Dielectric measurements to characterize the microstructural changes of young concrete

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The dielectric properties of concrete can be used to characterize the development of the microstructure of the cement paste. During hydration the microstructure changes and so does the amount of water in the pore structure. The different dielectric properties of the pore water and the solid phases can be measured with microwave frequencies.
In this paper the dielectric properties of cement and concrete during hydration are discussed.
The interpretation of the measurements to microstructure-related properties is shown. The connectivity of the pore system has been related to the conductivity of the cement paste. The conductivity versus strength relationship has been used for a practical application. This application concerns the strength development in young concrete in a non-destructive way. This method of monitoring the strength development has proved to be successful. The practical application is now tested on site.

Keywords: **young concrete, dielectric properties, hydration, pore water, strength development**

1 Introduction

During construction the strength development of young concrete can be an important factor in the planning. When the strength development is exactly known the formwork can be removed and prestress can be applied at the right time. This can result in a shorter construction time, which reduces the costs.
For determining the strength development several methods can be used. For practical reasons these methods should preferably be non-destructive, robust, easy to use and reliable.
The concrete group of the TU-Delft, in a joint venture with the IMAG-DLO and OFFIS, has developed a new non-destructive monitoring device to determine the strength development of young concrete. This monitoring system is based on the changes in the microstructure of the cement paste during hydration. These changes determine the changes in the dielectric properties and the strength development.

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2 Theoretical background

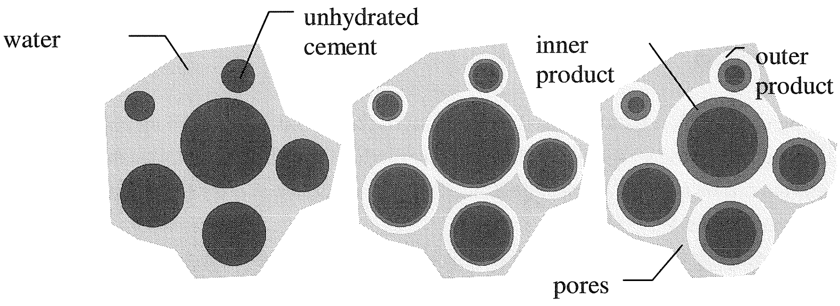
2.1 *Hydration of cement*

Sand and gravel in concrete are "glued" together with cement and water: the cement paste.
When cement reacts with water the cement paste changes into a solid matrix. In Figure 1 growing spheres are used to simulate the formation of a microstructure. The cement "spheres" grow in outward direction. When the cement grains start to interconnect the strength development starts and the pores gradually become blocked.
This hydration process is affected by external and internal parameters. The external parameters are, for example, the geometry of the structure and the weather conditions. The internal parameters are, for example, the type of cement that is used and the water/cement ratio.

Defining the degree of hydration by the amount of cement that has hydrated (Equation 1) is considered to be the most convenient starting point for considerations concerning the evolution of the microstructure [1].

$$\text{degree of hydration} = \frac{\text{amount of cement that has been hydrated}}{\text{initial amount of cement}} \tag{1}$$

For the determination of the degree of hydration several methods are available. In this research program the heat development is used. With this method the heat development of a case of complete hydration is compared with the actually produced heat [2]. The degree of hydration has been simulated with the program HYMOSTRUC [3]. With the calculated degree of hydration the dielectric measurements have been linked to the development of the microstructure.



Cement paste just after mixing

The hydration has started and hydration products are formed

The particles start to interconnect and the strength development starts

Fig. 1. Formation of the microstructure simulated by growing spheres.

2.2 *Dielectric properties*

The dielectric properties of a material determine the response of a material when placed in an electrical field. Dielectric properties (ε_r) are always presented relative to the dielectric constant of the vacuum (ε_0) (Equation 2). The dielectric properties of concrete are determined by the electrical permittivity (ε'), also known as the dielectric constant and the electrical conductivity (σ) (Equation 3 and 4). In figure 2 the permittivity is presented as a capacitor. The capacitor is placed parallel to the resistor, the latter representing the conductivity.

$$\begin{aligned} \varepsilon_r &= \frac{\varepsilon_m}{\varepsilon_0} \\ \varepsilon_0 &= 8.854\ 10^{-12}\mathrm{Fm}^{-1} \end{aligned} \tag{2}$$

$$\begin{aligned} \varepsilon_r &= \varepsilon' - j\left(\varepsilon'' + \frac{\sigma_{\text{ionic}}}{2\pi\varepsilon_0 f}\right) \\ j^2 &= -1 \end{aligned} \tag{3}$$

$$\sigma = 2\pi f\varepsilon''\varepsilon_0 + \sigma_{\text{ionic}} \tag{4}$$

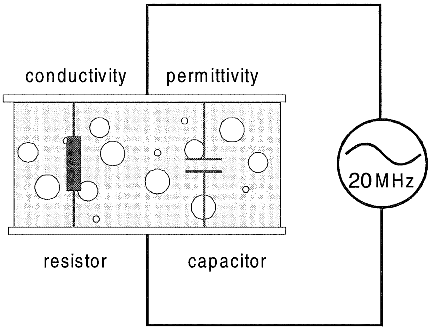


Fig. 2. Dielectric properties of concrete represented as resistor and capacitor.

The permittivity represents the electrical polarization. This is the amount of electrical energy that can be stored in a material. The electrical polarization can have several origins, occurring at different frequencies (f) of the electrical field (figure 3):

- Electronic polarization, which occurs due to the shifting of electrons in an atom.
- Atomic polarization, when atoms are combined the electrons will shift over several atoms to result in a polarization.
- Dipole polarization occurs at molecules, which have an a-symmetric structure like water. Due to the a-symmetric nature the molecule has an electrical charge. This charge can be polarized.
- Ionic polarization will occur when ions in water are transported through a material due to their electrical charge.

The conductivity has two components. Firstly there are the dielectric losses (ε'') of a material when placed in an alternating electrical field. These losses depend on the used frequency. Secondly the ionic conductivity (σ_{ionic}) of a material, which is a measure of the resistance of a material against conducting electricity through a material.

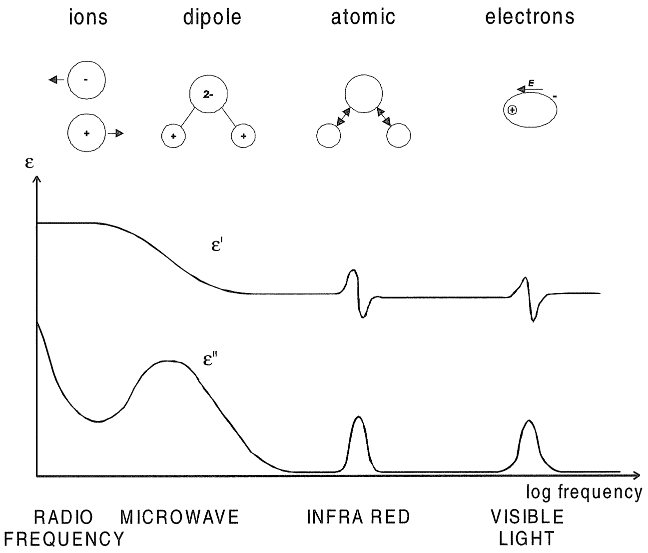


Fig. 3. Dielectric properties of a material at different frequencies.

2.3 *Dielectric properties of young concrete*

The dielectric properties of concrete can provide valuable information about the performance of the structure. Radar for example can be used to locate reinforcement in the structure. This method is based on the changes in dielectric properties of the different phases present in concrete. The different magnetic permeability of the concrete and the steel reinforcement makes it possible to detect the place of the reinforcement in the structure [4].

Young concrete has a lot of water in it, which will be consumed during hydration. The difference of the dielectric properties of the water and the solid phases makes it possible to detect these phases. The dielectric properties of water are based on the dipole structure of the molecule. Figure 3 shows already that the dipole structure of water can be detected with an alternating electrical field with microwave frequencies (100 kHz – 1000 MHz).

The hydration process in concrete is the reaction between water and cement. The water and cement phases in the concrete are the ones changing in volume during hydration, while the amount of aggregate does not change. This does not mean that the aggregate has no effect on the dielectric properties.

The water content and the way this water is structured in the cement matrix determine the dielectric properties of cement paste. Free water in the capillary pores has different dielectric properties than the physical or chemical bound water in the reaction products.

3 Experiments

3.1 *Research set-up*

The dielectric properties of young concrete have been examined with two measurement methods:

- The first method, the HP impedance analyzer, had as aim to examine the dielectric nature of cement and concrete at high frequencies (1–1000 MHz).
- With the second method, the 20 MHz dielectric sensor, the influence of material parameters like water / cement ratio and cement types on the relationship between dielectric properties and strength development has been examined at a frequency of 20 MHz.

3.2 *1–1000 MHz set-up*

To measure the dielectric properties of cement and concrete at high frequencies a HP- impedance analyzer has been used. This analyzer generates an electrical field in a frequency range of 1 MHz till 1000 MHz and registers the electrical capacitance and conductivity of the material. The test set-up is presented in Figure 4. The probe with which the electrical field was introduced into the cement and concrete is shown in Figure 5.

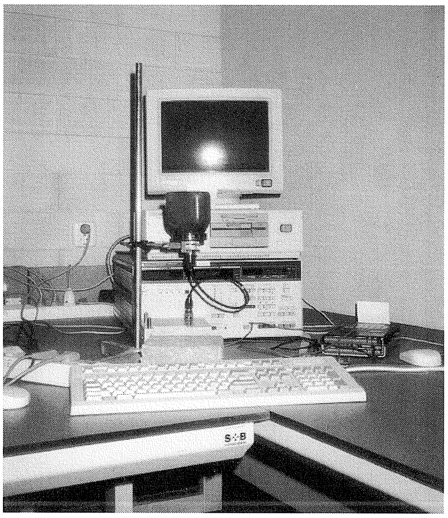


Fig. 4. Test setup for 1 – 1000 MHz measurements.

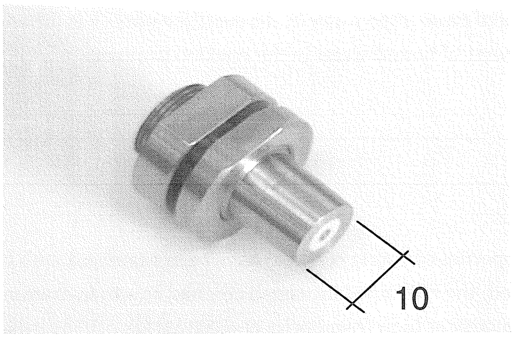


Fig. 5. Probe used for the 1–1000 MHz measurements.

3.3 *20 MHz setup*

The parameter test has been conducted with the 20 MHz dielectric sensor, which has been developed by the IMAG-DLO [5]. This sensor is small and easy to operate. Therefore it can be used for practical applications which will be mentioned later. The 20 MHz sensor does not only measure the dielectric properties. It also has the ability to measure the temperature. The basis of the 20 MHz sensor is a microchip of 4 by 4 mm developed by IMAG-DLO (Figure 6). The set of electrodes with which the electrical field was introduced into the concrete is shown in Figure 7.

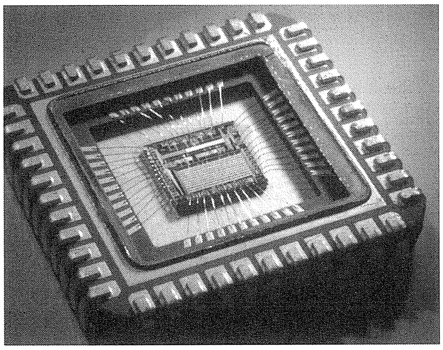


Fig. 6. The microchip of the 20 MHz sensor.

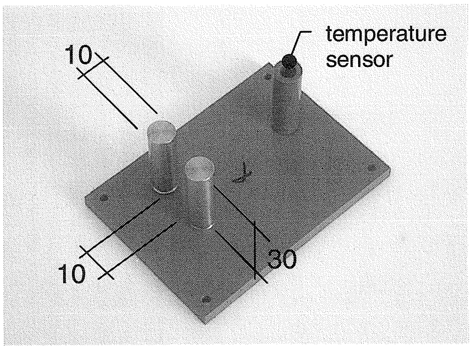


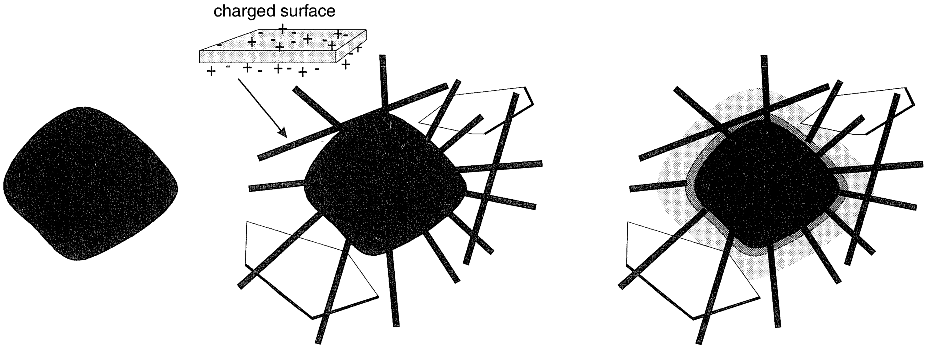
Fig. 7 Electrodes for 20 MHz sensor.

4 Test results

4.1 *Permittivity of cement*

The permittivity of cement is not only dependent on the amount of water in the paste. If that was the case the permittivity should only decrease with increasing degree of hydration. In the frequency range of 100 kHz – 100 MHz an increase of permittivity is often seen before the permittivity starts to decrease.

The increase of permittivity can be contributed to the unstable character of the system that is build up in the early stage of hydration Figure 8. This unstable microstructure contains a considerable amount of physically bound water with charged interfaces, which increases the permittivity but does not conduct electricity [6]. At frequencies lower than 100 KHz this increase in permittivity cannot be located any more because the electrode-interface effect dominates the measurements.



Just after mixing

Unstable reaction products with numerous charged surfaces

Stable products are formed, which decreases the amount of charged surfaces

Fig. 8. Subsequent stages in the formation of the microstructure.

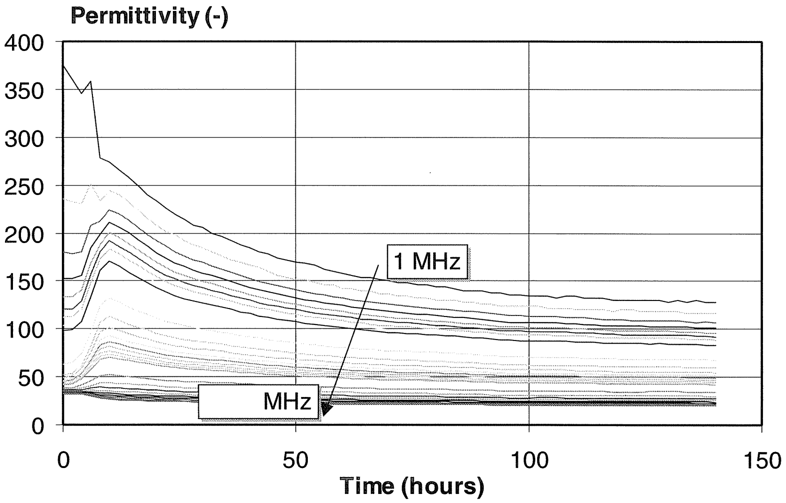


Fig. 9. Permittivity of cement paste in the first 140 hours after mixing.

4.2 *Conductivity of cement*

The conductivity is determined mainly by the amount of interconnected capillary pores in the cement matrix. The main conducting phase in cement paste is the capillary water. The reaction products that block the pores cause a reduction of the conductivity. (Figure 10). The decrease in conductivity is therefore not linearly related with the volume of capillary water. In other words, the decrease of connectivity results in a sharp decrease in the conductivity. The connectivity can thus be determined from the dielectric measurements.

In a frequency range of 1–1000 MHz the conductivity decreases during hydration. Just after mixing the differences between the conductivity at 1 MHz and 1000 MHz is rather small. At higher frequencies the conductivity increases due to the dielectric losses.

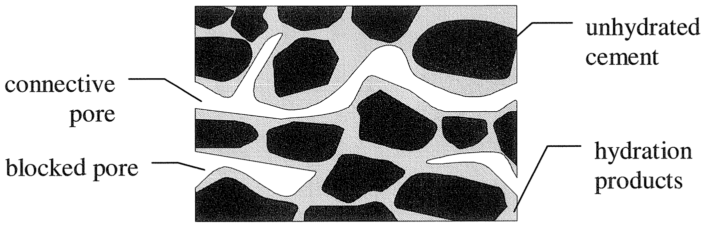


Fig. 10. Schematic presentation of the connectivity of capillary pores.

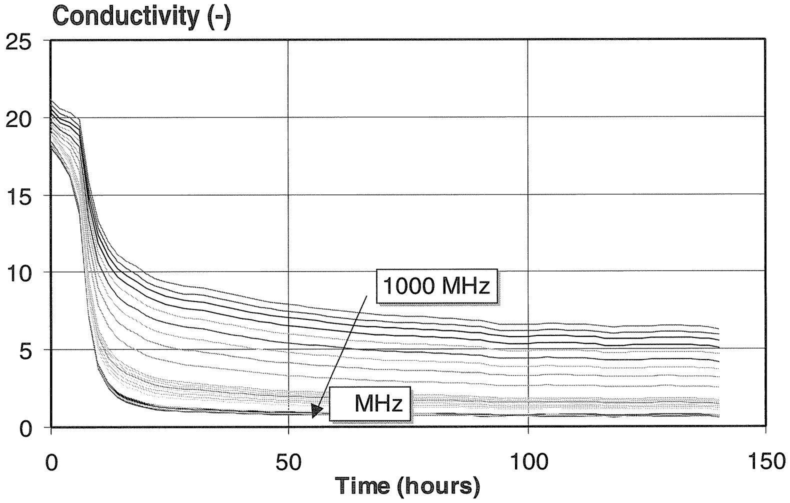


Fig. 11. Conductivity of cement paste in the first 168 hours after mixing.

4.3 *Connectivity*

The connectivity of the pores in the concrete is seen as an important factor for the durability of concrete. Transport mechanisms of aggressive materials like chlorides strongly depend on the pore system. The more pores are connected with each other the more chloride can penetrate into the concrete.

To relate the connectivity to the conductivity measurements the following model is used. It is assumed that open pores conduct an electrical current and closed pores do not. Directly after mixing the pores are all connected and the system is considered to be parallel. As soon as all the pores are blocked we have the other extreme system; the serial system. The real system will be a mixture of the parallel and serial system. The deviation of the measured conductivity from the parallel and serial system is considered as an indicator for the connectivity (Equation 8).

To calculate the connectivity the following procedure is followed:

- From temperature measurements the degree of hydration (α) is calculated.
- The Volume of the capillary water (V_{cap}) can be calculated with Equation 5 [7].

$$V_{cap} = \frac{\mathrm{wcr} - 0.40\alpha}{\frac{\rho_w}{\rho_c} + \mathrm{wcr}} \tag{5}$$

α	degree of hydration		(-)
wcr	water/cement ratio	0.5-0.6	(-)
V_{cap}	Volume capillary water		(m^3/m^3)
ρ_c	density cement	3150	(kg/m^3)
ρ_w	density water	1000	(kg/m^3)

- The conductivity of capillary water is calculated from the starting point of hydration where all pores are connected and the cement paste can be seen as a parallel system. The result was a conductivity for the pore water (including dielectric losses) of 27 mS/cm. For the model the conductivity of the pore water is assumed to be constant during hydration.
- The matrix is normally considered as a material with a low conductivity. From measurements however, a rather high conductivity (0.4 till 1.0 mS/cm) was found even for old cement matrixes (several months). This is partly due to the high dielectric losses at 20 MHz for cement and the water in the gel pores. In the model a value of 0.5 mS/cm was taken for the conductivity of the cement matrix.
- With the conductivity of the capillary pore water and the cement matrix the expected conductivity of a parallel and a serial system can be calculated with equation 6 and 7.

Serial system:

$$\sigma_{tot,\,s} = \frac{\frac{\sigma_{pw}}{V_{cap}} \frac{\sigma_{matrix}}{1 - V_{cap}}}{\frac{\sigma_{pw}}{V_{cap}} + \frac{\sigma_{matrix}}{1 - V_{cap}}} \tag{6}$$

Parallel system:

$$\sigma_{tot,\,p} = V_{cap}\sigma_{pw} + (1 - V_{cap})\sigma_{matrix} \tag{7}$$

σ_{pw}	conductivity pore water	27	(mS/cm)
σ_{matrix}	conductivity cement matrix	0.5	(mS/cm)
$\sigma_{tot,s}$	conductivity serial system		(mS/cm)
$\sigma_{tot,p}$	conductivity parallel system		(mS/cm)

- The difference of the measured conductivity and the calculated conductivity are used to calculate the connectivity according to equation 8.

Connectivity indicator:
$$\Theta = \frac{\sigma_m - \sigma_{tot,p}}{\sigma_{tot,p} - \sigma_{tot,s}} \qquad (8)$$

σ	measured conductivity	(mS/cm)
Θ	connectivity	(–)

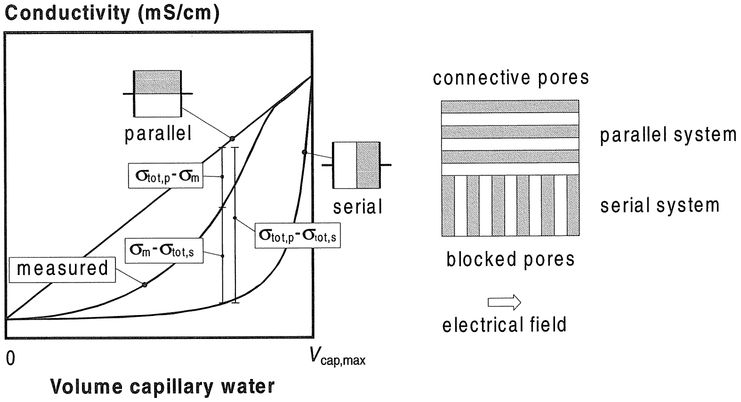


Fig. 12. The measured conductivity compared with the parallel and serial models.

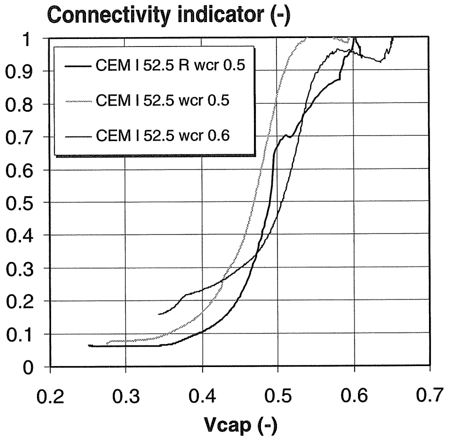


Fig. 13. Connectivity of pores in cement determined with the measured conductivity at 20 MHz.

4.4 *Concrete*

The difference between cement and concrete is the aggregate. The aggregate has normally a volume of about 60-80 % of the concrete and should therefore have a dominant effect on the dielectric properties. In this research a filling grade of 75 % normal aggregate was used. This resulted in a reduction of 15–40 % in the permittivity and conductivity.

For a wide range of concrete mixtures the dielectric properties at 20 MHz have been examined. These different concrete types were concrete made with ordinary Portland cement, concrete made with blast furnace slag cement, steel fiber reinforced concrete, high strength concrete and light-weight concrete. The range of water/cement ratios used for these concrete mixtures differs from 0.33 to 0.6. Most of the tests were done to get a relation between the dielectric properties and the strength development. This relationship can be useful for the practical application for example a non-destructive strength sensor.

5 Dielectric measurements for monitoring the strength development

5.1 *Laboratory studies*

A special practical device is made to determine the strength development of young concrete in a non-destructive way. The strength development can be important during construction for decisions concerning removal of the formwork and application of prestress. This dielectric strength sensor will be developed further for practical applications on site by OFFIS and will be called the CONSENSOR.

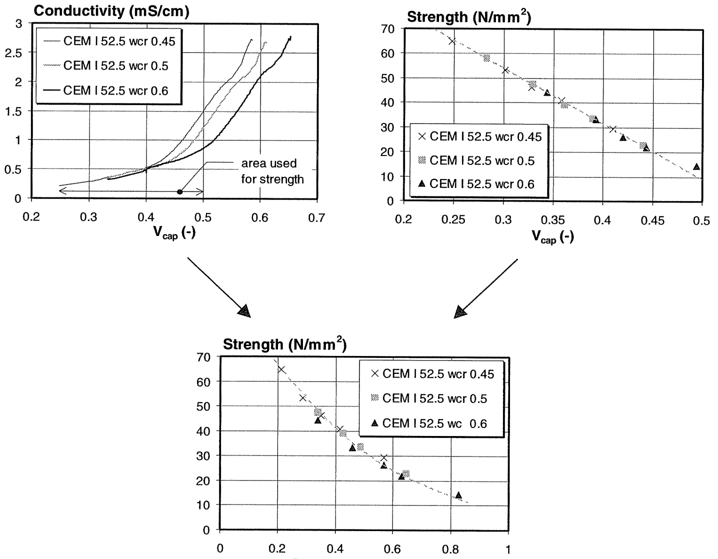


Fig. 14. The relationship between conductivity and capillary porosity and the relationship capillary porosity and strength resulting in the relationship between conductivity and strength.

The relation between the conductivity and strength has its basis in the relationship of these properties with the microstructure and the free water content in the capillary pores. For a wide range of concrete mixtures with Portland cement this relationship between capillary water, conductivity and strength has been examined (Figure 14).
In paragraph 4.2 it is mentioned that the water in the capillary pores determines the conductivity of concrete. The capillary porosity also determines the strength of the cement paste and therefore the strength of the concrete [8]. For the practical application the direct relationship between conductivity and strength will be used.

5.2 *Implementation in the practice*

The prototype of the sensor (Figure 15) has been tested in a concrete laboratory of the van der Velden concrete plant. The relationships for concrete with Blast Furnace Slag cement that were found in this plant, turned out to be consistent with those that were found in the laboratory at the TU-Delft (Figure 16). The results of these tests have been used to develop the consensor. At this moment two projects have been started to test the practical application on two structures of public works which are under construction.

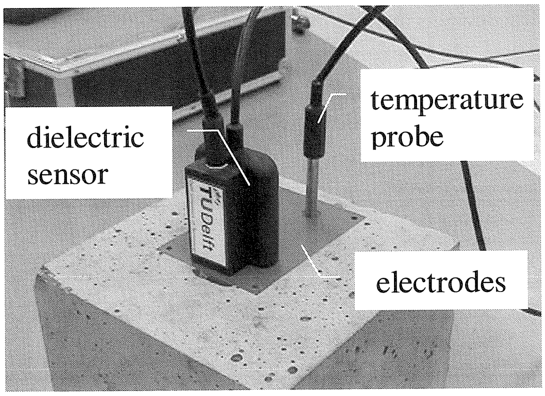


Fig. 15. Prototype of the dielectric measurement system.

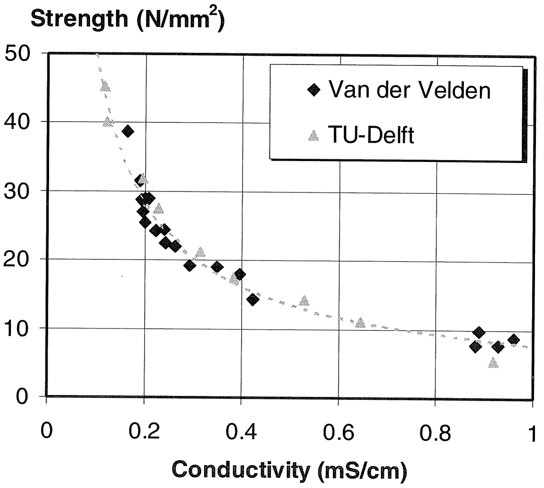


Fig. 16. Relationship between conductivity and strength for mixtures with blast furnace slag cement.

6 Further research

The dielectric properties have shown to be a good indicator for the changes of the microstructure in hydrating cement paste. The strength development, which is a direct result of the changes in the microstructure, can very well be monitored with the help of the conductivity.
To obtain more information from the dielectric measurements the TU-Delft develops a computer model, which calculates the evolution of the dielectric properties of hydrating cement. The model is based on a hydration model of growing spheres and an electrical circuit model.
The hydration model is based on the output of the program called HYMOSTRUC. This program gives as output the growth of spherical cement grains due to hydration. The output is used to generate a two dimensional picture of the microstructure of cement. This picture has been placed in a grid.
In this grid each pixel represents a phase of the hydrating cement, with each their specific dielectric properties. These dielectric properties are translated to capacitors and resistors. By connecting these capacitors and resistors with a system, which is proposed by Reinhardt [9], the overall dielectric properties of cement can be calculated (Figure 17).
With the results of these microstructural models more insight into the hydration process of cement can be generated and the interpretation of dielectric measurements can be improved.

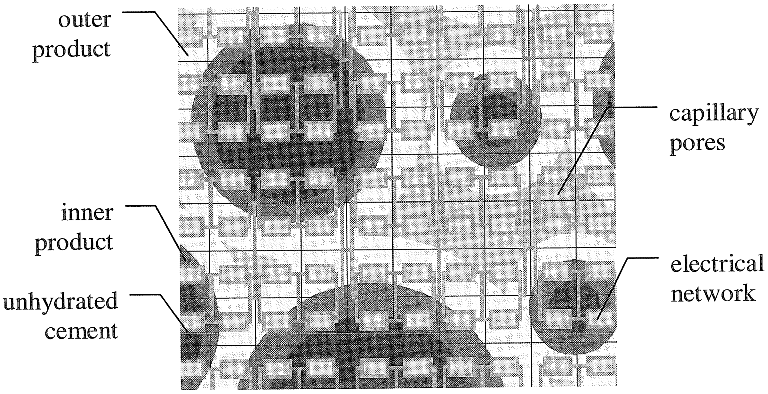


Fig. 17. Electrical network used as model to calculate the dielectric properties of a microstructure.

7 Conclusions

The dielectric properties can very well be used to characterize the changes in the microstructure during hydration. The dielectric properties can be linked to the microstructure with the degree of hydration. The degree of hydration, on its turn, is directly related to properties like strength.
The CONSENSOR is a practical application of the use of dielectric properties of young concrete.
The monitoring system is based on the relationship between conductivity and strength. It generates information that can be used for decision-making on-site concerning the removal of formwork and the application of prestress. The system is now tested in practice on structures, which are under construction.
To obtain more information from the dielectric measurements microstructural models can be used.
A microstructural model, which relates the dielectric properties to the hydration of cement, is now being developed.

Acknowledgements

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