Fabrication of piezoelectric vibration energy harvester using coatable PolyVinylidene DiFluoride and its characterisation

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A cantilever-type vibration energy harvester (VEH) made of PolyVinylidene DiFluoride (PVDF) was fabricated and characterised. PVDF is one of polymer piezoelectric material, which is more flexible than ceramic-based piezoelectric materials such as lead zirconate titanate. Since PVDF dissolves in organic solvent, it is coatable on substrate or parts, making it compatible with micro electro mechanical systems process. The fabrication process of VEH is as follows: a PVDF film (thickness: 30 μm) was coated on a phosphor bronze rectangular plate (length: 35 mm, width: 15 mm, thickness: 0.1 mm) by bar-coating method, followed by polarisation by corona discharge method. Aluminium top electrode (length: 30 mm, width: 10 mm, thickness: 0.3 μm) was deposited on the PVDF film by DC sputtering. One end of the plate was clamped by a fixture to form a cantilever, the length of which is 25 mm. Finally, a proof mass ($m = 0.2$ g) was attached to the free end of cantilever. Output power P at resonant frequency (= 55 Hz) was measured as a function of load resistance R, in which acceleration was set to 17 m/s^2. Maximum output of 4.3 μW was achieved at $R = 2.1$ MΩ, which is not inferior to those of reported VEHs using ceramic piezoelectric materials.

1. Introduction: In recent years, energy harvesting technologies have attracted much attention as an energy source for wireless sensor's battery [1, 2]. There are various energy sources for energy harvesting such as solar power, wind power, thermic energy, and vibration. Among these generating method, vibration energy harvester (VEH) is required under the environments where solar batteries are not usable, e.g. bridge, interior of a vehicle, place under elevated road, and building. There are three main types of generating method for VEH, that is, electromagnetic, electrostatic, and piezoelectric. Piezoelectric VEHs has high-electromechanical coupling factor and is suitable for size reduction because of its simple structure [3]. In many cases, the structure of piezoelectric VEH is like this: piezoelectric thin film is deposited on the surface of leaf spring made of metals or single-crystal silicon. A weight is set on the tip of spring [4–13]. They can generate electric power under low frequency vibration and low acceleration by adjusting the design of leaf spring to the vibration condition.

In previous study, piezoelectric VEHs were fabricated using ceramic piezoelectric material by Kanno *et al* [6–10]. They fabricated cantilever-type piezoelectric VEHs, which are composed of metal cantilever and lead zirconate titanate (PZT) film having highly piezoelectrisity on it. The output of 1–17 μW was reported in a vibration frequency range of hundreds hertz. While PZT have highly piezoelectrisity, it contains toxic lead, and it is a typical brittle material. Destruction of VEH devices under large vibration or shock has become a practical problem. These days, lead-based piezoelectric materials other than PZT and lead-free ones are being studied for VEH for the purpose of replacing PZT. Using these materials, micro-watt order power generation has been already realised [11, 12].

We focused on PolyVinylidene DiFluoride (PVDF), which is piezoelectric polymer material. PVDF can endure large deformation due to its large flexibility compared with ceramic PZT. PVDF can be dissolved in an organic solvent such as methyl ethyl ketone (MEK), forming PVDF solution. It is coatable on substrate or parts by several coating methods, making it compatible with micro electro mechanical systems (MEMS) to fabricate a miniaturised device. Toprak *et al.* fabricated miniaturised piezoelectric VEH composed of PVDF thin film coated on the cantilever made of single-crystal silicon [13]. However, its vibration frequency was 1,074 Hz and output was very small of pW order. There have not yet been reported any VEH, which uses coated PVDF film and is suitable for low frequency of 100 Hz or less.

In this study, we aimed to fabricate VEH using PVDF film coated on a leaf spring, which can generate power even at low frequency. At first, we fabricated PVDF thin film by bar coating method, followed by polarisation by corona discharge method. The mechanical and electrical properties of the fabricated piezo film, such as surface roughness, residual stress, Young's modulus, X-ray diffraction (XRD) pattern, piezoelectric constant, were characterised. Then we fabricated cantilever-type VEH using PVDF and evaluated its power generation ability.

2. Preparation of PVDF film by coating method

2.1. Formation of PVDF thin film: Spin coating method is typically employed in MEMS process. However, it was found that the thickness of PVDF film can be controlled only in the range of 5–10 μm by spin coating method. The spin-coated film of 5–10 μm in thickness was easily to be discharged in the polarisation process presumably due to cracks. It was necessary to obtain a thicker film to prevent cracks. As a method to obtain thick film, the bar coating method was employed.

Schematic illustration of bar-coating method is shown in Fig. 1. PVDF solution is dropped on the substrate. Bar coater is slid on spacers to expand the PVDF solution. Grooves are provided on the surface of bar coater for obtaining uniform film thickness.

PVDF solution was prepared by dissolving it in MEK. A bar coater (Model: 065-2-40μm, Allgood Corp.) was employed, of which diameter, length, groove depth are 9 mm, 320 mm, and 40 μm, respectively. After coating, the substrate was heated on a hot plate at 90°C for 10 min to evaporate MEK.

In this method, the film thickness could be controlled up to 50 μm by changing the height of spacer, on which the bar coater is moved while sliding. As a result of measurement, we found that thickness of PVDF film was linearly increased in proportion to the height of spacers (Fig. 2). However, the obtained surface undulation became larger in case the thickness is over 30 μm. Considering this situation, the thickness was decided to be set to 30 μm. Namely, the reason of deciding thickness is largely affected

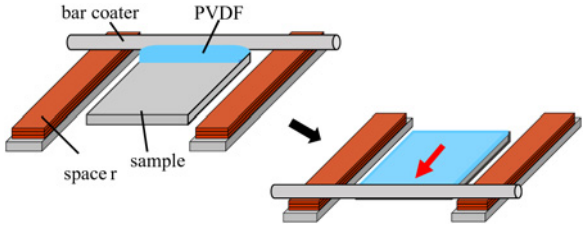

Fig. 1 *Schematic illustration of bar coating method*

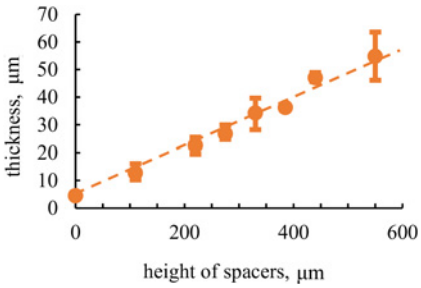

Fig. 2 *Relationship between obtained thickness of PVDF film and spacer height by bar coating method*

by the restriction of fabrication process. In the following of this paper, the films of 30 and 12 μm in thickness are prepared and used.

2.2. Characterisation of mechanical properties of PVDF film: Fig. 3 shows the surface morphology and roughness of obtained PVDF film measured by a stylus type step profiler (DekTakXT, Bruker Daltonics K.K). The surface waviness was within ± 1.5 μm along 25 mm line (see Fig. 3*a*). The surface roughness (*R*a) was estimated as 11 nm using the data shown in Fig. 3*b*.

Observing the film surface by scanning electron microscope, there were not any cracks or pinholes of several micro meter size.

Thin film stress measurement apparatus (FLX-2320-S, Toho technology Corp.) was used to measure the residual stress of PVDF film. The radii of curvature before and after the PVDF film deposition on a 4 inch silicon wafer were measured by this apparatus. Using the difference between these radii, the residual stress was estimated as 5 MPa, which is quite low stress compared with usual polymer films [14].

Young's modulus was measured by pulling PVDF film. Obtained stress-strain curve is shown in Fig. 4. It is reported that Young's modulus of bulk PVDF is 1–3 GPa, while that of thin PVDF film is approximately 0.5 GPa. Therefore, the result of 0.59 GPa, which is calculated by the initial inclination of curve, is thought to be reasonable value [15, 16].

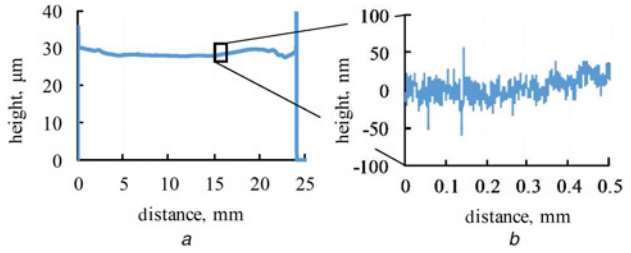

Fig. 3 *Surface morphology and roughness of obtained PVDF film measured by a stylus type step profiler*
a Surface morphology and
b Surface roughness of the PVDF film by bar coating method

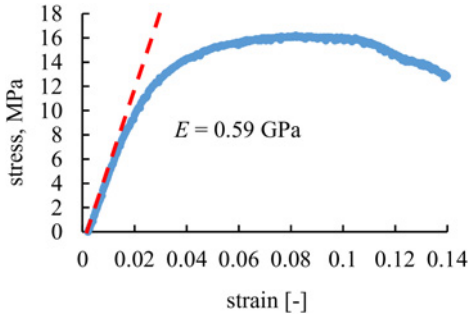

Fig. 4 *Stress-strain curve of PVDF film*

3. Polarisation of PVDF film

3.1. Corona polarisation method: Following coating a PVDF film, it was polarised by applying high voltage. In early stage of this study, top and bottom electrodes were deposited on PVDF film, then high DC voltage was applied between electrodes for polarising PVDF film; however, high electric field could not be applied, since discharge often occurred. In almost cases the discharge was creeping one, i.e., the route of discharge is on the substrate surface, followed by the route along the side wall to the electrical ground. It indicates that the film itself endures the applied high voltage, i.e., the discharge across the film thickness due to cracks or pinholes does not occur.

To overcome the discharge problem, we carried out charge injection into the surface of PVDF film using corona discharge. High electric field was applied to PVDF film before depositing top electrode. We call this method as corona polarisation. Schematic illustration of setup of corona polarisation is shown in Fig. 5. Procedures of corona polarisation are as follows: a needle electrode was located above the centre of PVDF film, followed by applying voltage of −10.0 kV for 10 min. Distance between the electrode and the test sample was set to 20 mm. In corona polarisation, discharge or any other problems did not occur.

3.2. Characterisation of piezoelectric property: XRD analysis result of the polarised PVDF film is shown in Fig. 6. The 20° peak indicates the dominant β-phase in PVDF film, which is beneficial for the piezoelectric effect. The measured XRD pattern matched well with that of a typical β-phase PVDF film discussed in previous studies [17, 18].

To confirm whether the polarised PVDF film has piezoelectricity or not, we measured piezoelectric constant d_{33} of it. The fabrication process of test sample to measure d_{33} is shown as follows: a PVDF film was prepared on a low resistance silicon substrate (size: 40 × 30 mm, resistivity: 0.2 Ωcm or less) by bar coating method, which is already explained in Section 2.1. One end of PVDF film was cut and a lead wire was connected to exposed low-resistance

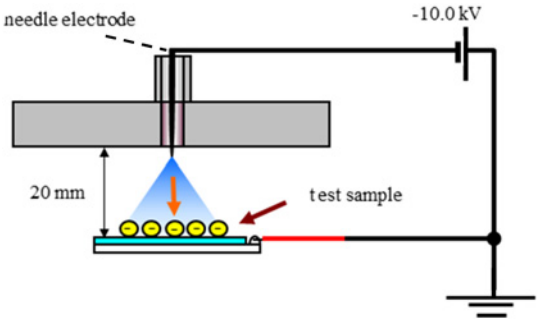

Fig. 5 *Schematic of corona polarisation*

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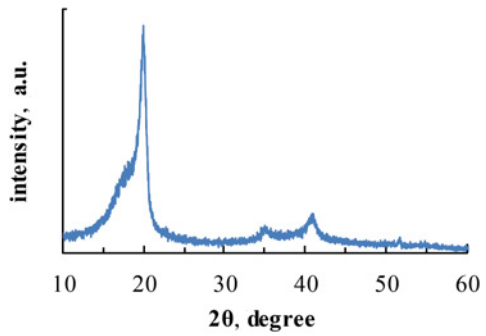

Fig. 6 *Result of XRD analysis of PVDF film*

silicon substrate by conductive glue. The effective area of PVDF film after cut was 30 mm square.

Piezoelectric constant d_{33} of PVDF film was measured by d_{33} meter (PIEZOTEST Corp.). The centre of PVDF film just below the needle electrode is the area where electric charge is easily implanted, and the amount of electric charge decreases as the area separates away from the centre. In other word, internal electric field occurring in PVDF film varies with locations and there is a possibility that PVDF film could not be polarised in places far from the electrode. We measured d_{33} of PVDF film at 17 points at intervals of 3 mm for investigating success range of one corona polarisation. The distribution of measurement points on PVDF film is shown in Fig. 7*a*. 17 points ((1)–(9) and A-H) shown in Fig. 7*a* were measuring points. Table 1 shows measurement result of d_{33}. The maximum value of d_{33} was −11.2 pC/N (see Table 1). Fig. 7*b* shows success or failure of corona polarisation at each point. When the value of d_{33} was greater than or equals to 8 pC/N, we considered polarisation was successful, otherwise we considered polarisation was failed. Success range of one corona polarisation was within the circle, of which diameter was

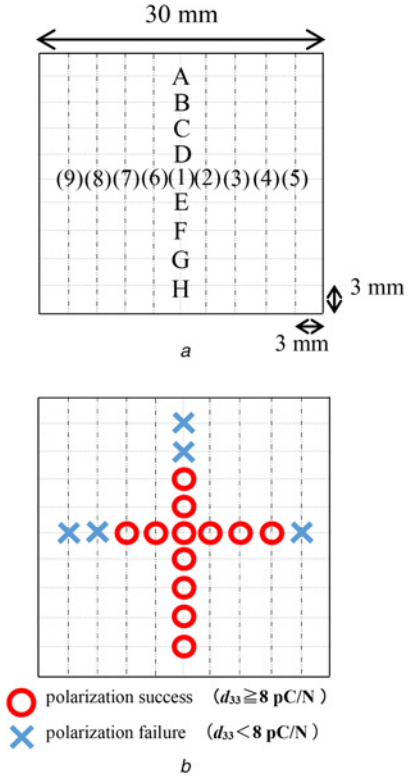

Fig. 7 *Distribution of measurement points on PVDF film*
a Measurement points of piezoelectric constant d_{33}
b Result of success or failure of corona polarisation

Table 1 Results of piezoelectric constant d_{33} of the PVDF film after corona polarization

d 33 [pC/N]	*d* 33 [pC/N]
(1) −9.93	A −5.59
(2) −8.86	B −7.72
(3) −10.7	C −8.01
(4) −9.81	D −9.46
(5) −6.89	E −10.6
(6) −10.0	F −11.2
(7) −8.85	G −11.1
(8) −3.40	H −8.02
(9) −3.43	

approximately 15 mm. In the outside area, sufficient piezoelectricity was not achieved.

The resistively of low resistance silicon is higher than typical metals and it may affect the piezoelectricity of PVDF films. We verified the effects of material difference in bottom electrode on piezoelectricity. The d_{33} of PVDF film of 30 and 12 μm in thickness coated on phosphor bronze ($2–6 \times 10^{-6}$ Ω cm [19]) were −8.44 and −10.13 pC/N (see Section 4.1 below), while that was approximately −10 pC/N (distributing among sampling points) on a low resistance single crystal Si (0.2 Ω cm). Considering these results, although the data is limited, the material of substrate, e.g. the resistivity of substrate, does not affect so much on the piezoelectricity of PVDF film.

4. Fabrication of cantilever-type VEHs

4.1. Preparation of PVDF film for VEHs: PVDF film was prepared on a phosphor bronze rectangular plate (length; 35 mm, width; 15 mm, thickness; 0.1 mm) by bar coating method. Phosphor bronze was adopted because of its good spring property. We fabricated two VEHs composed of different PVDF thicknesses; one is 30 μm (called sample 1), another is 12 μm (called sample 2). Dimensions of these samples are shown in Table 2.

After preparing PVDF film, lead wire was connected to the phosphor bronze plate and corona polarisation was applied. The conditions of corona polarisation were same as explained in Section 3.1. To surely polarise the whole area of PVDF film, corona discharge was carried out at six spots at intervals of 5 mm, as shown in Fig. 8.

Table 2 Dimensions of cantilever VEH devices

PVDF thickness sample1	30 μm
PVDF thickness sample2	12 μm
Phosphor bronze thickness	100 μm
Cantilever length	25 mm
Cantilever width	15 mm
Electrode length	22 mm
Electrode width	10 mm

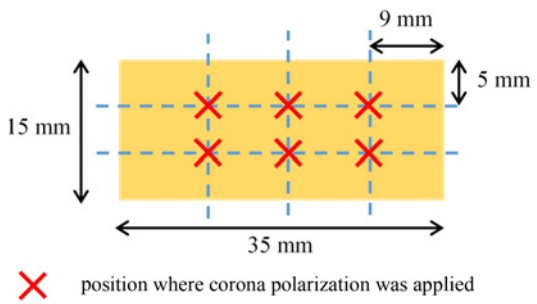

Fig. 8 *Schematic of points where corona polarisation was applied*

After polarisation, we measured d_{33} of these PVDF films. Average measurement values of d_{33} of polarised PVDF films were −8.44 pC/N (sample 1) and –10.13 pC/N (sample 2), respectively. For comparison, d_{33} before polarisation was −0.87 pC/N. These values (−8.44 and –10.13 pC/N) are not inferior to the typically reported ones ranging from −10 to −20 pC/N [20]. The difference of d_{33} between −8.44 and −10.13 pC/N may be affected by the difference of thickness to some extent. However, the difference would be larger only taking account of the difference of electric fields due to thickness. It is difficult to identify the reason at present.

In this study, PVDF film works in d_{31} mode. There are reports that d_{31} of PVDF film is ∼70% of its d_{33} [21, 22]. Considering that d_{33} of our PVDF film is ∼10 and it is same order as other reported values, d_{31} is expected to be not so bad.

Aluminium top electrode (length; 30 mm, width; 10 mm, and thickness; 0.3 μm) was deposited on PVDF film by DC sputtering. We measured the capacitance of fabricated VEHs by LCR meter (E4980A, Agilent Technology corp.). The capacitance and loss tangent were measured under the frequency from 1 kHz to 2 MHz. Measurement results are shown in Fig. 9. The capacitances of VEHs were 1.6 nF (sample 1) and 1.8 nF (sample 2) at 1 kHz, respectively. The dielectric constants of those calculated from the capacitance were ∼16 and 10, respectively. These dielectric constants are in the same order as the reported values such as 13 at 1 kHz [23]. Loss tangent were 0.013 (sample 1) and 0.021 (sample 2) at 1 kHz, respectively. These values are equivalent to the reported PVDF properties [24].

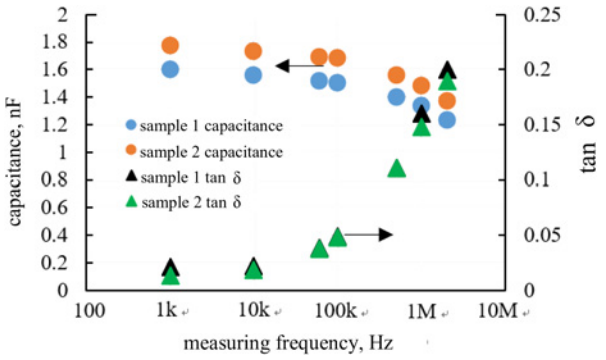

Fig. 9 *Capacitance and loss tangent of the VEH device*

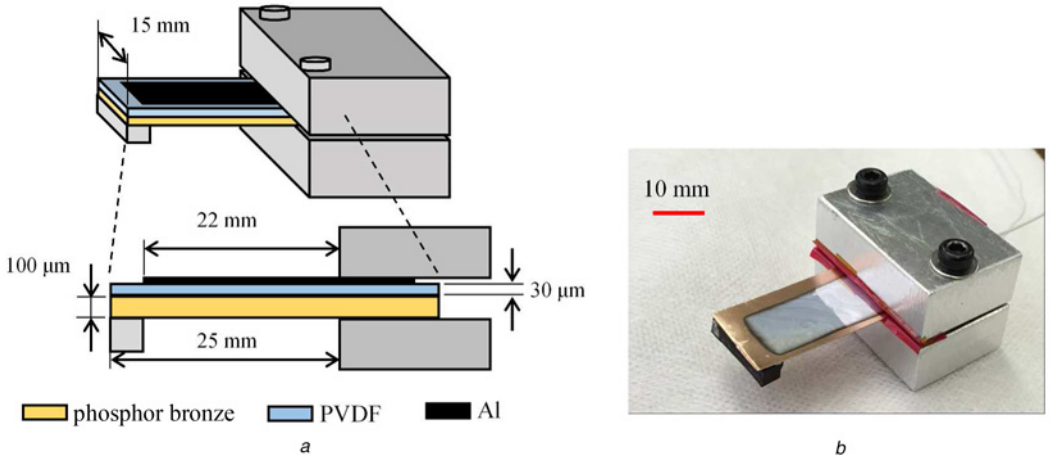

Fig. 10 *Schematic illustration and photograph of fabricated cantilever VEH*
a Schematic illustration and
b Photograph of the VEH using PVDF thin film on phosphor bronze cantilever

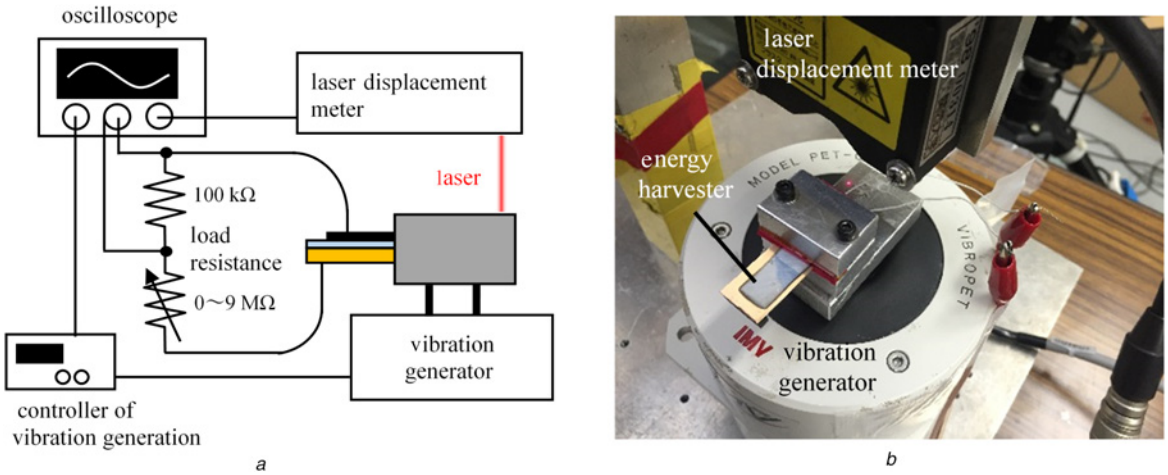

Fig. 11 *Measurement setup of vibration test*
a Schematic illustration and
b Photograph of experimental setup for verifying power generation for free vibration

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4.2. Vibration test and power generating result: one end of the plate was clamped by a fixture to form a cantilever. A proof mass ($m = 0.2$ g) was attached to the other end. Effective length of clamped antilever was 25 mm. Fig. 10 shows a schematic illustration and photograph of fabricated cantilever VEH.

Fig. 11 shows measurement setup of vibration test. Fabricated VEH was mounted on a vibration generator (PET-05, IMV Corp.). Lead lines between top and bottom electrodes were connected to the external load resistance R, which is composed of a fixed resistance (100 kΩ) and a variable resistance (from 0 to 9 MΩ) in series. A laser displacement meter was used for determining the acceleration. Output voltage $V(t)$ generated at the load resistance was measured by an oscilloscope. Output power P was calculated by

$$P = \frac{\int p(t)\mathrm{d}t}{T}, \tag{1}$$

where $p(t) = V(t)^2/R$, T is period. By changing R from 0.1 to 9.1 MΩ at intervals of 1 MΩ, the optimal value was searched.

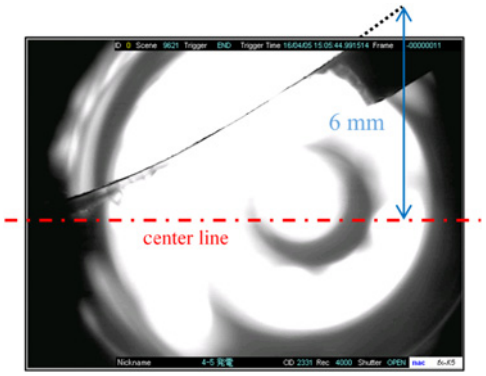

Fig. 12 *Image of the vibration of the VEH taken by high-speed camera*

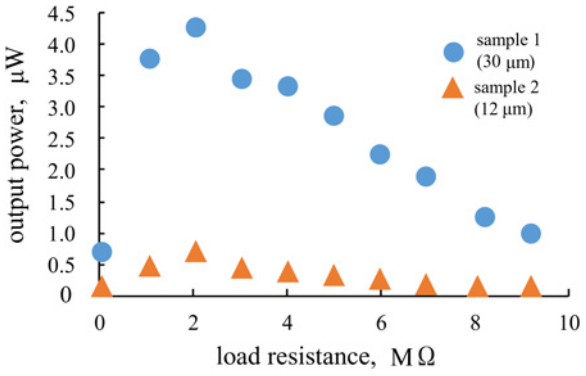

Fig. 13 *Output power vs. load resistance for free oscillation at 17 m/s², 55 Hz*

Acceleration and frequency of vibration were set to 17 m/s² and 55 Hz (it is resonant frequency of the cantilever), respectively. At the same time, state of vibrations was taken using a high-speed camera (Memrecam fx-k5, Nac Corp.). In this vibration conditions, half amplitude of the free end of cantilever was ~6 mm, as shown in Fig. 12.

Measurement result of the vibration test is shown in Fig. 13. Maximum output of 4.3 μW was achieved at $R = 2.1$ MΩ in case of sample 1. Looking at this result, the thicker PVDF film is, the higher output power is obtained. The reason is considered as follows: in the thicker PVDF film of VEH (sample 1), the distance between neutral axis and PVDF film is greater than that in the thinner PVDF film of VEH (sample 2). Therefore, stress and strain induced on the PVDF film of sample 1 are higher than those of sample 2. In simple and approximately calculation, the $30/12 = 2.5$ times larger strain would lead to $2.5^2 = 6.25$ times larger output power. Looking at Fig. 13, the peak power of sample 1 is 4.3 μW and that of sample 2 is 0.7 μW; the former is 6.1 times larger than the latter, which agrees with the calculation.

Table 3 shows comparison of our study with other cantilever type piezoelectric VEHs. According to the article of Kanno *et al.*, output powers of their VEHs using PZT ceramic films reached 1–17 μW [6–10]. However, vibration frequency of their vibration test was 108–890 Hz, which was higher than our study. Since theoretical output is proportional to the square of frequency, our VEH using polymer PVDF has performance equivalent to other VEHs using ceramic PZT.

Although piezoelectric constant of PVDF is lower than PZT [27], we could obtain equivalent power. This is caused by large deformation due to flexible polymer PVDF, which increases power generation.

5. Conclusion: PVDF film was formed by bar coating method and its mechanical and piezoelectric properties were characterised. A cantilever type VEH composed of PVDF thin film on a phosphor bronze rectangle plate was fabricated. The dimension of cantilever was 25 mm long, 15 mm wide. After corona polarisation, piezoelectric constant d_{33} of PVDF were estimated as −8.44 and −10.14 pC/N for 30 and 12 μm thickness, respectively. As a result of vibration test, maximum output power of 4.3 μW was achieved at $R = 2.1$ MΩ, which is equivalent to other cantilever type VEHs having ceramic PZT with high piezoelectricity, which is due to flexibility of polymer PVDF of generating large strain.

Thickness of PVDF film and corona polarisation condition should be optimised for enhancing the piezoelectric constant of PVDF in the future. Optimising the structure of VEH should be also carried out in the future by such a wary of employing MEMS technology.

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Table 3 Comparison of VEHs performance

	Piezoelectric material	Effective volume, mm³	Frequency, Hz	Acceleration, m/s²	Output power, μW	Power density, μW/mm³
Kanno [8]	PZT	0.2	344	25	2.5	12.5
Sung Sik Won [11]	KNN	2	132	9.8	3.62	1.8
Tang [12]	PMN-PT	0.6	237	19.6	5.93	9.83
Cao [25]	AlN	1.7	69	15.7	8.7	5.18
Wang [26]	PZT	4.3	89	9.8	15.4	3.62
This study	PVDF	6.6	55	17	4.3	0.65

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