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STRESS DEVELOPMENT IN KAOLIN BODY DURING FIRING IN THE DEHYDROXYLATION REGION

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The authors demonstrate a simplified calculation of the radial, tangential and axial components of the stress arising during isothermal and non-isothermal firing of a cylindrical kaolin body in the region of dehydroxylation brought about by temperature and concentration gradients.

It was found that a tensile stress develops at the surface, and a compressive one at the body axis on both isothermal and non-isothermal firing. During non-isothermal firing, the surface stress is decreased by the compressive stress due to the temperature gradient. An increase in the rate of firing a cylindrical body from 1 K min^{-1} to 5 K min^{-1} was found to raise by 30 % the maximum of resultant tensile stress at the surface.

INTRODUCTION

Firing of ceramic bodies in the dehydroxylation region represents a combined process of mass and heat transfer. It was found [1] that mass transfer can be regarded as diffusion involving a chemical reaction, and heat transfer as conduction of heat with a source. A mathematical model of the firing over the dehydroxylation region, worked out on the basis of this idea, allowed the time development of concentration and temperature profiles to be calculated for a body having the form of an infinite cylinder.

Occurence of these fields is closely associated with the stress in the body. Unsuitably conducted firing may bring about stresses exceeding the instantaneous strength of the body and cause its failure. Knowledge of concentration and temperature profiles in the body is indispensable to calculating the stress, from which limit rates of firing in the dehydroxylation range can be derived.

Studies of firing in the dehydroxylation region have so far been concerned with calculation of stress due to the temperature gradient [2, 3, 4, 5]. However, temperature and concentration fields coexist in the body during its non-isothermal firing in the dehydroxylation region, so that the stress results from both gradients. During isothermal firing the stress occurs as a result of the concentration gradient only.

The present study had the aim to estimate the stress in a body, developed during its firing over the dehydroxylation range by both the temperature and the concentration gradients. It is also assumed that in the course of firing in the dehydroxylation range, the ceramic body conforms to the conditions of a classical continuum.

THEORY

Firing of a ceramic body in the dehydroxylation region brings about formation of temperature and concentration gradients in the body, and their existence is responsible for a stress.

On the assumption that the temperature dependence of material quantities can be neglected, analytical solutions concerning the stress in a rotary symmetrical cylindrical body are available. One of the procedures is the method of thermoelastic potential displacement [6, 7] which requires analytical description of the temperature field in the body.

The temperature field arising during firing at constant rate in a cylindrical body is parabolic and can be described by the following equation in terms of a dimensionless radius:

$$T(\nu) = 2T_s - T_p + 2(T_p - T_s)\,\nu^2, \tag{1}$$

where $T_s = (T_c + T_p)/2$ is the mean temperature of the cylinder, T_c is the temperature at the cylinder center, T_p is the temperature at the cylinder surface and $\nu = \frac{r}{R}$ is the dimensionless radius.

For the given conditions, the following expression can be derived for the radial component of the thermal stress [6]:

$$\sigma_{rr}^{T} = \frac{E\alpha_T}{2(1-\mu)}\,(T_p - T_s)\,(1 - \nu^2), \tag{2}$$

where the symbol T indicates stress due to the temperature field, E is Young's modulus of elasticity, μ is Poisson's constant and α_T is the coefficient of thermal expansion.

The final expression for the tangential component of the thermal stress has then the form

$$\sigma_{tt}^{T} = \frac{E\alpha_T}{2(1-\mu)}\,(T_p - T_s)\,(1 - 3\nu)^2. \tag{3}$$

Derivation of the expression for the axial component of the temperature state of stress is based on generalized Hook's law [6]. The final form of this expression for a generalized planar case is as follows:

$$\sigma_{zz}^{T} = \mu(\sigma_{rr}^{T} + \sigma_{tt}^{T}) - E\alpha_T[T(\nu) - T_s]. \tag{4}$$

The analytical expressions (2) through (4) for the main components of thermal stress are of limited validity for the case of a parabolic temperature field and a homogeneous material [6].

For orientation calculation of the radial, tangential and axial components of the concentration stress, use can be made of equations (1) through (4) by replacing the temperatures with the corresponding concentrations of water, and the coefficient of thermal expansion α_T with the coefficient of concentration deformation α_c [8]. The adjustments bring about the following equations for the individual components of the stress:

$$\sigma_{rr}^{K} = \frac{E\alpha_c}{2(1-\mu)}\,(x_{Ap} - x_{As})\,(1 - \nu^2), \tag{5}$$

$$\sigma_{tt}^{K} = \frac{E\alpha_c}{2(1-\mu)}\,(x_{Ap} - x_{As})\,(1 - 3\nu^2), \tag{6}$$

$$\sigma_{tt}^{K} = \mu(\sigma_{rr}^{K} + \sigma_{tt}^{K}) - E\alpha_c[x_A(\nu) - x_{As}], \tag{7}$$

where $x_{As} = (x_{Ap} + x_{Ac})/2$ is the mean concentration in the body, x_{Ap} is the concentration at the body surface, x_{Ac} is the concentration at the body center and

$\nu = \frac{r}{R}$ is the dimensionless radius. The top index K designates stress due to concentration gradient in the body.

Relations (5) through (7) can be applied on the assumption that the concentration field in the body is again parabolic and that the material is homogeneous.

The above equations for the calculation of thermal and concentration stress in a ceramic body indicate that the following material quantities or their dependence on temperature or concentration have to be known:

— Young's modulus of elasticity E,
— Poisson's constant μ,
— coefficients of thermal expansion α_T,
— coefficients of concentration deformation α_c.

Poisson's constant of ceramic materials has the value of 0.25 to 0.3 according to [2, 9].

Young's modulus of elasticity of ceramic material containing a clay component, namely the Podbořany kaolin, was determined by static measurement on three-point bending [9]. Over the temperature range of 473 to 1073 K, Young's modulus was found to be indistinctly temperature dependent. The mean value was 6000 MPa.

The α_T values can be obtained directly from dilatometric curves of fired bodies by the conventional method [10].

Determination of the α_c coefficient may be based on the assumption that the concentration deformation of an unfired ceramic body with a content of kaolinitic clay component can be generally described similarly to thermal expansion [8]:

$$\frac{\Delta l(\Delta x_A)}{l_0} = \alpha_1 \Delta x_A + \alpha_2 (\Delta x_A)^2 - \alpha_n (\Delta x_A)^n, \tag{8}$$

where the coefficients α_n are determined e.g. by minimizing the quadratic deviations of experimental points from the functional values (8). For smaller concentration gradients ΔX_A and changes in length, the linear part only of (8) is usually considered, where $\alpha_1 = \alpha_c$ can be called the coefficient of concentration deformation. It is then possible to write

$$\alpha_c(\Delta x_A) = \frac{\Delta l(\Delta x_A)}{l_0 \Delta x_A} . \tag{9}$$

DETERMINATION OF MATERIAL QUANTITIES

The required material quantities for the calculation of the time development of the stress during the body firing up to melt formation were measured on the Podbořany kaolin.

The value of α_c can be established by measuring changes in length of an unfired body in terms of the concentration of water remaining in the body at the time the length change is measured. The residual water concentration value can be determined from the change in weight of a body which has been removed from the dilatometer, cooled down and fired at 1173 K to constant weight. The bodies employed were platelets 1×11×50 mm in size, prepared by grinding of cylindrical bodies made by drawing on a vacuum auger. The platelets were annealed at 573 K to remove possible rests of technologically necessary water and the interlattice molecular water originating from the illitic-montmorillonitic component of Podbořany kaolin [10].

The changes in length were measured on the platelets by the conventional method [11] in a quartz tube dilatometer. At the various changes in length and corresponding temperatures, the platelets were removed from the furnace and cooled down to 298 K. The value of residual water concentration was determined by subsequent final firing at 1173 K.

The experimentally established points of dependence of the relative body length on residual concentration of water were found to form a straight line by approximation (cf. Fig. 1) whose slope corresponded to the value of the coefficient of concentration deformation, which in the case of the Podbořany kaolin had the value

$$\alpha_c = 0.05. \tag{10}$$

The value of the coefficient of thermal expansion α_T was determined from the dilatation curve of bodies of fired Podbořany kaolin, measured on specimens of the same shape and dimensions as those used in the measurement of α_c. The coefficient of thermal expansion had the value

$$\alpha_T = 0.4 \times 10^{-5}\ \mathrm{K}^{-1}. \tag{11}$$

Young's modulus of elasticity was taken to have the value

$$E = 6000\ \mathrm{MPa}, \tag{12}$$

and Poisson's constant the value

$$\mu = 0.25. \tag{13}$$

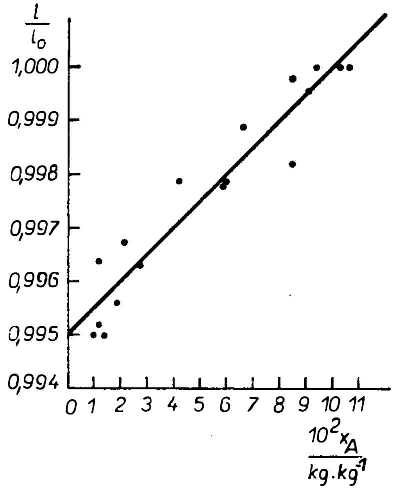

Fig. 1. Relative length of a platelet of Podbořany kaolin in terms of residual water concentration.

CONCENTRATION STRESS DURING ISOTHERMAL FIRING OF BODIES IN THE DEHYDROXYLATION REGION

As the concentration field in a body during isothermal firing was found to be parabolic [1] and the material quantities independent of concentration, the main components of the concentration stress were calculated according to analytical relationships (5) through (7) with the use of the material quantities (10), (12) and (13).

The values of the three main components of the concentration stress were calculated for the isothermal firing temperatures of 798 K, 823 K and 873 K. A typical example of a character and distribution of stress in a body at the given time at 873 K is shown in Fig. 2. Calculation showed that the concentration field creates in the body the stress whose radial component σ_{rr}^K is compressive in character over the entire body diameter. The tangential component σ_{tt}^K as well as the axial component σ_{zz}^K are tensile in character at the body surface and compressive at the centre. The same conditions were found for all the times at the other temperatures.

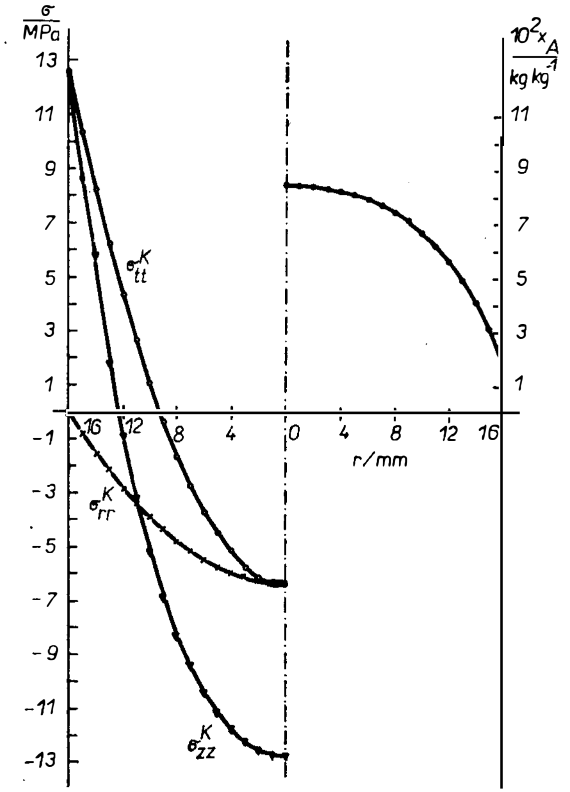

Fig. 2. Example of the courses of radial σ_{rr}^K, tangential σ_{tt}^K and axial σ_{zz}^K components of stress due to concentracion field in the body in the 40th minute of isothermal firing at 873 K.

The calculations further showed that the maximum values of tensile concentration stress occur at the body surface where it holds that $\sigma_{tt}^K = \sigma_{zz}^K$. The time course of the maximum values of tensile stress for isothermal firing temperatures of 798 K, 823 K and 873 K are plotted in Fig. 3. It appears that the rate at which tensile stress increases at the surface is highest at 873 K (cf. Table I).

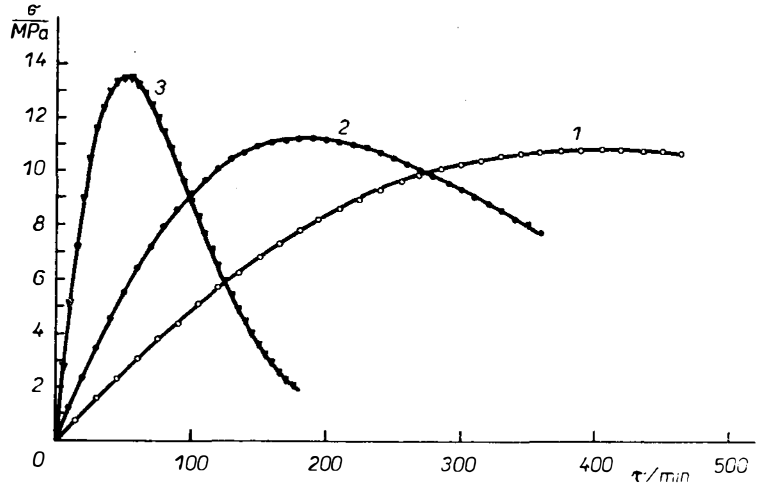

Fig. 3. Time dependence of maximum tensile concentration stresses σ_{tt}^{K} and σ_{zz}^{K} at the body surface during isothermal firing in the dehydroxylation range at 798 K (1), 823 K (2) and 873 K (3).

Table I

Comparison of the rates of increase in tensile stress at the body surface at various temperatures

$\frac{T}{K}$	$\frac{w}{\text{MPa min}^{-1}}$
798	≈ 0.05
823	≈ 0.1
873	≈ 0.4

STRESS DURING NON-ISOTHERMAL FIRING OF A BODY IN THE DEHYDROXYLATION REGION

The following can be said of the conditions in a body during non-isothermal firing:

— over the temperature region before the beginning of dehydroxylation, there is just a temperature field in the body,

— over the dehydroxylation region, both temperature and concentration fields exist in the body,

— after concluded dehydroxylation, only the temperature field remains again in the body.

This means that before the onset of dehydroxylation and after its conclusion, the stress occurring in the body is due to the temperature field only.

It may therefore be expected that in the dehydroxylation region thermal stress σ_{rr}^{T}, σ_{tt}^{T}, and σ_{zz}^{T} (cf. equations (2) through (4)), and concentration stress σ_{rr}^{K}, σ_{tt}^{K} and σ_{zz}^{K} (cf. equations (5) through (7)) develop in the body whose effects at one point of the body add up to the resultant stress $\sigma_{rr}^{K,T}$, $\sigma_{tt}^{K,T}$ and $\sigma_{zz}^{K,T}$.

A simplifying idea of deformation behaviour of ceramic material in the dehydroxylation region was used in calculating the stress in a body during non-isothermal firing. The ceramic material was assumed to expand over the entire temperature interval of 573 K to 1073 K and its behaviour was considered to be characterized by the coefficient of thermal expansion α_T. It was further assumed that the non-linearity of changes in length over the dehydroxylation region was due to changes in concentration in the material, and that its behaviour in this region was characterized by the coefficient of concentration deformation α_C.

The components of concentration and thermal stress were calculated according to the above equations using the material quantities (10) through (13).

Adding the individual components σ_{rr}^T and σ_{rr}^K, σ_{tt}^T and σ_{tt}^K, σ_{zz}^T and σ_{zz}^K at the same coordinate yielded the courses of the individual components of the stress and their resultants $\sigma_{rr}^{K,T}$, $\sigma_{tt}^{K,T}$ and $\sigma_{zz}^{K,T}$ for the temperature increase rates, at the body surface, of 1 K min^{-1} and 5 K min^{-1}.

The individual components of the stress in the body in the dehydroxylation region are plotted in Fig. 4 for the heating rate of 1 K min^{-1} at the body surface. The character of the components of concentration stress is obviously the same as in the case of isothermal firing. During non-isothermal heating, the stress is also affected by thermal stress, whose radial component σ_{rr}^T is tensile in character over the entire

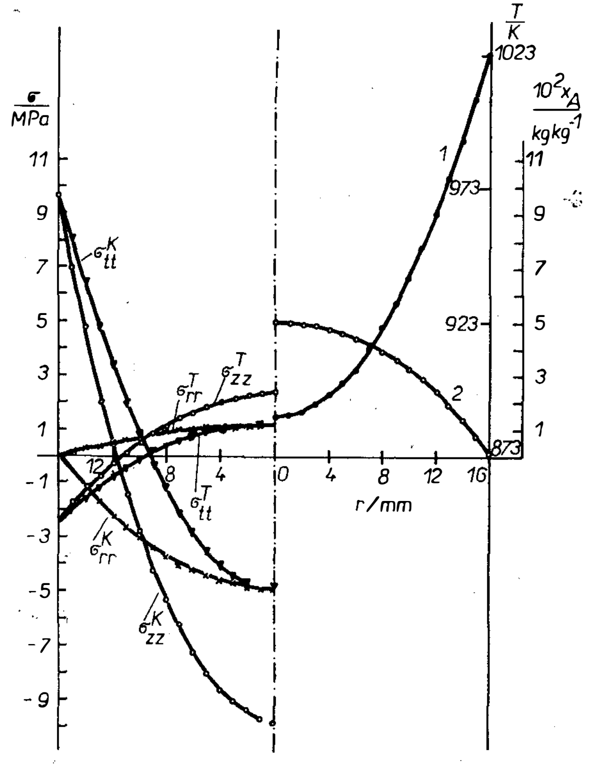

Fig. 4. The courses of radial σ_{rr}^T, σ_{rr}^K, tangential σ_{tt}^T, σ_{tt}^K and axial σ_{zz}^K, σ_{zz}^K components of the stres s due to temperature (1) and concentration (2) field in the body on firing at a rate of 5 K min^{-1}.

body diameter. The tangential component σ_{tt}^{T} and the axial one σ_{zz}^{T} are compressive in the surface areas of the body and tensile at its centre. Fig. 4 indicates that the greatest tensile stress occurs at the body surface as a result of components σ_{tt}^{K} and σ_{zz}^{K} while the effect of component σ_{tt}^{K} reaches over greater distances from the body surface than that of component σ_{zz}^{K}.

An example of the course of the tangential component of the concentration and thermal stress and their resultant for a heating rate of 5 K min^{-1} for one time of firing is shown in Fig. 5. It appears that according to assumptions introduced with respect to deformation behaviour of material in the dehydroxylation region, thermal stress σ_{tt}^{T} at the body surface decreases the value of tensile concentration stress σ_{tt}^{K}, so that the resultant tensile stress at the body surface, $\sigma_{tt}^{K,T}$, is smaller than the value of σ_{tt}^{K}. The same conditions were also found for the axial component $\sigma_{zz}^{K,T}$ (cf. Fig. 4).

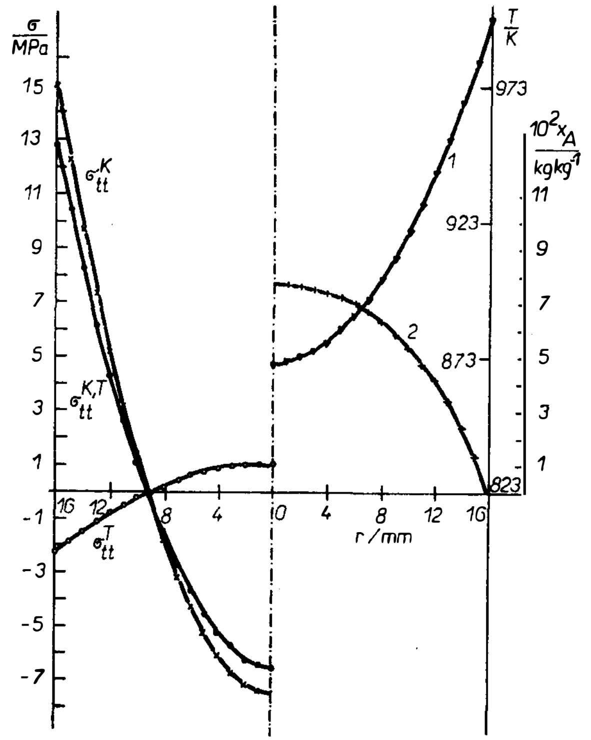

Fig. 5. The course of the tangential component of thermal σ_{tt}^{T} and concentration σ_{tt}^{K} stress and their resultant $\sigma_{tt}^{K,T}$, arising in the body in consequence of the temperature field (1) and concentration field (2) at a firing rate of 5 K min^{-1} in the 65th minute of firing from temperature $T_0 = 673$ K.

The shape of the time course of the maximum value of component $\sigma_{tt}^{K,T}$ and $\sigma_{zz}^{K,T}$ at the body surface is associated with the firing rate (cf. Fig. 6). At a firing rate of 5 K min^{-1} the rate of increase in $\sigma_{tt}^{K,T}$, $\sigma_{zz}^{K,T}$ at the body surface is about 0.6 MPa min^{-1}, and at a firing rate of 1 K min^{-1} the value is about 0.15 MPa min^{-1}, i.e. four times lower.

On comparing the maxima of these courses of tensile stress at the body surface at both heating rates one sees that at the firing rate of 5 K min^{-1} the value of the maximum is higher by 30 % than that at the firing rate of 1 K min^{-1} (cf. Fig. 6).

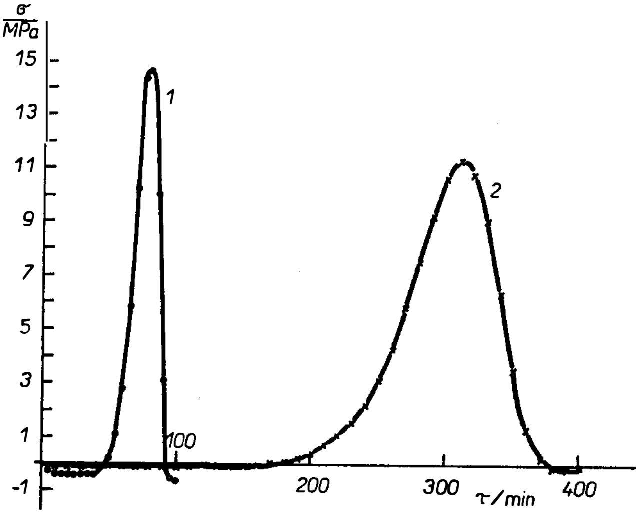

Fig. 6. Time course of the maximum values of the tangential component of stress $\sigma_{zz}^{K,T}$ and maximum values of the axial component $\sigma_{zz}^{K,}$ at the body surface at a firing rate of 5 K min^{-1} (1) and of 1 K min^{-1} (2). The initial time $\tau = 0$ corresponds to equalized body temperature of 573 K.

CONCLUSION

Calculations of the development of stress during isothermal as well as non-isothermal firing of cylindrical bodies of Podbořany kaolin, having the coefficient of thermal expansion $\alpha_T = 0.4 \times 10^{-5}\ \text{K}^{-1}$ and the coefficient of concentration deformation $\alpha_c = 0.05$, allowed the findings to be summarized into the following points:

— on the assumption that kaolinite being fired in the dehydroxylation region conforms to the conditions of an elastic continuum, it is possible to use the equations to estimate the radial, tangential and axial components of the stress arising as a result of temperature and concentration gradients,

— during isothermal firing, the stress σ^K is associated solely with the concentration gradient. The maximum tensile stress arises at the body surface and is represented by components σ_{tt}^K and σ_{zz}^K, the components being equal. The maximum compressive stress at the body centre is due above all to component σ_{zz}^K,

— during non-isothermal firing in the dehydroxylation region, the resulting stress $\sigma^{K,T}$ is due to both gradients. The resulting stress at the body surface $\sigma_{tt}^{K,T} = \sigma_{zz}^{K,T}$ is tensile in character and due to concentration gradient associated with components $\sigma_{tt}^K = \sigma_{zz}^K$. The stress is favourably decreased by compressive stress due to temperature gradient, and associated with components $\sigma_{tt}^T = \sigma_{zz}^T$,

— an increase in the rate of firing a cylindrical body from 1 K min^{-1} to 5 K min^{-1} raises the maximum of resultant tensile stress at the body surface by 30 %.

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VÝVOJ NAPĚTÍ V KAOLINOVÉM TĚLESE PŘI VÝPALU V OBLASTI DEHYDROXYLACE

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V práci jsou uvedeny vztahy pro výpočet radiální σ_{rr}, obvodové σ_{tt} a osové σ_{zz} složky napjatosti při výpalu válcového tělesa vyvolané koncentračním a teplotním gradientem. Vztahy vycházejí z předpokladu, že keramické těleso v oblasti dehydroxylace splňuje podmínky elastického kontinua. Výpočet je proveden pro izotermní a neizotermní výpal tělesa kaolínu Podbořany, pro který jsou v práci experimentálně stanoveny hodnoty koeficientu teplotní $\alpha_T = 0{,}4 \,.\, 10^{-5}\ \mathrm{K}^{-1}$ a koncentrační $\alpha_c = 0{,}05$ deformace. Bylo zjištěno, že při izotermním i neizotermním výpalu je na povrchu tahová a v ose tělesa tlaková napjatost. Při neizotermním výpalu je na povrchu výsledná tahová napjatost snižována tlakovou napjatostí vyvolanou gradientem teploty. Současně bylo zjištěno, že zvýšení rychlosti výpalu válcového tělesa z 1 K min^{-1} na 5 K min^{-1} zvyšuje maximum výsledné tahové napjatosti na povrchu tělesa o 30 %.

Obr. 1. Relativní délka destičky z kaolínu Podbořany v závislosti na koncentraci zbytkové vody.
Obr. 2. Příklad průběhu radiální σ_{rr}^{K}, *obvodové* σ_{tt}^{K} *a osové* σ_{zz}^{K} *složky napjatosti vyvolané v tělese koncentračním polem ve 40. minutě izotermního výpalu při teplotě 873 K.*
Obr. 3. Časová závislost maximálních tahových koncentračních napětí σ_{tt}^{K} *a* σ_{zz}^{K} *na povrchu tělesa při izotermním výpalu v oblasti dehydroxylace při teplotách 798 K (1), 823 (2) a 873 (3).*
Obr. 4. Průběh radiální σ_{rr}^{T}, σ_{rr}^{K}, *obvodové* σ_{tt}^{T}, σ_{tt}^{K} *a osové* σ_{zz}^{T}, σ_{zz}^{K} *složky napjatosti vyvolané teplotním (1) a koncentračním (2) polem v tělese při výpalu rychlostí 5 K min*$^{-1}$.
Obr. 5. Průběh obvodové složky teplotní σ_{tt}^{T} *a koncentrační* σ_{tt}^{T} *napjatosti a jejich výslednice* $\sigma_{tt}^{K,T}$ *vyvolané v tělese teplotním (1) a koncentračním (2) polem při rychlosti výpalu 5 K min*$^{-1}$ *v 65. minutě výpalu od teploty* $T_0 = 673$ *K.*
Obr. 6. Časový průběh maximálních hodnot obvodové složky napjatosti $\sigma_{tt}^{K,T}$ *a maximálních hodnot osové složky* $\sigma_{zz}^{K,T}$ *na povrchu tělesa při rychlosti výpalu 5 K min*$^{-1}$ *(1) a 1 K min*$^{-1}$ *(2). Počáteční čas* $\tau = 0$ *odpovídá ustálené teplotě v tělese 573 K.*

Stress Development in Kaolin Body during Firing in the Dehydroxylation Region

РАЗВИТИЕ НАПРЯЖЕНИЯ В КАОЛИНОВОМ ТЕЛЕ ПРИ ОБЖИГЕ В ОБЛАСТИ ДЕГИДРОКСИЛЯЦИИ

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В работе приводятся отношения для рассчета радиального σ_{rr}, контурного σ_{tt} и осевого σ_{zz} компонента напряженности при обжиге цилиндрического тела, вызванной градиентом концентрации и температуры. Отношения основываются на предположении, что керамическое тело в области дегидроксиляции отвечает условиям эластического континуума. Рассчет проводили для изотермического и неизотермического обжига тела из каолина Подборжаны, для которого в работе установлены величины коэффициента температурной $\alpha_T = 0{,}4 \cdot 10^{-5}$ K^{-1} и концентрационной $\alpha_c = 0{,}05$ деформации. Было установлено, что при изотермическом и неизотермическом обжиге находится на поверхности напряженность на растяжение и в оси напряженность на сжатие. При неизотермическом обжиге на поверхности окончательная напряженность на растяжение понижается в результате напряженности на сжатие, вызванной градиентом температуры. Одновременно было установлено, что повышение скорости обжига цилиндрического тела с 1 К мин$^{-1}$ до 5 К мин$^{-1}$ повышает максимум окончательной напряженности на растяжение на поверхности тела на 30 %.

Рис. 1. *Относительная длина пластинки из каолина Подборжаны в зависимости от концентрации остаточной воды.*

Рис. 2. *Пример хода радиального σ_{rr}^{K}, контурного σ_{tt}^{K} и осевого σ_{zz}^{K} компонента напряженности, вызванной в теле концентрационным полем в сороковой минуте изотермического обжига при температуре 873 К.*

Рис. 3. *Временная зависимость максимальных концентрационных напряжений на растяжение σ_{tt}^{K} и σ_{zz}^{K} на поверхности тела при изотермическом обжиге в области дегидроксиляции при температурах 798 К (1), 823 (2) и 873 (3).*

Рис. 4. *Ход радиального σ_{rr}^{T}, σ_{rr}^{K}, контурного σ_{tt}^{T}, σ_{tt}^{K} и осевого σ_{zz}^{T}, σ_{zz}^{K} компонента напряженности, вызванной температурным (1) и концентрационным (2) полем в теле при обжиге скоростью 5 К мин$^{-1}$.*

Рис. 5. *Ход контурного компонента температурной σ_{tt}^{T} и концентрационной σ_{tt}^{K} напряженности и их равнодействующая $\sigma_{tt}^{K,T}$, вызванной в теле температурным (1) и концентрационным (2) полем при скорости обжига 5 К мин$^{-1}$ в 65-ой минуте обжига при температуре $T_0 = 673$ К.*

Рис. 6. *Временный ход максимальных величин контурного компонента напряженности $\sigma_{tt}^{K,T}$, и максимальных величин осевого компонента $\sigma_{zz}^{K,T}$ на поверхности тела при скорости обжига 5 К мин$^{-1}$ (1) и 1 К мин$^{-1}$ (2). Начальное время $\tau = 0$ отвечает установившейся температуре в теле 573 К.*

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