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Proceedings of Symposium on Ultrasonic Electronics, Vol. 34 (2013) pp. 27-28
20-22 November, 2013

A Study on High Temperature Behavior of PZT/PZT Piezoelectric Composite
圧電複合体 PZT/PZT の高温における挙動に関する研究

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1. Introduction

Demand on continuous monitoring of nuclear power plants during operation and/or cold shutdown increases for safety assurance. For continuous monitoring during plant operation, sensor should have long term high temperature durability. In addition, temperature of nuclear power plants could rise up suddenly once serious problem happens even during cold shutdown. It was very challenging to apply traditional ultrasonic transducers for such conditions.[1)] Ultrasonic transducers made by piezoelectric sol-gel composites could operate in such severe conditions because of thermal shock resistance and no necessity of backing and/or couplant. Especially, PZT/PZT showed best sensitivity among developed sol-gel composites.[2)] However, Curie temperature of PZT is 300-350°C and there was anxious point whether PZT/PZT ultrasonic transducer could survive after experience of abnormal temperature rise.

Thus high temperature behavior of PZT/PZT was investigated in order to study about abnormal temperature rise durability. PZT/PZT films were fabricated onto metal substrate with high temperature durability such as titanium and stainless steel. In concrete explanation, real-time ultrasonic monitoring during heating up to 550°C was examined first. Another sample was heated up at certain temperature then cooled down to room temperature. After cooling, piezoelectric constant d_{33} was measured and ultrasonic monitoring at room temperature was attempted to acquire recovery possibility of PZT/PZT ultrasonic transducer after abnormal temperature rise incidents.

2. PZT/PZT ultrasonic transducer fabrication

Fabrication process of PZT/PZT ultrasonic transducer is as following; First, PZT powder and sol-gel solution was mixed by ball milling. Next, the mixture was sprayed directly onto metal substrates. Drying process at 150°C, and firing process at 650°C were operated. Those spray coating process and thermal process were repeated several times in order to obtain ~50μm thickness. After corona poling at room temperature, ~5mm diameter top electrodes were fabricated onto piezoelectric films.

3. Experimental results

3.1 Real time ultrasonic monitoring

PZT/PZT ultrasonic transducer onto ~3mm thick titanium substrate was heated by a hot plate from room temperature up to 550°C, and every 50°C ultrasonic response in pulse-echo mode was monitored and recorded. **Fig. 1** showed ultrasonic measurement result at 400°C. Clear multiple echoes from back surface of titanium substrate could be observed with reasonable SNR. This result indicated that ultrasonic response still existed even above Curie temperature of PZT, i.e. 300-330°C.[3)] This phenomenon could come from electrostrictive effect[4)] or momentum poling by Pulser/Receiver instrument. **Fig. 2** showed temperature dependency on sensitivity. The sensitivity was calculated by -1 multiply Pulser/Receiver gain to achieve $1V_{p-p}$ of 3rd reflected echo. The results of 500-550°C were omitted since silver paste top electrode deterioration was found. It seems that sensitivity decreased linearly, even though signal was still very clear at 400°C in Fig.1.

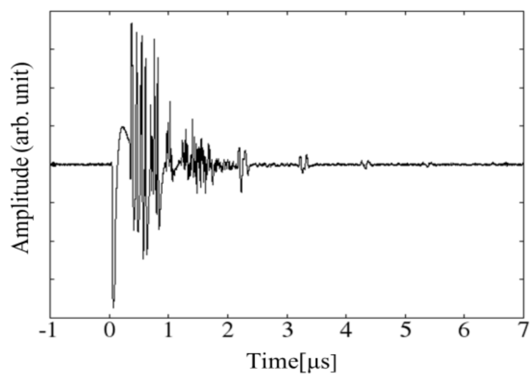

Fig.1 : Ultrasonic response of PZT/PZT ultrasonic transducer onto ~3mm thick titanium plate at 400°C.

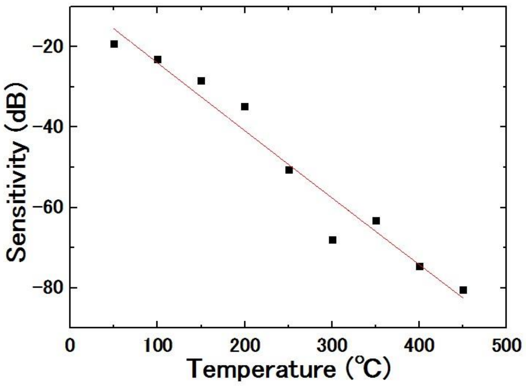

Fig.2 : Temperature dependency on sensitivity of PZT/PZT ultrasonic transducer.

3.2. High temperature history test

PZT/PZT ultrasonic transducer onto ~1.5mm thick stainless substrate was heated by a hot plate up to 50, 100, 150, 200, 250, 300, 350, 350, 400, 450, 500 and 550°C. After reaching those temperatures, sample was immediately removed and after cooling to room temperature, piezoelectric constant d_{33} by ZJ-3EN Piezo d33 meter supplied by Institute of Acoustic, Chinese Academy of Science first, then ultrasonic response in pulse-echo mode was monitored and recorded and **Fig. 3** showed temperature dependency on sensitivity and piezoelectric constant d_{33}. In this time, the sensitivity was calculated by -1 multiply Pulser/Receiver gain to achieve 1Vp-p of 7th reflected echo. It seems that sensitivity corresponded d_{33} value very well. Up to 400°C, reasonable piezoelectricity was still remained whereas above 450°C, piezoelectric became very low. Ultrasonic response of PZT/PZT ultrasonic transducer onto ~1.5mm thick stainless steel after 400°C heating was shown in **Fig. 4**. In Fig.4, clear multiple echoes were still observed whereas SNR was significantly deteriorated above 450°C. It should be mentioned that non-poled PZT/PZT sample could be poled by normal Pulser/Receiver operation and the sensitivity was very similar to above 450°C. It indicated that even by momentum exposure, PZT/PZT could be significantly depoled, though certain degree of ultrasonic performance could be recovered by Pulser/Receiver poling.

5. Conclusions

PZT/PZT ultrasonic transducers were fabricated onto few mm thick metal plates with high temperature durability in order to study about high temperature history influence. For real-time monitoring at elevated temperatures, clear multiple echoes at 400°C, which was above Curie temperatures of PZT powder phase and PZT sol-gel phase, were observed with reasonable SNR, though sensitivity showed linear decrease. High temperature history test showed similar tendency between sensitivity measurement and d_{33} measurement and the results indicated that PZT/PZT ultrasonic transducer could operate up to 400°C, and even after 550°C exposure experience, ultrasonic response could be recovered when it was poled by Pulser/Receiver instrument. Therefore it could mentioned that PZT/PZT ultrasonic transducer could operate after abnormal temperature increase.

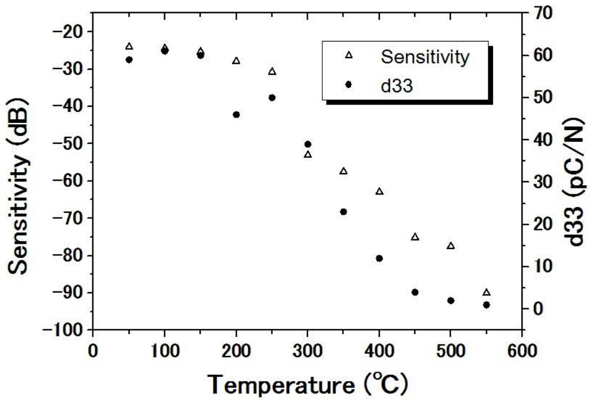

Fig.3 : Maximum temperature history dependency on sensitivity and piezoelectric constant d_{33} of PZT/PZT ultrasonic transducer.

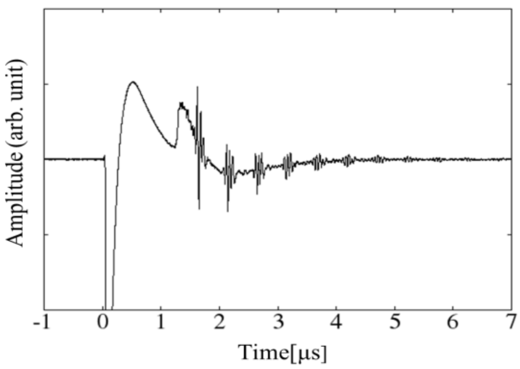

Fig.4 : Ultrasonic response of PZT/PZT ultrasonic transducer onto ~1.5mm thick stainless steel at room temperature after 400°C heating.

References

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