PERFORMANCE OF HYDROTHERMAL PZT FILM ON HIGH INTENSITY OPERATION

Minoru Kuribayashi Kurosawa*, Hidehiko Yasui**, Takefumi Kanda** and Toshiro Higuchi**
*Tokyo Institute of Technology, Dept. of Advanced Applied Electronics,
4259 Nagatsuta, Midori-ku, Yokohama 226-8502, Japan, e-mail: mkur@ae.titech.ac.jp
**The University of Tokyo, Dept. of Precision Machinery Engineering,
7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656, Japan

ABSTRACT

A lot of processes about PZT thin film were reported, but there performances at high intensity operation have not been reported yet at all. Hydrothermally deposited PZT film transducers were examined on large vibration amplitude level at resonance frequency. It was demonstrated that the hydrothermal PZT film has superior performance on linearity and maximum vibration velocity. The linearity of frequency responses around the resonance were excellent and superior than bulk material up to high intensity operation. The maximum vibration velocity of a flexural transducer was 1.3 m/sec. Concerning a longitudinal transducer, two deposition processes were tried. The maximum vibration velocity was 0.53 m/sec and 0.9 m/sec. These value were almost two time larger than bulk PZT materials for high power applications.

INTRODUCTION

Several kinds of deposition processes for piezoelectric materials have been developed for many applications such as electronic devices and electro mechanical devices. Especially for actuators and sensors, high electro mechanical coupling materials are preferred. Hence, the piezoelectric factors such as d_{31}, d_{33}, and/or e_{31} were measured to evaluate the performance of the material. Correctly, these factors are very important to estimate static characteristics of devices. For example, sensitivity of a sensor or elctro mechanical transformation ratio are calculated using these factors.

However, we can hardly guess the dynamics of devices from these statically defined piezoelectric factors. For example, in the case of ultrasonic motor, the maximum rotation speed is limited by the vibration velocity of transducer. Therefore, to know the limit of the vibration velocity is important to know the mechanical output of a device. The static factors indicate only the proportion ratio between an electric port and a mechanical port. They gives few information of the performance of working condition.

Recently, in the world of normal size piezoelectric devices for power application such as piezo transformer, ultrasonic processing tools and ultrasonic motors, dynamic performance of piezo materials have been attracted researchers attention. Linearity and dynamic range are also important factor for high performance electro mechanical devices.

Hydrothermally deposited PZT film transducers were examined on large vibration amplitude level around resonance frequency. It was demonstrated that the hydrothermal PZT film has superior performance on linearity and maximum vibration velocity. The linearity of frequency responses around the resonance were excellent and superior than bulk material up to high intensity operation. The maximum vibration velocity of a longitudinal transducer was 0.9 m/sec. About a flexural transducer, it was 1.3 m/sec.
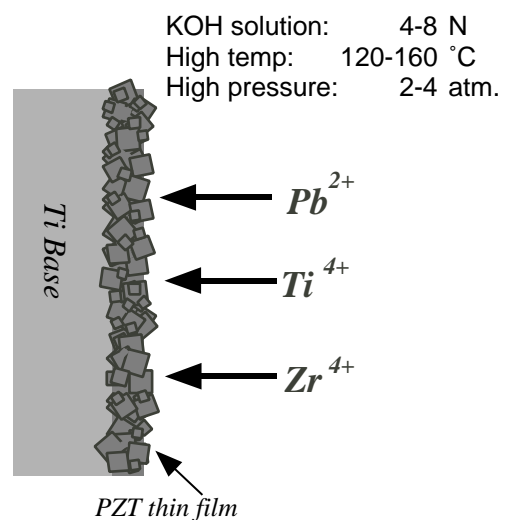

KOH solution: 4-8 N
High temp: 120-160 °C
High pressure: 2-4 atm.

Pb^{2+}

Ti^{4+}

Zr^{4+}

Ti Base

PZT thin film

Figure 1: Schema of the hydrothermal method for PZT thin film.

HYDROTHERMAL DEPOSITION OF PZT FILM

We have reported a micro ultrasonic motor[1] and a vibration touch probe sensor[2],[3] using hydrothermal PZT film. Hydrothermal method utilizes the chemical reaction between titanium base metal and the melted ions at high temperature. The chemical reaction is carried out in the solution as shown in Fig. 1. To sum up other merits of hydrothermal method, thick deposited PZT film, automatic polarization, and unnecessary of annealing process are attractive characteristics[4].

The process has two steps. Both processes are curried out in an autoclave at high temperature and high pressure. The first step is called nucleation process. During this process, nuclear crystals of PZT are deposited on the titanium base. For tight bonding of the film to the base material, the mechanism of crystal generation using the base material has an advantage.
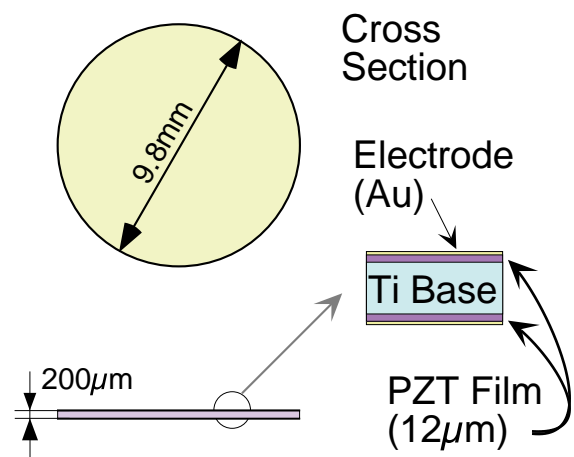
Table 1: Ingredient of the hydrothermal process so called "single process": Process A.

Process	Material	Concentration	
1st	$ZrOCl_2 8H_2O$	0.535 mol/l	0.507 g in 2 ml H_2O
	$TiCl_4$	1.603 mol/l	0.35 ml
	$Pb(NO_3)_2$	0.520 mol/l	1.204 g in 7 ml H_2O
	KOH	4 N	2.693 g in 11 ml H_2O
at 140 ℃, 24 hours			
2nd	$ZrOCl_2 8H_2O$	0.803 mol/l	0.761 g in 2 ml H_2O
	$TiCl_4$	1.603 mol/l	0.53 ml
	$Pb(NO_3)_2$	0.780 mol/l	1.806 g in 7 ml H_2O
	KOH	4 N	2.693 g in 11 ml H_2O
at 120 ℃, 24 hours, x2 times			

Table 2: Ingredient of the hydrothermal process so called "improved nucleation process": Process B.

Process	Material	Concentration	
1st	$ZrOCl_2 8H_2O$	2.09 mol/l	1.078 g in 2ml H_2O
	$TiCl_4$	1.97 mol/l	0.0433 ml
	$Pb(NO_3)_2$	1.06 mol/l	2.317 g in 7 ml H_2O
	KOH	8 N	5.47 g in 12 ml H_2O
at 160 ℃, 12 hours			
2nd	$ZrOCl_2 8H_2O$	1.24 mol/l	1.013 g in 2 ml H_2O
	$TiCl_4$	1.97 mol/l	1.707 ml
	$Pb(NO_3)_2$	1.04 mol/l	2.407 g in 7 ml H_2O
	KOH	4 N	2.69 g in 12 ml H_2O
at 120 ℃, 24 hours, x5 times			

Figure 3: Schematic view and cross section of circular disk PZT transducer for flexural vibration.

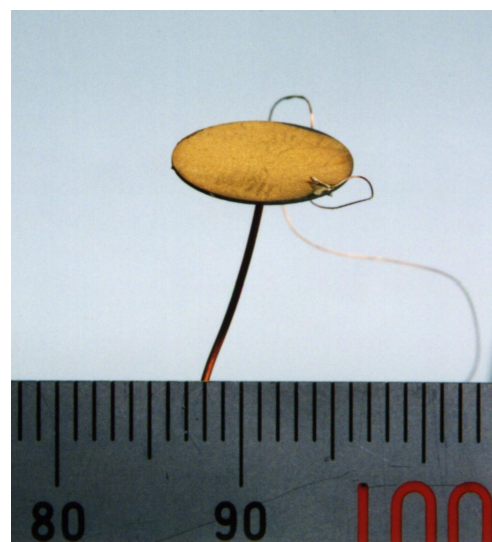Figure 2: SEM photograph of the hydrothermal PZT film surface.

Figure 4: Photograph of the circular disk transducer and feeding wire.

The original process did not contain titanium ion at the 1st step[4]. We proposed the improved processes [5] for electro mechanical devices. The reaction conditions which were adopted for transducer fabrication are shown in Table 1 and 2. The process is splitted two different ways for different applications. The process A indicated in Table 1 was applied to sensors[6]. This process is based on "single process"[5] for the first step. The process B indicated in Table 2 was applied to micro ultrasonic motors[7] and an ear phone[8]. The process B contains "improved nucleation process" and "crystal growth process".

The second step process was carried out at lower temperature of 120 degree Celsius as indicated at Table I

and 2. Concentrations of each ion were adjusted to obtain proper ingredient ratio. During the second process, PZT crystal are expected to grow up. The thickness of the film increased by repeating the crystal growth process. After the two kind of processes, we obtained 7 or 12 µm thick film for each side of the titanium base.

The deposited film is crystallized as shown in Fig. 2. The polycrystal structure is a distinctive feature of this process material. The piezoelectric factor d_{31} of deposited film was measured using the bimorph element. From the measurement of the vibration amplitude at low frequency drive, deformation was estimated. The piezoelectric factors d_{31} were estimated to be -34 pC/N or -25 pC/N from the measurements. The factor of bulk material is 3 times higher than these values.

SAMPLE TRANSDUCERS

The flexural vibration transducer is illustrated in Figs 3 and the photograph is shown in Fig. 4. This element was fabricated with the process B which was applied to the actuator[7],[8]. The vibration mode is 0th order flexural disk which was supported at the center. The diameter and the thickness of the base material of titanium circular plate were 9.8 mm and 0.2 mm. The thickness of the deposited PZT film was 12 µm on both side.

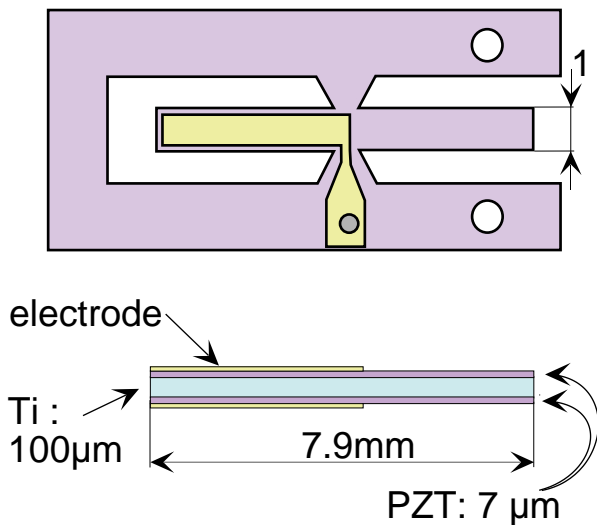Gold electrode was deposited on both side. The driving electrical field was supplied from the center support and

Figure 5: Schematic view of the rectanglar PZT transducer for longitudinal vibration; half wave length.
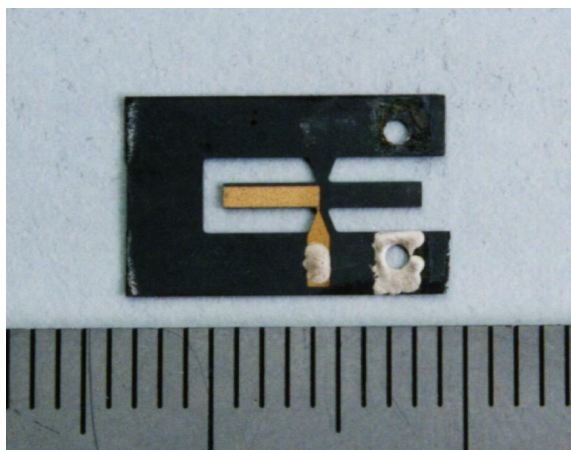
Figure 6: Photograph of the rectangular transducer.
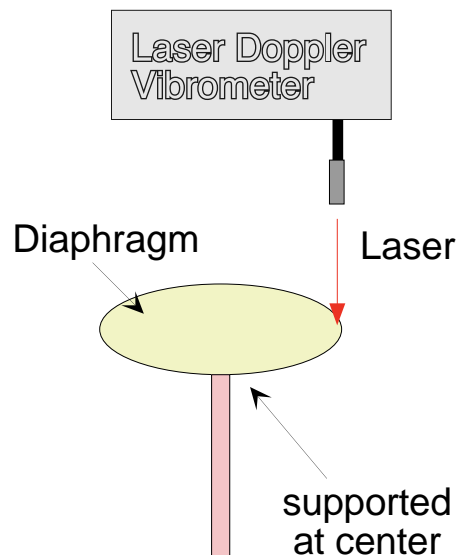
Figure 7: Schematic view of the measurement of the vibration velocity using laser Dopplar vibrometer.

the other side capper wire as shown in Fig. 4. Hence the polarization direction of the PZT films is titanium base, the flexural mode is excited.

A rectangular shaped longitudinal transducers which were 7.9 mm long and 1 mm width were fabricated. The schematic shape of the longitudinal transducer is shown in Figs. 5. The vibration mode was half wave length longitudinal vibration. The element was supported at the center nodal point of the axial vibration component. The

PZT film was deposited 7 μm or 12 μm on both side of 100 μm titanium base.

The photograph of the longitudinal transducer is shown in Figs. 6. Two elements were fabricated with the process A[5],[6] and also with process B which was applied to the touch probe sensor. The PZT film thickness was 7 μm with the process for the sensor. The electrical supply was fed from the gold electrode which was deposited on the quarter wave length part of one side and the titanium base.
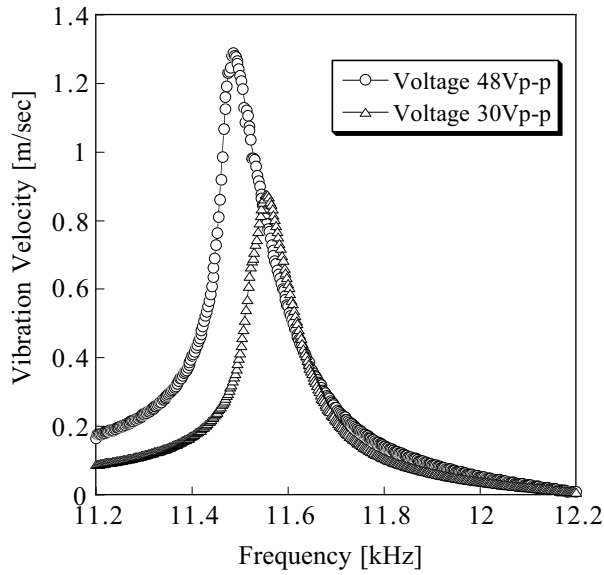

Figure 8: Frequency response of the circular transducer at middle and high driving voltages.
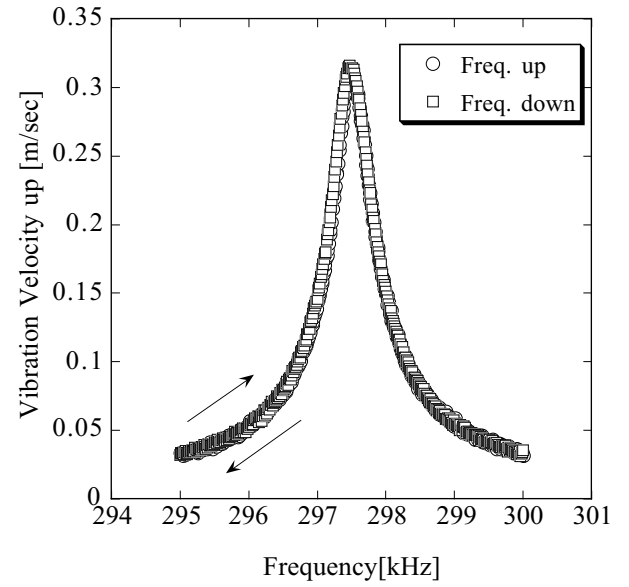

Figure 10: Frequency response of the rectangular transducer at middle driving voltage.
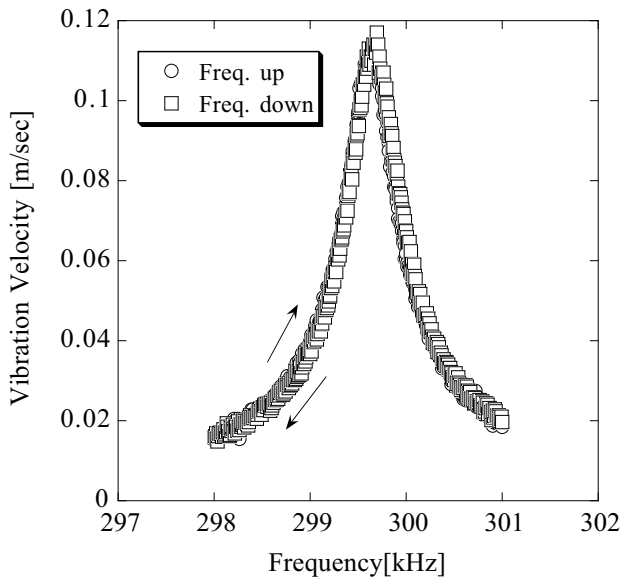

Figure 9: Frequency response of the rectangular transducer at low driving voltage.
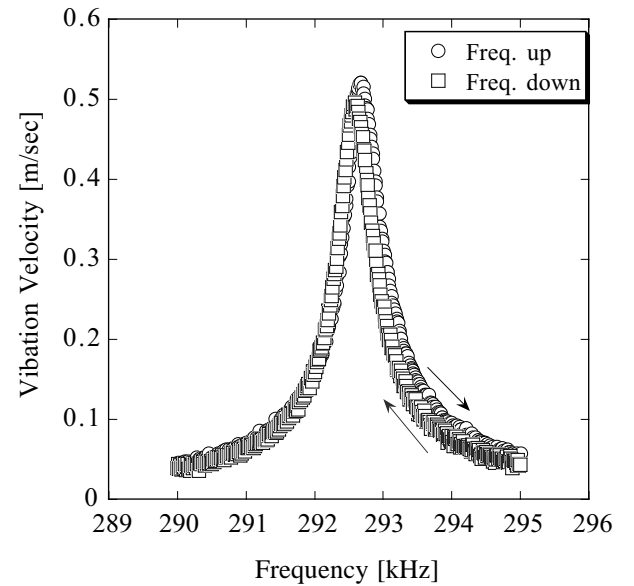

Figure 11: Frequency response of the rectangular transducer at high driving voltage.

The measurements of the vibration velocity was carried out with a laser Doppler vibrometer. As shown in Fig. 7 in the case of circular bending element, the bending vibration velocity at the edge was measured. In the case of the longitudinal vibration elements, the axial vibration direction at the end tip was measured with the laser Doppler vibrometer.

FREQUENCY RESPONSE

Under the condition that the driving voltage was constant, the vibration velocity was measured with changing driving frequency. In the case of bulk materials, it is well known that the response curves are distorted and jumping phenomenon are observed. In the case of hydrothermal PZT film devices, however, the response curves were smooth and continuous as shown in Figs 8, 9, 10 and 11.

Figure 8 shows the circular disc flexural transducer's response curves. The driving frequency changed from the lower frequency to the upper in both curves. From these responses, it is found that the Q factor of the material was constant up to extremely high intensity vibration level of 1.3 m/sec. In spite of the constant voltage drive, jumping phenomenon was not observed. Only the resonance frequency was slightly changed. At the low driving voltage, it was about 11.6 kHz. Although, the resonance frequency dropped to 11.5 kHz.

About the longitudinal vibration mode transducer, Q factor of the element was about 600 up to high intensity vibration level of 0.53 m/sec as shown in Figs 9, 10 and 11. This transducer was fabricated with the process A to deposit the PZT film. In these experiments, the frequency was changed upward and downward as indicated in the figures.

The resonance frequency depended on the strength of the driving electric field. At the lower driving voltage as shown in Fig. 9, the resonance was higher than 299 kHz. However, it was 297. 5 kHz at the medium and at last it was below 293 kHz as can be seen from Figs 10 and 11. It seems that the piezoelectric factor and/or the elastic modulus changed by the driving voltage.

Regardless of the resonance frequency shift, the Q factor of the transducer was constant against the driving voltage and the response curves were very smooth and continuous. The element seems to be good for sensor device. At least, it is superior than bulk PZT transducer which has strong non-linearity.

MAXIMUM VIBRATION VELOCITY

The maximum vibration velocity depends on material strength and is independent to frequency. We obtained the maximum vibration velocity of 1.3 m/sec by the flexural vibrator, 0.53 m/sec by the longitudinal vibrator with the process of sensor application and 0.9 m/sec with the
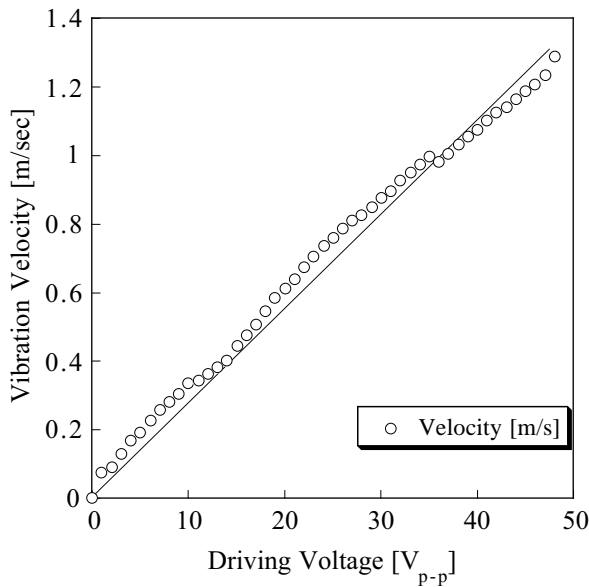
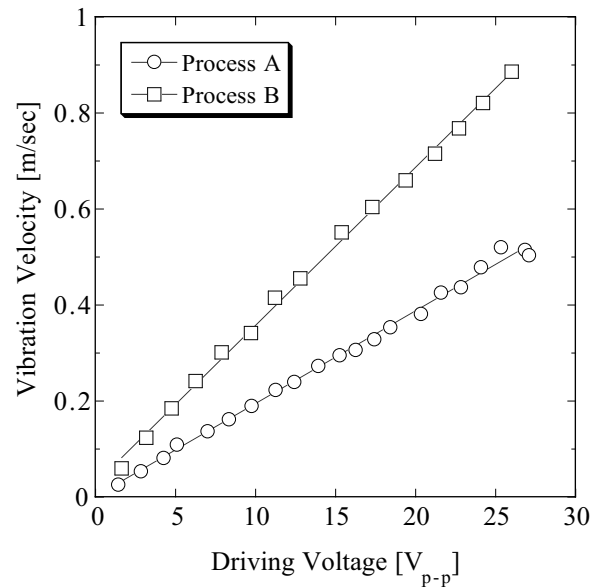Figure 12: Vibration velocity of the circular transducer; flexural vibration.

Figure 13: Vibration velocitis of the rectangular transducers; longitudinal vibration.

process of actuator application as indicated in Figs 12 and 13. The vibration velocity did not saturate, but the devices brake down due to high electric field. From calculation based on the experimental data, the piezoelectric coefficient of e_{31} of the longitudinal transducer was estimated to be 0.23 C/m^2 by process B and 0.10 C/m^2 by process A.

The maximum vibration level of bulk PZT elements was 0.3 m/sec[9]. Recent report of improved PZT with dopant of Fe ions has better performance, but the maximum vibration velocity is around 0.6 m/sec on the condition of without Q factor dropping. In case of single crystal material such as LiNbO$_3$, this limit is 3 m/sec[9]. The hydrothermal PZT material is intermediate of ceramics and single crystal.

CONCLUSION

Dynamic performance of the hydrothermally deposited PZT film transducers were measured. Two different processes were tested to fabricate the transducers and two different vibration modes, namely longitudinal and flexural, were tested as the transducers. Linearity of the transducer frequency response was very excellent without jumping effect and decrease of Q factors. The maximum vibration velocity was superior, 1.3 m/sec in flexural vibrator and 0.9 m/sec in longitudinal vibrator. It is concluded that the hydrothermal PZT has fine linearity and high intensity output. These merits are good for MEMS transducers. They seem to be based on poly-crystal structure of the material.

ACKNOWLEDGMENTS

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