# High Temperature Immersion Ultrasonic Probe Development using PbTiO<sub>3</sub>/PZT Ultrasonic Transducer

PbTiO<sub>3</sub>/PZT 超音波トランスデューサを用いた高温水浸超音波 探触子の開発

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# 1. Introduction

In the facilities such as nuclear reactors, development of the ultrasonic probe for the inspection of high-temperature liquid molten metal because an accident could be resulted in tragetity. In these facilities, non-destructive testing (NDT) is required since fatigue cracks and partial corrosions could happen due to thermal non-uniformity. Because molten metal is opaque and conductive, NDT by ultrasound is effective method because it can obtain the internal image without damaging.

However, ultrasonic measurement at high temperature was generally difficult because of low high temperature durability of piezoelectric material, couplant, and backing material which are elements of ultrasonic transducer. In the past studies, the high probes immersion temperature had been accomplished by a delay line.<sup>1-3)</sup> In this method, the size of probe with deley line became large sacale and mechanical scanning could be difficult. In previous study, high temperature immersion ultrasonic probe without delayline was developed by using Lead zirconate titanate (PZT)/PZT sol-gel composite.<sup>4)</sup> Sol-gel composite is composites by ferroelectric powder and dielectric sol-gel derived material and it has several advantages such as thermal shock resistance, good signal-to-noise ratio (SNR), and no neccessity of backing and couplant. However, PZT/PZT sol-gel composite could not serve above 200°C for long term. Relatively low SNR was also concerned.)

In this research, high temperature immersion ultrasonic probe using PbTiO<sub>3</sub>(PT)/PZT sol-gel composite was fabricated and it's acoustic performance and heat resistance were tested. In past study, PT/PZT sol-gel composite showed superior sensitivity to PZT/PZT from room temperature to 360°C.<sup>5</sup>) Therefore improvement of high temperature durability and SNR was expected.

# 2. Immersion probe fabrication

Piezoelectric film of the ultrasonic probe was made by PT/PZT sol-gel composite. For PT/PZT film fabrication, the mixture of PZT sol-gel solution and PT powder were ball milled for more than one day. Then PT/PZT film was sprayed onto 50µm thick stainless steel substrate. After spray coating, drying process at 150°C for 5min, and firing process at 650°C for 5min were operated. Those spray coating process and thermal process were repeated until the target thickness was achieved. In this research, those processes were repeated 15 times in order to obtain 100µm thickness of PT/PZT film. After electrical poling by corona discharge, silver paste top electrode was fabricated onto the PT/PZT film. Fig. 1 shows the fabricated sample.

The diameters of PT/PZT film and silver top electrode were ~30mm and ~4mm, respectively. After PT/PZT film fabrication as shown in **Fig. 1**, the substrate was bonded to the stainless steel pipe using high temperature waterproof adhesive material.



Fig. 1: Optical image of 100  $\mu$ m thick PT/PZT film onto 50  $\mu$ m thick stainless steel substrate.

### 3. Measurement setup

To determine the possibility of fabricated immersion probe, that probe was tested in the water at various temperature. The experimental setup schematic was shown in **Fig. 2**. An aluminum plate with 4mm thickness was set into the bottom, and the immersion probe was placed over the aluminum plate. The distance of the immersion probe and the

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aluminum plate was ~10mm. The probe was connected to the digital oscilloscope and pulser/receiver, and the measurements were carried out pulse-echo mode. It was preferable to obtain at least first and second reflected echoes from top surface and bottom surface so that the possibility of acoustic imaging of top surface and crack detection could be confirmed.

Thermal cycles from room temperature to 95°C were operated three times to examine the probe operation stability. Every 10°C temperature rise, the ultrasonic response was recorded.



Fig. 2: Schematic of measurement setup.

## 4. Experimental results

Typical measurement result at 90°C was shown in **Fig. 3**. Multiple reflected echoes from the top and bottom surfaces of the aluminum plate were confirmed. The pulse-echo at 14 $\mu$ s (A) considered the one round trip reflected echo between the probe and top surface of the aluminum plate. And the pulsed-echo at 15 $\mu$ s (B) is the one round trip reflected echo between the probe and bottom surface of the aluminum plate. These results were determined by delay time and distance to the aluminum plate. It indicated potential use for acoustic imaging.



Fig.3: Ultrasonic response in the water at 90°C during the second cycle.

The result of sensitivity of the echo (B) temperature dependency was shown in **Fig. 4**. During three cycles, the sensitivity of reflected echoes under the 60°C was almost same. It indicates that PT/PZT immersion probe did not deteriorate up to 90°C. The sensitivity started to decrease and the sensitivity varied between 80-90°C. This phenomena was caused by the bubbles attached with stainless steel just beneath PT/PZT. Further research is required to measure the sensitivity in the liquid with higher boiling point than water such as the silicone oil to confirm the high temperature durability of the probe above 100°C.



Fig.4: Sensitivity temperature dependency during 3 thermal cycles.

#### 5. Conclusions

An immersion ultrasonic probe using PT/PZT was developed and it could operate at the temperature of 90°C water. Reflected echoes from top and bottom surface of a aluminum plate were clearly measured and no deterioration furing 3 thermal cycle. Measurement in silicone oil bath will be presented.

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