Physics of the Solid State, 2024, Vol. 66, No. 3

14,13

The influence of heat treatment on the dynamic strength characteristics of cement mortars

© L.A. Igusheva[1], Yu.V. Petrov[1,2]

[1] St. Petersburg State University,
St. Petersburg, Russia
[2] Institute for Problems in Mechanical Engineering of the Russian Academy of Sciences,
St. Petersburg, Russia
E-mail: igusheva15@gmail.com

Received February 7, 2024
Revised February 7, 2024
Accepted February 15, 2024

The dynamic fracture of pre-heat-treated cement mortars was analyzed from the point of view of a structural-temporal approach based on the incubation time concept. The rate dependences of fracture toughness and compressive strength of standard cement mortar and mortar with an admixture of barium sulfate were constructed, taking into account the heat treatment influence. Based on known experimental data, the values of incubation times were estimated. According to the theoretical calculation, the compressive strength inversion effect and the fracture toughness inversion effect were shown.

Keywords: mortar, incubation time, dynamic fracture toughness, dynamic compressive strength, heat treatment.

DOI: 10.61011/PSS.2024.03.57951.21

1. Introduction

Mortar is one of the main components of and the most often used materials for construction. Mortar is used in fabricating concrete, construction masonry mortar, plaster, screed and, together with reinforced concrete it is more than a half of materials produced by humans [1]. The physical and mechanical properties of the cement mortar directly affect safety of constructions, buildings and structures, so it is the most important task to study strength characteristics of the cement mortars.

Due to various external factors, both of man-made and natural ones, the buildings and structures are subjected to high temperatures. Thermal impact takes place in fires as well as during extraction of geothermal energy and burial of radioactive waste. Besides, the critical constructions (tunnels, mines, nuclear reactors) can be subjected to high-speed loads — explosions and impacts that are caused by accidents, terrorism acts, earthquakes, extracting works, i. e. joint impact of the temperature and impact loads. In this regard, mathematical models that can calculate values of dynamic strength and dynamic fracture toughness of construction materials are to be constructed in order to determine bearing strength of the constructions as well as to rebuild the constructions damaged by the high temperatures.

The experimental studies have shown that the strength characteristics of concretes and cement mortars usually decrease with increase in the pre-treatment temperature [2–5]. In case of static loads, compression strength and fracture toughness are material constants and they can be measured in standard experimental tests [6,7] Under the high-speed loads, the strength characteristics change depending on a type of external loads (the amplitude, the pulse form, the loading rate) [8,9]. Thus, it is not enough to use the classic strength theory criteria when calculating the dynamic strength and fracture toughness of the materials.

It is promising for describing a reaction of the materials to the dynamic loads to apply a structural-temporal approach, whose main tool is a incubation time fracture criterion [10]. This approach can take into account phenomena that are specific for the high-rate loads: dependence of the material strength on the parameters of external load [11] and the effect of fracture delay [12]. The structural-temporal approach was successfully used for predicting fracture of the materials subjected to various additional external effects, such as the temperature [13] and moisture saturation [14].

Based on the incubation time criterion, the present study has constructed the dependence of dynamic compressive strength on the loading rate and the dependence of dynamic fracture toughness on the rate of stress intensity factor change for a standard cement mortar and a cement mortar with an admixture of barium sulfate. The obtained results are verified by means of the known experimental data.

2. Incubation time criterion

The fracture process occurs in time, and unlike the classic strength theory, the incubation time criterion makes it possible to take it into account. Prior to material fracture at the macro-level, both for the high-rate loads and the static loads there are preliminary relaxation processes that take the lead over macro-fracture of the sample and are related to formation of damage at the microlevel. Duration of these processes is called the incubation time τ; it is a fundamental property of the material that characterizes the material reaction to the high-rate loads.

Generally, the incubation time criterion [15–18] is represented by the following condition:

$$\frac{1}{\tau}\int\limits_{t-\tau}^{t}\left(\frac{F(t')}{F_s}\right)^{\alpha}dt' \leq 1, \qquad (1)$$

where $F(t)$ — the intensity of the local force field; F_s — the limit of the local force field for the case of static loading; τ — the incubation time; α — the dimensionless parameter reflecting the material reaction to the value of force field strength(when reviewing the problem in elastic formulation $\alpha = 1$). At the point of time t_*, when the equality of the condition (1) is fulfilled, then there is fracture of the material.

This approach is characterized by its applicability both for the static loads and the dynamic loads; it allows describing material fracture for the entire range of the possible loading rates. For the case of static loading, the incubation time fracture criterion is turned into the classic criterion of the strength theory.

The incubation time criterion is applicable both for calculating the strength of initially defectless materials and for determining fracture toughness of the pre-cut samples.

3. Dynamic compressive strength

Let us study the dynamic compressive strength of two different-composition cement mortars based on the experimental data of the study [19]. The first cement mortar consists of sand, mortar, water and barium sulfate with the weight fractions 0.62, 0.25, 0.07 and 0.06, respectively, while the second one consists only of sand, mortar and water with the weight fractions 0.48, 0.37 and 0.15.

The standard cement mortar is widely used in the construction industry due to its low cost and use simplicity, whereas baryte concretes and mortars are radiation-protective materials [20–22]. The cement mortar and concrete with an admixture of barium sulfate are used in construction of special very important structures, such as nuclear power stations, nuclear reactors, military facilities. These materials are also recommended for use in construction of medical facilities — for protection of medical personnel and patients against radioactive radiation. The baryte cement mortars have high heat resistance and can be used at the high temperatures for a long time [20,23,24].

In order to determined the dynamic compressive strength, the study [19] uses the split Hopkinson pressure bar system. Before the tests, the cylindrical samples of the cement mortars are pre-heated to the temperature of 150, 250, 350, 450, 600 and 850°C, then the dynamic fracture experiments occur at the room temperature of 25°C. The experiment diagram for determining the dynamic compressive strength of the cylindrical sample of the cement mortar is shown on Figure 1, *a*. The experiments were conducted at the various loading rates.

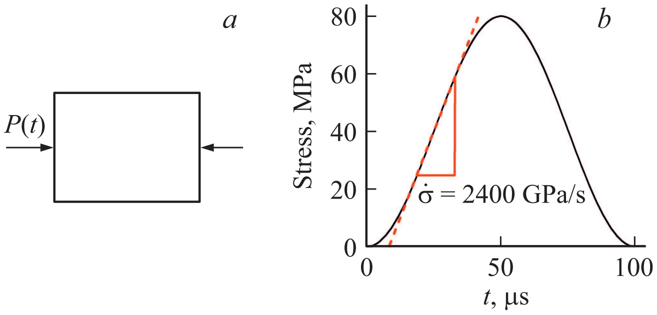

Figure 1. *a* — the experiment diagram for dynamic compressive strength of the cylindrical sample; *b* — the graph of dependence of stress on time in the experiment for the dynamic compressive strength, determination of the loading rate.

The incubation time criterion for determination of the material's compressive strength is written as follows

$$\frac{1}{\tau_\sigma}\int\limits_{t-\tau_\sigma}^{t}\sigma(s)ds \leq \sigma_s, \qquad (2)$$

where σ_s — the ultimate compressive strength for the case of static loading, τ_σ — compressive fracture incubation time. The fracture time t_* is determined as a point, in which the equality of the expression (2) is fulfilled.

In accordance with the data obtained in the study [19], the sample stresses linearly depend on the time at a certain loading interval; fracture occurs at the stress growth stage as well. Thus, for the calculations, the dependence of the stresses on the time is expressed by the following formula

$$\sigma(t) = \dot{\sigma}tH(t), \qquad (3)$$

where $\dot{\sigma}$ — the stress change rate, $H(t)$ — the Heaviside function.

The loading rate is equal to a tangential angle at the linear section of the graph of the dependence of the stress on time (Figure 1, *b*). By applying the incubation time criterion (2) for finding the dependences of the dynamic compressive strength σ_d on the stress increase rate $\dot{\sigma}$, we obtain the following expression:

$$\sigma_\mathrm{d}(\dot{\sigma}) = \begin{cases} \sigma_s + \dfrac{\tau_\sigma}{2}\dot{\sigma}, & \dfrac{\sigma_s}{\dot{\sigma}} \geq \dfrac{\tau_\sigma}{2}, \\ \sqrt{2\sigma_s\tau_\sigma\dot{\sigma}}, & \dfrac{\sigma_s}{\dot{\sigma}} < \dfrac{\tau_\sigma}{2}. \end{cases} \qquad (4)$$

In order to construct the theoretical curves (4), it is necessary to determine two material constants: the static compressive strength σ_s and the value of the incubation time τ_σ. The values σ_s are given in the study [19], τ_σ is determined by the least-square method as per the experimental data [19]. Table 1 shows the values of σ_s and τ_σ.

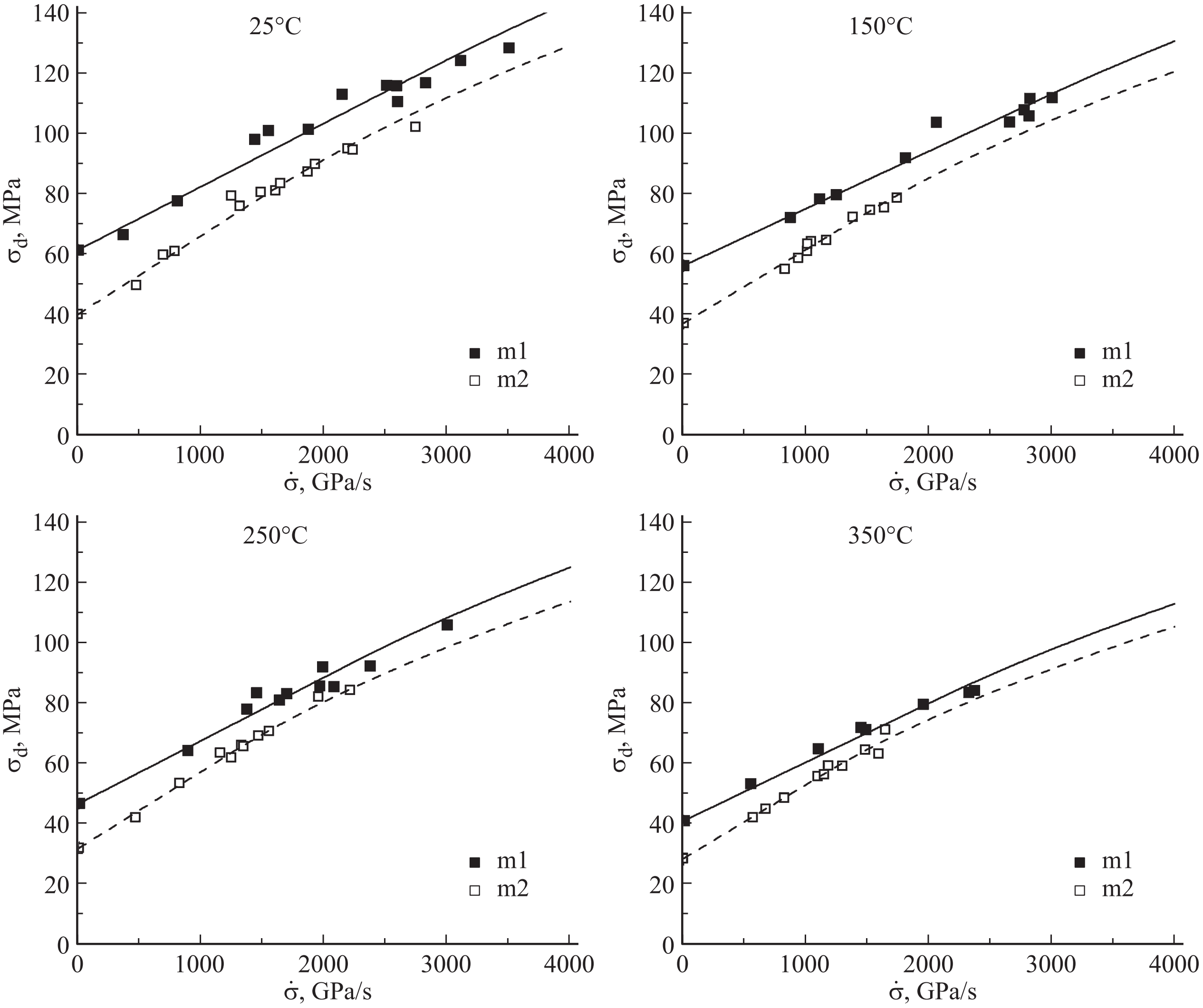

Figure 2. Rate dependences of the dynamic compressive strength for the cement mortars at the temperatures 25–350°C (the solid lines — calculation for the baryte cement mortar, the dashed curves — calculation for the standard cement mortar), the experimental data [19] (m1 — the baryte cement mortar, m2 — the standard cement mortar).

Table 1. Summary table of the values of the static ultimate compressive strength and the fracture incubation time for the cement mortars (m1 — the baryte cement mortar, m2 — the standard cement mortar)

	T, °C	25	150	250	350	450	600	850
m1	τ_σ, μs	42	38	42	39	44	41	62
	σ_s, MPa	61.41	56.19	46.34	40.55	32.44	25.49	7.72
m2	τ_σ, μs	52	49	51	49	51	45	69
	σ_s, MPa	40.12	37.12	31.56	28.1	22.09	18.78	6.46

The Figures 2 and 3 show the dependences of the compressive strength for the two cement mortars on the stress change rate at the various pre-treatment temperatures. The theoretical curves (4) qualitatively correspond to the experimental results described in the study [19]. The obtained results show that with increase in the pre-treatment temperature there is evidently decrease in the compressive strength for both the cement mortars. For all the temperatures, the compressive strength increases with increase in the loading rate. The baryte cement mortar has a higher compressive strength in comparison with the standard cement mortar at the same pre-treatment temperatures, wherein the higher pre-treatment temperature, the smaller the compressive strength difference between the two cement mortars.

When comparing the two cement mortars, there is evidently an compressive strength inversion effect (Figure 4). At the different pre-treatement temperatures the baryte cement mortar has a higher compressive strength under external loads with the small loading rate. But, it becomes less strong under the dynamic loads comparison with the standard cement mortar. A point of intersection of the compressive strength's rate curves of Figure 4

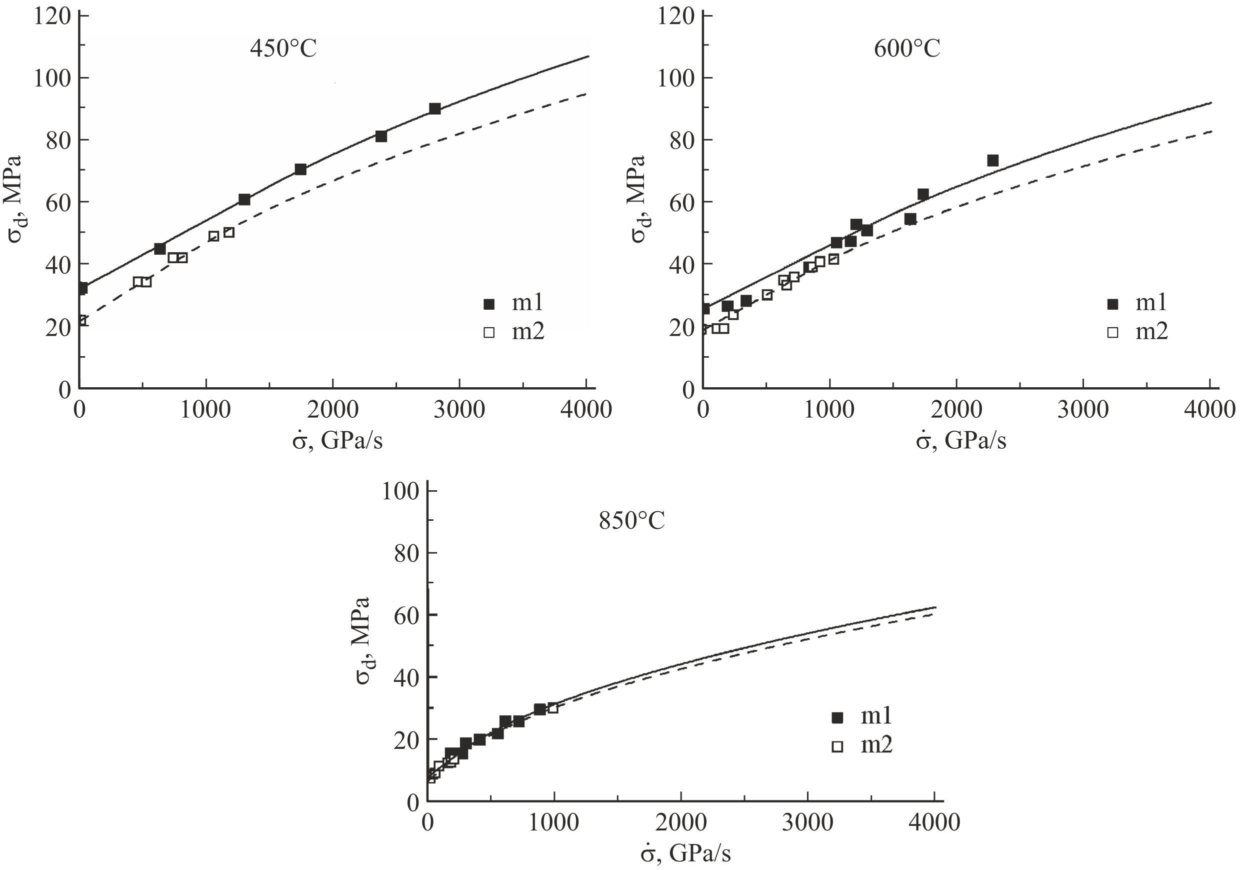

Figure 3. Rate dependences of the dynamic compressive strength for the cement mortars at the temperatures 450−850°C (the solid lines — calculation for the baryte cement mortar, the dashed curves — calculation for the standard cement mortar), the experimental data [19] (m1 — the baryte cement mortar, m2 — the standard cement mortar).

Table 2. Comparative characteristic of the compressive strength for the two cement mortars (m1 — the baryte cement mortar, m2 — the standard cement mortar)

Cement mortar having the higher compressive strength at slow loads; treatment temperature, °C	Cement mortar having the higher compressive strength at high-rate load; treatment temperature, °C	Loading rate, at which there is inversion of compressive strength, GPa/s
m1; 250	m2; 25	1216
m1; 350	m2; 25	67
m1; 350	m2; 150	714
m1; 350	m2; 250	1481
m1; 450	m2; 250	233
m1; 600	m2; 450	667

corresponds to the transitional loading rate, at which there is inversion of the compressive strength. Table 2 shows the comparative characteristic of the strength properties of the two cement mortars processed at the various temperatures.

4. Dynamic fracture toughness

Let us study the effect of impact of the heat pretreatment on the dynamic fracture toughness for the two cement mortars (with the admixture of barium sulfate and with no admixture) using the experimental data [25]. The compositions of the cement mortars are the same as specified in the previous section. Using the split Hopkinson pressure bar system, the study [25], has tested the semi-circular notched samples for three-point bending (Figure 5, *a*) in order to determine the dynamic fracture toughness. The samples are pre-heated to the temperatures of 150, 250, 350, 450 and 600°C to be followed by

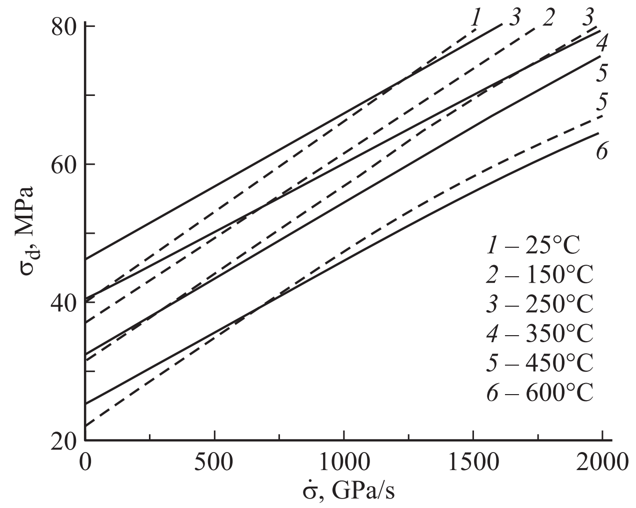

Figure 4. Inversion of the curves of dynamic compressive strength for the cement mortars (the solid lines — the baryte cement mortar, the dashed lines — the standard cement mortar).

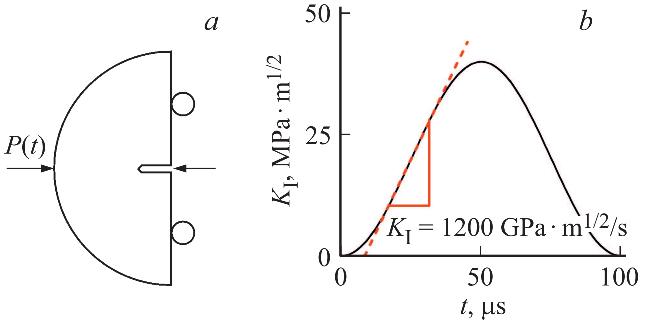

Figure 5. *a* — The experiment diagram for a three-point bending of a semi-circular notched sample, *b* — determination of the stress intensity rate.

the tests at the temperature of 25°C. In the experiment, fracture occurs as per the mode I: the crack banks move perpendicular to a plane of cracking in the sample.

The dynamic fracture toughness of the cement mortars is calculated using the incubation time criterion, which is presented by the following condition:

$$\frac{1}{\tau_K}\int_{t-\tau_K}^{t} K_I(t')dt' \le K_{Is}, \tag{5}$$

where K_{Is} — the ultimate value of the stress intensity factor of the fracture mode I for the case of static loading, τ_K — the fracture incubation time corresponding to the condition for determination of the fracture toughness. At the point of time when the equation of the expression (5) is fulfilled, the sample crack starts propagating.

The study [25] specifies the dynamic fracture toughness as a maximum value of the stress intensity factor in the sample for the entire loading history. The rate of stress intensity factor change is equal to the tangential angle at the linear section of the graph of the dependence of the stress intensity factor on time (Figure 5, *b*). The study [25] has shown that there is a time interval, during which the loading force changes by a the linear law, so for the theoretical curves it is believed that the dependence of the force on time is expressed as follows: $P(t) = \dot{P}tH(t)$, where $\dot{P}$ — the force increase rate, $H(t)$ — the Heaviside function.

Table 3. Summary table of the values of the static ultimate stress intensity factor and the fracture incubation time for the cement mortars (m1 — the baryte cement mortar, m2 — the standard cement mortar)

	T, °C	25	150	250	350	450	600
m1	τ_K, μs K_{Is}, MPa · m$^{1/2}$	94 1.11	93 0.94	116 0.78	126 0.67	117 0.48	128 0.36
m2	τ_K, μs K_{Is}, MPa · m$^{1/2}$	95 0.92	106 0.7	103 0.6	125 0.51	134 0.4	153 0.23

In the experiments for three-point bending of the semi-circular notched sample, the dependence of the stress intensity factor on the external force is presented as follows [26]:

$$K_I(t) = \frac{P(t)S}{BR^{3/2}} Y\left(\frac{a}{R}\right), \tag{6}$$

where $P(t)$ — the external force, R — the sample radius, B — the sample thickness, S — the distance between two support pins, $Y(a/R)$ — the dimensionless function depending on the sample geometry, which can be calculated by means of the software application package (for example, ANSYS) [26,27]. Thus, the stress intensity factor linearly depends on the external force acting on the sample [28]. Inserting the formula for force expression into (6), we obtain the following expression for determining the stress intensity factor

$$K_I(t) = \dot{K}_I tH(t), \tag{7}$$

where $\dot{K}_I$ — the rate of stress intensity factor change.

Inserting the expression (7) into the incubation time criterion (5), we obtain the formula for determining the dynamic stress intensity factor K_{Id} depending on the rate of stress intensity factor change $\dot{K}_I$:

$$K_{Id}(\dot{K}_I) = \begin{cases} K_{Is} + \dfrac{\tau_K}{2}\dot{K}_I, & \dfrac{K_{Is}}{\dot{K}_I} \ge \dfrac{\tau_K}{2}, \\ \sqrt{2K_{Is}\tau_K\dot{K}_I}, & \dfrac{K_{Is}}{\dot{K}_I} < \dfrac{\tau_K}{2}. \end{cases} \tag{8}$$

Figure 6 shows comparison of the theoretical calculation of dynamic fracture toughness for the baryte cement mortar and the cement mortar with no admixture (8) with the experimental results of the study [25]. The incubation time is determined by the least-square method as per the experimental data, the static ultimate stress intensity factor is determined in the study [25] (Table 3).

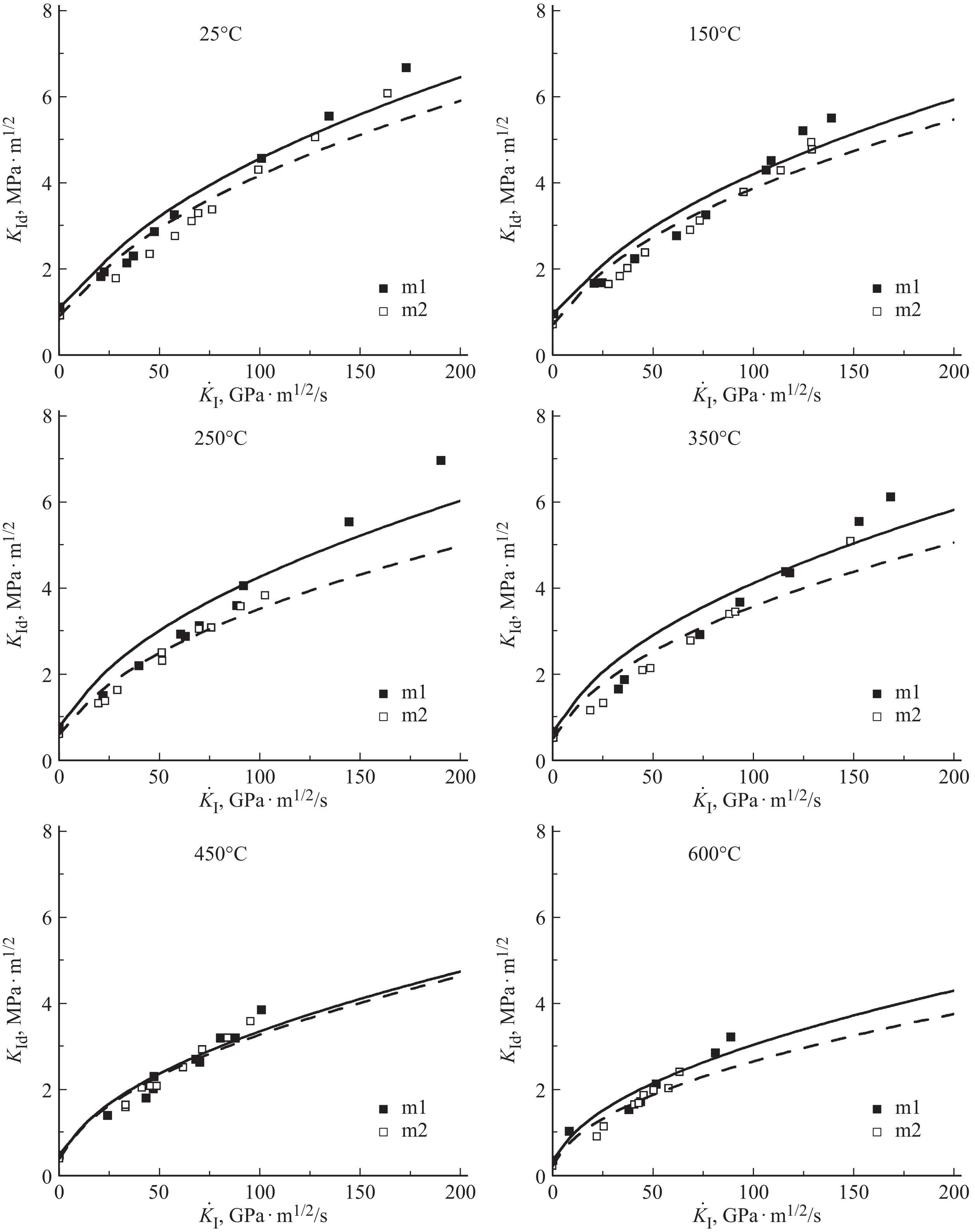

Figure 6. Rate dependences of the dynamic fracture toughness for the cement mortars at the various temperatures (the solid line — calculation for the baryte cement mortar, the dashed curves — calculation for the standard cement mortar), the experimental data [25] (m1 — the baryte cement mortar, m2 — the standard cement mortar).

Table 4. Comparative characteristic of fracture toughness for the cement mortars (m1 — the baryte cement mortar, m2 — the standard cement mortar)

Cement mortar having the higher fracture toughness at slow loads; treatment temperature, °C	Cement mortar having the higher fracture toughness high-rate loads; treatment temperature, °C	Loading rate, at which there is inversion of fracture toughness, GPa · $m^{1/2}$/s
m1; 150	m1; 250	14.8
m2; 25	m1; 250	13.3
m2; 150	m1; 350	3.1
m2; 250	m2; 350	8.4

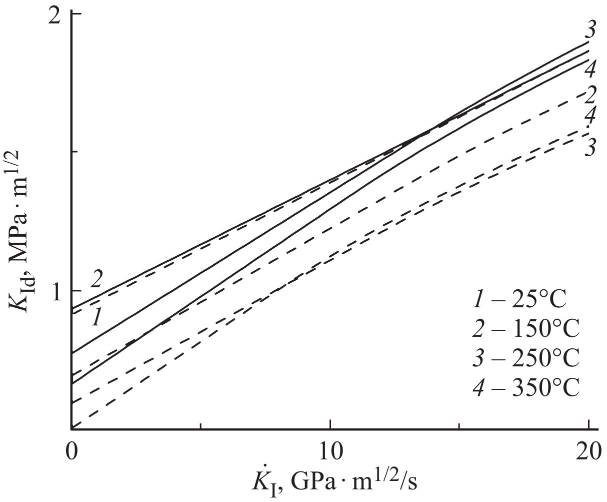

Figure 7. Inversion of the curves of dynamic fracture toughness for the cement mortars (the solid lines the baryte cement mortar, the dashed lines — the standard cement mortar).

The obtained calculated data qualitatively describe tendencies of fracture toughness change when varying the loading rate as well as allow taking into account impact of the heat pre-treatment. Bot the cement mortars exhibit a similar reaction to increase in the loading rate and increase in the heat pre-treatment. The dynamic fracture toughness increases with increase in the loading rate with all the treatment temperatures. The dynamic fracture toughness for the baryte cement mortar is higher in comparison with the cement mortar with no admixture at the same values of heat treatment for all the values of the loading rate, wherein the higher loading rate, the more significant difference between the values of the dynamic fracture toughness. With increase in the value of the pre-heat temperature for the cement mortars, there is evidently reduction of the fracture toughness for both the cement mortars.

In the case under study, there is also evidently an effect of inversion of the fracture toughness (Figure 7) when comparing the characteristics of the cement mortars that are pre-heated to the various temperatures. Similar to the compressive strength, the fracture toughness for one cement mortar treated at one temperature is higher that for the second cement mortar treated at the other temperature, at slow loads; at the same time for the dynamic loads the higher fracture toughness is exhibited by the second cement mortar. The point of intersection of the rate dependences of fracture toughness is a point of inversion of fracture toughness. The summary table for comparison of the dynamic and the static fracture toughness for the cement mortars treated at the various temperatures is given in Table 4.

It should be noted that the same cement mortar treated at the different temperatures exhibits the fracture toughness inversion effect. For example, at the rate of stress intensity factor change below 14.8 GPa · $m^{1/2}$/s, the higher fracture toughness belongs to the baryte cement mortar treated at the temperature of 150°C, in comparison with the baryte cement mortar treated at the temperature of 250°C. When the rate of stress intensity factor change is above 14.8 GPa · $m^{1/2}$/s, the higher fracture toughness corresponds to the baryte cement mortar treated at the temperature of 250°C.

5. Comparison of the incubation times

Tables 1 and 3 show the incubation time dependence on the pre-treatment temperature for the two cement mortars. It turns out that the incubation time corresponding to the condition for determination of the fracture toughness τ_K is approximately in two times higher that the incubation time corresponding to the condition for determination of the compressive strength τ_σ — both for the baryte cement mortar and the cement mortar with no admixture. On average, the incubation time both for the compressive strength tests and the tests for determination of fracture toughness for the cement mortar with the admixture of barium sulfate is below the fracture incubation time for the cement mortar with no admixture at the same treatment temperatures. The dependences of the fracture incubation time on the pre-heating temperature are of a similar nature for the cement with the admixture of barium sulfate and for the standard cement mortar. During heat treatment of both the cement mortars within the temperature range from 150 to 600°C, the incubation time corresponding to the condition for determination of the compressive strength τ_σ is almost unchanged, and its value insignificantly differs from the value of the incubation time of the heat-untreated cement mortar. When heating the samples to the temperature of 850°C, the value of the fracture incubation time τ_σ rapidly increases for both the cement mortars. On average, the fracture incubation time corresponding to the condition for determination of the dynamic fracture toughness τ_K increases with increase in the temperature.

6. Conclusion

The study has investigated the dynamic compressive strength and the dynamic fracture toughness for the cement mortar and the cement mortar with no admixture, which were heat-treated. The material fracture has been described using the incubation time fracture criterion. It has been shown that this approach allows qualitatively describing material behavior both at the high-rate and the static loads.

The dynamic compressive strength and the fracture toughness for the baryte cement mortar is higher than for the cement mortar with no admixture for all the values of heat treatment. With increase in the temperature, the strength properties of both the cement mortars decrease. With increase in the loading rate, the values of the dynamic strength and the fracture toughness for the heat-treated cement mortars increase.

The compressive strength and fracture toughness for the two cement mortars treated at the different temperatures have been compared; it has been shown that depending on the loading rate one cement mortar can be stronger at the quasi-static loads and at the same time less strong at the impact loads.

It has been found that pre-heating of the samples to the temperatures below 600°C had almost no influence on the incubation time value, which corresponds to the condition for determination of the compressive strength for the cement mortars. But, with further heating of the samples, there is evidently fast FIT increase. Whereas, on average, the incubation time corresponding to the condition for determination of the fraction toughness increases with increase in the temperature.

Based on the obtained results, it can be concluded that the structural-temporal approach can predict fracture of the cement mortars within a wide range of the fast effects taking into account influence of heat pre-treatment, relying on a minimum set of the material characteristics: time, the static compressive strength and the ultimate value of the stress intensity factor for the case of static loading.

Funding

The study was carried out with the support of the Ministry of Science and Higher Education of the Russian Federation (Agreement No. 075-15-2022-1114 dated June 30, 2022). The sections 2, 3, 5 were carried out by Yu.V. Petrov as supported by the Russian Science Foundation (No. 22-11-00091).

Conflict of interest

The authors declare that they have no conflict of interest.

References

[1] K.L. Scrivener. Indian Concr. J. **88**, *7*, 11 (2014).
[2] M.S. Cülfik, T. Özturan. Cem. Concr. Res. **32**, *5*, 809 (2002).
[3] J. Xiao, G. König. Fire Saf. J. **39**, *1*, 89 (2004).
[4] H.L. Malhotra. Mag. Concr. Res. **8**, *23*, 85 (1956).
[5] S.Y.N. Chan, G.-F. Peng, J.K.W. Chan. Mater. Struct. **29**, *10*, 616 (1996).
[6] Y. Zhou, J. Gao, Z. Sun, W. Qu. Constr. Build. Mater. **98**, 137 (2015).
[7] C.H. Popelar, C.E. Anderson Jr, A. Nagy. Exp. Mech. **40**, *4*, 401 (2000).
[8] R. Cai, J. Mao, H. Qi, J. Qu, Q. Guo. Theor. Appl. Fract. Mech. **128**, 104098 (2023).
[9] Z.P. Bazant, R. Gettu. ACI Mater. J. **89**, *5*, 456 (1992).
[10] Y.V. Petrov, B.L. Karihaloo, V.V. Bratov, A.M. Bragov. Int. J. Eng. Sci. **61**, 3 (2012).
[11] H. Qiu, Z. Zhu, M. Wang, F. Wang, Y. Ma, L. Lang, P. Ying. Eng. Fract. Mech. **228**, 106798 (2020).
[12] N.V. Mikhailova, I.V. Smirnov, V.V. Balandin, V.Vl. Balandin, A.M. Bragov, Y.V. Petrov. Int. J. Impact Eng. **164**, 104194 (2022).
[13] N.F. Morozov, Y.V. Petrov. Eur. J. Mech. A **25**, *4*, 670 (2006).
[14] N.S. Selyutina, Y.V. Petrov. Eng. Fract. Mech. **225**, 106265 (2020).
[15] Yu.V. Petrov. Dokl. AN SSSR **321**, *1*, 66 (1991) (in Russian).
[16] Y.V. Petrov. Phys. Solid State **38**, *11*, 1876 (1996).
[17] Y.V. Petrov, A.A. Utkin. Mater. Sci. **25**, *2*, 153 (1989).
[18] Y.V. Petrov, N.F. Morozov. J. Appl. Mech. **61**, *3*, 710 (1994).
[19] W. Yao, H.-W. Liu, Y. Xu, K. Xia, J. Zhu. Constr. Build. Mater. **136**, 139 (2017).
[20] K. Sakr, E. El-Hakim. Cem. Concr. Res. **35**, *3*, 590 (2005).
[21] T.-C. Ling, C.-S. Poon, W.-S. Lam, T.-P. Chan, K.K.-L. Fung. Mater. Struct. **46**, *7*, 1133 (2013).
[22] H. Binici. Int. J. Mater. Res. **101**, *8*, 1052 (2010).
[23] M.C.R. Farage, J. Sercombe, C. Gallé. Cem. Concr. Res. **33**, *7*, 1047 (2003).
[24] B.M. Luccioni, M.I. Figueroa, R.F. Danesi. Eng. Struct. **25**, *6*, 729 (2003).
[25] W. Yao, Y. Xu, H.W. Liu, K. Xia. Eng. Fract. Mech. **169**, 74 (2017).
[26] Y.X. Zhou, K. Xia, X.B. Li, H.B. Li, G.W. Ma, J. Zhao, Z.L. Zhou, F. Dai. Int. J. Rock Mech. Min. Sci. **49**, 105 (2012).
[27] F. Dai, R. Chen, K. Xia. Exp. Mech. **50**, *6*, 783 (2010).
[28] I.L. Lim, I.W. Johnston, S.K. Choi. Eng. Fract. Mech. **44**, *3*, 363 (1993).

Translated by EgoTranslating