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Real time in situ characterization of concrete at very early age

Project No 200009 - Final Report to Cemsuisse Work completed to end of June 2003

Introduction

This report concerns the project "Real Time In Situ Characterization Of Concrete At Very Early Age" which began with a proposal to Cemsuisse in 1999 entitled "Testing of Concrete, Mortar and Cement Paste at Very Early Age using Fiber Optic Sensors". The success of this proposal led to initial development, tests and measurements which provided support to a joint project to the Commission for Technology and Innovation (CTI). The title of the CTI project is the same as the title of this report. Although the CTI funded only 50% of what was proposed, the work has been very successful.

The research has involved developing a methodology for monitoring concrete during the hardening phase in order to determine key material characteristics, such as thermal expansion coefficient. Results confirm the hypothesis formulated in the proposal and they support innovative approaches that were projected. Work is continuing within the scope of a doctoral thesis (M. Viviani).

Objectives and tasks

Objectives of this project are to:

- Increase understanding of the real behaviour of a concrete structure from pouring to the end of exploitation
- Develop a software product for real time in-situ characterisation of the structure at very early age
- Calibrate numerical models
- Provide support for adjustments to concrete mix design during construction (if necessary)
- Evaluate the efficiency of strengthening (refurbishment) of existing structures
- Propose standard procedures in order to ensure valorisation and wide dissemination.

The project thus has practical and theoretical parts. The practical part consists of laboratory research, in-situ applications and software development while the theoretical part consists of development and adaptation of models. The following tasks set was proposed (taken from the CTI proposal, May 2000) :

- A. **Development of a mortar sensor,** able to measure the very early age deformations of thin structural elements such as the strengthening or refurbishing of concrete layers and thin mortar layers.
- B. In situ determination of the **hardening** time.

- C. In situ identification and evaluation of the **most important components** of very early age deformation, such as **thermal deformation**, **autogenous deformation**, **drying deformation**, **deformation due to loading and creeping deformation**.
- D. **Development of software allowing real time identification of** the parameters in concrete.
- E. Monitoring of the evolution of the thermal expansion ratio.
- F. **Development of numerical and mathematical models** describing the evolution of the thermal expansion ratio and hardening time of concrete. Comparison with measurements

Task A deals with the development of a mortar sensor. Final test of the mortar sensor through laboratory and in-situ tests are planned. The mortar sensor measurements is then compared with the standard sensor measurements (Subtask A5).

Task B deals with the in situ determination of the hardening time of concrete. The hardening time depends of the rigidity of the stiff sensor. Rigidity depends upon Young's modulus and cross-section area of the sensor. Optimal rigidity of the stiff sensor is then established (Subtask B2), as well as the thermal expansion coefficient (TEC) of the stiff sensor (Subtask B3).

Task C is the core of the project. It involves in-situ identification and evaluation of the most important components of very early age deformation (Subtask C1). Next steps include the laboratory characterization of the specimens (Subtask C2), where the methodology developed to separate the contribution to the total deformation will be tested. Then the strategy will be tested in-situ (Subtask C3).

Task D takes into account practical aspects of the project by the development of software that facilitates real time identification of parameters. This software includes an interface between the software that manage the measurement and the subroutine that computes the output data (Subtask D1). A subroutine that analyses the measurements in real time is necessary in order to determine the evolution of the TEC, identify the hardening time of concrete, and separate different components of the total concrete deformation (Subtask D2). The development of an interactive tool for the visualization of results in a form of tables and diagrams (Subtask D3) will complete Task D.

In Task E, Monitoring of the concrete Thermal Expansion Coefficient (TEC) evolution is proposed. TEC is one of the most useful concrete properties and its value evolves continually in the early age of concrete.

In Task F, the result of the previous tasks is used to write and validate a simplified numerical model, which describes the evolution with respect to maturity of the TEC (Subtask F1)

	Description	Detai	ls	Current work		
A	Development of Thin (Mortar) Sensor	A1	Selection of the protective tube		End of taskA5	
		A2	Selection of the shape, dimension and material of the anchor pieces			
		A3	Conception of the passive zone			
		A4	Establishing of the sensor assembling procedure			
		A5	Final tests			
в	In situ determination of the hardening time of concrete	B1	Adaptation and changes of the dimensions of sensor.		Beginning of task B2	
		B2	Establishing of rigidity of the stiff sensor			
		B3	Establishing of TEC of the stiff sensor			
	Research and development of the method for very early age characterisation in the laboratory	C1 C2	Specim	en shape selection		
			C1.1	Shape of the sample	Beginning of	
			C1.2	Dimension and		
С				sensor equipment of		
				Choice of the sample		
			Laborat	ory characterisation of	task C3	
			the specimens		-	
		C3	Real time in situ characterisation of the structures			
						Development of software for real time identification of above mentioned parameters
	between the software that					
	manage the measurement and					
_	the subroutine that computes					
D		Development of the subroutine				
	D2					
	D3	Development of the user				
		interface				
F	Monitoring of the concrete	⊏1	Real-tin	ne monitoring of the	Completed	
	TEC evolution		TEC ev	olution	Completed	
F	Numerical modelling	F1	Development a of simplified		Not	
			numerical model for the IEC			
		F2	Numeria	cal modelling of the	performed	
			hardening time of concrete			
L			narueilli	is the of concrete		

Task changes during the project

Several modifications of the plan occurred during the project. Theses are principally related to tasks A, C and F.

Task A was almost completed. Two prototypes of the mortar sensors have been developed and their functionalities have been confirmed during laboratory tests. However, for the main goals of this project, the standard sensor provides enough flexibility. Thus, preference has been given to ensuring reliability results from the standard sensor. Nevertheless in future work, the mortar sensor will be embedded in very thin layers of mortar for tests that examine this kind of hardening behaviour.

Task C included the laboratory characterization of the moulds. This has been performed together with the separation of the effect through continuous measurements of the radial and vertical expansion of the moulds. Also, due to reduced CTI funding, Task C3 has been delayed.

Task F includes the development of simplified models of the thermal expansion coefficient. This task was not performed due to CTI funding limitations.

Progress of the work

The work completed is briefly listed for every task set as follows :

A. During the task set A, it was recognized that the standard sensor is soft enough for most requirements. Nevertheless, a new sensor, with very low axial stiffness has been developed. This sensor will be useful for monitoring hardening behaviour of very thin layers of mortar and low density materials.



Figure 1: Mortar sensor



Figure 2: A schematic of the mortar sensor

B. The determination of the hardening time in-situ was one of the major goals of the proposal "Testing of concrete mortar and cement paste at very early age". The in-situ detection of the hardening time is now proposed by a company in Switzerland (see <u>www.smartec.ch</u>). Within the scope of this project, the equipment for detection of the hardening time has been modified in order to insure repeatability and avoid errors due the positioning of the sensors and their holding apparatus. The deformation measurement of **fresh concrete** requires the use of sensors that are

directly coupled with the material, with a minimum effect of the holding and positioning system. The positioning apparatus (see figure 1), supports the sensors in the middle by the piece A, while the pieces B and C have just the function to keep the sensors straight only during pouring and vibration of the concrete.



Figure 3 Holding-positioning system for standard and stiff sensors

- C. This part of the work is the scientifical core of the project. Separation of contributions to the total deformation, in real-time and in-situ is useful for decision-making during every stage of construction. In the original project, a maturity technique was proposed to separate contributions of the total measured deformation. This technique requires a knowledge of reaction trends in order to be applicable. The available methods (ASTM C-1074, ACI 228) are both expensive and time consuming. Thus, a **new methodology** has been developed. This methodology uses the same measurements mentioned in the CTI plan in order to obtain values of the activation energy, a key constant in the Arrhenius equation. Testing has demonstrated the validity of the approach and the possibility to decompose the total deformation into components that reflect physical and chemical phenomena.
- D. Software, written in Matlab, is capable of acquiring the data in Microsoft Excel format and reduce them, giving the maturity history of all deformations in form of graphs. Matlab interfaces allow the maximum freedom for data screening.
- E. The thermal expansion coefficient (TEC) is one of the main characterising parameters of concrete. The knowledge of TEC evolution helps determine

countermeasures that avoid early cracking. The evolution of the TEC has been successfully measured during the project

Testing

The results obtained during testing are summarized in this section. Separation of the effects is based on the observation that strains in hardening materials are caused by phenomena such as humidity loss, temperature variations and autogenous desiccation. Through curing specimens such that conditions are created where one phenomenon is suppressed, relative influences can be identified. Pours of the same material, hardening in various environments enable comparison through Arrhenius-based laws (see Appendix A).



Figure 4 A schema of the monitoring strategy

Two test series have recently been performed, the first test has been held at the IMM laboratory (Lugano) at the end of January 2003, the second at the LMC-EPFL (Prof. Scrivener) at the beginning of June 2003 (see Fig. 5). The tests lead to improvements of knowledge and promising results. A complete separation of the effect (due physical phenomenon) has been possible in both cases, with a notable improvement in the second test. In both tests, the activation energy has been carried out with the newly developed methodology. Maturity histories of the deformations were calculated. Maturity histories allow direct comparisons of the deformations. With reference to Fig 4, specimen S1 is temperature insulated and hydro insulated, specimen S2 is hydro insulated, specimen S3 has no insulation. Size and material of the moulds are the same, as well as the positions of the sensors. Concrete is the same in the three cylinders. Deformations and temperatures are monitored in all cylinders. The mix design used for the test in IMM is shown in Table 1, while the one used in LMC is described in Table 2.

Experimental details

During testing, several sensors have been used. The deformation sensors are SOFO sensors of two types; stiff and standard (see figure 3). Thermocouples have also been used; all of them of type K. The data logging and reading equipment includes a SOFO V reading unit, a 30-channel optical switch and 3 ADAM-SOFO modules and HP generalpurpose reading unit. For the tests at IMM, in order to increase the temperature of concrete in the specimen S1 (see Fig. 4), high power bulbs have been used, while in the second test has been preferred a 10 cm wrap of rock-wool. The specimens used to decouple the drying effect (specimen S3, see Fig 4) are different for the two test series. In the IMM tests, a 73 X 55 X 32 cm box has been used while in the second test, a cylinder with diameter of 31.5 cm and 100 cm high was employed.



Figure 5 Installation of the specimens, tests at LMC

In order to test the applicability of the methodology, testing has been carried out with two distinctly different concretes (see Tables 1 and 2). Both of them are used for large civil engineering structures.

Water/cement Ratio	0.45
Cement II / A-LL 42.5 R (Morbio inferiore)	325 Kg/m ³
Superplasticizer	0.9%
Air Entrainer	0.1%
Aggregate	0-32 Hüttwangen
Maximum temperature difference	5 °C

Table 1 Concrete used for the tests at IMM

Water/cement ratio	0.48
Cement I 42.5 (Eclèpens)	350 Kg/m ³
Superplasticizer	0.8%
Air Entreiner	No
Aggregate	0-32 Sergey
Maximum temperature difference	15 °C

Table 2 Concrete used for the tests at LMC

Results

Data analysis and reduction

Results of the IMM test are presented in Fig. 6, 7 and 8 (see Fig. 4 for specimen labels). In the first two figures, the measurements of stiff and standard sensors, as well as the temperature measurements are presented. The measurements were carried out over more than 350 hours. The measurements in specimen S1 (see fig 6) underwent changes in magnitude at around 25 hours. This is due to an unexpected flow of cold air on the cylinders. Nevertheless, results remain useable.



Figure 6 Deformations and temperature of specimen S1, tests at IMM



Figure 7 Deformations and temperature of specimen S2, tests at IMM



Figure 8 Deformation and temperature of specimen S3, tests at IMM

The results shown in Figs. 8, 9 and 10 are used to determine the activation energy. Then, maturity histories of deformation have been calculated in terms of equivalent age, see Appendix A. Maturity histories are directly comparable and thus, because the only difference in terms of external conditions between the specimens S1 and S2 is the temperature, the thermal expansion coefficient can be calculated (see Fig. 9)



Figure 9 Evolution of the thermal expansion coefficient, tests at IMM

The evolution of the thermal expansion coefficient evolution is coherent with values found in the literature. Once the TEC is available, the deformation due to thermal effects can be decoupled in the specimens S1 as well as in the specimen S2. Thus, the autogenous deformation is found (see Fig. 12). The values obtained for the autogenous deformation are also coherent with those found in literature.



Figure 10 Evolution of the autogenous deformation, tests at IMM

Knowing the TEC and the autogenous deformation, the effect of the external conditions on the free surface of the specimen S3 can be decoupled. This is presented in Fig. 11



Figure 11 Evolution of the drying deformation, tests at IMM

Results of the LMC tests are presented in Fig. 12, 13 and 14. In the first two figures, the measurements of stiff and standard sensors as well as temperature measurements are presented. Measurements were carried out for more than 200 hours. All results are coherent with expected trends. The stiff sensor of the specimen S2 (see Fig. 13) malfunctioned at 60 hours. However, data were sufficient to obtain desired results.



Figure 12 Deformations and temperature of specimen S1, tests at LMC



Figure 13 Deformations and temperature of specimen S2, tests at LMC



Figure 14 Deformation and temperature of specimen S3, tests at LMC

In the LMC test, the use of rock-wool wrap instead of high power bulbs, in order to heat the specimen S1, improved the quality of results. The graph of TEC, autogenous deformation and drying effect, presented in Fig. 15, 16 and 17, are coherent with the examples found in literature.



Figure 15 Evolution of the thermal expansion coefficient, tests at LMC



Figure 16 Evolution of the autogenous deformation, tests at LMC



Figure 17 Evolution of the drying deformation, tests at IMM

Activation energy

As mentioned above, values for the **activation energy** (see Appendix A) are needed within the scope of this project. This parameter has been evaluated with a new methodology, together with the **rate of reaction**, which is another hydration parameter. These two parameters allow separation of the contributions to total deformation, as well as **prediction of key mechanical properties**, such as compressive strength, a short time after the pouring of concrete. The evolution of compressive strength has been predicted within 100 hours and compared with values obtained with classical compressive tests (see Fig. 18 and 19). The results indicate that early predictions of the activation energies and their use in an empirical strength formula (Appendix A) constitutes a promising methodology for strength prediction.



Figure 18 Compressive strength prediction, tests at IMM



Figure 19 Compressive strength prediction, tests at LMC

Conclusion

The work performed in this project demonstrates that a decomposition of the effect on the total deformation according to various physical phenomenon is possible in real-time and insitu. More specifically, during this work the following results have been acheived:

- Autogenous deformation, thermal expansion coefficient and drying deformation have been calculated for two types of concrete mixes
- A new, low axial stiffness sensor has been developed
- A software for the treatment and interpretation of data has been implemented
- The activation energy has been approximated using a new methodology. This lead to prediction of strength evolution, including 28-day strength, within 100 hours

These results contribute to a greater understanding of important aspects of concrete hardening. Autogenous deformation, for example, is the subject of thousands of scientific papers and several research groups around the word are attempting to monitor trends. The combination of new sensors, measurements, strategies and data reduction methodologies allow calculation of autogenous deformation in real time and in-situ. The same conclusions can be drawn for the thermal expansion coefficient and drying deformation. Moreover, the the approximation of the activation energy, allows the early prediction of the evolution of important mechanical properties of concrete, such as compressive strength. Further research into all of these topics is under way.

Prof. Ian Smith Lausanne, 25.08.2003

Appendix A

The Arrhenius Law

Hardening reactions are characterized by a change of state of the reactant when they lead to products (liquid suspension to solid). The resulting solid includes both the reaction products and the surplus of reactant. A typical example is cement paste. If the paste has a water to cement ratio of 0.1 the solid will be a composite of hydration product (CSH) and not-hydrated cement. A good choice of reactant concentrations is thus necessary in order to ensure desired solid phase characteristics. The speed of a chemical reaction is expressed in term of its rate of reaction:

RATE OF REACTION = $\frac{\text{Increase in concentration of products}}{\text{Time in which the increase take place}}$ mole/dm³ * Sec

Causes of rate of reaction changes are:

- a) CONCENTRATION: If there is more of a substance in a system, there is a higher chance that molecules will collide and speed up the rate of the reaction.
- b) TEMPERATURE: When the temperature of a system increases, the molecules bounce around more (because they have more energy), thereby making them more likely to collide. This means they are also more likely to combine. When the temperature lowers, the molecules are slower and collide less, thereby lowering the rate of the reaction.
- c) PRESSURE: Pressure affects the rate of reaction -especially when reactants are gases. When the pressure increases, the molecules have less space to move around. Greater concentration makes them collide with each other more often.
- d) Increased agitation
- e) Use of a catalyst
- f) Increase surface area of reactants

A synthesis of such concepts is the law established by Svante Arrhenius [1]. The rate of reaction, k is defined as

$$k = Aexp \frac{-E_a}{RT}$$
 Eq. 1

- T Absolute temperature (°K)
- E_a Activation energy (KJ/mole)
- **R** Gas constant (KJ*mole⁻¹* $^{\circ}$ K⁻¹)
- A Frequency factor (s^{-1})

This law states that the **rate of a chemical reaction increases exponentially with the absolute temperature**, regardless of the degree of reaction already obtained. A reversible reaction might cease and invert its direction according to the [Eq. 1]. This rule is valid for a wide range of reaction –from the formation of the iron rust to the baking of cakes– and it has been employed in research and practice. Through this equation, is possible to quantify the **degree of reaction** by integrating the rate of reaction rate [Eq. 2]. The degree of reaction, α , is useful because it computes the percentage of products that are generated during an elapsed time [2].

$$\alpha = \int_{t_0}^{t} Aexp \frac{-E_a}{RT} dt \qquad \qquad Eq. 2$$

For every process starting with the same reactant in terms of concentration and quality, the final value of the degree of reaction will be the same (100%). At every value of the degree of hydration, a new composite material is created with its own thermo-chemo-mechanical properties.

Use of the Arrhenius equation in civil engineering

The use of temperature-kinetic principles in civil engineering started at the end of the 1940s when researchers discovered the influence of temperature on the strength gain of concrete. The combined effect of elapsed time and temperature are included in the 'Maturity index', [3 and 4]. Maturity is the area under the temperature-time history graph (see Eq. 3). According to Saul, it has the following meaning: "Concrete of the same mix at the same maturity (reckoned in temperature-time) has approximately the same strength whatever combination of temperature and time go to make up that maturity."



- *M(t)* Maturity (degree hours)
- Δt Time interval (hours)
- T_a Average concrete temperature during the time interval Δt (°C)
- T_0 datum temperature (°C)

It is possible to correlate the maturity index with the strength gain for a certain mix-design. Once the strength-maturity curve is experimentally known for a given mix design, the strength is estimated in-situ by the knowledge of the temperature history (see Eq. 3). While this principle is suitable for the later phases of concrete life, is not accurate for hardening stages. In the 1970s, construction companies began attempting to complete work as soon as possible, especially by removing formwork earlier.

Studies related to concrete maturity began in the 1970s and these results are synthesized in the "Maturity Method" [2]. The concept of maturity remained substantially Saul's until, in 1977, Freiesleben-Hansen and Pedersen [5] presented a new maturity index based on the Arrhenius equation. Observing that the hardening of concrete is a chemical reaction; the authors applied the Arrhenius law to describe it through a new index, called Equivalent age (Et) (see Eq. 4).

This index is particularly interesting because it allows comparisons of concrete pours (or specimens) that are hydrating at different speeds. The parameter, Et is the integral in time of the ratio between the rates of reaction of two processes occurring with the same reactants. One specimen is a reference for the degree of temperature history. The other specimen at every time t* has an equivalent age, Et. This means that at that time t* it has the same degree of reaction that the reference process had after the time Et [7]. Thus, if

the time history of a physical property is known for the reference specimen, physical properties are known also for the other specimen (see Fig. 2).

$$Et = \int_{t_0}^{t} \left[expQ\left(\frac{1}{T_a} - \frac{1}{T_r}\right) \right] dt \qquad Eq. 4$$

- *Et* Equivalent age (hours)
- T_a Temperature of concrete (OK)
- T_r Reference temperature (^{OK})
- *t* Time (hours)
- Q Activation energy divided by gas constant (E_a / R)

Properties of concrete that are related to the degree of hydration can be traced using the same procedure when two requirements are fulfilled

- Kinetic parameters are known a priori
- Relations between physical properties of concrete and degree of reaction are already established

Procedures to fulfil both of these requirements are explained in codes such as ASTM C1074; ACI 228 and SHRP C376. Unfortunately these methodologies require equipment, professionals [2] and time (it could take up to about 40 days). This impedes use of non-destructive testing methodologies as a means for replacing less accurate concrete acceptance criteria such as the 28-day compressive strength.

Examples of relations are given below. The relation of Knudsen [Eq. 5] and [6] is of greatest interest.

$$S(k,t) = S_{u} \frac{\sqrt{k_{r} (t - t_{0})}}{1 + \sqrt{k_{r} (t - t_{0})}}$$
 Eq.5

- S Compressive strength at age t,
- S_u Ultimate strength,
- k_r Rate of reaction at the reference temperature Tr,
- t_0 Age at start of strength development.

This links the compressive strength to the rate of reaction (k). It has been widely used to extract kinetic parameters, and many codes apply it in practice.

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