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Characterization of heterogeneity in concrete and cement by mechanical spectroscopy

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Abstract

We propose a method to obtain the degree of heterogeneity of samples of cement or concrete due to the presence of cracks, bubbles or simply the native ingredients of the material. A number of samples are prepared in a cylindrical shape, and their transverse vibration resonant frequencies are measured. A given mode of oscillation will correspond to slightly different frequencies in different samples due to the random nature of the system. For example, for a given mixing formula, the ratio of sand to cement may be known, but the precise position of sand grain cannot be determined. We studied the statistical distributions of frequencies for each mode and found that there exists a relationship between the width of the probability functions and the degree of heterogeneity.

Keywords: Particle size distribution; Microstructure; Pore size distribution; Characterization; Spectroscopy

1. Mechanical spectroscopy

In the past, we have studied the time evolution of concrete as a function of hardening stage. The nondestructive technique used (Fig. 1), *mechanical spectroscopy*, is based on the propagation of resonant audio frequency modes of oscillation along the long axis of cylindrical samples. This technique, together with a theoretical model [1], is similar to standard C 215 of the American Society for Testing and Materials (ASTM) [2], with the advantages of being simpler, cheaper and possible to do with the equipment available at any university physics laboratory.

An audio-generator-fed piezoelectric transducer—the actuator—at one end of the rod excites vibrations in the sample. Off resonance, these vibrations do not form standing waves. When a resonance is attained, the vibration extends all over the bar. A second mechano-electric transducer—the sensor—is placed at the other extreme of the cylinder.

To measure the resonant frequency, we connect the two transducers to an oscilloscope in the x–y mode and search for simple Lissajous figures. This parameter provides an indirect measurement of the Young's modulus, which was measured once a day during the 28-day hardening time.

A thorough mathematical analysis that allows linking the vibrational frequencies of the sample with its average elastic constants has been developed by the authors [3,4]. In this paper, we use those results and extend them to account for local density fluctuations and their concomitant fluctuations of elastic constants about its average. Due to the many degrees of freedom involved and the intrinsic random nature of this system, this problem is solved numerically.

The method that we propose here can be used to study the heterogeneity of the concrete sample in terms of the curing stage. This application is essential to a deep understanding of the curing process itself and to characterize the conditions under which undesirable effects, such as cracks, are reduced to a minimum.

2. Characterization of the degree of heterogeneity

Our current goal is to extend this characterization technique to provide information about the components that

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Fig. 1. Experimental setup used to validate the theoretical modeling. A cylindrical bar of concrete is mechanically excited by acoustic transducers. The transmission down the bar is detected by a second transducer. The voltage from both transducers is combined to form Lissajous figures on the oscilloscope screen.

make up the sample. For this purpose, we have made samples with various contents of gravel, sand and cement, and we have measured their frequency spectra.

The *statistics* of these spectra provides information about elemental elastic constants, relative content and density fluctuations of the constituents. In this work, we present a theoretical model to obtain the density fluctuations of inclusions in cementitious materials.

Previous theoretical studies exist [5,6] in which spherical inclusions to define an effective elastic medium were present in an otherwise uniform matrix. We extend those results beyond effective medium theory and we consider explicitly the heterogeneous nature of the sample.

3. Model system

We study the random frequency spectra of 50,000 samples obtained from the same statistical ensemble. Fig. 2 shows a schematic representation of a typical sample.

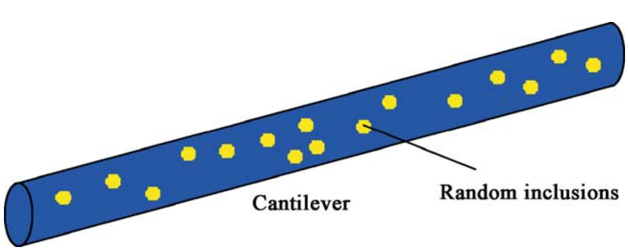

Fig. 2. Cylindrical sample under study with randomly located inclusions.

```
e = Table[Table[Random[Real, {1 - 1/6, 1 + 1/6}],
    {i, Nmax + 2}], {k, Nsystems}];
```

Fig. 3. The parameter *e* is the relative Young's modulus at a given location *i* along the lever. Nsystems in our study was 50,000, the size of the ensemble.

Each sample is characterized numerically. First, the sample is divided in many small slices. Each slice is assigned a random elastic modulus according to

$$E = \langle E \rangle \mathrm{N}(1, \sigma)$$

where $\langle E \rangle$ is the average elastic constant, typically about 30 GPa, and N(1,σ) is the normal distribution with mean 1 and variance σ^2.

In Fig. 3, we show a statement in Mathematica to generate Nsystems random values for the elastic modulus.

Fig. 4 introduces the definition of the matrix **Γ** as entered into the Mathematica code.

We introduce a dimensionless frequency squared by:

$$\mu \equiv \frac{2}{3\pi} \frac{ML^3}{N_{\max}^4 a^4 \langle E \rangle} \omega^2$$

where M is the mass of the bar, L is the length of the bar, a is its transverse radius, ω is the normal mode frequency and $N_{\max}$ is the number of sections along the bar.

The solution to

$$|\Gamma| = 0$$

provides the values of μ and thus the resonant frequencies of the system.

```
Γ = Table[Table[

    If[(j - i) == -2, e[[k]][[i - 1]],

     If [(j - i) == -1,
      -μ - 2 (e[[k]][[i]] + e[[k]][[i - 1]]),

      If[j == i,
       (4 e[[k]][[i]] + e[[k]][[i - 1]] +
          e[[k]][[i + 1]]) - 4 μ,

       If [(j - i) == 1,
        -μ - 2 (e[[k]][[i]] + e[[k]][[i + 1]]),
        If[(j - i) == 2, e[[k]][[i + 1]],
         0]   ] ] ] ],

    {j, 2, Nmax + 1}, {i, 2, Nmax + 1}],
   {k, Nsystems}];
```

Fig. 4. The matrix **Γ** corresponds to the eigenvalue problem of coupled regions with locally random elastic properties. The frequencies are obtained through the dimensionless parameter μ as defined in the text.

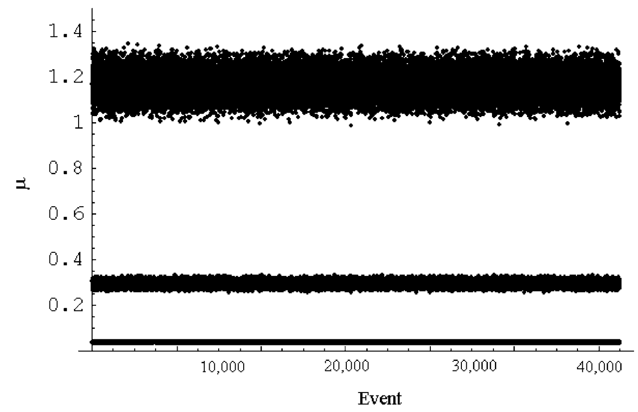

Fig. 5. First three lower normalized frequencies squared for 50,000 samples.

For a few simple cases, we first checked the program to make sure that the frequencies that it generated were reasonable.

For example, a typical matrix with $N_{\max} = 5$ is

$$\Gamma = \begin{pmatrix} 6.0425 - 4\mu & -4.24576 - \mu & 1.11112 & 0 & 0 \\ -4.24576 - \mu & 6.53178 - 4\mu & -4.37332 - \mu & 1.07554 & 0 \\ 1.11112 & -4.37332 - \mu & 6.55049 - 4\mu & -4.4255 - \mu & 1.13721 \\ 0 & 1.07554 & -4.4255 - \mu & 6.737 - 4\mu & -4.49967 - \mu \\ 0 & 0 & 1.13721 & -4.49967 - \mu & 6.64053 - 4\mu \end{pmatrix}$$

After solving the eigenvalue problem for many bars, we construct a plot of μ versus event (Fig. 5).

We make a histogram of the data above and fit it to an analytical probability distribution as shown in Fig. 6.

The curve in Fig. 6 is given by

$$P(\mu) = .89e^{-\left(\frac{\mu-0.03}{0.0025}\right)^2} + 0.6e^{-\left(\frac{\mu-0.19}{0.02}\right)^2} + 1.37e^{-\left(\frac{\mu-0.78}{0.065}\right)}$$

When we use a continuous model for the bar, the first few frequencies are given by

$$\mu_1 = 0.033 \quad \mu_2 = 0.17 \quad \mu_3 = 0.76$$

in excellent agreement with the numerical values that we obtained experimentally.

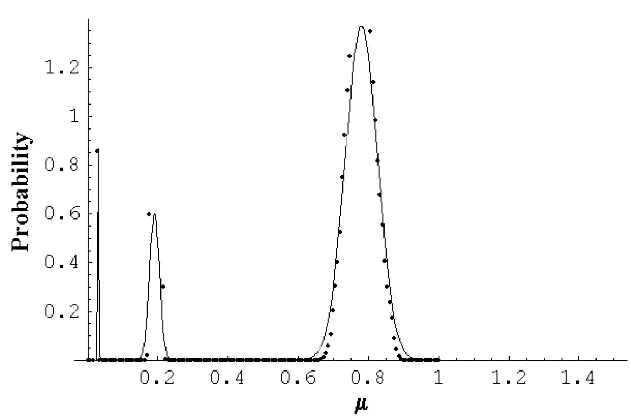

Fig. 6. Probability distributions of the lower three frequencies.

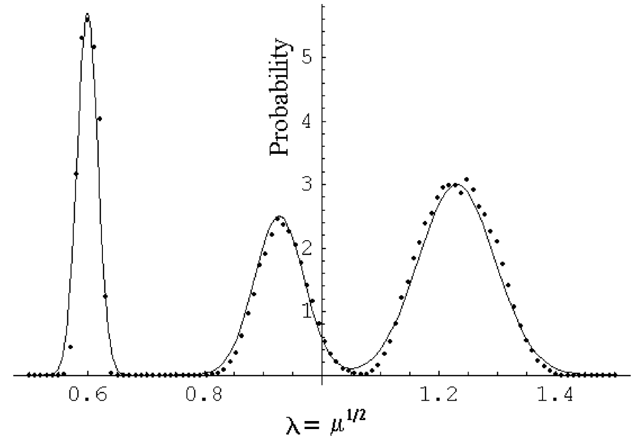

Fig. 7. Probability distribution for the level differences.

Finally, we perform a statistical analysis of the differences of frequency levels normalized to the density of frequencies. According to the Random Matrix Theory, this quantity is universal, independent of the nature of the bar and the inclusions.

From the numerical probability distribution (Fig. 7), we find the following expression for the distribution of $\lambda \equiv \mu\sqrt{\mu}$:

$$P(\lambda) = 5.7e^{-\left(\frac{\lambda-0.6}{0.025}\right)^2} + 2.5e^{-\left(\frac{\lambda-0.927}{0.06}\right)^2} + 3e^{-\left(\frac{\lambda-1.23}{0.092}\right)^2}$$

We established that the width of the peak increases with its order and with the uncertainty in inclusions concentration.

Call S the slope of a generic curve in Fig. 8. Then $\sigma(S)$ is well approximated by

$$\sigma = 10S$$

This provides a method to obtain information about the distribution of inclusions: One would measure S experimentally and, from the last equation, obtain σ, the particle concentration distribution.

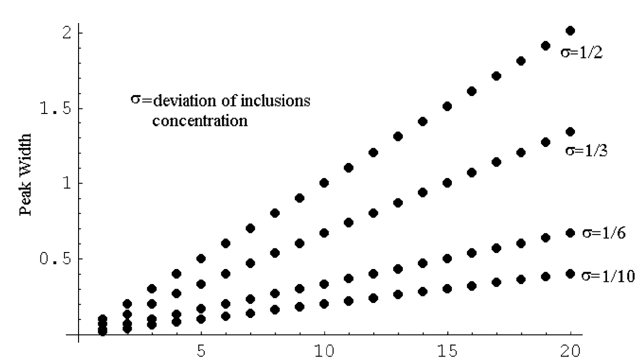

Fig. 8. The width of the probability distribution peaks increase with order (horizontal axis) and with inhomogeneity of inclusions.

4. Conclusions

Currently, we have made 50 concrete bars with the same formula and tested them with the setup shown in Fig. 1. However, as explained before a formula for concrete does not deal with the micro position of the constituents of concrete. In fact, we can safely assume that all 50 bars are microscopically different. We measure the statistical distribution of the lowest three or four frequencies and, finally, we analyze the results using Fig. 8, from which we can extract the degree of heterogeneity of the ensemble. This is of clear industrial relevance as the method can quantify the "smoothness" of a sample produced with a given formula and under specific environmental conditions. If the heterogeneity so predicted were larger than a nominal accepted value for a certain application, the manufacturing process would have to be rejected.

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