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Effect of damage on permeability and hygro-thermal behaviour of HPCs at elevated temperatures: Part 1. Experimental results

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Abstract. This paper presents an analysis of some experimental results concerning micro-structural tests, permeability measurements and strain-stress tests of four types of High-Performance Concrete, exposed to elevated temperatures (up to 700°C). These experimental results, obtained within the "HITECO" research programme are discussed and interpreted in the context of a recently developed mathematical model of hygro-thermal behaviour and degradation of concrete at high temperature, which is briefly presented in the Part 2 paper (Gawin, *et al.* 2005). Correlations between concrete permeability and porosity micro-structure, as well as between damage and cracks' volume, are found. An approximate decomposition of the thermally induced material damage into two parts, a chemical one related to cement dehydration process, and a thermal one due to micro-cracks' development caused by thermal strains at micro- and meso-scale, is performed. Constitutive relationships describing influence of temperature and material damage upon its intrinsic permeability at high temperature for 4 types of HPC are deduced. In the Part II of this paper (Gawin, *et al.* 2005) effect of two different damage-permeability coupling formulations on the results of computer simulations concerning hygro-thermo-mechanical performance of concrete wall during standard fire, is numerically analysed.

Keywords: high-performance concrete; permeability; micro-structure; elevated temperature; micro-cracking.

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1. Introduction

Prediction of concrete performance at high temperature is of great practical interest, especially in safety evaluation in tall buildings and in tunnels. In the past years several major, high profile and costly tunnel fires have taken place in Europe that resulted in significant loss of life (about 500 persons) and damage to the structures. Channel Tunnel, Mont-Blanc, Tauern, Kaprun and St. Gotthard are examples of tunnels where accidents occurred due to fire, thus clearly indicating the inadequacy of current design procedures. In the cases referred above, the duration and extent of the fire were longer than expected and, in some cases, the temperature regimes were much more severe than those considered in the codes. Moreover the inspection after fire showed extensive damage to the concrete elements, e.g., in the affected sections of the Channel Tunnel the concrete lining was almost completely removed by spalling.

Spalling, which may be explosive, (Bazant and Kaplan 1996), is the final step of a continuous damage process which starts to develop at relatively low temperature. Spalling is mainly due to different coexisting coupled processes, such as thermal (heat transfer, thermal dilatation of concrete components), chemical (dehydration of cement paste and resulting decrease of its strength properties), hygral (transfer of moisture, shrinkage, increase of vapour pressure) and mechanical degradation (cracking).

Because of the important consequences of loss of concrete integrity under fire, many researchers studied problems related to high temperature behaviour of concrete, see Bazant (1988), Bazant and Kaplan (1996), Bazant and Thonguthai (1978, 1979). The mathematical model proposed by Bazant and Thonguthai (1978, 1979) is widely used for this purpose. However that model has some limitations, e.g., moisture is considered as a one-phase fluid and phase changes are omitted. Moreover, it admits some situations which are physically incorrect e.g., relative humidity greater than one is attributed to material fully saturated with water. Hence there is still need for a more accurate and realistic description of mass and energy transport in concrete in high temperature environment.

Recently a new fully coupled and non-linear mathematical model for computer simulation of hygro-thermal and degradation phenomena in concrete structures exposed to high temperature was presented by Gawin, *et al.* (1998, 1999, 2002a, 2002b, 2003, 2004).

In a previous paper, Gawin, *et al.* (2002b), a general form of the relation describing effect of damage on concrete intrinsic permeability at high temperature was proposed. However, its parameters were not determined experimentally, because of lack of any suitable data for concrete at high temperature. In this paper, some experimental results of the "HITECO" research project, (Brite Euram III, 1999), are analysed and discussed in the context of mathematical modelling of damage and hygro-thermal behaviour of concrete at high temperature, and in particular, damage-permeability coupling.

These results deal with various types of High Performance Concrete (HPC), which have low porosity and consequently low permeability, being particularly endangered by deterioration at high temperature.

Constitutive relationships describing influence of damage on concrete intrinsic permeability, as well as its parameters for 4 types of HPC are deduced from these tests. A first attempt to estimate the values of the thermal (i.e. induced by thermal strains at micro- and meso-level) and chemical (i.e. caused by dehydration) components of the thermo-chemical damage of concrete at high temperature is made. In the Part 2 paper (Gawin, *et al.* 2005) effects of the new mechanistic

formulation of damage-permeability coupling on results of computer simulations are numerically analysed by comparing them with the results obtained by means of the previously proposed, phenomenological formula, that does not take into account the thermo-chemical damage of concrete in a direct way.

2. Results of micro-structural and gas permeability tests of HPCs

Several micro-structural and material parameters of 4 HPCs and 3 UHPCs subjected to elevated temperatures were versatilely tested within the framework of the "HITECO" project, (Brite Euram III, 1999). In this paper only a part of these experimental results, related to the main subject of the paper, is presented and discussed. Micro-structural and permeability tests were carried out at ambient temperature (20°C) after heating to the testing temperature (200°C, 300°C, 400°C, 500°C, 600°C and 700°C) and cooling again; therefore, they may be considered as "residual" material characteristics. These tests were made on three specimens submitted to the same regime of heating and also selected from three different original concrete specimens. Below the mean values obtained from the tests are given. Some basic data about composition of the HPCs used in these tests are summarized in Table 1.

To obtain an information about total porosity and pore size distribution of concretes at elevated temperature, both nitrogen adsorption and mercury intrusion porosimetry (MIP) tests were carried out. The latter technique allowed the measurement of macro-pores and larger meso-pores, within the range from 6 nm to 200 μ m. This range of pore sizes has a dominant influence on concrete

Concrete symbol	<i>w/b</i> ratio	Binder content	Binder composition	Aggregate type
	[—]	kg/m ³		
C60	0.36	450	OPC-100%	Calcareous: max. size-20 mm
C60 SF	0.354	367	OPC-85%, fly ash-10%, microsilica-5%	Granite: max. size-16 mm
C70	0.32	490	OPC-100%	Gabbro: max. size-16 mm
C90	0.29	561	OPC-91%, microsilica-5%	Gabbro: max. size-16 mm

Table 1 Basic data about composition of the analysed HPCs

Table 2 MIP results of total	porosity measurement	s at various temperature	es for the analysed HPCs

Temperature	C60	C60 SF	C70	C90
20°C	8.99 ± 2	10.2 ± 2.23	6.21 ± 1.34	5.97 ± 0.79
200°C	13.44 ± 0.64	11.97 ± 1.01	9.19 ± 2.50	11.79 ± 0.24
300°C	12.02 ± 1.92	12.32 ± 5.07	11.63 ± 1.21	9.63 ± 1.72
400°C	13.59 ± 1.54	14.06 ± 3.54	13.16 ± 0.60	14.27 ± 1.63
500°C	16.71 ± 0.68	15.74 ± 0.01	13.42 ± 1.63	14.96 ± 2.96
600°C	19.93 ± 1.83	15.22 ± 0.16	16.63 ± 1.74	16.38 ± 1.15
700°C	20.19 ± 4.31	15.99 ± 6.05	18.99 ± 3.85	16.60 ± 0.30

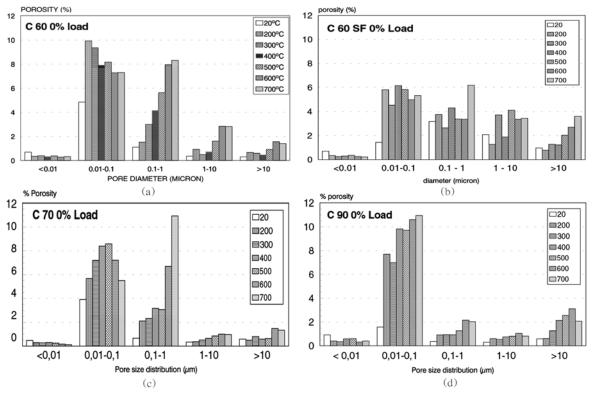


Fig. 1 Changes of pore size distribution in concrete after exposure to high temperatures: (a) C60, (b) C60 SF, (c) C70, (d) C90

permeability at temperatures higher than 105°C, Bazant and Kaplan (1996), and covers also the range of micro-cracks and cracks developing at elevated temperatures, see e.g., Hinrichsmeyer (1987) and Schneider and Herbst (1989). The samples, of 1-3 cm³ size, were obtained from fracture of a larger specimen. Aggregates bigger than medium size of the sample were eliminated. Each sample was dried using a vacuum pump for at least one week, before starting with the test. The highest mercury filling pressure used was 30,000 psi (~206.85 MPa), to minimize harmful effect of excessive pressures. MIP results concerning total porosity of the analysed HPCs at various temperatures are summarized in Table 2, and the pore size distributions are shown in Fig. 1a-d.

The residual gas permeability of the analysed concretes, heated to a given temperature and then cooled to room temperature, was determined by means of the Cembureau equipment using air as flowing gas. The samples used were slices of concrete with diameter of 100mm and height of 50mm. The air flux was measured at various pressures and the mean value of permeability coefficient was calculated. The range of pressures used during the tests was chosen in such a way to maintain gas flux in a measurable range of the equipment. Hence for higher temperatures, when micro-cracks and cracks appeared in the concrete samples, use of lower gas pressures was necessary. Nevertheless, the samples of C90 concrete at temperatures exceeding 500°C were cracked to such an extent that performing any flux measurement was impossible. The results of residual gas permeability tests for the analysed HPCs heated to various temperatures are summarised in Table 3.

Table 3 Values of intrinsic permeability coefficient, $k [10^{-18} \text{ m}^2]$, obtained from gas permeability tests of the analysed HPCs

Temperature	C60	C60 SF	C70	C90
20°C	45.60 ± 4.70	51.39 ± 5.30	55.00 ± 3.10	75.8 ± 26.8
200°C	280.3 ± 32.4	180 ± 27	577.0 ± 14.3	1750 ± 252
300°C	1507 ± 184	630 ± 27	1880 ± 246	10200 ± 2130
400°C	4281 ± 1148	1799 ± 268	2738 ± 212	42600 ± 12400
500°C	12663 ± 3106	5067 ± 370	7448 ± 545	176000 ± 26200
600°C	39060 ± 13160	17500 ± 4800	11833 ± 1043	-
700°C	79960 ± 30330	33000 ± 4600	19110 ± 409	-

Table 4 Values of intrinsic permeability coefficient, $k [10^{-18} \text{ m}^2]$, of the analysed HPCs at various temperatures, calculated from Klinkenberg's relation

Temperature	C60	C60 SF	C70	C90	
20°C	2.43	4.50	3.58	6.64	
200°C	69.2	16.6	214	161	
300°C	613	173	187	2800	
400°C	829	812	375	19200	
500°C	3040	2390	2080	82800	
600°C	8190	8240	3540	_	
700°C	26100	15500	5900	_	

Table 5 Values of Young's modulus, *E*, and thermo-chemical damage, *V*, for the analysed HPCs at various temperatures

Temperature	C60		C60 SF		C70		C90	
	E [GPa]	V [-]						
20°C	34.50	0.00	36.70	0.00	34.61	0.00	36.70	0.00
200°C	24.82	0.28	20.90	0.43	31.07	0.10	20.90	0.43
300°C	21.81	0.37	15.30	0.58	31.16	0.10	15.30	0.58
400°C	19.00	0.45	9.40	0.74	15.71	0.55	9.40	0.74
500°C	14.34	0.58	7.80	0.79	14.30	0.59	7.80	0.79
600°C	12.30	0.64	6.90	0.81	6.81	0.80	6.90	0.81

It is known that the measured gas- and water-permeability values of concrete may differ by even 2-3 orders of magnitude as a result of gas slippage phenomenon. This effect depends on the mean gas pressure used during the gas permeability test, as was shown for concrete by Bamforth (1987). To compare permeability values measured at various pressures, water permeability coefficients were calculated from the mean values given in Table 3, using Klinkenberg's relation with coefficients modified for concrete, (Bamforth 1987). The results are shown in Table 4.

To obtain information about material damage, the results of residual elastic strain and compressive strength measurements were used, (Brite Euram III, 1999). The specimens were heated at a rate 2° C/minute to the test temperatures, that were maintained for one hour, and then they were slowly

cooled to ambient temperature. At each temperature three specimens were tested. The values of Young modulus obtained from these measurements and thermo-chemical damage, V, calculated using Eq. (3), are given in Table 5. In reality, in the direct measurements of Young modulus one determines the total effect of damage, i.e. the total damage parameter D defined by Eq. (8). However, in the analysed situation, due to small dimensions of the sample, a small heating rate and no external stress load present, one can assume that the mechanical damage component d equals to zero, and the actual value of Young modulus corresponds to that of undamaged material $E_0(T)$.

3. Modeling of concrete damage

Degradation of concrete at high temperature, arising from a coupled hygro-thermal, chemical (dehydration) and mechanical interaction can be modelled by means of the isotropic non-local damage theory of Mazars (1984, 1986) and Mazars and Pijaudier-Cabot (1989), as done for example by Gawin, *et al.* (1998, 1999, 2002a, 2002b). Thermo-chemical effects are also taken into account, as proposed by Gerard, *et al.* (1998), Nechnech *et al.* (2001) and Gawin, *et al.* (2003).

The damage theory defines a "modified effective stress" $\tilde{\sigma}$ and takes into account both the mechanical damage d ($0 \le d \le 1$), as a parameter measuring the reduction of resistant area due to cracks, and thermo-chemical damage V ($0 \le V \le 1$) as a parameter describing thermo-chemical material degradation at elevated temperatures (mainly due to micro-cracking and cement dehydration) resulting in a reduction of the material strength properties,

$$\tilde{\sigma} = \frac{\sigma'}{(1-d)(1-V)} \tag{1}$$

where σ' is the effective stress (called also Bishop's stress), responsible for deformations of the material skeleton.

The mechanical damage parameter can be determined using the experimental data of the Young's modulus for a mechanically undamaged material at a given temperature T (i.e. only heated, without any mechanical load), $E_0(T)$, and for the mechanically damaged material at the same temperature, E(T), as follows (Mazars 1984, 1989, and Mazars and Pijaudier-Cabot 1989),

$$d = 1 - \frac{E(T)}{E_0(T)} \tag{2}$$

The thermo-chemical component of damage is described in terms of experimentally determined relation of Young's modulus for mechanically undamaged material as a function of temperature, $E_0(T)$, according to the relation, (Gawin, *et al.* 2003, Gerard, *et al.* 1998),

$$V = 1 - \frac{E_0(T)}{E_0(T_0)}$$
(3)

where T_0 is room temperature.

4. Analysis of the tests results

The results presented in the previous section are analysed now in the context of the mathematical

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model proposed by Gawin *et al.* (1998, 1999, 2002a, 2002b, 2003, 2004), briefly presented in the Part 2 paper Gawin, *et al* (2005), and in particular, damage-permeability coupling which is an important constitutive relationship of the model.

The cracking phenomenon has a key role for increase of concrete permeability at elevated temperatures. As pointed out in Brite Euram III (1999), analysing all micro-structural tests results, including Scanning Electron Microscopy (SEM) and Back Scattered Electron Microscopy (BSEM), micro-cracks in the paste, resulting from autogenous shrinkage, may be present even at ambient temperature (e.g. C60, C90). They are paths for further growing at higher temperatures. Micro-cracks surrounding the aggregates or anhydrous components, usually appear above 300°C. As temperature increases, cracks grow preferably through paste, deteriorated due to the dehydration process, and above 500°C they start growing around aggregates, reaching the size greater than 10 μ m. At higher temperatures cracks grow in size and can cross aggregates.

In a phenomenological approach, e.g., Bazant and Kaplan (1996), Bazant and Thonghutai (1978, 1979), a formula relating permeability directly to temperature is needed. Usually it is expressed in one of the two possible exponential forms, [5]:

$$\kappa = k_0 \times \exp[C_T(T - T_0)] = k_0 10^{A_T(T - T_0)}$$
(4)

where k_0 is the intrinsic material permeability at reference temperature $T_0 = 293.15 \text{ K}$, C_T and $A_T = C_T \cdot \log(e)$ - are material dependent parameters.

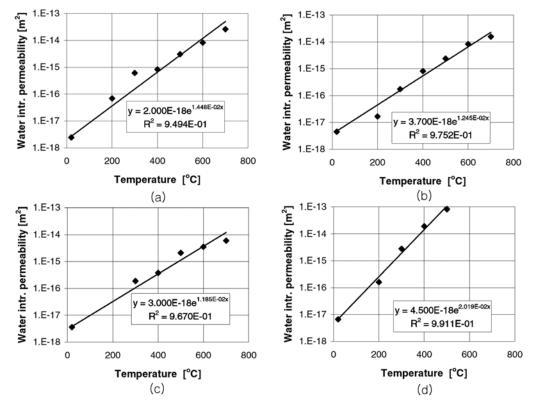


Fig. 2 Dependence of water intrinsic permeability on temperature for HPCs at elevated temperatures: (a) C60, (b) C60 SF, (c) C70, (d) C90

Using the data from Table 4, dependence of the water intrinsic permeability on temperature for the analysed types of HPCs is presented in Fig. 2a-d, together with the C_T parameter values of formula (4), obtained by means of the least square method, and the correlation coefficients. For the C70 the permeability value at temperature of 200°C is omitted in further analysis because it is inconsistent with other results. As it may be seen in Fig. 2, formula (4) is in a good accordance with the experimental results for all the considered HPCs.

However, using a mechanistic approach, changes of intrinsic permeability should be expressed in terms of state variables describing the real physical reasons of the phenomenon, i.e., increase in porosity and material cracking in our case. The latter may be described by means of the thermochemical damage -V, see Gawin, *et al.* (2003) and Nechnech, *et al.* (2001). For this purpose one of the two exponential type formula may be used, Gawin, *et al.* (2002b), Bary (1996),

$$k = k_0 \times \exp([C_V V] \ k_0 10^{A_V V})$$
(5)

where k_0 means intrinsic material permeability of undamaged material, C_V and $A_V = C_V \cdot \log(e)$ - are material dependent parameters.

The relations between intrinsic permeability and damage parameter, V, for the considered HPCs are shown in Figs. 3a-d. The values of the C_V parameters of the formula (5) have been obtained by

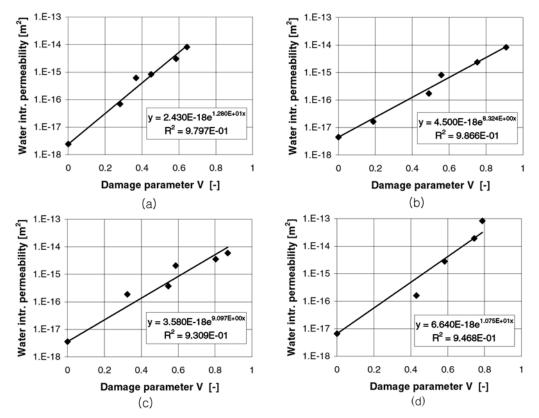


Fig. 3 Dependence of water intrinsic permeability on thermo-chemical damage for HPCs at elevated temperatures: (a) C60, (b) C60 SF, (c) C70, (d) C90

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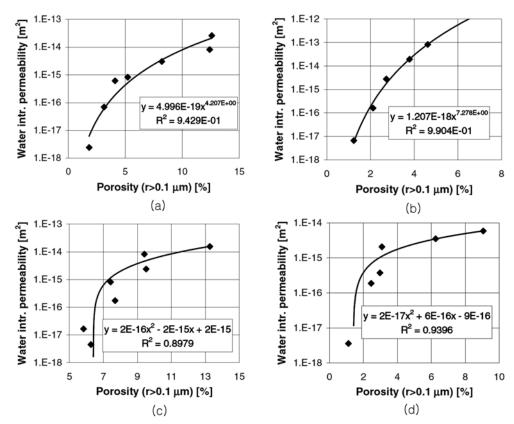


Fig. 4 Dependence of water intrinsic permeability on micro-cracks and cracks porosity of HPCs at elevated temperatures: (a) C60, (b) C60 SF, (c) C70, (d) C90

means of the least square method. In this case the correlation coefficients between the physical quantities are high as well.

Thermo-chemical damage parameter takes into account decrease of Young's modulus caused both by chemical changes of concrete at elevated temperature (dehydration) and its mechanical deterioration (cracking). The latter process is obviously influenced by concrete dehydration, which is in turn governed by the temperature changes. However, we do expect a stronger correlation between total volume of micro-cracks and cracks, and material intrinsic permeability. Such a correlation is analysed in Figs. 4a-d, assuming that micro-cracks and cracks correspond to pores of dimensions greater than 0.1 μ m. Of course, cracks influence significantly also pores tortuosity, hence the latter correlation is not so distinct as in the previous cases, Figs. 2-3. Nevertheless, a certain correlation seems to exist.

To explore this problem more in detail, we shall try to estimate what part of thermo-chemical damage is related directly to the decrease of Young's modulus due to cement dehydration process (chemical component of damage, V_{chem}). The remaining part, called further the thermal component of damage, V_{therm} , is caused mainly by temperature-induced changes of material structure, i.e., mainly micro-cracking and cracking, since porosity increase due to dehydration is relatively small and may be neglected in the analysed temperature range. For these components of thermo-chemical damage holds a multiplicative relation, i.e., $(1-V) = (1-V_{chem}) \cdot (1-V_{therm})$.

The estimation is done by using the following, approximate relation between relative hydration, w_n/w , and compressive cylinder strength, f_c , obtained by Persson (1996), for HPCs (60 MPa $< f_c$ < 125 MPa):

$$f_c = 260 \times (w_n / w) \ 0.085 \tag{6}$$

where w_n means non-evaporable water content in 1 m³ concrete and w - water content in 1 m³ of concrete mix. It was assumed that the same relation describes the relative changes of concrete strength caused by the non-evaporable water losses due to dehydration at high temperatures.

Additionally, the following approximate relation between compressive strength of HPC - f_c [MPa], and its Young's modulus - E [MPa], see Neville (1995),

$$E = 3320 \sqrt{f_c} + 6900 \tag{7}$$

is used.

To estimate the changes of the non-evaporable water content, the results of thermo-gravimetric analyses (TG and DTA) of the considered HPCs in the temperature range from about 105°C to 650°C, Brite Euram III (1999), are used. The endo-thermic peaks related to the concrete components transformations (i.e. portlandite, quartz and aggregate) are not taken into account. Most

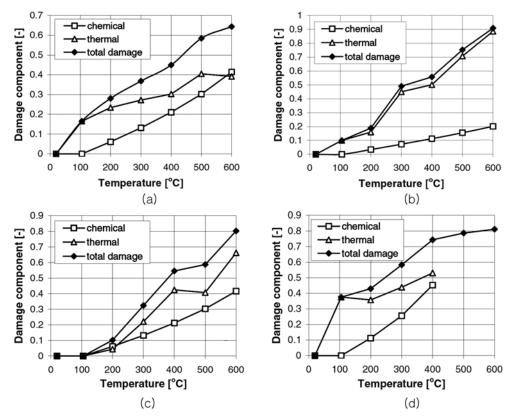


Fig. 5 Approximate decomposition of thermo-chemical (total) damage of HPCs at elevated temperatures into chemical and thermal components: (a) C60, (b) C60 SF, (c) C70, (d) C90

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of the rest weight losses may be attributed to continuous dehydration of the CSH gel and caused by its losses of the non-evaporable water.

The results of this approximate, multiplicative damage decomposition into chemical and thermal components are presented in Figs. 5a-d. The latter component, calculated from the experimentally determined values of thermo-chemical (total) and chemical damage, is slightly decreasing in single points of Figs. 5a,c,d. This is thermodynamically inadmissible, because material damage is an irreversible physical phenomenon. This inconsistency of the presented decomposition may be explained by the fact that it is based on the test on different specimens (tested at precise temperatures). However, the relation found enables to assume that general trends and orders of magnitude for the damage components may be deduced from the analysis properly.

The chemical damage of the C90 concrete, resulting from the approximated formulas (6) and (7), seems overestimated, especially for higher temperatures. Indeed, this component starts to exceed the total (thermo-chemical) damage above temperature of about 490°C, what is physically not admissible.

Despite of approximate character of the presented damage decomposition, one may expect a correlation between value of thermal damage and total volume of micro-cracks and cracks. Results of such an analysis are presented in Figs. 6a-d. A similar analysis has also been done for the thermo-chemical damage (not presented here), giving distinctly lower correlation coefficients, what

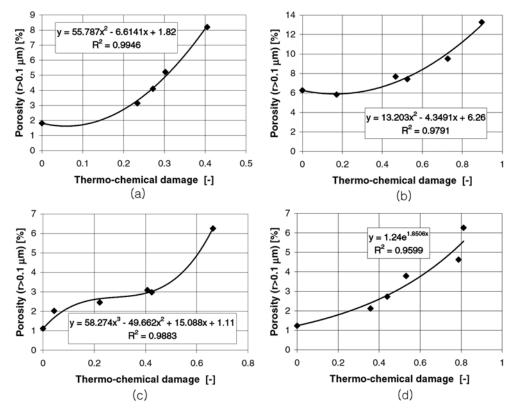


Fig. 6 Dependence total porosity of micro-cracks and cracks for HPCs at elevated temperatures, upon thermo-chemical damage: (a) C60, (b) C60 SF, (c) C70, (d) C90

qualitatively confirms the correctness of our assumptions and reasoning concerning the thermochemical damage decomposition.

The results presented in Fig. 5 show, that for the analysed concretes in the temperature range up to 450-500°C (and for the C60 SF even in the whole considered temperature range), the dominating reason of material damage is micro-cracks and cracks development, causing an increase of intrinsic permeability, Fig. 4. Hence, taking into account the approximate character of the thermal damage component evaluation, it seems reasonable to use in mathematical modelling a relationship of the type of Eq. (5), i.e., with total thermo-chemical damage, V, which is well defined by Eq. (3) and relatively easy for experimental determination. Moreover, one may expect, that similar relationship will be also valid for purely mechanical damage, d. Indeed, an exponential type formula was proposed by Bary (1996) for dependence of intrinsic permeability upon damage parameter. The value of the C_V parameter estimated there was equal to about 9.21 ($A_V = 4$) for normal concrete, what is quite similar to the values for the HPCs, presented in Fig. 3, varying from 8.24 to 12.8. Considering all of these facts, it seems reasonable to assume, that the influence of purely mechanical damage on permeability of HPCs is described by the same exponential relation as in the thermo-chemical damage case. However, one should take into account that total effect of the mechanical and thermo-chemical damages acting at the same time is multiplicative, i.e., the total damage D, defined by formula, (see Gawin, et al. 2003, Nechnech, et al. 2001),

$$D = 1 - \frac{E(T)}{E_0(T_0)} = 1 - \frac{E(T)}{E_0(T)} \frac{E_0(T)}{E_0(T_0)} = 1 - (1 - d) \cdot (1 - V)$$
(8)

has to be used in (5), and not the sum of the thermo-chemical damage, V, and mechanical damage, d.

Concluding, it is worth to underline, that despite qualitative similarities, every analysed HPC behaves at elevated temperatures differently, see Figs. 2-6. Thus it seems that one cannot expect to find any universal correlation between temperature induced material damage, changes in porosity micro-structure and resulting permeability variations with the type of assumptions made until now. Consequently every type of HPC should be profoundly tested and analysed until more knowledge could verify whether any general relation could be established.

6. Conclusions

Analysis of the results concerning micro-structural tests, permeability measurements and strainstress tests of four types of High Performance Concrete exposed to elevated temperatures (up to 700°C), allowed for formulation of the constitutive relationship describing influence of damage on concrete intrinsic permeability. The latter has been expressed both in terms of temperature (phenomenological approach) and damage parameter (mechanistic form). A correlation between the concrete inner micro-structure and intrinsic permeability has been shown to exist. Contributions of the chemical (decrease of strength properties due to dehydration) and cracking phenomena into the total concrete damage process have been quantitatively estimated.

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