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Cement & Concrete Composites 28 (2006) 299–306
Cement & Concrete Composites
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Determination of initial degree of hydration for improvement of early-age properties of concrete using ultrasonic wave propagation

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Available online 30 March 2006

Abstract

During the first few hours after mixing, the properties of concrete change between different types of material behaviour. Fresh concrete is during mixing a Bingham material, gradually attaining solid body properties with considerable compressive strength and stiffness. The development of mechanical properties can be described by the degree of hydration. For the prediction of mechanical properties of early-age concrete as well as for the prediction of stresses caused by differences of temperature and autogenous shrinkage, it is essential to know the initial degree of hydration, from which on the development of strength and stiffness can be assumes to begin. This paper deals with the determination of the end of the dormant phase by using ultrasonic pulse velocity techniques. Using compression wave and shear wave transducers the hardening of concrete is observed under adiabatic curing conditions. From the development of dynamic Young's modulus and Poisson's ratio a model of the initial degree of hydration is derived to improve existing models of the development of tensile strength and modulus of elasticity for very early-age concrete. A procedure to determinate an upper and lower bound for the end of setting time is presented. Typical results are presented for different concrete compositions, especially for high strength concrete.

Keywords: Ultrasonic testing; Young concrete; Setting and hardening; On-set time; Degree of hydration; Young's modulus; Poisson's ratio

1. Introduction

Ultrasonic testing for controlling the quality of metallic and composite materials is one of the most successful and most frequently applied testing methods in this field. The development of electronics as well as of microprocessor techniques during the last 20 years led to considerable improvements of automatic testing systems and their integration into production process. A comparable successful application of ultrasonic methods for testing mineral building materials has so far not been attained. The development of such systems started in the early 50s of the past century. Two main objectives can be discerned. The first one relates to the application of ultrasonic testing technique to measure the development of mechanical properties of concrete during hardening. The second one deals with the assessment of damage and cracks. An overview of activities in these areas was reported in [1–3] and others.

Early reports of iBMB in the field of ultrasonic testing are presented by Eisenmann et al. [4,5] between 1951 and 1956. They investigated the influence of cracks in concrete on ultrasonic pulse velocity. In addition, they described the difference between static and dynamic modulus of elasticity. This work was continued by Kordina et al. [6], who investigated the development of ultrasonic pulse velocity of shear and compressive waves and the relationship of these parameters to compressive strength. In 1974 Neisecke [7] presented a new method to control the quality of mineral building materials. He measured the transverse and longitudinal velocity as well as the intensity of the incoming signal. In principle, it was possible to simultaneously measure the development of dynamic Poisson's ratio and dynamic Young's modulus. Among the things, the main application of this method was the observation of the hardening process of concrete in the first 72 h. Hillger [8] continued this work. In 1983 he presented an enhanced method

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doi:10.1016/j.cemconcomp.2006.02.007

by which the spectrum of the incoming signal could be analysed. Besides the research conducted at the iBMB in Braunschweig, several research groups in the USA and in Europe were involved in NDT of cement based materials at early ages. An excellent literature review regarding this topic is given in [9].

In order to predict mechanical properties of concrete at very early age, non-destructive test methods can be of advantage. By combining such methods with destructive tests reliable information regarding the mechanical properties can be acquired. In this paper a test method is presented that determines the initial setting time, which represents the earliest time at which mechanical properties such as Young's modulus and tensile strength can be measured destructively. The method was developed in order to improve existing models of mechanical concrete properties and to obtain a better understanding of hardening concrete.

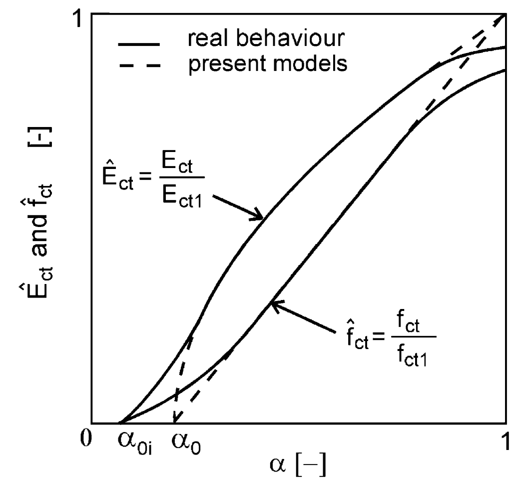

Fig. 1. Normalized tensile strength and modulus of elasticity dependent on degree of hydration – models and real behaviour.

2. Modelling of mechanical properties of concrete at very early ages

During the first hours and days after mixing, compaction and properties of concrete rapidly change within a wide range. Fresh concrete is a Bingham type of material, gradually attaining solid body properties with increasing compressive strength and stiffness. The development of mechanical properties can be described by the degree of hydration. For the prediction of mechanical properties of early-age concrete as well as for the prediction of stresses caused by differences of temperature and autogenous shrinkage, it is essential to assess the initial degree of hydration, from which on the development of strength and stiffness can be assumed to begin.

Several researchers have shown that the development of the mechanical properties can be modelled with the degree of hydration or equivalent age. Especially compressive strength, tensile strength and modulus of elasticity show a significant dependence on the degree of hydration; e.g. [10,11].

All models as described in the literature depict the test results well, but also contain uncertainties, in particular for a low degree of hydration. Fig. 1 shows the dependency between the modulus of elasticity $\hat{E}_{ct}$ and uniaxial tensile strength $\hat{f}_{ct}$ on the degree of hydration α as described by models [10] and also their real behaviour. Parameter α_0 is the degree of hydration that marks the end of the dormant phase and the on-set of strength and stiffness evolution. The value of the parameter α_0 is determined by regression of measured tensile strength data with a linear f_{ct}–α relationship. But this method exhibits two main technical difficulties. The first one relates to the fact, that reasonable results for the tensile strength can only be measured if α is greater 0.4 , because of necessary preparation time. In addition, the scatter of test results at very early-age is rather large. The second difficulty relates to modelling the degree of hydration at very early ages with sufficient precision. One possibility to obtain more precise values of α_{0i}, is to perform tests under adiabatic conditions and in combination with in situ measurements of mechanical properties. Fig. 1 also suggests that the evolution of mechanical properties starts much earlier than described by the theoretical value α_0. From this point of view it is clear that determining the corresponding smaller initial degree of hydration α_{0i} is more advantageous.

Non-destructive methods based on ultrasonics are especially suitable for this kind of investigations since the velocities of ultrasonic waves directly depends on the development of the elastic properties of the material tested.

3. Experimental equipment and testing

Fig. 2 shows the test set-up for the experiments described in this paper. It consists of an adiabatic calorimeter, its controlling unit, an ultrasonic measurement device and a controlling unit for the external water bath. All controlling units are integrated in a standard PC. An appropriate software steers all controlling tasks in this apparatus. The water bath is used to store the concrete specimens for destructive testing under adiabatic curing conditions.

Immediately after mixing of the concrete the measurements start. A steel bucket (volume = 11.6 dm^3, $\varnothing$ = 205 mm) contains a tube to protect the temperature sensor in the center of specimen. This tube is filled with 3 ml silicon oil. The sample is insulated by an air filled chamber surrounded by a metal jacket. Multiple layers of insulation are inserted between the jacket and external shell. A ventilator at the top of the jacket is installed to guarantee good circulation of air in the chamber. The air temperature is measured by a temperature sensor placed directly in the air stream of the ventilator and heated by a silicon-oil-filled heat exchanger. The temperature difference between specimen and air ($\Delta T < 0.01$ K, thermal loss <0.02 K/h) is controlled by the PC. The weight of the specimen is measured before and after testing.

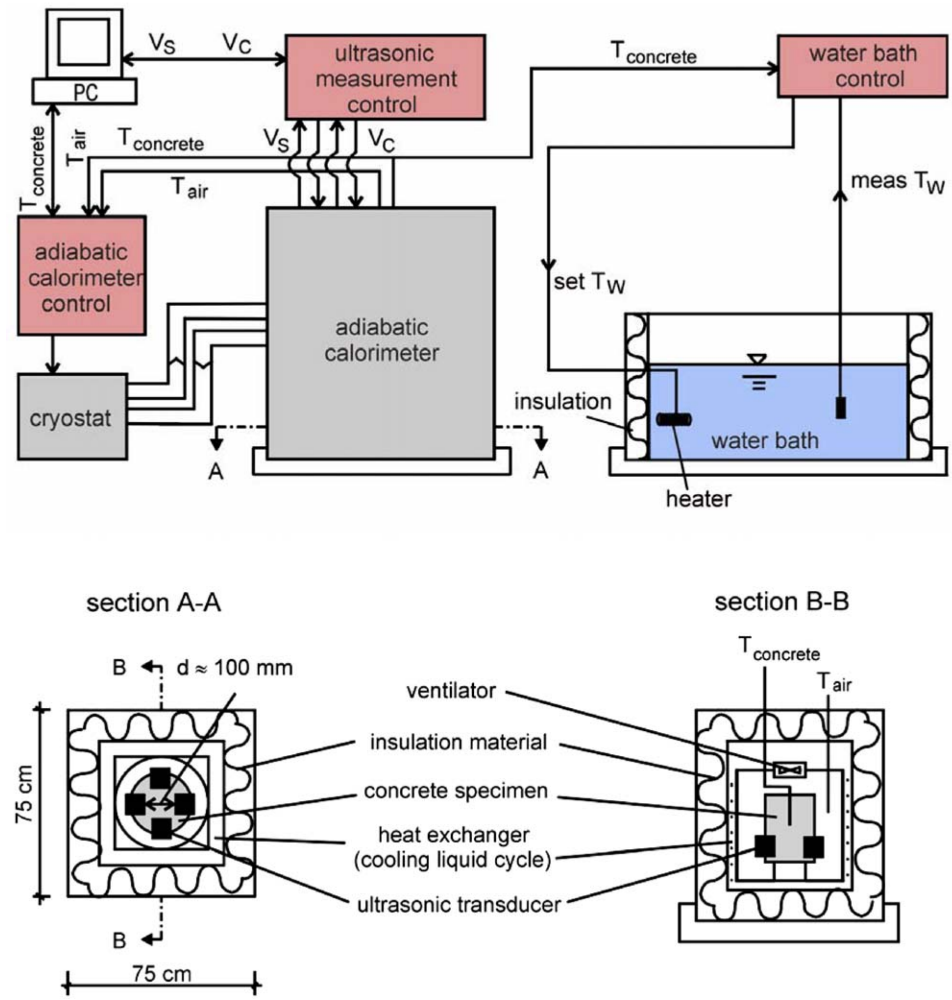

Fig. 2. Test set-up for ultrasonic testing under adiabatic curing conditions.

The ultrasonic transducers are integrated in the steel bucket (see Fig. 2). The transducers are protected by a plastic tube and held in place by a rubber seal ring. This construction guarantees that no moisture is lost during testing. The transducers are coupled directly to the concrete. For the receiver and transponder a commercial transition time measurement system is applied. Two types of commercial ultrasonic shear wave and compression wave transducers with a frequency of 50 kHz and 100 kHz respectively are used.

By measuring the ultrasonic pulse velocity of shear and compression waves the dynamic Young's modulus $\text{dyn}\,E$ and the dynamic Poisson's ratio $\text{dyn}\,\mu$ can be calculated with [7]

$$\text{dyn}\,\mu = \frac{1 - 2(v_s/v_c)^2}{2 - 2(v_s/v_c)^2} \tag{1}$$

and

$$\text{dyn}\,E = \frac{(1 + \text{dyn}\,\mu)\cdot(1 - 2\text{dyn}\,\mu)}{(1 - \text{dyn}\,\mu)} \cdot v_c^2 \cdot \rho_c \tag{2}$$

with ρ_c as the density of (fresh) concrete; v_c, as the velocity of compression wave and v_s, as velocity of shear wave propagation. The velocities can be calculated from the measured transition times t_c and t_s with $v_c = d_c/t_c$ and $v_s = d_s/t_s$, where d_c and d_s denote the thickness of the specimen in the corresponding direction. The dimensions d_c and d_s were determined for each single test and the values range from 90 mm up to 110 mm.

Usually the transition time of compression waves is defined as the first significant change in the amplitude of the measured signal. Such definition is not useful for young concrete because of its excellent damping properties. The expected signals are flat and have small impulses. It is hence difficult to identify the transition time. Other difficulties are the disadvantageous signal–noise ratio and the age dependent transducer–specimen system. To take these factors into account, and to ensure stable measurements the following procedure was applied. If the first maximum amplitude of the incoming signal is detected, this maximum amplitude is amplified to 100% of its intensity. The transition time is then determined by defining the point of time, at which the first amplitude of the incoming signal reaches

5% of the first maximum amplitude of the entire signal. To control this procedure A-scans are evaluated by hand and PC (see Fig. 3). Every 300–3600 s, 10 evaluations are made and the average value is stored.

The identification of the on-set time of shear pulses is much more complicated, because the transition time cannot be evaluated as the first significant change of the signal from the zero-line. The reason is that the shear wave transducer cannot generate a natural shear wave. But if the ratio of shear wave signal to compression wave ratio is big enough, it is possible to identify t_s [12].

It can be assumed that the measured signal is a natural combination of a compression wave and shear waves. Without any kind of restriction we can suggest, that the known compression wave can be described as a sine wave. From this point of view it is possible to define the starting point of transversal impulse as the first significant difference of the received signal from a sine wave. In general there exist four different possibilities to analyse the incoming signal. The starting point can be located between a minimum and maximum (a), maximum and minimum (c) or at a minimum (d) or maximum (b) of the measured signal. In Fig. 4 the procedure is shown.

For the calculation of the transition time, the point of time is used, which exhibits the first significant change of the compensated ultrasonic signal. For the evaluation of the s-waves, the procedure for evaluation of c-waves is adopted.

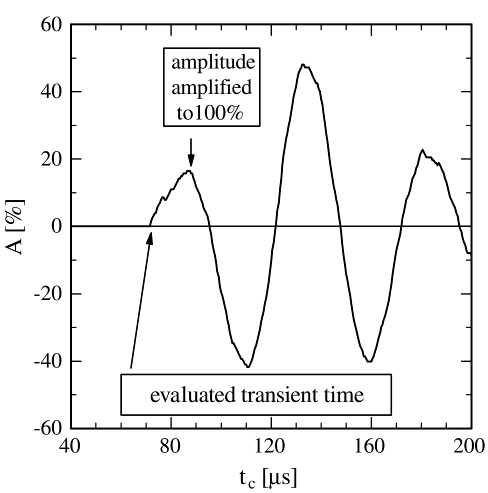

Fig. 3. Identification of transition time of compression waves.

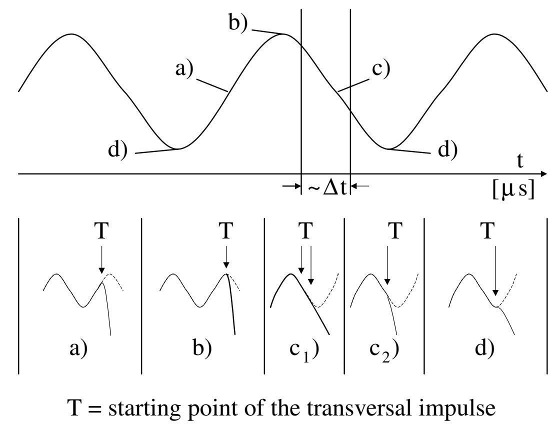

Fig. 4. Identification of transition time of shear waves.

In this paper the investigations of two HPC compositions are presented. The composition "German HPC" is a comparative German HPC. The second one is denominated "Norwegian HPC" because all constituents stem from Norway. This type of concrete was used for the recent strait crossings, e.g. Øresund bridge. All concrete compositions were comprehensively tested. Table 1 contains the main data of the tested concrete presented in this paper.

Further investigations regarding the heat liberation and the development of mechanical properties were reported in [13,14].

4. Models and test results

For the description of mechanical properties of the two investigates of concrete mixtures, models developed at the iBMB and those applied in Scandinavia are presented. More information about these models can be found in [15,16].

In the iBMB-model, the heat of hydration $Q(t_e)$ and the degree of hydration $\alpha(t_e)$ are expressed by Eqs. (3) and (4) where t_e is the equivalent age (Hansen–Pedersen approach); Q_{pot}, maximum heat of hydration (Bogue calculus); t_k, concrete specific parameter [h]; c_1, concrete specific parameter [–].

$$Q(t_e) = Q_{pot} \cdot \alpha(t_e), \tag{3}$$

$$\alpha(t_e) = \exp\left(-\left[\ln\left(1 + \frac{t_e}{t_k}\right)\right]^{c_1}\right). \tag{4}$$

The uniaxial tensile strength f_{ct}, compressive strength f_c and the Young's modulus E_{ct} can be expressed by

Table 1
Concrete compositions

Concrete no.	Concrete type	Cement content [kg/m^3]	w/c [–]	T_{c0} [°C]	Cement type	Remarks
CO21/R1–R2	OPC	370	0.40	22.2	CEM I 52.5R	20.0 [kg/m^3] SF[a] German HPC
CO11/I1–I5	OPC	370	0.40	20.4	CEM I 52.5R-LA	18.4 [kg/m^3] SF[a] Norwegian HPC

[a] SF = silica fume.

$$X_i(\alpha) = X_{i1}\left(\frac{\alpha - \alpha_0}{1 - \alpha_0}\right)^{n_i}, \quad X_i(\alpha) \in \{E_{ct}, f_{ct}, f_c\},$$
$$n_i = \begin{cases} \frac{1}{2} & \text{for } E_{ct}, \\ 1 & \text{for } f_{ct}, \\ \frac{3}{2} & \text{for } f_c, \end{cases} \tag{5}$$

where X_{i1} are the hypothetical end value of property X_i at $\alpha = 1$; and n_i, property specific exponent. Eq. (5) shows the values of n_i used by the iBMB model [15]. The values X_{i1} and α_0 are determined by regression of test results.

In the Scandinavian models, the heat of hydration, degree of hydration, strength and stiffness are generally expressed by

$$Y_i(t_e) = Y_{i1} \exp\left(-\left(\frac{t_e}{a_i}\right)^{b_i}\right). \tag{6}$$

The parameters Y_{i1}, a_i and b_i must be determined on basis of tests [14].

In this paper only a brief description of the development of the material properties of the two concrete mixtures is given. In Figs. 5–8 the development of the investigated material properties vs. equivalent age are shown. The calculated form parameters of the iBMB and the Scandinavian model are documented in Table 2.

It can be noticed, that referring to the development of degree of hydration determined from adiabatic heat release, the heat release of the German HPC starts earlier. This is caused by the higher amount of C_3A. It can be seen in Fig. 8 that the compressive strength of both mixes reaches values from 68 to 75 MPa. For the maximum values of uniaxial tensile strength values between 3.1 MPa and 4.1 MPa are measured (Fig. 6). The static Young's tensile modulus reaches values between 28 GPa and 34 GPa after 672 h (Fig. 7).

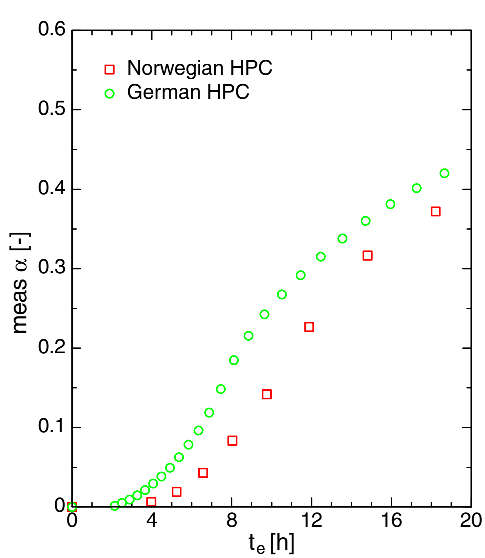

Fig. 5. Development of degree of hydration vs. equivalent age.

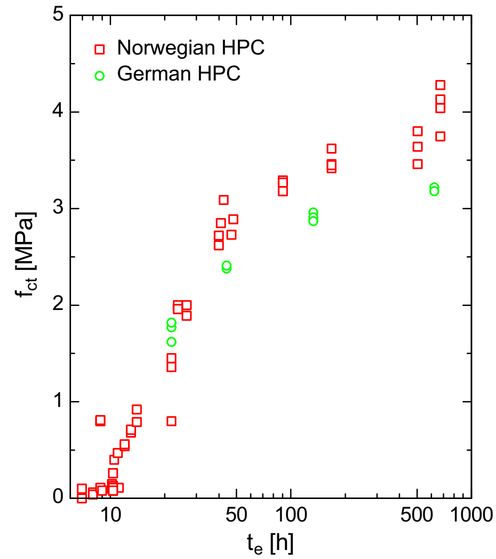

Fig. 6. Development of uniaxial tensile strength vs. equivalent age.

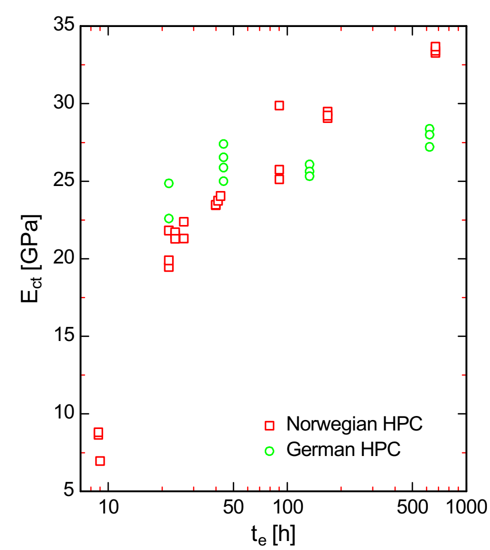

Fig. 7. Development of Young's modulus vs. equivalent age.

The most interesting point of these investigations is the development of the mechanical properties at very early ages. As deduced from the diagrams in Figs. 6–8 all mechanical properties of the Norwegian HPC start at nearly the same equivalent age. This initial setting time is about $t_{e0} \approx 9$ h. From Fig. 5, a degree of hydration of $\alpha_{0i} \approx 0.1$ can be derived. It can be noticed, that the end of the dormant phase for the Norwegian HPC is reached at $t \approx 8$ h and at $t \approx 5$ h for the German HPC.

Fig. 9 shows the original plots of the compressive and shear wave velocity for the "Norwegian mix" with respect to the equivalent age. One will see that – as expected – the compressive wave velocities are higher than the shear wave velocities. In Figs. 10 and 11 the evaluation of dynamic Poisson's ratio and dynamic Young's modulus vs. equivalent

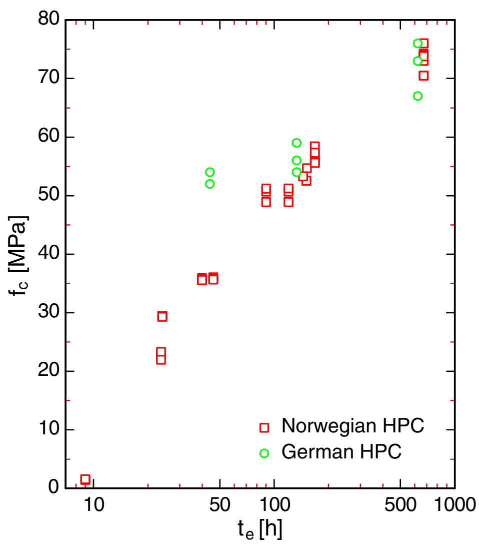

Fig. 8. Development of compressive strength vs. equivalent age.

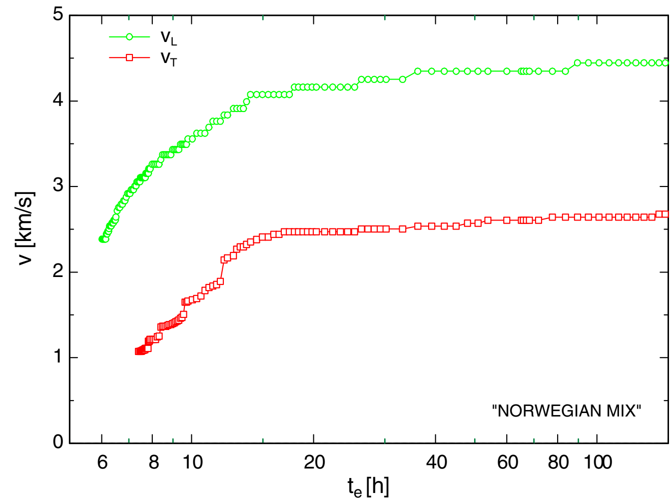

Fig. 9. Development of ultrasonic pulse velocity vs. equivalent age.

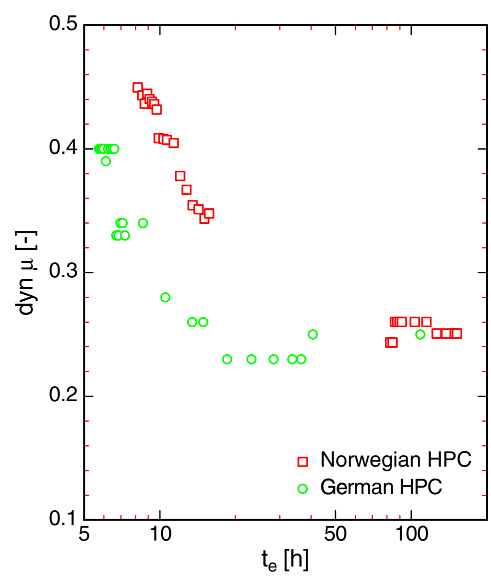

Fig. 10. Development of Poisson's ratio vs. equivalent age.

Table 2
Parameters of shape functions for Norwegian mix and German mix

				Norwegian mix	German mix
α	iBMB	Q_{pot}	kJ/m^3	174 864	192 831
		t_k	h	9.141	6.87
		c_1	–	−40.853	−0.816
	Skan	Q_{pot}	kJ/m^3	174 864	192 831
		a	h	22.21	15.80
		b	–	0.456	0.352
f_{ct}	iBMB	f_{ct1}	MPa	5.71	4.49
		α_0	–	0.17	0.12
	Skan	f_{ct1}	MPa	3.99	3.27
		a	h	36.32	14.33
		b	–	0.832	0.978
E_{ct}	iBMB	E_{ct1}	GPa	36.63	34.22
		α_0	–	0.17	0.12
	Skan	E_{ct1}	GPa	33.85	28.49
		a	h	16.11	0.427
		b	–	0.603	0.464
f_{cc}	iBMB	f_{c1}	MPa	124	117
		α_0	–	0.17	0.12
	Skan	f_{c1}	MPa	88.97	–
		a	h	94.16	–
		b	–	0.444	–

age for very early ages is presented. It can be noticed, that the end of the dormant phase for the Norwegian HPC is achieved after equivalent age of 8 h and after 5 h for the German HPC. These result confirm the observations of the tests presented above. The development of both properties starts nearly at the same time (6.5 h for dyn E and 11 h for stat E). To model the dynamic measurements also the equations presented above are used. These models (Eq. (5)) predict the test results well, as Fig. 12 shows. In the next step the gap in the development of mechanical properties between α_{0i} and α_0 has to be closed.

5. Improvement of the models of iBMB

On the basis of the test results discussed in the previous sections, the models described with Eq. (5) and (6) can be improved to better account for the early-age growth of strength and stiffness is taken into account. Fig. 13 shows the attempt. It is assumed that the value α_0 has been established with uniaxial tensile strength tests and that the value α_{0i} has been accordingly measured by ultrasonic tests under adiabatic conditions. The transition of value α_{0i} to the models, Eq. (5), is described by a linear function between α_{0i} and $\gamma\alpha_0$, with $\gamma \leqslant 1.\gamma$ is determines such that the slope of the extended model written as

$$\widetilde{X}_i(\alpha) = \begin{cases} X_i(\gamma \cdot \alpha_0)\left(\frac{\alpha-\alpha_{0i}}{\gamma \cdot \alpha_0 - \alpha_{0i}}\right) & \text{for } \alpha_{0i} \leqslant \alpha \leqslant \gamma \cdot \alpha_0, \\ X_{i1}\left(\frac{\alpha-\alpha_0}{1-\alpha_0}\right)^{n_i} & \text{for } \gamma\alpha_0 < \alpha \leqslant 1 \end{cases} \quad (7)$$

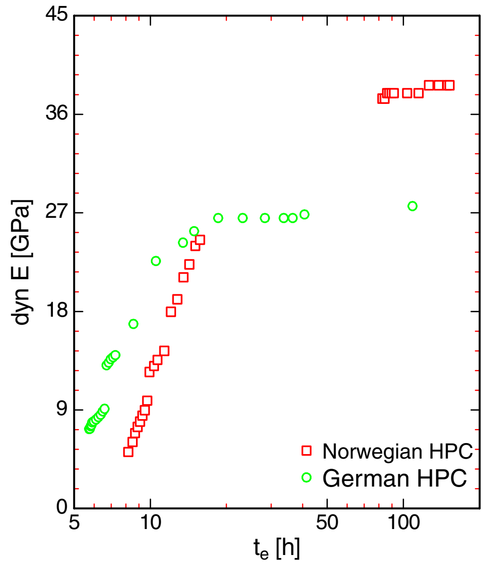

Fig. 11. Development of dynamic Young's modulus vs. equivalent age.

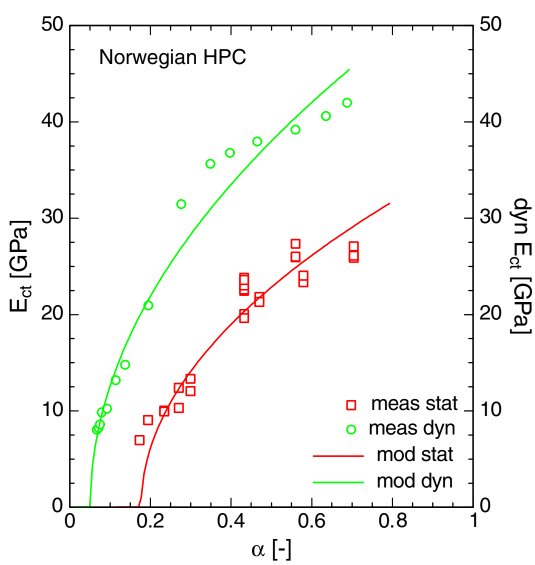

Fig. 12. Comparison of dynamic Young's modulus and static modulus of elasticity vs. degree of hydration.

and the E_{ct}-curve are of the same magnitude for $\alpha = \alpha_0\gamma$. Herein the abbreviations of Eq. (4) are used. With the initial value α_{0i} the associated effective age t_{e0i} is known. With that, the Scandinavian models, Eq. (6) can be revised by substituting t_e by $(t_e - t_{e0i})$. Table 3 shows the relevant parameters of both modifications.

6. Conclusions

For the prediction of thermal stresses and cracking, the realistic modelling of the mechanical properties of early-age concrete is essential. However many material models describe the material behaviour at very early ages, that is during setting and shortly after, only with insufficient accuracy. These problems are caused by difficulties to perform destructive tests at that age and to unambiguously relate the results to degree of hydration. The gradual transition of concrete into solid matter can only be distinctly described by non-destructive tests.

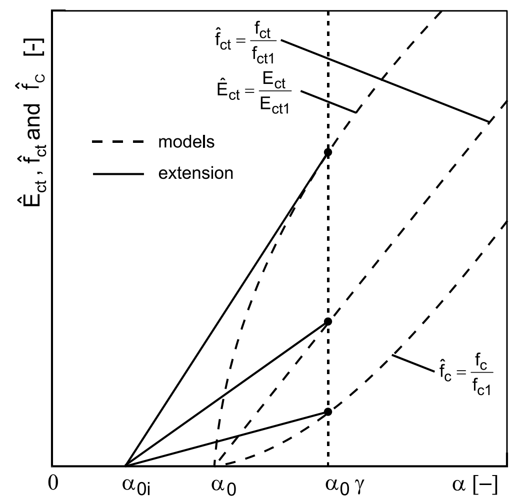

Fig. 13. Improvement of the models according to Eq. (7).

Table 3
Shape parameters of improved models for Norwegian and German HPC

	Parameters of iBMB model Eq. (7)			Parameters of Scandinavian model Eq. (6)		
Norwegian HPC	E_{ct1}	GPa	36.44	E_{ct1}	GPa	26.26
	n_i	–	0.5	a	h	11.94
	α_0	–	0.176	b	–	1.539
	α_{0i}	–	0.05	t_{e0i}	h	11
	γ	–	0.71	–	–	–
German HPC	E_{ct1}	GPa	34.22	E_{ct1}	GPa	28.59
	n_i	–	0.5	a	h	0.427
	α_0	–	0.12	b	–	0.464
	α_{0i}	–	0.03	t_{e0i}	h	6
	γ	–	0.77	–	–	–

By measuring the ultrasonic pulse velocities of compression and shear waves it is possible to determine the end of the dormant phase for arbitrary concrete compositions. The iBMB-model was extended in such a way, that the end of the dormant phase α_0 determined by extrapolation of destructive test results was combined with an initial end of the dormant phase α_{0i} determined via ultrasonic pulse velocity measurement for compression and shear waves. Satisfactory results can be obtained if these measurements are performed under adiabatic curing conditions.

Due to the fact that the presented procedure needs the evaluation of compressive and shear wave velocities in concrete, it is necessary to have an ultrasonics measurement device able to measure both types of waves for getting α_{0i}. Despite this the calibration of the presented model needs several uniaxial tensile test results including Young's modulus of elasticity at different equivalent ages to obtain a value for determining α_0 as well as a model curve for E_{ct} vs. α.

Practical application for this model is mass concrete RC- or PC-members since the conventional models are used to underestimate concrete tensile capacity at very early-ages leading to higher steel contents as necessarily.

Acknowledgements

The authors would like to thank the German Research Council (Deutsche Forschungsgemeinschaft) for financial support. The authors would also like to thank their teacher em. Prof. Dr.-Ing. Dr.-Ing. E.h. F.S. Rostásy for his valuable help over the years.

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