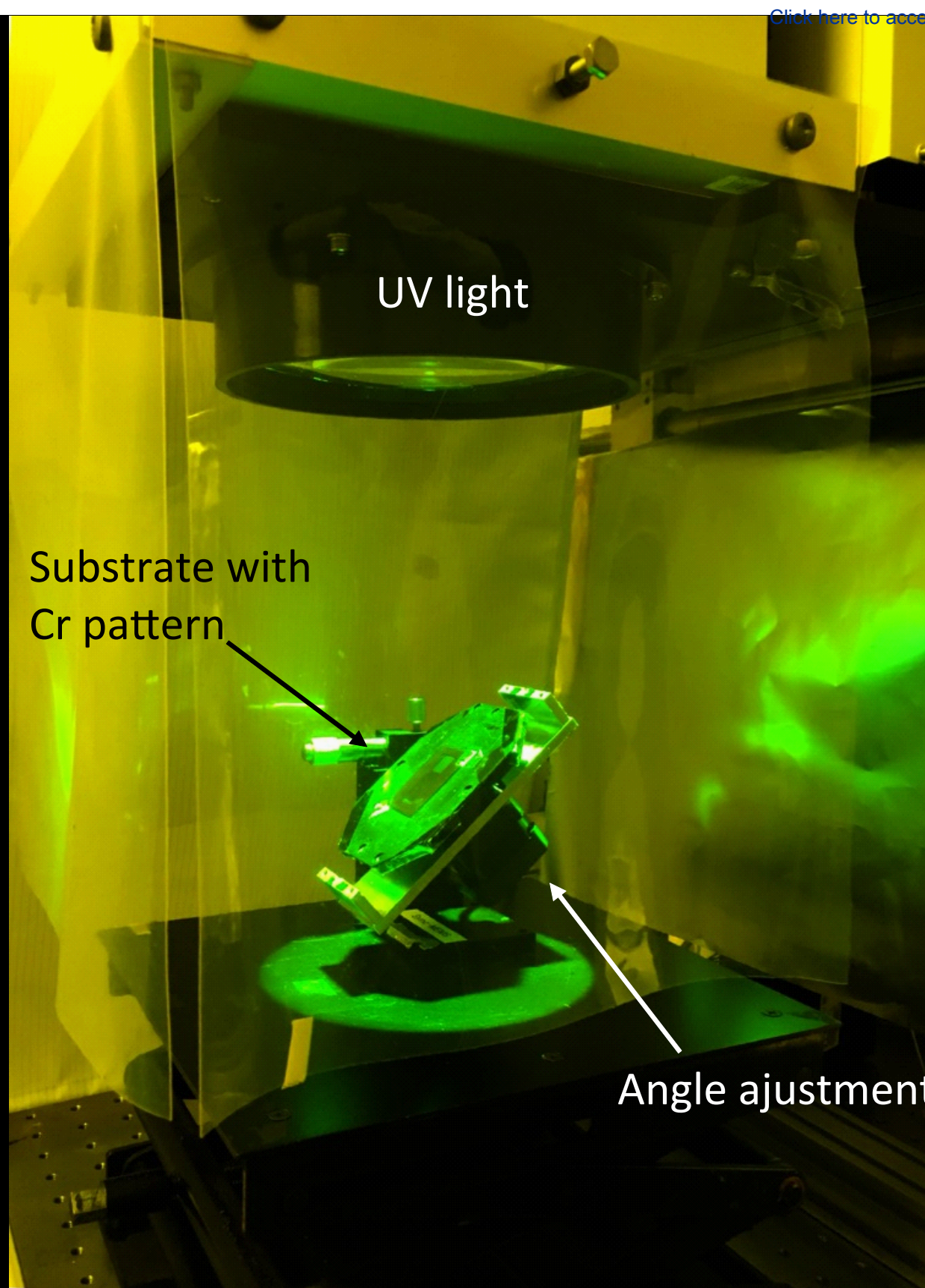
36. Schubert, C., Van Langeveld, M.C., Donoso, L.A. Innovations in 3D printing: A 3D overview from optics to organs. *British Journal of Ophthalmology*. **98** (2), 159-161 (2014).
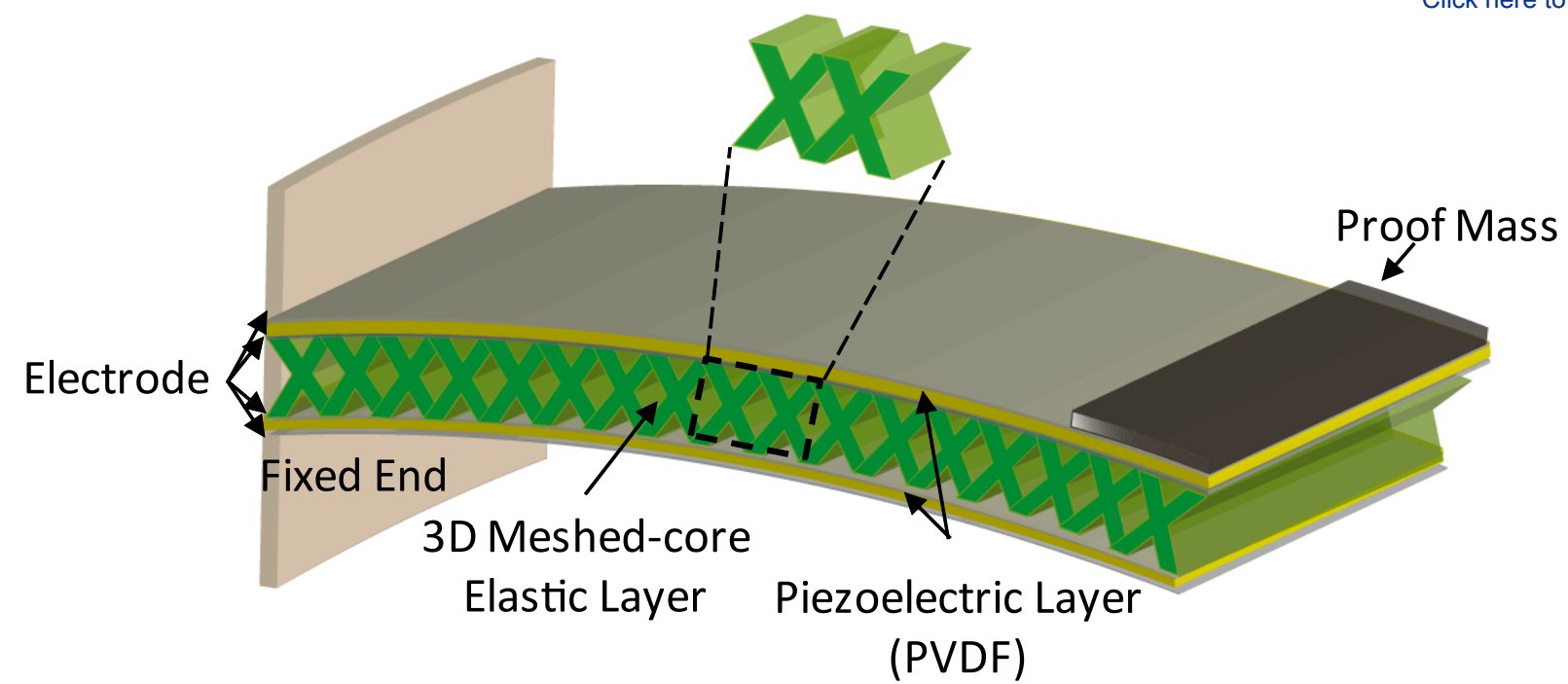
37. Muthalif, A.G.A., Nordin, N.H.D. Optimal piezoelectric beam shape for single and broadband vibration energy harvesting: Modeling, simulation and experimental results. *Mechanical Systems and Signal Processing*. **54**, 417-426 (2015).

38. Tai, W.C., Zuo, L. On optimization of energy harvesting from base-excited vibration. *Journal of Sound and Vibration*. **411**, 47-59 (2017).

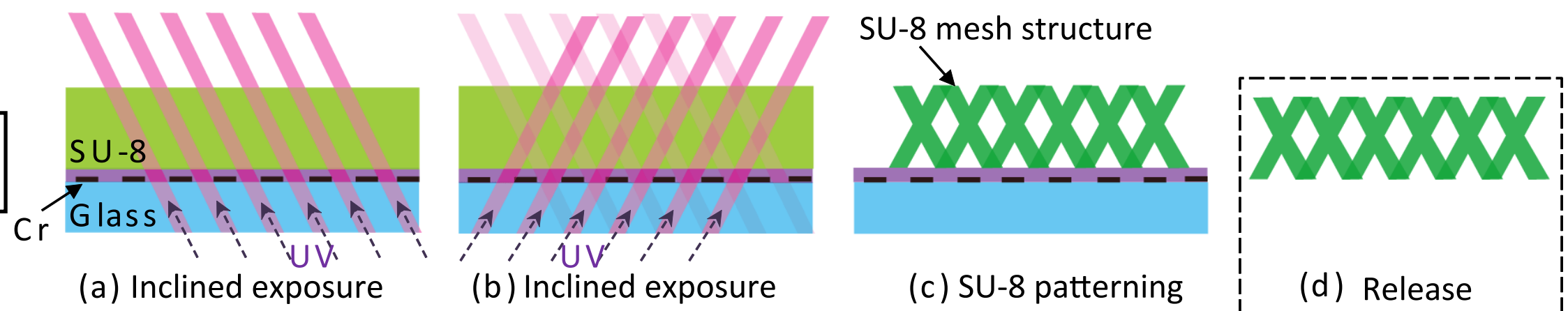39. Song, J., Zhao, G., Li, B., Wang, J. Design optimization of PVDF-based piezoelectric energy harvesters. *Heliyon*. **3** (9), e00377 (2017).
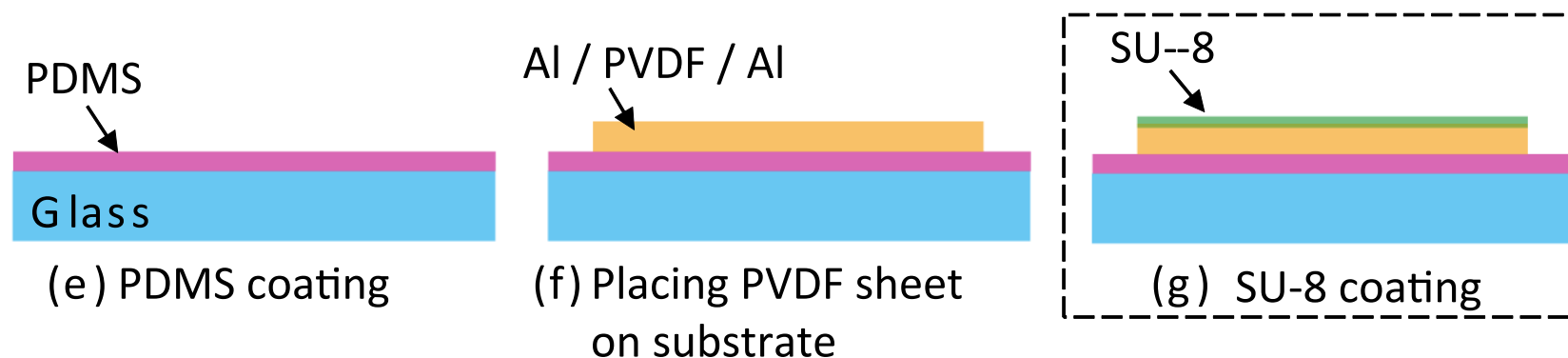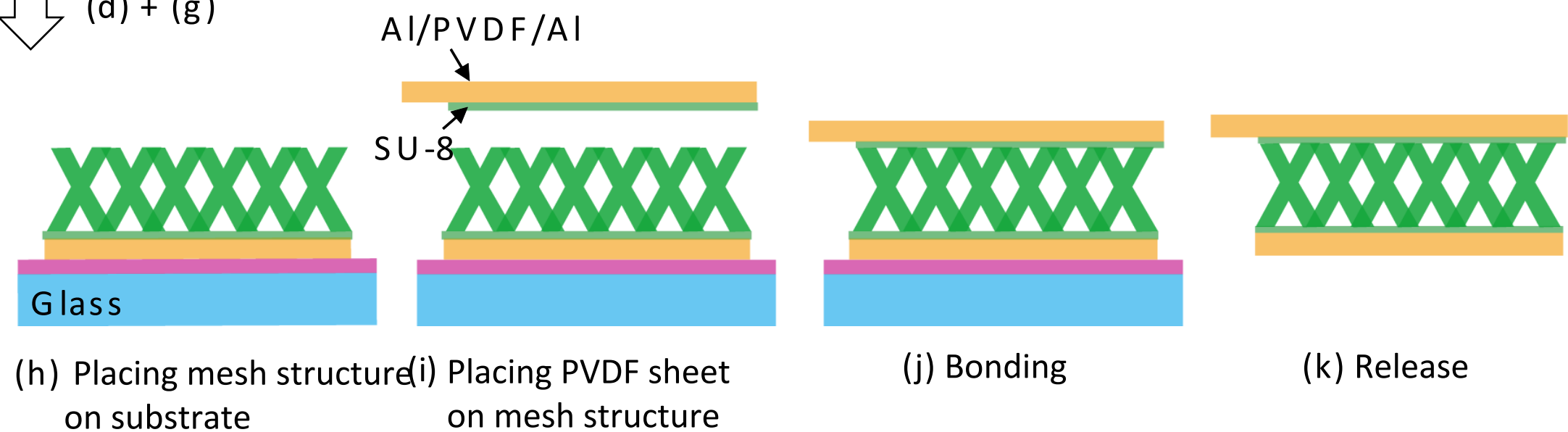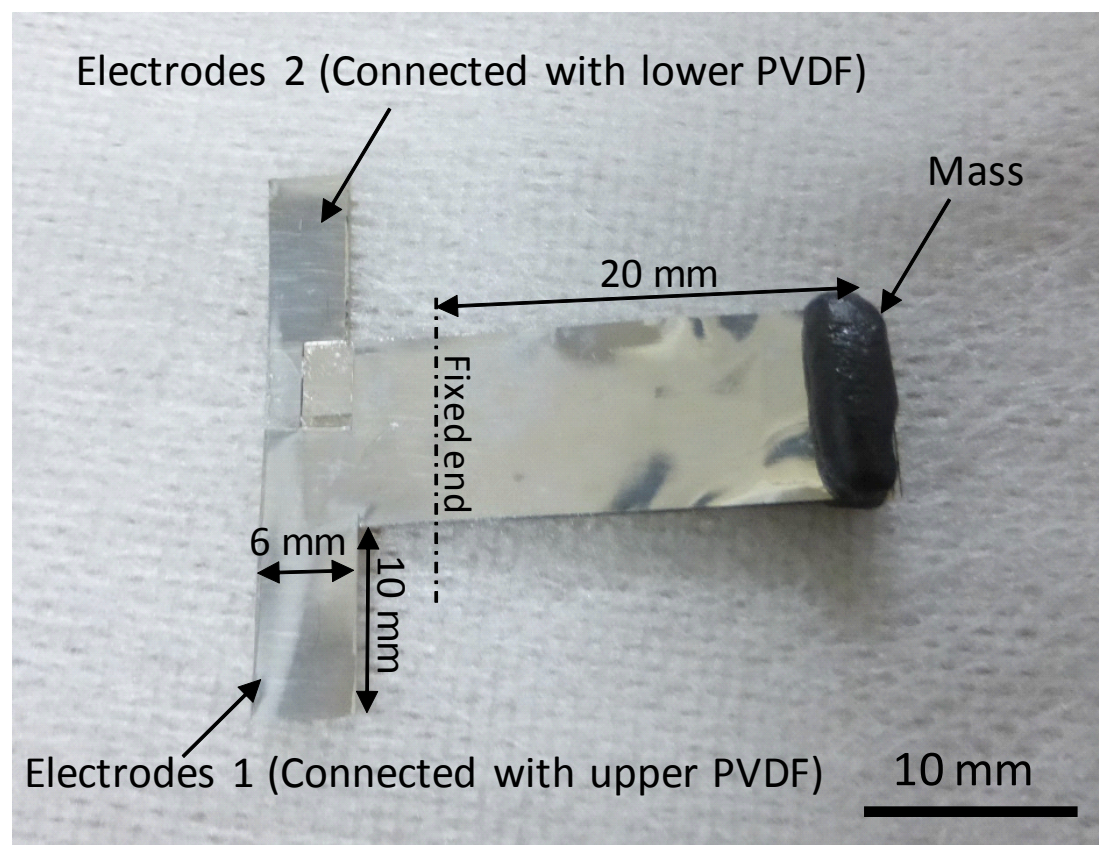
Figure 1

(a)

(b)

Figure 2

UV light

Substrate with
Cr pattern

Angle ajustment table

Figure 3

Click here to access/download;Figure;Figure_3_r.ai ±

Proof Mass

Electrode

Fixed End

3D Meshed-core
Elastic Layer

Piezoelectric Layer
(PVDF)

Preparation of mesh structure

SU-8 mesh structure

SU-8
Cr
Glass
UV

(a) Inclined exposure

UV

(b) Inclined exposure

(c) SU-8 patterning

(d) Release

Preparation of PVDF sheet

PDMS
Glass

(e) PDMS coating

Al / PVDF / Al

(f) Placing PVDF sheet
on substrate

SU--8

(g) SU-8 coating

(d) + (g)

Bonding process

Al/PVDF/Al

SU-8

Glass

(h) Placing mesh structure
on substrate

(i) Placing PVDF sheet
on mesh structure

(j) Bonding

(k) Release

Figure 4

Click here to access/download;Figure;Figure_4_r.ai ⬇
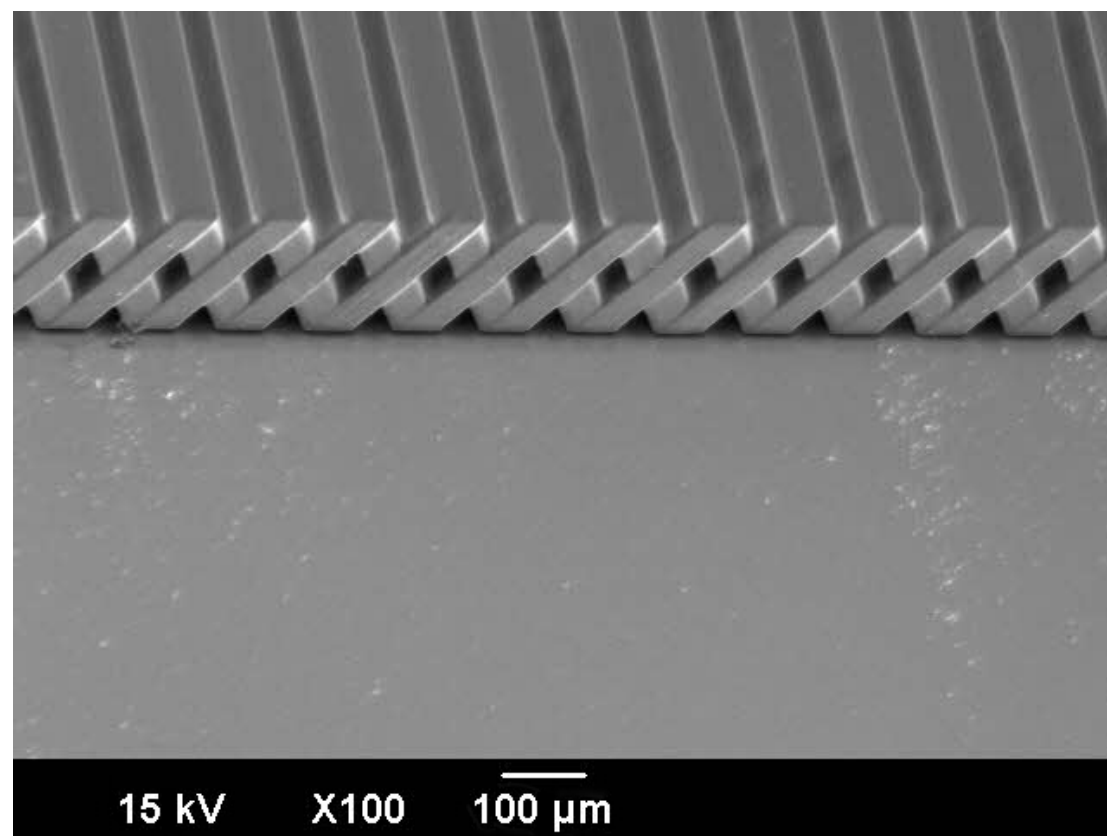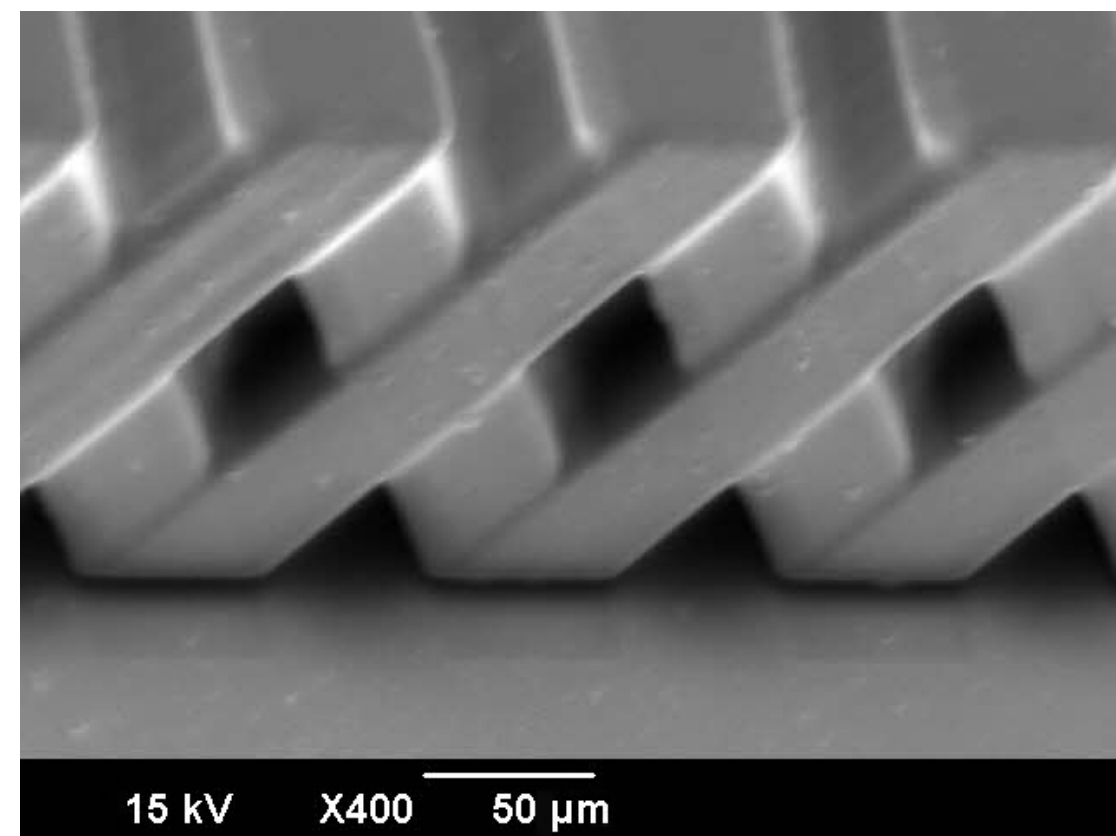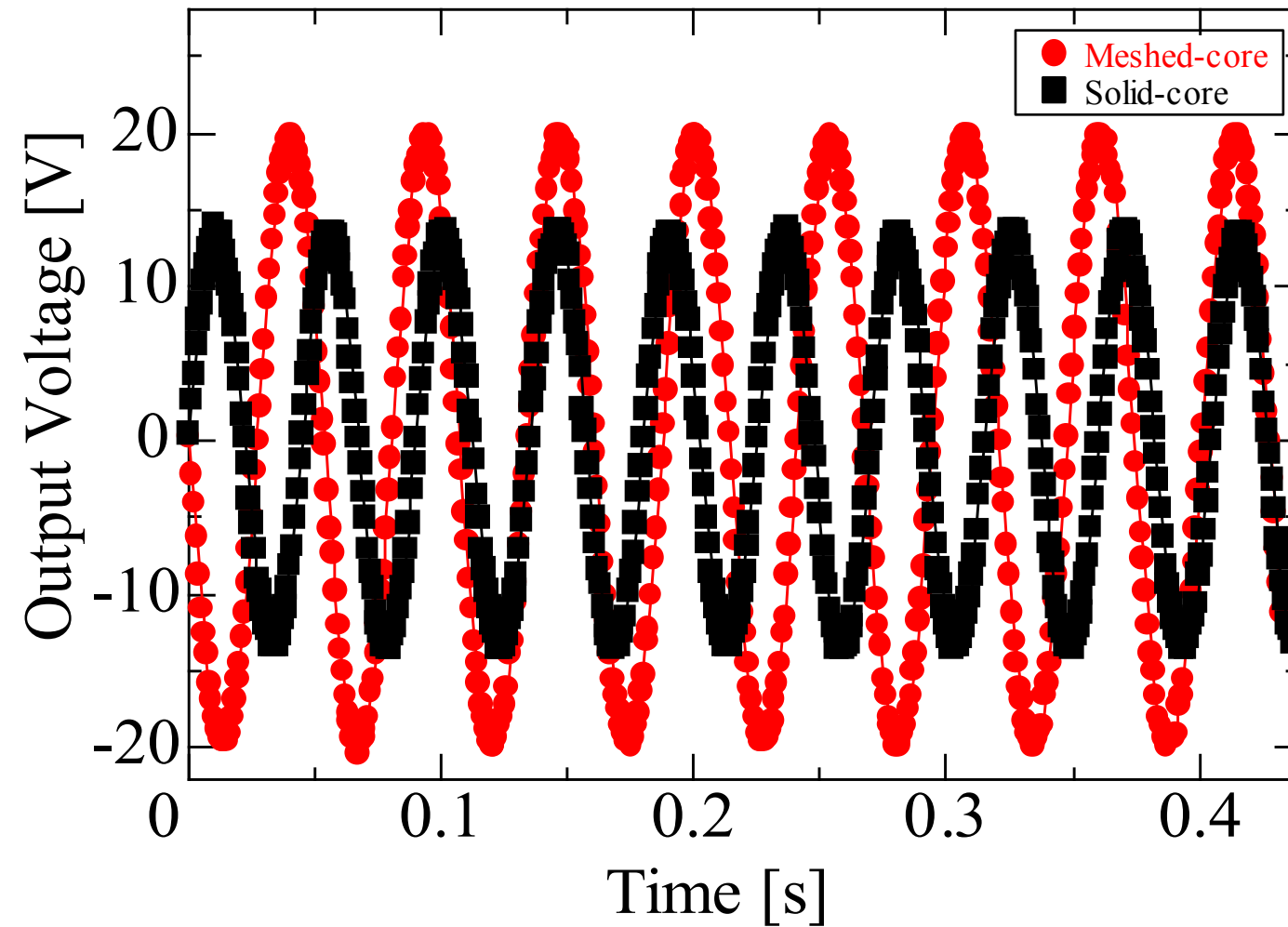

(a) Fabricated device.
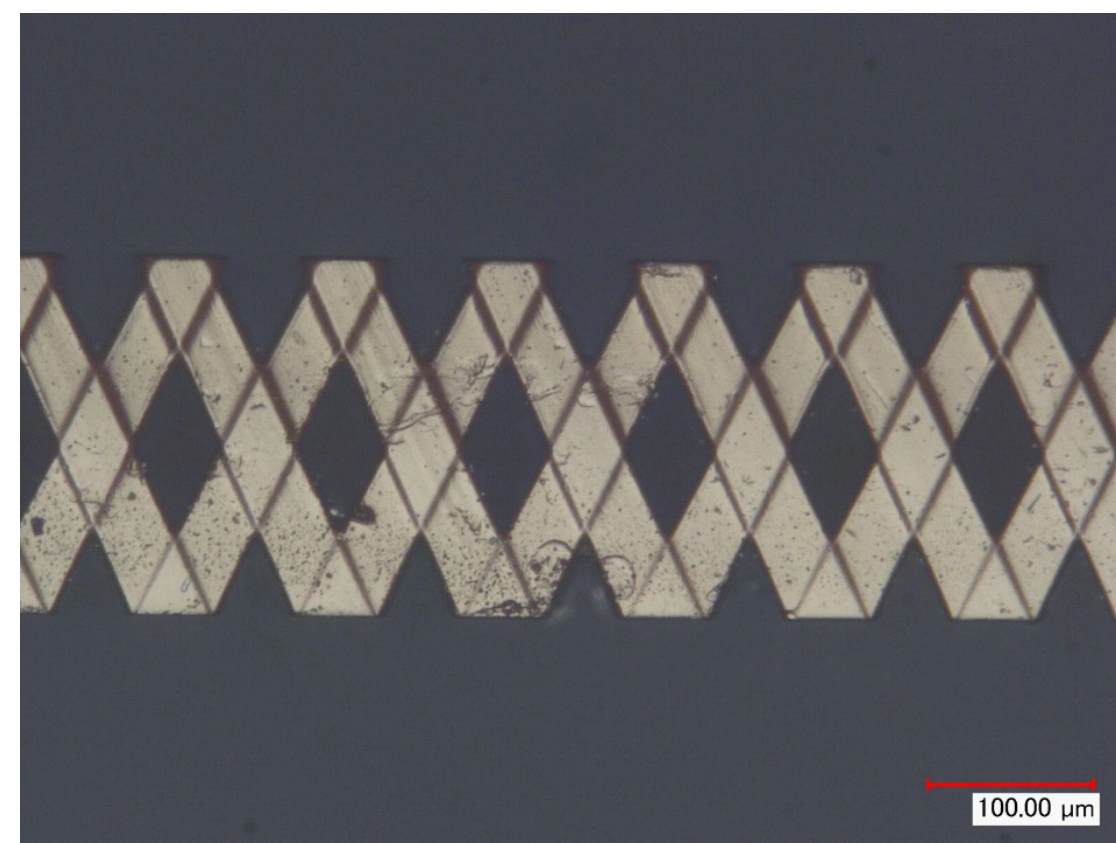
(b) Cross-sectional image.
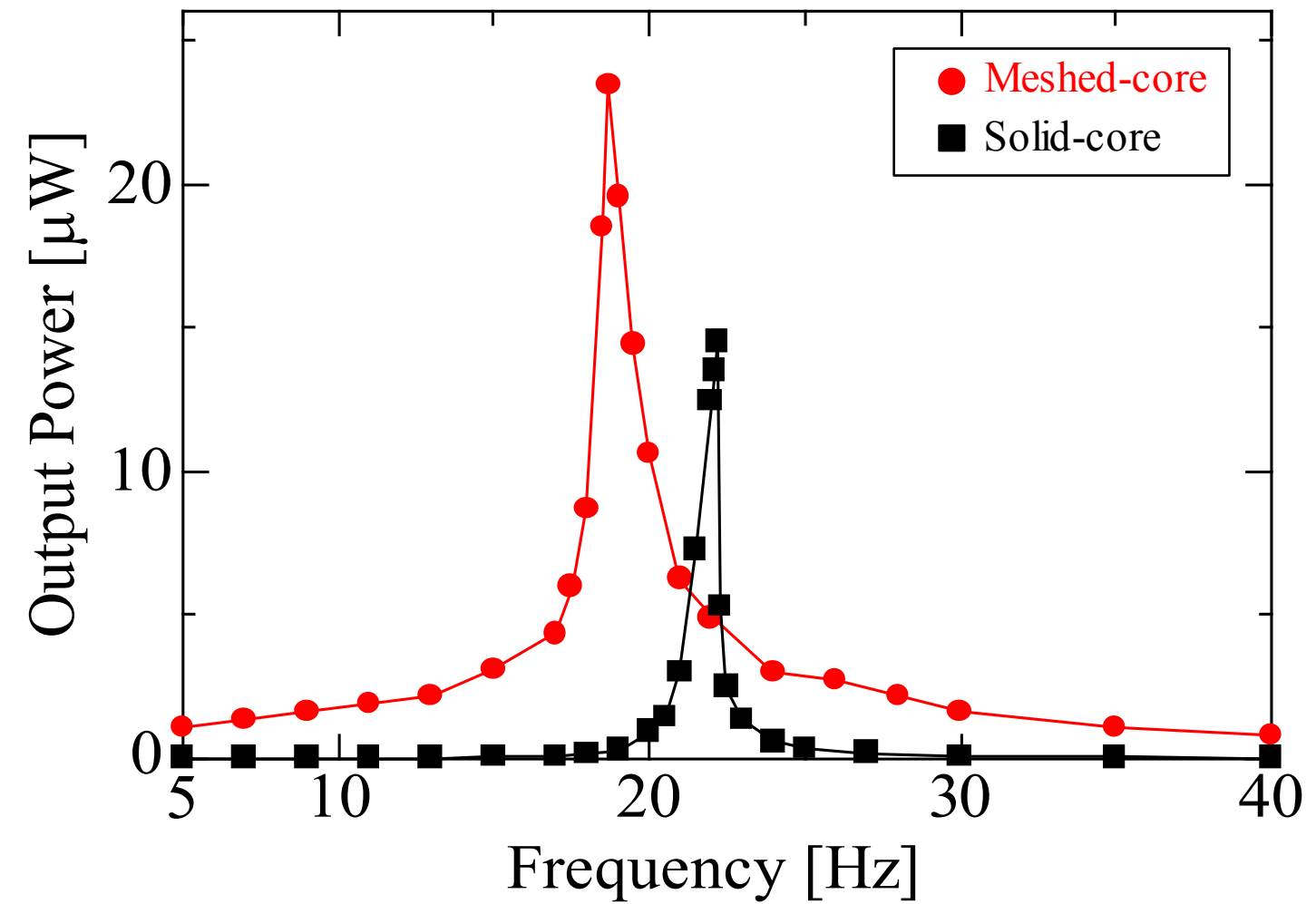
(c) SEM image (×100).

(d) SEM image (×400).

Figure 5

(a) Output voltage at resonance frequency.

(b) Frequency response.

Name of Material/ Equipment	Company	Catalog Number	Comments/Description
SU-8 3005	Nihon Kayaku		Negative photoresist
KF Piezo Film	Kureha		Piezoelectric PVDF film, 40 mm
Vibration Shaker	IMV CORPORATION	m030/MA1	Vibration Shaker
Spray coater	Nanometric Technology Inc.	DC110-EX	
Sputtering equipment	Canon Anelva Corporation	E-200S	
PDMS	Dow Corning Toray Co. Ltd	SILPOT 184 W/C	Dimethylpolysiloxane
Spin coater	MIKASA Co. Ltd	1H-DX2	
Digital oscilloscope	Teledyne LeCroy Japan Corporation	WaveRunner 44Xi-A	
SEM	JEOL Ltd.	JCM-5700LV	
Digital microscope	Keyence Corporation	VHX-1000	

jove
JOURNAL OF VISUALIZED EXPERIMENTS

1 Alewife Center #200
Cambridge, MA 02140
tel. 617.945.9051
www.jove.com

ARTICLE AND VIDEO LICENSE AGREEMENT

Title of Article: Fabrication method of polymer-based piezoelectric vibration energy harvester with flexible 3D mesh structure fabricated by 3D photolithography

Author(s): Takuya Tsukamoto, Yohei Umino, Kotaro Hashikura, Sachie Shiomi, Kou Yamada, Takaaki Suzuki

Item 1: The Author elects to have the Materials be made available (as described at http://www.jove.com/publish) via:

☑ Standard Access   ☐ Open Access

Item 2: Please select one of the following items:

☑ The Author is **NOT** a United States government employee.

☐ The Author is a United States government employee and the Materials were prepared in the course of his or her duties as a United States government employee.

☐ The Author is a United States government employee but the Materials were NOT prepared in the course of his or her duties as a United States government employee.

ARTICLE AND VIDEO LICENSE AGREEMENT

1.   **Defined Terms.** As used in this Article and Video License Agreement, the following terms shall have the following meanings: "**Agreement**" means this Article and Video License Agreement; "**Article**" means the article specified on the last page of this Agreement, including any associated materials such as texts, figures, tables, artwork, abstracts, or summaries contained therein; "**Author**" means the author who is a signatory to this Agreement; "**Collective Work**" means a work, such as a periodical issue, anthology or encyclopedia, in which the Materials in their entirety in unmodified form, along with a number of other contributions, constituting separate and independent works in themselves, are assembled into a collective whole; "**CRC License**" means the Creative Commons Attribution-Non Commercial-No Derivs 3.0 Unported Agreement, the terms and conditions of which can be found at: http://creativecommons.org/licenses/by-nc-nd/3.0/legalcode; "**Derivative Work**" means a work based upon the Materials or upon the Materials and other pre-existing works, such as a translation, musical arrangement, dramatization, fictionalization, motion picture version, sound recording, art reproduction, abridgment, condensation, or any other form in which the Materials may be recast, transformed, or adapted; "**Institution**" means the institution, listed on the last page of this Agreement, by which the Author was employed at the time of the creation of the Materials; "**JoVE**" means MyJove Corporation, a Massachusetts corporation and the publisher of The Journal of Visualized Experiments; "**Materials**" means the Article and / or the Video; "**Parties**" means the Author and JoVE; "**Video**" means any video(s) made by the Author, alone or in conjunction with any other parties, or by JoVE or its affiliates or agents, individually or in collaboration with the Author or any other parties, incorporating all or any portion of the Article, and in which the Author may or may not appear.

2.   **Background.** The Author, who is the author of the Article, in order to ensure the dissemination and protection of the Article, desires to have the JoVE publish the Article and create and transmit videos based on the Article. In furtherance of such goals, the Parties desire to memorialize in this Agreement the respective rights of each Party in and to the Article and the Video.

3.   **Grant of Rights in Article.** In consideration of JoVE agreeing to publish the Article, the Author hereby grants to JoVE, subject to **Sections 4** and **7** below, the exclusive, royalty-free, perpetual (for the full term of copyright in the Article, including any extensions thereto) license (a) to publish, reproduce, distribute, display and store the Article in all forms, formats and media whether now known or hereafter developed (including without limitation in print, digital and electronic form) throughout the world, (b) to translate the Article into other languages, create adaptations, summaries or extracts of the Article or other Derivative Works (including, without limitation, the Video) or Collective Works based on all or any portion of the Article and exercise all of the rights set forth in (a) above in such translations, adaptations, summaries, extracts, Derivative Works or Collective Works and (c) to license others to do any or all of the above. The foregoing rights may be exercised in all media and formats, whether now known or hereafter devised, and include the right to make such modifications as are technically necessary to exercise the rights in other media and formats. If the "Open Access" box has been checked in **Item 1** above, JoVE and the Author hereby grant to the public all such rights in the Article as provided in, but subject to all limitations and requirements set forth in, the CRC License.

612542.6   For questions, please contact us at submissions@jove.com or +1.617.945.9051.

jove
JOURNAL OF
VISUALIZED EXPERIMENTS

1 Alewife Center #200
Cambridge, MA 02140
tel. 617.945.9051
www.jove.com

4. **Retention of Rights in Article.** Notwithstanding the exclusive license granted to JoVE in **Section 3** above, the Author shall, with respect to the Article, retain the non-exclusive right to use all or part of the Article for the non-commercial purpose of giving lectures, presentations or teaching classes, and to post a copy of the Article on the Institution's website or the Author's personal website, in each case provided that a link to the Article on the JoVE website is provided and notice of JoVE's copyright in the Article is included. All non-copyright intellectual property rights in and to the Article, such as patent rights, shall remain with the Author.

5. **Grant of Rights in Video – Standard Access.** This **Section 5** applies if the "Standard Access" box has been checked in **Item 1** above or if no box has been checked in **Item 1** above. In consideration of JoVE agreeing to produce, display or otherwise assist with the Video, the Author hereby acknowledges and agrees that, Subject to **Section 7** below, JoVE is and shall be the sole and exclusive owner of all rights of any nature, including, without limitation, all copyrights, in and to the Video. To the extent that, by law, the Author is deemed, now or at any time in the future, to have any rights of any nature in or to the Video, the Author hereby disclaims all such rights and transfers all such rights to JoVE.

6. **Grant of Rights in Video – Open Access.** This **Section 6** applies only if the "Open Access" box has been checked in **Item 1** above. In consideration of JoVE agreeing to produce, display or otherwise assist with the Video, the Author hereby grants to JoVE, subject to **Section 7** below, the exclusive, royalty-free, perpetual (for the full term of copyright in the Article, including any extensions thereto) license (a) to publish, reproduce, distribute, display and store the Video in all forms, formats and media whether now known or hereafter developed (including without limitation in print, digital and electronic form) throughout the world, (b) to translate the Video into other languages, create adaptations, summaries or extracts of the Video or other Derivative Works or Collective Works based on all or any portion of the Video and exercise all of the rights set forth in (a) above in such translations, adaptations, summaries, extracts, Derivative Works or Collective Works and (c) to license others to do any or all of the above. The foregoing rights may be exercised in all media and formats, whether now known or hereafter devised, and include the right to make such modifications as are technically necessary to exercise the rights in other media and formats. For any Video to which this **Section 6** is applicable, JoVE and the Author hereby grant to the public all such rights in the Video as provided in, but subject to all limitations and requirements set forth in, the CRC License.

7. **Government Employees.** If the Author is a United States government employee and the Article was prepared in the course of his or her duties as a United States government employee, as indicated in **Item 2** above, and any of the licenses or grants granted by the Author hereunder exceed the scope of the 17 U.S.C. 403, then the rights granted hereunder shall be limited to the maximum

rights permitted under such statute. In such case, all provisions contained herein that are not in conflict with such statute shall remain in full force and effect, and all provisions contained herein that do so conflict shall be deemed to be amended so as to provide to JoVE the maximum rights permissible within such statute.

8. **Protection of the Work.** The Author(s) authorize JoVE to take steps in the Author(s) name and on their behalf if JoVE believes some third party could be infringing or might infringe the copyright of either the Author's Article and/or Video.

9. **Likeness, Privacy, Personality.** The Author hereby grants JoVE the right to use the Author's name, voice, likeness, picture, photograph, image, biography and performance in any way, commercial or otherwise, in connection with the Materials and the sale, promotion and distribution thereof. The Author hereby waives any and all rights he or she may have, relating to his or her appearance in the Video or otherwise relating to the Materials, under all applicable privacy, likeness, personality or similar laws.

10. **Author Warranties.** The Author represents and warrants that the Article is original, that it has not been published, that the copyright interest is owned by the Author (or, if more than one author is listed at the beginning of this Agreement, by such authors collectively) and has not been assigned, licensed, or otherwise transferred to any other party. The Author represents and warrants that the author(s) listed at the top of this Agreement are the only authors of the Materials. If more than one author is listed at the top of this Agreement and if any such author has not entered into a separate Article and Video License Agreement with JoVE relating to the Materials, the Author represents and warrants that the Author has been authorized by each of the other such authors to execute this Agreement on his or her behalf and to bind him or her with respect to the terms of this Agreement as if each of them had been a party hereto as an Author. The Author warrants that the use, reproduction, distribution, public or private performance or display, and/or modification of all or any portion of the Materials does not and will not violate, infringe and/or misappropriate the patent, trademark, intellectual property or other rights of any third party. The Author represents and warrants that it has and will continue to comply with all government, institutional and other regulations, including, without limitation all institutional, laboratory, hospital, ethical, human and animal treatment, privacy, and all other rules, regulations, laws, procedures or guidelines, applicable to the Materials, and that all research involving human and animal subjects has been approved by the Author's relevant institutional review board.

11. **JoVE Discretion.** If the Author requests the assistance of JoVE in producing the Video in the Author's facility, the Author shall ensure that the presence of JoVE employees, agents or independent contractors is in accordance with the relevant regulations of the Author's institution. If more than one author is listed at the beginning of this Agreement, JoVE may, in its sole

![jove logo] 1 Alewife Center #200
Cambridge, MA 02140
tel. 617.945.9051
www.jove.com

ARTICLE AND VIDEO LICENSE AGREEMENT

discretion, elect not take any action with respect to the Article until such time as it has received complete, executed Article and Video License Agreements from each such author. JoVE reserves the right, in its absolute and sole discretion and without giving any reason therefore, to accept or decline any work submitted to JoVE. JoVE and its employees, agents and independent contractors shall have full, unfettered access to the facilities of the Author or of the Author's institution as necessary to make the Video, whether actually published or not. JoVE has sole discretion as to the method of making and publishing the Materials, including, without limitation, to all decisions regarding editing, lighting, filming, timing of publication, if any, length, quality, content and the like.

12.   **Indemnification.** The Author agrees to indemnify JoVE and/or its successors and assigns from and against any and all claims, costs, and expenses, including attorney's fees, arising out of any breach of any warranty or other representations contained herein. The Author further agrees to indemnify and hold harmless JoVE from and against any and all claims, costs, and expenses, including attorney's fees, resulting from the breach by the Author of any representation or warranty contained herein or from allegations or instances of violation of intellectual property rights, damage to the Author's or the Author's institution's facilities, fraud, libel, defamation, research, equipment, experiments, property damage, personal injury, violations of institutional, laboratory, hospital, ethical, human and animal treatment, privacy or other rules, regulations, laws, procedures or guidelines, liabilities and other losses or damages related in any way to the submission of work to JoVE, making of videos by JoVE, or publication in JoVE or elsewhere by JoVE. The Author shall be responsible for, and shall hold JoVE harmless from, damages caused by lack of sterilization, lack of cleanliness or by contamination due to

the making of a video by JoVE its employees, agents or independent contractors. All sterilization, cleanliness or decontamination procedures shall be solely the responsibility of the Author and shall be undertaken at the Author's expense. All indemnifications provided herein shall include JoVE's attorney's fees and costs related to said losses or damages. Such indemnification and holding harmless shall include such losses or damages incurred by, or in connection with, acts or omissions of JoVE, its employees, agents or independent contractors.

13.   **Fees.** To cover the cost incurred for publication, JoVE must receive payment before production and publication of the Materials. Payment is due in 21 days of invoice. Should the Materials not be published due to an editorial or production decision, these funds will be returned to the Author. Withdrawal by the Author of any submitted Materials after final peer review approval will result in a US$1,200 fee to cover pre-production expenses incurred by JoVE. If payment is not received by the completion of filming, production and publication of the Materials will be suspended until payment is received.

14.   **Transfer, Governing Law.** This Agreement may be assigned by JoVE and shall inure to the benefits of any of JoVE's successors and assignees. This Agreement shall be governed and construed by the internal laws of the Commonwealth of Massachusetts without giving effect to any conflict of law provision thereunder. This Agreement may be executed in counterparts, each of which shall be deemed an original, but all of which together shall be deemed to me one and the same agreement. A signed copy of this Agreement delivered by facsimile, e-mail or other means of electronic transmission shall be deemed to have the same legal effect as delivery of an original signed copy of this Agreement.

A signed copy of this document must be sent with all new submissions. Only one Agreement is required per submission.

CORRESPONDING AUTHOR

Name: Takaaki Suzuki

Department: Division of Mechanical Science and Technology

Institution: Gunma University

Title: Professor

Signature: 鈴木孝明   Date: September 6, 2018

Please submit a **signed** and **dated** copy of this license by one of the following three methods:
1.   Upload an electronic version on the JoVE submission site
2.   Fax the document to +1.866.381.2236
3.   Mail the document to JoVE / Attn: JoVE Editorial / 1 Alewife Center #200 / Cambridge, MA 02140

We wish to express our appreciation to the reviewers for their insightful comments, which have helped us significantly improve the paper. The followings are our point-by-point responses:

< Editorial comments >

Comment 1.

Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues.

Response 1.

Thank you very much for the comment. We have reviewed to proofread the manuscript so that there are no spelling or grammar issues.

Comment 2.

Figure 4: Please provide a title for the whole figure in Figure Legend.

Response 2.

We have added a title for the whole figures in Figure 4.

Comment 3.

Figure 5: Please provide a title for the whole figure in Figure Legend.

Response 3.

We have added a title for the whole figures in Figure 5.

Comment 4.

Please do not highlight notes for filming.

Response 4.

We have fixed the corresponding sentences not to be the highlight.

Comment 5.

Step 1.2.1: Please add more details to your protocol steps. Please ensure you answer the "how" question, i.e., how is the step performed? Alternatively, add references to published material specifying how to perform the protocol action.

Response 5.

We have added more details and answered the "how" questions in the Step 1.2.1. (Lines: 121-124)

Comment 6.

1.2.5: What's the concentration of NMD-3? What's the solvent?

Response 6.

We have added the concentration and the solvent. (Line: 138)

Comment 7.

1.3.1: What's the concentration of acrylic resin?

Response 7.

We have added the concentration of acrylic resin. (Line: 165)

We wish to thank the reviewers again for the valuable comments.