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The influence of fire temperatures on cement composites

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ABSTRACT : *The paper presents the issues of the influence of fire temperatures on cement composites: cement paste, mortar and concrete. The composites did not contain any additives or admixtures. The composites were tested, among others, for compressive strength, tension by bending and splitting, as well as fracture toughness and their critical stress intensity factor K_{IC} was determining. The tests were carried out at a normal temperature of 20^oC and after application of temperatures of 500, 600 and 700^oC. The essence of the paper are relationships of to fracture toughness of studied composites after application of fire temperatures.*

KEYWORDS: *fire temperatures, cement composites, fracture toughness, stress intensity factor.*

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I. INTRODUCTION

On the basis of national and foreign literature it can be said that there are not many studies, especially in the Polish language, on the effects of high temperatures on the fracture toughness of concrete, of paste and mortar. Researchers have dealt mainly with the influence of high temperatures on the strength of concrete in the context of changes in its physical, chemical, mechanical and structural features as well as its phase composition, while there are few results of research, especially on the national scale, on the influence of high temperatures on the stress intensity factor K_{IC} of concrete, paste and mortar. Own research showed that destruction created during the initial hardening of paste, mortar and concrete before and during the exploitation, such as pores, technological cracks and other damage, develops under the influence of fire temperature and determine the physical and mechanical properties of paste, mortar and concrete, while their size can be assessed by means of the stress intensity factor K_{IC}.

II. TRANSFORMATIONS OF CEMENT COMPOSITES UNDER THE INFLUENCE OF FIRE TEMPERATURES

Transformations that occur in concrete with increase of fire temperatures are described, among others in [1],[2],[3]. They can be quite accurately determined in individual temperature ranges:

- 105°C - evaporation of free water. Beginning of ettringite dehydration and its reduction in the matrix,
- 120 - 163°C - decomposition of gypsum and its content drop in the cement matrix.
- 180°C - total water reduction - increase in porosity and microcracks. Dehydration of ettringite and hydrated calcium silicates CSH,
- 190 ÷ 300°C - sudden breakout at the edges and scaling of the concrete surface (spalling),
- 300°C - end of ettringite dehydration and further dehydration of CSH,
- 300°C - beginning of decrease in strength, oxidation of iron compounds,
- from 500°C - dehydroxylation of calcium hydroxide (portlandite) that passes into free lime with the possibility of rebinding: $Ca(OH)_2 \rightarrow CaO + H_2O$ and reduction of chemically bound water, followed by rehydration of CaO (from atmospheric moisture or water poured over concrete while extinguishing the fire [4]): $CaO + H_2O \rightarrow Ca(OH)_2$.
- 600 ÷ 700°C - due to the above reaction and due to the decomposition of calcium carbonate ($CaCO_3 \rightarrow CaO + CO_2$) there is a large increase in the volume of calcium compounds (about 40%), which causes increased cracking of the concrete and a significant decrease in its strength. Beginnings and development of cracks and weakening of bonds between aggregate and paste in concrete, possible beginnings of degradation. Increase in porosity of the paste. Concrete becomes unsuitable for construction after heating at temperatures of 550-600°C - strength drops above 50%, and for temperatures of 800°C approx. 80%

- 573°C - phase transformation of quartz from the β low temperature variant to the α high temperature variant; the process of structure reconstruction takes place with intensive enlargement of concrete volume (on aggregate with silicon content) and is the main factor of its low strength during high temperatures operation'
- 100 ÷ 800°C - dehydration of CSH cement matrix components,
- 350 ÷ 900^{o}C - transformations occurring in the aggregate: volume changes, phase transitions and chemical decomposition reactions,
- 700 ÷ 800°C - decomposition of $CaCO_3$ (calcium carbonate) limestone aggregates,
- 1200 ÷ 1350°C - melting (softening) of concrete.

The process of changing the strength of concrete depending on the temperature results, among others, from its composition (type of aggregate, w/c ratio, presence of additives, etc.), but also depends on the speed of heating and the duration of action of temperatures. Gradual degradation of concrete is the result of physico-chemical changes taking place in concrete at fire temperatures.

The influence of high fire temperatures on the basic characteristics of concrete is very important. Its compressive and tensile strength (which determines cracking and fracture) as well as the elastic modulus (also the dynamic module) are reduced, whereas the deformations increase, often to the point of destruction of the structure.

The main reason for the drop in the strength of concrete at high temperatures is the reverse orientation of the paste and aggregate deformation, which causes a weakening of adhesion between them.

III. OWN RESEARCH

In fire conditions, heat penetrates slowly into the cement composites, but as a result of the slow pace of its course, there are significant differences in temperature in the concrete, paste and mortar between the surface of the element and its interior.

The effect of temperature gradients is the degradation of the material, mainly defined by the reduction in compressive strength f_c and tensile $f_{ct,sp}$, but also, as our own research [5] indicates, a decrease in fracture toughness expressed in the critical of stress intensity factor K_{IC}.

In our own studies, intermediate temperatures between 20^{o}C and 500^{o}C were omitted. It was done so consciously, because in the literature one can find descriptions of concrete tests in the temperature ranges of up to 450^{o}C. Therefore, in our own research, there remains a "empty space", which must be remembered, because in it the strength of the concrete may rise to a certain value, and then decrease with further temperature increase [10]. However, not all researchers confirm this temporary increase in strength at elevated temperatures and record a decrease in the compressive strength of concrete from the beginning of the rise in temperatures in the case of conventional concretes, eg [2], [6].

Therefore, in our study the temperature of min. 500°C is considered fire temperature, also as a reference to the standard isothermal method 500.

3.1 Description of the tested samples

The test specimens made of the concrete contained the same amount of coarse aggregate with variable w/c ratios, making it possible to determine the effect of w/c and the created structure on the K_{IC} factor and other strength parameters of materials. Concretes were made with ratios of w/c = 0,40; 0,50 and 0,60. No additives or admixtures were used.

The compressive strength of the concrete was determined on cubic samples with a side of 150 mm. The stress intensity factor was tested on beam-samples recommended by RILEM [[7]] with dimensions of $L \times b \times W$ = 700 × 80 × 150 mm, a span (spacing of supports) S = 600 mm, with a primary notch with a length of a = 50 mm and a width of 3 mm with a bevel of 1,5/2,6 = 0,577. The proportions of the dimensions of the samples were: $S = 4W$; b = about $0,5W$; $a = 0,333W$.

At the same time, there were made paste and cement mortar beam-samples with dimensions $L \times b \times W$= 160×40×40 mm. Spacing of supports S = 100 mm was adopted in accordance with the rules for testing the strength of bending beams [[8]]. In the beams intended for testing the stress intensity factor, notches were made through cuts with a diamond circular saw due to the impossibility of making notches, as in concrete samples. The length of the primary notch a = 13.3 mm and the width 3 mm. Differences observed in the fracture toughness of samples with notches cut by saws and contoured during their concreting are negligible [[9]].

The strength stretching when bending and compressive strength of the paste and cement mortar were tested on the beams without notches.

3.2 Components of concrete, pastes and mortars

The samples were made of cement concrete using Portland cement with high early strength CEM I 42.5 R, with gravel aggregate with fractions 2 ÷ 8 and 8 ÷ 16 mm and with sand (0 ÷ 2 mm). The composition of concrete mixes is presented in Tab. 1.

While calculating the contents of cement paste and mortar there is assumed the principle of preserving the proportions of concrete mix components. Such a criterion allows to make comparisons of the results of study of concrete, paste and mortar samples as the contents of paste and mortar are in a way a derivative of concrete composition.

Tab. 1. Contents of concrete mixes.

w/c ratio	Unit	0,4	0,5	0,6
Cement CEM I 42,5 R	kg/m^3	320	320	320
Sand (0 - 2 mm)	kg/m^3	652	625	598
Coarse aggregate gravel (2-8 mm)	kg/m^3	531	531	531
Coarse aggregate gravel (8-16 mm)	kg/m^3	797	797	797
Water	dm^3/m^3	128	160	192

The content of cement paste and mortar calculated on the basis of the percentage of their components in a concrete mix is presented in Tab. 2 and Tab. 3.

Tab. 2. Contents of cement pastes.

w/c ratio	0,4	0,5	0,6
Cement CEM I 42,5 R kg/m^3	1734	1622	1524
Water dm^3/m^3	694	811	914

Tab. 3. Contents of cement mortars.

w/c ratio	0,4	0,5	0,6
Cement CEM I 42,5 R kg/m^3	706	705	703
Sand (0 - 2 mm) kg/m^3	1439	1376	1313
Water dm^3/m^3	283	352	422

3.3 The course of research

Beam-samples intended for testing at fire temperatures, both concrete and paste and mortar, were heated in a chamber furnace at temperatures of 500, 600 and 700°C. The rate of sample heating to the set temperature was 4 ÷ 6°C/min. The rate of sample heating was comparable to the values reported in the literature (eg [[10]], [[11]]). For this reason, as well as descriptions of the occurrence of explosive phenomena of heated concrete samples [[12]], it was not decided to heat them according to the norm, standard time-temperature heating curve [[13]], according to which the initial rate of temperature increase is too high (115°C)/min) and may cause explosion of samples and destruction of the furnace. The slower pace of heating is also justified by the fact that in many practical design cases (eg: auditoria, sports halls or other facilities with low fire load), the use of a nominal curve is not economically justified [[14]].

It should be added here that the RILEM recommendations [[15]] entitled "Test methods for mechanical properties of concrete at high temperatures" determine the heating and cooling speed of samples at temperatures of 20 ÷ 200°C at a rate of only 0,1°C/min, and in emergency conditions, in the temperature ranges of 200°C ÷ 750°C from 0,5 to 2°C/min, depending on their diameter. They also provide warning about the possibility of explosive chipping of concrete during heating. Regarding the time of annealing, it was stated that during the simulation of the emergency conditions (fire), the maximum surface temperature reached should be maintained for 60 ± 5 minutes. Our own research, however, was modeled on the already mentioned literature items due to the more balanced nature of the recommendations contained therein.

The exposure time of samples during own tests, i.e. 1,5 hours, at a given temperature, was also modeled on the technical literature, among others [[11]] to allow for a possible comparison of results. Cooling of samples to ambient temperature proceeded with the furnace door open, one can repeat after [[2]] that "the samples were cooled together with the furnace". After cooling the furnace to a temperature of about 50°C and removing them, they cooled in a room outside the furnace at a temperature of about 30°C. Ultimately, the

samples cooled down under laboratory conditions and under such conditions were tested (bent) until they were destroyed.
All samples were weighed before the tests, due to the need to include their mass in the formulas for calculating the stress intensity factor.

In our research, a common procedure was applied: the two-parameter Jenq and Shah crack model described in [[7]]. During the test studies, there was made a graph of load - width of opening of the edge of the CMOD primary notch.. The maximum load was achieved in 5 min., further the load was reduced to 95% of maximum and then reduced to zero. The samples were repeatedly loaded and unloaded according to this principle until fracture. Fracture parameters were determined from the relation: load - the width of the crack opening (P - CMOD) and load - displacement of the point of force application (P - f).

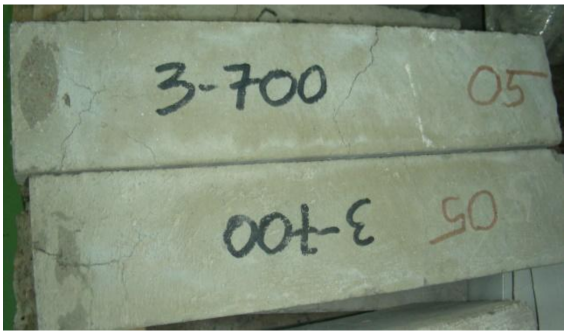

Fig. 1. Concrete sample for w/c = 0,5 after application of fire temperature 700^{o}C.

Fig. 1 shows a concrete sample after application of temperature of 700^{o}C and after testing (fracture). Visible are peeling and surface cracks as well as spalling. The breakthroughs of this sample are shown in Fig. 2. Apart from the damage mentioned, longitudinal cracks are also noticeable. In breakthroughs, damages in concrete under the influence of fire temperature, were formed mainly on the boundary of grains of coarse aggregate and paste.

Fig. 2. Breakthroughs of the sample from Fig. 1.

This is proof of the decrease in strength under the influence of high temperatures, especially the transition zone between the matrix and the aggregate, but also the decrease in the strength of the paste and cement mortar contained in the concrete.

During and after heating at 600 and 700^{o}C, a certain amount of cement paste samples with w/c = 0,5 and 0,6 were damaged. Some directly in the furnace, others during cooling outside the furnace. In turn, paste samples with a w/c ratio = 0,4 theoretically withstood the temperatures of 600 and 700°C, but they were so cracked that they were not suitable for placing in the strength machine, they simply crumbled and could not be tested. An example of such a damaged paste sample is shown in Fig. 3.

Fig. 3. A sample of paste w/c = 0,4 after heating in an furnace at 700°C.

The phenomenon of damage of paste samples at high temperatures is confirmed in the literature [[16]]. It stated, among others, that the temperature of 400°C is critical for cement paste, which completely loses its strength due to the dehydration of $Ca(OH)_2$ and the hydration of CaO. The picture shown in [[16]] of the damage of the cement paste sample (although cylindrical) after the application of temperature of 800°C is essentially identical to the view of the damage in Fig. 3.

Mortar samples after heating to high temperatures mostly survived this heating, especially those of w/c = 0,4. Some of the samples with the w/c ratio = 0,5, as in the case of paste, were not resistant to heating at 700°C, and samples with a w/c ratio = 0,6 did not withstand heating at 600 and 700°C. In the available literature [[17]], it is stated that at 900°C, mortar samples could not be tested because of their insufficient strength, while heating these samples at lower temperatures of 500°C and 700°C often resulted in their damage.

IV. THE RESULTS OF SELECTED STUDIES

From the multifaceted work [[5]] this paper only quotes and compares the results of the relationships of the critical of stress intensity factor K_{IC} on the temperature for concrete, paste and mortar. In Fig. 4, Fig. 5 and Fig. 6 there are "collective" charts on which these relationships were presented. The charts contain very interesting information despite the seeming lack of unambiguous possibility of comparing these relationships between differing, even phase composition, composites. It should be remembered, however, that the proportions of the ingredients of the paste and mortar were assumed to be identical to those in concrete.

The charts clearly show for all the studied ratios of w/c (0,4, 0,5 and 0,6), the practically identical course of relationship. The shape and arrangement of the graphs, in spite of different values, for all tested fire temperatures is basically identical. One should pay attention to the initial values of fracture toughness parameters at 20°C: in all charts at this temperature, the critical stress intensity factor is the highest, and at fire temperatures the parameter decreases very rapidly in concrete. In the fire temperatures, a smaller decrease of the K_{IC} factor in mortar and of paste than in concrete is particularly characteristic, expressed both in absolute numbers and percentages.

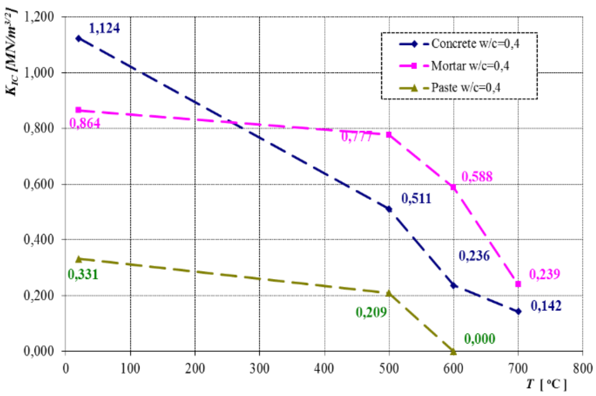

Fig. 4. Relationships between K_{IC} - T of concrete, paste and mortar for w/c = 0,4 under the influence of fire temperatures.

Very important information was provided by the results of tests on concrete, paste and mortar on one graph (Fig. 4, Fig. 5 and Fig. 6). For better readability, the charts were made separately for each w/c. A comparison of the percentage decrease in the K_{IC} factor of concrete, paste and mortar under the influence of fire temperatures leads to quite interesting conclusions: practically in all the examined w/c ratios, concrete showed the greatest decrease in the K_{IC} stress intensity factor.

The decrease was (depending on w/c) at 500°C from about 55% to about 77%, at 600°C from about 79% to 82%, while at 700°C - about 88% for all w/c [[5]].

In the case of the w/c ratio = 0.4 (Fig. 4), the smallest drop in the K_{IC} factor was observed in mortar - only 10% at the temperature 500°C, and slightly bigger drop in cement paste at this temperature - approx. 37%. All decreases in the K_{IC} factor for individual composites were as follows:
- 500°C: concrete about 55%, paste about 37%, mortar about 10%,
- 600°C: concrete about 79%, paste by 100%, mortar about 32%,
- 700°C: concrete about 87%, mortar about 72%.

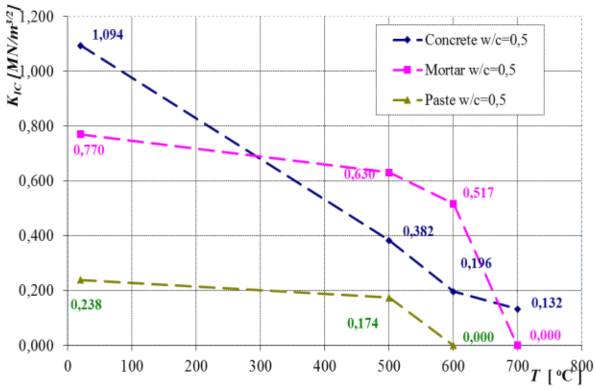

Fig. 5. Relationships of K_{IC} - T of concrete, paste and mortar for w/c = 0,5 under the influence of fire temperatures.

At w/c = 0,5 (Fig. 5), the smallest decrease in the K_{IC} factor was found in the cement mortar - about 18% at 500°C, in the remaining composites the following decreases were observed:
- 500°C: concrete about 65%, paste about 27%, mortar about 18%,
- 600°C: concrete about 82%, paste by 100%, mortar about 33%,
- 700°C: concrete about 88%, mortar 100%.

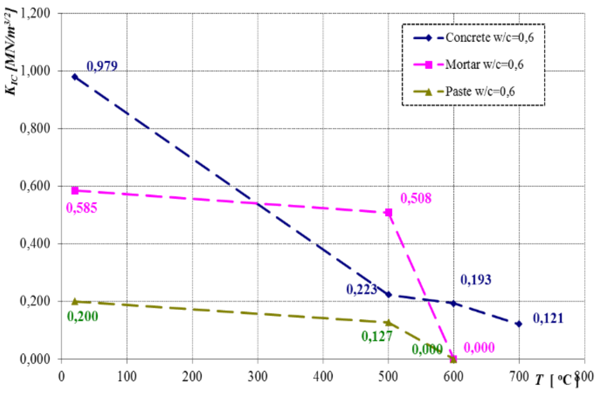

Fig. 6. Relationships between K_{IC} - T of concrete, paste and mortar for w/c = 0,6 under the influence of fire temperatures.

However, with the w/c ratio = 0,6 (Fig. 6), concrete, paste and mortar can only be compared up to the temperature of 500°C, because above this temperature samples of paste and mortar have been destroyed. The decrease of the concrete K_{IC} factor at this temperature was approx. 77%, of paste approx. 36%, and of mortar only approx. 13%.

As can be seen from the charts and sets of declines expressed in [%], for all tested w/c, the highest relative and absolute fracture toughness at high temperatures was exhibited by cement mortar, of course in the temperature range in which it has not yet been damaged.

V. THE RESULTS OF THE SEM STUDIES

Significant depth of field of the scanning microscope enables fractographic studies (topographies) of breakthroughs, which consist in the evaluation of their surface resulting from the stress tearing the sample material. These studies also make it possible to learn the mechanisms of cracking. As it is known, cracks are formed and develop in the weakest places of samples, so in the breakthroughs are visible various details of the structure, eg the separation of foreign phases, material defects (pores, voids and microcracks). In the course of our own research, photographs were taken by SEM scanning electron microscopy of concrete, paste and mortar samples exposed to fire temperatures. Performed X-ray microanalysis (*Energy Dispersive X - Ray Spectroscopy*) allowed the identification of the elemental composition of the test materials. Structural changes taking place in the tested composites were compared depending on w/c and temperatures, also at 20°C. There were presented exemplary photos of concrete and mortar structure samples of w/c = 0,4 and their X-ray microanalysis after the application of high temperatures.

The SEM photo (Fig. 7, magnification 500×) indicates the location of EDS research after application of fire temperature of 700°C - portlandite crystals (or similar plates [[2]]) are visible, which due to the high temperature have cracked, the structure of their columns has loosened and they lost their hexagonal shape. This is confirmed by the fact that at fire temperatures as a result of dehydration of calcium hydroxide and reduction of chemically bound water, the portlandite is decomposed [[18]].

The EDS microanalysis (Fig. 7), also taking into account the oxide composition, showed that the ratio of calcium oxide to silicon dioxide is: C/S = 0,81 < 1,0, i.e. it is phase "α" according to Nonat's classification.

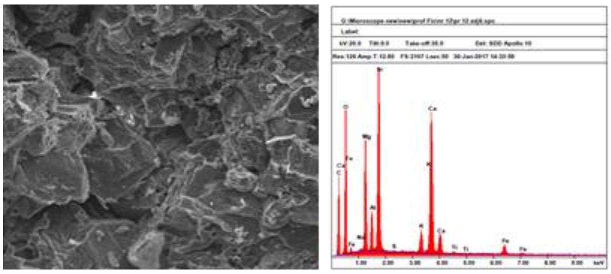

Fig. 7. Sample SEM photo and EDS microanalysis of concrete for w/c = 0.4 after exposure to fire temperatures.

According to [[19]], the composite has the highest fracture toughness when the C/S ratio is 0,99 in the CSH phase.
Fig. 8 shows a SEM photo of mortar after applying the temperature of 500°C (magnification 5000×).

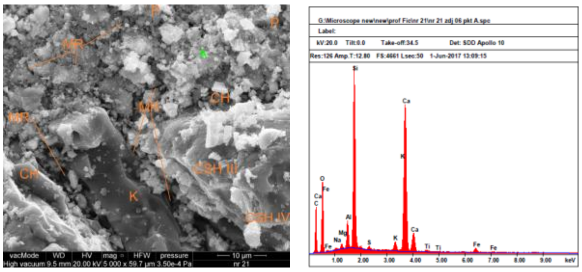

Fig. 8. Sample SEM photo and EDS analysis of mortar for w/c = 0.4 after exposure to fire temperatures: CH - portlandite, CSH phase, MR - micro-cracks, P - pores, K - aggregate.

EDS analysis (Fig. 8) shows that in the CSH III phase the C/S ratio = 0,97 according to Nonat's classification. This value is very close to C/S = 0,99, at which the composite has the highest fracture toughness (max K_{IC}).

This confirms the results of the tests described in point 4 by SEM photos and EDS microanalysis, stating that cement mortar has greater fracture toughness at high temperatures than concrete

VI. SUMMARY

The above studies and analyzes show that despite higher resistance of concrete to fire temperatures, as evidenced by concrete samples surviving temperature of 700°C, the decrease in K_{IC} is much faster in concrete than in paste and mortar, which in fact have a lower fire resistance than concrete, which is proved by the damage and even destruction of these samples (especially for w/c = 0,6) at temperatures above 500°C.

The reason for this behavior of these cementitious composites is the lack of coarse aggregate, and therefore a different level of mortar structure (micro) and cement paste (submicro) than concrete (macro). The lack of coarse aggregate also reduces the transition (contact) zone at the interface of cement matrix and aggregate, which eliminates the weakest link, i.e. this zone in concrete. Although in mortar, the transition zone also exists, but due to the small size of the fine aggregate grains, there is no so-called "Wall effect" on such a scale as it happens at the interface of coarse aggregate and cement matrix, which probably affects the relatively higher strength of the ITZ (*Interfacial Transition Zone*) in mortar.

This is confirmed by the damages in concrete after exposure to fire temperatures described in point 3.3, which are mainly formed on the grain boundaries of coarse aggregate and paste, without going through the grains of the coarse aggregate and without breaking them. This is the result of the decrease in strength under the influence of high temperatures, especially the transition zone between the matrix and the aggregate, but also, though to a lesser extent, of the paste and cement mortar contained in the concrete.
In the case of paste, due to the absence of aggregate, the transition layer is virtually residual, only on the remains of parts of unhydrated cement grains [[20]], which explains a smaller decrease in the K_{IC} factor at fire temperatures.

Conflict of interest
There is no conflict to disclose.

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