EVALUATION OF THE STRUCTURAL INTEGRITY
OF GRANULAR COMPOSITES BY ULTRASONIC TECHNIQUES

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ABSTRACT

The development of the mechanical strength of composite materials which are consolidated by cementaceous bonds has been monitored by observation of the ultrasonic properties over the evolution of bond growth. The granular nature and weak bonding of these materials produce high attenuation of the ultrasonic signal and observation is difficult, particularly in larger structures. In order to obtain ultrasonic parameters with high resolution and to overcome the effects of grain scattering, a system based on a minicomputer and array processor has been implemented which provides time and frequency-domain processing and aperture synthesis. This ultrasonic data provides a reliable measure of the structural integrity of these materials.

INTRODUCTION

The quality of composite materials has been traditionally evaluated by the measurement of compressive strength (ASTM-C39). This technique is well established, but is destructive, requires considerable replication, and is of limited value in determining local inhomogeneity and structure. Ultrasonic techniques can provide a non-destructive method of analysis and have been used on larger structures as in the concrete industry to predict the viability of structural materials. A class of composite materials consisting of fly-ash and a form of gypsum (CaSO₄) which are bonded into a solid by various cementaceous reactions are currently under study to determine the feasibility of using this process for the ocean disposal of waste from coal-fired power plants. This program involves a five-year study of the integrity of these materials both in the laboratory and on a large scale at a test-site (reef) in the ocean.

Although ultrasonic methods offer promise in providing a method of non-destructive evaluation of these materials, it was found that excessive ultrasonic attenuation due to grain scattering and bond softening may be present and that adequate correlation of ultrasonic properties to the "actual" compressive strengths of the material would have to be developed within the program.

We report here on the first phase of this program in which the materials are characterized ultrasonically by a system which is capable of measuring these properties at three levels. Our results show that the ultrasonic modulus provides a sensitive indication of the compressive strength of these materials (within the statistical limits of the fracture process). Furthermore, the modulus is shown to be a sensitive indicator of the growth of the cementaceous bond and hydration process.

MATERIAL STRUCTURE

Figure 1 is an SEM of the internal structure of a test block of fly-ash and scrubber sludge (CaSO₄) mixed in a mass ratio of 3 : 1 which has undergone consolidation in sea water for a period of 6 months. The sphericules are fly-ash granules, while the scrubber sludge appears as an amorphous intergranular mass. Two other structural forms are evident and appear only in consolidated materials. The small crystals are believed to be ettringite while the larger crystallites, more evident in Fig. 2, are known to be gypsum (CaSO₄ • 2H₂O). These micrographs illustrate the complexity of the material under study. We believe at this time that the increase of modulus of these materials is entirely attributable to the growth of these crystallites in the intergranular voids of the block.

INSTRUMENTATION

The apparatus developed for the detailed evaluation of ultrasonic properties of these materials is shown in Fig. 3. A test tank is provided which mounts a pair of ultrasonic transducers. The receiving transducer is under the control of a mechanical translation device which makes it possible to position the receiving array precisely over a range of 50 cm. The fluid media, in this case water, provides an effective coupling medium and facilitates the rapid interchange of test samples. All work reported here was carried out at 162 kHz which was found to be the best compromise between grain scattering attenuation and resolution.

The ultrasonic system is under control of a PDP 11/34 computer which also provides for storage, processing, and display of the voluminous data. The system can be used as either an intervalometer (time domain) or dispersometer (frequency domain). The mechanical translation system makes it possible to use synthetic aperture analysis to obtain additional resolution and discrimination against multiple path effects. After amplification and filtering, the received ultrasonic data can be sent directly to a dedicated 100 KHz A/D converter, or to a high-speed sample-and-hold circuit under control of a synchronizer. The second alternative provides a method of digitizing repetitive ultrasonic signals of much higher bandwidths at the expense of longer acquisition time.

Control software has been developed so that the extensive processing required can be carried out optionally either in software or through use of an array processor (Model MSP-3 manufactured by Computer Design and Applications Inc.). This feature not only makes it possible to retain greater functional control of the process, but provides software backup (at slower speeds) in the event of
limited hardware failure. The logical flow of this process is illustrated in Fig. 4. At each step in the movement of data through the ultrasonic test system, a given process, such as synthetic aperture processing, may or may not be used at the discretion of the operator. If single channel operation were desired, as in low resolution intervalometer work, the data would be passed directly to time domain analysis or storage.

RESULTS

The velocity evolution of two groups of six blocks, with each group having a 1 : 1 (E2Cl) or a 3 : 1 (E2C3) fly-ash to sludge concentration is illustrated in Fig. 5. These blocks were aged in laboratory tanks of sea water held at room temperature for more than a year. The blocks were periodically measured for ultrasonic velocity, attenuation, and density. It will be noted that the initial velocities of both compositions were nearly the same, but the growth in velocity for the 1 : 1 material was more rapid and achieved higher values. Since the change in density in this process is small, the increase in velocity is attributable to a growth in the modulus of the composite material, which in turn is due to cementation or crystalline growth in the voids.

This observation is supported by a concurrent study of the density of these materials over the same time period as illustrated in Fig. 6. Our observations indicate that increases in density due to the uptake of water into the pores of these materials is complete in 1 - 2 hours after placement in the tank. The growth in density indicated in Fig. 6 is primarily due to chemical hydration. Hence, crystalline growth is correlated with the evolution of the ultrasonic modulus.

It is highly desirable to use these ultrasonic methods under in-situ conditions to predict the structural integrity of composite materials. An evaluation of a large number and variety of blocks was carried out in which the blocks were aged in the laboratory or at sea, measured for ultrasonic velocity, and then tested for compressive strength. These results, after calculating the modulus of each material group from velocity data, are displayed in Fig. 7. The correlation between the compressive strength and the modulus of these materials is indicated by these data. It should be noted that each data point in this figure represents the average of the compressive strength of several blocks. The standard deviation for compressive strength on these materials is high (25 - 50%) due to the inhomogeneity of the blocks and statistical nature of the fracture process.

The results of these studies provide guidelines for the estimation and prediction of the compressive strength of composite materials of this type. Such techniques are not only invaluable for field studies of these and similar engineering materials but also provide a precision method for the controlled study of the cementation process which should be applicable to a wide variety of materials.

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REFERENCES


Fig. 1 The internal structure of a composite with a fly-ash to scrubber sludge mass ratio of 3:1 and 5% lime which has been aged in sea water for six months. The fly-ash granules are spheroids. Scanning electron micrograph (SEM) at x = 2600.

Fig. 2 Same material as in Fig. 1 in area that emphasizes the growth of crystallites in the inter-granular voids. The larger crystal is gypsum while the needle-like structure may be ettringite (see text). (x = 3200).
Fig. 3 Laboratory system with dedicated computer facility for the acquisition and processing of ultrasonic data. The sample-and-hold system makes possible the analysis of frequencies to 10 mHz.

Fig. 4 Conceptual organization of the analysis process. The system may be operated as an intervalometer and dispersometer.
Fig. 5 The velocity evolution of two material compositions aged in laboratory tanks. The more rapid cementation of the 1:1 material (E2C1) is clearly indicated by the increase in velocity associated with the increasing elastic modulus of this material.

Fig. 6 The density evolution of the same test group as that in Fig. 5. The density increase in the E2C1 group is attributable to crystallite growth through hydration resulting in cementation and increase in elastic modulus.
Fig. 7 Correlation of the average compressive strengths of groups of materials (consolidated in sea water for various times) to their elastic moduli as determined by ultrasonic data at 162 kHz. This correlation forms a basis for the prediction of the integrity of materials of similar composition from ultrasonic velocity data.