
VERY HIGH TEMPERATURE ULTRASONIC TRANSDUCERS FOR *IN-SITU* AND INTERMITTENT APPLICATIONS

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An Ultran Technical Publication #TP-794

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ABSTRACT

For materials process monitoring and for their reliable performance at elevated temperatures, a suitable nondestructive analytical technique is highly desired. Although ultrasound offers excellent possibilities for the solution of critical industrial problems, the reliability and performance of transducers for their high temperature applications have generally been major hurdles in ultrasonic testing. This paper describes applications-oriented temperature-dependent observations from our recently developed high temperature transducer devices. While the new transducers have been successfully evaluated between $\sim -50^{\circ}\text{C}$ to $\sim 600^{\circ}\text{C}$, it is believed that with suitable modifications they can be applied in excess of $1,500^{\circ}\text{C}$ without extrinsic cooling mechanisms. It will be seen that these devices are not only characterized by thermo-acoustic stability and relatively short pulse widths, but also by high sensitivities even after prolonged exposure to high temperatures.

1. INTRODUCTION

In-situ quality and process control for intelligent processing of materials during various stages of their manufacture are critical for meeting the industry's insatiable demand for the Total Materials Quality (TMQ). Similarly, for Reliable Performance of In-service Components (RPIC), it is imperative to know their overall structure and condition. The significance and realization of nondestructive materials analysis have been discussed extensively by numerous authors during the last decade.^{1,2,3}

Recently considerable strides have also been made in the NonDestructive Characterization (NDC) of materials in the early stages of their manufacture.^{4,5} However, for TMQ and RPIC NDC at high temperatures is a pre-requisite. To this effect, electromagnetic acoustic transducers and eddy current method have been used for the inspection of steel tubes,⁶ and laser-induced ultrasound has been applied for the characterization of graphite epoxy composites.⁷ Although useful information can be derived by these methods, they are, nevertheless, either limited to certain materials composition, or too cumbersome for general purpose usage in high temperature characterization.

Obviously, these problems could be circumvented if ultrasonic transducers, emitting and receiving bulk waves, could be directly or indirectly placed on the hot surfaces of a test material. Commercial high temperature applications transducer devices are limited by the temperature resistance of materials surrounding and protecting the fragile piezoelectric material. For example, commonly used protective polyamid delay materials in front of the active piezoelectric element are not only limited to $\sim 250^{\circ}\text{C}$ continuous and $\sim 450^{\circ}\text{C}$ intermittent use, but from the acoustic standpoint they are even worse. Polyamids are characterized by extremely high frequency dependence of ultrasound attenuation, thereby limiting the usable transducer frequency to well below 2MHz at elevated temperatures. These materials are also

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very sensitive to velocity variations (time of flight fluctuations) as a function of temperature, thus decreasing the reliability of high temperature data.

Attempts have been made to reduce these problems by cooling the critical piezoelectric region of the transducer device and implanting internal or external water cooling coils. Devices based upon this concept have been produced with and without high temperature resistant and acoustically transparent metal and ceramic delay lines. Needless to say, such high temperature transducers are extremely cumbersome and bulky for routine use. On the other hand, by utilizing suitable high temperature brazing techniques, low frequency and undamped long pulse duration high temperature transducers have also been recently reported.⁸

It is well known that with the exception of specialized experimental studies, a piezoelectric element alone cannot be used to perform routine ultrasonic examinations at high temperatures. The fragile piezoelectric material must be fully protected and suitably treated for the production of reliable high temperature device. There are several functions that need to be considered for maximum performance and operation of high temperature transducer devices for NDC tasks. These are summarized in Table-I.

TABLE-I. Functions and NDC tasks required by high temperature ultrasonic transducer devices.

FUNCTION	HIGH TEMPERATURE YIELD	NDC TASKS AND THEIR SIGNIFICANCE
Thermo-mechanical stability of the device	No physical degeneration of the device	Long-term exposure to high temperatures. <i>In-situ</i> use for process monitoring.
Thermal fatigue resistance	No physical degeneration of the device	Continuous, but intermittent use. Corrosion, velocity, microstructure, etc.
Acoustic characteristics	Frequency range: <0.5 to >10MHz Bandwidth: =/>50% of bcf	High resolution of relatively thin sections. High reliability of data.
Thermo-acoustic stability	Minimum to no change in acoustic parameters, including time of flight variations as a function of temperature	Materials research. High reliability of data.
Temperature resistance	Optimum in two steps: Direct contact device: Continuous ~350°C Intermittent: >600°C Delayed contact device: Continuous: >600°C Intermittent: >1,000°C	Multiple.

2. DEVELOPMENT AND SALIENT FEATURES OF HIGH TEMPERATURE TRANSDUCER DEVICES

For nearly 20 years we at Ultrason have been acutely aware of the difficulties in producing reliable and ergonomic "true" high temperature ultrasonic transducer devices useful for materials processing and high temperature monitoring of properties, microstructure, corrosion, and other significant applications where temperatures in excess of 250°C are involved. After a considerable amount of "hit-and-trial," in 1987 we were successful in producing between <500KHz to ~10MHz direct contact devices for continuous operation up to 250°C.⁹ Besides

having thermo-mechanical stability, these devices typically generate short ultrasonic pulse widths, i.e., between two to three wavelengths, even at elevated temperatures. However, due to the limitations of high temperature mechanical loading and bonding materials, this design could not be operated in excess of 250°C continuous, and intermittently to ~400°C.

Rigorous efforts towards the production of reliable ultrasonic devices characterized by "the highest possible temperature resistance" in conjunction with "the shortest possible pulse widths" at high temperatures have generated novel design,¹⁰ that also meet the criteria stipulated in Table-I. Based upon the thermo-mechanical compression phenomenon, we have successfully contained the critical transducer assembly in a specially formulated compression chamber constructed from high temperature and acoustically compliant materials. By utilizing this concept, two transducer types have been produced.

- 2.1 **Direct contact type:** Shown in figures 1 and 2, this type of transducer can be used for continuous operation up to ~350°C, and intermittently up to ~500°C. Besides its use as a direct contact device, it can also be adapted to high temperature anglebeam and immersion techniques.

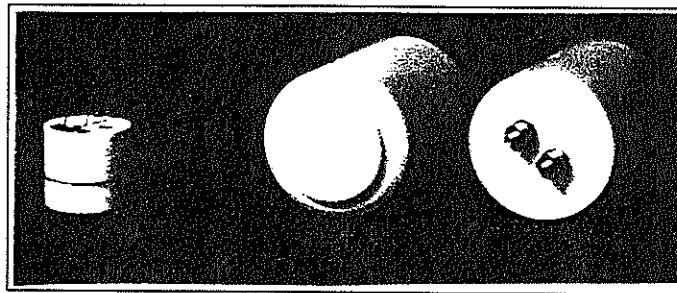


Fig. 1. Direct contact type high temperature transducers. Left hand: without protective TEFLON shield. Right hand: with protective TEFLON shield.

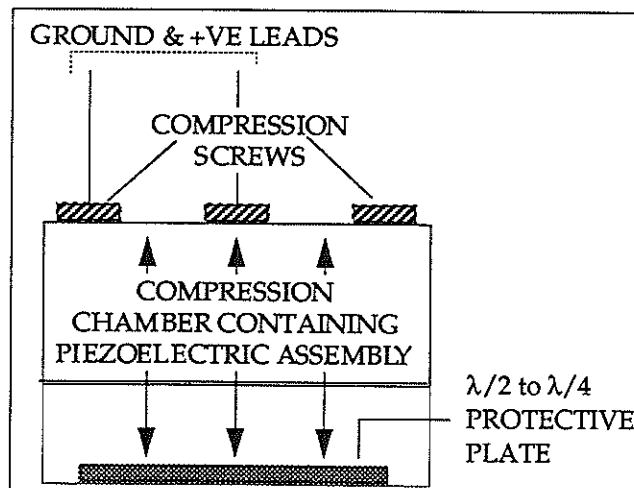


Fig. 2. Schematic of direct contact high temperature ultrasonic device.

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- 2.2 **Delayed contact type:** Shown in figures 3 and 4, this transducer type can be used in excess of 600°C continuously, provided the ambient temperature does not exceed 350°C. This design can also be suitably modified to operate >1,500°C.

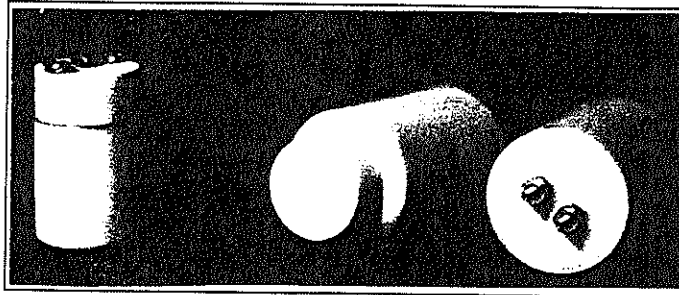


Fig. 3. Delayed contact type high temperature transducers. Left hand: without protective TEFLON shield. Right hand: with protective TEFLON shield.

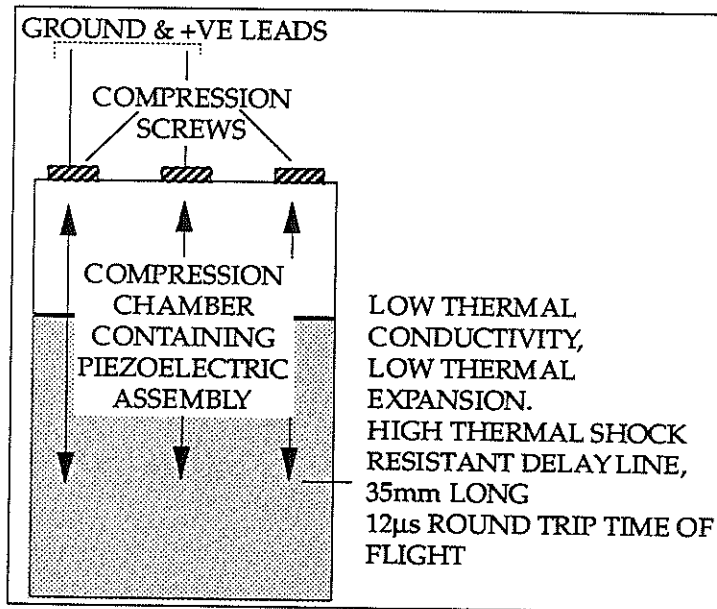


Fig. 4. Schematic of delayed contact high temperature ultrasonic device.

3. EXPERIMENTAL PROCEDURE

The observations reported in this paper were obtained by exciting the transducers with a ~15ns negative spike pulser. Depending upon the objective of a given experiment, the signals were amplified by a broadband 1KHz to 35MHz receiver, or directly by the amplifier of a broadband 500MHz Tektronix 7854 waveform calculator oscilloscope. By utilizing this mechanism, two sets of temperature dependent experiments were performed as following:

- 3.1 **Analysis of piezoelectric materials:** Commercial grade samples of lead zirconate - lead titanate, PZT ($t_c = 350^\circ\text{C}$), lead meta-niobate, PMN ($t_c = 550^\circ\text{C}$), and lithium niobate, LN ($t_c = 1,250^\circ\text{C}$) were used to study the effect of temperature on their sensitivity. All samples are 19mm diameter and approximately 2.5MHz resonant frequency. These

materials were coupled* to a 25mm thick carbon steel block placed on a hot plate, capable of generating temperature $>500^{\circ}\text{C}$. A schematic of this experimental setup is shown in Fig. 5.

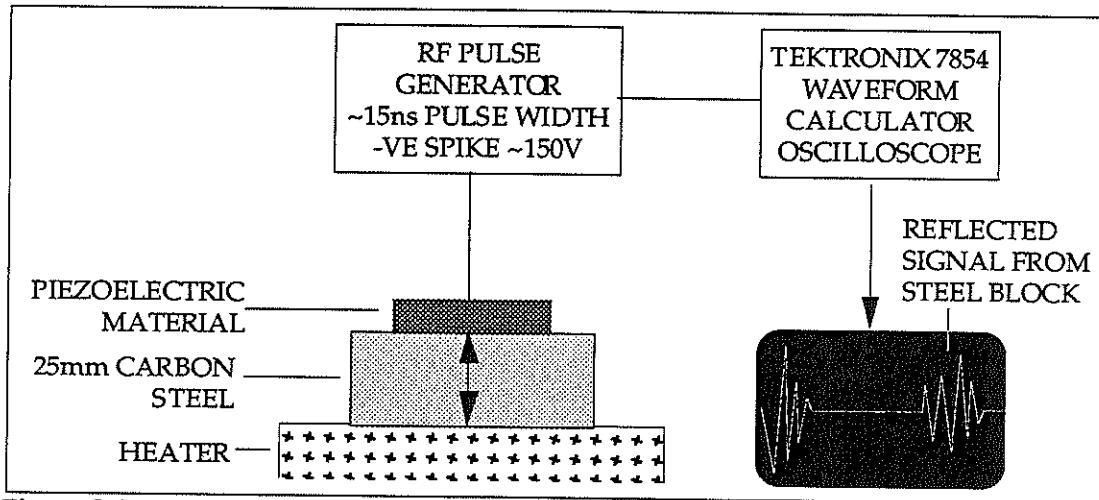


Fig. 5. Schematic of the experimental set up used for high temperature characterization of selected piezoelectric materials.

3.2 Analysis of high temperature transducer device: A delayed contact high temperature device, typically, 5MHz nominal frequency and 12.7mm active area diameter was analyzed according to the setup shown in Fig. 6.

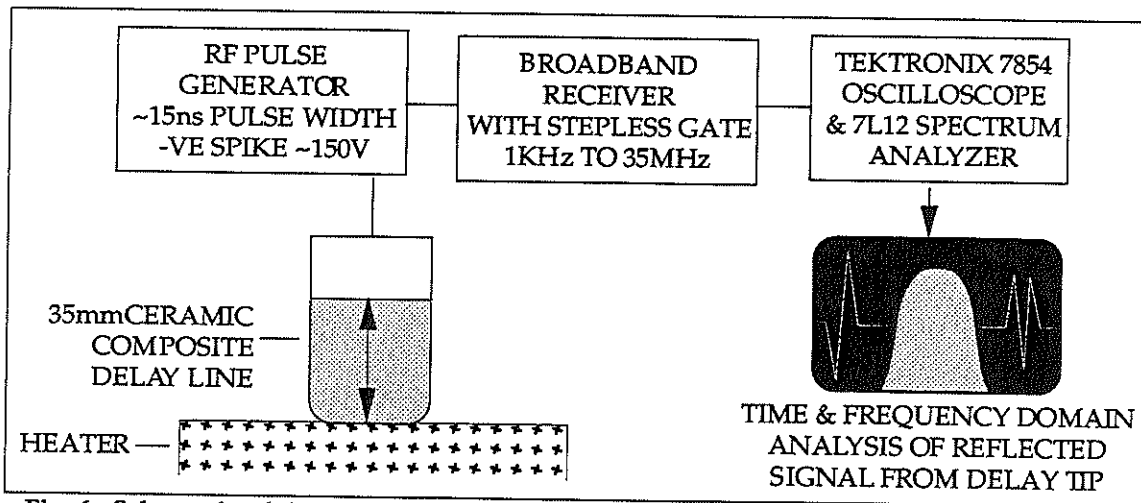


Fig. 6. Schematic of the experimental set up used for high temperature characterization of delayed contact transducer devices.

*High temperature couplant provided by Sonicoat, Japan.

4. OBSERVATIONS AND INFERENCES

4.1 **Examination of piezoelectric elements:** Before applying a high temperature suitable active piezoelectric material, it was necessary to determine the sensitivities of selected materials that are commercially available and are characterized by relatively high Curie points. To this effect, three piezoelectric material categories, represented by lead zirconate lead titanate ($t_c = 350^\circ\text{C}$), lead meta-niobate ($t_c = 550^\circ\text{C}$), and lithium niobate ($t_c = 1,250^\circ\text{C}$) were chosen. Besides the high Curie points, these materials are also characterized by significant acoustic, mechanical, and thermal properties of value from the standpoint of an NDC ultrasonic transducer device, as shown in Table-I. Sensitivities of these materials were determined by measuring the amplitudes of reflected signals from a 25mm thick carbon steel reference block, i.e.,

$$\text{Sensitivity (dB)} = -\text{Log } 20 A_x/A_0 \quad (1)$$

where A_x is the output amplitude of a reflected signal from a specified target and A_0 is the excitation voltage.

Observations from three piezoelectric materials are shown in Fig. 7. As expected, PMN and LN exhibit minimum sensitivity loss, i.e., less than ~~10%~~ ^{6 dB} from room temperature to $>500^\circ\text{C}$. However, PZT showed dramatic losses beyond $\sim 200^\circ\text{C}$. While both PMN and LN have favorable high temperature acoustic characteristics, the latter does not possess good mechanical stability when used in conjunction with mechanical dampening loads and front protective materials, as does the PMN. This appears to be particularly true when LN is used beyond a 2MHz frequency.

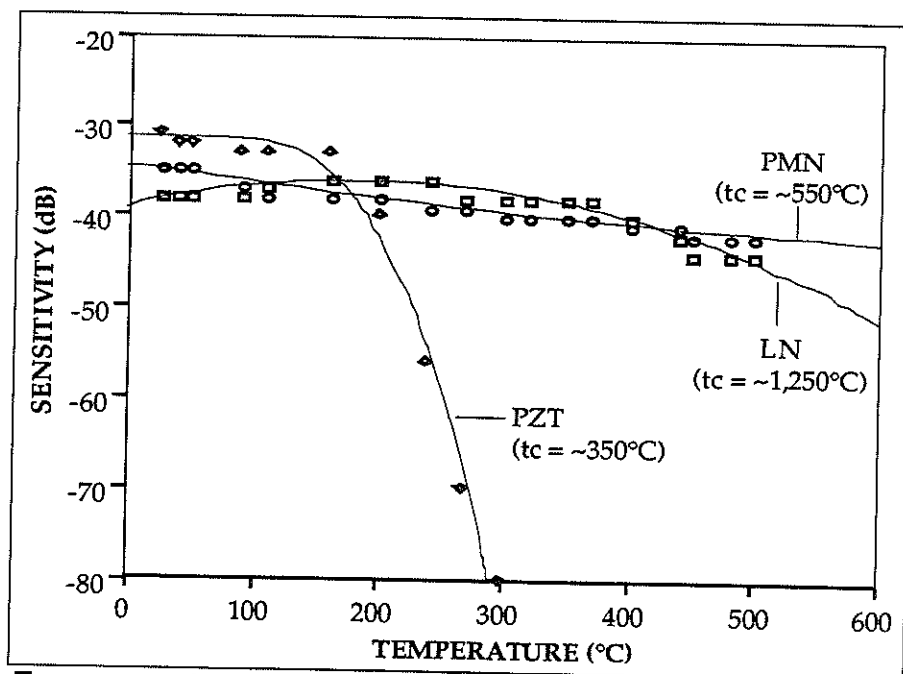


Fig. 7. Temperature dependence of sensitivity for selected piezoelectric materials. All materials are typically, 2.5MHz resonant frequency and 19mm active area diameters.

4.2 **Characterization of high temperature ultrasonic devices:** Several devices, typically between 3 to 5MHz nominal frequencies, 12.7mm active area diameter and 12 μ s round trip time of flight delay of a high temperature compliant and acoustically transparent ceramic composite, were analyzed as a function of temperature from \sim -50°C to 600°C. Analysis relative to sensitivity, time and frequency domain, time of flight variations of delay line, and aging were carried out, as reported in the subsequent sections.

4.2.1 **Sensitivity characterization:** Fig. 8 shows typical observations relative to sensitivity, determined per equation #1. It is very interesting to note that the sensitivity loss is approximately <15% within the range of temperature investigation. Furthermore, the trends of temperature-sensitivity curves for the device and for the PMN alone (Fig. 7) are very nearly the same. This observation proves that the compression chamber design does not produce ghost mechanisms which would otherwise result in severe losses in sensitivity as a function of temperature.

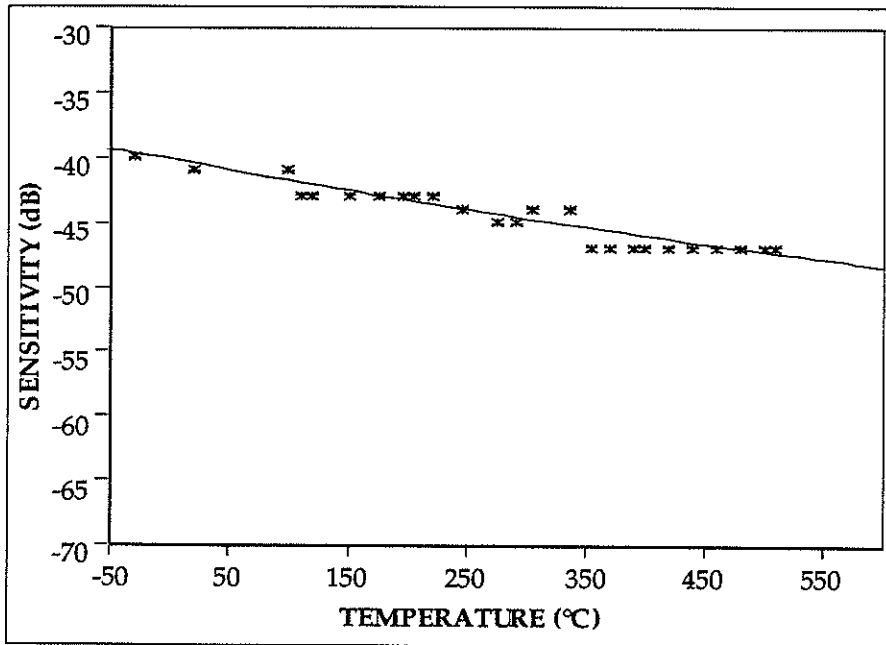


Fig. 8. Temperature dependence of sensitivity for a typically 5MHz, 12.7mm active area diameter and 12 μ s round trip time of flight delay line transducer.

4.2.2 **Acoustic characterization:** Figures 9 and 10, respectively, show time and frequency domain analysis at room temperature and at \sim 510°C for a typically 3.5MHz high temperature device. Captions of these figures give the details of the analyzed parameters. Table-II provides a comparison of acoustic and other salient observations at room and at \sim 600°C performance of a similar device, but 5.0MHz nominal frequency.

4.2.3 **Aging and thermal fatigue characterization:** It was observed that after less than 0.5hr at \sim 600°C, the sensitivity dropped to ~~25%~~ of the room temperature value.

by <10dB

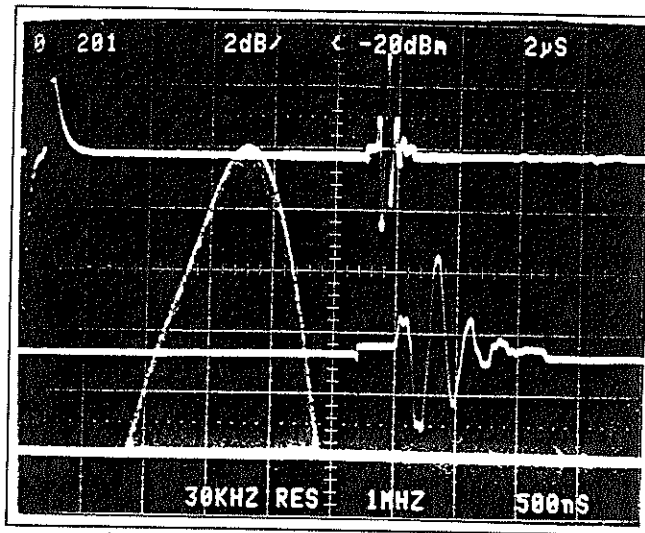


Fig. 9. Time and frequency domain analysis of a typically 3.5MHz nominal frequency, 12.7mm active area diameter, and 12µs round trip time of flight delay high temperature transducer at room temperature.

TOP TRACE: Horizontal Scale: 2µs/d; Vertical Scale: 20mV/d @ 50Ω. BOTTOM TRACE: Horizontal Scale: 500ns/d; Vertical Scale: 20mV/d @ 50Ω. FREQUENCY ENVELOPE: Horizontal Scale: 1MHz/d; Vertical Scale: 2dB/d

Measured parameters as a function of delay tip reflection: Bandwidth Center Frequency (bcf): 3.4MHz. Bandwidth at -6dB: 2.2MHz or ~65% of bcf. Pulse Width: <800ns. Sensitivity: -40dB

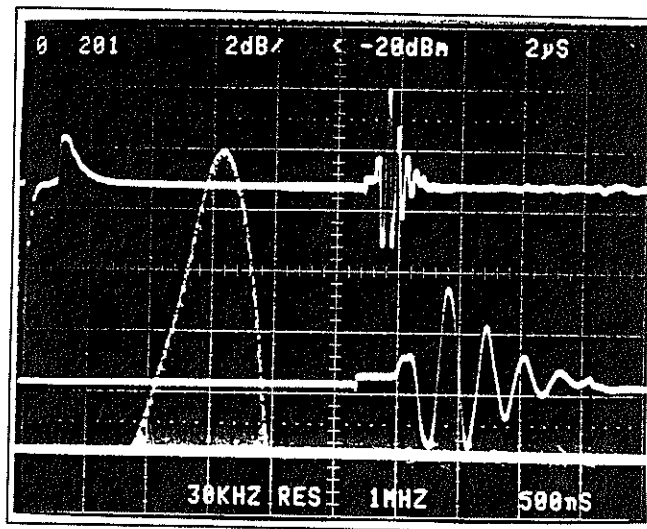


Fig. 10. Time and frequency domain analysis of a typically 3.5MHz nominal frequency, 12.7mm active area diameter, and 12µs round trip time of flight delay high temperature transducer at room temperature.

TOP TRACE: Horizontal Scale: 2µs/d; Vertical Scale: 20mV/d @ 50Ω. BOTTOM TRACE: Horizontal Scale: 500ns/d; Vertical Scale: 20mV/d @ 50Ω. FREQUENCY ENVELOPE: Horizontal Scale: 1MHz/d; Vertical Scale: 2dB/d. Measured parameters as a function of delay tip reflection: Bandwidth Center

Frequency (bcf): 3.1MHz. Bandwidth at -6dB: 1.4MHz or ~45% of bcf

Pulse Width: <1.2µs. Sensitivity: -44dB

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TABLE-II. Comparison of acoustic and other parameters at room and at ~600°C temperatures for a typical 5MHz, 12.7mm active area diameter and 12µs round trip time of flight delay line transducer.

MEASUREMENT TEMPERATURE (°C)	PULSE ¹ WIDTH (µs)	SIGNAL TO ² NOISE RATIO (dB)	SENSITIVITY ³ (dB)	BANDWIDTH ⁴ (%)	FREQUENCY LOSS	δ _{tof} ⁵ (%)	MEASUREMENT ⁶ RANGE WITHIN DELAY SPACE (µs)
20	0.7	>34	-40	>50%	---	---	<1 to ~12
~600	1.0	>30	-44	>40%	NONE	<1	~1 to ~12

1. Measured from trailing to leading edge of rf envelope from delay tip reflection.
2. Measured by correlating the highest noise signal between the first two delay reflections with the first delay reflection.
3. $-20 \text{ Log } A_x/A_0$, where A_x is the amplitude of delay tip reflection and A_0 is the amplitude of the excitation pulse.
4. Measured as %age of bandwidth center frequency at -6dB level.
5. Reported as %age increase in time of flight (tof) from 20°C to ~600°C.
6. Between the first two delay reflections, for example, in steel from ~2mm to >35mm. Measurements can also be made well beyond 2nd and 3rd reflections.

After this time no significant alteration in sensitivity or in other characteristics was observed for prolonged use of these devices. Several devices were examined for nearly 60 days with 8 hour operation each day without failure. Similarly, they were subjected to room temperature and 500°C temperatures several times daily with intervals of a few minutes with no apparent degradation of any device. As a precaution, however, we suggest that when temperatures higher than 400°C are involved, heat treatment to 200°C should further insure the mechanical stability of these transducer devices.

5. CONCLUSIONS

In this paper we have described the successful completion of reliable, optimum, and high acoustic quality very high temperature transducer devices from our laboratory. It is important to note that the high temperature resistance of these devices is primarily limited by the intrinsic properties of modern piezoelectrics and other materials necessary for obtaining the desired thermo-acoustic parameters. However, with the use of suitable high temperature coupling media, it is now possible to apply ultrasound for high temperature materials characterization, for process monitoring, for thickness and corrosion, and for other applications where elevated temperatures are involved.

ACKNOWLEDGEMENTS

We are pleased to acknowledge with much thanks the assistance of Hollie A. Holderman who was actively involved in this development during its early stages. This project was supported by Ultran's on-going R&D into ultrasound.

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July 22, 1994
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