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ULTRASONIC CHARACTERIZATION AND SPECTROSOPIC ANALYSIS OF CERAMIC MATERIALS

V. K. Ravindran, Binu P. Thomas***
R. Sunderasan and B. C. Bhaumik**
**QRPG/SR, **EXMD/SDEG*
Vikram Sarabhai Space Centre
Thiruvananthapuram

ABSTRACT

Ceramic materials have an important and critical role in the thermostructural applications. In rocketry these critical materials are required in only small quantities. However they are required to be characterized in total to make rocket performance predictable to the finest details. If local variation exists in the material, non-uniform behaviour of the material results in performance variation. Non destructive material characterization enables one to find out the local variation of the material property very effectively. This paper describes the development of an ultrasonic spectroscopic methodology and application of advanced existing methodologies for ceramic materials. The results are discussed in details with tables, graphs, and theoretical analysis.

1. INTRODUCTION

Nondestructive characterization is essential wherever complete material volume evaluation is called for. Ultrasonic methodologies are the most practically feasible nondestructive characterization application in the area of material characterization. This is mainly because ultrasonic methodologies are non hazardous, applicable in small samples to very large samples, mechanical / physical properties of material can be very easily related to features of ultrasound through well established theories and empirical relations , availability of low cost equipments where full use of present day computing capability can be made.

2. PROPERTIES OF CERAMIC MATERIALS

Features of ultrasound and properties of materials through which sound travel can be related empirically as well as theoretically. It is difficult to establish empirical relationships for immediate practical purposes because it would require sample preparation and testing in large numbers in optimum conditions. Theoretical relations on the other hand can be easily applied.

Material density is empirically related to velocity and attenuation of ultrasound in the material [1]

$$D = a + bv + c\alpha \qquad (1)$$

D - density
v - velocity of ultrasound in the material
α - attenuation
a, b and c - constants

Modulus of elasticity of material is related theoretically to features of ultrasound

$$E \approx v^2 d \qquad (2)$$

v - velocity of ultrasound
d - density

Another important characteristic of the material that can be related to features of ultrasound is the grain size or porosity size in the material. The scattering theory established that when wavelength of sound is closer to the size of scatterers in the material (pore / grain) it is more difficult to transmit sound through the material. When the wavelength is below ten times the grain size, it is almost impossible to transmit the sound [2]

3. ULTRASONIC NDE OF CERAMIC MATERIALS

Unlike metals, ceramic materials are rarely tested in conventional NDE laboratories. Hence the available knowledge and expertise in this respect is poor. However literature available indicate that certain ceramic materials transmit ultrasound with very high speed.
The relation between velocity and wavelength of in a material is

$$v = f\lambda$$
$$\text{i.e., } \lambda = v/f \qquad (3)$$

v - velocity
f - frequency
λ - wavelength

Generally, velocity of sound is considered as constant for a material. Thus when frequency increases wavelength decreases. For same frequency, in these ceramic materials, which are having very high sound velocity, compared to conventional metals, wavelength is very large. Thus according to scattering theory, these ceramic materials exhibit less scattering, which enables the use of high frequencies for NDE. Thus conventional NDE laboratories can be very effectively utilized for NDE of ceramic materials.

Certain ceramic materials like ceramic cement compositions can also be tested because these are comparable to evaluation of cement structures, which are widely evaluated using ultrasonic methodology.

4. ULTRASONIC SPECTROSCOPY

As detailed in section 2 above, frequency analysis of ultrasound is essential to study the material structure like porosity, grain size and their distribution in material volumes. For this, spectrums of frequencies (with known profile) are sent through the material. This sound spectrum after passing through the material will be analyzed to locate the transmissibility of different frequencies through the material. If a frequency is located beyond which sound is not transmitted an estimate of grain size / porosity size can be made very easily. (Fig. 1)

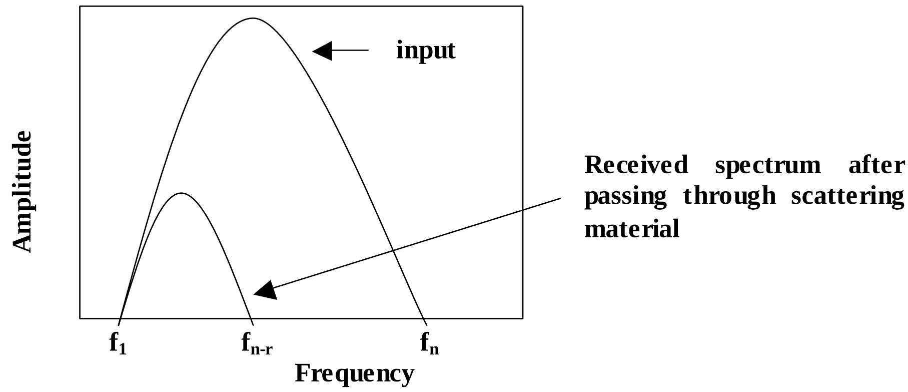

Fig. 1 Frequency spectrum before and after transmission through scattering material

As seen in fig. 1 the transducer produces a range of frequency f_1 to f_n. Corresponding to this range of frequencies there is a range of wavelengths λ_1 to λ_n in the material (equation 3). When higher frequencies (smaller wavelength) encounter scatterers (grain) of size $1/10^{th}$ of λ they get scattered fully and are lost from the pulse. If the frequency f_{n-r}, (fig. 1) is the highest frequency that can be transmitted in a material, it can be estimated that there are sufficient scatterers in the material of size of $1/10^{th}$ of λ_{n-r}.

5. SOFTWARE DEVELOPMENT FOR SPECTROSCOPY.

For practical applications of material characterization a large number of signals are to be analyzed. Thus the development of an user-friendly software is essential for spectral analysis and measurement of attenuation at each location in the material. This was done by the authors and the software developed provides all operational features suitable for ultrasonic NDE (fig. 2). The main features of this software which is developed under WINDOWS environment includes plotting of signals, selecting the range to be processed using a window, plotting of frequency spectrum, superimposing of the spectrum and calculating different parameters like energy, attenuation etc. from the spectrum.

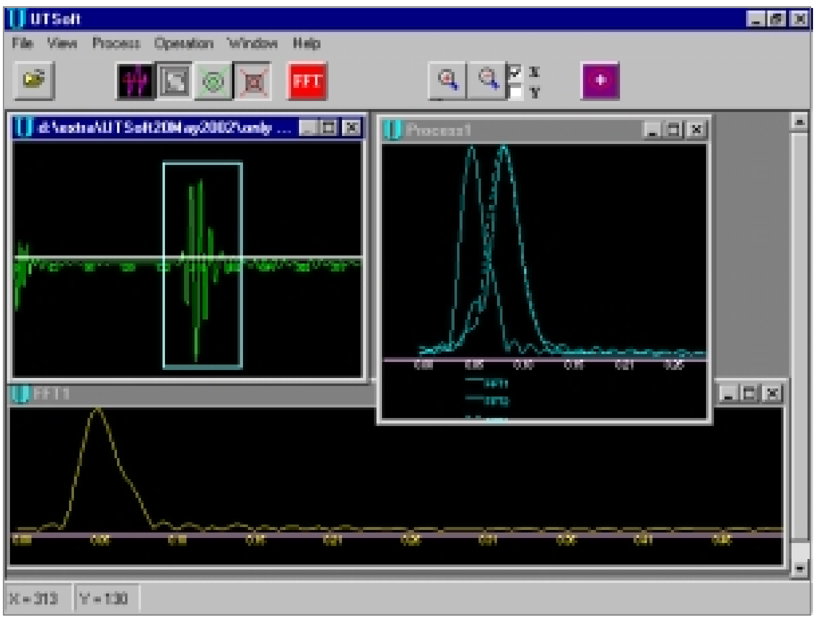

Fig. 2 Software Environment

6. SPECIMEN PREPARATION

Not much effort is required to prepare specimen for finding constants in equation 1. In equation 2, there is no constant for finding through experimentation. However, for understanding the effects of scatterer size on frequency dependent attenuation (fig. 1) controlled specimen are required to be prepared. A method was evolved by the authors to prepare specimens with, known scatterer size embedded in it.

Specimens are prepared using ceramic cement with known size of sand in it. During preparation of ceramic cement block, only scatterer particle size was varied between blocks. (Table 1)

Sl. No	Specimen details	Quantity	Sand size
1	Only ceramic cement	4 Nos.	Nil
2	Fine sand specimen	4 Nos.	0.2 mm
3	Coarse sand specimen	4 Nos.	0.5 mm to 1.0 mm

Specimens with different densities of Alumina are selected for finding constants in equation 1.

7. DATA COLLECTION AND EVALUATION

Table 2 gives the values of ultrasonic parameters collected for finding the constant in equation 1

Table 2. Parameters for finding constants in equation 1

Sl. No.	Specimen	Attenuation dB/mm	Velocity m/s	Measured density g/cc
1	Alumina	0.445	10614	3.85
2	Alumina	0.505	10588	3.80
3	Alumina	0.6608	9782	3.63

Constants $a = +2.328729$
$b = +0.0001515026$
$c = -0.307373$

Tables 3 to 5 gives the parameters measured with respective ceramic cement blocks.

Table 3. Cement only blocks

Time taken by sound to travel microseconds	Sound amplitude %FSH at gain 65dB	Velocity mm / microsecond
18.07	99	2.636
17.197	69	2.77
17.497	73	2.726
16.997	56	2.800

Table 4. Blocks using fine grain of sand

Time taken by sound to travel microseconds	Sound amplitude %FSH at gain 65dB	Velocity mm / microsecond
16.797	16	2.84
16.697	14	2.85
17.097	25	2.78
16.897	27	2.82

Table 5. Blocks using coarse grain sand

Time taken by sound to travel microseconds	Sound amplitude %FSH at gain 75dB	Velocity mm / microsecond
14.480	11	3.294
14.598	17	3.267
14.490	21	3.290
14.698	12	3.245
14.390	17	3.314

Table 6 gives the density and Table 7 gives the modulus of elasticity calculated using equation 2

Table 6. Density of blocks

Specimen	Weight gm	Volume m^3	Density gm/cc
Cement only	147.8852	0.1046	1.414
Fine grained	166.8384	0.1056	1.595
Coarse grained	180.8050	0.1046	1.729

Table 7. Modulus of elasticity

Specimen	Velocity m/s	Modulus of elasticity MPa
Cement only	2733	10561.58
Fine grained	2823	12711.08
Coarse grained	3282	18623.97

Fig. 3 shows the spectral display of the sound after passing through the various ceramic cement blocks. The frequency spectrum of the ultrasound signals gives the highest frequency that can be transmitted through each specimen. The corresponding wavelength can be calculated using equation 3. According to scattering theory, the grain size is equal to $1/10^{th}$ of the above wavelength.

Table 8 gives the grain size (scatter particle) estimated from the specimens.

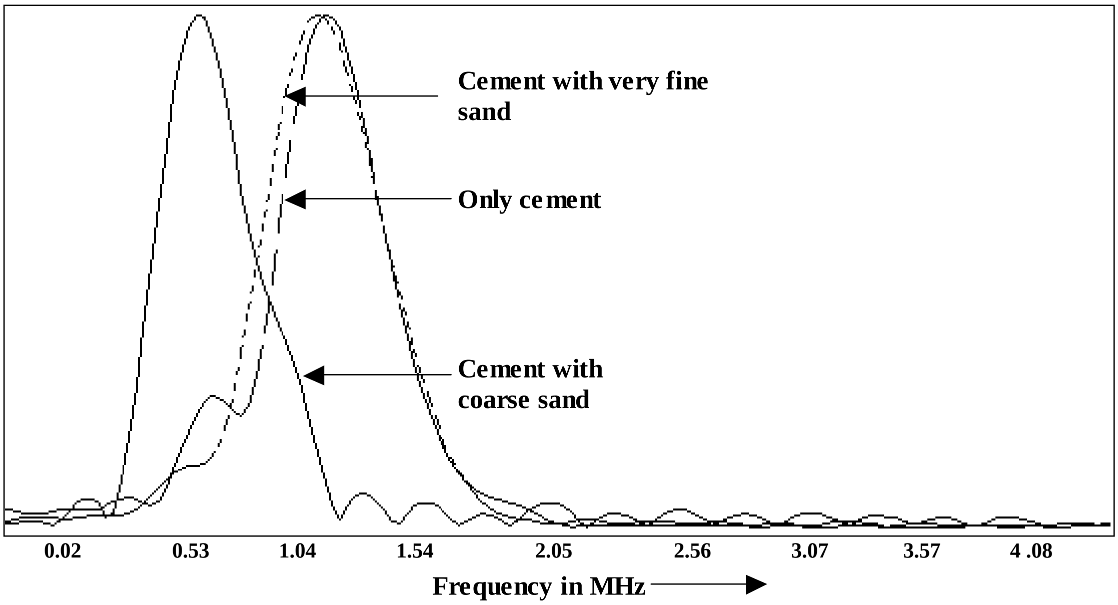

Fig. 3 Ultrasonic spectroscopic analysis of ceramic cement blocks

Table 8. Grain size.

Specimen	Velocity mm/µs	Highest frequency in the spectra MHz	Wavelength mm	Grain size mm
Cement only	2.733	1.43	1.91	0.191
Fine grained	2.823	1.24	2.27	0.227
Coarse grained	3.282	0.95 0.30(peak)	3.45 10.94	0.345 1.094

8. DISCUSSION OF THE RESULT

From the result, it can be seen that ceramic materials can be very easily characterized using ultrasonic NDE. Scatter particle sizes estimated using the methodology developed by the authors are having very good match with the known sizes (Table 8 and fig. 3).

From the other tables of ceramic cement blocks tables 3 to 7 it can be seen that variation in material structure can be very easily detected by ultrasonic features like velocity, attenuation etc. Using equation 1, density measurements were also done for mapping the density variations.

9. CONCLUSION

Ultrasonic non-destructive material characterization is very effective in complete material evaluation of ceramics having fine-grained Alumina to coarse-grained ceramic cement. This methodology can very easily locate non-uniformity in the material with normal conditions of specimen that are practiced in shop floor. It can also be used for estimation of elastic modulus as well as grain size (scatterer size).

10. REFERENCE

1. Klaus Goebbels, *Material characterization for process control and product conformity*, CRC Press
2. Metals Handbook, vol. 17 NDE and Quality Control