NON-CONTACT ULTRASONIC CHARACTERIZATION OF CERAMICS AND COMPOSITES

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ABSTRACT

Conventional wisdom stipulates that ultrasonic analysis of ceramics and of other solids cannot be accomplished without direct transducer contact with test materials. Thus, non-contact ultrasound-- i.e., through air coupling--is viewed as impossible. This impossibility is entirely due to the phenomenal acoustic impedance mismatch between the coupling air and the test media. After many years of struggle, this barrier has been broken through novel transducer designs, characterized by extremely high transduction in air and in other gaseous media. Now it is possible to propagate ultrasound in a non-contact mode through a wide variety of materials. We have successfully built and tested such transducers within the <250kHz to >5MHz frequency range on very thick to very thin, green and sintered ceramics under ambient atmospheric conditions.

This paper describes the quantitative and qualitative observations on a variety of ceramic and other materials by utilizing the newly developed non-contact methodology for density-velocity relationships and for defect detection. Also shared are some reflections about ultrasound from the author's experience.

INTRODUCTION

Analogous to any materials characterization method that utilizes a wave as the characterizing tool, ultrasound can also provide significant information about the medium through which it is propagated. The reflected and transmitted ultrasonic signals can be correlated and manipulated to detect internal discontinuities, to measure the physical properties and microstructure, and to generate the surface and internal images of a medium: Table-1. Besides being nondestructive, the advantages of ultrasound are numerous: it does

Table I. Significance of Ultrasonic Analysis, modified after Vary [1].

MEASUREMENT CATEGORY	MEASURED PARAMETERS	INFORMATION REVEALED
Time Domain	Velocities of Longitudinal, shear, and surface waves.	Direct correlation with density, porosity, defect detection, mechanical properties, residual & applied stresses, interfacial analysis, etc.
Frequency Domain	Frequency dependence of ultrasound absorption and attenuation. (Ultrasonic Spectroscopy)	Direct correlation with atomic structure, inter-granular relationships, phase detection, etc.
Image Domain	Time of flight, attenuation, or frequency domain monitoring of ultrasonic signals by raster or multi-transducer scanning.	Planar or 3D images of surface and internal features of materials, including graphical representation of material heterogeneity in terms of microstructure, defects, and physical properties.

not require special sample preparation; it can be adapted to on-line manufacturing or testing environments; unlike x-ray and γ-ray methods, ultrasound is non-hazardous; the equipment involved is relatively easy to transport; and it can be used to test transparent or opaque materials. In fact, ultrasound can be used to characterize *any* medium through which it can be propagated. For example, with the exception of a vacuum, all three states of matter--solid, liquid, and gas-- can be analyzed by ultrasound. However, the complexity of the test material geometry can be sometimes arduous to overcome. Also, the selection and design of suitable transducers can be extremely frustrating, particularly for the NDC of complex materials.

These impediments can be greatly minimized through the development of NDC educational and training curricula which are targeted towards the objectives of materials characterization [2]. It is a young field that needs to be understood, taught, and applied properly. Otherwise, there is a real danger of making poor choices, and of developing even poorer conclusions. Despite the dedicated efforts of a handful of educational and research institutions [3,4,5,6], it is this author's experience that this discipline--from the standpoint of

materials science and engineering--is not yet fully developed. For example, the practical aspects of a wave-material interaction with respect to the crystallo-chemical makeup of the media through which ultrasound is propagated are not addressed in a manner that are congruent to other methods of materials characterization [2].

A one-to-one analogy and correlation with optics can create unreal perceptions about ultrasound, leading to erroneous expectations and conclusions. We forget that in ultrasound we do not have the luxury of light's rectilinear propagation and its small frequency and wavelength range: ~3.00 to ~7.00nm. In ultrasound we are dealing with an enormous frequency range--<50kHz to >1GHz--and an equally large range of wavelengths due to the diversity of test media through which it (ultrasound) can be propagated. When variations in apertures and attenuation are added, a complex spectrum of ultrasonic fields ensues. These seemingly academic subjects are of real value in defining the suitability of ultrasound and its limitations with respect to resolution, detectability, scattering, propagation, etc. Knowledge of these issues from a practical standpoint is vital.

Since its inception, ultrasonic NonDestructive Testing (NDT) has been preoccupied with the overt flaw detection in metals and alloys by either physically coupling transducers to test materials with oil and grease, or by submerging both the transducers and the test materials in water or like media. However, materials--such as powder compacts, green, liquid-sensitive and porous materials--cannot be reliably tested by conventional ultrasonics. On the other hand, the advantages of Non-Destructive Characterization (NDC) are obvious and of major significance at a time when the issues of quality and economics of the manufactured materials are extremely demanding. Ultrasound must first be developed in such a way that it is applicable to the wide range of materials at various stages of their processing without damaging or altering them during testing.

WORK RELATED TO NON-CONTACT ULTRASONICS

Recognizing the necessity of total materials quality (including the accuracy and reliability of the analyzed information), we have advanced ultrasonic NDC to new heights through the essential transducer and applications R&D with respect to the modern materials characterization objectives [7]. In order to facilitate the NDC of green, fragile, liquid-sensitive, and porous materials, we introduced the dry coupling longitudinal and shear wave transducers in 1983. These were

quickly adapted to our earlier development of the short unipolar time envelope transducers. Dry coupling provided the means for test material protection, while the short pulse permitted accurate time of flight measurements. The combination of transducer acoustics with the coupling methodology facilitated quantitative NDC of a wide range of green and sintered ceramics and composites [8,9,10,11,12]. While the uses of the new transducers in aerospace, nuclear, materials, and bio-medical industries are numerous today, the proprietary nature of their applications prevents further disclosure.

Despite the arduous nature of non-contact ultrasonics, our struggle for its development did not cease with the advances in dry coupling technique. Transducers for air/gas propagation (<250kHz to ~20MHz) were a by-product of the dry coupling transducers. They found limited applications for high frequency receivers, surface profiling, proximity analysis, level sensing, and non-contact characterization of paper products. These earlier air/gas propagation transducers failed to generate high-strength quality transmitted signals required for the NDC of ceramics or metals.

In 1993 our struggle finally produced transduction in gaseous media to such an extent that--leaving aside non-contact ultrasound propagation in papers, rubbers, and low density materials--it was possible to accomplish this through materials such as dense ceramics, metals, and biological structures [13]. Based upon the development of non-contact ultrasound, work is also in progress for the critical medical diagnostic applications such as burnt skin and bed sore characterization [14]. Recently an excellent methodology for the measurement of elastic constants of polymer matrix composites has also been reported [15]. For a review on non-contact or air-coupled ultrasound, see reference [16].

COMPLEXITY OF NON-CONTACT ULTRASOUND

In principal, the non-contact ultrasonic *modus operandi* is analogous to the well-known contact and immersion techniques, but it varies dramatically in magnitude. For example, while the acoustic impedance mismatch between the transducer and the test materials is greatly minimized in contact ultrasound, it must be overcome in non-contact modality. The mismatch is of several orders of magnitude when one compares the acoustic impedances of air (4.0×10^2 kg/m^2.s) and of most industrial materials (30×10^6 kg/m^2.s). In addition, the phenomenal absorption of ultrasound by ambient air--particularly at

frequencies greater than 1.8MHz--multiplies the complexity by magnitudes. Until the acoustic barrier between air and solids is broken, non-contact ultrasound cannot be realized. As will be seen in this paper, this is the barrier that has finally been broken.
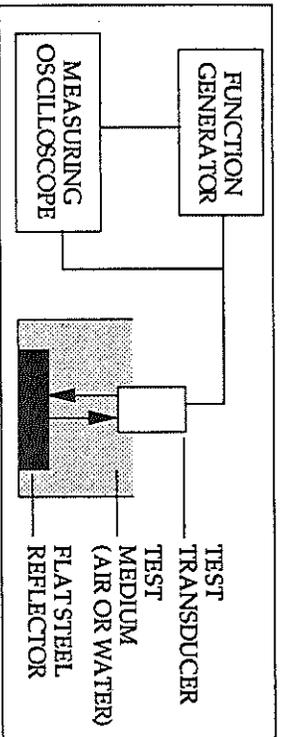

Fig. 1. Transducer characterization scheme for sensitivity comparison.

COMPARISON OF NON-CONTACT AND CONVENTIONAL NDT TRANSDUCERS

A simple, yet effective, unbiased transducer characterization scheme was developed to evaluate and to compare the sensitivity of non-contact transducers with those of conventional high-sensitivity NDT transducers. Transducers in pulse-echo reflection mode were excited by a single-burst 16V sine wave pulse from a function generator, Fig. 1. The reflected signal from a flat steel target--10mm away from the transducer in the test medium, received by the same transducer--was fed directly into the oscilloscope. The frequency of each test transducer was adjusted in the function generator to yield the maximum reflected signal amplitude. The sensitivity (loop gain) was determined according to:

$$\text{Sensitivity (dB)} = -20 \log V_x/V_o \qquad (1)$$

where V_x is the received signal amplitude in volts directly measured from the oscilloscope, and V_o is the voltage of the excitation pulse, in this case, 16V. Table II provides a one to one comparison for the sensitivities of non-contact and the conventional high sensitivity immersion NDT transducers, respectively, in ambient air and water. All transducers are planar, and typically, 25mm active area in diameter.

CURRENT LIMITATIONS OF NON-CONTACT ULTRASOUND

While the non-contact transducers have accomplished the task of propagation through virtually all materials, it is still not easy to apply them in the popular pulse-echo mode for the NDC of ceramics or metals. However, the transmission technique is perfectly applicable.

Table II. Comparison of the reflected sensitivities of non-contact transducers and the conventional NDT immersion transducers.

FREQUENCY (MHz)	TRANSDUCER SENSITIVITY (dB)	
	Non-Contact (In Ambient Air)	NDT Immersion (In Water)
0.25	-62	-36
0.5	-66	-32
1.0	-72	-30
2.0	-84	-28

Note that despite the exceptionally high transduction of current non-contact transducers (only about 40dB lesser than that in water, Table II), still high amplification of the received signals through test materials is required. This requirement increases as a function of increasing acoustic impedances of materials, further exacerbating the problems of interpreting transmitted signals. For example, besides the key signals corresponding to ultrasound propagation in the materials, one sees a number of unwanted signals related to material interfaces and edges, and from any other reflector (walls, equipment, operator, etc.) upon which ultrasound falls through air from the transducers. In time domain, the majority of such signals arrive much later than the signals of interest. It is observed that the first arrival always corresponds to the ultrasound transmitted signal through the test material. However, sometimes signals internally reflected through test materials can also be seen. The appearance of such signals, though of great value, depends upon the acoustic impedance and the frequency-dependent attenuation of the test material. Usually the internally reflected signals are observed when the test material impedances and the attenuation are relatively low. Figures 2 and 3 show the location of various signals when ultrasound is propagated through a material in the non-contact transmission mode under ambient atmospheric conditions. Reflected signals corresponding to air distances between the material surfaces and transmitting and receiving transducers can be removed from the
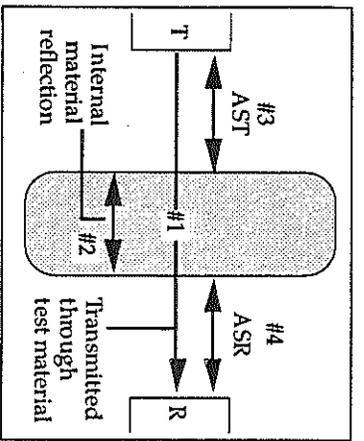
Fig. 2. Location of key ultrasonic signals in non-contact transmission mode. #1--Straight through material. #2--Internally reflected in material. #3 & 4--Reflections from Air Separation Transmitter (AST) and Air Separation Receiver (ASR) from material surfaces.
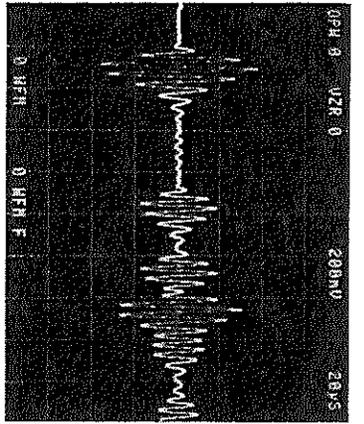

Fig. 3. Demonstration of key ultrasonic signals from an 80mm thick polystyrene. From left to right: Transmitted through material, internal reflection, AST (15mm) ASR (20mm).

region of interest in time domain by increasing the transducers' air separation distances.

VELOCITY MEASUREMENT

There are several ways to determine longitudinal wave velocity in the test materials by non-contact transmission mode. Here we describe a simple two-step technique, as shown in Fig. 4.

Measurement of Air Velocity

The first step is to align both transmitting and receiving transducers that are facing each other in air and are separated by a distance, da. The next step is to read the Time of Flight (ToF), ta, corresponding to ultrasound travel in air directly from the oscilloscope, and then to determine the air velocity,

$$V_a = d_a/t_a \qquad (2)$$

Measurement of ToF in the Test Material and its Velocity

After inserting the test material between the transmitting and receiving transducers, the complete ToF, Tc--corresponding to ultrasound travel in air as well as in the test material--is read directly
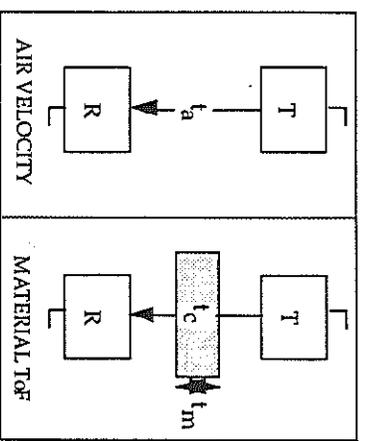
Fig. 4. Alignment of transducers and insertion of the test material for velocity measurements.

from the oscilloscope. The ToF corresponding to the delay time in air is determined as:

$$t_0 = (t_a - t_{am}) \qquad (3)$$

where tam is the ToF in air relative to the material thickness, dm, i.e.,

$$t_{am} = d_m/V_a. \qquad (4)$$

The ToF through the test material is thus

$$t_m = (t_c - t_0). \qquad (5)$$

Therefore, the test material velocity, Vm can be determined as:

$$V_m = d_m/t_m \qquad (6)$$

or

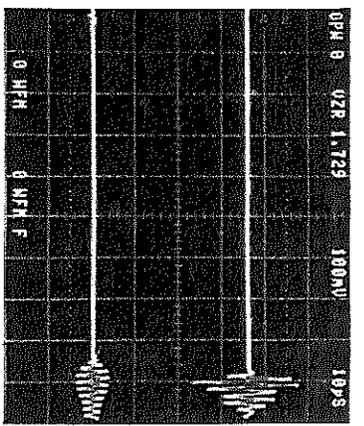$$\boxed{V_m = \frac{d_m}{t_c - (t_a - t_{am})}} \qquad (7)$$

Fig. 5. Top trace: T and R in air 30mm apart. Bottom trace: With a 1.6mm green multi-layer ceramic.

As an example, Fig. 5 shows the transmitted signals corresponding to ultrasound travel in air and in a test material, in this case a green multi-layer ceramic. From these observations:

$$t_a = 86.13\mu s, \quad V_a = 348.13 m/s, \quad t_c = 82.3\mu s, \quad V_m = 2,100 m/s.$$

Using equation 7, the velocity of this material is 2,100m/s. The velocity determined by the dry coupling technique of this sample is 2,240m/s. Assuming our measurements of the ToFs are accurate, it is reasonable to conclude that the velocity determined by the non-contact technique is more representative of the true value. Accuracy of the velocity calculation is a sensitive function of the accuracy of the measured ToF. For example, a variation of 10μm distance in air will introduce an error of ±20ns in the ToF measurement. For thin and for high velocity materials, this could make a considerable difference. Therefore, in order for the non-contact ultrasonic velocities to be reliable, it is imperative to make accurate and repeatable ToF measurements.

DEFECT DETECTION

Should ultrasound encounter a region in the test material characterized by defects such as internal cracks, pores, gross texture or compositional variations, its transmitted signal would be correspondingly different from that in the defect-free region. This is demonstrated in Fig. 6 on a 47mm thick polystyrene block.

The most frequently asked questions in ultrasonic NDC concern the resolution and detectability. The maximum resolution of two closely lying surfaces or discontinuities is the major goal in the pulse-echo or the direct reflection technique. While resolution is not the main issue in the direct transmission technique (applied in the present work), the ability to detect the smallest defect in a test medium is relevant. Detectability is directly proportional to the wavelength and to the size of the interrogating beam, particularly that of the receiving transducer. Table III provides an idea about the non-contact detectability limits in various materials as a function of the ultrasonic velocities.

VELOCITY-DENSITY RELATIONSHIPS

According to the velocity measurement scheme described in this paper, green and sintered ceramics (those varying in density or in other
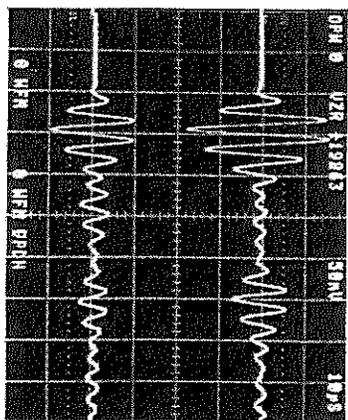
Fig. 6. An example of defect detection in a 47mm thick polystyrene block. Top Trace: The defect-free region. The first signal corresponds to transmission through the material, and the second to its internal reflection. Bottom trace: Reduced amplitudes with a 4mm cylindrical hole. Also observe the appearance of the hole indication between the transmitted and reflected signals.

Table III. Non-Contact ultrasonic detectability limits*

MATERIALS & VELOCITIES (m/s)	DETECTABILITY (mm), function of FREQUENCY				
	100kHz	200kHz	500kHz	1.0MHz	2.0MHz
1,500 Liquids, visco-elastic materials, tissues, green materials, etc.	1.6	0.8	0.3	0.15	0.08
3,000 Polymers, composites, bones, porous and granular materials.	3.3	1.6	0.6	0.3	0.15
6,000 Metals, ceramics, glasses, and composites.	6.6	3.3	1.2	0.6	0.3
12,000 Dense ceramics, oxides, nitrides, carbides, etc.	13.0	6.6	2.5	1.2	0.6

*NOTES
1. Detectability is the smallest detectable discontinuity in the test medium.
2. These calculations are based upon the interrogating beam size being equal to one wavelength. Note further that this requirement is not easily realized in non-contact applications.
3. The effects of attenuation and diffraction have not been considered in these calculations.

features) were investigated. The transmitting transducer was excited by a 400 volt pulse. Transmitted signals were received by a receiving transducer and amplified by a broadband amplifier. Depending upon the acoustic impedance and the frequency attenuation behavior of the test materials, transducer frequencies varied from 250kHz to 2.0MHz.

Figures 7 and 8, respectively, show rf A-scans of green ceramics for velocity measurements, and the velocity-density relationship for green Al2O3. Similarly, figures 9 and 10, respectively, show rf A-scans and the velocity-density relationship for sintered Al2O3.
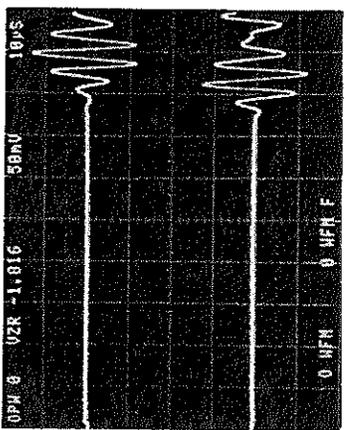
Fig. 7. Full transmitted signals of green ceramics. Top trace: Slip-cast porcelain. $Z = 19X10^6 kg/m^2.s$. $V = 1,180 m/s$. Bottom trace: Pressed titania. $Z = 4.2X10^6 kg/m^2.s$, $V = 1,900 m/s$.
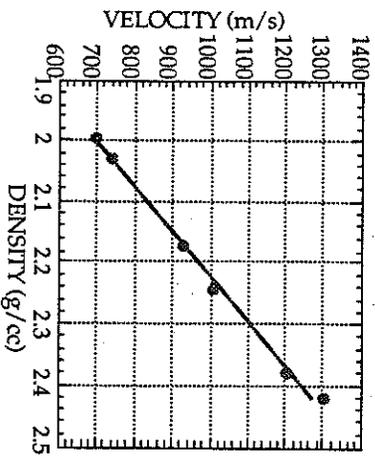
Fig. 8. Relationship between density and non-contact ultrasonic velocity for green Al2O3.

QUALITATIVE CHARACTERIZATION

We have applied the non-contact ultrasonic technique on a wide range of industrial materials and biological structures in order to demonstrate its potential and feasibility for various applications [11]. As stated earlier, if ultrasound can be propagated through a medium, then interpretation of transmitted signals holds the key to important information relative to quality and internal features of the medium. Here we provide the self-explanatory evidence of qualitative analysis for selected green and sintered ceramics, rocks, and composites. The observations reported here show not only feasibility of non-contact
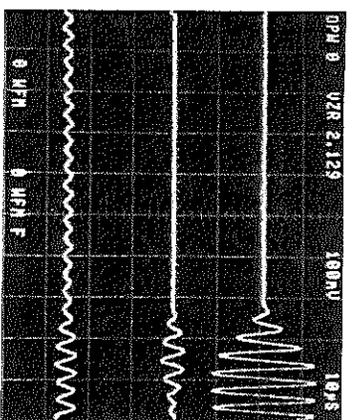
Fig. 9. Full transmitted signals of sintered Al2O3. Top trace: 65% dense. $Z = 15X10^6 kg/m^2.s$. $V = 5,900 m/s$. Middle trace: 77% dense. $Z = 25X10^6 kg/m^2.s$, $V = 8,100 m/s$. Bottom trace: 92% dense. $Z = 37X10^6 kg/m^2.s$. Velocity = ~10,000m/s.
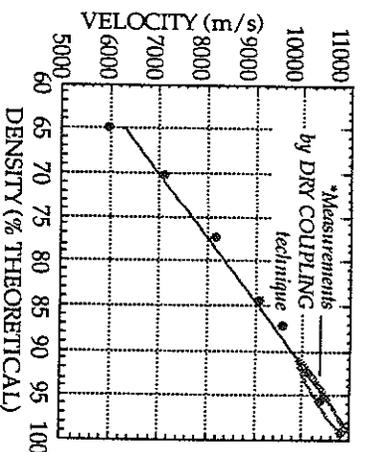

Fig. 10. Relationship between density and non-contact ultrasonic velocity for sintered Al2O3.

*Non-contact technique could not be used reliably for very dense ceramics.

analysis of compositionally variant materials, but also of micro-structurally diverse materials of varying thicknesses.

Green Ceramics

Fig. 11 shows transmitted ultrasonic signals in the rf A-scan mode from a 76mm thick section of ZrO2 brick. As expected, when ultrasound propagates through a defective region, the amplitude of the transmitted ultrasound is substantially decreased. Fig. 12 shows similar signals from a green ceramic multi-layer package.

Sintered Ceramics

Fig. 13 shows transmitted signals from two samples of a porous ZrO2 refractory. Visually both look alike. However, when they were examined ultrasonically, one of them produced results typical of internal cracks. Fig. 14 is the same as Fig. 13, with the observations produced by dry coupling, presented here for comparison purposes.

Fig. 15 shows transmitted signals through various regions in a SiO2 fiber block with porosity >50%. Fig. 16 shows similar signals at various points in a glazed ceramic tile.
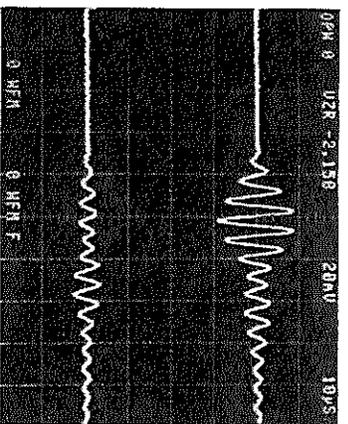
Fig. 11. Green ZrO$_2$ refractory - 76mm. AST: 20mm. ASR: 20mm. Top trace: Defect-free region. Bottom Trace: With 3mm cylindrical hole.
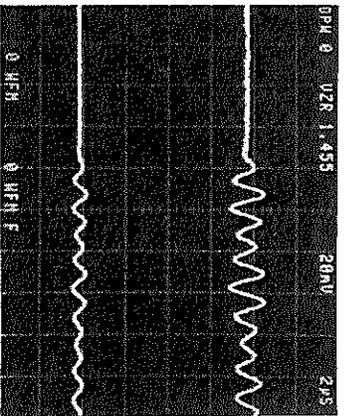

Fig. 12. Green ceramic multi-layer package 4.4mm. AST: 5mm. ASR: 5mm. Top trace: Defect-free region. Bottom trace: Presumably delamination or an internal crack.
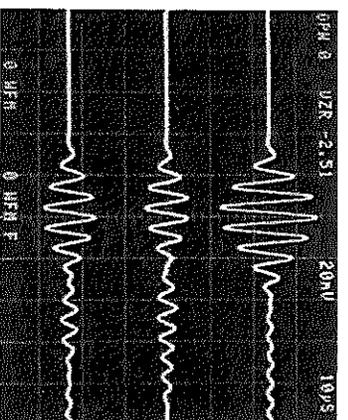

Fig. 13. Sintered ZrO$_2$ refractory - 100mm. AST: 20mm. ASR: 20mm. Top trace: Defect-free sample. Bottom trace: Internally cracked sample.

Fig. 14. Same as Fig. 13, but the analysis is performed by dry coupling for comparison.

Rocks

Fig. 17 shows transmitted signals through a core-drilled sample of sandstone at different locations.

Fig. 15. Transmitted signals at various regions in a SiO$_2$ fiber block - 10mm. Porosity: >50%. AST: 10mm. ASR: 10mm. Significant variations in signal strength could be indicative of density variations or internal defects.
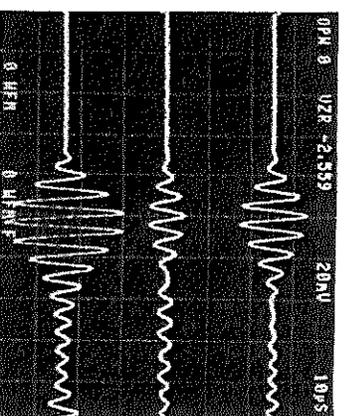

Fig. 16. Transmitted signals at various regions in a glazed ceramic tile - 6mm. AST: 10mm. ASR: 10mm. Significant variations in signal strength could be indicative of tile and/or glaze quality.
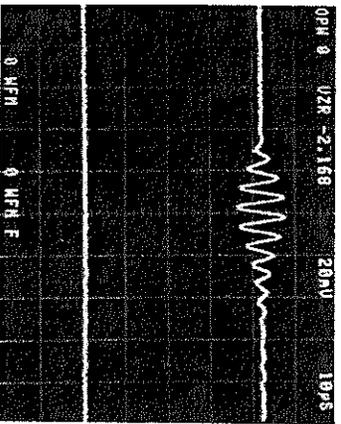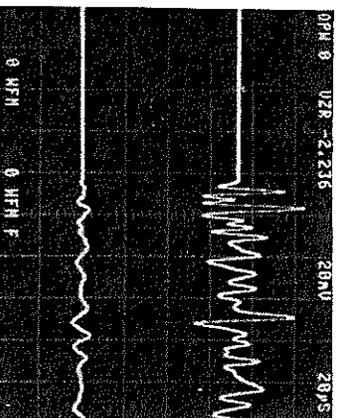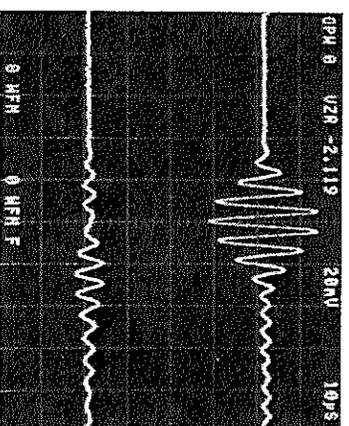

Fig. 17. Transmitted signals through different regions of a core-drilled sample of sandstone - 100mm. Top trace: Defect-free region. Bottom Trace: Presumably a cracked or a different texture region.

Composites

2D Carbon-Carbon disc brakes for large aircraft are rather difficult to characterize nondestructively even by conventional ultrasonic means.
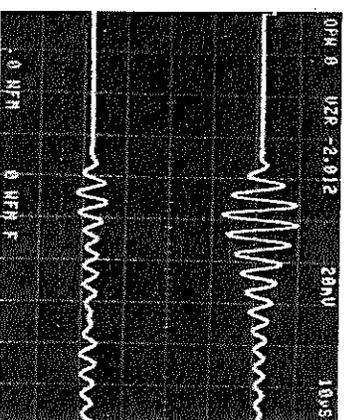

Fig. 18. Transmitted signals through 2D C-C disc brake - 20mm. AST: 20mm. ASR: 20mm. Top trace: Defect-free region Bottom trace: Presumably an internal heterogeneity or a defective region.

To our surprise, we discovered that ultrasound could be easily propagated through this material by non-contact means, Fig. 18.

Fig. 19 shows transmitted signals from a defect-free region and from an impact-damaged region in a 1.5mm 8-ply graphite fiber-reinforced plastic composite. Fig. 20 is a similar example for 6.3mm 12-ply glass fiber-reinforced plastic composite.
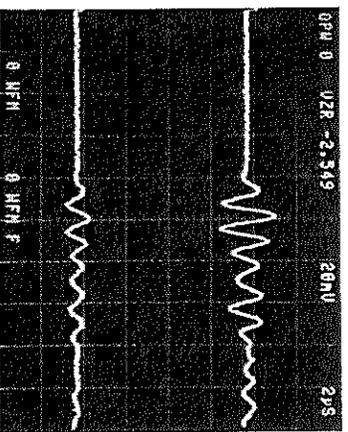
Fig. 19. Transmitted signals through a 1.5mm 8-ply graphite fiber reinforced plastic composite. AST: 5mm. ASR: 5mm. Top trace: Defect-free region. Bottom trace: Impact-damaged region.
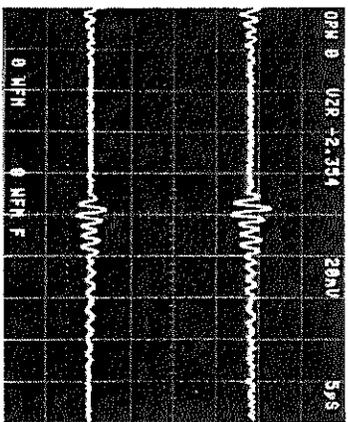
Fig. 20. Transmitted signals through a 6.3mm 12-ply glass fiber reinforced plastic composite. AST: 5mm. ASR: 5mm. Top trace: Defect-free region. Bottom trace: Impact-damaged region.

CONCLUSIONS

In this paper we have described the exciting practical development of non-contact ultrasonic methodology of immense value to the industry and to the society in general. However, this development should not be considered a panacea. This technique cannot replace the high frequency and high resolution ultrasonics necessary for microstructural evaluation and for imaging and microscopy of dense and high acoustic impedance materials. On the other hand, as shown in this paper, non-contact ultrasound can be relatively easily and reliably used for green and for porous materials characterization. In the author's opinion, in many cases, such applications have not been reliably done by conventional non-destructive ultrasonics.

Non-contact ultrasonic applications are naturally limited by the laws of physics. For example, under ambient conditions we are presumably limited to ~5MHz frequency for thin green and sintered ceramics and for thick materials to ~500kHz frequency. Similarly, for gross defect detection and for density-velocity estimations in dense and high velocity materials, the current non-contact methodology is adequate. For such materials, it should not be trusted for small defect detection and for precise velocity-density relationships.

Some of these problems can be minimized by subjecting transducers and test materials to environments of high air pressures, or by using other gases, particularly those that are less attenuative (such as hydrogen or helium). While investigating high velocity materials (dense oxides, carbides, nitrides, borides and diamond and diamond-like materials) by the non-contact ultrasonic technique, one should also be fully conscious of deleterious diffraction effects since the applicable non-contact frequencies are still very low. All these factors are deemed significant and should be evaluated with respect to test material composition and microstructure as well as with the objectives of NDC.

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