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Effect of Temperature on Structural Quality of High-strength Concrete with Silica Fume

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ABSTRACT

Experimental investigation conducted to study the thermo-mechanical properties of concrete at Temelin (Czech Republic), Mochovce (Slovakia), and Penly (France) nuclear power plants reveals structural integrity degradation between 100°C and 200°C due to both a loss of water bound in hydrated cement minerals and subsequently air void formation. Test results indicate changes in strength, average pore radius and calculated permeability coefficients for Mochovce specimens exposed to temperatures up to 400°C. It demonstrates that the permeability coefficient measured on the basis of pore sizes using mercury intrusion porosimetry is suitable technique for the evaluation of concrete quality. It confirms that strength and permeability coefficient are equivalent structural quality variables of concrete. At 400°C gel-like hydration products are decomposed, at 600°C $Ca(OH)_2$ is dehydroxylated, and $CaCO_3$ dissociation to CaO and CO_2 accompanied with the re-crystallisation of non-binding phases from hydrated cement under re-combustion are dominant processes between 600°C and 800°C. This stage of concrete is characterised by the collapse of its structural integrity, revealing residual compressive strength**.**

This paper reports high-strength concrete behaviour subjected to temperatures up to 200°C. In accordance with previous results, research studies of structure-property relation show the changes in strength, dynamic modulus of elasticity, strain-stress behaviour, and shrinkage induced deformations influenced by a hydrate phase decomposition, Volume reduction of the hydrate phase due to the loss of bound water mass is the cause of air void formation, and pore structure coarsening. The main attention is herein devoted to the evaluation of utility property decrease of high-strength concrete attacked by elevated temperatures. For this aim concrete with 28-day compressive strength of 78.5 MPa (150^3 mm, 20°C/95% R.H.) was tested.

A great amount of attention to the study of structure – property relation of concrete attacked by elevated temperatures is recommended, especially from the viewpoint of design of concrete mixture with a better ability to withstand the action of elevated temperatures.

KEY WORDS: concrete, elevated temperatures, strength, shrinkage, stress-strain curve, Poisson's ratio, volume deformations.

INTRODUCTION

Concrete is exposed to elevated temperatures in an accidental building and tunnel fire, or when it is near to furnaces and reactors [1]. Its mechanical properties such as strength, modulus of elasticity and volume deformation decrease remarkably and this results in undesirable structural quality deterioration of concrete [2, 3]. Of particular importance are loss in strength [4] and elastic modulus [5], cracking and spalling, ductility, and loss of bond with any steel reinforcement [6, 7]. The related studies show that hardened cement paste plays a key role in this deterioration process. Loss in structural quality of concrete, especially the strength and fracture generally exhibits a complex dependency on the developed phase composition and pore structure of hardened cement paste [8]. High temperatures induce a loss in strength and elastic modulus (dynamic and Young's) [9] and increase both the elastic deformability and creep by altering the physico-chemical composition of the cement paste [4, 10]. Factors affecting the shape of stress-strain curve are the type of binder and aggregate, the type of admixtures and aggregate, the aggregate – cement ratio and storage conditions [8, 11]. It was found that the loss in structural quality of concrete due to a rise of temperature is influenced by its degradation through changes induced in basic processes of cement hydration and hardening of the binding system in the cement paste of concrete [12, 14]. Cement pastes cured at high temperatures have more heterogeneous microstructure and coarser pore structure then those exposed only to ambient temperature [15]. The higher the temperature, the coarser the pore structure and the higher the permeability coefficient of concrete becomes. These changes, however, appear more expressly when the temperature is over 400°C. Permeability is sometimes even regarded as a direct measure of durability of concrete structure quality [16].

This paper presents experimentally observed behaviour of high-strength concrete with silica fume intended for the use in nuclear power plants subjected to temperatures up to 200°C, followed by the recovery period at 20°C and 60% R. H. – air cure simulating normal curing conditions after temperature elevation. The aim of our investigation was to verify behaviour of concrete and the cement paste not only at temperature attack but also after a temperature elevation when cementitious material is returned back to normal exposure at 20 °C and kept further in such conditions. Concrete elements attacked by a high temperature are left to serve in the structures for a long time. It is of great importance to study structure – property relations in such material in a more detail for better prediction of their residual serviceability.

EXPERIMENTAL DETAILS

Materials

Ordinary Portland cement (CEM I 42.5, Holcim, a. s., Rohožník) was chosen for the investigation. The cement was used in combination with silica fume (Sioxid < 98 % SiO_2, OFZ a. s., Istebné), superplasticizer Melment (Stachema Ltd., Bratislava) and siliceous river (Danube) aggregate. The composition of the fresh concrete mixture and its properties are listed in Table 1.

Casting

Cubes (100^3 mm, 150^3 mm), prisms (100x100x400 mm) and cylinders (diameter of 100 mm and height of 400 mm) were made in steel moulds on a vibration table (50 Hz, 0.35 mm) with vibration time of 60 seconds. Compressive strength of concrete after 28-day basic curing at 20°C and 100 % R. H. – wet air is reported in Table 2. For comparison with concrete the cement paste specimens (20^3 mm and 40x40x160 mm) with the same mixture proportions (cement, silica fume, superplasticizer and water / cement ratio of 0.32) and vibration time of 60 seconds were made and subjected to 28-day basic curing before temperature elevation.

Curing

Prismatic concrete specimens (21 altogether) and cylinders (21 altogether) following removal from the moulds and after 28-day basic curing were stored at temperatures of 40°C, 60°C, 100°C, and 200°C as seen in the Fig. 1. A part of the specimens was subjected to 28-day recovery period at 20°C / 60% R. H. – air cure after the exposure at 100°C and 200°C, respectively. Reference concrete properties were studied in 140-day / 20°C / 60% R. H. – air cure after basic curing in a wet air. The specimens of a cement paste were kept in the same curing regime than those of concrete.

Table 1. Concrete mixture composition

Components	Per m^3	
Portland cement of class 42.5	425	kg
Silica fume	32	kg
Aggregate: 0 / 4 mm	865	kg
Aggregate: 4 / 8 mm	393	kg
Aggregate: 8 / 16 mm	593	kg
Superplasticizer Melment L10	5.6	l
Water	136	l
Water / cement ratio	0.32	
Fresh concrete		
Temperature	16	°C
Volume density	2440	$kg.m^{-3}$
Slump	60	mm
Air content	2	% vol

Table 2. Compressive strength of concrete after 28-day basic curing at 20°C and 100 % R. H.

Size and shape of the specimens	Compressive strength (MPa)
150^3 mm	78.5
100^3 mm	81.8
100x100x400 mm	63.1

Testing

Concrete specimens were tested for weight (Fig. 2) and length changes (Fig. 3), dynamic modulus of elasticity (Fig. 4), prism compressive strength (Fig. 5), and impact strength (Fig. 6). All measurements were carried out immediately after the specimens were taken out of the hot air drier. In addition, stress-strain curve in compression of the concrete specimens exposed to temperatures at 100ºC and 200ºC was determined (Fig. 7) as well as the relations between the compressive stress, Poisson’s ratio (Fig. 8) and volume deformations (Fig. 9). These tests were performed after two or three days of cooling in the ambient temperature. The cement pastes were tested for cube compressive strength. The pore structure study was performed by mercury intrusion porosimetry (MIP) using a Carlo Erba porosimeter with a macroporosimetry unit (Milan) enabling measurements of pore radius between 3.75 nm and 0,2 mm.

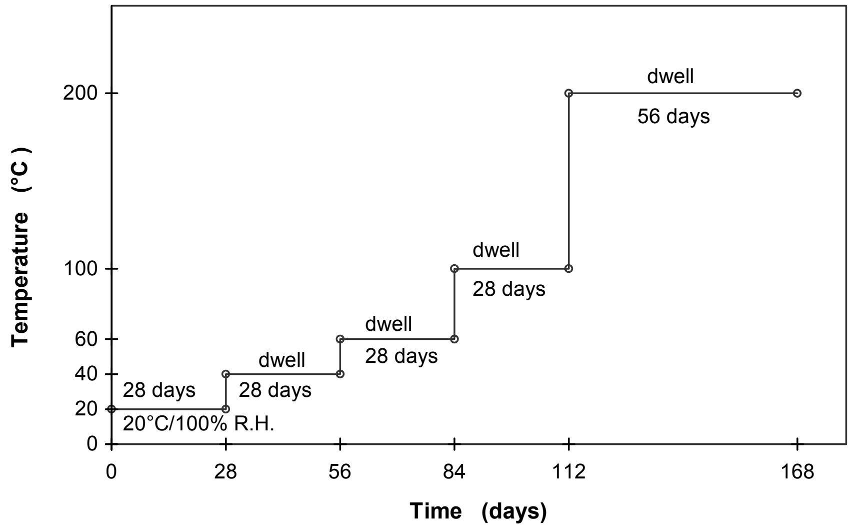

Fig. 1 Temperature regime of tested concrete specimens

RESULTS AND DISCUSSION

The evident drop in weight of the specimens due to released water is found at any temperature elevation, Fig. 2. By contrast with it, weight increase indicating the "self-curing" of concrete during the recovery periods is observed. However, one would take into consideration that structural quality of concrete has been deteriorated at temperature elevation already and the recovery at the ambient temperature may only very hardly achieve the improvement of physical state of concrete comparable to that of concrete before temperature attack. The degradation of concrete quality

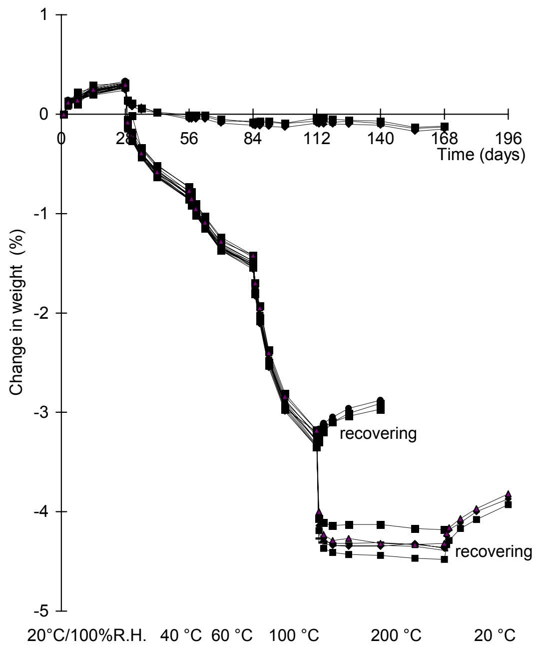

Fig. 2 Loss in weight

is an irreversible process. The shape of curves illustrating changes in shrinkage/expansion behaviour and dynamic modulus of elasticity is also characterised by the sudden turns corresponding to the sudden temperature changes which are then followed by a relatively stabilised stage, Fig. 3 and Fig. 4. At 200°C the thermal expansion begins to exceed pronouncedly the shrinkage. The immediate cooling of the specimens after the exposure to 100°C and 200°C evokes the thermal shock connected with rapid and large shrinkage remaining then on approximately constant levels during

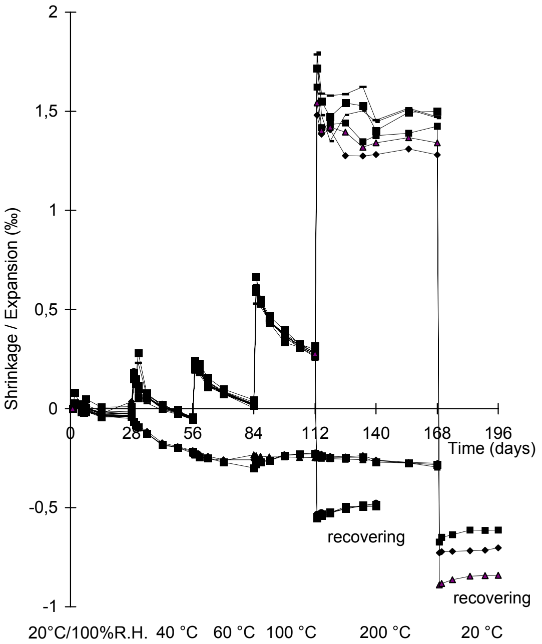

Fig. 3 Length changes

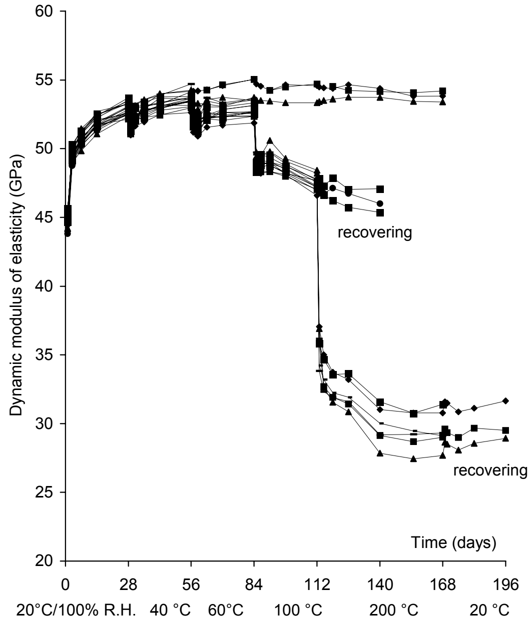

Fig. 4 Changes in dynamic modulus of elasticity

recovery periods at 20°C / 60% R.H. – air cure, Fig.3. Such a concrete behaviour at sudden temperature elevations is contributed to the quick release of bound water from hydrated cement paste. An expansion of concrete specimens proportional to the released amount of bound water in this short time is observed. In the following stabilised stage, no significant bound water release takes place, and a stabilised behaviour of concrete is found in this period. Instead of bulky hydration material due to released bound water from the cement paste, air voids occur in concrete. The structural integrity of the specimens is deteriorated as confirmed by losses in weight, Fig. 2 and dynamic modulus of elasticity, Fig. 4 at any temperature elevation. Loss in the weight confirms loss in the mass of concrete material, and the growth of the air voids portion. The immediate cooling of the specimens after exposure at 100°C and 200°C causes the extreme shrinkage, Fig. 3. It is believed that this shrinkage has only physical character due to the absence of previously released bound water from the cement paste. This finding is also supported by negligible changes in weight, Fig. 2 and dynamic modulus of elasticity, Fig. 4. A sudden turn of the length of concrete specimens exposed to 100°C and then cooled to the 20°C from +0.3‰ to –0.6‰, and that at 200ºC then cooled to 20ºC from +1.5‰ to –0.8‰ indicates that such length changes are the reason of significant deterioration of structural quality connected with the crack propagation and loss in mechanical properties. It is seen that structural integrity of concrete suffers pronouncedly either after a quick increase or after a quick decrease of temperature during a very short time interval.

Changes in prism compressive strength and impact strength of concrete specimens are illustrated in Fig. 5 and Fig. 6, respectively. Slight increase in prism compressive strength up to 100ºC is observed. Differences in compressive strength of concrete cured at 100ºC and 200ºC and control specimens kept only at the ambient temperature are negligible. Contrary, compressive strength significantly lowers after the recovery periods. The strength decrease of

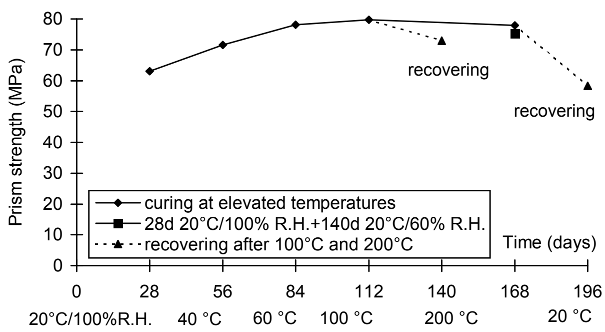

Fig. 5 Changes in prism compressive strength

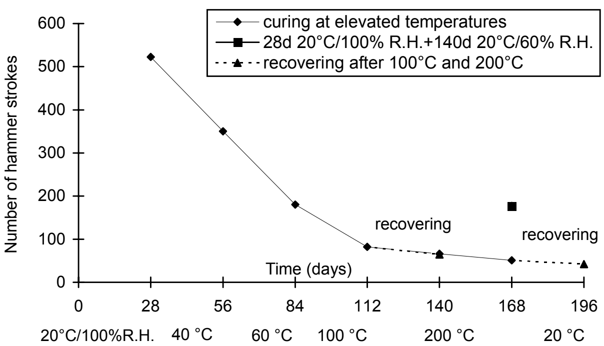

Fig. 6 Changes in impact strength

concrete after the exposure to 200ºC and that after a following recovery period is even 16 MPa, Fig. 5. Similar effect of temperature elevation and recovering is observed for the impact strength of the specimens. Evident losses in impact strength are found at any temperature elevation, and also after recovery periods, Fig. 6. Strength measures show that structural degradation of concrete is markedly influenced by the temperature elevation. The effect of temperature on prism compressive strength and impact strength is quite different. Recovery of concrete at 20ºC previously attacked by the temperature of 100ºC and 200ºC does not contribute to strength increase. This can be explained by the phenomenon of rapid cooling connected with extreme shrinkage of the specimens and crack propagation leading in the final effect to strength losses. The results show that rapid cooling of hot concrete surfaces exposed to 100ºC and 200ºC are for structural quality deterioration equally dangerous as temperature elevations.

The stress-strain curves of the specimens cured at 100ºC and 200ºC are illustrated in Fig. 7. In contrary to the prism strength measured immediately after the specimens were taken out of the hot air drier, the peak stress of the specimens cured at 200ºC is significantly lower than that of the control specimens. The Young's modulus decreases

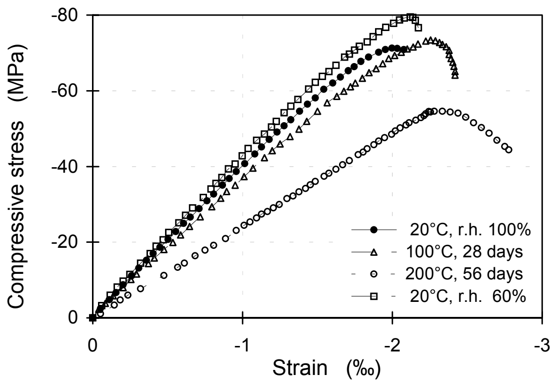

Fig. 7 Stress-strain curves

slightly after exposure to 100ºC and considerably after specimens were exposed to 200ºC. The course of the stress-strain curve after temperature attack of 200ºC exhibits a pronounced "softening" of concrete visible on both ascending and descending branch. Changes in elastic properties of concrete after the exposure to the elevated temperatures are demonstrated also by the change of the Poisson's ratio, which is illustrated in Fig. 8. As it is known, the Poisson's ratio

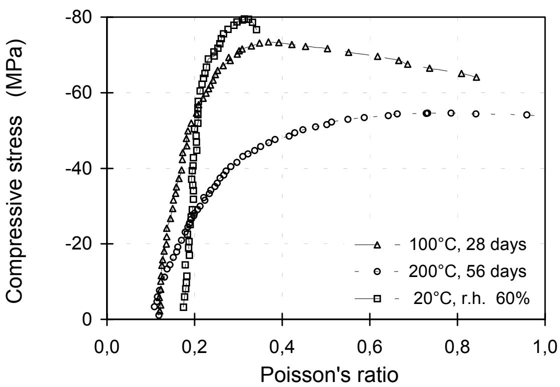

Fig. 8 Poisson's ratio vs. compressive stress

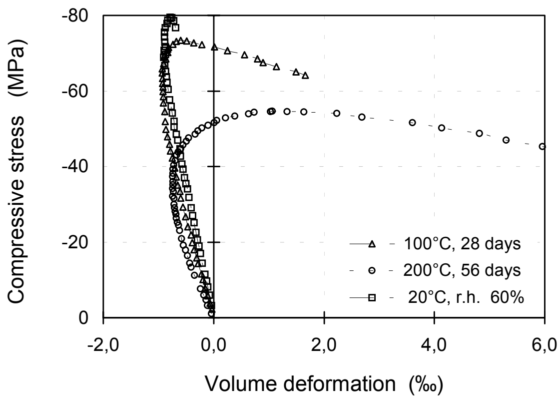

Fig. 9 Volume deformation vs. compressive stress

of concrete is stress independent and is about 0.2, as can be seen in Fig. 8 for the control specimens kept in the ambient temperature. The Poisson's ratio of the concrete cured at 100ºC and 200ºC becomes stress dependent, starting with the value of 0.12 and growing with increasing stress, the growth being more pronounced for concrete exposed to 200ºC. The course of the volume deformation plotted in Fig. 9 also shows that concrete specimens exposed to 200ºC start to expand before the peak stress is reached which confirms the structural deterioration of the concrete.

Compressive strength of the cement paste, Fig. 10 with the same mixture composition than that in concrete, and the same temperature elevations, exposure times and curing conditions is always higher relative to the compressive strength

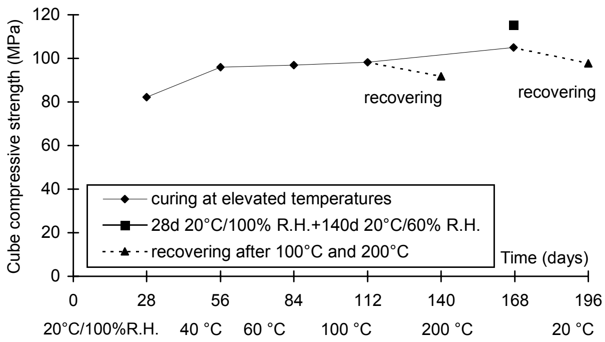

Fig. 10 Cube compressive strength of cement pastes related to the cement paste in concrete specimens

of concrete, Fig. 5. This is due to strength losses in concrete caused by interfacial zones between the cement paste and aggregate being regarded as the weakest of bulky concrete material. Compressive strength of the cement paste is decreased after recovery periods showing similar trends of strength losses as observed in concrete specimens. Surprisingly, the cement paste recovering 28 days at 20 ^{o}C / 60 % R.H. – air after temperature elevation to 100 ^{o}C and 200 ^{o}C, respectively, reveals decreased compressive strength than those found only after exposures to 100 ^{o}C and 200 ^{o}C. This indicates lasting and irreversible character of a cement paste deterioration. It is seen that the cement paste (or concrete) once damaged by a higher temperature keeps its structural integrity disqualification even in the recovery period being regarded as "self-curing". The recovery at the ambient curing conditions after temperature attack does not contribute to the strength increase of a cement paste (or concrete). Contrary, significant decline in strength was observed. Such behaviour of the cement paste can be explained by changes in pore structure matrix at temperature elevations followed by rapid cooling and lasting exposure at 20 ^{o}C. It is believed that just a rapid cooling at the beginning of recovery periods is the reason of pore matrix damage due to extreme shrinkage in a few minutes. Relevant length changes of concrete are illustrated in Fig.3. It is clear that shrinkage of a neat cement paste without aggregate is higher than that of concrete but the tendency to the immediate shrinkage after temperature drops from 100 ^{o}C and 200 ^{o}C to 20 ^{o}C is evident. Negligible changes in all significant parameters of the formed pore structure matrix in the cement paste show its high ability to withstand temperature attack up to 200 ^{o}C. This is confirmed by strength values of the cement paste, Fig. 10, and compressive strength of concrete specimens reported in Fig. 5. No clear dependence between strength increase and temperature elevations was found. By contrast with it, compressive strength of the cement paste and concrete are decreased after recovery periods at 20 ^{o}C in comparison with those at 100 ^{o}C and 200 ^{o}C. This is caused by the pore structure coarsening due to increases in volume of macropores (pore radius over 7 500 nm), pore median radius and total porosity (Tab. 3). The compressive strength dependence on parameters of the formed pore

Table 3. Pore structure study of cement pastes

Curing regime used	Volume of macropores ($mm^3.g^{-1}$)	Portion of macropores (%)	Median of micropore radius (nm)	Median of pore radius (nm)	Total porosity (3.75 nm – 0.2 mm) (% vol.)
28 days basic	50.4	0	23.5	23.5	9.4
To 100ºC	46.6	0	18.9	18.9	9.2
100ºC + recovery	48.4	6.7	21.6	23.6	10.3
To 200S	55.9	4.4	23.1	23.7	11.2
200ºC + recovery	41.7	5.2	31.8	32.6	11.9
20ºC/60% R.H.	49.2	5.8	26.2	26.8	10.8

matrix is clearly reported in Table 4. The cement paste recovering after temperature elevation gives in the final effect the higher volume of macropores, pore median radius and total porosity values. The larger air voids portion at lower compressive strength compared to the measured pore structure parameters and compressive strength of cement paste kept only at 100 ^{o}C and 200 ^{o}C are observed. The detrimental effect of temperature changes on structural integrity of concrete and cement paste is markedly higher after a cooling from a higher temperature to the ambient 20 ^{o}C than that when concrete and cement paste are kept only at elevated temperature. This is contributed to extremely high shrinkage during cooling connected with the pore structure coarsening, air voids portion increasing and decline in strength. Recovery of concrete and cement paste after a rapid cooling does not enhance physico – mechanical characteristics during a " self-curing ". Contrary, concrete and the cement paste are still evidently deteriorated. Recovering at 20 ^{o}C does not provide the wishing improvement of their physical state. One would also take into consideration this fact anticipating future service-life of concrete structures exposed to higher temperatures. Concrete in temperature - attacked

Table 4. Changes in strength of cement paste related to pore structure development at ambient curing and temperature elevations

Curing regime used	Portion of macropores (%)	Pore median radius (nm)	Total porosity (% vol.)	Compressive strength (MPa)
28 days basic	0	23.5	9.4	82.2
To 100°C	0	18.9	9.2	98.2
To 200°C	4.4	23.7	11.2	105.0
20°C/60% R.H.	5.8	26.8	10.8	115.3
To 100°C	0	18.9	9.2	98.2
100°C + recovery	6.7	23.6	10.3	91.8
To 200°C	4.4	23.7	11.2	105.0
200°C + recovery	5.2	26.8	11.9	97.7

elements remains in bad physical conditions and the consequent recovering at 20 °C does not contribute to the wishing improvements. One would also take into account that the higher the temperature elevation, the higher strength losses and coarser pore structure matrix, and the lower significance of "self-curing" recovery period with expectations of concrete property improvement. Our long – term measurements (after basic curing at 20 °C/100 % R.H. – wet air, 100 °C and 200 °C temperature exposure) clearly show that shrinkage of the cement paste is forever negatively affected by a previous temperature elevation, Tab.5. This means that the cement paste is not able to achieve its original physical

Table 5. Long-term shrinkage of the cement paste at various exposure conditions using 40x40x160 mm specimens

Exposure condition	Time (days)	Character of the length change	Shrinkage (‰)	Relative shrinkage (%)
Basic curing, 20°C/100% R.H. - air	28	shrinkage	-0.3	100
After 40 °C	56	shrinkage	-2.2	733
+ 1 day at 60 °C	57	expansion	-1.6	533
After 60 °C	84	shrinkage	-3.2	1 066
+ 1 day at 100 °C	85	expansion	-3.05	1 016
After 100 °C	112	shrinkage	-5.8	1 933
+ 1-day recovering after 100 °C	113	shrinkage	-6.6	2 199
+ 28-day recovering after 100 °C	140	expansion	-5.4	1 799
+ 429-day recovering after 100 °C	541	expansion	-3.8	1 266 (158)
+ 1 day at 200 °C	113	shrinkage	-8.2	2 733
+ 28 days at 200 °C	140	shrinkage	-11.05	3 583
+ 56 days at 200 °C	168	shrinkage	-11.1	3 699
+ 1-day recovering after 200 °C	169	shrinkage	-13.0	4 333
+ 28-day recovering after 200 °C	196	expansion	-11.15	3 716
+ 373-day recovering after 200 °C	541	expansion	-7.9	2 633 (329)
+ 513 days after basic curing at 20 °C/60 % R.H.-air (reference)	541	shrinkage	-2.4	799 (100)

state comparable with those: 1.) before temperature attack even at a lasting recovery period, and 2.) in the reference 20 °C/60 % R.H. – dry air cure. Relative shrinkage values related either to the 28-day basic curing or 541-day curing at 20 °C/60 % R.H. air (in brackets) show the impossibility of the cement paste to acquire the origin shrinkage values. The observed changes in shrinkage are irreversible either after temperature elevations, either after lasting recovery periods at 20 °C. As it has been reported already, the cement paste plays a key role in concrete. Bearing in mind well-known retarding effect of aggregate on the extent of shrinkage it is clear that concrete attacked by high temperatures indicates similar behaviour as seen in Fig. 3. The temperature elevation followed by a rapid cooling period that is changed by a lasting recovering proves persistently deteriorated physical state of the cement paste in concrete with the whole consequence on its residual strength, elasticity, deformation, pore structure and permeability. We suggest that the assessment of concrete structures that have once in the past suffered by a high temperature at arbitrary long periods of the attack would concern this fact as a basic tool for better prediction and modelling their further service-life.

CONCLUSIONS

The following conclusions are applicable to the particular concrete, cement paste and test conditions employed:

1.) The strength, elasticity modulus and deformation of concrete are irreversibly influenced by temperature elevation mainly to 100 ^{o}C and 200 ^{o}C. The decisive changes leading in the final effect to the structural deterioration are taking place immediately after the temperature elevation (the observed time-limited loss in weight and elasticity modulus, and expansion increase). Concrete exhibits a pronounced "softening" though the formed pore matrix and compressive strength are influenced at individual temperature levels only negligibly.

2.) The effect of pore structure coarsening at the end of recovery periods after 100 ^{o}C and 200 ^{o}C exposures very evidently results in significant concrete and cement paste strength decrease. Rapid cooling after temperature elevations evokes equal and irreversible structural quality deterioration of concrete and cement paste. The " self-curing " of concrete and cement paste after short and long recovering at 20 ^{o}C does not contribute to the structural integrity improvement. Concrete and cement paste is persistently deteriorated, and the impossibility to acquire their origin physical state before temperature attack is observed.

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