

MODEL OF NON-ISOTHERMAL FIRING OF CERAMIC BODY IN THE DEHYDROXYLATION REGION

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A general mathematical model of firing a ceramic body in the dehydroxylation region allows the time development of concentration and temperature fields in the body to be calculated. The model is based on the assumption that kaolinite is a binary mixture of incompressible components, i.e. water bound in the form of OH⁻ groups in the structure of kaolinite and anhydrous kaolinite, that dehydroxylation is a first-order chemical reaction, and that firing the body in the dehydroxylation region is a combined process of mass and heat transfer. Mass transfer is regarded as diffusion involving a chemical reaction, and heat transfer as conduction of heat with a source. Using the general model, a solution for firing a body in the form of an infinite cylinder in the dehydroxylation region is suggested.

INTRODUCTION

Quantitative description of firing a ceramic body in the region up to melt formation requires knowledge of the processes characteristic of the ceramic mix in question over this interval.

Classical ceramic mixes contain certain proportions of a plastic component, mostly of kaolinitic type. Dried bodies then contain, apart from residues of technologically necessary water, also water bound in the form of OH⁻ groups in the structure of kaolinite. Molecular water is formed from the OH⁻ groups in the crystals of kaolinite on firing. This reaction is known under the term dehydroxylation. If one considers that the residual technological water is removed in the beginning of thermal exposure and that no changes due to temperature arise in the other mix components, then the changes in body weight during its firing over the interval up to melt formation are associated with dehydroxylation of the plastic components and transport of water vapour through the decomposition product. Dehydroxylation of kaolinite was the subject of numerous studies [1 through 30]. The lack of agreement with respect to the controlling process of dehydroxylation can be attributed above all to the evaluation of measuring results obtained from experiments using different arrangements, i.e. different layer thicknesses of the powdered sample or of bodies, different vapour pressures in the ambient atmosphere, use of different methods of study, etc.

If dehydroxylation is described as a chemical reaction yielding molecular water from OH⁻ groups in the structure of kaolinite, study of the reaction would be bound to a specimen having the size of the two-layer which constitutes the basic structural unit of kaolinite. As such an experiment is unviable, dehydroxylation of kaolinite is studied on thin layers of kaolinite. The results of such an approach must necessarily include the effect of the subsequent process, i.e. transport of water vapour through the decomposition product.

The resistance to vapour transport decreases with decreasing layer thickness

and the diffusion path also decreases. At a certain layer thickness, the chemical reaction will become the prevailing controlling process. Under isothermal conditions, the controlling process will also be determined by water vapour pressure in the ambient atmosphere and in the decomposition product. The situations that can occur are the following (cf. Fig. 1):

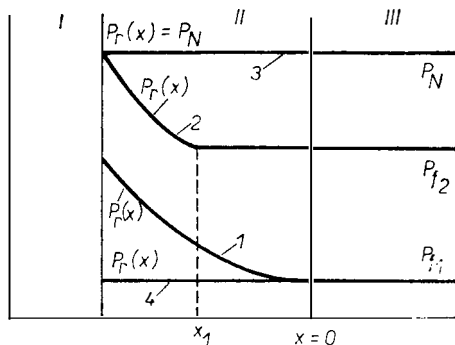


Fig. 1. Concept of the effect of water vapour in the ambient atmosphere on the process controlling dehydroxylation of kaolinite: I — kaolinite, II — decomposition product, III — ambient environment; P_N — equilibrium water vapour pressure at the given temperature, $P_{f1,2}$ — water vapour pressure in the ambient atmosphere, $P_r(x)$ — pressure profile in the decomposition product.

1. If the vapour pressure in the ambient atmosphere, P_f , is much lower than the equilibrium pressure P_N at the given temperature, or $P_{f1} = 0$ in the case at vacuum, i.e. $P_{f1} \ll P_N$ and water vapour pressure P_s in the decomposition product will increase in the direction from the layer surface towards the reaction zone (i.e. $P_r = P_r(x)$, where x is the ordinate in the direction of the thickness of the decomposition product), while $P_r(x) > P_{f1}$, then diffusion of vapour, driven by the pressure gradient, will be the controlling process.

2. If the water vapour pressure in the ambient atmosphere, P_{f2} , conforms to the relations $P_{f2} \gg P_{f1}$ and $P_{f2} < P_N$, the reaction will be the controlling process until $P_r = P_{f2}$ holds in the reaction product having the thickness $x = x_1$. On attainment of pressure P_r according to the relation $P_{f2} < P_r = P_N$, diffusion of water vapour will then be the controlling process.

3. No chemical reaction takes places if the vapour pressure in the ambient atmosphere is $P_{f3} = P_N$.

4. The chemical reaction as the only controlling mechanism can be determined in the system where no pressure gradient occurs in the decomposition products (i.e. $P_r(x) = \text{const.}$) and in the ambient atmosphere, $P_f < P_N$.

On using the situations indicated above to analyze existing knowledge of dehydroxylation, one finds that in studies [3, 4, 5, 6, 10] where the specimen thickness and the vapour pressure in the atmosphere corresponded to these situations, chemical reaction was established as the controlling mechanism of dehydroxylation. In the case of larger specimen thickness and vapour pressure corresponding to the given situations, diffusion of water vapour was determined as the decisive mechanism [5, 6, 7, 8, 28, 29]. In studies [7, 12] it was also found

that an outer vapour pressure of 0.006 MPa slowed down the reaction distinctly.

On the basis of the above findings it can be said that diffusion of vapour through the reaction product is the decisive mechanism in the firing of bodies of larger dimensions in the dehydroxylation region at a low vapour pressure in the ambient atmosphere, and that the process can be regarded as diffusion involving a chemical reaction. It should also be borne in mind that in the given case, diffusion of vapour through the body consists of diffusion of vapour through the particle of kaolinite and diffusion through the decomposition product surrounding the particle.

Firing further involves transfer of heat. As dehydroxylation is an endothermal reaction and heat transfer occurs in a solid body, conduction of heat through the body is the decisive mechanism and the heat conduction balance must also respect the heat consumed by the chemical reaction.

From this point of view, the firing of a body in the dehydroxylation region therefore represents a combined process of heat and mass transfer.

The present study had the object to work out a general mathematical model of the firing of a ceramic body in the dehydroxylation region on the basis of the concept described above.

MATHEMATICAL MODEL

If the initial kaolinite is regarded as a binary mixture consisting of component *A* (i.e. water bound in the form of OH⁻ ions in the structure of kaolinite) and component *B* (i.e. anhydrous kaolinite), then ϱ_A is the concentration by weight specifying the weight of component *A* contained in unit volume of the mixture, and the density of the mixture is then [31, 32]

$$\varrho = \varrho_A + \varrho_B, \quad (1)$$

where ϱ_B is the concentration by wt. of component *B*.

On defining the mass fraction of component *A* by the equation

$$x_A = \varrho_A/\varrho, \quad (2)$$

then for mass fractions in the binary mixture it holds that

$$x_A + x_B = 1. \quad (3)$$

The balance of weight of component *A* for the binary mixture has then the form (34)

$$\frac{D\varrho_A}{D\tau} = -\varrho_A \operatorname{div} \mathbf{v} - \operatorname{div} \mathbf{j}_A + r'_A, \quad (4)$$

where the substantial differential $D\varrho_A/D\tau$ is defined to the mean mass rate \mathbf{v} of the mixture, and \mathbf{j}_A is the diffusion flux of component *A* defined by the equation

$$\mathbf{j}_A = \varrho_A(\mathbf{v}_A - \mathbf{v}), \quad (5)$$

and r'_A is the specific rate of formation of component *A* in the mixture.

On considering an incompressible mixture, i.e. $\varrho = \text{const.}$, it holds that

$$\operatorname{div} \mathbf{v} = 0 \quad (6)$$

and the mass balance (4) acquires the form

$$\frac{D\rho_A}{D\mathbf{x}} = -\operatorname{div} \mathbf{j}_A + r'_A. \quad (7)$$

In the case of purely concentration diffusion in a binary mixture, the following relation holds between mass flux and the driving force:

$$\mathbf{j}_A = -\rho D_{AB} \operatorname{grad} x_A, \quad (8)$$

which for $\rho = \text{const.}$ can be written in the form

$$\mathbf{j}_A = -D_{AB} \operatorname{grad} \rho_A, \quad (9)$$

where D_{AB} is the diffusion coefficient of component A in component B .

Joining of equations (7) and (9) yields the following form for the mass balance:

$$\frac{D\rho_A}{D\tau} = \operatorname{div} (D_{AB} \operatorname{grad} \rho_A) + r'_A; \quad (10)$$

on considering an isobaric process, D_{AB} in the case of an isotropic mixture is a function of temperature and concentration.

Adjustment of substantial derivative (10) yields the form

$$\frac{\partial \rho_A}{\partial \tau} + \mathbf{v} \operatorname{rad} \rho_A = \operatorname{div} (D_{AB} \operatorname{grad} \rho_A) + r'_A. \quad (11)$$

With a system free of convective flow, i.e. $\mathbf{v} = 0$, equation (11) acquires the form

$$\frac{\partial \rho_A}{\partial \tau} = \operatorname{div} (D_{AB} \operatorname{grad} \rho_A) + r'_A, \quad (12)$$

or, on introduction of mass fractions, the form

$$\frac{\partial x_A}{\partial \tau} = \operatorname{div} (D_{AB} \operatorname{grad} x_A) + r_A, \quad (13)$$

while it holds that $r_A = r'_A/\rho$.

The term r_A takes into account the rate of formation of component A in unit volume of the mixture. In the resolving of concentration fields this term is a function of position and time in the most general case. On considering a homogeneous reaction usually taking place throughout the volume of the mixture of reacting components, the volume rate of formation of component A will appear in term r_A . For example, for a n -th order reaction, the term r_A can be expressed by the equation

$$r_A = -k_n x_A^n, \quad (14)$$

where n is the dimensionless order of the reaction, k_n is the rate constant of n -th order reaction. The k_n constant depends on pressure and in most instances distinctly on temperature according to the equation

$$k_n = k_{n0} \exp(-B_i/T), \quad (15)$$

where B_1 is the characteristic temperature of the process and k_{n0} is a constant. On considering a 1-st order reaction with the kinetic equation

$$r_A = -kx_A, \tag{16}$$

its resolving yields the following relationship for the time dependence of the concentration of component A :

$$x_A = x_{A0} e^{-k(T)\tau}, \tag{17}$$

where x_{A0} is the initial concentration of component A .

Substitution of (16) into (13) yields mass balance for a binary incompressible mixture involving a 1-st order chemical reaction without any convective flux (flow) being present:

$$\frac{\partial x_A}{\partial \tau} = \text{div} (D_{AB} \text{grad } x_A) - k(T) x_A, \tag{18}$$

where $D_{AB} = D_{AB}(T, x_A)$.

The temperature dependence of the diffusion coefficient can be expressed by the equation

$$D_{AB} = D_{AB0} \exp (-B_2/T), \tag{19}$$

where B_2 is the characteristic temperature and D_{AB0} is a constant.

If a chemical reaction takes place in the body, the energy balance has the form (33)

$$\rho c_p \frac{\partial T}{\partial \tau} = \text{div } \lambda \text{grad } T + A', \tag{20}$$

where λ is the thermal conductivity, c_p is the specific heat under constant pressure, T is temperature and A' is the specific heat source given by the chemical reaction.

If λ , ρ and c_p are constant, it holds that

$$a = \frac{\lambda}{\rho c_p}, \tag{21}$$

where a is thermal diffusivity. Substitution of (21) into (20) yields the following form of the energy balance:

$$\frac{\partial T}{\partial \tau} = a \text{div grad } T + A, \tag{22}$$

while it holds that

$$A = \frac{A'}{\rho c_p}. \tag{23}$$

To resolve equation (22) one has to know the source A describing the consumption of heat for the endothermal reaction. Expressing of the source term A may be based on the assumption that partial dehydroxylation has taken place in kaolinite with initial concentration of chemically bound water x_{A0} so that in time τ its weight is $m(\tau)$ and it contains an already decomposed proportion free of chemically bound water and weighing m_R , and a non-decomposed proportion containing the original concentration of chemically bound water x_{A0} and weighing $m_N(\tau)$. It then obviously holds that

$$\lim_{\tau \rightarrow \infty} m(\tau) \rightarrow m_R. \tag{24}$$

The heat consumed by the endothermal reaction of kaolinite dehydroxylation, the weight of kaolinite being $\Delta m_N(\tau)$ can be expressed by the equation

$$Q = \Delta H \Delta m_N(\tau), \quad (25)$$

where ΔH is the reaction enthalpy per unit weight of the mixture, $\Delta m_N(\tau)$ is the change in weight of the non-reacted proportion of kaolinite in time τ .

The weight of water m_{H_2O} released in time τ by dehydroxylation from kaolinite weighing $m(\tau)$ can be expressed by the equation

$$m_{H_2O} = \Delta x_A m(\tau), \quad (26)$$

where $\Delta x_A = x_{A0} - x_A$,

and at the same time by equation

$$m_{H_2O} = x_{A0} \Delta m_N(\tau). \quad (27)$$

In view of the validity of equations (26) and (27) it can be written

$$\Delta m_N(\tau) = \frac{\Delta x_A}{x_{A0}} m(\tau). \quad (28)$$

Substituting equation (28) into equation (25) yields an expression for the amount of heat consumed by the endothermal reaction in kaolinite weighing $m(\tau)$, in the form

$$Q = \Delta H \frac{\Delta x_A}{x_{A0}} m(\tau). \quad (29)$$

At the same time, endothermal dehydroxylation will bring about a temperature decrease by T in the kaolinite weighing $m(\tau)$. It is therefore possible to write the equation

$$Q = c_p \Delta T m(\tau). \quad (30)$$

From the equation of the right-hand sides of equations (29) and (30) it follows that

$$\Delta T = \frac{\Delta H}{c_p} \frac{\Delta x_A}{x_{A0}} \quad (31)$$

On expressing the change in temperature as a time interval and the time change in concentration by means of equation (17), one obtains the following expression for heat source A :

$$A = - \frac{\Delta H}{c_p} \frac{1}{x_{A0}} k(T) x_A. \quad (32)$$

Joining of equations (22) and (32) yields an energy balance in the form

$$\frac{\partial T}{\partial \tau} = a \operatorname{div} \operatorname{grad} T - \frac{\Delta H}{c_p} \frac{1}{x_{A0}} k(T) x_A. \quad (33)$$

DISCUSSION AND CONCLUSION

A general mathematical model of the firing of a ceramic body over the dehydroxylation region, considering dehydroxylation as a 1-st order chemical reaction, is expressed by a mass balance having the form

$$\frac{\partial x_A}{\partial \tau} = \operatorname{div} (D_{AB}(T, x_A) \operatorname{grad} x_A) - k(T) x_A \quad (34)$$

and by an energy balance having the form

$$\frac{\partial T}{\partial \tau} = a \operatorname{div} \operatorname{grad} T - \frac{\Delta H}{c_p} \frac{1}{x_{A0}} k(T) x_A. \quad (35)$$

The time development of concentration and temperature fields in the body can be determined by resolving equations (34) and (35). However, the calculation requires the material quantities or their temperature and concentration dependence to be known, i.e.

$$\Delta H, a, c_p, D_{AB} = D_{AB}(T, x_A), k = k(T). \quad (36)$$

If the quantities cannot be found in the literature, they have to be determined experimentally.

Knowledge of initial and boundary conditions is required for the solving of balance equations (34) and (35).

On considering, for example, firing of an infinite cylindrical body in the dehydroxylation range, introduction of cylinder ordinates gives equation (34) the form (32)

$$\frac{\partial x_A}{\partial \tau} = \frac{1}{r} \frac{\partial}{\partial r} \left(D_{AB}(T, x_A) r \frac{\partial x_A}{\partial r} \right) - k(T) x_A \quad (37)$$

while balance (35) acquires the form (33)

$$\frac{\partial T}{\partial \tau} = a \left(\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} \right) - \frac{\Delta H}{c_p} \frac{1}{x_{A0}} k(T) x_A. \quad (38)$$

The example of firing a cylindrical body of infinite length in the dehydroxylation region can then be formulated by the following conditions:

— initial conditions

$$x_A(r, 0) = x_{A0}, \quad (39)$$

$$T(r, 0) = T_0, \quad (40)$$

— boundary conditions

$$x_A(R, \tau) = x_{A0} \exp(-k(T)\tau), \quad (41)$$

$$T(R, \tau) = f(\tau), \quad (42)$$

$$\frac{\partial x_A(0, \tau)}{\partial r} = 0, \quad (43)$$

$$\frac{\partial T(0, \tau)}{\partial r} = 0 \quad (44)$$

where R is the radius of the cylinder.

Equations (36) through (44) then represent the complete set of equations for resolving the firing of a body, having the form of a cylinder of infinite length, over the dehydroxylation region.

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MODEL NEIZOTERMNÍHO VÝPALU KERAMICKÉHO TĚLESA
V OBLASTI DEHYDROXYLACE

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Východím předpokladem modelu neizotermního výpalu keramického tělesa s obsahem kaolinitu v oblasti dehydroxylace je, že v tělese dochází ke společnému sdílení vody difúzí a tepla vedením. Sdílení hmotnosti je považováno za difúzi s chemickou reakcí a sdílení tepla za vedení se zdrojem. Dehydroxylace kaolinitu je považována za chemickou reakci 1. řádu. Za těchto předpokladů byly získány základní bilanční a konstitutivní rovnice obou procesů. Je naznačeno jejich řešení pro počáteční a okrajové podmínky definující neizotermní výpal nekonečného válce v oblasti dehydroxylace. Jsou vymezeny nezbytné materiálové veličiny a jejich odpovídající závislosti pro řešení modelu.

Obř. 1. Předřtava o vlivu tlaku vodní páry v okolní atmosféře na řídící proces dehydroxylace kaolinitu: I — kaolinit, II — produkt rozkladu, III — okolní prostředí; P_N — rovnovážný tlak vodní páry za dané teploty, P_{f12} — tlak vodní páry v okolním prostředí, $P_T(x)$ — tlakový profil v produktu rozkladu.

МОДЕЛЬ НЕИЗОТЕРМИЧЕСКОГО ОБЖИГА КЕРАМИЧЕСКОГО ТЕЛА В ОБЛАСТИ ДЕГИДРОКСИЛИРОВАНИЯ

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Исходным предположением модели неизоатермического обжига керамического тела с содержанием каолинита в области дегидроксилирования является то, что в теле происходит общая передача воды диффузией и тепла проводностью. Массопередача считается диффузией с химической реакцией и теплопередача теплопроводностью с источником. Дегидроксилирование каолинита считается химической реакцией первого порядка. При таких предположениях были получены основные уравнения баланса и конституции обох процессов. В работе намечено их решение при начальных и граничных условиях, определяющих неизоатермический обжиг бесконечного цилиндра в области дегидроксилирования. Определяются необходимые величины вещества и им отвечающие зависимости для решения модели.

Рис. 1. Понятие о влиянии давления водяного пара в окружающей атмосфере на управляющий процесс дегидроксилирования каолинита: I — каолинит, II — продукт разложения, III — окружающая среда; P_N — равновесное давление водяного пара при заданной температуре, P_{f12} — давление водяного пара в окружающей среде, $P_T(x)$ — профиль давления в продукте разложения.

HYDROTALKIT, horečnato-hlinitý hydroxokarbonáthdrát $Mg_6Al_2(OH)_{16}CO_3 \cdot 4H_2O$, resp. $[Mg_{3/4}Al_{1/4}(OH)_2] [(CO_3)_{1/8}(H_2O)_{1/2}]$ vzniká popri vápenato-horečnatých alumináthdrátoch, resp. hydrogranátoch v hydratačných produktoch troskových cementov. V zmesnej troskovej malte vystavenej pôsobeniu 0,3 molárneho roztoku $MgSO_4$ dokázali D. M. Royová, E. Sonnenthal a R. Prave (Cement and Concrete Research, vol. 15, 914—916 (1985)) vznik hydrotalkitu, v ktorého štruktúre už pred časom zistili možnosť náhrady aniónov CO_3^{2-} aniónmi NO_3^- . Rastrovacím elektrónovým mikroskopom dosiahli autori článku výrazné rozlíšenie hydratačných produktov (doštičky hydrotalkitu, prizmy sádrovca, romboédre calcitu) v porovnaní s rtg. difraktografom.

L. Števlá

Dva závody patřící podniku United Glass Containers v Anglii se umístily jako druhý a třetí v soutěži o úsporu energie a získaly odměnu. Oba závody docílily úspory tím, že přešly z plynového vytápění dávkovačů skla na vytápění elektrické.

Firmě Corning se podařilo v letech 1983 až 1985 zlepšením tvarů varného nádobí ze skla Pyrex a zlepšením servisu zvýšit jeho prodej o 80 %.

Zájem o nové materiály z keramiky a skla potvrzuje také to, že od 1. 1. 1986 začínají vycházet dva časopisy věnované výzkumu a použití těchto nových materiálů. Jeden z časopisů se nazývá Advanced Ceramic Materials a vydává jej Americká keramická společnost a bude vycházet čtvrtletně. Bude přinášet články o biokeramice, katalyzátorech, keramice pro obtížné prostředí, řezných nástrojích, elektrických izolátorech, vodičích s rychlými ionty, feritech, keramice pro tepelné stroje, sklech pro infračervenou část spektra, optických vláčknech, zátaových sklech, sklech pro vakuovou techniku, piezoelektrických materiálech, senzorech a průhledné keramice. Druhý časopis vydává Elsevier a nazývá se Ceramics International a bude rovněž vycházet čtvrtletně. Bude se zabývat zejména metodami k přípravě surovin, výrobními procesy (lisování, tažení, vstřikování, válcování, lití, tažení, lisování za tepla a vysokého tlaku, plasmové sintrování) i vlastnostmi výrobků a inženýrskými problémy při jejich výrobě. Členem redakční rady tohoto časopisu je prof. V. Šatava, DrSc., z katedry technologie silikátů VŠCHT v Praze.

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