Experimentální Analýza Napětí 2010
Experimental Stress Analysis

Monitoring of cement-based material solidification, focusing on electrical properties

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Abstract: Nondestructive diagnostics of concrete materials and non-cement-based materials is the subject of current research for developed methods of testing building materials. Behavior of concrete components in the early stages of solidification also gives information about future product qualities of concrete. Common methods include monitoring the volume change components, thermal characteristics and approaches to the study of electrical properties. Our comprehensive measurements carried out continuously and at 28 days. The values of electrical quantities of material during solidification and possible association with stress in the material are correlated. Electrical parameters are evaluated: in particular, capacity, resistance and loss factor at frequency of alternating electric field. During hardening of the concrete panel is an internal tension, which is trying to describe by mentioned electric quantities.

Keywords: Impedance Spectroscopy, Dielectric Losses, Loss Factor, Conductivity Losses, Polarization Losses

1. Introduction

The quality of concrete depends on the setting and hardening. The pursuit of this plot is possible by using some NDT methodes. The impedance spectroscopy, which belongs to the methods of nondestructive testing of concrete samples, were characterized and monitored changes in the spectrum in its hydration. Differences were observed in the spectras of tan δ (f) and C (t), respectively. R (T) for samples and its quality was described by the dominant type of loss in material.

Change of electrical parameters is caused by a chemical process and associated aspects such as stress in the material or creating porous structures. The presence of the mixing water for making concrete affects the quality of the material in the form of the resultant tension and compressive strength, affects porosity.

Just pores and their formation provide a source of information about many properties of the substance [1]. Pores informed of the structure deformation, the

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degree of mechanical stress on the technological implications of the porous substance (strength, water resistivity, frost resistance, shrinkage, etc.).

Porosity of cement stone is composed by both pores resulting in fresh concrete (and which remained in the hardened concrete), and secondly of the pores caused by cement hydration. Pores can be divided into macropores (diameter d> 1 μm, technological P_T and P_P as aeration) and the micropores with a diameter up to 1 μm (some authors suggest up to 10 μm), which are further subdivided into gel-P_G, P_H as hydration and capillary P_K.

The total porosity of cement stone P_{CK} is the sum of [1]:

$$P_{CK} = (P_T + P_P) + (P_G + P_H + P_K) \tag{1}$$

2. Measured material and electrodes description

Electrodes (Fig. 1) were made of two types. The first of brass sheet thickness of 1 mm flat dimensions 25x40 mm. Second are of polished steel with a diameter of 6 mm and length of 75 mm (inset length 65 mm). Recessed electrodes (Fig. 2) are mated with the help of teflon plate, which guarantees electrode distance 3 cm and their parallelism of its longitudinal axis.

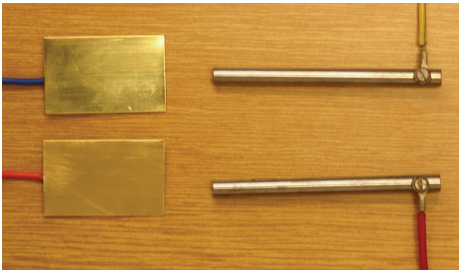

Fig. 1. Type of electrodes sunk into concrete.

Table 1. Recipe of concrete and its mechanical properties

Recipe for concrete	1 m^3	**Mechanical properties**	
w-c ratio	0,33	Workability	F4
CEM I 42,5 R Mokrá	420	f_{c1} [MPa]	48,4
Metakaolin Mefisto K05	35	E_1 [GPa]	29,0
Water	150	f_{c2} [MPa]	60,5
Superplasticizer	7,5	$E_{2,rh}$=95 [GPa]	-
Sand 0/4 Kinsky	625	$E_{2,rh}$=60 [GPa]	30,4
Crushed 4/8 Litice	245	f_{c28} [MPa]	105,1
Crushed 8/16 Litice	975	$E_{28,rh}$=95 [GPa]	40,5
		$E_{28,rh}$=60 [GPa]	37,3

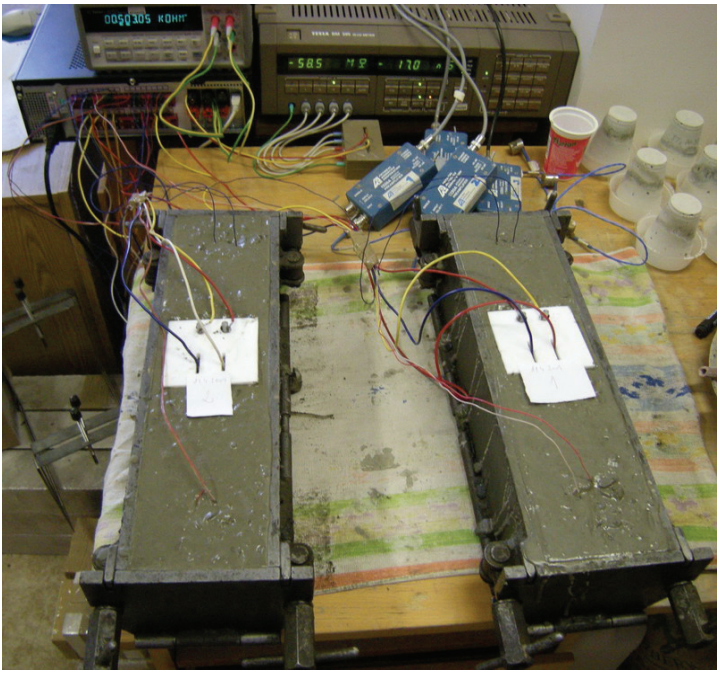

Fig. 2. Display of work place (concrete samples, devices and embedded electrodes). Sample No. 1 is labeled on the right, left sample is labeled number 2 during the experiment wrapped in shrink foil.

Concrete was mixed in a laboratory blender at number 35 l. To measure of compressive strength f_c has been constructed on a cube edge 150 mm and method for measuring the impedance spectroscopy and the measurement of static elastic modulus E prisms with dimensions 100x100x400 mm. The values of tensile strength and modulus were measured at age 24-26 hours, next at age of 2 days and 28 days, both on samples stored in wet storage (r.h. 95%) and in laboratory air (r.h. 60%).

The electrodes were inserted into the temperature of the mixture for about 15 minutes after mixing, and the measurement started after 1.5 hours.

For this mixture with a w/c ratio 0.33 shows that the static modulus of elasticity and compressive strength with increasing age. Its role is apparently played by water in the structure and formation of the aforementioned porous structure.

3. Results

Implemented continuous measurements in the range up to 1080 hours provide a number of time-dependent electrical parameters. The analysis presented below were choice to discus.

The graph in Figure 3 compares the electrical capacity of the sample in the electrode at frequency 1 kHz of electric field. Capacity is slightly different for

surface electrodes and flux cored electrodes. It's the shape of electrodes, their surface, electrodes distance and orientation effects due to metal electrodes in the preparation of the experimental sample. It is not our intention to compare the absolute value of the electric capacity between the different electrodes.

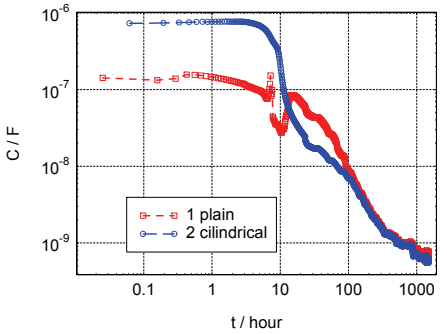

Fig. 3. El. capacity of sample for two different type of electrodes, f=1kHz.

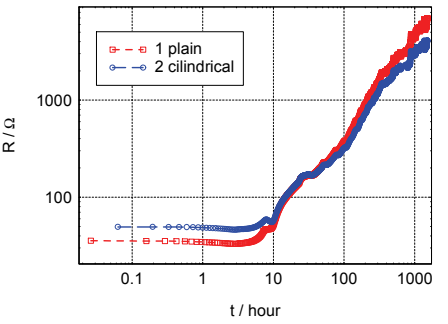

Fig. 4. El. rezistance of sample for two different type of electrodes, f=1kHz.

In Figure 3 we observe in the first hour the minimum increase of capacity at both type of electrodes, the first hour of the start of measurement, corresponding to approximately 1.5 hours of blending, resulting in a decrease of one order of capacity. Shortly before 10th hour we observe for the flat electrode capacity increases of curve, which may be caused by switching between measuring ranges used RLCG bridge, but also the possible manipulation of the sample when applying the method of acoustic emission sensors. However, substantial growth in capacity for flat electrodes after 10th hour, the values of the capacity for cylindrical electrodes increase not, only with the falling value of the capacity becomes calmer. Mixing water still fills the space between particles of the mixture, the sample is still plastic and tension in the sample is minimal, this is compensated for transporting water and change the position of certain particles. Water is increasingly linked to the binder particles. Porosity increases.

After 15th hour sample capacity of the electrode area is falling again. After 23.5th hour occurs in both curves sharper declines, however, the curve will follow the initial downward trend in the 35th hour. Gauging the remaining water is now more reminiscent of an electrolyte with a dielectric constant higher than the permittivity of the air continues to disappear from the binder and macropores. Permittivity of the material decreases and electrical capacity too decreases. It can be assumed that the sample increases dramatically tension specimen ceases to be plastic, increasing the frequency of microcracks.

After approximately 100th hours of measurements have comparable values of capacitance values and the rest of the concrete curing time is significantly different values. Shape of the curves in the logarithmic on both axes in time from 10th hour is not a straight line across the interval, can be used to approximate logC = k*logT + q,

where k and q are factors, because of electrical capacity doesn't change exponentially in progress throughout the remainder of the interval.

The logarithmic graph of the electrical resistance dependence on the logarithm of time for description log R (log t), we compare the electrical resistance at frequency 1 kHz of electric field excitation of both types of used electrodes. Again, not to compare the measured values, only changes in the spectrum and trends. Again we see the shape of the spectrum comparable in both types of electrodes until 8 hours. Then rise and fall for several hours at the curve of electrical resistance determined using cylindrical electrodes, while the resistance values at the second curve are not falling. Tension in the concrete panel is small, this is compensated by material plasticity. From 10^{th} hour held two curves of the same trend. Electrical resistance increases, porosity increasing too and strain in the material. After 13^{th} hour until 100^{th} hour of electrical resistance values are comparable. Values of electrical resistance measured plate electrodes are growing more rapidly than the cylindrical electrodes values. In conclusion, we observe the interval value of 6000 Ω and 3500 Ω. Looking at the whole spectrum can`t be replaced once the pattern of change of electrical resistance during hydration exactly exponential dependence, we can now say that close to an exponential curve is the resistance of concrete at the cylindrical electrode from 8^{th} hour. When searching for a trend in this curve, we come to form an virtual envelope curve in the form: logR = 0,832 *log T +0,971. after calculating: R = 9.4 * (T ^ 0.8). The most important parameter is the exponent 0.8, which seems very similar with other cement mixtures.

The graph in Figure 4 we can see from about the 5^{th} hour hydration exponential increase in electrical resistance of the sample. Assumed to be a close link with the increase in porosity that occurs presence gel pores, hydration and capillary porosity of pores. By using Fagerlund empirical formula for the degree of hydration of cement will be matched with the proportion of species in the total porosity of pores and its dependence on hydration time.

Very interesting is complex spectrum of the dissipation factor on frequency tandelta dependency for the entire period of hydration. In Figures 5 and 6 are shown spectra of cylindrical electrodes.

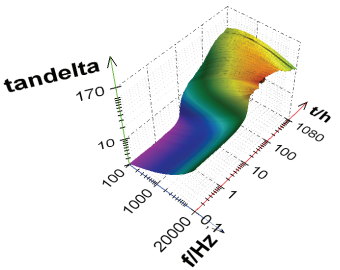

Fig. 5. Dielectric loss factor at time dependency - sample no.1, cylindrical electrodes measured, face 1.

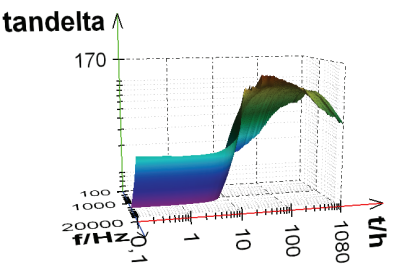

Fig. 6. Dielectric loss factor at time dependency - sample no.1, cylindrical electrodes measured, face 2.

After 10^{th} hour, the value increases, this corresponds to an increase in compressive strength, tension grew in the sample. Thence discovered the maximum surface moves with time to lower parts of the spectrum and its absolute value decreases. The presence of arc-shaped curve shows the polarization dominant losses from conductivity.

In Figures 7 and 8, we see again the spectrum dissipation factor generalized chart for measurements using planar electrodes, very similar values as the previous case. Very alarming is the presence of narrow extremes. The first is located at the first stage of solidification only at the highest frequency electric field, but only to show the influence of electrode shape on the measurement result. Much more complicated, it appears the presence of a narrow mound about 10 hours, but starts earlier at lower frequencies and ends later at higher frequencies. It corresponds with the observed anomalies in electrical capacity, depending on the time interval in the same setting, as well as to decrease the electrical conductivity in that interval.

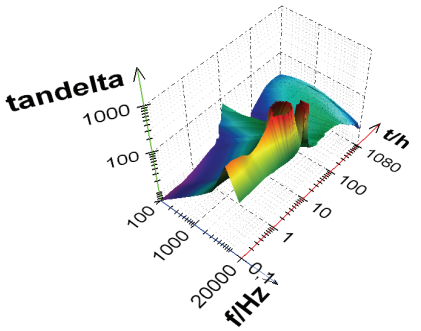

Fig. 7. Dielectric loss factor at time dependency - sample no.1, planar electrodes measured, face 1.

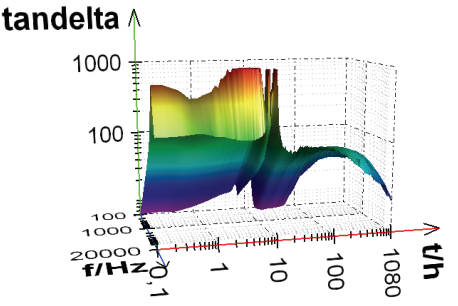

Fig. 8. Dielectric loss factor at time dependency - sample no.1, planar electrodes measured, face 2.

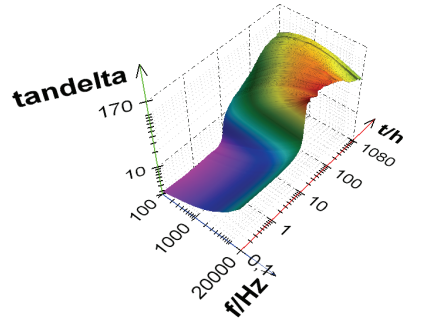

Fig. 9. Dielectric loss factor at time dependency - sample no.2, cylindrical electrodes measured, face 1.

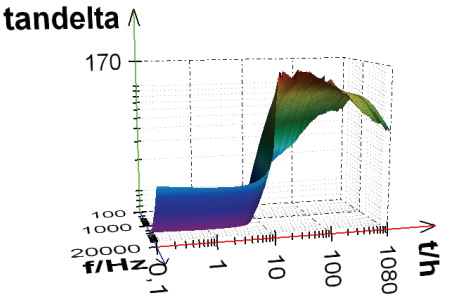

Fig. 10. Dielectric loss factor at time dependency - sample no.2, cylindrical electrodes measured, face 2.

In figures 9 and 10 there are described dielectric loss factor spectra, obtained by using cylindrical electrodes, for sample no.2 hydration, which was for all experimenting time covered by plastic foil. Spectra are again smooth continuity, the maximum value that reach is 166, which is comparable to the value of non covered sample No.1, which value is 140. For no coated sample (No. 1) observed in the first 10 hours of hydration at the highest frequencies higher dissipation factor and at the end of measurement (time t ~ 1000hours) is seen moving faster maximum curves to lower frequencies than the spectra of the coated sample (in this chart indicates narrower single column).

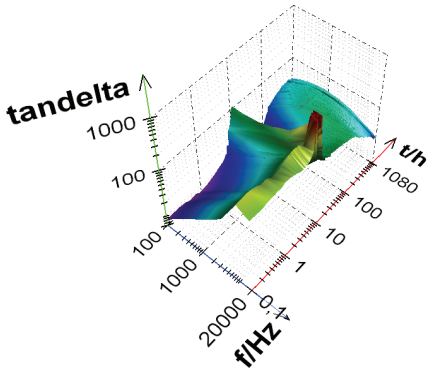

Fig. 11. Dielectric loss factor at time dependency - sample no.2, planar electrodes measured, face 1.

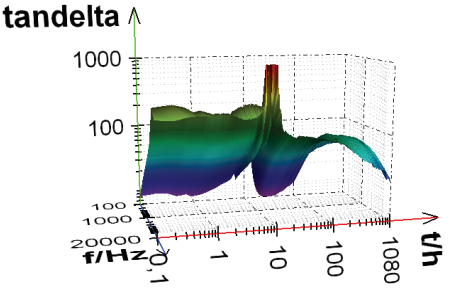

Fig. 12. Dielectric loss factor at time dependency - sample no.2, planar electrodes measured, face 2.

The coated sample is surrounded only autogenous shrinkage, while the sample is reflected noncoated plastic shrinkage and drying shrinkage. Each contraction is due, inter alia, changes in capillary pressure. Arguably, the high values of dissipation factor at no coated sample, using electrodes printed on the implementation of the measure, are a manifestation of more species and higher shrinkage stress, whereas the wrapped sample is not achieved such high values of dissipation factor and stress in the sample is lower.

4. Conclusion

During the 45 days was observed hydration and hardening of the concrete composition, using two types of electrodes. Obtained time and frequency dependences showed some differences in the spectra of electrical parameters depend on the type of electrodes. During hydration of the concrete sample to avoid in the first hours of the dramatic changes in the measured electrical parameters such changes occur at intervals of 4 to 50 hours (approximately). Change of electrical resistance and capacity of the sample indicates the strength changes in the sample to reach higher intensities varied rapidly. Taking away pan of sample had no significant effect on the course of hydration.

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