Construction and Building Materials 47 (2013) 502–510

Contents lists available at SciVerse ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Air voids size distribution determined by ultrasonic attenuation

Hong Jae Yim [a], Jae Hong Kim [b,*], Bang Yeon Lee [c], Hyo-Gyoung Kwak [d]

[a] Department of Civil and Environmental Engineering, Korea Advanced Institute of Science and Technology, 335 Gwahangno, Yuseong-gu, Daejeon 305-701, Republic of Korea
[b] School of Urban and Environmental Engineering, Ulsan National Institute of Science and Technology, 50 UNIST-gil, Eonyang-eup, Ulju-gun, Ulsan 689-798, Republic of Korea
[c] School of Architecture, Chonnam National University, 300 Yongbong-dong, Buk-gu, Gwangju, Republic of Korea
[d] Department of Civil and Environmental Engineering, Korea Advanced Institute of Science and Technology, Daejeon, Republic of Korea

HIGHLIGHTS

- An ultrasonic method to determine the air voids size distribution is proposed.
- The measured ultrasonic wave attenuation increases with the amount of air voids.
- The size distribution of air voids affects the wave frequency-attenuation curve.
- The maximum likelihood for the air voids size distribution is log-normal distribution.
- We compare the results obtained by the proposed method and the microscopical determination.

ARTICLE INFO

Article history:
Received 4 January 2013
Received in revised form 4 April 2013
Accepted 4 May 2013
Available online 10 June 2013

Keywords:
Ultrasonics
Image analysis
Microstructure
Air voids
Cement paste

ABSTRACT

Voids in cement-based materials influence their material properties. While air voids within a certain range are beneficial for freezing-and-thawing resistance, large-size voids decreases strength and durability. This paper proposes a method to quantify the air voids size distribution, which use the concept of ultrasonic wave attenuation. The wave attenuation increases with the inclusion of air voids. Its measurement is compared with a theoretical model, and then three-dimensional information of air voids can be obtained. An application example of cement paste is presented and the results are discussed with the measurement of permeable pores and the result of a microscopical determination.

1. Introduction

Air voids in concrete affect and determine its resistance to stress and durability [1,2]. The void is inherently entrapped when a cement-based material is being mixed and sometimes it is intentionally entrained to enhance freezing-and-thawing durability. The entrained air void is distributed in a range of 50–200 μm [1]. The void of such a size adjusts to water expansion when concrete is freezing, and consequently reduces the imposed hydraulic pressure and the freezing-and-thawing damage [3]. Even though an exact physical theory of the freezing-and-thawing resistance is still hardly formulated, it is doubtless fact that the dimensions and spacing of air voids are the most important parameters.

The dimension and spacing of air voids is usually characterized by microscopical determination, in accordance with ASTM C457 [4]. A saw-cut section of a sample is captured via a microscopy, where the chord lengths of voids are measured in the cross section. The size distribution of the air voids is obtained from the measurement records of the chord lengths. A spacing factor is then calculated from a model, called spacing equation. The measured chord lengths and the numbers of air voids are input parameters in the model. Various spacing equations including the model adopted for ASTM C457 can be found in the literature [5] and they have provided reasonable evaluation based on the size distribution of air voids. Nevertheless, the air voids characterization still needs to be verified in terms of the size distribution of air voids. The microscopical determination is based on two-dimensional cross section images. The chord length measurement has lack of three-dimensional information. In addition, the reliability on the cross-sectional measurement is probably obtained with a number of sectional images, even more than 100.

On the other hand, many researchers have recently performed characterization of air voids in hardened cement-based materials using ultrasonic waves. The ultrasonic methods, nondestructive test, have been proposed to characterize the damage of concrete

---

* Corresponding author.
E-mail addresses: jaekim@unist.ac.kr, jhk3001@gmail.com (J.H. Kim).

0950-0618/$ - see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.conbuildmat.2013.05.070

such as crack depth or contact-type defect [6–8]. Especially, the measuring of amplitude decay has advantage of obtaining three-dimensional information on the path where stress wave propagates. A previous study measures pulse-echo attenuation and finally determines the average void size and volume fraction [9,10]. Yet, analyzing the full distribution of air voids has not been attempted because the previous study relies on the assumption of monosize spheres.

This paper proposes a method to obtain the air voids size distribution rather than the average value. The quantitative ultrasonic wave attenuation of longitudinal through-transmission wave was measured via a self-compensating technique [11]. This technique ensures the quantitative measurement of wave attenuation, which is discussed more in the paper. The measured wave attenuation was compared with a theoretical model prediction, and finally the air voids volume fraction and size distribution were obtained by minimizing the difference between the model prediction and the measurement. The theoretical model adopted the modified scattering Roney formulation [12,13], and considered normal, log-normal, and Weibull distributions for the air voids size distribution. The obtained air voids volume fraction was compared with the experimental results by a test method for estimation of the total volume fraction of permeable voids (ASTM C642) [14]. The obtained size distribution was also compared with that obtained on a typical section of the sample via an image processing technique. That can be considered as the microscopical determination (ASTM C457) [4].

2. Background

Ultrasonic wave attenuation, decay of the amplitude of a propagating wave, is the result of geometric spreading and intrinsic attenuation. Geometric spreading is due to the wavefront spreading out. Intrinsic attenuation consists of wave absorption and scattering if a medium is composed of a matrix and inclusions, such as air voids. An attenuation coefficient is said to be a material property when it expresses the amount of intrinsic attenuation excluding the geometric spreading [15].

While absorption is inherent in the matrix, the scattering due to the inclusions in media is more evaluated. The scattering phenomena due to spherical uni-diameter grains such as air voids in a medium can be explained with the Roney formulation [12]. The formulation assumes that (1) the medium is comprised of two phases, a solid matrix and spherical grains; (2) the spherical grains in the medium do not interact with each other; and (3) wave energy scatters on the boundaries of the grains. Wave attenuation due to scattering on the grain boundaries depends on the wavelength, the grain size, and the material properties (elastic moduli) of the solid matrix and spherical grains.

A modified Roney formulation to overcome the limitation of uniform grain size using a log-normal distribution has also been introduced [13,16–18]. The scattering attenuation coefficient, α_s, is given as follows [13]:

$$\alpha_s = N_T\left(\frac{\Delta k}{k}\right)^2\int_0^\infty p(D)\frac{2}{\mu}\sum_{m=0}^{\infty}(2m+1)\sin^2\delta_m dD \tag{1}$$

where D is the diameter of the spherical grains, $p(D)$ is the probability density function of the diameter ($\int_0^\infty p(D)dD = 1$). N_T is the total number of the spherical grains per unit volume of the medium, and $\left(\frac{\Delta k}{k}\right)^2$ is an average elastic mismatch parameter of which amount is similar to the ratio of acoustic impedances of two phases [12]. The spatial parameter μ is

$$\mu = \frac{\pi D_m}{\lambda} \tag{2}$$

where D_m is the mean diameter of grains ($D_m = \int Dp(D)dD$) and λ is the applied wave length, and then the corresponding parameter becomes

$$\tan\delta_m = \begin{cases} \frac{j_1(\mu)}{n_1(\mu)}, & m = 0 \\ \frac{mj_{m-1}(\mu)-(m+1)j_{m+1}(\mu)}{mn_{m-1}(\mu)-(m+1)n_{m+1}(\mu)}, & m \geqslant 1 \end{cases} \tag{3}$$

where $j_m(\mu)$ and $n_m(\mu)$ are spherical Bessel functions of order m and spherical Neumanns functions of order m, respectively,

As a result, the intrinsic attenuation coefficient α_i attributed from both matrix absorption and spherical inclusions scattering is represented as follows:

$$\alpha_i = \alpha_s + (1-\varphi)\alpha_a \tag{4}$$

where α_a is the absorption attenuation coefficient of the solid matrix and φ is the volume fraction of the spherical inclusions ($\varphi = N_T\int\frac{\pi D^3}{6}p(D)dD$).

3. Experiment and evaluation

3.1. Sample preparation

The objective of this study is to evaluate excessive or abnormal air voids with wave attenuation measurement. The air voids in cement paste samples were prepared with use of an Air Entraining (AE) admixture. Cement paste samples labeled as P1, P1A1, P1A2, and P1A3 were proportioned with admixture-to-cement weight ratios of 0%, 0.2%, 0.7%, and 1.0%, respectively. The water-to-cement ratio of all samples was 0.4 by weight. The total mixing time was about 10 min using Hobart planetary mixer with a paddle beater. The amount of air voids in each fresh mix was measured following the ASTM C231 pressure method [19] as reported in Table 1. The samples were subsequently cured in 20 °C water for 6 months. In order to visualize the air voids, a sample was cut with a diamond saw and then polished to obtain cross-sectional images. Images were acquired using an optical microscope with a charged coupled device, as shown in Fig. 1.

Table 1 also reports the permeable voids in the hardened samples measured in accordance with ASTM C642 [14]. In order to obtain the air voids volume fraction, it is necessary to subtract capillary pores from the permeable voids measurement. Fig. 2 shows the schematic for the calibration. When the total volume of samples is the unit volume, the volume of capillary pores in the reference sample is φ_{c1} (Fig. 2a); the volume fraction of permeable voids φ_1 can then be calculated as φ_{c1}. Other samples can also be represented by the volume of generated air voids φ_a and capillary pores φ_{c2} (Fig. 2b), and the volume fraction of permeable voids φ_2 is then calculated as ($\varphi_a + \varphi_{c2}$). The volume fraction of capillary pores excluding generated air voids in other samples is $\varphi_{c2}/(1-\varphi_a)$, which should correspond with φ_1. Hence, the volume fraction of increased air voids φ_a can be obtained using Eq. (5), and the results are given in Table 1.

$$\varphi_a = \frac{\varphi_2 - \varphi_1}{1-\varphi_1} \tag{5}$$

3.2. Wave attenuation measurement

In order to measure the wave attenuation, a quantitative attenuation measurement technique previously proposed by the authors was used [11]. A block diagram of the experimental system setup for the attenuation measurement is presented in Fig. 3. The most important component to quantitatively measure the wave attenuation is lead-zirconate–titanate (PZT) ceramics, which are located between the conventional transducers and the sample. The PZT ceramic excludes the effect of coupling between the transducer and the sample on the attenuation measurement via a self-compensating principle [11]. When longitudinal waves are generated separately in the forward and backward directions, from-left-to-right and from-right-to-left, the stress waves at the channels of X and Y are measured via the PZT ceramics. Applying the principle of self-compensating isolates the frequency response function of the sample, where the effect of the coupling conditions is robustly excluded. The derived wave attenuation is, which peak amplitude corresponding to generated frequency of both channels, as follows:

Table 1
Air voids volume fraction of the samples.

Label	AE dosage (%)	Air content (%)	Permeable void (%)	Air void (φ_a) (%)
P1	0	1.4	26	–
P1A1	0.2	3.8	29	4
P1A2	0.7	5.9	34	10
P1A3	1.0	More than 15	40	19