Transactions of the Materials Research Society of Japan 27 [1] 251-253 (2002)

Effects of Surface Condition on the Electrical and Chipping Properties of PZNT Single Crystal Vibrators for Medical Array Transducers.

Tsuyoshi KOBAYASHI, Yasuharu HOSONO, Mamoru IZUMI, Kazuhiro ITSUMI, Koichi HARADA and Yohachi YAMASHITA

Power Supply Materials and Devices Laboratory, Corporate R.& D. Center, Toshiba Co.

1, Komukai Toshiba-cho, Saiwai-ku, Kawasaki 212-8582, JAPAN

Phone : +81-44-549-2134, Fax : +81-44-520-1286, Tuyosi.kobayashi@toshiba.co.jp

Abstract — Effects of different surface conditions on the electrical and mechanical properties of lead zinc niobate titanate (Pb0.91(Zn1/3Nb2/3)0.09TiO3 (PZNT 91/9)) piezoelectric single crystal (PSC) have been investigated. The PZNT PSC wafers manufactured by different surface conditions, such as lapped by #2,000, #4,000, #6,000 Al2O3 powders and by diamond paste to mirror, were cut into many slivers elements by pitch of 180 μm with 30μm thick diamond blade for fabricating array medical transducer. The amounts of chippings were increased of the surface conditions of #6,000 and mirror. lapped wafers. In addition, a weak bonding strength of Au electrode on the PSC was seen of the mirror lapped wafers after dicing. Damaged PSC sliver showed many spurious modes on the piezoelectric impedance response. It is important to control the surface roughness of PSC to prevent chipping and to realize a good electrode bonding strength on the PSC after dicing.

1. Introduction

Recent reports indicate that some single crystals of the relaxor (Pb(B1,B2)O_3)-lead titanate $PbTiO_3$ (PT) binary system have excellent piezoelectric properties near the MPB[1-2]. The authors have conducted an investigation with a view to improving a high sensitivity and a broad bandwidth of ultrasonic transducers of echo ultrasound (Fig. 1) by using $Pb[(Zn_{1/3}Nb_{2/3})_{0.91}Ti_{0.09}]O_3$ (PZNT 91/9) single crystal[3-4] instead of the conventional $Pb(Zr_{1-x}Ti_x)O_3$ ceramics that have mainly been used for present transducers. Single crystals between $Pb(Zn_{1/3}Nb_{2/3})O_3$ and $PbTiO_3$ can be grown by a flux method over the whole composition range. Kuwata et al. reported an extremely large electromechanical coupling factor in the longitudinal bar-mode (k_{33} = 0.92) and piezoelectric strain constant d_{33}>1,500 pC/N for PZNT 91/9 PSC. The authors reported that large size PZNT 91/9 single crystals have been successfully obtained using a novel flux method[5] and solution Bridgman process[6] with a suitable ratio of 45mol% PZN-PT 91/9 : 55mol% PbO flux[5]. The largest crystal had dimensions of 43×42×40 mm and weighed 420 g. The transducer obtained from the ingot has an enough size (>15x25x0.3mm) and quality level for cardiac medical transducers.

In order to fabricate array type transducer, the PSC vibrator is cut into tiny slivers ranging from 100-200 microns pitch by diamond blade saw. In the dicing process, many chippings are occurred due to its low mechanical brittleness of the PSC compared to that of PZT ceramics. However, no reports on the effects of different surface conditions on the electrical and mechanical properties of PZNT PSC have been reported. The purpose of this report is to investigate chipping, mechanical strength and electrical properties of PZNT PSC with different surface controlled vibrators.

2. Experimental Procedure

The PZNT 91/9 single crystal was grown by solution Bridgman method using a PbO flux. A detail process of PSC growth has been reported.[6] Obtained PZNT ingot was sliced into 0.5mm thick (001) wafers. These wafers were lapped with different white Al_2O_3 powders, #2,000, #4,000 and #6,000. In addition, mirror lapped wafers were prepared by lapping of #6,000 wafers by using diamond paste. Thickness of 0.05 and 0.2 μm-Cr/Au electrodes were deposited on both sides of PSC by sputtering.

For the investigation of electrical and mechanical properties, the 10 pieces of 1mm width ×0.3 mm thick × 10 mm Length rectangular bars and 10 pieces of 0.15 mm width ×0.3 mm thick × 10 mm length sliver vibrator were formed from the each surface roughness (100) wafer. The sliver vibrator's size is a similar to that of the medical array ultrasonic transducers of 2.5 MHz center frequency. In order to measure the piezoelectric properties, the specimens were poled at 0.3 kV/mm in a silicone oil bath. The electric field was applied at a temperature of 200℃ for 10 min, and the specimens were cooled to 25℃ in the same field. The electromechanical coupling factor rectangular bar mode, k_{33}' was measured by the resonance-and anti-resonance method using the sliver vibrator.

The bending strength σ max was measured by 3-point bending test by using 1x10x0.3mm rectangular bar and 0.15x10x0.3mm sliver samples. The average value of 10 pieces of 1 mm width rectangular plates and 0.15 mm width sliver of each four kinds of surface roughness vibrators were measured.

3. Result and Discussions

Table I shows the summarized results.

Figure 3 shows a typical frequency response and an impedance of a damage-free PSC slier (A) and a damaged PSC sliver. Many spurious modes are seen if the sliver has many chipping or cracks after the dicing.

Figure 4 shows a surface condition of #2,000 sample view from the top. There are many scratches and small bumps caused by Al2O3 polishing powder. These small mechanical damages may cause a origin of the chippings or cracks of the sliver in dicing process. However, the sliver vibrators rapped by #2,000 or #4,000 showed a few amounts of chippings or cracks. In comparison mirror lapped sliver vibrators showed many chippings or cracks after dicing as seen in Fig. 5. Therefore, hidden cracks below the surface strongly affect the chipping and cracks of PSC sliver during the dicing.

In addition, samples of #6,000 and mirror showed a weak electrode bonding strength.

The chipping-less vibrators that of #2,000 and #4,000 showed high electro-mechanical coupling factor; *k33'* = 85%. However, the lower value of 79% and 75% were obtained and many spurious modes were seen in the #6,000 and mirror samples, respectively. These degradations of electro-mechanical coupling factor *k33'* caused by the defects of surface may affects the sensitivity scattering between channels of array type transducer. Three point bending strength of PSC 1mm bar sample increase with decreasing surface roughness, Rmax. up to 0.05 μ m. Surprisingly, the mirror lapped samples showed the lowest bending strength of the 1mm bar and 0.15 mm slivers samples. The cause is estimated that there are many hidden small cracks under the surface.

4. Conclusion

The relation between surface roughness, bending strength and its electrical properties of the PZNT 91/9 piezoelectric single crystal (PSC) were investigated. The results are as follows:

1) The bending strength increased with decreasing surface-roughness of the PZNT PSC vibrator if no chipping were seen.
2) The amount of chippings in the PZNT vibrator increased markedly in the vibrator lapped #6,000 and mirror.
3) The increasing of the chipping amounts affects a decreasing of the bending strength, deteriorated electromechanical coupling factors and boding strength of the electrodes on the sliver vibrators.
4) In the view point of the fine pitch dicing process for fabricating the array type ultrasonic transducers, the #4,000 lapping which has approximately the Rmax=0.1 μ m surface roughness is the best for the PZNT 91/9 PSC vibrators.

References:

1) J. Kuwata, K. Uchino and S. Nomura: Jpn. J. Appl. Phys. 21(1982)1298.
2) T. R. Shrout, Z. P. Chang, N. Kim and S. Markgraf: Ferroelectr. Lett. 12(1990)63.
3) S.Saitoh, M.Izumi, S.Shimanuki, S.Hashimoto and Y.Yamashita, U.S. Patent 5,295,487,(1994).
4) S.Saitoh, T.Takeuchi, T Kobayashi, K.Harada, S.Shimanuki and Y.Yamashita, Jpn. J. Appl. Phys.,Vol.38,No.5B,pp.3380-3384(1999).
5) T. Kobayashi, S. Shimanuki, S. Saitoh and Y. Yamashita: Jpn. J. Appl. Phys. 36(1997)6035.
6) K.Harada, S.Shimanuki, S.Saitoh, and Y.Yamashita, J.Am. Ceram. Soc., Vol.81,[11], pp.2785-2788(1998).

Table I Physical and electrical properties of PZNT 91/09 PSC vibrators with different surface conditions.

Surface condition		#2,000	#4,000	#6,000	Mirror
Al2O3 Powder particle size	(μ m)	12.7	6.4	4.2	<1.0
Surface roughness Rmax.	(μ m)	0.16	0.08	<0.05	<0.02
Bending strength (W=1.0mm)	(kg/mm3)	43	44	45	38
Bending strength (W=0.15mm)	(kg/mm3)	51	63		48
E/M coupling factor *k33'*	(%)	85	85	79	76
Spurious mode		few	few	some	many
Chipping		few	few	some	many
Electrode peeling off		none	none	few	many

Figure 1. Echo ultrasound and medical prove

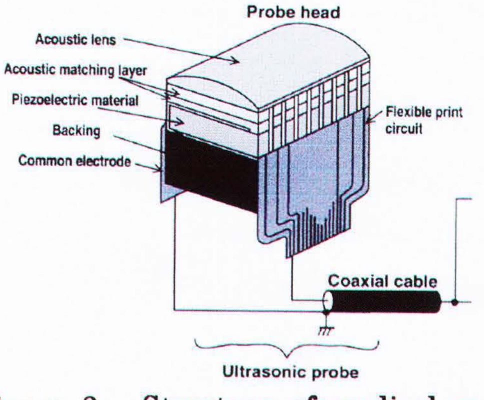
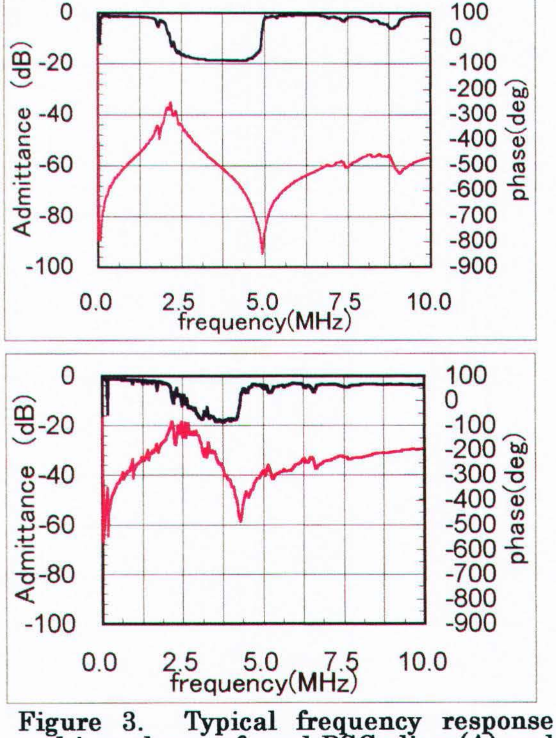


Figure 2. Structure of medical proves. Note: Piezoelectric material and matching layers are cut into small slivers by dicing.



(A)

(B)

Figure 3. Typical frequency response and impedance of good PSC sliver(A) and damaged PSC sliver(B). The damaged PSC sliver shows many spurious modes.

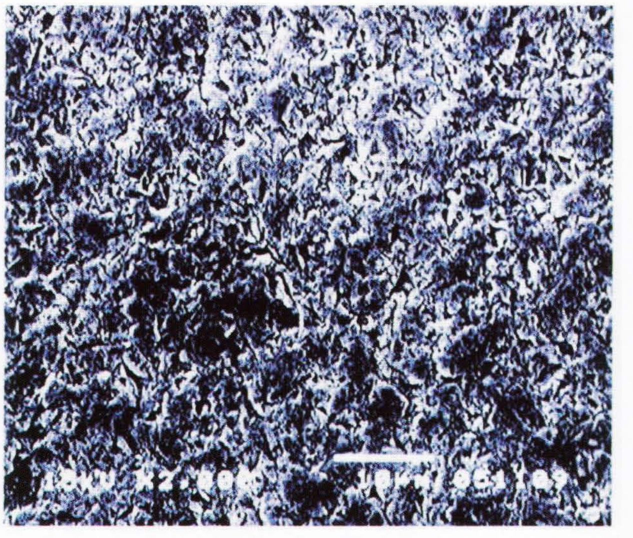


Figure 4. PZNT PSC surface lapped by #2,000 Al_2O_3 powder. Note: There are many scratches on the surface of the PSC.

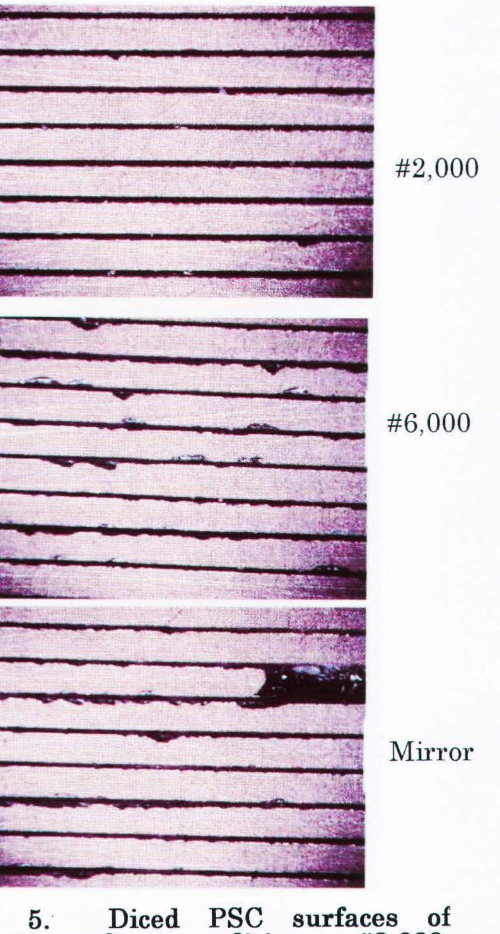


Figure 5. Diced PSC surfaces of different surface conditions, #2,000, #6,000 and mirror lapping (View from the top). Note: Mirror lapping and #6,000 lapped PSC show electrode peeling off and many chipping after dicing. Dicing pitch is 180 micron with 30 micron blade.

(Received December 21, 2001; Accepted January 31, 2002)