PRINCIPAL CRITERIAL RELATIONSHIPS IN THE KINETIC CHARACTERISTICS OF THE HEAT TREATMENT OF CEMENT MATERIALS

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Changes taking place in the Rb, Ko, and $\mathrm{Ki_m}$ numbers during heat treatment are studied. Analysis of the criterial relationships presented reveals the mechanism of heat and mass transfer for various methods of applying the heat, i.e., the mechanism which influences structure formation in cement stone.

According to a paper by Lykov [1], a mutual relationship may be established between the average integrated values of the moisture content $\bar{u}$ and the temperature $\bar{t}$, on the one hand, and the intensities of heat and mass transfer q_h and q_m (and hence the rate of heat treatment or drying), on the other, in the form of a heat-balance equation, by using the laws of energy and mass conservation. The principal equation governing the kinetics of drying or heat treatment takes the form [1]

$$q_n(\tau) = \gamma_0 R_V r \frac{d\bar{u}}{d\tau}(1 + \mathrm{Rb}). \tag{1}$$

This equation establishes a relationship between the heat transfer q_h and the moisture transfer $d\bar{u}/d\tau$ by way of the Rebinder number Rb. The Rebinder number, derived by A. V. Lykov, is determined from the relation

$$\mathrm{Rb} = \left(\frac{d\bar{t}}{d\bar{u}}\right)\frac{c}{r} = \frac{bc}{r}, \tag{2}$$

where $b = d\bar{t}/d\bar{u}$ is the temperature coefficient of drying.

According to Eq. (1), for calculating the intensity of heat transfer in the course of drying or heat treatment it is required to know the manner in which the Rb number depends on the moisture content of the material $\bar{u}$.

We calculated the Rb number from the results of an experimental investigation into the kinetic characteristics of the heat treatment of cement mortars and concrete, this treatment being applied either by means of an alternating electromagnetic field or by steaming. The method and technique of the experiments were set out in [2, 3]. The samples were prepared from mortar and ordinary heavy concrete 200 × 200 × 60 mm in size and subjected to heat and moisture treatment in metal molds, which restricted their volume on five sides. The moisture evaporated from the upper surface, i.e., a one-dimensional problem of mass transfer was presented for consideration. Heat treatment was carried out in the following optimum mode, as determined by preliminary investigations: the temperature was raised at a rate of 15°C/h to a maximum of 80°C in 4 h, held at 80°C for 3 h, and then reduced at 13°C/h for 3 h. The relative humidity of the medium was kept at a level of $\varphi = 90\%$. In all the experiments the concrete and mortar were initially held at $t = 20°\mathrm{C}$ for 4 h.

For the calculations we used curves representing the time dependence of the layer-by-layer moisture content and temperature [3].

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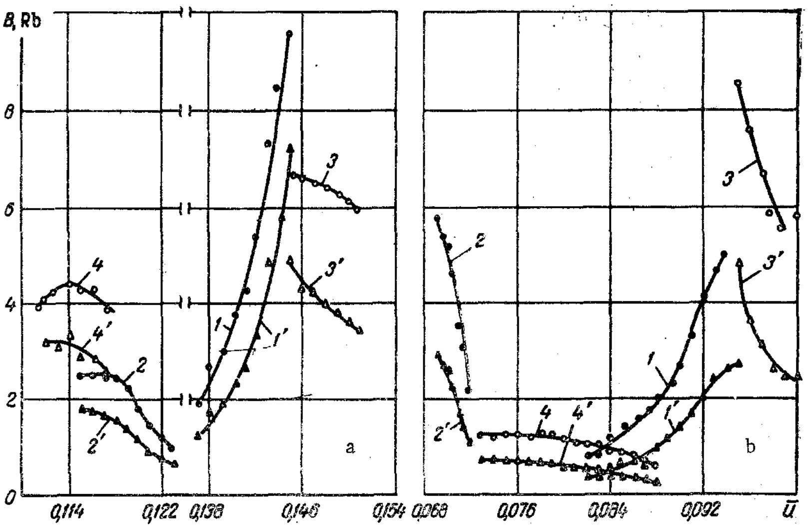

Fig. 1. The Rb number and coefficient B as functions of moisture content $\bar{u}$: a) cement mortar; b) concrete. During electromagnetic heat treatment: 1, 2) values of Rb in the periods corresponding to the heating of the material and the falling rate of moisture loss respectively; 1', 2') values of B in the same periods. During steam treatment; 3, 4) values of Rb in the periods corresponding to the heating of the material and the falling rate of moisture loss respectively; 3', 4') values of B in the same periods. $\bar{u}$, kg/kg.

The quantity Rb indicates the ratio of the amount of heat used in heating the sample to the amount of heat used in evaporating the moisture over an infinitely short period of time.

The value of the temperature coefficient of drying $b = d\bar{t}/d\bar{u}$ was calculated by A. G. Temkin's method, i.e., the discrete differentiation [4] of the $\bar{t}(\bar{u})$ curves during heat treatment. The values of the coefficients c and r (Tables 2 and 3) were determined from the equations [5]

$$c = c_0 + c_b\bar{u}, \tag{3}$$

$$r = 595 - 0.55\,(T - 273). \tag{4}$$

The values of the Rb numbers were calculated while heating the material and also in the period corresponding to the falling rate of moisture loss; in the period corresponding to the constant rate of moisture loss, Rb equalled zero.

The Rb number is shown in Fig. 1 as a function of the average integrated moisture content $\bar{u}$.

Analysis of the curves shows that, in the period during which the temperature of the mortar is rising (when the mortar is being hardened by heat treatment in an electromagnetic field) the value of the Rb number falls smoothly as the moisture content $\bar{u}$ diminishes (curve 1 in Fig. 1a). In the case of the steam treatment of mortar, the Rb number falls smoothly with increasing moisture content $\bar{u}$ over the same period (the process is accompanied by the soaking of moisture into the material) (curve 3 in Fig. 1a). The Rb number falls far more in absolute magnitude during this period for the electromagnetic mode of heat treatment than in the case of steaming. Here we may mention that, in electromagnetic heat treatment, rather less heat is spent in heating the material than in steaming. This is due to the difference in the mechanisms of heat and mass transfer, since in the first case the mass of material diminishes during heat treatment ($\bar{u} < \bar{u}_0$) while in the second it increases ($\bar{u} > \bar{u}_0$).

Analogous relationships are obtained in this period for concrete (curves 1 and 3, Fig. 1b). While the temperature is rising and the hardening material is in the form of a colloid, and capillary porosity is only beginning to develop, the osmotic form of moisture binding is predominant.

It follows from Fig. 1a that, in the period of falling temperature of the mortar (for both forms of heat supply), there is first of all a rise in the Rb number with decreasing moisture content and then a fall or stabilization of the Rb value (2 and 4, Fig. 1a). During this period, the development of the capillary porous structure is practically completed in the material, the moisture assumes an open capillary aspect, and

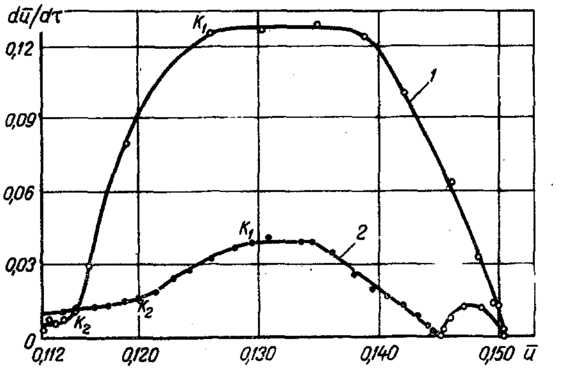

Fig. 2

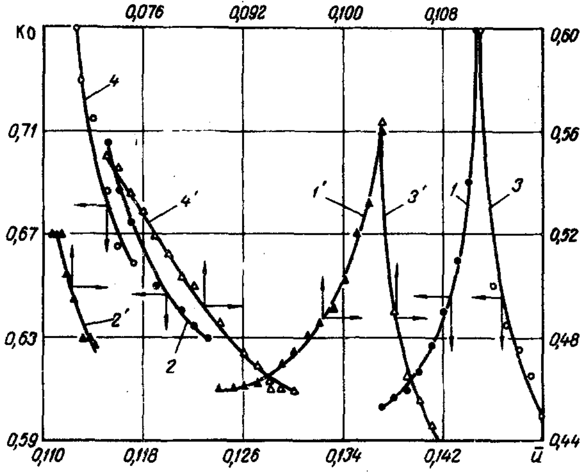

Fig. 3

Fig. 2. Curves giving the rate of moisture loss $d\bar{u}/d\tau = f(\bar{u})$ in the heat treatment of mortar: 1) steaming; 2) electromagnetic treatment. $d\bar{u}/d\tau$, kg/kg·h.

Fig. 3. Dependence of the Ko number on the moisture content $\bar{u}$ of mortar and concrete. For electromagnetic heat treatment: 1, 2) values of Ko in the heating period and the period of falling moisture loss rate for the mortar; 1', 2') the same for concrete. For steam treatment: 3, 4) values of Ko in the heating period and the period of falling moisture loss rate for mortar; 3', 4') the same for concrete.

meniscuses are formed, tending to pass down into the material as heat treatment proceeds. The moisture is chiefly displaced in the form of capillary moisture in this period.

The appearance of points of inflection on the $Rb(\bar{u})$ curves may be explained as being due to arrival at the boundary between the two basic states of the moisture in the capillaries: microcapillary and seam-type (pendular).

These conclusions are to a fair accuracy supported by the dispositions of the critical points (corresponding to the boundaries of the different forms of binding between the moisture and the solid phase of the mortar) on the $d\bar{u}/d\tau(\bar{u})$ curves in Fig. 2. As is apparent from Fig. 2, each of the curves passes through two critical points, K_1 and K_2. According to the fundamental principles of the doctrine relating to the forms of binding between moisture and material developed by Kazanskii [13], we may consider that the first critical point (K_1) on the $d\bar{u}/d\tau = f(\bar{u})$ curve corresponds to the instant at which the surface of the samples reaches hygroscopic moisture content. Between the first and second critical points (K_1 and K_2), the moisture associated with capillary forces is removed. Close to the second critical point, the moisture in the material passes into the pendular (seam) state, evaporating entirely within the material. The time at which the second critical point appears on the curves of moisture removal rate (Fig. 3) corresponds to the time of development of the bend on the $Rb(\bar{u})$ curves (Fig. 1a).

The bend appears far earlier on the $Rb(\bar{u})$ curve during electromagnetic heat treatment than it does in the course of steam treatment ($\bar{u} = 0.120$ kg/kg instead of 0.114 kg/kg); this evidently indicates the presence of a large amount of moisture with the adsorption type of binding in the cement stone. This in turn confirms the existence of a large internal specific surface of the capillaries in the cement stone, i.e., an improvement to the structure of the pore space by virtue of an increase in the volume of the microcapillaries ($r < 10^{-5}$ cm).

A distinguishing characteristic of the heat treatment of concrete by both methods of heat supply in the period of falling temperature is the transfer of moisture predominantly bound by capillary forces (curves 2 and 4, Fig. 1b). Thus for this mode of heat treatment (both in the case of the electromagnetic method of heat supply and in that of the steaming technique) no second critical point appears on the $d\bar{u}/d\tau(\bar{u})$ curves of concrete, nor are there any inflections on the $Rb(\bar{u})$ curves. This situation may be explained by considering the difference between the character and magnitude of the capillary porosity in mortar and concrete respectively.

The $Rb(\bar{u})$ relationships obtained for concrete and mortar in different periods of heat treatment were analyzed in the form of empirical formulas. For this purpose we plotted graphs in coordinates of log Rb and log $(\bar{u}-\bar{u}_p)$. The experimental points $[\log \mathrm{Rb}; \log(\bar{u}-\bar{u}_p)]$ then fell on straight lines. The equation of each of these lines may be written in the form

$$\lg \mathrm{Rb} = n \lg (\bar{u}-\bar{u}_p) + \lg A$$

or

$$\mathrm{Rb} = A(\bar{u}-\bar{u}_p)^n. \tag{5}$$

The constants A and n were found in the following way. The values of n were found from the tangent of the angle of inclination of each of the straight lines to the horizontal axis. The intercept cut off on the vertical axis by each line was numerically equal to logA. The values of A and n for various modes of heat treatment are given in Table 1.

The determination of the Rb number enables us to calculate the intensities of heat and mass transfer completely while the material is being heated and also in the period corresponding to the falling rate of moisture loss. The use of the Rb number for calculating the kinetics of the heat-treatment process is convenient because (as indicated by experimental data [6]) in the majority of cases it is independent of the parameters of the process and depends solely on the moisture content of the material $\bar{u}$.

The mutual relationship between the heat transfer $q_h(\tau)$ and the mass transfer $d\bar{u}/d\tau$ may be established for any method of heat treatment by using the Rb number or the B/Ko number from the fundamental equation of heat-treatment kinetics, which may be written in the form

$$q_n^*(\tau) = \left(\frac{d\bar{u}}{d\tau}\right)^* (1+\mathrm{Rb}) = \left(\frac{d\bar{u}}{d\tau}\right)^* \left(1+\frac{B}{\mathrm{Ko}}\right). \tag{6}$$

Hence the discovery of empirical formulas for Rb = f($\bar{u}$) and B = f($\bar{u}$) is of great interest not only for calculating the kinetics of the heat-treatment processes but also for the whole technology of heat treatment, since the fundamental technological properties of the material being processed are determined by its temperature and moisture content.

It is well known [6] that a relationship of the form

$$\mathrm{Ko} = B/\mathrm{Rb} \tag{7}$$

exists between the Ko and Rb numbers. The relative temperature coefficient of heat treatment B may be calculated [6] from

$$B = b\frac{\bar{u}_0}{T_\infty}, \tag{8}$$

where T_∞ equals T_{av} for the steaming treatment and the T of the cement mold and armature for the electromagnetic heat treatment.

These values of B, in conjunction with Eq. (7), enabled us to calculate the Ko number characterizing the ratio of the heat expended in evaporating moisture from the material to the heat required for raising the temperature from 0 to T_c.

TABLE 1. Coefficients of the Equation $\mathrm{Rb} = A(\bar{u}-\bar{u}_p)^n$

Material	Mode of heat treatment	Period of heat treatment	A	n
Cement mortar	B	b	8,67	0,125
		a	0,266	—1
	A	b	0,02	—0,84
		a	$0,653\cdot10^9$	5,05
Concrete	B	b	1,33	0,014
		a	$0,4\cdot10^{-2}$	—2,55
	A	b	0,1	—0,575
		a	$0,37\cdot10^7$	3,15

Note: Here and subsequently A signifies electromagnetic heat treatment, B steam treatment, a the period of heating the material, and b the period of falling moisture loss rate.

TABLE 2. Experimental and Calculated Kinetic Characteristics of the Heat Treatment of Mortar

Mode of heat treatment	Period of heat treatment	$\bar{u}$	$\bar{t}$	b	r	c	A	n	T_∞
B	b	0,117	80,0	6000	551,0	0,357	8,67	0,125	341,5
		0,116	76,0	6500	553,3	0,356			336,0
		0,115	68,0	6500	557,6	0,355			331,0
		0,114	61,0	6900	561,4	0,354			323,5
		0,113	55,0	6600	564,7	0,353			318,0
		0,112	48,0	6500	568,6	0,352			307,0
	a	0,145	20,0	—10000	584,0	0,385	0,266	—1	293
		0,146	31,0	—9900	578,0	0,386			332
		0,147	40,0	—9700	573,0	0,387			335,5
		0,148	50,0	—9300	567,5	0,388			338
		0,149	59,0	—9000	562,5	0,389			345,5
		0,150	68,0	—8700	557,6	0,390			342,5
		0,1505	76,0	—8500	553,3	0,3905			343,5
A	b	0,123	78,6	1500	551,8	0,363	0,02	—0,84	353
		0,122	77,7	1860	552,5	0,362			351
		0,121	75,6	2060	553,5	0,361			349
		0,120	73,0	2840	554,8	0,360			347
		0,119	70,0	3420	556,5	0,359			344
		0,118	66,0	3800	558,7	0,358			340,5
		0,117	62,0	4000	560,9	0,357			337
		0,116	58,0	4000	563,1	0,356			333
		0,115	54,0	4000	565,3	0,355			327
	a	0,145	18,0	14500	585,1	0,385	$0,653\times 10^{9}$	5,05	293
		0,144	35,0	—12600	575,8	0,384			315
		0,143	47,0	—10900	569,2	0,383			323,5
		0,142	56,0	—7900	564,2	0,382			333
		0,141	62,0	—6300	560,9	0,381			340
		0,140	67,0	—5420	558,2	0,380			344
		0,139	73,0	—4380	554,8	0,379			347
		0,138	77,6	—3940	552,3	0,387			350
		0,137	78,6	—2800	551,8	0,377			352

Expressed in terms of moisture content and temperature, the values of B and Ko are given in Figs. 1 and 3, respectively, for various methods of conveying the heat to the material.

It follows from Fig. 1 that the general run of the $B(\bar{u})$ curves is analogous to that of the corresponding $Rb(\bar{u})$ curves for both the mortar and the concrete. It is also interesting that the inflections on the $Rb(\bar{u})$ and $B(\bar{u})$ curves for the mortar coincide as regards moisture content in the period of falling temperature.

We see from Fig. 3 that in the period of rising temperature the values of the Ko number for both mortar and concrete fall with decreasing moisture content in the case of electromagnetic treatment and with increasing moisture content in the case of steam treatment. In the period of falling temperature, the relationship between Ko and $\bar{u}$ is the same for both modes of heat input, i.e., the Ko numbers rise with diminishing moisture content. However, it should be noted that the average values of the Ko number are considerably higher for the steam treatment than for electromagnetic heat treatment. This evidently indicates that in the latter case rather less heat is consumed than in steam treatment.

A knowledge of the $B = f(\bar{u})$ relation enables us to determine the temperature of cement materials at any specified instant of heat treatment, while the $Rb = f(\bar{u})$ relationship enables us to calculate the intensity of heat transfer during the whole heat-treatment process.

In the course of steam treatment, owing to the increase in the moisture content of the concrete and mortar samples, the excess internal pressure (which does not relax by virtue of the motion of the moisture) tends to increase. With increasing moisture content and excess internal pressure, a three-dimensional stressed state develops in the material, incorporating tensile tangential stresses; this distorts the structure of the material as it develops, leading to a loss of contact between the cement stone and the filler, and also to the formation of microcracks. In electromagnetic heat treatment the excess internal pressure is considerably lower. The moisture content gradient is also smaller than in the case of steam treatment. We should therefore expect less distortion of the reinforced concrete structures, both in the actual course of electromagnetic heat treatment and also after the completion of the latter; this conclusion is in agreement with experimental results.

TABLE 3. Experimental and Calculated Kinetic Characteristics of the Heat Treatment of Concrete

Mode of heat treatment	Period of heat treatment	$\overline{u}$	$\overline{t}$	b	r	c	A	n	T_∞
B	b	0,088	80,0	1200	551,0	0,328	1,33	0,014	351
		0,087	79,0	1340	551,6	0,327			340
		0,086	77,6	1420	552,3	0,326			346
		0,085	76,0	1600	555,2	0,325			344
		0,084	74,4	1600	554,0	0,324			341
		0,083	72,6	1740	555,0	0,323			338,5
		0,082	71,0	1760	559,9	0,322			336
		0,081	69,0	1900	557,1	0,321			335
		0,080	67,4	2070	558,0	0,320			331
		0,079	64,9	2150	559,3	0,319			328
		0,078	62,7	2230	560,6	0,318			325,5
		0,077	60,6	2130	561,6	0,317			323,0
		0,076	58,4	2160	562,8	0,316			320,5
		0,075	56,4	2160	564,0	0,315			318,0
		0,074	54,0	2160	565,3	0,314			315,5
		0,073	53,0	2200	566,4	0,313			313,0
	a	0,095	20,0	—15000	584,0	0,335	$0,4\times10^{-2}$	—2,55	293,0
		0,096	38,0	—12900	574,1	0,336			331,0
		0,097	50,0	—11300	567,5	0,337			345,5
		0,098	59,0	— 9800	562,6	0,338			347,0
		0,099	68,0	— 9300	557,6	0,339			349,0
		0,100	78,0	— 9500	552,1	0,340			353,5
A	b	0,072	78,0	4000	552,0	0,312	0,1	—0,575	352,0
		0,0715	77,0	5400	552,6	0,3115			350,0
		0,0710	74,0	6200	554,3	0,3110			346,5
		0,0705	70,0	8240	556,5	0,3105			343,5
		0,0700	66,0	9360	558,7	0,3100			340,0
		0,0695	60,4	9880	561,8	0,3095			334,5
		0,0690	55,4	10600	564,5	0,3090			328,6
	a	0,095	20,0	—8500	584,0	0,335	$0,37\times10^{7}$	3,15	293,0
		0,094	28,0	—8700	579,6	0,334			309,5
		0,093	37,0	—8200	574,6	0,333			317,0
		0,092	48,0	—7100	569,7	0,332			324,0
		0,091	52,0	—5800	566,4	0,331			329,5
		0,090	56,0	—4700	564,2	0,330			334,0
		0,089	61,0	—3900	561,4	0,329			338,0
		0,088	65,0	—3400	559,2	0,328			342,0
		0,087	67,0	—2900	558,1	0,327			345,5
		0,086	70,0	—2600	556,5	0,326			348,5
		0,085	73,0	—2380	554,8	0,325			351,0
		0,084	75,0	—1940	553,8	0,324			355,0
		0,083	76,4	—1640	553,0	0,323			353,5
		0,082	78,0	—1500	552,0	0,322			354,0

During the heat treatment of concrete, shrinkage takes place. A nonuniform distribution of the moisture content within the concrete (one involving high gradients) creates a three-dimensional stressed state within the material.

Hence, in addition to tensile and compressive stresses, cleaving (tangential) stresses also operate. Since in the case of moist solids, including hardening concrete, the limiting shear stresses at which rupture of the structure occurs are much smaller than the limiting normal stresses, the presence of the high tangential stresses constitutes a potent cause of crack formation.

As indicated in earlier papers [1, 8], the limiting tangential stress may, to a first approximation, be regarded as directly proportional to the moisture content gradient and the surface length of the material. As a criterion of crack formation we may take the fundamental criterion of the transfer of moisture during heat treatment – the Kirpichev mass-transfer number

$$\mathrm{Ki}_m = \frac{q_m(\tau) R_V}{a_m \gamma_0 \overline{u}_0}. \tag{9}$$

It is well known [9] that the Ki_m number characterizing the field of moisture content varies from 0 to 2 in the case of a parabolic distribution of moisture content in the material. The limiting value of Ki_m serves as an indicator of the limiting state as the structure of the material ruptures. It follows from Fig. 4 that the value of the Ki_m number increases at the onset of the concrete heating stage, its absolute increment being greater for concrete and mortar subjected to steam treatment than for those subjected to electromagnetic heat treatment. Starting from half an hour after the beginning of heat treatment, the Kirpichev

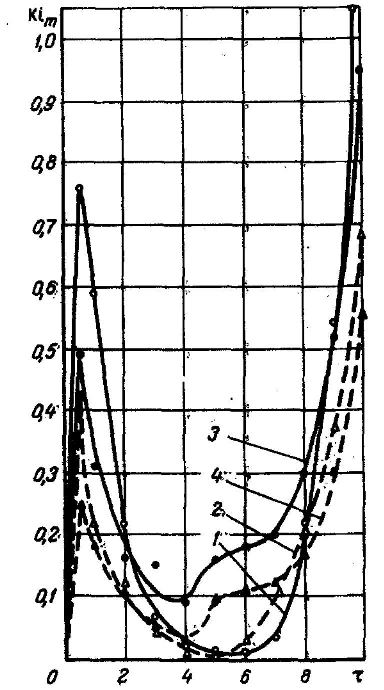

Fig. 4. Variation in the Ki_m number in relation to the kinetics of the heat-treatment process under optimum conditions: 1) steam treatment of concrete; 2) of mortar; 3) electromagnetic heat treatment of concrete; 4) of mortar. τ, h.

number diminishes until the beginning of the stage of isothermal treatment, independently of the form of the cement material and the method of heat treatment. At this stage Ki_m changes very little; for the steaming method its absolute value falls almost to zero for both concrete and mortar. This may be explained by supposing that, at the stage of isothermal treatment, with this method of heat treatment, there is a transition from the absorption of moisture to its evaporation from the material, since $\bar{u}$ approaches $\bar{u}_0$. At the beginning of the stage of falling temperature, the Ki_m number rises sharply, since, as cooling proceeds, the evaporation of the moisture intensifies, and so does the shrinkage of the concrete and mortar. The shrinkage stresses are added to the thermal stresses, and may produce cracks in the surface of the samples; this sharply reduces resistance to water penetration and frost. The sharpness of the rise in Ki_m and also its absolute magnitude are much greater for concrete and mortar subjected to steam treatment; this is because the gradient of the moisture content of the samples is greater than in the case of electromagnetic heat treatment. It follows from Fig. 4 that, in the course of steam treatment, the danger of crack formation in reinforced concrete structures is greater than it is in the course of electromagnetic heat treatment; this agrees with experiment [7].

The result of our investigations show that the greatest danger of the degradation of concrete as a result of crack formation arises during the rise and fall in temperature (Fig. 4); this agrees closely with the results of [10].

It is well known [1] that, the smaller the Ki_m number, the lower is the resistance to internal mass transfer. We see from Fig. 4 that, at the stage of rising temperature, the motion of the moisture in concrete and mortar hardening under electromagnetic heat treatment is much less than in the case of steam treatment. At the stage of isothermal treatment, when the structure of the material has already acquired strength sufficient to resist the stresses arising from the migration of moisture, the value of the Ki_m number is slightly higher for electromagnetic heat treatment. The migration of moisture is impeded in the latter case by the formation of a more compact structure, with an increased volume of microcapillaries and a closed porosity [12].

In the period of temperature drop, which is the most dangerous from the point of view of the probability of crack formation in concrete, the values of Ki_m are much greater for steaming than for electromagnetic heat treatment.

Thus the mechanism of moisture transfer has a considerable effect on the structure formation of cement stone, and depends on the mode of supplying the heat.

The criterial relationships thus obtained enable us to describe (to an accuracy sufficient for engineer's calculations) the kinetic characteristics of the heat treatment of cement materials in cases in which experimental data are available in relation to the coefficients of heat and moisture transfer, expressed as functions of the changes taking place in temperature $\bar{t}$ and moisture content $\bar{u}$ during the process.

NOTATION

q_h	is the heat flux density in kcal/m · h;
q_m	is the moisture flux density in kg/m · h;
q_h^*	is the ratio of heat flux in the period of falling moisture-loss rate (or the period of heating the material) to the heat flux in the period of constant moisture-loss rate;
$(d\bar{u}/d\tau)^* = (100/N)(d\bar{u}/d\tau)$	is the relative rate of heat treatment;
a_m	is the diffusion coefficient of moisture, m^2/h;
u_0	is the initial mean integrated moisture content of the material, kg/kg;
$\bar{t}$	is the mean integrated temperature of the material;
γ_0	is the density of the absolutely dry material, kg/m^3;
c	is the specific heat, kcal/kg · °C;
r	is the specific heat of vaporization, kcal/kg;
R_V	is the ratio of the volume of the body to its surface area;
K_1, K_2	are the arbitrary nomenclature for the first and second critical points respectively.

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