SIMPLE ULTRASONIC NDC FOR ADVANCED CERAMICS DEVELOPMENT & MANUFACTURE

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Abstract

Necessitated by the advances in structural and electronic materials, several technologies related to NonDestructive Characterization (NDC) have attracted the attention of the materials community. For both economy and safety, the role of nondestructive evaluation of materials cannot be overestimated.

This paper deals with the significance and applications of *materials-suitable* ultrasonic NDC by utilizing dry coupling techniques. Longitudinal and shear waves velocities determined by these methods have been directly related to elastic properties and microstructures of sintered, green, and porous ceramics without altering their original characteristics during the analytical process.

A few practical examples have been provided in order to aid the establishment of materials' quality and producibility parameters via ultrasonic NDC. Simplicity and accuracy of these methods are proposed for reliable NDC of advanced ceramics, powder metals and their composites.

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Introduction

Ultrasonic characterization is not new. It has been known for nearly 100 years, and has been used as NonDestructive Testing (NDT), primarily in the metals industries. The major goal of NDT is to identify and locate "overt" flaws in materials. Reviews of NDT methods and their applications have been given by McMaster [1] and Krautkramer [2].

Correlations of acoustic measurements (ultrasonic velocities, frequency dependence of ultrasound attenuation, and phase relations of reflected and transmitted ultrasonic signals) with test material properties (density, porosity, intergranular relationships, elastic and mechanical properties) establish the basis for NonDestructive Characterization (NDC), Fig. 1.

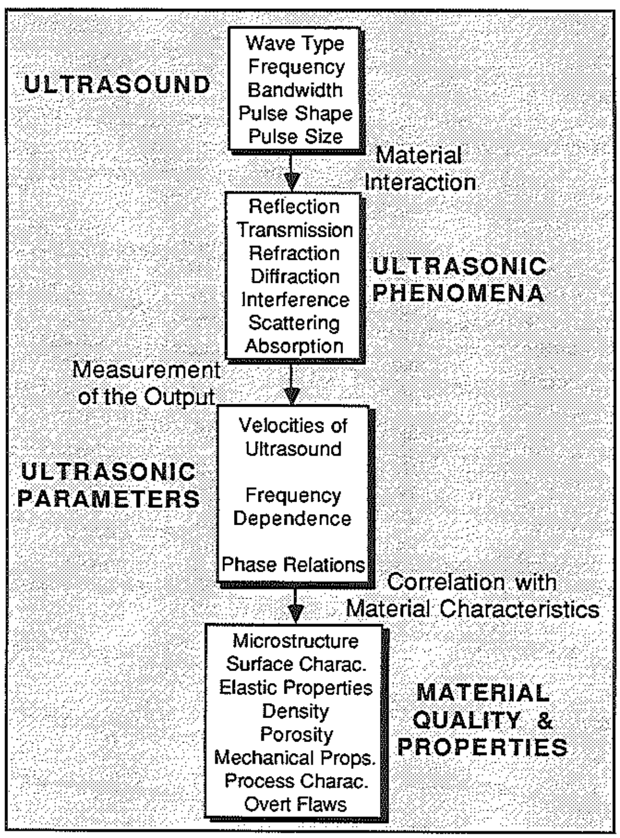

Figure 1 - Principles of ultrasonic NonDestructive Characterization (NDC) - applications of ultrasound-material interaction phenomena for materials quality and properties analysis.

To exhibit the reliability of ultrasound for meaningful NDC, Vary [3], Thompson [4], Bhardwaj [5-7], and others have demonstrated advancements in time and frequency domain analysis of a wide range of industrial materials. While the advantages of nondestructive evaluation are numerous and self-evident, NDC is a relatively new subject. In the following sections we show the systematic characterization of ceramic materials in time domain by applying *materials-suitable acoustics* and *material-suitable methodologies* developed in our laboratory.

Materials-Suitable Acoustics

Materials-suitable acoustics defines critical ultrasonic parameters (frequency, pulse shape and its size) applicable to the inherent characteristics (composition and microstructure) of a given test material. For example, while a 1MHz frequency may be adequate for the analysis of a 20mm coarse-grained fused SiO_2, it is too small for the interrogation of a 1mm dense SiC or Al_2O_3. The wavelength at 1MHz is ~4.0mm for fused SiO_2, and ~11mm for dense SiC or Al_2O_3. Considering that the pulse width of a 1MHz frequency is one wavelength, in the time domain envelope it is equal to 1μs. The time required to propagate ultrasound through a 20mm fused SiO_2 is 20μs; in dense 1.00mm SiC or Al_2O_3 it is only 90ns. Obviously, the pulse width of a 1MHz transducer -1μs - is too large to successfully interrogate dense 1.00mm SiC or Al_2O_3. Simply stated, the higher the velocity of a given test material, the higher the applicable frequency (or shorter pulse width) for its investigation.

Materials-Suitable Methodology

This refers to the applicable physical technique by which a given test material is coupled to an ultrasonic transducer. An impervious material can be coupled by applying a thin layer of oil, grease, or glycerene between the transducer and test material surface. The same cannot be said about green, porous, or liquid-sensitive materials (superconductors, ceramic electrolytes, salt-based compositions, etc.). Obviously, a portion of liquid couplant will penetrate inside the porous material or damage the fragile green material. Conventional liquid coupling is not a desirable ultrasonic method for the characterization of such materials.

When dealing with ceramics and metal matrix composites, we are actually addressing materials characterized by extremes in chemical composition, microstructure and ultrasonic characteristics. While base metals and non-metals (iron, aluminum, silicon, zirconium, etc.) exhibit relatively slower ultrasonic velocities; their oxides, nitrides, carbides and borides are characterized by much higher velocties. For example, the longitudinal wave velocity of zirconium is 4,800m/s, and for dense ZrO_2 it is 7,600m/s. Not only does the chemical composition affect velocities, but they are also greatly influenced by the material microstructure - grain or fiber shape & size, porosity, etc. Since velocity of ultrasound determines the wavelength (as a function of a given frequency), development of velocity-wavelength relationships with respect to ultrasonic detectability and resolution of surfacial and internal defects are extremely significant for proper NDC. Therefore, in order to accurately analyze this diversity of materials, we need to apply acoustically suitable and correct ultrasonic method(s), that will meet the NDC objectives without adversely affecting the test material during the analytical process.

Accurate Measurement of Ultrasonic Velocities

One of the simplest methods of NDC is correlating the measured ultrasonic velocities (longitudinal, shear, and surface waves) to the material characteristics of interest, such as density, texture, elastic properties, and so on. The value of such correlations depends upon the accuracy of the measured velocity.

Dry Coupling Direct Transmission Method

In order to determine ultrasonic velocities in test materials, pulsed ultrasound was used to measure Times-Of-Flight (TOF) by the dry coupling direct transmission method. Excitation of transducers and amplification of transmitted signals were performed by a fully computerized 5ns rise time pulser with a 35MHz amplifier, Fig. 2. When the test materials were too porous or too attenuative this system was interfaced with a high energy ultrasonic system featuring an RF burst pulser and a 3MHz amplifier. Measurements of TOF corresponding to the known distance traveled by ultrasound were made from a Tektronix 2432, a 300MHz digitizing storage oscilloscope.

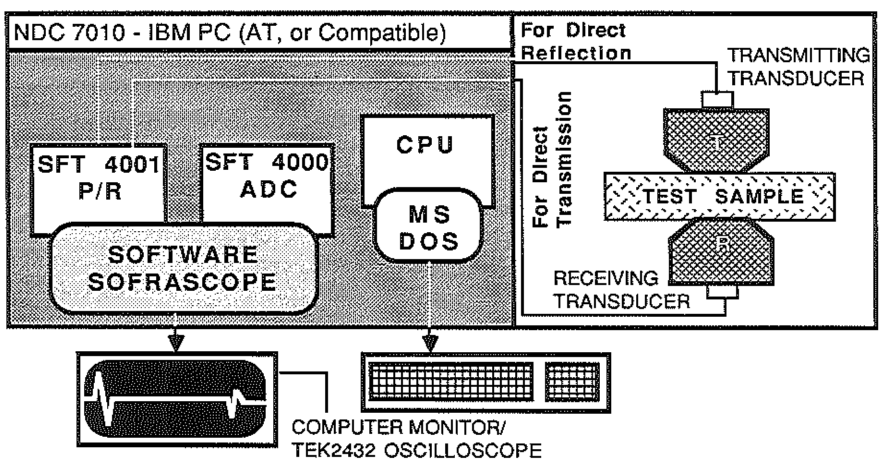

Figure 2 - Pulsed ultrasound setup of NDC 7010 used for the measurement of longitudinal and shear velocities by dry coupling methods.

The mechanism of the TOF measurement included the establishment of a reference signal corresponding to the "zero" position on the oscilloscope, as shown in Fig. 3. Fig. 3a shows the time domain trace of the triggered oscilloscope, i.e., without connecting cables and transducers. In order to establish the "zero" reference point, it is important that the TOF corresponding to the transducer matching/ protective layers be considered, lest an error be introduced in test sample TOF. This is shown in Fig. 3b. Here, the two transducers - transmitter and receiver were placed together with their active regions touching each other. The ensuing transmitted signal in this case corresponds to the TOF through the matching/ protective layers of two transducers. A cursor was placed at the trailing edge of this signal to indicate the zero or the reference point from where TOFs corresponding to test sample travel distance were measured. For example, Fig. 3c shows the appearance of a transmitted signal through a test sample. The right hand cursor of Fig. 3c is placed at the trailing edge of the transmitted signal. The difference between the

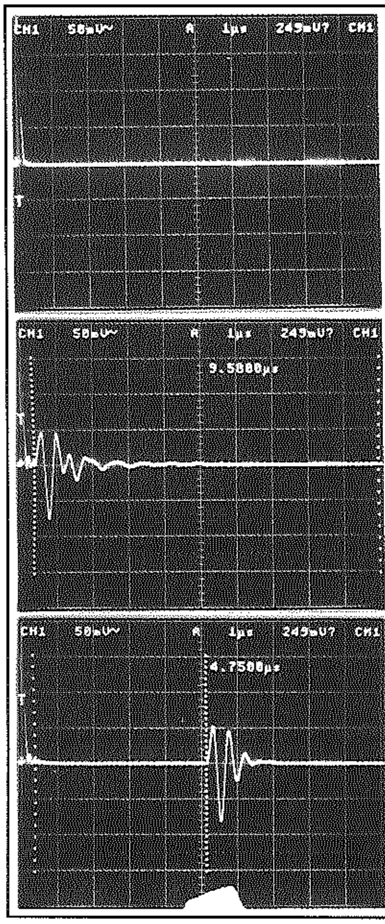

Fig. 3a.
Triggered oscilloscope trace, without transducers.

Fig. 3b.
Oscilloscope trace with two transducers coupled together.

Note the location of left hand cursor, adjusted at the transmitted signal from transducers' matching layers. This cursor establishes a reference or "0" point from where time-of-flight measurements are to be made.

Fig. 3c.
Oscilloscope trace with test material sandwiched between two transducers.

The position of left hand cursor is unchanged. The right hand cursor is placed at the trailing edge of the transmitted signal from the test material.

The actual time-of-flight is automatically measured between the two cursors and displayed on the oscilloscope screen - in the present case it is 4.75µs.

Figure 3 - A sequence for accurate time-of-flight measurement by direct transmission dry coupling mechanism.

two cursors is the true TOF of ultrasound through the test sample, and is directly displayed on the oscilloscope screen.

Limitations of Other Methods

Direct or delayed reflection methods may also be utilized for the measurement of TOF. However, such methods have limitations, particularly when studying relatively porous media. One technique utilizing overlapping of adjacent reflections corresponding to the distance between the top and bottom surfaces of a test specimen for TOF measurement is the *"pulse-echo overlap,"* described by Vary [8]. This technique is only applicable to those ultrasonic signals that are generated by direct reflection and that feature multiple reflections, generally corresponding to the far side of a test sample. However, a wide range of industrial materials (very-dense to very-porous, and chemically and physically heterogeneous samples) cannot be analyzed by *pulse-echo overlap* for accurate TOF measurements. This is due to frequency attenuation and other factors that are likely to curb the usage of direct reflection method, a prerequisite for *pulse-echo overlap*. Furthermore, for extreme accuracy in TOF measurements, it is highly desirable to work within a well-collimated beam of ultrasound. These requirements can be satisfied more easily by using the direct transmission method. The *pulse-echo overlap* technique on relatively thick sections may also introduce unwanted ultrasonic diffraction effects, thus adversely affecting the accuracy of TOF measurements. For materials characterization, a more universal method for accurate TOF measurement is the direct transmission of ultrasound. If, for some practical reasons this method cannot be used, then other methods need to be applied.

Applications of Dry Coupling Ultrasonics

Variable Density/Porosity Ceramics

In order to demonstrate time domain velocity relationships, various samples of sintered alumina* - 51mm diameter and 13mm thick - were studied by the experimental procedure described in the previous section. The porosity of these samples varies from 0.7% to 35.2% (theoretical density from 99.3% to 64.8%). Therefore, they were analyzed by "Dry Coupling" ultrasonic transducers developed in our laboratory. The significance of Dry Coupling over the conventional "Wet Coupling" has been described in detail by Bhardwaj [6] and Brunk, et al. [9]. Table I shows the

Table I - Salient characteristics of Dry Coupling Longitudinal and Shear wave Transducers used for the NDC of alumina.

ALUMINA DENSITY RANGE	DRY COUPLING TRANSDUCERS			
	LONGITUDINAL WAVE		SHEAR WAVE	
	Active Φ (mm)	Frequency (MHz)	Active Φ* (mm)	Frequency (MHz)
100-92.3%	3.0	20.0	6.0	10.0
87.2-77.3%	6.0	10.0	6.0	5.0
70.3-64.8%	12.7	5.0	12.7	2.0
"	12.7	2.0	12.7	1.0

*Φdiameter.

salient characteristics of longitudinal and zero degree incident beam shear wave dry coupling transducers used in this investigation. Fig. 4 shows the

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velocities of longitudinal and shear waves as a function of the density of sintered alumina. These velocities are directly related to the elastic properties of the travel medium as follows:

$$E = V_l^2 \cdot \rho \cdot (1+\sigma) \cdot (1-2\sigma)/(1-\sigma) \qquad (1)$$

$$G = V_t^2 \cdot \rho \qquad (2)$$

$$\sigma = (1 - 2b^2)/(2-2b^2) \text{, where } b = V_t/V_l \qquad (3)$$

$$K = E/[3(1-2\sigma)] \qquad (4)$$

where, V_l = Longitudinal wave velocity, m/s; V_t = Shear wave velocity, m/s; E = Young's modulus, Pa; G = Shear modulus, Pa; K = Bulk modulus, Pa; ρ = Density, Kg/m^3; and σ = Poisson's Ratio.

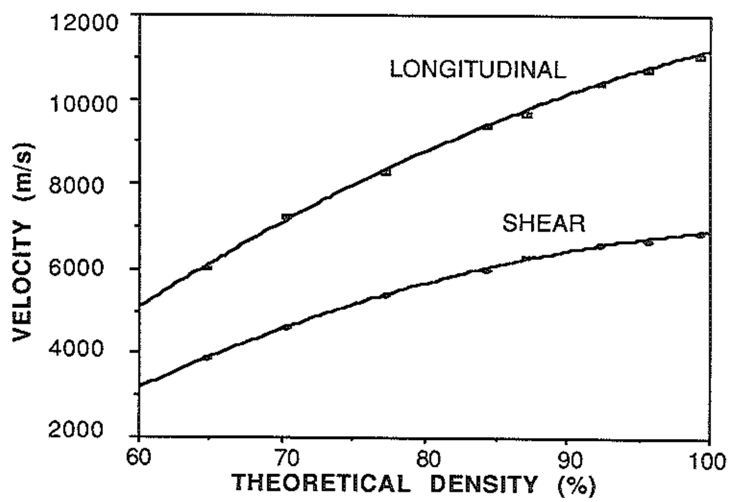

Figure 4 - Relationships between the density and longitudinal and shear wave velocities of sintered alumina determined by direct transmission dry coupling methods. Frequency of longitudinal wave transducers: 10MHz Frequency of shear wave transducers: 5MHz.

Utilizing these relations in conjunction with measured longitudinal and shear wave velocities, by 10MHz and 5MHz frequencies, respectively, Young and shear moduli of elasticity of sintered alumina were determined (Fig. 5). Fig. 6 is the comparison of destructively (static 4-point bending method) and nondestructively (as per the procedures described here) determined Young's modulus of elasticity of sintered alumina. This comparison indicates that ceramics of varying micro-structures and densities can be satisfactorily characterized by simple and easy ultrasonic methods. While this relationship is not novel, it should be noted that by utilizing "dry coupling" ultrasonics (on samples of varying porosity) the accuracy and reliability of NDC is established. Table II shows the comparison of acoustic and elastic properties of sintered alumina

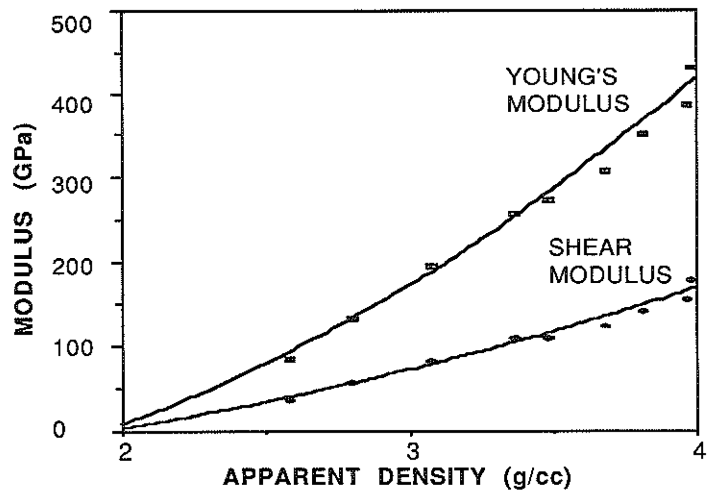

Figure 5 - Young's and shear moduli of elasticity as functions of density of sintered alumina generated from ultrasonic velocities data.

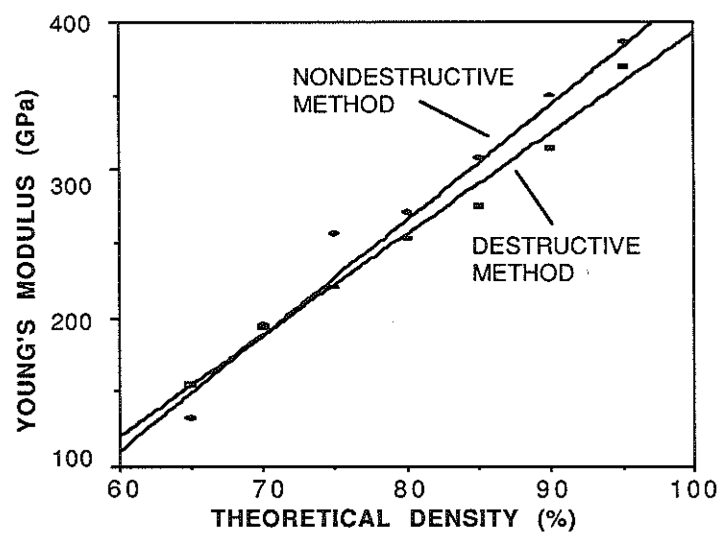

Figure 6 - Comparison of destructively (4-point bending method) and ultrasonically determined Young's modulus of alumina as functions of its density.

Table II - Comparison of acoustic and elastic properties of sintered alumina determined by Dry Coupling (DC) and conventional Wet Coupling (WC) ultrasonic methods. Erratic values of ultrasonic velocities, and thus the elastic properties, by WC method are attributed to unreliable effects of liquid couplant, particularly in the porous materials.

ALUMINA DENSITY g/cc (% Theo)	Vl (m/s)		Vt (m/s)		E (GPa)		G (GPa)		K (GPa)		σ	
	DC	WC	DC	WC	DC	WC	DC	WC	DC	WC	DC	WC
3.98 (100)*	11,055	11,000	6,700	6,600	432	423	178	173	248	250	0.210	0.218
3.96 (99.3)	10,830	10,870	6,270	6,380	388	398	156	161	255	253	0.247	0.238
3.81 (95.6)	10,340	10,530	6,115	6,220	351	363	142	147	216	226	0.230	0.233
3.68 (92.3)	9,915	10,250	5,815	6,025	308	330	124	133	195	208	0.237	0.236
3.48 (87.2)	9,470	9,650	5,700	5,745	272	281	111	114	161	171	0.222	0.226
3.37 (84.5)	9,130	9,225	5,660	5,540	258	252	109	103	134	149	0.181	0.218
3.08 (77.3)	8,280	8,330	5,190	5,070	195	191	83	79	101	108	0.177	0.206
2.80 (70.3)	7,140	7,180	4,500	4,410	133	130	57	54	67	72	0.170	0.197
2.58 (64.8)	5,930	6,080	3,700	3,750	85	87	36	36	41	47	0.164	0.192

*Sapphire - c - crystallographic direction determined by ultrasonic Dry Coupling (DC) and conventional Wet Coupling (WC) methods. This data was obtained by a 10MHz longitudinal wave and 5MHz shear wave transducers. Wet coupling analysis was conducted by applying isopropyl glycol between the longitudinal wave transducer and the test material surface; ordinary honey was used to couple the shear wave transducers. Discrepencies between the longitudinal and shear wave velocities by these methods are apparently caused by the couplant penetration inside the porous samples. Since the dry coupling does not alter the original characteristics of the test materials, it is a more reliable and accurate method for the NDC of microstructurally heterogeneous materials.

Characterization of Green ceramics

Correlation of ultrasonic velocities with compaction pressures or densities of green ceramics can also provide significant information prior to sintering. In order to illustrate the feasibility of green ceramic NDC, several samples of compacted alumina-polymer binder* were analyzed by the procedure described in the experimental section. Fig. 7 shows the relationship of longitudinal and shear wave velocities with compaction pressure of green ceramic. This method can be used for the analysis of green ceramic and powder metal composites for density/velocity variations at discrete points within a given sample or a component.

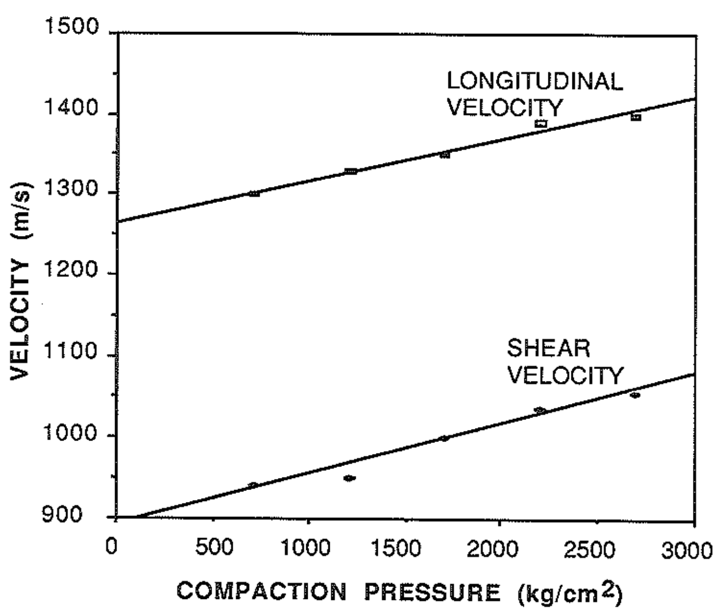

Figure 7 - Relationship between compaction pressure of green alumina and its longitudinal and shear wave velocities established by direct transmission dry coupling methods

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Examples of Ceramic Composites NDC

In the preceding sections we introduced the significance of dry coupling ultrasonics over the conventional "wet coupling" of transducers. By utilizing Al_2O_3 as an example material, we have also provided a systematic procedure for green and sintered materials' NDC for density and elastic properties correlations via longitudinal and shear wave velocity measurements. These procedures can also be applied for the evaluation of ceramic and metal matrix composites. Following examples illustrate specific applications of this method for the NDC of selected fibrous and particulate ceramic composites, significantly varying in densities.

Characterization of Fibrous Composites

Materials such as *rigid porous fibrous ceramic composites* were developed for the protection of the space shuttles. These materials are typically composed of 60% to 85% air and are being considered for other applications requiring thermal protection. In order to evaluate such materials nondestructively, both longitudinal and shear wave dry coupling velocity measurement techniques were applied by using 0.5MHz and 1.0MHz frequencies. By the application of *minimal* pressure on dry coupling transducers with the test sample sandwiched in-between, it was relatively easy to propagate longitudinal but not shear waves through such materials.

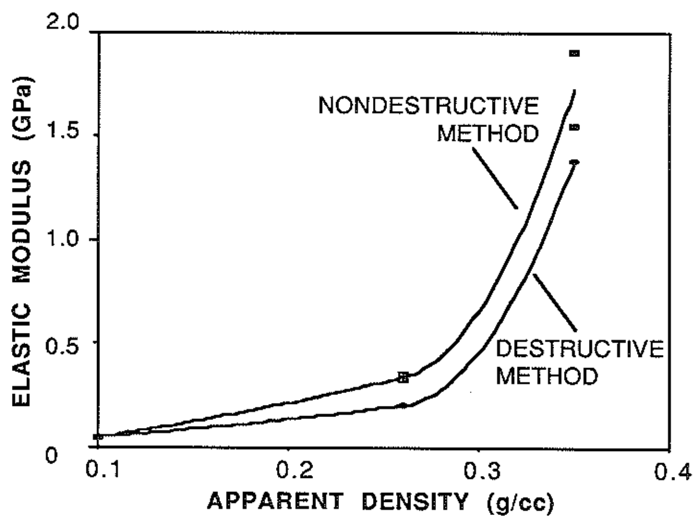

Figure 8 - Comparison of destructively and nondestructively determined elastic moduli of "ultra" porous rigid ceramics in thickness direction as functions of density.
Nondestructive method: Direct transmission dry coupling.

Provided by the Lockheed California Co.

Fig. 8 shows the comparison of elastic moduli determined from longitudinal wave velocities and conventional destructive testing, Banas et al. [10]. These observations indicate that reliable NDC of ultraporous materials is both feasible and relatively easy. However, attempts for dry coupling NDC of cellular ceramic composites, such as those used for liquid metal sieving, generated inconclusive results.

Characterization of particulate ceramic composites

Table III shows salient acoustic characteristics and elastic properties of sintered SiC and Si_3N_4 particulate composites determined through the correlation with longitudinal and shear wave velocities. As a reference similar parameters of single phase SiC and Si_3N_4 are also shown.

Table III. Ultrasonically determined elastic properties of single and multi phase SiC and Si_3N_4 particulate composites.

MATERIAL	DENSITY (g/cc)	VELOCITY (m/s)		ELASTIC MODULI (GPa)			POISSON'S RATIO
		LONGITUDINAL	SHEAR	E	G	K	σ
SiC - Single Phase[1]	3.01	11,500	7,320	374	161	184	0.161
	3.09	11,800	7,530	406	175	198	0.159
	3.15	12,050	7,700	431	186	210	0.157
SiC - Composite[2]	2.37	7,480	4,640	121	51.1	64.8	0.186
	2.40	7,540	4,750	126	54.0	64.2	0.172
Si_3N_4 - Single Phase[3]	3.05	11,050	6,225	299	118	215	0.268
Si_3N_4 - Composite[2]	2.30	6,390	4,010	86.6	36.9	44.3	0.174
	2.35	6,540	4,020	90.8	38.0	50.0	0.196

[1]SOHIO Engineered Materials, Niagara Falls, NY.
[2]Composition and source withheld at the request of the material developer.
[3]Commercial sample from Norton Co., Northboro, MA.

These observations exhibit the sensitivity of longitudinal and shear wave velocities toward subtle variations in test material densities, thus providing an accurate mechanism for material property determination. Through the applications of relatively high ultrasonic frequencies and conventional wet coupling, similar observations on dense SiC have been reported by Generazio et. al. [11]. They have further shown the sensitivity of ultrasonic NDC over x-radiography.

Conclusions

In this paper we have described simple and significant developments in ultrasonic NDC science and technology for materials property determination and for microstructural evaluation. We have also outlined important ultrasonic considerations, such as the relationship of transducer characteristics with respect to defects, properties, and microstructural characterization of materials. A few practical examples have been provided in order to demonstrate the applicability of modern ultrasound as a reliable tool for nondestructive characterization of green and sintered ceramics and their composites. The scope of our presentation will be undoubtedly enhanced by its applications at QC/QA and materials development functions.

Acknowledgements

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