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High-temperature integrated and flexible ultrasonic transducers for nondestructive testing

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ABSTRACT

Integrated ultrasonic transducers (IUTs) and flexible ultrasonic transducers (FUTs) are presented for nondestructive testing at high temperatures. These transducers are made of sol-gel-sprayed piezoelectric thick ($>40\ \mu\text{m}$) ceramic films. The ceramic materials are lead-zirconate-titanate, bismuth titanate and lithium niobate which are for thickness measurements up to 150, 400 and 800 °C, respectively. The IUT can also be deposited onto one end of a long ultrasonic delay line to perform nondestructive testing at the other end at even higher temperatures. FUTs made of bismuth titanate films onto thin stainless steel foils are also used for thickness measurements at 300 °C with a high-temperature couplant sandwiched between the FUT and a steel substrate. All experiments at high temperatures were performed in pulse-echo mode and ultrasonic echoes with signal-to-noise ratios above 20 dB were obtained. The center operation frequencies of both IUTs and FUTs range from 4.4 to 10.7 MHz.

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1. Introduction

Ultrasonic techniques employing piezoelectric ultrasonic transducers (UTs) have been widely used to perform nondestructive testing (NDT) on metallic structures such as airframes, engines, pipes, nuclear power plant structural parts, etc., because of the subsurface inspection capability, simplicity and cost-effectiveness [1–3]. Many of these applications such as corrosion, erosion, defect detection, etc., occur at high temperature (HT). Therefore, there is a demand of high-temperature ultrasonic transducers (HTUTs) [4–7].

The limitations of the current HTUTs are (1) complicated to be used for curved surfaces, (2) difficult to be applied in pulse-echo mode due to noises caused by imperfect damping in backing materials at HT and (3) not efficient at temperatures higher than 400 °C. In this investigation, sol-gel-sprayed thick ($>40\ \mu\text{m}$) piezoelectric ceramic films as HTUTs [8–10] are presented. Piezoelectric powders to be used are lead-zirconate-titanate (PZT), bismuth titanate (BIT) and lithium niobate (LiNbO_3) which have Curie temperatures of 350, 675 and 1210 °C, respectively. In one type these films are directly deposited onto the parts to be tested, and called integrated UTs (IUTs). Such IUTs do not require ultrasonic couplant. In another type these films are coated onto thin metal foils such as 38- μm -thick stainless steel (SS)

membrane and named as flexible UT (FUT) [11]. FUTs can be made off-line and require ultrasonic couplant to perform NDT on parts or structures.

Corrosion is a general safety concern for metallic parts and structures. In particular, stress corrosion cracking, corrosion pitting and exfoliation corrosion are commonly found in aircraft structures [12,13]. There is a critical need to perform in-situ quantitative thickness measurements to determine the degree of corrosion and provide correlation to component's residual fatigue life. The objective of this study is to develop HT IUTs and FUTs using the above-mentioned piezoelectric ceramics. These IUTs or FUTs will need to have good ultrasonic performance in pulse-echo mode which only requires one side access so that high thickness measurement accuracy can be obtained at temperatures up to 800 °C.

Another objective is to fabricate HT IUT onto one end of an ultrasonic delay line [14–16], and use the probing end (opposite to the IUT end) to perform at temperatures which can be higher than the maximum temperature of the HT IUT (e.g. $>800\ ^\circ\text{C}$). In certain situations parts or structures to be tested cannot be exposed to HTs generally used during certain fabrication procedures of the IUT. In such cases the HT FUTs can be fabricated off-line and used with a HT ultrasonic couplant between the FUT and the object to be tested. The evaluation of the ultrasonic strength of all IUTs and FUTs in this investigation will be based on a commercially available pulser-receiver device EPOCH (model LT) which has receiver gain up to 100 dB. This handheld device is commonly used in the NDT industry. The electrical contacts during all measurements were obtained using a spring-loaded two-pin probe.

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2. Fabrication and ultrasonic performance of IUTs

The sol-gel-based sensor fabrication process consists of six main steps [8–10]: (1) preparing high dielectric constant PZT solution, (2) ball milling of piezoelectric PZT, BIT or LiNbO_3 powders in PZT solution to submicron size, (3) sensor spraying using slurries from steps (1) and (2) to produce a film of thickness between 5 and 20 μm , (4) heat treating to produce a solid composite (PZT/PZT, BIT/PZT or LiNbO_3 /PZT) thick film, (5) electrical poling to obtain piezoelectricity, and (6) electrode painting or spraying for electrical connections. Steps (3) and (4) are used multiple times to produce optimal film thickness for specified ultrasonic operating frequencies. The heat treatments in step (4) are different for different ceramic powders. Colloidal silver, silver paste or platinum pastes were used to fabricate top electrodes. In this study, longitudinal (L) ultrasonic wave transducers are described. The relative dielectric constant is calculated using the measured capacitance by an impedance analyzer.

2.1. IUT made of PZT/PZT composite film

Fig. 1a shows an IUT made of 90- μm -thick PZT/PZT composite film and deposited onto a 12.7-mm-thick steel plate and measured by a handheld EPOCH LT pulser-receiver at 156 °C. The highest heat-treatment temperature for this sample in the furnace was 650 °C. The diameter of the silver paste top electrode of this IUT is 6.0 mm. The measured ultrasonic data in pulse-echo mode at 150 °C is presented in Fig. 1b, where L^n is the n th round trip L echo through the plate thickness. The signal-to-noise ratio (SNR) of the L^1 echo at 150 °C is 37 dB. In the measurement at 156 °C shown in Fig. 1a, 9.9 dB gain out of the available 100 dB receiver gain was used. This result indicates that this L wave IUT is very efficient. At 156 °C the center frequency and the 6 dB bandwidth are 8.0 and 7.8 MHz, respectively. It is noted that PZT/PZT IUT can function up to at least 200 °C. Three hundred and seventy five thermal cycles of such IUTs have been carried out. Each thermal cycle consisted of 5–10 min heating from room temperature to 150 °C, 30 min remaining at 150 °C and 10–30 min cooling from 150 °C to room temperature. There was no any deterioration of the ultrasonic performance after these cycles. The relative dielectric constant of PZT/PZT film is around 320.

2.2. IUT made of BIT/PZT composite film

An IUT made of 80- μm -thick BIT/PZT composite film and deposited onto a 12.7-mm-thick steel plate which is the same as the one used in Section 2.1 and measured by the EPOCH device at

400 °C. The highest heat-treatment temperature for this sample in the furnace was 650 °C. The dimension of the top rectangular silver paste electrode of this IUT is 8.0 mm by 8.0 mm. At 400 °C 47.4 dB out of the available 100 dB receiver gain was used the L^1 echo reflected from the end of the substrate. The SNR of the L^1 echo at 400 °C is 31 dB. The measured ultrasonic data at 400 °C in pulse-echo mode after passing through a high pass filter is presented in Fig. 2. At 400 °C the center frequency and the 6 dB bandwidth are 5.5 and 4.6 MHz, respectively. It is noted that BIT/PZT IUT can function up to at least 500 °C. 375 thermal cycles of such IUTs have been carried out. Each thermal cycle consisted of 15 min heating from room temperature to 400 °C, 30 min remaining at 400 °C and 20–45 min cooling from 400 °C to room temperature. There was no any deterioration of the ultrasonic performance. The relative dielectric constant of BIT/PZT film is around 80.

2.3. IUT made of LiNbO_3 /PZT composite film

An IUT was made of 125- μm -thick LiNbO_3 /PZT composite film and deposited onto a 25.4 mm diameter 26.3 mm long titanium rod for the measurement at elevated temperature using the EPOCH device. The titanium rod is chosen because of less oxidation at temperatures higher than 500 °C. The heat-treatment procedures used here are different from the one used in Sections 2.1 and 2.2. A special designed induction heating device was developed to perform the local heat treatment. The maximum temperature is higher than 700 °C. The dimensions of the square top platinum paste electrode of this IUT are 10 by 10 mm. The measured ultrasonic data at 800 °C in pulse-echo mode after passing through a high pass filter is shown in Fig. 3 and the center frequency and the 6 dB bandwidth were 4.4 and 3.2 MHz, respectively. At 800 °C 90.0 dB out of the available 100 dB receiver gain

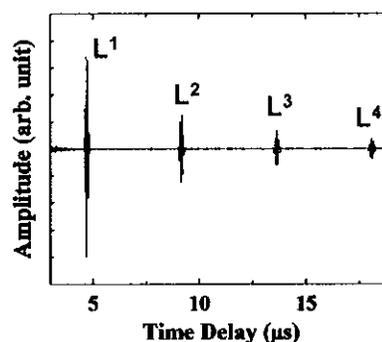


Fig. 2. Measurement data of an IUT made of BIT/PZT composite film in pulse-echo mode at 400 °C.

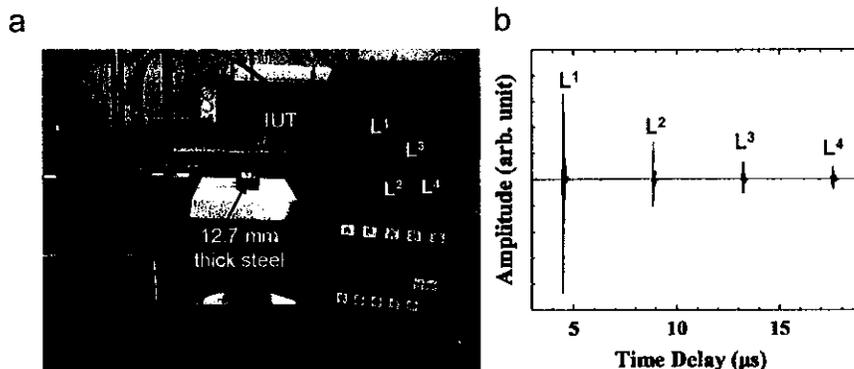


Fig. 1. (a) Measurement setup for an IUT made of PZT/PZT composite film at 156 °C using an EPOCH LT and (b) measured ultrasonic data in pulse-echo mode at 150 °C.

was used for producing the L^1 echo reflected from the end of the rod. The SNR of the L^1 echo at 800 °C is 20.3 dB. It is noted that $\text{LiNbO}_3/\text{PZT}$ IUT can function at even more than to 800 °C and its relative dielectric constant is ~ 2 . Five thermal cycles of such IUTs from room temperature to 800 °C were performed using a gas torch heating method. Each thermal cycle took a total duration of 40 min. There was no any deterioration of the ultrasonic performance after five cycles.

2.4. Performance comparison of IUTs made of PZT/PZT, BIT/PZT and $\text{LiNbO}_3/\text{PZT}$ composite films

Fig. 4a and b shows the signal strength variation and SNR, respectively, of PZT/PZT composite film on steel described in Section 2.1, BIT/PZT composite film on steel described in

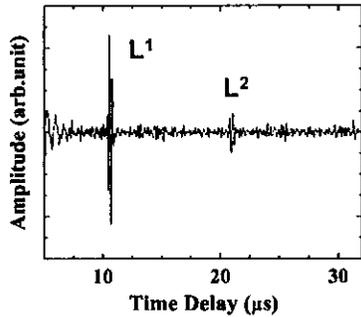


Fig. 3. Measurement data of an IUT made of $\text{LiNbO}_3/\text{PZT}$ composite film in pulse-echo mode at 800 °C.

Section 2.2 and $\text{LiNbO}_3/\text{PZT}$ composite film on titanium described in Section 2.3 in pulse-echo mode at different temperatures using an EPOCH LT. Fig. 4b indicates that IUTs made of PZT/PZT, BIT/PZT and $\text{LiNbO}_3/\text{PZT}$ composites films mentioned above have SNR more than 37, 31 and 20 dB, respectively at all temperatures displayed. Their signal strengths are sufficiently high for many NDT applications.

2.5. IUT deposited onto a clad buffer rod

As mentioned in the Introduction, another objective of this study was to deposit HT IUT onto one end of a long ultrasonic delay line (or buffer rod) [14–16], the probing end (opposite to the IUT end) can perform NDT at temperatures even higher than the maximum temperature of the HT IUT (e.g. >800 °C). Fig. 5a presents IUTs made of $\sim 110\text{-}\mu\text{m}$ -thick PZT/PZT composite film and deposited onto two clad steel buffer rods [15,16] measured by an EPOCH LT system. The temperature at the IUT end is 151 °C and that at the other rod end (probing end) is 182 °C. The heat-treatment procedures used here are also different from the one used in Sections 2.1 and 2.2. A specially designed induction heating device was developed to perform the local heat treatment and the maximum temperature was higher than 700 °C.

The clad steel buffer rod [15,16] consists of a steel core and a SS cladding made by thermal spray process. As shown in Fig. 5b the clad steel rod shown on the left has a core diameter of 12.7 mm and another shown on the right has a core diameter of 25.4 mm. Both rods have $\sim 1\text{-mm}$ -thick SS cladding and a length of 102 mm. The clad steel rod is chosen because of its high SNR, in particular, in pulse-echo

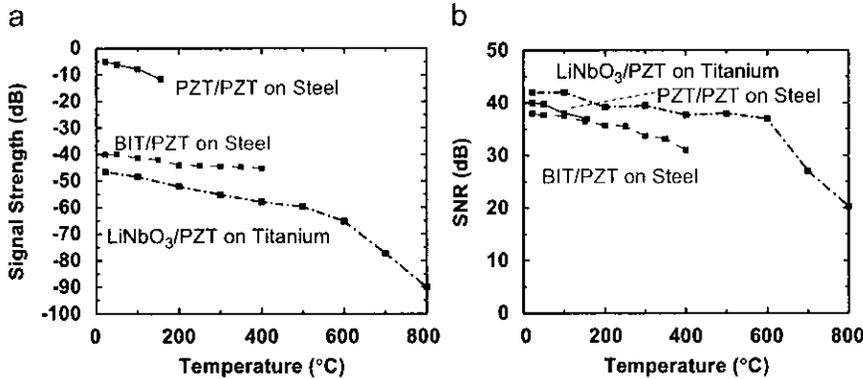


Fig. 4. (a) Signal strength variation and (b) SNR of PZT/PZT composite film on steel, BIT/PZT composite film on steel and $\text{LiNbO}_3/\text{PZT}$ film on titanium in pulse-echo mode at different temperatures using an EPOCH LT.

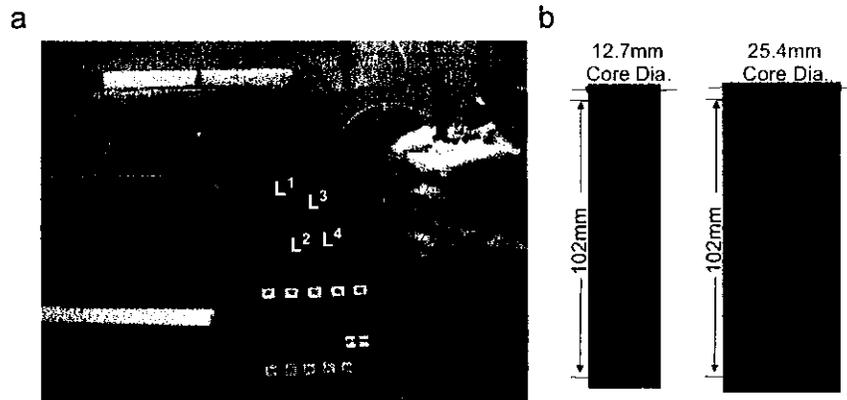


Fig. 5. Measurement setup for an IUT made of PZT/PZT composite film at room temperature using an EPOCH.

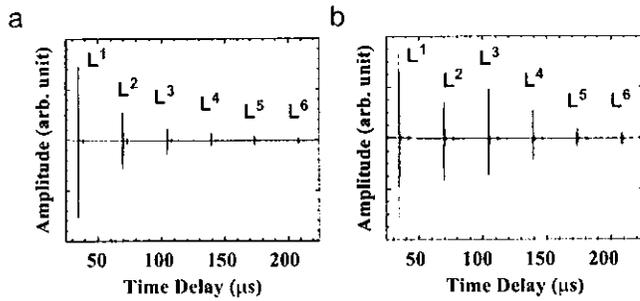


Fig. 6. Measured ultrasonic data of IUT at 150 °C in pulse-echo mode for (a) small and (b) large diameter clad steel rod shown in Fig. 5b.

mode which provides advantages for in-line ultrasonic monitoring of industrial material manufacturing such as polymer extrusion [17] and molten metal processes [16]. The diameter of the silver paste top electrode of the IUT is 6.5 and 7.0 mm, respectively, on the small and large diameter. When the IUT was at room temperature, 50, 100 and 150 °C, only 5.0, 5.5, 7.1 and 10.1 dB gain, respectively, out of the available 100 dB receiver gain were used for the small diameter clad steel rods for producing the same signal strength of the L^1 echo reflected from the end of the rod. L^n is the n th round trip L echo through the rod length. At the probing end (the rod end opposite to the IUT) the measured temperature was 182 °C which is 31 °C higher than 151 °C. It is noted that the length of the probe can be made much longer because of the high signal strength of the IUT, the temperature at the probing end can be much higher than 151 °C. For the large diameter clad buffer rod IUT at room temperature, 50, 100 and 150 °C 18.0, 18.1, 18.3 and 20.0 dB gains, respectively, were used. The relative dielectric constants of the IUT for small and large diameter clad rod are 290 and 190, respectively. The difference between the relative dielectric constants of the PZT/PZT deposited on the clad rods shown in Fig. 5b and that of the steel plate shown in Fig. 1b comes from the different heat treatment procedures. The measured ultrasonic data at 150 °C in pulse-echo mode for the small and large diameter rods shown in Fig. 5b are shown in Fig. 6a and b, respectively. At 150 °C the center frequency and the 6 dB bandwidth of the L^1 echo were 7.0 and 5.9 MHz, respectively for the small diameter rod and 6.8 and 3.7 MHz, respectively for the large diameter rod. The SNRs of the L^1 echoes for IUTs at 150 °C are 26 and 30 dB, respectively, for the small and large diameter clad rods. In Fig. 5b the reason of the amplitude of L^3 echo is larger than that of L^2 echo is due to the diffraction effect. Fig. 6a and b confirm that PZT/PZT IUTs have excellent performance at 150 °C. It is expected that these IUTs deposited onto these clad steel rods will perform the same during thermal cycles as the PZT/PZT film coated onto steel substrate as mentioned in Section 2.1.

3. Fabrication and ultrasonic performance of FUTs

The other type of sensor developed is the HT FUT which can be made off-line. Since in certain situations parts or structures for NDT cannot be exposed to high fabrication temperatures required for IUTs, then such HT FUTs may be used for HT NDT. In this situation, HT ultrasonic couplant must be used between the FUT and the sample to be tested. FUT made of PZT/PZT composite film has been reported previously [11]. Here, only the BIT/PZT composite film made onto SS foils is presented. The fabrication procedure is the same as those described for BIT/PZT ceramic powders in Section 2. Many FUTs using PVDF [18] and polymer composites [19,20] were reported; however, they may not be effectively used at temperatures higher than 150 °C. The FUT made of BIT/PZT film coated onto a 38- μ m-thick SS foil has been

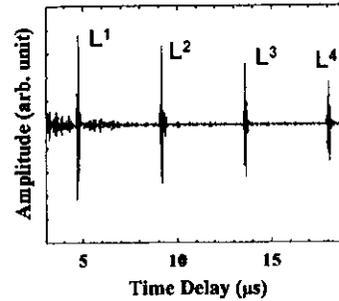


Fig. 7. Measurement data of an FUT in pulse-echo mode at 303 °C. This FUT was made of BIT/PZT composite film deposited onto a 38- μ m-thick SS foil and bonded onto a 12.7-mm-thick steel substrate.

examined after one thousand times bending test with a curvature of 25 mm diameter. There is no observable damage both in visual appearance and ultrasonic performance. Such FUT was bonded onto a 12.7-mm-thick steel substrate using a metallic adhesive (from Cotronics Corporation, Brooklyn, NY), cured at 300 °C for 2 h. Fig. 7a shows the measurement setup at 303 °C using the EPOCH device and the measured ultrasonic data after passing through a high pass filter is presented in Fig. 7b. The center frequency and the 6 dB bandwidth of the L^1 echo at 300 °C were 10.7 and 8.2 MHz, respectively. The SNR of the L^1 echo is 22 dB. The 303 °C test temperature is limited due to the high-temperature bonding material used. When the FUT was operated at 303 °C 69 dB gain \pm 1 dB out of the available 100 dB receiver gain were used. Since the bonding material is used as the HT ultrasonic couplant, one desires such bonding material which not only provides good ultrasonic coupling between the FUT and the surface of the sample to be tested at HTs, but also do not induce high noises. Fig. 7b demonstrates the capability of FUT for NDT at 303 °C. It is noted that such FUT itself can function at least up to 500 °C and its relative dielectric constant is 60. It is expected that these FUTs made of BIT/PZT film and deposited onto SS foil will perform the same during thermal cycles as the BIT/PZT film coated onto steel substrate mentioned in Section 2.1.

4. Thickness measurement accuracy estimation

As mentioned in the Introduction corrosion monitoring is an important NDT application. Therefore, an example of the thickness measurement accuracy at HT is presented here using the two clad steel rods shown in Fig. 6a and b. The temperature at the IUT side is 151 °C and that at the other end of the rod is 182 °C.

$$\sigma(\Delta t - \Delta t') \geq \sqrt{\frac{3}{2f_0^3 \pi^2 T (B^3 + 12B)} \left(\frac{1}{\rho^2} \left(1 + \frac{1}{\text{SNR}_1^2} \right) \left(1 + \frac{1}{\text{SNR}_2^2} \right) - 1 \right)} \quad (1)$$

Assuming these two clad rods are under a constant temperature of 150 °C Eq. (1) (Eq. (19) in Ref. [21]) is used here for the estimation of the measurement accuracy for the time delay and then length of the clad steel rod using IUTs, where f_0 is the center frequency, T the time window length for the selection of e.g. L^1 and L^2 echoes for the cross-correlation, B the fractional bandwidth of the signal (the ratio of the signal bandwidth over f_0), ρ the correlation coefficient used in cross-correlation, SNR_1 and SNR_2 the SNR of the 1st echo (e.g. L^1 in Fig. 6a and b for small and large clad rod) and 2nd echo (e.g. L^2 in Fig. 6a and b for small and large clad rod), respectively, $\sigma(\Delta t - \Delta t')$ the standard deviation of the measured time delay (Δt the true time delay; $\Delta t'$ the estimated time delay), and V_L (5884 m/s) the measured L velocity in the steel core. Since a sampling rate of 100 MHz is used in the

Table 1
Parameters for Eq. (1).

Parameters	Small diam. buffer rod at 150 °C	Large diam. buffer rod at 150 °C
f_0	7 MHz	6.8 MHz
T	0.6 μ s	0.6 μ s
B	5.9/7	3.7/6.8
ρ	0.68	0.7
SNR_1	26 dB	30 dB
SNR_2	19 dB	25 dB
$\sigma(\Delta t - \Delta t')$	9.0 ns	11.7 ns
Digitization resolution (100 MHz) together with cross-correlation method	2 ns	2 ns
V_L	5884 m/s	5884 m/s
Thickness accuracy	32 μ m	40 μ m

experiment, with the use of cross-correlation method including interpolation [22] the time measurement error which may be additionally introduced is estimated to be 2 ns. The estimated rod length measurement accuracies (assuming under the constant temperature of 150 °C) for small and large clad steel rods with a length of 102 mm using the above parameters given in Table 1 are 32 and 40 μ m, respectively. This evaluation demonstrates that IUTs having broad bandwidth and high SNR can be used for accurate corrosion evaluation which may be in certain aspect considered as thickness reduction.

5. Conclusions

Integrated ultrasonic transducers (IUTs) made of PZT/PZT, BIT/PZT and LiNbO₃/PZT piezoelectric ceramic composite films are presented for thickness measurement at temperatures up to 150 °C, 400 °C and 800 °C, respectively. These films ranging from 40 to 125 μ m were made by a sol-gel-sprayed technique. No couplant is required for IUTs to carry out NDT. IUTs made of PZT/PZT composite film on steel plate, BIT/PZT composite film on steel plate and LiNbO₃/PZT composites film on titanium rod have SNR more than 37, 31 and 20 dB, respectively at temperature up to 150, 400 and 800 °C. The center frequencies of these IUTs ranged from 4.4 to 8.0 MHz. Their signal strengths evaluated by a hand held EPOCH LT pulser/receiver equipped with 100 dB receiver gain showed that these IUTs have sufficiently high signal strength for many NDT applications.

PZT/PZT composite films were also deposited onto the end of one 12.7 mm diameter and 102 mm long and that of one 25.4 mm diameter and 102 mm long clad steel rod ultrasonic delay lines to perform ultrasonic measurement at 150 °C. At 150 °C the center frequencies and the 6 dB bandwidths of the first round-trip echoes reflected from these two rod ends were 7.0 and 5.9 MHz, respectively for the small diameter rod and 6.8 and 3.7 MHz, respectively for the large diameter rod. The signal strengths and SNRs of these echoes at 150 °C were 10 and 20 dB, and 26 and 30 dB, respectively, for the small and large diameter clad rods. At 150 °C only 10 dB of the available 100 dB receiver gain was used. Because of such high signal strength and high SNR of the first reflected echo from the rod end, if the length of the clad steel rod is made much longer, the temperature at the probing end can be much higher than 150 °C.

In certain situations parts or structures for NDT cannot be exposed to HT fabrication procedures of the IUT, then HT FUT fabricated off-line can be used for HT NDT except that HT ultrasonic couplant must be used between the FUT and the sample to be tested. A FUT made of BIT/PZT film coated onto a

38- μ m-thick SS foil was bonded to a steel plate using a HT metallic adhesive and ultrasonic measurement was performed up to 303 °C. The center frequency and the 6 dB bandwidth of the first round-trip echo reflected from the back of the steel plate at 300 °C were 10.7 and 8.2 MHz, respectively and its SNR was 22 dB.

Using Eq. (1) (Eq. (19) in Ref. [20]) the estimated rod length measurement accuracies (assuming under a constant temperature of 150 °C) for small and large clad steel rods shown in Fig. 4b with a length of 102 mm using the parameters given in Table 1 are 32 and 40 μ m, respectively. This evaluation demonstrates that the presented IUTs having broad bandwidth and high signal-to-noise ratio can be used for accurate corrosion evaluation which may be in certain aspect considered as thickness reduction.

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