Setting and hardening concrete

Thermomechanical effects

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Lhe hydration of cement is an exothermic, heat-activated reaction. In solid (or heat-insulated) parts, exothermicity results in a rise in temperature which, in some areas, can reach around 50 degrees. In thinner parts, thermo-activation can be used on site, and especially in prefabrication plants, to accelerate resistance growth. After a review of the main thermal properties of cements and concretes, this article presents the three most-important features of these products.

These are the main thermomechanical effects that are important to the engineer:

*— the **risks of cracking** in massive structures or structures with complex geometries, and the numerical models that can be used to analyze these risks (and, where appropriate, to choose between techniques to limit crack openings);*
*— **industrial techniques for** accelerating the growth of a material's mechanical strength on site or in the factory, using various types of heat treatment (**thermomaturation**);*
*— **on-site methods** which, in the hours following concrete setting, enable us to estimate the mechanical strength acquired at selected points in the structure, based on temperature recordings in the structure itself (**maturometry**).*

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1. Hydration heat cement and concrete hardening

1.1 Cement hydration: an exothermic, heat-activated reaction

The setting and hardening of concrete is the result of a number of chemical reactions, the main ones of which - the hydration of the silicates in clinker - are accompanied by a high release of heat. In addition to the difficulty of defining and isolating these various reactions, the heats of hydration of pure species are rather tricky to measure, and the literature does not provide precise values.

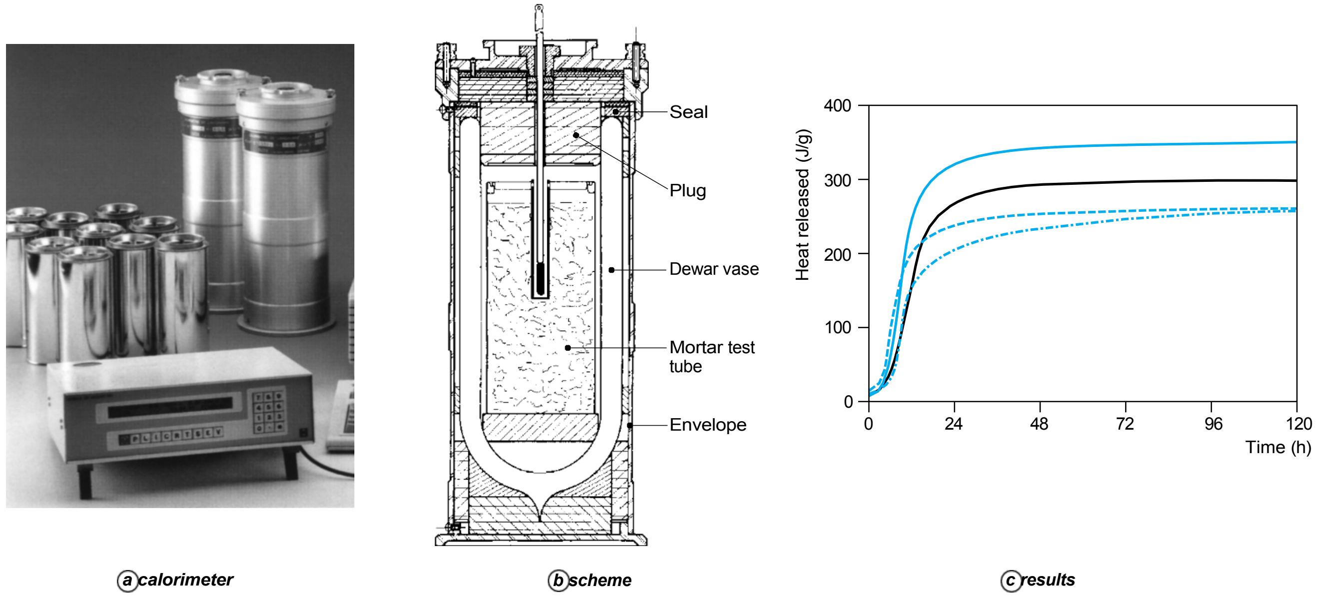
Note: the ***heat of hydration*** is the sum of the enthalpy of formation and the heat released by the absorption of adsorbed water molecules.

The following ranges (in joules per gram of anhydrous material) are used for the ***main constituents*** of clinker:

- — tricalcium silicate ($C_3 S$) .... 500 to 525 J/g ;
- — dicalcium silicate ($C_2 S$)........................... 250 to 270 J/g ;
- — tricalcium aluminate ($C_3 A$) ....1000 to 1200 J/g ;
- — tetracalcium aluminoferrite ($C_4 AF$) . . . . 395 to 425 J/g.

A cement's heat of hydration therefore depends on its chemical compo- sition. Given these values and the ranges of composition, it varies mainly with the $C_3 A$ content. In cement plants, it is systematically estimated by means of a standardized test (**the Langavant bottle test**, Figure **1**), which consists in recording, under semi-adiabatic conditions, the heat of hydration of the cement. temperature rise of a mortar with a fixed composition and a specified sand. This test, which provides reliable and practically interesting values at 12 h and 24 h, does not, however, provide the *final* values, as the error due to heat loss from the device becomes very large after a few days, when the hydrata- tion reactions are far from complete. Values at 12 h and 24 h depend on both total heat and reaction kinetics, which in turn depend on chemical composition (as the various hydration reactions do not have the same activation energy) and clinker fineness. Adiabatic calorimeters provide more reliable final values [3]. Depending on the cement, the total amount of heat released is between 200 and 500 joules per gram of cement. Most of this heat is produced within a few hours, in a flow that passes through one or sometimes two peaks, with a long period of decay, characteristic of the evolution of the material, the flow becoming weaker and weaker, but may remain significant over the very long term (slowdown due to the existence of an increasingly thick and compact layer of hydra- tes, which coats the grains of anhydrous cement).

For the moment (in 1997), we can't predict the total amount of heat that will be generated in a concrete, or the kinetics of its pro- duction, from cement data alone, its chemical composition or its specific heat of hydration. We simply note that the total heat released in concrete often increases proportionally faster than the cement dosage. On the other hand, it's easy to ***characterize*** a concrete mix using a test that involves recording the rise in temperature in a test specimen placed under **quasi-adiabatic** conditions (**QAB** test, figure **2**). The curve thus obtained is an expression of the material's *behavior law*, and is used to calculate temperature fields in concrete structures (§ 2.3).



ⓐ *calorimeter* ⓑ *scheme* ⓒ *results*

Figure 1 - The Langavant calorimeter, with diagram and results obtained with four different types of cement

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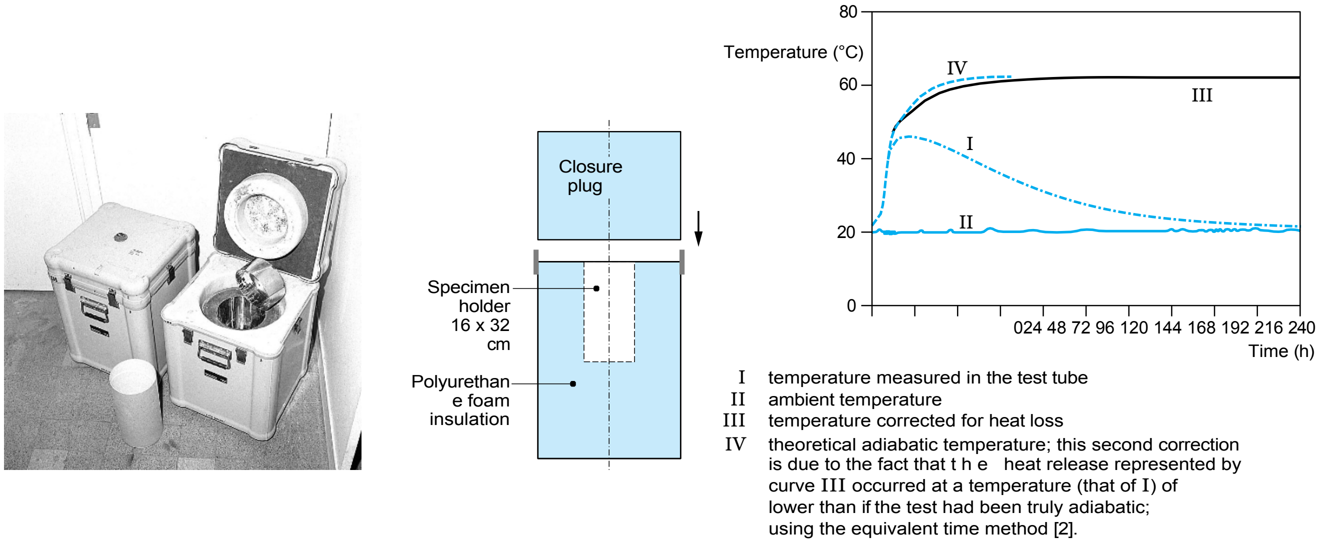


ⓐ *calorimeter* ⓑ *scheme* ⓒ *results*

Figure 2 - QAB calorimeter test, with diagram and results obtained with ordinary concrete [2].

1.2 Mechanical properties of concrete. Changes during hardening

Mechanical properties (compressive strength, tensile strength, Young's modulus) develop continuously as the material hardens, starting from *zero* (Figure **3**). Moreover, the growth of these characteristics is strongly *accelerated* by heat. As with many chemical reactions, the kinetics of hydration correspond fairly well to Arrhenius' law [19].

- **Arrhenius' law** states, for example, that if isothermal tests are carried out at different (constant) temperatures, the evolution curves obtained in all these tests are superimposed (reduced to a single curve, or *master curve*) with an *affinity* on the time scale.

The ***affinity ratio*** [an exponential of the inverse of the absolute tempé- rature, exp(- k/T)] depends only on temperature and, as a result (because there is thus *decoupling* between the two state variables of temperature and the degree of advancement of the reac- tion), this law is a true *law of behavior*, in the sense that it allows us to predict, from the mere knowledge of two isothermal curves, the evolution that would be obtained in any temperature history.

- The application of this law opens the way to models for calculating the temperature fields produced by hydration, as well as models for predicting mechanical properties. A simple formulation of these models uses the notion of concrete *maturity,* which is defined as the (theoretical) age at which the material would have reached, had it been kept at a constant reference temperature T_0 , the same state as that which it has actually reached under actual conditions of temperature $T(t)$, which may itself be variable. In the context of Arrhenius' law, maturity is written as follows:

$$\mu = \int_0^t \exp\left(\frac{k}{T_0} - \frac{k}{T(\tau)}\right) d\tau$$

where k is a material constant, expressed in kelvins.

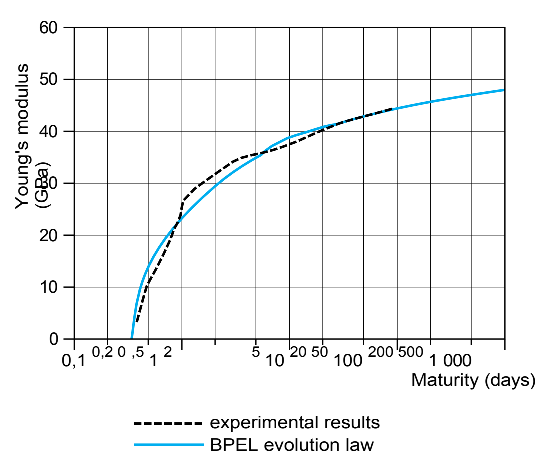


Figure 3 - Evolution of the modulus of elasticity of concrete over time: experimental values and law provided by the regulations calculation of BPEL concrete structures (prestressed concrete in limit states)

For CPA cement, k is of the order of 5,000 K, which means that the concrete hardening ***rate doubles every 12 or 13 Kelvin*** [3] [7].

Modulus (or tensile strength) can then be considered as a continuous, monotonically increasing function of the degree of hydration, i.e. of maturity, which can be calculated at any time. point of the structure from its temperature (calculated, as in paragraph 2, or measured, as in paragraph 4).

■ The **Young's modulus of concrete** (*E*) plays a major role in all structures subject to boundary conditions of more or less impeded displacements, particularly when these conditions apply from a very early age (pavements, dams, composite bridge slabs, rafts or thick walls cast in layers or successive lifts).

Under conditions of totally restrained deformation, for example, the intensity of thermally induced stresses is given by :

$$d\sigma = E\alpha\, dT\ ,$$

with α coefficient of thermal expansion.

If you want to influence Young's modulus values (as is sometimes the case with pavements or dams, for example, when you have a choice of aggregates), the most effective approach is to select aggregates according to their own modulus, firstly because coarse aggregates "weigh" more than the cementitious matrix on the value of the composite's modulus [16], but also because we generally have much less freedom over the parameters that vary the modulus of the cement paste, because of their impact on the value of the mechanical strength and rheological properties of the fresh concrete, which are fixed elsewhere.

1.3 Calorific and thermal properties of concrete

■ The **coefficient of thermal expansion (CTE)** plays an important role in parts that are subject to conditions of restrained movement and, moreover, are either thick (such as dams) or subject to significant temperature variations (such as pavements). Like Young's modulus, and for the same reasons of volume ratios, concrete's TDC depends more on that of the aggregates than on that of the cement paste.

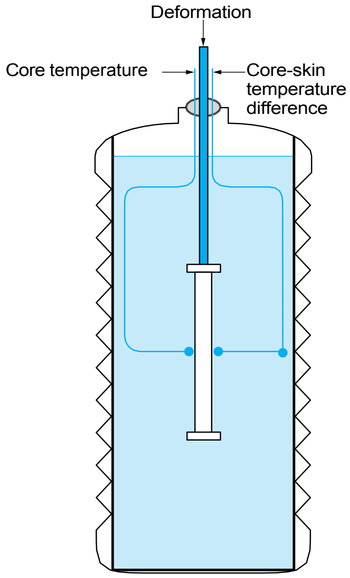
Its value can range from 6 × 10^{-6} or 7 × 10^{-6} for a concrete of limestone aggregates to 12 × 10^{-6} or 13 × 10^{-6} for all-silica aggregate concrete.

This is why limestone aggregates are often chosen for concrete pavements, although limestone with an excessively high Young's modulus should be avoided (stresses are proportional to the product of the two characteristics).

On the other hand, unlike Young's modulus, TDC is ***practically constant*** from the very first hours after setting. In experimental procedures, Arrhenius' law makes it possible to separate endogenous shrinkage and thermal deformation, to show that these two deformations are additive, and that TDC is constant after a few hours. To achieve this, two tests are carried out in flexible molds (Fig. ***4a)***, one under quasi-isothermal conditions and the other under quasi-adiabatic conditions. The measured deformations are then expressed as ***a function of the*** respective ***maturities***, and the difference between the two deformations ***at equal maturities*** is plotted as a function of the difference between their temperatures at the same maturity (Fig. ***4b***). Starting from point A (corresponding to cement setting), this relationship, whose slope represents the CDT, is linear ***and reversible*** [13].

The higher value observed until around 4 p.m. can be explained by the contribution of the liquid phase, as water has a much higher TDC. However, this phase has no mechanical consequences, given the very low value of Young's modulus in this (short) period [6] [13].

In most cases, **heat exchange and heat transfer coefficients** can be assumed to ***remain constant*** during hardening, as can the **heat content** of the material [5] (Fig. **5**).



The concrete is poured into a flexible PVC mold. An extensiometer is positioned in the center to measure core and skin temperatures.

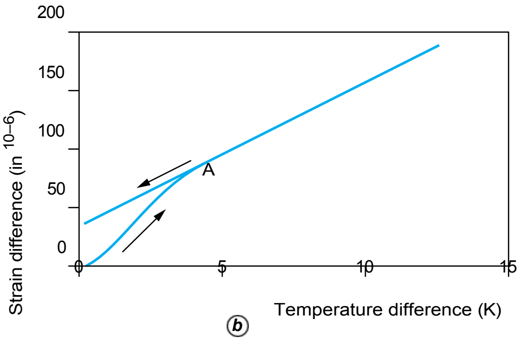


Figure 4 - Determination of the coefficient of thermal expansion at young age, by decoupling with endogenous shrinkage; a clear change in slope is observed (A), which corresponds to setting, beyond which the CDT remains constant [13].

1.4 Endogenous withdrawal

Cement hydration continues well after setting, as shown by the sometimes significant changes in the mechanical properties of concrete over the long term (Figure **3**). However, setting consumes a relatively small proportion (except for high-performance concretes) of the water used to make the concrete: 15 to 20 liters per 100 kg of cement, or less than half the initial water content of ordinary concrete. This is the main reason for the very high porosity of ordinary concretes: 8 to 16% of total volume, which represents 20 to 50% of the volume of the binder paste.

On the other hand, despite this excess of water, continued hydration leads to desiccation within the material (self-desiccation) as soon as setting begins.

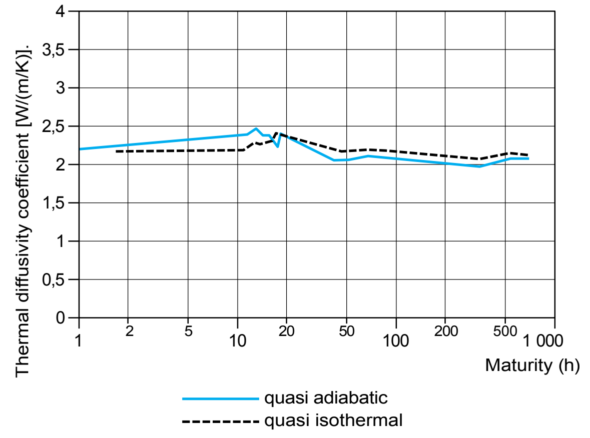


Figure 5 - Evolution of the coefficient of thermal diffusivity of concrete before, during and after setting, which in this case is between 10 and 20 hours [5].

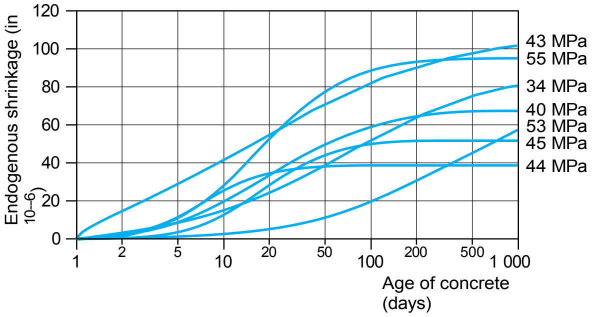


Figure 6 - Evolution of endogenous shrinkage for various ordinary concretes (E/C > 0.45) [16].

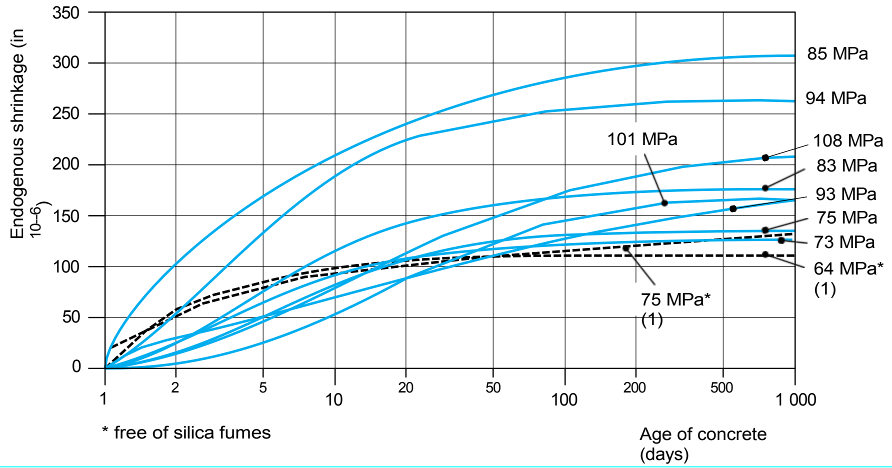


Figure 7 - Evolution of endogenous shrinkage for various high-performance concretes [16].

This is simply because the increase in solid volume is less than the volume of water consumed in the reaction. The volume balance shows a deficit of around 10% of the volume of hydrates formed [14]. This reduction in the relative volume occupied by the liquid phase in the pore space, together with desiccation, leads to deformation of the mineral matrix. If we observe that pressurizing a fluid to fill the pores would lead to apparent swelling, we see that, conversely, the tensions that exist in the liquid phase (and which, as we know today, respect the laws of surface tension, down to pore sizes of a few nanometers [9]) cause a contraction of the matrix, known as ***shrinkage***. Numerical homogenization techniques have made it possible to calculate this contraction from mercury porosimetry and water content data. The effect of capillary tension on the mineral matrix clearly outweighs other mechanical effects, and is therefore the origin of water shrinkage [11].

Endogenous shrinkage (shrinkage measured in the absence of any water exchange with the surrounding environment, and corrected for thermal deformation) remains below $^{10-4}$ (figure **6**) for concretes with a water/cement ratio (*W/C*) greater than 0.45, but increases rapidly when this ratio falls below 0.40, reaching 300 × $^{10-6}$ (figure **7)** [16]. This is simply an effect of pore size; stresses in the liquid phase (which cause compression of the mineral matrix) vary as the inverse of pore size at the interface with the gas phase. The time evolution of this deformation is directly linked to the cement hydration kinetics, which also control the evolution of the concrete's mechanical strength. The parameters that influence these kinetics are therefore also those that influence the rate of strength growth: firstly, the nature and fineness of the cement, and secondly*, the* W/C ratio.

The ***evolutionary kinetics of*** endogenous shrinkage follow those of the evolution of mechanical resistances quite closely: very rapid in the first few days, it progresses by 60 to 90% at 28 days. The total intensity of endogenous shrinkage remains moderate, but when added to other forms of shrinkage, can be significant: 100 × $^{10-6}$ to 300 × $^{10-6}$. The rapidity of its development at a young age means that this shrinkage has long been ignored by practitioners, since classic shrinkage tests - at least those used to provide the laws retained in calculation regulations (originally essentially for estimating precontract losses) - began at 48 h or 3 days.

As hydration is generally accompanied by a reduction in the moisture imbalance with the surrounding environment (except in the case of high-performance concretes with very low W/C ratios), this does not activate diffusion processes, and therefore gradients, and endogenous shrinkage is essentially an ***intrinsic phenomenon***, which can almost always be considered uniform throughout the volume of a part, at least within a zone corresponding to the same concreting operation (in the case of re-concreting, it's the dephasing of kinetics that generates mechanical effects). This means that, in a prefabricated part, or one-phase or continuous casting, and not blocked by its supports or formwork, this shrinkage has no mechanical effect.

On the other hand, when shrinkage is prevented, or even simply hindered, from *the very start of setting* (continuous rigid support, fixed supports, concreting resumption, etc.), it often constitutes a significant (and often overlooked) component of early cracking. It's important to remember that, under conditions of totally hindered deformation (as in the cracking bench test, for example, which is used to assess the risk of cracking in concrete pavements), the specimen will always end up breaking, This means that, despite stress relaxation (which is particularly rapid in young age), stresses due to endogenous shrinkage alone will always reach values equal to those of the material's tensile strength.

2. Controlling cracking at an early age

2.1 Stresses produced during setting and curing

The mechanical effects resulting from the temperature fields produced by cement hydration are often considerable, and in some structures (dams, foundations, etc.) can be far more dominant than strictly mechanical actions, especially in the early stages.

The heat produced by cement hydration creates an imbalance with the surrounding environment, generating chain flows and consequently temperature gradients.

■ The **deformations** resulting from temperature variations are therefore never uniform, which generates stresses. The deformations observed are therefore not a simple function of *local* temperature variation alone, but always involve ***structural behavior***. This structural behavior is often quite simple to analyze: for example, for a *non-planar* temperature field (i.e. a field that is, in a straight sec- tion, a non-linear function of spatial coordinates), the strength of materials allows, under the conditions of application of beam theory, a very simple decomposition of mechanical effects into two ***components*** [3] :

— plane deformation ;
— a field of self-constraints.

- The ***plane deformation*** itself comprises :

— elongation or (more frequently in the case of concrete) average shortening, which is obtained by calculating the average value of the imposed deformation over the cross-section;
— when the imposed field is not symmetrical, a rotation, which is obtained simply by writing the equality of its moments with those of the real field.

- The *self-stress* ***field*** is obtained by the difference between the real field and the equivalent linear field, defined and calculated as above.

■ Thermal **cracking** can be of two types:

— or skin cracking;
— or localized cracking.

- ***Skin cracking*** (as in continuously cast foundation blocks, headers or voussoirs on piles) is due, not to the local gradient, but to *the deviation of* the local temperature from the equivalent linear field; this skin cracking is, however, rarely very open, as the distance between two consecutive main cracks is of the same order as the depth of the tensioned zone, which cannot exceed a quarter of the thickness.

- In the case of repeated concreting (thick veil cast in successive lifts) or embedment (dam, veil embedded on a foundation mas- sif or on a continuous footing, pavement on a rigid layer or soil), ***the cracking is localized*** and often much more widely spaced, and therefore much more open; on continuously reinforced concrete (CRCP) pavements, for example, distances of over 50 m between cracks have been observed. Crack spacing can thus be highly variable (the range of distances observed on structures goes from a few centimetres to several tens of metres), and the great extent of crack openings observed is therefore linked to the crucial role of the structure's *mechanical boundary* conditions, much more than to the material's thermal and mechanical parameters.

To understand, analyze and quantify these effects, the engineer needs data on the following four elements:
1) the law that describes heat release during hydration (and which must reproduce the thermo-activation phenomenon);
2) values of heat transfer and exchange coefficients, by volume and by surface;
3) the value of the coefficient of thermal expansion (CTE);
4) that of the Young's modulus [*E-modulus*] of concrete, with its law of evolution over time.

2.2 Thermal shrinkage

The hydration *heat of* cement plays a major role in massive structures (dams, foundation blocks), but also in more modestly thick structures (from 20 cm, when one face is thermally insulated), when these are subjected to boundary conditions of prevented movement, either totally (pavements), or strongly (composite bridge slabs). In both types of structure, the effects of thermal shrinkage are added to those of endogenous shrinkage; concretes with high heat of hydration are also often those with high endogenous shrinkage, as are most high-performance concretes (HPC).

Site experience clearly shows that, as long as the concrete thickness remains below 30 cm (or 20 cm when only one side is cooled), thermal effects are nil or very slight. Conversely, as soon as there is a zone of concrete whose distance from the nearest cooled surface exceeds 50 cm, the temperature of the concrete will rise by 30 to 50 Kelvin (or even 55 cm with high-dosage CPA HP or HPR), and cracking is then practically inevitable during cooling. Recent observations on construction sites using high-performance concretes - which often have higher heats of hydration, but also faster hydration kinetics than conventional concretes - show, however, that non-negligible thermal effects can be achieved with HP concretes at thicknesses of less than 30 cm (figure **8**).

Depending on the dosage and type of cement, thermal shrinkage can reach 400 × $^{10\text{-}6}$ to 500 × $^{10\text{-}6}$ at the core of the parts once the thickness exceeds a certain value. The kinetics of this

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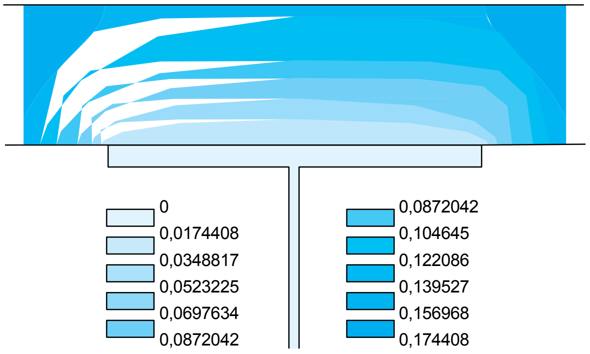
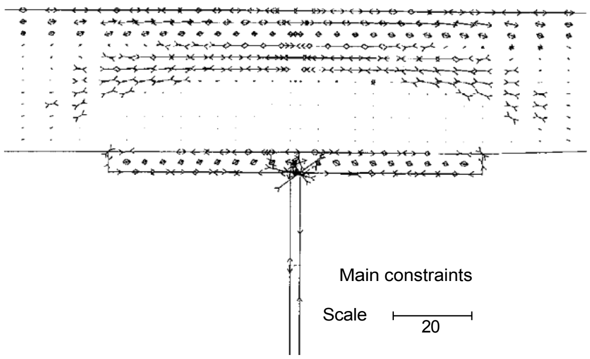


Hydration degree field



Stress field on support. Partial connection

Figure 8 - Thermal stress field in the slab of a composite bridge, for form stripping at 4 p.m.

shrinkage is simple: it begins with the end of setting (the maximum temperature is reached between 20 and 40 h) and the duration of cooling (which has the appearance of a decreasing exponential) is proportional to *the square of* the thickness (the average temperature is divided by two: for a 20-centimeter-thick slab ventilated on both sides, every hour; for a 40-centimeter-thick slab, every 4 h; for a 100-centimeter-thick slab, every 25 h, etc.).

2.3 Stress calculation. Controlling the risk of cracking

The data provided in paragraph 1 make it possible to carry out a complete numerical ana- lysis of the mechanical effects produced in concrete structures by cement hydration. Full details of these numerical models, their mathematical formulation and their theoretical and experimental bases, can be found in [1]. They use the heat equation with a source term in the second member and, to account for the thermo-activation phenomenon, Arrhenius' law. This calculation is thus coupled with diffusion: in the vicinity of the surface, for example, which is cooled by the outside temperature, the release is slowed down, and therefore lasts longer, the slower growth of modulus and resistance in these zones is thus restored by the model. This kind of calculation is It has been widely validated by a large number of applications, which have confirmed the predicted values through *in situ* measurements [20]. It is systematically used for exceptional structures or where there is a risk of thermal cracking (figure **9**).

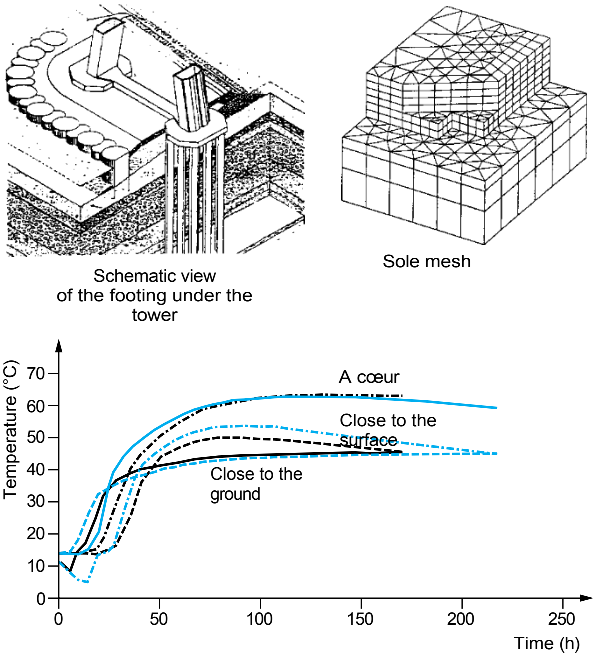


Figure 9 - Calculated temperatures at several points on the footings of the Pont de Normandie, and comparison with on-site measurements [20].

3. Heat treatment of concrete

3.1 General

The addition of thermal energy during setting accelerates concrete hardening. This thermo-mechanical coupling, often referred to as thermo-activation, can be put to good use on construction sites and, above all, in precast plants. In 1983, Marc Mamillan wrote, in the introduction to the previous version of this article: "*... heat treatment is the most effective way of obtaining the strength required for demolding in just a few hours...*". Today, this is no longer entirely true. The development of high-performance concretes, with the use of admixtures and mineral additives (in particular silica fume, which is now a component of certain cements), has shown that it is possible to obtain very high mechanical strengths at ages of less than 24 hours (the target times are often 18 or 20 hours, as these are the times which guarantee compliance with a schedule based on daily cycles), and at an overall cost which is of the same order.

It should be added that any heat treatment leads to a reduction in long-term mechanical performance, whereas, conversely, high-performance formulations lead to an increase in mechanical performance, as well as an improvement in most of the characteristics that contribute to the material's durability.

High-performance formulations, however, are not always the best. The recent example of composite bridges, for which this type of formulation was frequently proposed by companies in the early 90s, has shown that, where there is a risk of early-age cracking due to shrinkage prevention, the use of these concretes is not the best solution.

This example of composite bridges (consisting of a metal girder connected by connectors to a concrete slab) also shows that these two methods of accelerating hardening are not equivalent: the high-performance concrete method is only acceptable in the case of prefabrication of the hourdis elements (but this raises the problem of the bonding mortar); the heat treatment method is only acceptable if the metal girders are heated at the same time as the hourdis concrete.

Finally, it should be added that active heat treatments can give rise to disorders that are difficult to control, when the heat input coincides with the end of the dormant period. The (strong) couplage between this external input and the heat of hydration can lead to temperature rises well in excess of fifty Kelvin (above 80°C, there is a rapid degradation of all the material's mechanical performance).

In practice, several technologies can be used for heat treatment of concrete.

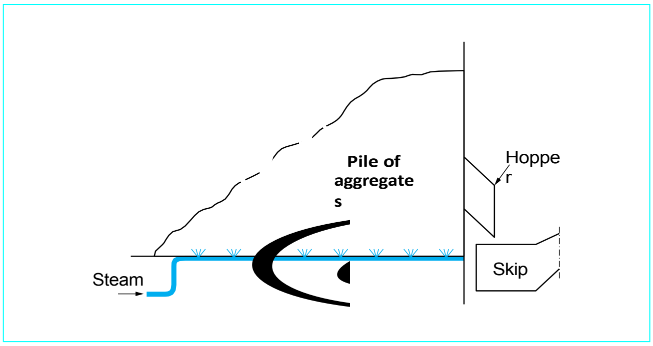


Figure 10 - Preheating concrete by heating aggregates

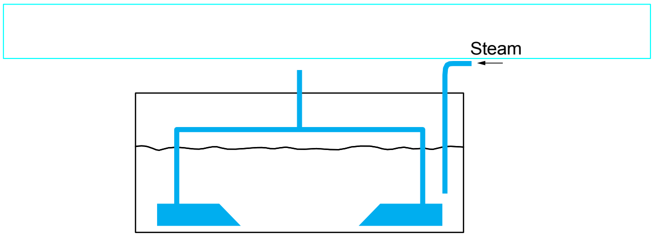


Figure 11 - Preheating concrete by injecting steam into the mixer

3.2 Passive heat treatment or "self-baking

The simplest method is to reduce heat loss during setting by using insulating formwork, sometimes simply covered on the outside with a coating or spray of insulating material (vermiculite, etc.), or by covering the entire structure with an insulating tarpaulin. The tarpaulins used on site, as well as formwork coated with 5 cm of insulating mortar, reduce the surface heat-exchange coefficient by more than 100 compared to bare metal formwork (whose ribs and reinforcements act like the fins on a radiator). This method is often the most economical (no heat is supplied) and the safest; ultimately, eliminating all exchange with the surrounding environment eliminates any temperature gradient [2].

3.3 Heating the concrete before placing

This technique is also known as **preheating** [17]. Preheated concrete can be obtained in three different ways:

— or by ***heating the aggregates*** and/or water before mixing, the aggregates generally being heated by steam injection in the lower part of the storage piles (figure **10**);

— or by heating the mixture ***by injecting steam into the mixer*** during concrete production (figure **11)**;

or, finally, by electrically ***heating*** the concrete ***in a bucket*** after mixing, using electrodes to pass current through the fresh concrete for a few minutes, which then acts as an electrical resistance.

The latter technique is the most energy-efficient method of prefabrication. It was initially developed in what was then the USSR, where it was observed and analyzed by Jay and Caumette [12], who called it the "*heated bucket"* technique and introduced it to France in the early '80s, demonstrating that its benefits were not limited to cold-weather concreting, but that it could also be used - particularly in conjunction with heat-insulated formwork - to accelerate material hardening, taking advantage of the thermo-activation phenomenon. They then developed the bucket and electrodes (Figure **12**).

When using pumped concrete, a similar technique, also based on conduction heating, consists of placing an element at the end of the pump pipe (heat exchanger with heat-transfer fluid circuit or electrical resistors) which heats the concrete during pumping.

At a flow rate of 15 m^3 /h, the temperature of the concrete can be raised by around 20°C.

3.4 Heating concrete after placing

For a long time, this was the most widely used technique [10] [18]. Two principles can be distinguished: internal heating, applied in the mass, and external heating, through the walls, the most frequently used method.

- For **indoor heating**, the main processes are as follows:

— ***Reinforcement*** heating, used for beams prestressed with bonded wires; it requires electrically insulated formwork;

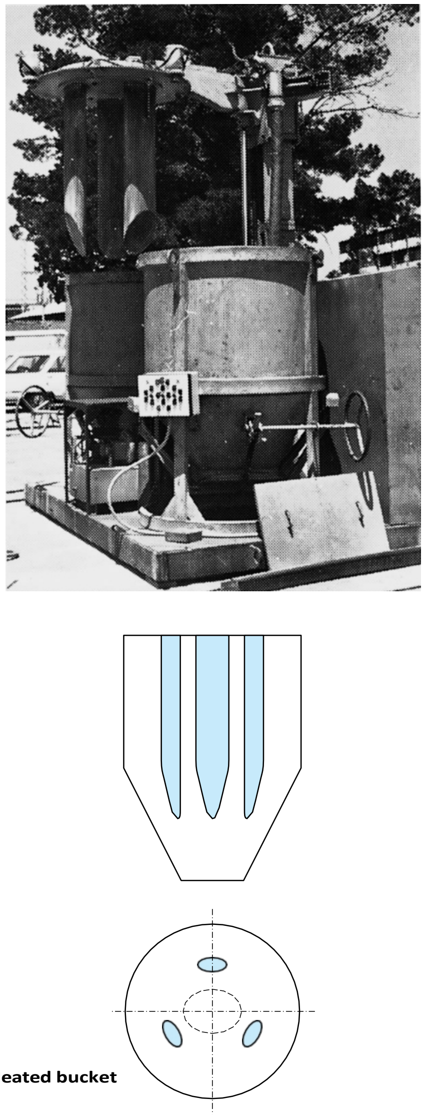


Figure 12 - Heated bucket

— heating ***by means of*** insulated ***electrical resistors,*** embedded in the concrete, at low voltage (typically 42 V); in the case of parts with variable cross-sections, this method allows good temperature distribution within the parts; it is economically attractive when the number of parts to be manufactured does not exceed a few dozen.

■ **External heating** is obtained by heating the mold, either directly or by heating the air surrounding the concrete part:

— ***direct*** heating of ***the metal mold*** is achieved by applying electric resistors or fluid-carrying pipes (water or steam) to the formwork (figure **13**);

— the air surrounding the mold is heated by placing the part(s) ***in a drying oven*** (or by placing the part(s) around (e.g. a tent or tarpaulin) and blowing in steam or hot air.

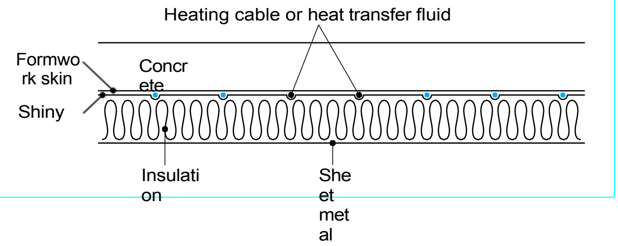


Figure 13 - Heated formwork

3.5 Infrared radiation

This process, initially used to work with concrete in cold weather, has been used in a number of industrial applications for accelerated curing of parts less than 10 cm thick.

The energy required for concrete treatment is supplied by heating panels, consisting of *infrared emitters* that can be powered by electricity or gas (propane or butane), and whose positioning ensures even temperature distribution.

This process is highly flexible in use and requires little investment, but its effectiveness decreases with the thickness of the part to be treated: for a thickness greater than 25 cm, the treatment cycle needs to be extended beyond 4 hours. Its use is also limited with low-thermal-conductivity concretes such as lightweight concretes, and it is ruled out for elements with thermal discontinuities, such as sandwich panels.

3.6 Autoclave

Autoclaving is a method used to accelerate the hardening of various materials, by combining heat and high pressure, typically 180°C and 1 MPa. With concretes, a reaction between lime and silica takes place.

This method is hardly ever used outside the manufacture of small prefabricated elements (breeze blocks, kerbstones, etc.), as it requires considerable investment (hermetically sealed processing chambers capable of withstanding pressure).

4. Maturometry

4.1 Objectives and principle of the maturometry method

A variety of equipment and methodologies are now being used on construction sites to *replace the crushing of information specimens* (specimens used to check that the required strength has been reached for form stripping or prestressing) by indirect evaluation of this strength, using Arrhenius' law, based on temperature recordings (by thermistors or thermocouples embedded in the concrete) of the structure.

Arrhenius' law, which applies remarkably well to a large number of chemical reactions whose speed depends on temperature, expresses that the speed of the reaction is related to temperature by an exponential, as follows:

$$\dot{\alpha} = \frac{d\alpha}{dt} = f(\alpha)\exp\left(-\frac{E}{RT}\right)$$

with α degree of reaction,

E activation energy of the reaction,

R perfect gas constant,

T absolute temperature.

When the temperature varies and its value $T(t)$ is known at all times, the following variable can be defined:

$$t_{eq} = \int_{0}^{t} \exp\left(\frac{k}{T_0} - \frac{k}{T(t)}\right) dt$$

variable that has the dimension of time and is equal to the real age t when the temperature is constant and equal to T_0 . It's called ***equivalent age*** or ***maturity***.

Arrhenius' law means that the degree of advancement of the reaction at time t is the same as that which would have been reached at time instant t_{eq}, if the temperature had been kept constant and equal to the *reference temperature* T_0 (reference generally taken to be 293 K, or 20°C).

Numerous authors [4] [8] [19] have shown that the compressive strength of concrete (or, more precisely, the ratio between its value at age t and its long-term value) verifies this law quite satisfactorily, even for *variable temperature* histories.

This means that, if we know the law of evolution over time, $R_c(t)$, of compressive strength at 20°C, all we need to do is record the temperature at a point to predict, at any instant, the strength acquired at that point, by replacing, in this law, the real age t by the equivalent age t_{eq}. This law of evolution at 20°C then becomes *a material characteristic*, an equation of state that fully describes the relationship between resistance, time and temperature. This is the concept of ***maturity***, a parameter that can be defined, indifferently, either by *equivalent age* or by the ratio of attained strength to final strength; in practice, we prefer the former definition (*equivalent age*), as the latter loses all precision at higher ages.

4.2 On-site methods

Various methods are used on building sites today. All are based on the calculation of an equivalent time, according to Arrhenius' law, from the data of one or more temperatures measured by thermistors or thermocouples embedded in the concrete of the structure. Two types of device can be distinguished.

4.2.1 Using an electronic box

It provides, via digital display and/or printout on a paper ribbon, measurements and calculation results: concrete age, temperature, equivalent time. The most recent versions of these devices allow the nature of the cement to be taken into account to a greater or lesser degree (via the parameter used in the calculation of thermal activation); strength is estimated on the basis of an evolution curve determined experimentally at the start of the worksite.

4.2.2 Using a portable microphone

It does the same thing as the electronic box, while offering more possibilities and flexibility in the calculations, with the ability to take into account a greater number of parameters (Campenon Bernard's Bistre® program, for example [15]); this type of approach also makes it possible to take into account acquired experience and the evolution of knowledge.

4.3 Method limitations and current developments

In fact, in its application to concrete hardening, Arrhenius' law (and the concept of maturity that goes with it) has **three important limitations,** listed here in order of increasing importance.

- The **final value of the mechanical resistance depends on the temperature**. In the case of heat-treated concrete, for example, this value can be 10 to 15% lower than that of the same untreated concrete, and vice versa, according to the known formula : "... *winter concrete, iron concrete*". Gautier [19], in particular, had clearly shown that the law only applies to the ratio of the acquired resistance divided by the final resistance, and cannot, on its own, predict the resistance if the long-term resistance is not known; part of the error induced by not taking into account, in the calculation, the effect on the final value (because this, in practice, is never measured, nor even estimated), can be reduced by calibration (the determination of the coefficient k which enters into the formula can, voluntarily or not, integrate this effect); but this nevertheless remains a *masked* error.

- **Arrhenius' law**, which is widely verified for many simple chemical reactions, **does not** a priori **apply to a cortège of chemical reactions**, especially one as complex as that occurring during the dormant period and during setting; its application, for example, to the calculation of temperatures and thermal stresses in massive structures (§ 2 and [20]) sometimes reveals shifts on the time scale, especially in the case of heavily admixed concretes (in practice, these shifts have no impact on thermal stresses or cracking risks); the reason for this is undoubtedly that the various reactions that occur during setting each have their own activation energy (the coefficient k that enters into the exponential of the law), which leads to an error on the very first phases of the process, whereas, very soon after setting, there is only one main chemical reaction governing the evolution of mechanical resistances; the evolution law, which consists of integrating temperature-corrected time intervals, only retains an error shift on the initial time.

- **Temperature** also **affects the activity of admixtures**, especially superplasticizers, which have a *retarding* effect, particularly in HP concretes (increasingly used), and this can amplify prediction errors: it is not uncommon, on HP concrete sites, to see setting times of 10 to 15 h, and sometimes more; this effect of initial temperature is generally taken into account by the model, but with the same activation coefficient k as that governing hardening, but which has no reason to apply to the retarding effect of the admixture! The error thus induced on the equivalent time can then be, at least in some cases, much greater.

4.4 Calibration

Given the current state of knowledge, maturometric methods cannot yet be considered totally validated or qualified. Any proposed application therefore requires, at least for future projects, the implementation of a qualification and calibration procedure.

A procedure of this kind has been defined by the Ponts et chaussées network, based on the elements outlined above. It is based on the laboratory production of 21 16 × 32 cm specimens (ten of which are fitted with a thermocouple at the core) for the following tests.

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4.4.1 Isothermal testing at 20 °C: calibration and reference

Twelve of these 21 test tubes are made up in the late afternoon at 20°C and protected from drying out immediately after manufacture (in plastic bags or with self-adhesive aluminum foil), then stored at 20°C. Temperature is recorded continuously for 72 h, at the center of four test tubes (at least 10 measurements per hour for the first 18 hours, then at least one per hour).

Three test tubes are crushed at each of the following ages: 18, 24, 36 and 48 h.

4.4.2 Isothermal test for *k* coefficient calibration

Three test specimens are made at 20°C and protected from drying out as soon as they have been manufactured (by the same means as the previous ones), then placed in an oven or in *heated hoods* and maintained until 19 h at a temperature of between 30 and 35°C.

The temperature of at least two test pieces is continuously recorded.

These three test tubes were crushed at 8pm.

The ratio between this age and the age (obtained by interpolation on the resistance curve at 20°C) at which the same resis- tance is reached at 20°C is used to calculate a value for the activation coefficient *k*.

4.4.3 Non-isothermal validation tests

Six test specimens are manufactured using a method that achieves a temperature as early as possible in the manufacturing process.

The temperature range for three of them is between 5 and 10°C, and for the other three, between 12 and 15°C (ice water, liquid nitrogen, etc.).

They are then protected from desiccation (by the same means as the previous ones) and each group of three is placed, until 21 h, in a closed box made of 10 cm thick expanded polystyrene sheets. The temperature of two specimens from each group was recorded continuously.

These six test tubes were crushed at 10 pm.

The difference between the measured resistances and the resistances predicted by the calculation provides an assessment of the method.

In practice, however, determination of the *k* constant requires at least one test on a specimen kept at a constant temperature different from the reference, in addition to the reference compression tests. This test is sometimes replaced by the use of an activation energy value which has already been used or which, for certain cements, can be found in the literature (notably in [7]).

4.5 Prospects and avenues for improvement

The various avenues being explored are as follows:

— ***detection of the start of setting*** using a threshold based on the difference between temperatures inside and outside the concrete, and start of calculation of the equivalent time at this point;

— ***addition of a term or coefficient*** to capture the effect of admixtures, e.g. by using a different value for the dormant period for the activation term (this approach involves carrying out tests on non-admixed control concrete in parallel with the tests described above);

— ***use of flux sensors***, glued to the surface of the concrete, either to detect and evaluate the heat release itself, or, in addition to the probes, to detect the start of setting.

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