Continuous Strength Measurements of Cement Pastes and Concretes by the Ultrasonic Wave Reflection Method

Didier Lootens^a, Marc Schumacher^a, Maxime Liard^{a,*}, Scott Z. Jones^b, Dale P. Bentz^b, Stefano Ricci^c, Valentino Meacci^c

 ^aSika Technology AG - Central Research Tueffenwies 16 CH-8048 Zürich, Switzerland
 ^bNational Institute of Standards and Technology Materials and Structural Systems Division
 100 Bureau Drive Gaithersburg, Maryland 20899
 ^cDepartment of Electronics and Telecommunications Florence University
 Via S. Marta, 3, 50139 Florence, Italy

Abstract

Concrete compressive strength is a critical design criterion for concrete elements and should, as a consequence, be carefully controlled to ensure structural integrity and intended functionality. As the cementitous binder of concrete hydrates, its strength and elastic modulus increase with time as concrete transitions from a fluid with suspended particles to a rigid but porous solid. Porosity of the material decreases as hydration products fill available space to create a densified structure. Ultrasonic instruments are able to continiously measure the material properites of cementitious materials. This is a significant advantage over destructive, quasi-static compression test of cylinders or cubes at discrete time intervals. Here, we estimate the elastic modulus and compressive strength of a cement paste or concrete from the amplitude of a reflected ultrasonic wave. A series of cement pastes and concretes are tested in quasi-static compression tests. The differences between the compressive strengths obtained by quasi-static compression tests and ultrasonic wave reflection differ by ± 20 % over a range of compressive strengths spanning more than 3 decades.

Keywords: Compressive Strength Measurements, Early-age hydration, Non-destructive Testing, Setting time

1 Introduction

2 Compressive strength testing is a commonly utilized early-age test to characterize cementitious materi-

³ als [1]. Strength evolution is a key parameter in construction, and consequently, all product development

⁴ or quality control operations make extensive use of destructive compression and tensile strength tests. Due

^{*}Corresponding author

Email address: liard.maxime@ch.sika.com (Maxime Liard)

to the random and heterogeneous nature of the cement-based pastes, mortars, and concretes, an average of
at least 3 specimens at a given age is typically required to obtain an acceptable precision of compressive
strength results, often cited as the multi-laboratory coefficient of variation of about 7 % for mortar cube
testing according to the ASTM C109 standard test method [2]. The process of preparing specimens for testing by ASTM C109 requires mixing, casting, curing, and destructive testing. This process is labor intensive
and cannot be easily automated. Replacing this method of testing with one that does not require this type
of sample preparation, even on a partial basis, would represent a gain in material testing efficiency, as well
as a reduction in material and labor costs.

Ultrasonic methods are used in industrial application to measure the evolution of the elastic modulus of 13 a cementitious materials over time [3]. Three techniques are commonly used: (i) compression sound wave 14 propagation through the concrete [3, 4, 5], (ii) speed of the surface wave at the interface between concrete and 15 air [6], (iii) and wave reflection at the interface between the concrete and a wave guide [7, 8, 9, 10]. The three 16 techniques measure the acoustic properties of the materials of interest, which are related to their mechanical 17 properties. The attenuation of the ultrasonic wave through the material may be used to estimate the evolution 18 of the shear or bulk modulus of material, respectively G and K. Acoustic impedance measurements of shear 19 waves have been successfully used to monitor the flocculation and setting times of cement paste [11, 12]. 20 Akkaya et al. [8], estimated the compressive strengths of concretes with aggregate volume fractions from 21 50 % to 70 % using ultrasonic wave reflection techniques. Results indicate the reflection loss coefficient 22 is sensitive to cement hydration and, after calibration, the reflection loss change may be used to predict 23 concrete strength at early ages. 24

This study estimates the strength of concrete, with aggregate volume fractions ranging from 10 % to 25 70 %, using ultrasonic wave reflection techniques. Accelerating admixtures are added to the concrete as-26 sess the ability of this technique to estimate the strength of samples with a rapidly changing compressive 27 strength. A custom-built ultrasonic device is used to measure the reflection loss coefficient of a reflected 28 wave generated at the interface of a waveguide and a hydrating cementitious material. The shear modulus of 29 the sample is estimated from this measurement, which is related to the elastic modulus and, ultimately, the 30 strength. Compressive strengths estimated by this method are compared to traditional quasi-static compres-31 sive strength measurements to assess the suitability of replacing these measurements with non-destructive 32 assessments of strength. 33

34 Materials and Methods

35 Mixture Proportions

Both samples of cement paste and concrete, containing aggregates up to 70 % by volume, are evaluated in this study. An ASTM C150 Type III ordinary portland cement (OPC) is used to limit the impact of the temperature increase during curing on the hydration kinetics of the samples [13]. Cement pastes were prepared using three non-commercial accelerators: two alkali-free sulfoaluminate suspensions, called

accelerators A1 and A2, and a sodium silicate-based accelerator. The cement was mixed with a limestone 40 powder, having a similar particle size distribution to the cement, and water with a Hobart¹ mixer at a speed 41 setting of 2 (285 rev/min \pm 10 rev/min) for 3 min. Samples were prepared for quasi-static compression 42 testing by spraying the materials into the mold using the the device described in [14]. In the case of 43 ultrasonic measurements, the paste was sprayed directly onto the instrument. The accelerator and cement 44 paste are mixed before the material exits the nozzle. Both paste and accelerator are pumped at a constant 45 flow rate to a mixing chamber. Compressed air at 200 kPa creates a homogeneous mixture of the two 46 components. Quasi-static compression testing samples are prepared using 40 mm x 40 mm x 40 mm cube 47 molds. Samples for ultrasonic measurements are cylinders with a diameter of 100 mm and a thickness of 48 20 mm. Samples for both test are shown in Figure 1. The samples were kept at a temperature in the range 49 of 23 °C to 27 °C. The paste formulations are summarized in Table 1. Concrete mixtures were prepared per 50 the formulations provided in Table 2 and cast into the same molds used for the paste specimens. 51



(a) Cylinder Specimens



(b) Cube Specimens

Figure 1: Samples of concrete for testing, including cylinder (100 mm diameter and 20 mm height) and cube (40 mm) geometries, shown after compression tests.

	Cement	Limestone Powder	Water	Accolonation	Accelerator Concentration	
	(kg)	(kg)	(kg)	Accelerator	(by mass of cement)	
Paste 1	1	1	0.46	Alkali-free A1	6~%	
Paste 2	1	1	0.46	Sodium Silicate	10~%	
Paste 3	1	1	0.36	Alkali-free A2	6~%	
Paste 4	1	1	0.38	Sodium Silicate	10~%	

Table 1: Formulation of the sprayed cement paste.

¹Certain commercial products are identified in this paper to specify the materials used and the procedures employed. In no case does such identification imply endorsement or recommendation by the National Institute of Standards and Technology, nor does it indicate that the products are necessarily the best available for the purpose.

				Aggregates			Admixture
Aggregates	Cement	Limestone Powder	Water	4 mm - 8 mm	1 mm - 4 mm	$< 1 \mathrm{~mm}$	$(\rm SiO_2)_nO$
(vol. %)	(kg)	(kg)	(L)	(kg)	(kg)	(kg)	$2~\%/{\rm kg-cement}$
10 %	3.15	3.15	1.48	0.14	0.35	0.21	0.12
30~%	2.45	2.45	1.24	0.42	1.05	0.63	0.09
50~%	1.75	1.75	1.01	0.70	1.75	1.05	0.06
70~%	1.05	1.05	0.77	0.98	2.45	1.47	0.04

Table 2: Concrete formulations used for quasi-static and ultrasonic testing.

52 Derivation of Equations

The ultrasonic wave speed of an isotropic material is a function of the Lamé coefficients and the density 53 of the material. A sample may be considered homogenous with respect to the propagating ultrasonic wave 54 when the largest heterogeneity of the material is smaller than the wavelength. A wave propagating at 55 1000 m/s with a frequency of 1 MHz will have a wavelength of 1 mm. The particle size for cement powder is 56 approximately $< 100 \,\mu$ m, indicating cement paste may be treated as a homogenous material for this particular 57 propagating wave. For concrete or mortars containing aggregates (or other heterogeneities) larger than 1 mm, 58 the heterogeneities will cause the propagating wave to scatter, further complicating the assessment of the 59 concrete's elastic properties. To overcome this, the reflected wave generated at the wave guide-sample 60 interface may be used to infer the changing elastic properties of the paste portion of the concrete. The 61 amplitude of the reflected wave can be used to estimate the strength of the sample by recognizing that 62 the amplitude of the reflected wave will decrease as the acoustic impedance of the binder portion of the 63 sample increases. When the binder is composed of OPC, the increase in the acoustic impedance is a result of hydration reactions between the cement and water which create hydration products, such as calcium silicate 65 hydrate (C-S-H), which create percolated network of particles. 66

Concrete may be considered a two phase composite material consisting of a paste phase (binder and water) 67 and an aggregate phase. The volume fraction of the aggregates in concrete is spatially dependent near the 68 surface of a mould or a form (the wall effect) [15]. Numerical simulation in three dimensions have shown 69 the volume fraction of aggregates converges to the theoretical volume fraction (volume of aggregates/total 70 volume) at approximately 10 mm from the wall [16]. This result holds for the range of volume fractions of 71 interest to this study and indicates that, for the first few millimeters from a surface, the primary constituent 72 of the material is cement paste. The shear modulus of the cement portion of a concrete sample may be 73 estimated by generating a reflected wave at the interface between the waveguide and the sample. The 74 reflected wave generated at the waveguide/sample interface is assumed to follow the theory outlined in [17] 75 and is assumed to probe the cement paste portion of the sample due to the wall effect. A shear wave with 76 amplitude, A_i , is generated within a waveguide with an acoustic impedance Z_{wg} . At an interface of the 77 waveguide and the sample the medium experiences a sudden change in acoustic impedance. A portion of 78

the incident wave is transmitted through the interface to the sample medium with impedance, Z_s , while the 79 remaining portion is reflected from the interface to ultrasonic sensor. The reflection coefficient, r, is the ratio 80 of the reflected wave amplitude, A_r , to the amplitude of the incident wave and is related to the impedance 81 of the two mediums by relationship, $r(t) = A_r/A_i = (Z_{wg} - Z_s(t))/(Z_{wg} + Z_s(t))$ [17], where t is the time 82 after mixing. $Z_{s}(t)$ is assumed to represent the time-dependent impedance of the cement paste portion of 83 the concrete. The increase in the portion of the incident wave which is transmitted through the samples and 84 the decrease in the amplitude of the reflected wave generated at the waveguide-sample interface has been 85 attributed to the formation of a percolated network of cement particles which is a result of the formation of 86 hydration products within the cement paste. [18, 19, 20]. 87

The reflection coefficient is estimated from amplitude measurements of the incident and 1st reflected wave as a function of time. The reflection coefficient is used to estimate the shear modulus of the paste portion of the concrete. The elastic modulus of the paste is calculated assuming the sample obeys linear elastic, isotropic theory and a Poisson's ratio, ν . The range of expected values of ν is 0.2 to 0.3 [21, 22]. Here a value of $\nu = 0.3$ is used in computing the elastic modulus. The effective elastic modulus of the concrete is estimated using the Hashin-Shtrikman model and the concrete strength is calculated assuming a power-law relationship between elastic modulus and compressive strength.

95 Relationship between ultrasound and mechanical properties

⁹⁶ Ultrasonic waves propagate through solids in either compression or shear modes. Both the shear and ⁹⁷ elastic modulus capture the time-dependent evolution of the material. The shear modulus experiences an ⁹⁸ increase by more than 5 orders of magnitudes from the fresh state after mixing to the final setting of the ⁹⁹ paste which enables accurate study of the paste flocculation and its early age setting [23]. Thus, shear waves ¹⁰⁰ are preferred for recording the evolution of the mechanical properties at early ages [24].

In this study, a shear wave is generated by one of the transducers and propagates through the wave guide until a reflection is created at the wave guide-sample interface. The reflected wave, which is detected by the same transducer that generated the pulse, is the first signal received by the transducer (at time t_1 in Figure 2). The following reflected wave is a result of the sample-air interface (t_2) . Figure 2 is a schematic representation of the ultrasonic test configuration.



Figure 2: Echo mode measurement of ultrasonic wave propagation. A pulse generated from the transducer travels through the wave guide were a reflection wave is created. The arrival time of this wave at the transducer is t_1 .

All the pulse energy is reflected when the wave guide is in contact with the air, as the acoustic impedance of air is much less than that of the wave guide $(Z_{air} \ll Z_{wg})$. In the case of cementitious materials, the amplitude of the reflected wave changes with time as hydration reactions create a hardened material [9]. The shear modulus, G(t), of the sample is estimated using Equation 1, where $Z_s(t)$ is the acoustic impedance of the sample, ρ_s is the density of the sample, and r(t) is the reflection coefficient, which is estimated from the ratio of the amplitude of the reflected wave to the amplitude of the incident wave.

$$G(t) = \rho_s^{-1} Z_s(t)^2 = \rho_s^{-1} Z_{wg}^2 \left(\frac{1-r(t)}{1+r(t)}\right)^2$$
(1)

¹¹² Under the assumption of a homogeneous and isotropic material, the shear modulus, G, is related to the ¹¹³ elastic modulus, E, through Poisson's ratio, ν .

The relationship between the elastic modulus and the compressive strength, σ , of a cement paste or a mortar is obtained by a power law function as reported [25], and denoted in Equation 2, where k and n are the fitting parameters. Here, we assume n = 0.5 [26].

$$E = k\sigma^n \tag{2}$$

The relationship between compressive strength and elastic modulus, given in Figure 2, is acceptable over a broad range of compressive strengths and elastic moduli, even if concrete is not strictly a homogenous, linear elastic, isotropic material. The shear modulus may be expressed as a function of compressive strength as shown in Equation 3.

$$G = \frac{k\sigma^n}{2\left(1+\nu\right)}\tag{3}$$

Therefore, by measuring the shear modulus G with the ultrasonic device and using Poisson's ratio ν for a cement paste or concrete, one can use Equation 3 to calculate σ_u by Equation 4.

$$\sigma_u = \left(\frac{2G\left(1+\nu\right)}{k}\right)^{\frac{1}{n}} \tag{4}$$

When the sample undergoing test may be treated as homogenous with respect the the ultrasonic waves, e.g., cement paste, the compressive strength of a sample is related to the reflection coefficient, r, by introducing Equation 1 into Equation 4 to produce Equation 5.

$$\sigma_u(t) = \left(\frac{2(1+\nu)}{k} \frac{Z_{wg}^2}{\rho_s} \left(\frac{1-r(t)}{1+r(t)}\right)^2\right)^{\frac{1}{n}}$$
(5)

When the sample undergoing testing is heterogenous, e.g., concrete, the Hashin-Shtrikman lower bound model (see [27]) is used with Equation 1 to compute the composite shear modulus of the sample. The composite shear modulus is then used with Equation 4 to compute the compressive strength. The relationship between the compressive strength measurement by quasi-static compression test and shear modulus correlation is estimated using linear regression techniques. The uncertainty of the compressive strengths predicted by ultrasonic measurements is estimated at a 95 % confidence level.

The objective of this study is to compare measurements of compressive strengths of cement paste and concrete samples made by physical testing (σ_c) and estimated from Equation 5 (σ_u).

134 Equipment

The ultrasonic device used in this study is presented in Figure 3. It is a custom-built, non-commercial 135 device for evaluating the material proprieties of cement-based materials. The device is composed of 8 cells 136 which record and process data independent of each other. Each cell each contains three ultrasonic shear 137 wave transducers bonded to the wave guide, operating at a frequency of 0.8 MHz. The three transducers 138 operate sequentially. Each transducer will generate an incident wave and detect the 1st reflected wave to 139 estimate the reflection coefficient at the wave guide/sample interface. The amplitude of the detected waves 140 is calculated by taking the root mean square of the signal. The amplitudes from the three transducers are 141 averaged to account for heterogeneities, such as air voids and inclusions, in the sample volume that may 142 affect the acquired signal. Multiple independently operating cells are used to assess the sample variation 143 for one mixture formulation or they can be used to test multiple mixture formulations simultaneously. The 144 user can program the sampling interval, length, and test duration according to their needs. The signal is 145

- processed by a low noise amplifier, sampled at 75 MHz with 16-bit resolution, and saved for post processing.
- ¹⁴⁷ Full details of the ultrasonic device used in this study are reported in [28].



Figure 3: Ultrasonic device used in this study. Device consists of 8 cells, each with 3 transducers and one temperature sensor.

148 Results

Comparisons between quasi-static compressive strength measurements and compressive strengths estimated by Equation 5 are of interest to this study. The presented results first address the case of cement paste samples and then address the case of concrete samples with two aggregate shapes at four volume fractions.

153 Cement Paste

Figure 4 reports the relationship between compressive strengths estimated by Equation 5 and quasi-static compression testing of the four paste formulations reported in Table 1. The mean quasi-static compressive strengths and standard deviations for 10 replicate specimens, are reported in Table 3. The maximum coefficient of variation of the quasi-static compressive strengths is 10 % of the measured value. Figure 4c reports the quasi-static compressive strengths as a function of the curing time. Compressive strength measurements span a range of two orders of magnitude in an approximately 24 h period.

As can be observed in Figure 4a, there is a power-law relationship between the two methods of determination of the compressive strength. To assess the relationship between the compressive strength of the paste determined by quasi-static compression tests (σ_c) and ultrasonic measurements (σ_u), linear least squares regression was performed to determine the coefficients *a* and *m* of the power-law equation described in Equation 6.

$$\sigma_c = a \sigma_u^m \tag{6a}$$

$$\log(\sigma_c) = \log(a) + m \log(\sigma_u) \tag{6b}$$

Performing linear least squares regression on the linearized form of the data produces the estimates for 165 $\log(a)$ and m given in Table 4. The expanded uncertainty of $\log(a)$ at a 95 % confidence level is computed 166 by multiplying the standard error in Table 4 by the t-statistic, $t_{0.975,18} = 1.762$. The lower bound of the 167 confidence interval for $\log(a)$ is -0.0008 and the upper bound is 0.077, corresponding to a confidence interval 168 of 19.6 %. The plot of the residuals of the fitted values versus the quasi-static compressive strengths is 169 shown in Figure 4b. Akaike Information Criterion (AIC) was used to assess the suitability of the model in 170 Equation 6 compared to a linear model of the form $\sigma_c = \beta_1 \sigma_u + \beta_0$. The small sample AIC values for the 171 model of Equation 6 and the linear model are -43.92 and 82.53, respectively, with a probability that the 172 linear model minimizes information loss compared to power law model of 1.22×10^{-55} , indicating the model 173 of Equation 6 is suitable for this data set. 174



(a) Relationship between ultrasonic computed and quasi-static compressive strength measurements

(b) Residuals of linear least squares regression of Equation 6

(c) Quasi-static Compressive Strengths

Figure 4: (a) Relationship between the strength measured with ultrasound and with the compression of the cubes. The representations are all made in logarithmic scales. (b) Residuals plotted versus cube break strengths. (c) Evolution of paste strength as a function of time for the four pastes.

time	Paste 1		Paste 2		I	Paste 3	Paste 4	
(h)	\bar{X}	$\sigma_{ar{X}}$	\bar{X}	$\sigma_{ar{X}}$	\bar{X}	$\sigma_{ar{X}}$	\bar{X}	$\sigma_{ar{X}}$
1.5	_	_	0.56	$1.1 imes 10^{-3}$	_	_	0.65	$1.3 imes 10^{-3}$
2.5	0.62	1.2×10^{-3}	1.03	2.1×10^{-3}	_	_	_	_
3.0	_	_	_	_	_	_	1.82	3.6×10^{-3}
3.5	_	_	1.76	3.5×10^{-3}	_	_	_	_
4.0	1.13	2.3×10^{-3}	_	_	_	_	_	_
4.5	_	_	3.98	1.6×10^{-2}	_	_	_	_
5.5	_	_	_	_	1.18	2.4×10^{-3}	5.87	2.9×10^{-2}
6.0	1.97	3.9×10^{-3}	_	_	_	_	_	_
7.5	_	_	_	_	3.25	9.7×10^{-3}	_	_
8.5	_	_	8.38	6.7×10^{-2}	4.90	2.5×10^{-2}	10.61	1.1×10^{-1}
9.0	5.05	2.5×10^{-2}	_	_	_	_	_	_
25.5	_	_	_	_	40.54	1.6	28.03	7.8×10^{-1}
26.0	14.75	2.2×10^{-1}	24.92	$6.2 imes 10^{-1}$	_	_	_	_

Table 3: Mean (\bar{X}) quasi-static compressive strengths and standard deviation $(\sigma_{\bar{X}})$ of 10 replicate samples for the paste mixtures described in Table 1. Units: MPa.

Table 4: Parameter estimates determined by linear least squares regression. Experimental data was linearized by taking the base 10 logarithm of each observation.

	Estimate	Standard Error
$\log(a)$	0.038	0.022
m	0.883	0.025

It is possible to estimate the compressive strengths of cement paste with Equation 5 and then one can correct the measured values using the power-law relationship $\sigma_c = a\sigma_u^m$ determined previously with a = 1.09and m = 0.88 which enables continuous and reproducible measurement of the compressive strength of a cement paste.

179 Concrete

In this section, we study the feasibility to use the ultrasonic device to measure the compressive strength of a concrete sample. As previously discussed, when the size of the aggregates are larger than the wavelength of the ultrasonic wave, scattering effects begin to dominate the acquired signal. The elastic properties of a concrete sample are estimated from the wave that is reflected at the sample-wave guide interface, i.e., the 1st reflected wave in Figure 2.

185 Quasi-static Compression Test

The quasi-static compressive strength development of concretes created with crushed or rounded aggregate, at various volume fractions, is reported in Figure 5. The quasi-static compressive strengths range between 0.2 MPa and 20 MPa over a time period ranging from 4 h to 30 h. Tables 5 and 6 report the mean quasi-static compressive strength of the cubes composed of crushed aggregates and rounded aggregates, respectively.

Table 5: Mean (\bar{X}) quasi-static compressive strengths and standard deviation $\sigma_{\bar{X}}$ of 10 replicate samples for the concrete mixtures with crushed aggregates described in Table 2. Units: MPa.

time	VF 1	.0 %	VF 30 %		VF 50 $\%$		VF 70 $\%$	
(h)	\bar{X}	$\sigma_{\bar{X}}$	\bar{X}	$\sigma_{\bar{X}}$	\bar{X}	$\sigma_{\bar{X}}$	\bar{X}	$\sigma_{\bar{X}}$
4	0.47	0.06	0.39	0.04	0.35	0.02	0.44	0.04
6	0.94	0.12	0.94	0.1	0.73	0.1	0.58	0.03
8	1.82	0.13	1.78	0.11	1.21	0.07	1.05	0.05
14	_	_	4.99	0.22	3.62	0.18	2.33	0.15
20	_	_	10.4	0.25	7.33	0.20	5.11	0.28
30	20.09	0.64	15.77	5.73	13.21	0.64	10.81	0.37

Table 6: Mean (\bar{X}) quasi-static compressive strengths and standard deviation $\sigma_{\bar{X}}$ of 10 replicate samples for the concrete mixtures with rounded aggregates described in Table 2. Units: MPa.

time	VF 1	0 %	VF 30 % VF 50		50 % VF 70 %		0 %	
(h)	\bar{X}	$\sigma_{\bar{X}}$	\bar{X}	$\sigma_{\bar{X}}$	\bar{X}	$\sigma_{\bar{X}}$	\bar{X}	$\sigma_{\bar{X}}$
4	0.69	0.06	0.39	0.06	0.54	0.05	0.44	0.03
6	1.66	0.09	0.89	0.07	1.26	0.1	0.94	0.06
8	3.24	0.2	1.83	0.09	2.16	0.12	1.55	0.17
14	8.51	0.26	4.63	0.34	4.63	0.34	2.95	0.2
20	15.45	0.6	9.29	0.78	9.92	0.63	6.03	0.19
30	24.06	0.98	17.15	1.06	18.6	0.59	14.97	0.98

The quasi-static compressive strength of the two concretes with crushed or rounded aggregates is reported in Figure 5 as a function of aggregate volume fraction and age of the specimen. For both the rounded and crushed aggregates, the strength is globally decreasing with an increasing volume fraction of aggregates, indicating a weak interface between cement paste and the aggregates. As the strength of the paste increases
from approximately 0.2 MPa to 20 MPa, fracture begins to occurs preferentially at the interface between
the paste and the aggregates, increasing the path length of the fracture, which increases the bulk fracture
toughness of the material.



Figure 5: Quasi-static compressive strength of concrete cubes with various volume fractions of (a) crushed aggregates and (b) rounded aggregates.

¹⁹⁸ Ultrasonic Measurement of Concrete Strength

The ultrasound technique is measuring amplitude of the reflected wave generated at the wave guide/sample, 199 which is assumed to be primarily composed of cement paste. As a consequence, this technique does not ac-200 count for the impact of the aggregates or air voids (or fibers when present) on the total strength. An 201 estimation of the concrete elastic modulus, E_c , is required to compute the concrete strength. E_c is computed 202 using the lower bounds of the Hashin-Shtrikman equation [27]. Calculations require measurements of the 203 paste elastic modulus, which have be computed by ultrasonic measurements, and the elastic modulus of 204 the aggregates. Aggregate elastic modulus is dependent upon the mineralogy and can have a wide range 205 of reported values such as those reported in [25]. Siliceous aggregates used in this study are assumed to 206 have an elastic modulus of 80 GPa. The strength of the bond between the aggregate and the cement paste 207 is an important factor. The nature of the interfacial transition zone (ITZ) is dependent on time, type of 208 aggregate, and its reactivity with the cement paste, and is not easy to quantify. In fact, both strength of 209 the aggregates and their ITZ are difficult to access [14, 12, 20]. The Hashin-Shtrikman equation does not 210 account for such effects and as such, the ultrasonic-based predictions of concrete strength are insensitive to 211 the nature of the paste-aggregate bond. 212

The dependence of the concrete elastic modulus (and strength) on aggregate content is schematically represented in Figure 6a where the range is bounded by the strength of the cement paste (VF = 0 %) and that of the aggregate (VF = 100 %). As the aggregate strength remains constant as cement undergoes

hydration reactions, and is higher than the strength of the cement paste at early ages, the concrete strength is 216 only a function of the cement paste strength and time. Assuming the aggregates do not change the reactivity 217 of the cement, one can use the strength evolution of the cement paste to compute the concrete strength using 218 the Hashin-Shtrikman model. This method is demonstrated in Figure 6b where both the cement strength 219 (black line) and the computed concrete strength (cyan line) are plotted. Penetration test results are displayed 220 as cyan squares. The agreement between the results of the penetration test and ultrasonic measurements 221 is interpreted to validate the use of the Hashin-Shtrikman model for indirect concrete compressive strength 222 measurements. 223



(a) Hashin-Shtrikman Lower Bound

(b) Development of compressive strength with time

Figure 6: (a) Elastic modulus of concrete computed by Hashin-Shtrikman lower bound equation. The elastic modulus of the cement paste phase changes with time. (b) Evolution of the strength as a function of the time for a cement and a concrete. The points are obtained with mechanical tests and the upper line calculated from the ultrasonic measurements made on the cement paste.

A direct measurement of concrete compressive strength may be obtained by exploring the relationship 224 between σ_c and σ_u for samples containing aggregates. Compressive strengths from ultrasonic measurements 225 are computed using Equation 1 and the Hashin-Shtrikman lower bound equation. The results are reported 226 in Figure 7 and show that ultrasonic measurements of compressive strength appear to be independent of 227 volume fraction and shape. This is expected as the amplitude of the 1^{st} reflected wave is dependent upon the 228 acoustic impedance of the sample, which changes as a result of the formation of hydration products and does 229 not directly assess the interior. Moreover, the compressive strength measurements made with ultrasound 230 are different from those obtained by quasi-static compression, reported in Figure 5, as the strength for the 231 rounded aggregates increases with increasing aggregate content for all time points. 232



Figure 7: Compressive strength of mortar cubes, measured by ultrasonic methods, at various volume fractions of (a) crushed aggregates and (b) rounded aggregates.

The relationship between the strengths measured with ultrasound and quasi-static compression for all concentrations and type of aggregates are represented in Figure 8. The cube strength measured by quasistatic compression testing as a function of compressive strength measured by the ultrasonic method follows a power-law relationship for volume fraction of aggregates up to 50 %. At 70 % volume fraction of aggregates, excess entrained air during the spray process causes the quasi-static compressive strength to be lower than the strength estimated by the ultrasonic method.

For the crushed aggregates, the quasi static compressive strength fall within the 95 % confidence intervals demonstrating the possibility to use ultrasonic measurements to estimate the compressive strength of a concrete. In the case of the rounded aggregates, the quasi static compressive strengths predictions are also within the 95% except for the 10% volume fraction test. In both case, the power law exponent for the correction is about 0.9 which a value identical to the one obtained for the cement paste. Table 7 reports the parameters log(a) and m, in Equation 6, estimated by linear least squares regression for the cases of the crushed and rounded aggregates.



Figure 8: Relationship between compressive strengths of mortar cubes with (a) crushed aggregates and (b) rounded aggregates measured by the ultrasonic method and quasi-static loading. The solid line represents the results of a linear least squares regression of Equation 6b onto the data in (a) an (b). The shaded region is the estimated 95 % confidence interval of the regression.

Table 7: Parameters estimated by linear least squares regression of the data reported in Figure 8 using Equation 6

	Crushed	Aggregates	Rounded Aggregates		
	Estimate	Std. Error	Estimate	Std. Error	
$\log(a)$	-0.011	0.040	0.036	0.060	
m	0.865	0.054	0.900	0.077	

For the case of crushed and round aggregates, the relationship $\sigma_c = a\sigma_u^m$ may be used with a equal to 0.97 246 and 1.14, respectively, and m equal to 0.865 and 0.900, respectively. With these parameters, the difference 247 is compressive strengths measured by quasi-static compression testing and the ultrasonic method is \pm 20 % 248 over the 3 decades of compressive strengths in this study. As σ_u is a function of E_{agg} , this relationship 249 is very likely to be dependent of the nature of the aggregates. Nevertheless, we demonstrate the possibility 250 to predict indirectly the compressive strength of a mortar or a concrete over time. This method is useful 251 during the development of a new mortar/concrete when a lot of screening experiments are required, because 252 the ultrasonic device enables quick and precise comparison between samples. Obviously, this method does 253 not aim to replace all the compressive strength measurements but could be used to reduce drastically the 254 amount of samples to be crushed during a development campaign. 25!

256 Conclusions

The compressive strength of concrete containing rounded and crushed aggregates at volume fractions from 257 10~% to 70~% was measured in quasi-static compression and estimated from the reflection of an ultrasonic 258 wave. When the sample contains heterogeneity larger than the wavelengths of the ultrasonic waves, the 259 strength of the sample is estimated by computing the composite shear modulus of the sample using the 260 Hashin-Shtrikan lower bound model, which is used to compute the elastic modulus and, finally, the strength. 261 Results from both methods were compared where it was found that the dependence of the compressive 262 strength of the sample measured by quasi-static compression on the compressive strength estimated using 263 the ultrasonic method can be described by a power-law function. The difference in compressive strengths 264 measured by both methods is estimated to be \pm 20 % over a range of compressive strength spanning 3 265 decades. As the volume fraction of aggregates increase to 70 %, the quasi-static compressive strengths 266 deviates from the power-law dependence on the compressive strengths measured by the ultrasonic method. 267 This is attributed to excess air which is entrained in the sample during mixing. The results presented in this 268 study suggest that ultrasonic wave reflection is a suitable technique for compressive strength measurements; 269 however, further tests are required to assess the validity of this method for other mixtures such as high 270 strength, low permeability concretes with aggregates greater than 8 mm and mixtures with entrained air. 271

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