Advances in Ultrasound for Materials Characterization

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ULTRASONIC CHARACTERIZATION AND ITS SIGNIFICANCE

As the developments and applications of "yesterday's test-tube materials" phenomenally increase, the industry is fast seeking techniques that give significant information about these materials without destroying them. Goals for nondestructive characterization (NDC) are important in the development and uses of materials, particularly in structural, electronic, and biological applications.

Beginning with the significance and place for NDC, this paper describes the advances in ultrasonic technology from Ultran Labs during the last 10 years. Several ideas, observations, and concepts are presented to exhibit the validity, accuracy, and reliability of modern ultrasound for the NDC of a vast family of ceramic materials.

Although a comprehensive definition of characterization was presented by the National Research Council in 1967,[1] the term still means different things to different people. In the broadest terms, "Characterization describes those features of composition and structures (including defects) of a material that are significant for a particular preparation, study of properties, or use, and suffice for the reproduction of a material."[1]

How a material is characterized is of importance to those involved with its development, manufacture, and eventual use. Total and absolute material characterization by a single method is impossible—each method is limited in the information it provides, besides being either entirely destructive (wet chemical methods) or partially destructive (dry physical methods). Mankind does not yet possess a futuristic tool that would yield "all" the information about a material "without ever touching or destroying it." However, sound waves are capable of providing significant information without destroying the material.

Ancient civilizations used the quality of sound to determine the quality of materials, such as bricks, pottery, utensils, etc., by tapping them. Geophysicists use sound via earthquakes and microseisms to decipher the earth's structure and to prospect mineral and oil deposits in the earth. Applications of similar methods by increasing the frequency of sound, generally into several megahertz, can provide useful information about material properties, composition, and defects.

For all practical purposes, ultrasound propagated in a material behaves much like other waves. It generates the phenomena of reflection, refraction, transmission, diffraction, interference, absorption, and scattering of ultrasonic waves when they interact with a material. Investigation of these phenomena in realtime offers significant information about the test material.[2]

SCOPE OF ULTRASOUND IN MATERIALS AND PROCESSES

The NDC use of ultrasound has been known since the phenomenon of piezoelectricity—the cornerstone of modern ultrasonic technology—was discovered in 1880. Until quite recently, it was generally confined to overt flaw detection in some primary metals. Today ultrasound is undergoing widespread use to characterize diverse families of materials and processes: ceramics, plastics, rubbers and tires, tissues, bones, lumber, pulp and papers, liquids, concretes, composites (combination of several materials), rocks and minerals, gases, crystallization, polymerization, liquefaction, solubility, and densification.

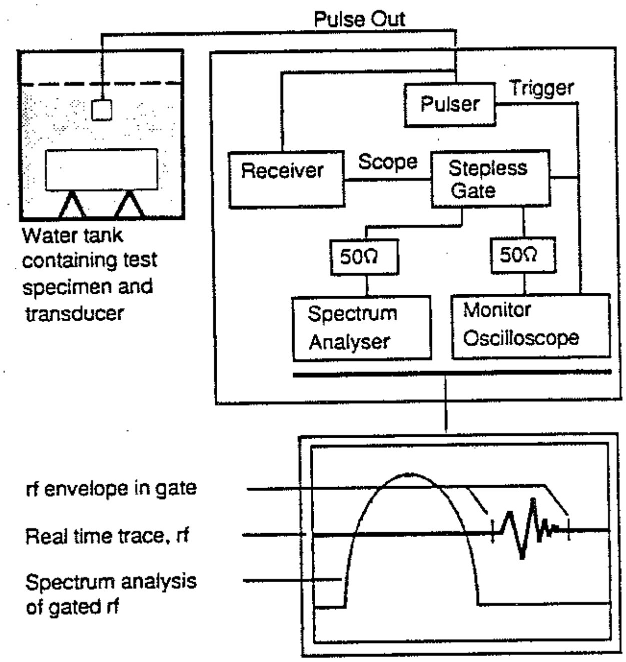

Fig. 1. Setup for pulsed ultrasonic real time and frequency domain analysis used in transducer and materials characterization. Test specimen to transducer coupling in this example is accomplished by the water-delay method.

SIGNIFICANT INFORMATION REVEALED BY ULTRASOUND

Besides the well-known defect detection use of ultrasound in the metals industry, it is capable of providing even more useful information about other materials and processes. Proper relations between ultrasonic and material parameters offer vital information, such as: texture, microstructure, density, porosity, elastic constants, liquid level sensing, dimensional analysis, surface profiling, object imaging, internal microscopy, mechanical properties, thickness/thinness gaging, corrosion monitoring, applied stresses, invisible defects, internal imaging, robotics, artificial intelligence, residual stresses, and in-situ materials testing.

IMPORTANT ELEMENTS OF ULTRASOUND IN NDC

When one is using, or plans to use ultrasound for NDC, the key to success lies in the proper selection and identification of an ultrasonic transducer—the heart of the ultrasonic system as well as the generator and receiver of ultrasound. Its selection must complement the objectives of a given application as closely as possible. The following elements of the transducer should be considered: frequency, pulse width, pulse size, bandwidth, acoustic impedance matching, electrical matching, active area dimensions, beam size, beam shape, sensitivity, geometric precision, test environment suitability, geometrical acoustics, and physical style.

TRANSDUCER ACOUSTICS IN NDE

The critical parameters of practical importance to a specific NDC application are the acoustics of the transducer, i.e.

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the incident characteristics of ultrasound. Nearly all NDC of materials as well as those of transducers are conducted by exciting the transducer with a short-pulse-width electrical impulse, one featuring fast rise time and an equally fast decay time. While analyzing the NDC transducer, the amplifier bandwidth should also complement that of the transducer. Thus, a suitable amplifier is the one that exhibits a frequency bandwidth exceeding that of the transducer. A typical setup for pulsed ultrasonic NDC is shown in Fig. 1. Here, the information about the characteristics of ultrasound (after excitation and amplification of the transducer response) is displayed on an oscilloscope.

TIME DOMAIN CHARACTERISTICS OF ULTRASOUND

Acoustic characteristics of an NDC transducer are achieved and controlled by careful utilization of piezoelectric materials as well as by their relationships with a number of other primary and secondary materials used in the preparation of NDC transducer.[2] Further modifications in transducer acoustics are accomplished by varying the pulse width and electrical impedance of the transducer excitation system.

Because the nature of materials characterization is to achieve extreme time/distance resolution, it is very important that a transducer be prepared and analyzed with extreme precision. This critical requirement is particularly more significant while analyzing relatively "long wavelength" materials, such as most dense ceramics. To characterize the time domain features of a transducer, ultrasound is reflected from a standard target, such as an optically flat and polished clear fused quartz or silica. The ensuing result is known as the real time rf envelope, such as that shown in Fig. 2. In this illustration, real time response of a "broad bandwidth" 10.0 MHz transducer is analyzed. The measured pulse width of this transducer is 150 ns or 1.5 wavelengths. In practical terms this information is of extreme value in deciding time/distance resolution of defects/targets/reflectors within the test material. For example, this transducer will resolve 0.9 mm in steel (assuming longitudinal wave velocity 5970 m/s) and 1.6 mm in dense Al_2O_3 (assuming longitudinal wave velocity 10,900 m/s).

The minimum measurable thickness of a material, or the minimum distance within it at which ultrasonic measurements can be made, depends on the transducer pulse width, not necessarily on the frequency. Experience proves that an indiscriminate increase in the transducer frequency generally leads to severe problems. These are related to system limitations and exponential decay of otherwise significant reflections as a function of increasing frequency.

FREQUENCY DOMAIN CHARACTERISTICS OF ULTRASOUND

Frequency characteristics of ultrasound are measured by performing spectrum analysis or Fast Fourier Transformation of the "rf envelope." Figure 3 shows such an analysis of the transducer in Fig. 2. The measured peak frequency of this transducer is 10.8 MHz, whereas its bandwidth at -6 dB points is 10.0 MHz. The bandwidth center frequency (bcf), which is the arithmetic mean of the lowest and the highest frequencies at -6 dB points, assumes more significance, especially when the frequency spectrum is asymmetrical. The bcf for the transducer in Fig. 3 is 10.6 MHz, indicating near-perfect symmetrical distribution of frequencies in this transducer.

Bandwidth defines the resolution capabilities of a transducer as well as allowing the analysis of a material at frequencies other than the peak or bcf. For example, the transducer in Fig. 3 can be used from 6 to 16 MHz at a -6 dB level. This is generally done by introducing band pass filters of known values while amplifying the received signals. By so doing, a certain reduction in amplitude will be observed. For example, if the transducer in Fig. 3 was used with 6- and 16-MHz band pass filters, an $\approx$6 dB loss in the reflected signal will be observed when compared to the value at 10.0 MHz–the peak frequency of the transducer. This use of ultrasound is very important in determining the frequency-dependent effects of a material upon

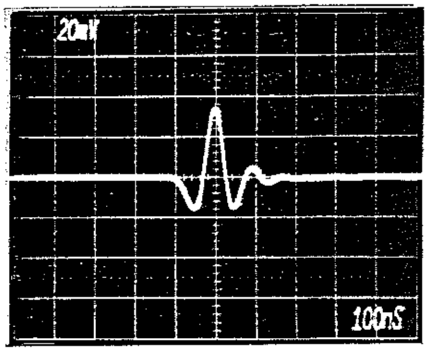

Fig. 2. A real time oscilloscope trace generated by pulsed ultrasound for a "broad bandwidth" ultrasonic transducer. Center of photograph represents the "rf envelope" of ultrasound reflected from the surface of an optically flat and polished clear fused silica (horizontal scale: 100 ns/div, vertical scale: 20 mV/div, and measured pulse width of "rf envelope": 150 ns or 1.5 wavelengths).

the incident characteristics of ultrasound, known as ultrasonic spectroscopy.

DEVELOPMENT OF ULTRASOUND FOR MATERIALS NDC

Broadly speaking, if ultrasound can be propagated in a medium, then it is capable of providing significant information about the medium. However, the diversity of media that can be investigated by ultrasound is extensive, as we have seen earlier. Thus a given transducer may have only specific uses. In general, materials such as concretes, refractories, lumber, fiber-based composites, and most viscoelastic materials require relatively lower frequencies (<100 KHz to 2 MHz) and longer pulse widths (several wavelengths); whereas most metals, glasses, and dense ceramics require higher frequencies (5 MHz to >100 MHz) and shorter pulse widths (few wavelengths). The diversity of materials dictates that a number of transducers be designed for optimum use of ultrasound in NDC. Because of modern research into ferroelectric materials, we have today a number of piezoelectric materials from which to choose to make proper NDC devices. This, in conjunction with several acoustically active and passive materials developed at Ultran Labs, has resulted in the development of a broad range of NDC transducers. Known as five "basic acoustic series" of NDC transducers, Table I shows their general characteristics and appli-

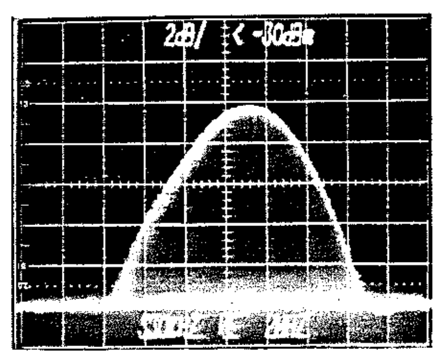

Fig. 3. Spectrum analysis of "rf envelope" shown in Fig. 2, exhibiting the frequency domain characteristics of the transducer (horizontal scale: 2.0 MHz/div, vertical scale: 2 dB/div, measured peak frequency: 10.8 MHz, bcf: 10.6 MHz, bandwidth at -6dB points: 10.0 MHz or 94% of bcf, and usable frequency range at -6 dB level: 6 to 16 MHz).

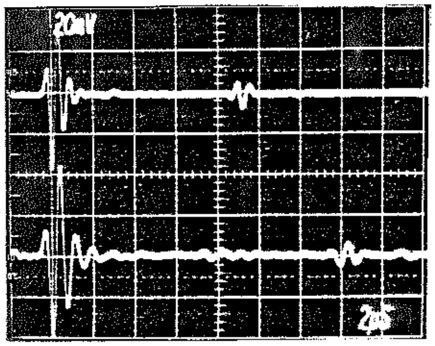

Fig. 4. Real time oscilloscope traces of longitudinal (*top*) and shear (*bottom*) waves from direct-bonded fused-silica refractory (porosity: 20 to 25%, grain size: 100 to 200 μm, thickness of material investigated: 19mm; longitudinal transducer: 12 mm diameter and 1.0 MHz, velocity: 4140 m/s; shear wave transducer: 12 mm diameter and 1.0 MHz, velocity: 2700 m/s, elastic modulus: 30 GPa, shear modulus: 13 GPa, and Poisson's ratio: 0.11).

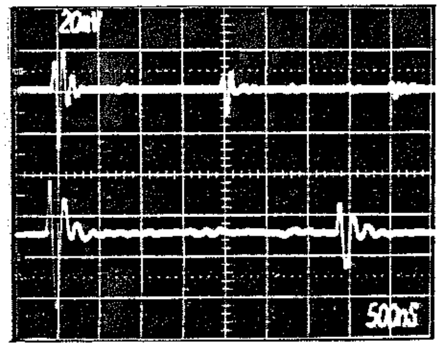

Fig. 5. Real time oscilloscope traces of longitudinal (*top*) and shear (*bottom*) waves from dense Al_2O_3 (porosity: none, grain size: submicrometer, thickness of material investigated: 10.8mm; longitudinal transducer: 6mm diameter and 20.0 MHz, velocity: 10 540 m/s; shear wave transducer: 6 mm diameter and 10.0 MHz, velocity: 6220 m/s, elastic modulus: 370 GPa, shear modulus: 150 GPa, and Poisson's ratio: 0.23.

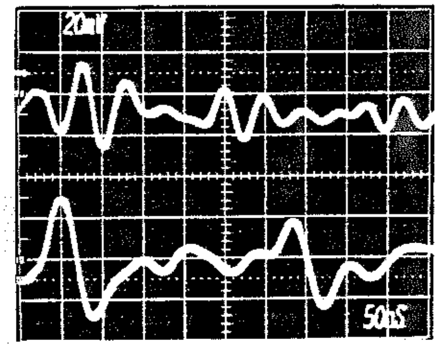

Fig. 6. Real time oscilloscope traces of longitudinal (*top*) and shear (*bottom*) waves from dense BeO (porosity: none, grain size: submicrometer, thickness of the material investigated: 1.07 mm; longitudinal transducer: 6 mm diameter and 20.0 MHz, velocity: 12 190 m/s; shear wave transducer: 6 mm diameter and 10.0 MHz, velocity: 7360 m/s, elastic modulus: 396 GPa, shear modulus: 162 GPa, and Poisson's ratio: 0.21).

cations. These transducers, spanning in frequency range from <100 KHz to >100 MHz and in pulse widths from a theoretical minimum of 0.5 wavelength to over 6 wavelengths have been developed and optimized in our laboratory.

APPLICATIONS OF MODERN ULTRASOUND IN MATERIALS CHARACTERIZATION

As we stated earlier, NDC use of ultrasound is quite recent—at best $\approx$10 years old. For newcomers to NDC, it should be interesting to note that developments in ultrasonic technology are also quite current. Not too long ago, reliability and performance of transducers were lacking and generally challenged by materials researchers. In the view of this investigator, because of the lack of developments in basic ultrasonics, this important wave did not receive the due attention of modern materials scientists for its NDC use. Nondestructive testing, as it is popularly known in the QC/QA functions of steel and aluminum industries, was looked down upon in the R&D and materials labs of industries and research institutions. Once described as "black art" and "voodoo," NDC has come a long way in becoming a respectable technique in materials characterization. The progress in ultrasonic transducer technology, not the applications of computers, is chiefly responsible for easing the apprehensions of materials scientists in the use of ultrasound as a reliable and accurate tool for NDC. In this section we show some of the major and practical applications of ultrasound that are the direct result of advanced transducer developments at Ultran Labs.

DEVELOPMENT AND APPLICATIONS OF DRY OR SELF-COUPLING TRANSDUCERS

Those familiar with ultrasonic NDC methods know that the transducer "must" be coupled physically to a test part with liquid couplants such as oil, grease, water, etc. This chronic limitation of ultrasound has been eliminated by the development of a longitudinal- and zero-degree incident beam, shear-wave transducer capable of efficient transfer of ultrasound into test materials without the use of liquids. Originally, dry or self coupling transducers were developed for the examination of aircraft/aerospace composites; however, they soon found applications in a wide range of materials, including ceramics. The following observations on some relatively coarse-grained and dense ceramics were made possible by dry coupling transducers. Figure 4 shows ultrasonic observations of longitudinal and shear waves from a direct-bonded fused-silica grain. Figure 5 shows ultrasonic observations of longitudinal and shear waves from dense Al_2O_3. Figure 6 shows ultrasonic observations of longitudinal and shear waves from dense BeO. Dry coupling ultrasonic techniques are applicable when: the use of liquid couplants is a nuisance, liquid couplants are expected to damage or alter the test material properties, and accurate measurements of porous materials are desired.

DEVELOPMENT AND APPLICATIONS OF LAMBDA (UNIPOLAR) TRANSDUCERS

The most critical requirement in materials characterization is the ability of the characterizing technique to provide the utmost in resolution. As we have seen, the shorter the pulse width of the transducer, the higher the resolution of the reflecting target/surfaces of the test material. Ideally, the maximum achievable resolution:

$$d_{min}=\lambda/2 \tag{1}$$

i.e. the minimum distance that can be resolved by a wave is equal to half the wavelength of that wave in a given material. This is only possible if the pulse width of the wave is also $\lambda/2$. Lambda transducers, developed in our lab nearly 10 years ago, are characterized by the "rf envelope" occupying time corresponding to $\lambda/2$, thereby emitting a near-perfect unipolar impulse.[3] Figures 7 and 8 show the time and frequency domain

Table I. General Characteristics of Five "Basic Acoustic Series" of NDC Transducers*

Series	Frequency range	Bandwidth % at −6 dB	Pulse width wavelengths	Sensitivity relative	Applications general
W	<100KHz–>25MHz	50–100	1–2	Mod–high	High resolution
P	<500KHz–>25MHz	40–70	2–4	High–moderate	General purpose
K	<100KHz–>20MHz	20–50	3–6	Very high	High sensitivity
M	<10MHz–>100MHz	30–100	1–6	Mod–high	V. high frequency
λ	<500KHz–30MHz	100–300	0.5–1	Low–moderate	V. high resolution

*Derived from modern piezoelectric materials in conjunction with several acoustically active and passive materials surrounding the piezoelectric elements.

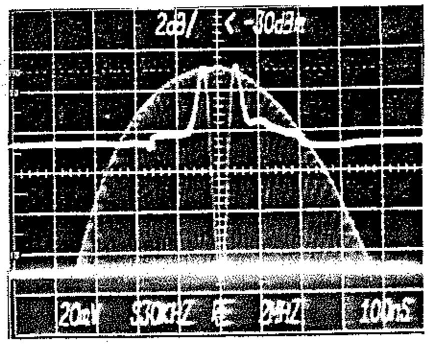

Fig. 7. Time and frequency domain response of a typical planar-wave, unfocused lambda transducer (peak frequency: 10.0 MHz and bandwidth: 12.0 MHz).

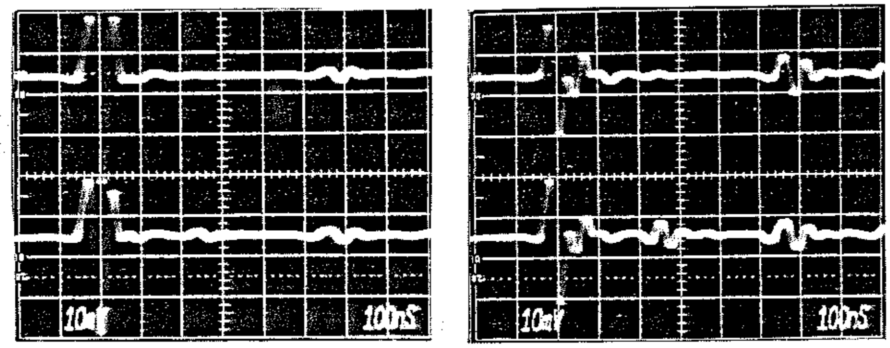

Fig. 9. Top traces defect-free zone in MLCC and bottom traces indication of $\approx$25 μm defect in the center. (Thickness of samples: 1.5 mm; lambda transducer's nominal frequency: 15.0 MHz; 6 mm diameter, and 19 mm focus in water). (*A*) was generated by optimizing the focal point on the sample surface. (*B*) was generated by optimizing the focal point on the defect zone.

responses of typical unfocused and focused lambda transducers, respectively.

Since their development, lambda transducers have generated much interest for their possible use in advanced materials characterization. Before these devices, one would use very high frequencies to achieve high resolution and minute defect detection. By reducing the transducer pulse to a bare minimum of half the wavelength in lambda transducers, it is not necessary to apply cumbersome, very-high-frequency ultrasonics, even in the characterization of relatively long wavelength materials, such as nearly all superdense ceramics and composites. This spectacular development, until recently "ignored" by the NDT industry, has not only expanded the horizons in NDC of denser materials, but it has also substantially reduced the cost and complexity, which would otherwise be wasted on very-high-frequency ultrasonic systems. Another obvious advantage of lambda transducers is that its exceptionally broad bandwidth (100 to 300% of bcf) now makes it easier to conduct ultrasonic spectroscopy (frequency dependence of ultrasonic attenuation/absorption as a function of material microstructure or atomic structure) without using a number of transducers![4]

The following examples of the applications of lambda transducers could not easily be generated even using conventional frequencies in the neighborhood of 100 MHz. Figures 9(*A*) and 9(*B*) show a small defect in a multilayer ceramic chip capacitor, and Figs. 10 and 11 show a diamond layer bonded to a WC substrate.

Examination of a Plasma-Sprayed Cr_2O_3 Coating on Inconel Substrate. A thin and relatively dense coating of Cr_2O_3 on inconel is generally extremely difficult to measure, particularly if the test must be performed from the coating side. The complexity of this test is further aggravated by the fact that both Cr_2O_3 and inconel are closely matched with respect to their acoustic impedances ($Z_{Cr_2O_3}=44\times10^5$g/cm^{2}s and $Z_{inconel}=64.5\times10^5$g/cm^{2}s), thus yielding a very low reflection coefficient at the interface, i.e. an R value (reflection coefficient at Cr_2O_3/inconel interface) of 3.5%. Although the interface between these two materials is somewhat faint because of a poor reflection coefficient, nonetheless a lambda transducer clearly resolved 180-μm-thick plasma sprayed Cr_2O_3 on inconel, Figs. 12 and 13.

Measurement of Flame Sprayed Fe_3O_4 on Carbon Steel. Similar to the previous example, this application is also quite cumbersome because of the close similarities between the acoustic impedances of two materials involved ($Z_{Fe_3O_4}=35.6\times10^5$g/cm^{2}s and $Z_{steel}=51\times10^5$g/cm^{2}s), thus yielding a very low reflection coefficient at the interface, i.e. an R value (reflection coefficient at Fe_3O_4/steel interface) of 3.2%. However, in this case the coating is relatively thick, 0.55 mm. This material is clearly resolved by a mere 10 MHz lambda transducer. Fig. 14.

Lambda transducers are proposed for the following materials applications: extremely high resolution, phase transformation studies, ultrasonic microscopy, studies in which collimation of ultrasound is desired, minute defect detection, fre-

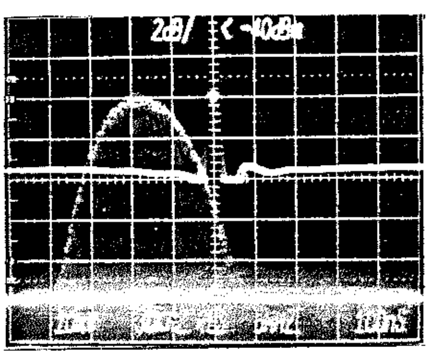

Fig. 8. Time and frequency domain response of a typical point focused lambda transducer (peak frequency: 15.0 MHz, bandwidth: 18.0 MHz, and 19 mm point focus in water).

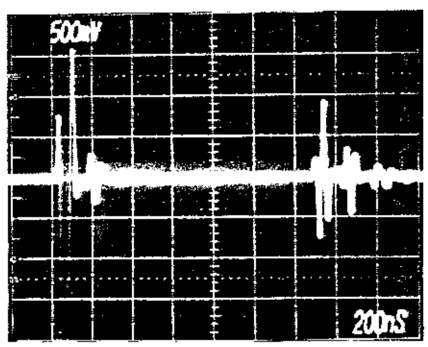

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Fig. 10. Real time rf A-scan from a diamond cutting tool (*extreme left*) interface between transducer delay and diamond surface. No. 2 interface between diamond and WC. No. 3 bottom surface of WC. Test conducted from the diamond side. (Measured velocity of WC: 7750 m/s, thickness of WC: 4.1 mm; lambda transducer's nominal frequency: 20.0 MHz, 3 mm diameter with a clear fused-quartz delay tip.)

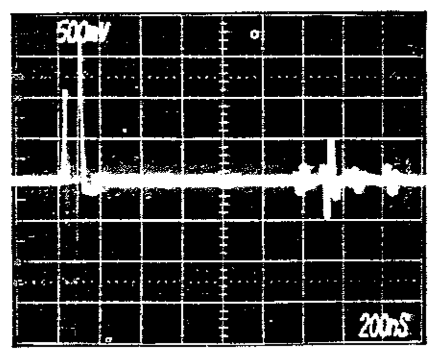

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Fig. 11. Real time rf A-scan from a diamond cutting tool (*extreme left*) interface between transducer delay and WC surface. No. 2 interface between WC and diamond. No. 3 bottom surface of diamond. Test conducted from the WC side. (Measured velocity of diamond: 7940 m/s, thickness of diamond: 0.63 mm; lambda transducer's nominal frequency: 20.0 MHz, 3 mm diameter with a clear fused-quartz delay tip.)

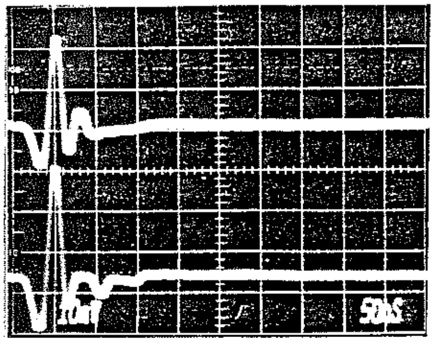

Fig. 12. Real time rf A-scan of a Cr_2O_3 plasma-sprayed coating on Inconel (*top*) reflection from optically flat fused-quartz front surface establishing zero point and (*bottom*) from the coating side. No. 1 interface between Cr_2O_3 and transducer delay. No. 2 interface between Cr_2O_3 and Inconel.

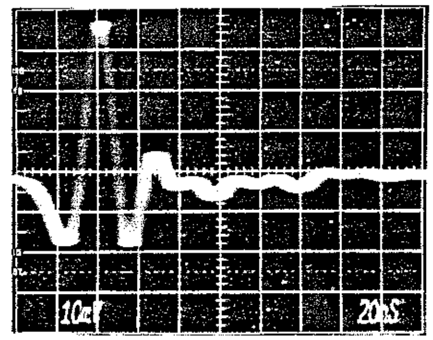

Fig. 13. Bottom trace of Fig. 13, except with horizontal scale enlarged from 50 to 20 ns/div (coating thickness: ≈ 180 μm, lambda transducer's nominal frequency: 30.0 MHz and 3 mm diameter, bandwidth: 30.0 MHz, flat or unfocused in water).

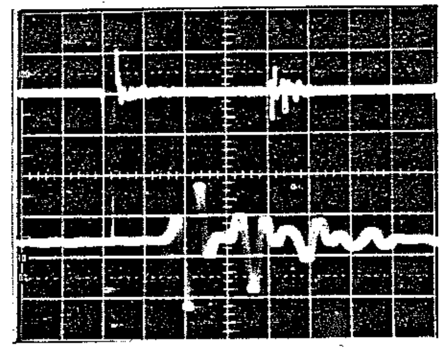

Fig. 14. Real time rf A-scan of a flame-sprayed Fe_3O_4 on carbon steel (*top*) extreme left: transducer delay and steel interface; extreme right: multiple reflections from the coating and (*bottom*) same as (*top*) except the multiple reflection region amplified from 500 to 100 ns/div in horizontal scale. (Lambda transducer's nominal frequency: 10.0 MHz, bandwidth: 13.0 MHz, 6 mm diameter, unfocused in water.)

quency dependence of ultrasonic attenuation/absorption, and studies in which near-field effects of ultrasound must be eliminated.

DEVELOPMENT OF CONTINUOUS-USE, HIGH-TEMPERATURE TRANSDUCERS

It is highly desired to investigate temperature-dependent characteristics of materials by nondestructive means. However, all previous attempts to accomplish this by ultrasound utilized complex cooling mechanisms around the transducer or high loss and unreliable buffer rods to protect the critical transducer elements in the device. These approaches were obviously based on "the-then inevitability" of the transducer and its knowledge. Although strides are being made to further increase the temperature stability of ultrasonic transducers, these devices have been successfully developed and tested to withstand temperatures exceeding 250°C continuously. With the applications of suitable buffer zones, such as those made of machinable ceramics or quartz glass, it is possible to make reliable ultrasonic measurements exceeding 1000°C.[5]

To determine ultrasonic losses caused by the temperature effects on the piezoelectric and other materials of the transducer, a thermal hysteresis study was conducted on a 5-MHz transducer (12 mm diameter intermediate range—250°C). From the observations in Fig. 15, it was noted that, while the losses entirely attributed to the transducer materials are in the neighborhood of 6 dB, the losses caused by the couplant—either its attenuative behavior as a function of temperature or by its evap-

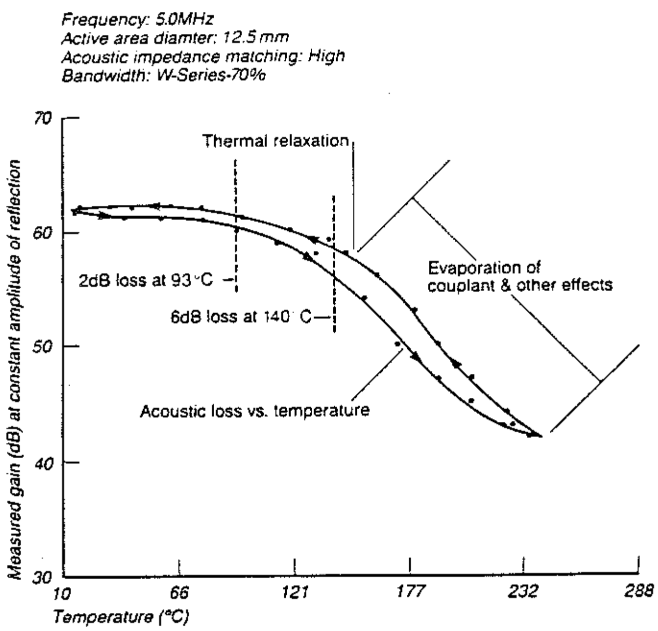

Fig. 15. Thermal hysteresis loop for an intermediate range, 250°C ultrasonic NDC transducer. Example shown is for a 5 MHz, 12-mm-diameter device. High-temperature loss attributed to the piezoelectric and other transducer materials: 6 dB. Losses attributed to couplant: 14 to 20 dB.

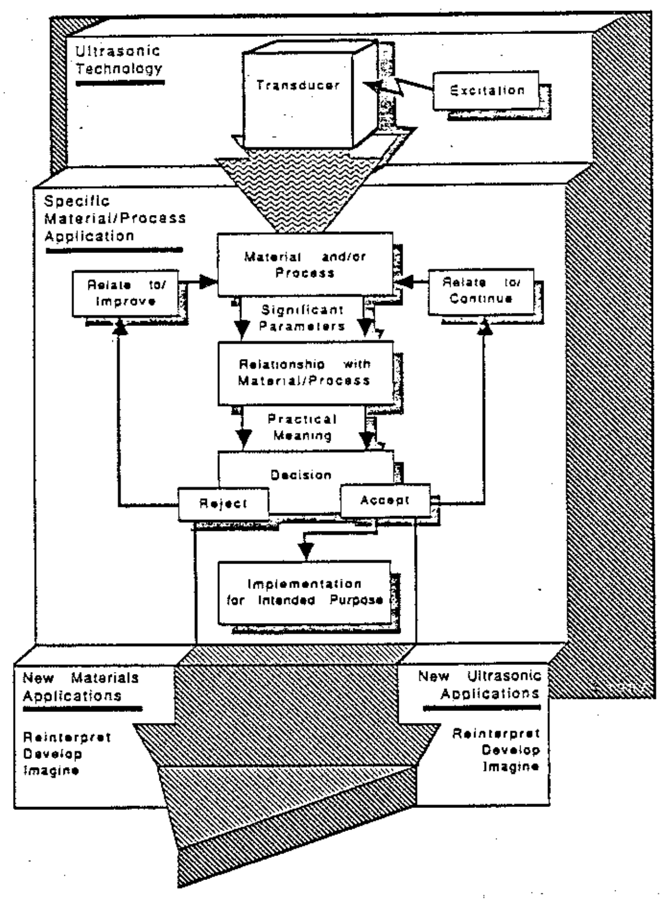

Fig. 16. Perspective on ultrasonic characterization of materials and processes.

oration—were of the order of 14 to 20 dB. Although we have developed both longitudinal- and shear-wave transducers for continuous high-temperature exposure, note that transducer coupling at high temperature remains cumbersome.

RELATION BETWEEN ULTRASONIC NDC AND MATERIALS DEVELOPMENT AND MANUFACTURE

In this paper we have described the significance, reliability, and some recent developments in ultrasonic technology from our lab. The subject concerning the ultrasonic NDC is relatively new and it needs to be further applied as well as further perceived in light of other materials characterization techniques. Although the uses of ultrasound are numerous, there are only a few parameters that are measured in ultrasonic testing. Direct relationships of the following ultrasonic parameters provide significant information about the media through which ultrasound travels: velocity of longitudinal waves, velocity of shear waves, velocity of surface waves, and frequency-dependent aspects of longitudinal, shear, and surface waves.

From the practical industrial standpoint materials characterization serves the following functions: (1) Characterized parameters determine the suitability and applicability of a given material in one or more uses of that material. (2) Characterized parameters also shed light on those parameters of material preparation that are significant in its reproduction or in determining the conditions of a particular material preparation.

From the foregoing sections of this paper, we have shown that ultrasonic nondestructive characterization serves these functions well. The relationship between ultrasonic NDC and materials characterization from the standpoints of quality control, development, and manufacture, and applications of materials is shown in Fig. 16. Note that, while a number of applications of ultrasound can be realized relatively easily, to a great extent there is an acute need for further development of more meaningful relationships. In short, the theories on ultrasound-material interaction are well-developed, but they need to be proved through further empirical and experimental investigations. Although a QC/QA engineer can apply ultrasound easily to efficiently and accurately measure porosity, density, elastic constants, etc., a materials researcher needs to further enhance relationships between materials and various types of ultrasonic waves in time and frequency domains. The mere examination of a sample or two, varying in a property or two, is not sufficient to make mathematical equations and conclusions. In our lab we have witnessed several industrial researchers whose observations were fundamentally proven wrong by refined acoustics—not by expensive electronics—yet at one time they made broad generalizations based on their improper ultrasonic observations. Further research and education are the only methods to eradicate currently prevalent confusion on this subject—after all, major implications of ultrasonic NDC are in the economy- and safety-related materials applications. Being relatively new in the field of materials science and technology, ultrasound offers unlimited excitement and challenges to a materials developer and researcher.

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