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High-Temperature and Broadband Immersion Ultrasonic Probes

Yuu Ono, *Member, IEEE*, Makiko Kobayashi, *Member, IEEE*, Jean-François Moisan, and Cheng-Kuei Jen, *Senior Member, IEEE*

Abstract—Immersion ultrasonic probes for measurements and imaging at high temperature are presented. The probes consist of sol-gel-sprayed thick films as piezoelectric ultrasonic transducers (UTs) directly deposited onto steel buffer rods. They operate in pulse-echo mode at temperatures up to 500 °C. The operating ultrasonic frequency is between 5 MHz and 20 MHz, controlled by the film thickness. The ultrasonic thickness measurement of a steel plate with the probe fully immersed in molten zinc at 450 °C was demonstrated using ultrasonic plane waves. For imaging purposes, the probing end of the steel buffer rod was machined into a semi-spherical concave shape to form an ultrasonic lens and achieve high spatial resolution with focused ultrasound in liquids. Ultrasonic surface and subsurface imaging using a mechanical raster scan of the focused probe in silicone oil at 200 °C was also carried out. The importance of the signal-to-noise ratio (SNR) in the pulse-echo measurement is discussed.

Index Terms—High-temperature ultrasonic transducer (HTUT), immersion probe, sol-gel-spray technique, thick film, ultrasonic imaging.

I. INTRODUCTION

ULTRASONIC pulse-echo techniques [1], [2] are often used to perform nondestructive testing and characterization of materials due to their simplicity, speed, economy, and capability to probe interiors of opaque materials. Such techniques sometimes require ultrasonic transducers (UTs) of large bandwidth, high signal-to-noise ratio (SNR), and to operate at elevated temperatures. Various efforts have been devoted to develop high-temperature (HT) piezoelectric UTs [3]–[15]. Commercially, several piezoelectric HTUTs are also supplied by several companies, such as Etalon (Lizton, IN), RD-Tech Panametrics-NDT (Waltham, MA), RTD (Rotterdam, The Netherlands), Ultrat (Boalsburg, PA), and Ishikawajima Inspection and Instrumentation (Tokyo, Japan), etc. Most of these HTUTs use buffer rods (delay lines) to facilitate the fabrication or HT measurements and to increase the ultrasonic frequency bandwidth.

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In certain situations, UTs must be immersed in liquids or gases at elevated temperatures [4], [5], [9], [12]–[15]. The reported examples were performing ultrasonic measurements in Canola oil at 125 °C using a 1–3 PZT piezocomposite transducer [14]; viewing under liquid sodium coolant in order to inspect in-vessel structures in nuclear reactor using lead zirconate titanate (PZT) ceramics up to 220 °C [12], lead zirconate ceramics up to 285 °C [4] or lithium niobate crystals up to 600 °C [5]; and measuring deformation of workpieces in a hot isostatic pressure vessel using an aluminum nitride film transducer at temperatures above 1000 °C and pressures above 150 MPa [9]. For most of these HTUTs, the piezoelectric materials were bonded [4], [5], [12], [14] or deposited [9] onto the substrates. In this study, we present an alternative method to fabricate immersion HT ultrasonic probes using a sol-gel-spray technique. Wall thickness measurements and ultrasonic imaging at temperatures up to 450 °C will be demonstrated in ultrasonic pulse-echo technique.

II. FABRICATION

A piezoelectric bismuth titanate, $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ (BIT), could be an attractive candidate for devices such as sensors and actuators operating at elevated temperatures due to its high resistivity and high Curie temperature (675 °C) [13], [16], [17]. The BIT powder was dispersed into PZT solution by a ball milling method to achieve the gel. An air gun was then used at room temperature to spray the sol-gel BIT/PZT composite directly onto metallic substrates, which can have a flat or curved surface. Spray conditions, such as air pressure, distance, and angle of the air gun to the substrate, and spray time were carefully adjusted to achieve homogeneous films. After spray coating, thermal treatments of drying, firing, and annealing were carried out. Multiple layers were made in order to reach the desired film thickness. The piezoelectric film operating at an ultrasonic frequency range from 5 to 20 MHz can be manufactured by controlling the total film thickness. For this investigation, BIT/PZT films of 90 μm thickness were made. Films were then electrically poled using a corona discharge technique. Finally, a top electrode was fabricated on the BIT/PZT film. The detailed fabrication process can be found in [18].

Fig. 1 shows the BIT/PZT film with a top electrode fabricated on a steel buffer rod with a length of 51 mm. The diameters at the UT end and the probing end of the rod were 32 and 30 mm, respectively, in order that the periphery of the rod had a 2° taper angle from the UT to probing ends for assembling the rod in a holder with a good seal preventing the liquid contacting the film UT. We used a platinum film fabricated by vacuum sputtering or a silver paste via painting at room temperature to form the top

Fig. 1. Ultrasonic probe consisting of a BIT/PZT film deposited onto a steel buffer rod.

Fig. 2. Ultrasonic probe mounted in a holder.

electrode. The electrodes of the platinum and silver paste were tested, and their operating temperature could be above 500°C. However, our experiments showed that the platinum electrode had better performance than the silver paste in an aspect of endurance at temperatures above 250 °C. The detail will be discussed later.

The platinum film of 0.1 μm thick and 10 mm in diameter was fabricated at the center of the buffer rod as seen in Fig. 1. The optimum diameter of the top electrode for this UT in order to obtain the strongest ultrasonic signal was around 10 mm. A coaxial cable made of stainless steel with magnesium oxide insulators was used as an electrical cable. This coaxial cable is applicable up to 900 °C. An alumina ceramic plate (6.35 mm thick and 21.6 mm in diameter) was used to press an inner lead of the coaxial cable on the platinum top electrode with three stainless steel screws. Thus, a steady electric contact between the inner lead and the top electrode and a perfect electric isolation between the top and bottom electrodes (steel buffer rod) were realized. The HT probe mounted in a stainless steel (S316) holder, which can seal the BIT/PZT film HTUT from liquids, is shown in Fig. 2.

In order to investigate ultrasonic performance of the HT probe at elevated temperatures, the probe shown in Fig. 2 was set in an electric resistance furnace and heated. Fig. 3(a) shows ultrasonic signals (L^1) measured at 22 °C and 450 °C with the ultrasonic pulse-echo technique. The L^1 signal is the first round-trip echo reflected from the probing end shown in Figs. 1 and 2. Fig. 3(b)

Fig. 3. Ultrasonic performance of the HT probe in (a) time and (b) frequency domains at 22 °C and 450 °C.

presents the frequency spectrums of the L^1 shown in Fig. 3(a). The center frequency was 8 MHz and the 6-dB bandwidth was 4 MHz for the L^1 at 450 °C. The signal strength of the L^1 at 450 °C was 1.5 dB smaller than that at 22 °C at 8 MHz. The SNR of more than 40 dB was obtained both at 22 °C and 450 °C. Such ultrasonic performance is sufficient for thickness measurements to be presented in Section III.

The SNR, which is one of the important parameters to evaluate performance of ultrasonic probes, is defined as the strength of the echo reflected at the probing end over that of the unwanted echoes in the probe (buffer rod here). The undesired echoes are called “spurious signals” throughout this paper. The spurious signals mainly come from the following sources: mode conversion, wave reverberation and diffraction within the rod of finite diameter and specific shape and scattering echoes from random grains or voids in the rod materials. High SNR can make many practical ultrasonic monitoring applications feasible. We also fabricated a PZT/PZT film UT by the sol-gel-spray technique and compared its ultrasonic performance with that of BIT/PZT film UT. The PZT/PZT UT had about 10 dB stronger ultrasonic signals than the BIT/PZT UT. The SNR of the PZT/PZT UT was almost the same as that of the BIT/PZT UT. However, operation temperature of the PZT/PZT UT was limited up to 200 °C.

In order to test their durability, the HT probes were submitted to thermal cycles between room temperature and elevated temperatures using a furnace. During each thermal cycle, it took about 1 h to heat the probe from room temperature to elevated

temperatures and then to cool it to room temperature. The durability test of the probe with the silver paste top electrode up to a furnace temperature 250 °C verified that the strength of the signal reflected from the probing end gradually decreased about 4 dB in 100 thermal cycles, but there was no further deterioration of the signal observed after 100 cycles up to 180 cycles. The test was terminated in 180 cycles. In the test of the probe with the platinum top electrode from room temperature up to 500 °C, the signal strength gradually decreased with -0.05 dB/cycle in 140 cycles. This may be due to the following reasons: partial detachment of the electrode–film interface because of difference of thermal expansion coefficients of the materials; improper contact of an electrical cable onto the top electrode; and/or deterioration of piezoelectricity of the BIT/PZT film because of minor depoling at elevated temperatures.

III. ULTRASONIC THICKNESS MEASUREMENT OF A STEEL PLATE IN MOLTEN ZINC AT 450 °C

Galvanization is one of common techniques to prevent corrosion of steel components. The steel parts are dipped into molten zinc (Zn) at temperatures 450 °C–460 °C for coating. The molten Zn is contained in a steel kettle with a size of, for example, $6 \times 3 \times 1$ m. There is a concern that the kettle will corrode from the inside and might cause disastrous failure in which the dangerous and costly spillage of the molten Zn happens. Ideally, the kettles are heated uniformly and there is a gradual loss of the kettle wall thickness as an iron–Zn layer forms on the inside of the kettle. However, nonuniform heating of the burners and the build-up of dross at the bottom of the kettle can lead to accelerated corrosion. Therefore, the kettle is required to be examined periodically to ensure its safe operation. Previously, an electromagnetic acoustic transducer (EMAT) was used to show the feasibility to measure the wall thickness at the external side of the kettle wall [19]. However, a mechanical raster scanning of the EMAT to perform the wall thickness profile could be difficult since furnaces are installed near the external wall of the kettle.

In our previous work, we performed ultrasonic measurements and imaging in a molten Zn bath using long buffer rods [20]. However, the UT was outside of the molten Zn, and the UT was cooled by compressed air. Here, an immersion HT probe described above with a compact size as an alternative approach was used. The advantage is that this probe of light weight can be easily scanned inside the molten Zn, which acts as a liquid couplant between the probing end and the inner kettle wall to obtain the wall thickness profile.

Fig. 4 shows a schematic diagram of the ultrasonic thickness measurement in molten Zn using the pulse-echo technique. The purity of the Zn used was 99.5%. The reported longitudinal wave velocity and the density of the molten Zn at 420 °C are 2790 m/s and 6.76 g/cm^3 , respectively [21]. The HT probe shown in Fig. 2 was completely immersed into the molten Zn at 450 °C together with a steel sample of a thickness of 24.5 mm. The distance between the probing end and the front surface of the sample was about 17 mm. The probe radiated and received pulsed ultrasound with a pulser–receiver. The Zn contained in a stainless steel (SS) crucible was heated by the furnace. A temperature controller controlled temperature of the molten

Fig. 4. Schematic diagram of ultrasonic thickness measurement of a 25.4-mm-thick steel plate in molten Zn at 450 °C.

Fig. 5. Ultrasonic signals for thickness measurement in molten Zn at 450 °C.

Zn using temperature values measured by a thermocouple immersed in the molten Zn. Ultrasonic signals were recorded using a data acquisition board with a resolution of 12 bits and a sampling rate of 100 MHz.

The ultrasonic thickness measurement in the molten Zn is given in Fig. 5. Two echoes, indicated by L_{2F} and L_{2B} , reflected from the front and back surfaces of the sample, respectively, were clearly observed. Provided that the longitudinal ultrasonic velocity (v) in the steel at 450 °C is known experimentally or from the literature, the thickness (H) of the sample can be calculated using the measured time delay difference (T , indicated in Fig. 5) between the L_{2F} and L_{2B} by $H = v \times T/2$. This steel probe was immersed in the molten Zn for many hours, and the

Fig. 6. Focused HT probe with an ultrasonic lens.

Fig. 7. Ultrasonic imaging setup.

probing end surface was only slightly corroded after even 9 h. It means that the surface remained acceptably flat. In fact, when this probe was placed facing the SS container, the wall thickness of the SS container could be measured as well.

IV. ULTRASONIC IMAGING IN SILICONE OIL AT 200 °C

In order to achieve high spatial resolution, the probing end of the steel buffer rod of the HT probe shown in Fig. 1 was machined into a semispherical concave surface as an ultrasonic lens, which focuses ultrasound in liquids, as shown in Fig. 6. The radius of curvature and the aperture diameter of the lens were 11.1 and 14.9 mm, respectively. A schematic diagram of the ultrasonic imaging set up in water at room temperature or in silicone oil at 200 °C is shown in Fig. 7. The focused HT probe with a holder was mounted on a manual vertical-translation stage (Z-stage) to adjust the distance between the probing end and the sample. In addition, the probe was transferred horizontally using an XY-stage driven by stepping-motors. A sample for imaging was fixed at the bottom of the crucible by a sample

Fig. 8. Optical photograph of U.S. one-cent coin used for imaging experiments. The tail side had its figures as an imaging side while the head side was polished to be flat.

holder. The data acquisition system was the same as the one used in Section III.

Fig. 8 shows an optical photograph of a sample used for imaging experiments, which was an American one-cent coin. The head side of the coin was polished to a flat surface while the tail side was left as it was for imaging. The diameter and thickness of the sample were 19.1 and 1.1 mm, respectively. The line thickness of the smallest characters (E·PLURIBUS·UNUM·) on the coin was about 0.2 mm.

Fig. 9(a) shows the signals obtained when the front surface of the sample (figure side) was located at the focus of the lens in water at room temperature. The first (L^1) and second (L^2) round trip echoes of longitudinal waves in the rod were observed at the time delay of about 16 μ s and 32 μ s, respectively. The L_{2F} was a signal reflected from the front surface of the sample at the focus and used for constructing the surface images. Besides those signals, spurious signals caused by the semispherical surface of the lens can be seen. This semispherical surface causes different ultrasonic wave path lengths, thus inducing spurious signals arrived at different time delays. If such spurious signals interfere with the desired signals from the sample, it may result in deterioration of imaging quality. The L_{2F} shown in Fig. 9(a) had enough SNR for constructing ultrasonic images in water at room temperature.

The signals obtained in silicone oil at 200 °C when the front surface of the sample was located at the focus of the lens are presented in Fig. 9(b). The L^3 observed at 48 μ s was the third round-trip echo in the rod. The L_{2F} signals indicated in Fig. 9(b) was weak and about 20 dB smaller than that in water at room temperature under the same measurement conditions of electric energy applied to the UT and electronic amplification of the signals. This is due to the following reasons: The ultrasonic attenuation in silicone oil at 200 °C is significant; and the acoustic impedance mismatching between the steel rod and silicone oil (200 °C) is larger than that between the steel rod and water (24 °C), resulting in less ultrasonic energy transmitted from the rod to silicone oil than to water.

In addition, the spurious signals in silicone oil at 200 °C shown in Fig. 9(b) were much larger than those in water shown in Fig. 9(a) because of the 20-dB larger electronic amplification

Fig. 9. Reflected signals from the probe and the sample (a) in water at room temperature and (b) in silicone oil at 200 °C.

of the signals than in water, which reduce the SNR of the L_{2F} . However, it is noted that amplitude and time delay of these spurious signals are fixed at a constant temperature because they are mainly the echoes reflected inside the rod. Therefore, such spurious signals can be eliminated significantly by subtracting background signals, measured without the sample, from the signals measured with the sample by digital signal processing techniques. In the following imaging experiments, the data after the subtraction are presented. It is noted that the attenuation of ultrasound in the steel rod at 200 °C is negligible because the signal strength of the L^1 at 450 °C was only 1.5 dB smaller than that at 22 °C as shown in Fig. 3.

A. Surface Image

We evaluate the focusing ability of ultrasound in water at room temperature and in silicone oil at 200 °C using this focused HT probe at an ultrasonic frequency f of 8 MHz in order to estimate spatial resolution for imaging in these liquids. The lateral resolution dr and focusing depth dz are approximately calculated using the following equations [22]:

$$dr \cong 1.02\lambda \cdot \frac{F}{D} \quad (1)$$

$$dz \cong 7.1\lambda \cdot \left(\frac{F}{D}\right)^2 \quad (2)$$

TABLE I
COMPARISON OF FOCUSING ABILITY OF ULTRASONIC
FOCUSED PROBE, SHOWN IN FIG. 6, IN WATER AT 23 °C AND
SILICONE OIL AT 200 °C. dr : LATERAL RESOLUTION, dz : FOCUSING DEPTH

	In water at 23°C	In silicone oil at 200°C
dr	0.19 mm	0.065 mm
dz	1.31 mm	0.38 mm

where λ is the wavelength of the longitudinal waves in the liquids, F is the focal length, and D is the aperture diameter of the lens. The λ and F are calculated by $\lambda = v_{\text{liquid}}/f$ and $F = R/(1 - v_{\text{liquid}}/v_{\text{steel}})$, respectively, where v_{liquid} and v_{steel} are longitudinal wave velocity in liquids and steel, respectively, and R is a curvature radius of the lens. The calculated results are summarized in Table I. The v_{liquid} for water at 23 °C is 1491 m/s [23], and the v_{steel} at 23 °C and 200 °C are 5923 and 5843 m/s [24], respectively. The v_{liquid} for silicone oil at 200 °C was estimated 612 m/s experimentally using the time delay of the signals reflected from the sample surface at the focus of the lens.

The dr in water (0.19 mm) is almost comparable to the smallest line thickness (0.2 mm) of the characters on the sample shown in Fig. 8. Due to the lower longitudinal wave velocity in silicone oil at 200 °C (612 m/s) than in water at 23 °C (1491 m/s), the higher lateral resolution ($dr = 0.065$ mm) is expected in silicone oil. However, the acoustic impedance ($= \rho v$) of the silicone oil at 200 °C is about 60% smaller than that of water, resulting in less transmission of ultrasonic energy from the steel rod to silicone oil than to water as mentioned previously, where ρ is the density of the liquids that are almost the same between silicone oil and water. This causes the lower SNR of the desired signals in silicone oil than in water.

The imaging experiment was first carried out in water at room temperature, and then that in silicone oil (200 Fluid-100 cst, Dow Corning, Midland, MI) at 200 °C using the same probe and sample. The sample shown in Fig. 8 was placed with the tail side (figure side) upward so that the figures were on the front surface and the back surface was flat. Fig. 10(a) and (b) shows the measured signals (L_{2F}) reflected from the sample in water at 24 °C and in silicone oil at 200 °C, respectively, when the front surface of the sample was placed at the focus of the lens. It is noted that subtraction of the background spurious noises had been conducted on the data, shown in Fig. 10, by the digital signal processing technique as mentioned previously. The time delay of the L_{2F} in silicone oil at 200 °C was about 20 μ s greater than that in water at 24 °C since ultrasonic velocity in silicone oil (612 m/s) is smaller than that in water (1491 m/s).

Fig. 11(a) and (b) shows ultrasonic images obtained in water at room temperature and in silicone oil at 200 °C, respectively, using the L_{2F} signals shown in Fig. 10. The scan step was 100 μ m for XY raster scanning of the focused HT probe. The left and right figures were constructed from the amplitude and time delay of the L_{2F} , respectively. The darker color represents the larger amplitude for amplitude images and the longer time delay for time delay images. The images of the figures including the characters having 0.2-mm line thickness on the tail side of the one-cent coin were clearly observed. Slight deformation of

Fig. 10. Ultrasonic signals obtained when a front surface of the sample is at the focus of the lens (a) in water at room temperature and (b) in silicone oil at 200 °C.

Fig. 12. Ultrasonic signals obtained when a back surface of the sample was at the focus of the lens (a) in water at room temperature and (b) in silicone oil at 200 °C.

Fig. 11. Ultrasonic images of front surface of the sample measured (a) in water at room temperature and (b) in silicone oil at 200 °C.

Fig. 13. Ultrasonic images of back surface of the sample measured (a) in water at room temperature and (b) in silicone oil at 200 °C.

the images was caused by movement errors of the *XY*-stage used for raster scanning.

B. Subsurface Image

In order to investigate the performance of the focused HT probe for inspection of defects inside materials, experiments for subsurface imaging were carried out. The same sample used for the surface image was placed at the bottom of the crucible with the flat side upward so that the front surface was flat and the figure was on the back surface. Fig. 12(a) and (b) shows the measured signals reflected from the sample in water at 24 °C

and in silicone oil at 200 °C, respectively, when the back surface of the sample was placed at the focus of the lens. In addition to the L_{2F} reflected from the front surface of the sample, one can see the signals (L_{2B} , L_{4B}) reflected from the back surface of the sample in water and in silicone oil, where the L_{2B} and L_{4B} indicate the second and third round-trip signals, respectively, in the sample.

Fig. 13(a) and (b) shows ultrasonic images of the back surface (subsurface) of the sample obtained in water at 24 °C and in silicone oil at 200 °C, respectively, using the L_{2B} signals shown in Fig. 12. The subsurface images were observed in silicone oil

[Fig. 13(b)] but not as clear as those in water [Fig. 13(a)] due to insufficient SNR of the L_{2B} in silicone oil at 200 °C. The imaging quality could be further improved by optimizing the lens design, shown in Fig. 6, such as radius of the curvature and aperture diameter of the lens. In addition, the SNR of the probe can be increased by fabricating cladding layers on periphery of the rod [25] and acoustic impedance matching layers at the lens–silicone oil interface [26], [27].

V. CONCLUSION

Thick-film (90 μm) piezoelectric BIT/PZT high-temperature immersion ultrasonic probes have been successfully fabricated by a sol–gel-spray technique. The operating frequency was between 5 and 20 MHz, controlled by the BIT/PZT film thickness. The operating temperature reached up to 500 °C, and no cooling system was required. The ultrasonic probe developed and used in the experiments had a center frequency of 8 MHz and the 6-dB bandwidth of 4 MHz at 450 °C. The signal strength of the echo reflected from the flat probing end at 450 °C was 1.5 dB smaller than that at 22 °C at 8 MHz. The SNR of more than 40 dB was obtained both at 22 °C and 450 °C. Such ultrasonic performance is sufficient for thickness measurements in liquids at elevated temperatures. With the sol–gel-spray technique, the BIT/PZT films can be easily manufactured at desired locations even on large and/or heavy metal substrates having a flat or curved surface for which other UT fabrication methods, such as spin coating, bonding, vacuum sputtering, and chemical-vapor-deposition techniques, are often difficult to apply. Currently, we are further developing a local heating technique using a portable heater in order to eliminate a furnace used for the thermal treatments in the process, which enable on-site fabrications of these UTs.

The ultrasonic measurement of the thickness of a steel plate with the probe fully immersed in the molten Zn at 450 °C was demonstrated using ultrasonic plane waves. For imaging purposes, the probing end of the steel buffer rod was machined into a semispherical concave shape to form the ultrasonic lens and achieve the high spatial resolution with focused ultrasound in liquids. Ultrasonic surface and subsurface imaging using a mechanical raster scan of the probe in silicone oil at 200 °C were successfully demonstrated. The imaging quality can be improved by optimizing the lens design, fabricating cladding layers on the periphery of the rod [25] and forming acoustic impedance matching layers at the lens–silicone oil interface [26], [27]. All of these steps can increase the SNR of the probe. These HT probes may be used to measure the ultrasonic reflection coefficients, velocities, attenuations, and elastic properties of materials immersed in liquids at elevated temperatures. Further possible applications could be ultrasonic imaging in nuclear reactors [4], [5], [12], inspection of reactor pressure tubes [15], thickness measurements at elevated temperatures [7], [19], etc.

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