Percolation theory usage for water transfer simulation while drying out of refractory castables

Andrew V. Zabolotsky

***Abstract*—**A percolation structure model of refractory material was provided for mass transfer with temperature gradient (water evacuation while drying out) simulating. This allow providing pore structure model for material, which fits the experimental data. Coefficient of mass transfer for water, obtained with help of the suggested model was used for drying out process simulating for gunning mass, considering simultaneous chemical processes of hydration - dehydration. The results of simulation were not against data received by investigation of the gunning mass drying out at real metallurgical plant (damage character, observed at real installation can be explained using the suggested structure and mass transfer computer model).

***Keywords*—**percolation theory, refractory concrete, gunning mass, drying out, computer simulation, mass transfer.

I. Introduction

DESTRUCTION of ceramics concrete products during their drying out is a serious problem for refractory castables usage. Such destruction can occur with explosion and can cause serious damage of equipment or personnel maiming. The cause of concrete destruction is high inner pressure of water vapor, which grows rapidly with temperature increase and reach maximal possible value of 22 MPa at 373 °C [1]. At the same time typical tensile strength of ceramic castables is between 0,5 and 10 MPa, furthermore, structure defects such as pores or micro-cracks prove to be "stress concentrators", so mechanical stress values grow significantly near such damages [2]. The author tried to calculate stress multiplication factor for some pores types - spherical and discus and it was 1,46 for spherical and more than 7 for thin discus (ANSYS Mechanical program was used for these calculations) [3]. So, temperature more than 130 °C may be and more than 200 °C must be dangerous for humid ceramics material (vapor pressure is 0,3 and 1,5 MPa respectively).

Water evaporation can occur with mass transfer through solid phase of concrete or blowing out through channel pores. Process rate is different for these two ways of evaporation, but driving force of both is water vapor pressure. The part of water evacuated with one or another way depends on pore structure of concrete - whole porosity and ratio of closed and channel pores. Investigation of pore structure and its 3D modeling, using the results of microscope testing, is hard and time consuming task, so express methods of modeling are demanded. This article is illustrating usage of the mathematics percolation theory for concrete structure modeling and this model application for simulation of water evacuation.

Percolation theory generally is used for modeling of multi-phase substances structure and movement through such structure, if some phases are permeable, for example - porous ceramic body or electric conductivity of conductors and insulators mixtures. In the previous article [4], percolation theory was used for prediction a situation when a ceramic body will become permeable, if pores share is increasing. Generally pores can have any geometric form (from cubes to long nails), but according to experiment data, simple forms, such as cubes and spheres give fine correlation between experiment and calculation. Meanwhile, real porosity level of refractory concretes leads to structure models, which contain both channel and closed pores. So, the article [5] also discuss a method of closed - channel pores ratio calculation and fixing of geometrical distance between different pores types, which is needed for simulation of water transfer through solid phase.

The obtained method was used for analysis of water evaporation from refractory concrete and gunning mass samples during their drying out.

II. Calculation and Experimental Procedure

A. Percolation structure modeling

Water amount, used for castable processing is always more, than needed for chemical reactions of hardening, so before drying beginning, two types of humid can be found in body: free and chemically bonded water. According to Neville [6] free water can be present at that moment only inside pores, but not inside crystals as defect of lattice. Obviously, free water would be divided between closed and channel pores in the proportion of these pores ratio. So, the first task is to provide a model, allowing to calculate amount of pores of all possible types. The simple percolation model, based on same sized spherical elements, was used for this purpose.

The idea was to create a volume area filled with spherical solid elements of the same size and then replace some part of them with porous elements and calculate weather porous

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elements would be surrounded with only solids, or they would form some clusters, surrounded with solids or they would create some channel structures, connecting opposite surfaces of the whole area. The self-provided software was used for this research and volume area of 10^6 elements (100x100x100) was investigated. Simple search method was chosen for types of porous clusters fixing. Also, this software was used for calculation of distances between different types of pores and between random solid element and nearest pore. Monte Carlo method was used for the last purpose [5].

B. Mass transfer modeling

Mechanism of mass (water) transfer while drying out process is different for channel and closed pores of castable material. Pouiseuille formula is used for water evacuation modeling from channel pores [7]:

$$V = \frac{r^2}{8\mu} \cdot \frac{\Delta P}{h}. \quad (1)$$

V - velocity of water or vapor movement, r - channel pore radius, μ - liquid water or vapor viscosity, ΔP - pressure difference between atmospheric pressure and pressure inside pore, which is determined by hydrostatic, capillary pressure and equilibrium water vapor pressure for the temperature inside pore, h - water displacement in the pore. Pressure difference between atmosphere and inner pore vapor could be significant, because drying out process usually takes place at high temperature.

Water transfer from closed pores to nearest channel pore or sample border can be simulated using Fick's equation [8]:

$$\frac{dm}{dt} = D \cdot \frac{\partial c}{\partial x} \cdot dS, \quad (2)$$

mass transfer ($\frac{dm}{dt}$) through elementary area dS is proportional to concentration gradient (or pressure gradient for gases) ($\frac{\partial c}{\partial x}$) for transfer direction, proportional coefficient D is called mass transfer coefficient, this value is individual for different substances and needed to be fixed experimentally.

Calculations were done with self-provided software, using finite element method.

C. Experimental procedure

An aim of experimental testing was to fix the value of water transfer coefficient for explored ceramics materials. Samples were rapidly heated up to different temperatures between 100 and 200 °C: they were installed into hot stove and their mass loose was fixed while next several hours. The results were analyzed, using equations (1) and (2) for free water. Equation (1) was used for modeling of water evacuation from channel pores and equation (2) - for simulation of water transfer from closed pores to nearest channel pore.

Data of calcium hydro aluminates temperature dissociation was used for simulation of chemical bonded water evaporation. The data analysis for computer simulation was done by author and briefly shown at fig. 1. This figure shows molar amount of water, which can appear as a result of calcium hydro aluminates temperature dissociation depending on calcium oxide amount in dry castable and temperature of its hardening. These two factors are enough to predict the concrete chemical composition at the moment of drying out beginning [9 - 11].

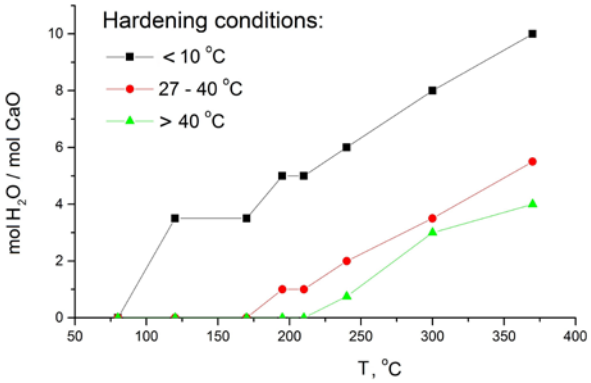

Fig. 1. Free humid formation while thermal dissociation of calcium hydro aluminates, depending on concrete hardening temperature.

We met a serious lack of information about velocity of synthesis and dissociation of magnesium hydroxide for simulation of chemical processes while hardening and drying out of castables, containing magnesia binder (gunning masses for example). So, we used data from [11, 12] and prepared fig. 2 as temperature - time map for magnesium hydroxide synthesis. The area above the graph at this picture corresponds with conditions of full hydroxide synthesis, the area with temperature below 50 °C - is the area there no reaction occur, all other areas are zones of partly completed reaction. The analysis of dissociation velocity for magnesium hydroxide was found only at [13], its value was fixed at this work as from $3 \cdot 10^{-5}$ up to 10^{-4} kg·m^{-2}·s^{-1}, depending on powder particles size, for temperature more than 270 °C: if temperature is lower magnesium hydroxide is stable and does not dissociate. This value was used for further simulating and calculations.

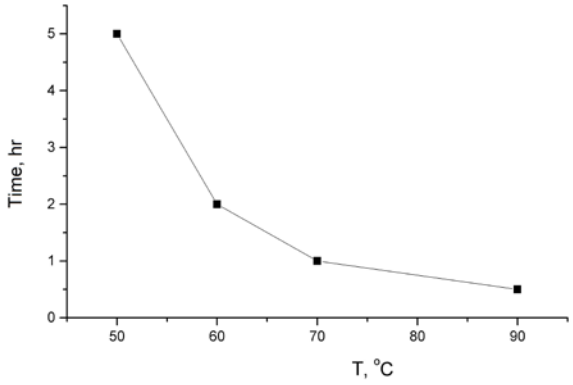

Fig. 2. "Temperature - time" map of magnesium hydroxide synthesis.

III. Results and Discussion

A. Percolation structure models for refractory concretes and gunning masses

Results of structure modeling were presented as graphs, illustrating correlation between opened and full porosity (fig. 3). Open porosity means volume of pores, having connection with sample surface. We can see several important points at this graph. First - when full porosity exceeds value of 30 - 32 %, we can see so called percolation threshold, when open porosity grows rapidly. Channel pores, connecting opposite surfaces of the sample, appear at the same point and material becomes permeable for gas or liquid. Open porosity, which could be measured with standard tests is equal to about 7 % at this moment. This conclusion is valid for mono fraction grain composition of material. Real poly fraction materials has another percolation threshold value, which could be calculated using hierarchical structure models [4]. That entry shows that real ceramic materials could be presented as mono fraction matrix (second hierarchical level), including big grains (first level) and percolation threshold must be fixed for the matrix. In this case percolation threshold for whole two-level material can be 9,6 % of open porosity or more, depending on grains - matrix ratio.

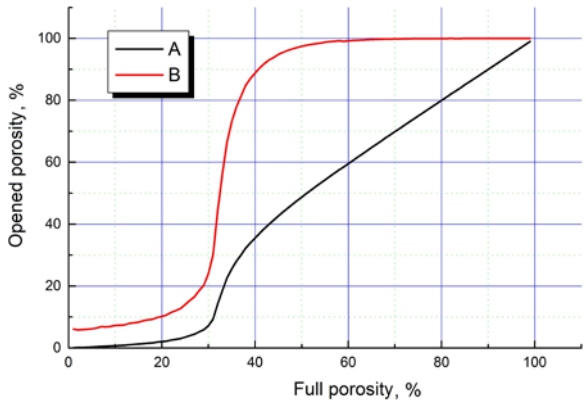

Fig. 3. Full and open porosity obtained with percolation model: A - absolute open porosity value, B - open - full porosity ratio.

Full porosity interval between 30 and 60 % (10 - 60 % opened porosity) is characterized by presence of both pores types - opened and closed. And the second point at the fig. 3 graph is 60 % of full or opened porosity: if porosity exceeds this level it becomes all opened - closed pores share becomes inessential.

In fact, refractory concretes have open porosity about 15 % and gunning masses - about 40 - 45 %. So, according to the mono fraction spherical percolation model these materials must be permeable. By the way, numerous percolation models, based on extended pores shape are known. But percolation thresholds for such structure (really such structures can appear after thermal removal of organic fiber) can be as low as 0,6 volume % for thin long fiber [14]. So, if pores shape is not spherical - material becomes permeable with lower porosity level, than with spherical pores.

Such structure characteristics as distance from random closed pore to nearest channel pore and from random point inside solid phase to channel pore depend on average grain size of binding phase, which can be fixed by microscope investigation [5]. Different character of these two values is logical: opened pores grow inside material from all sides slowly with full porosity increasing and closed pores are distributed evenly, so the average distance between them changes evenly too. The situation is different for distance from solid phase to channel pore: because if porosity level is below percolation threshold - there are opened pores at the sides of the sample and central solid zones are far away from these pores. After percolation threshold is exceeded, open pores reach all zones of the sample and distance reduces at the moment and significantly.

So, this geometrical data is enough to fix the needed length of way for free water to reach from closed to opened pore and for chemically bonded water to reach from the point of dissociation inside solid phase to nearest channel pore. This data and average grain size of the binding phase (which was equal to 30 μm for investigated materials) is enough for further modeling.

Two refractory concretes and one gunning mass were chosen for water transfer modeling. Concretes contained 0,5 and 1,0 % of calcium oxide respectively, the gunning mass contained 16 % of active fine powder of magnesium oxide. Opened porosity for concretes was 19,0 and 18,5 % respectively and 40 % for gunning mass. Table 1 presents results of percolation modeling for these materials, concretes are marked with numbers 1 and 2, gunning mass with 3. Open porosity was measured experimentally for these materials, other values were calculated, using percolation model [4]. So, opened porosity is given in volume percents to full volume of material, full porosity was calculated, using fig. 3 and opened porosity value. Such characteristics as single pores, closed clusters and percolation clusters shares are given in volume percents to full porosity, their meaning is the following:

Table 1. Porous structure characteristics

№	CaO, %	Open porosity, %	Matrix porosity, %	Single pores, %	Closed clusters, %	Percolation clusters, %
1	1	18,5	40	6	10	84
2	0,5	19,0	42	4	7	89
3	-	40,0	65	<1	~5	>94

- **single pores** - a structure which appears when one of solid grains is removed with void, so it is like small spherical pore and its size is equal to average size of solid grain
- **closed clusters** are couples of single pores, situated close to each other, having common border; closed clusters does not have any contacts with sample surface
- **percolation clusters**, unlike closed have exits to minimum 2 surfaces of the sample.

Fig. 4 illustrates one of possible closed clusters geometrical form, generated by used percolation models. It means that this

structure is surrounded by solid phase only. Single pore is equal to one sphere at this figure and percolation cluster is like very long complicated shaped cluster from one sample surface to another.

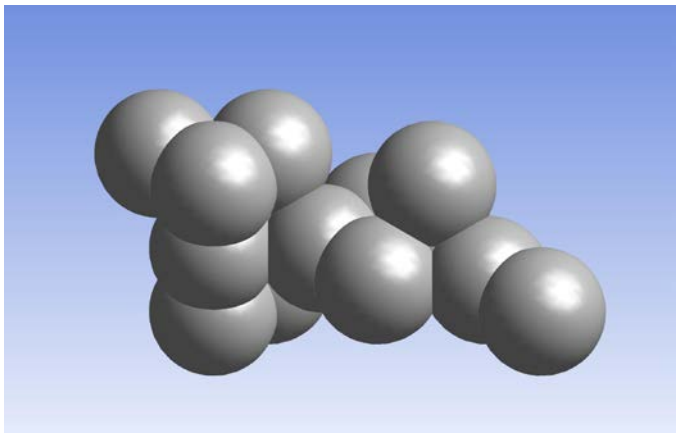

Fig. 4. Geometrical form of pores cluster (example).

B. Mass loss while drying out

Typical graph of concrete sample mass loss, while drying out at constant temperature, is shown at fig. 5. It is typical for capillary-porous body drying out. The following lots can be found at such graphs:

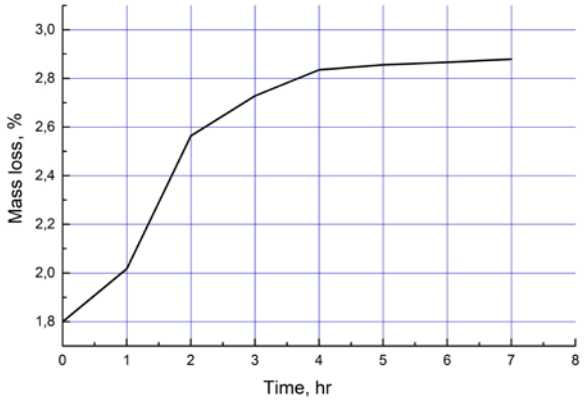

Fig. 5. Typical mass loss graph for refractory concrete (experimental).

- **constant drying speed** - when water or water vapor movement from sample core to surface has similar or more velocity, when its evaporation from surface. It refers to water evacuation from percolation (or opened) clusters and equation (1) can be used for it modeling. It is lot from 0 to 2 hours at fig. 5, different incline angle of graph for time, less and more than 1 hour takes place due to very rapid heating of sample at the beginning of the process, up to the used drying out temperature level.
- **decreasing drying speed** - water movement from core of the sample has lower velocity in comparison with its evaporation from surface. This period also refers to water evacuation from percolation clusters and corresponds to the graph lot between 2 and 4 hours at fig. 5.
- **the second constant speed** lot from 4 hours at fig. 5 means that water evacuation from closed pores takes place at the moment. Water need to overcome solid barrier between closed and open pores. This stage modeling needs equation (2) usage.

It can seem that water evacuation from closed pores can take place simultaneously with water movement from opened and percolation clusters. But driving force of water movement from closed pores is difference between vapor pressure in opened and closed pores, so while significant amount of water presents in opened clusters this pressure difference is very low. So, the significant vapor movement from closed pores begins after all water from opened pores is evacuated.

In this way mass transfer coefficient from equation (2) can be calculated, using parameters of the second constant speed lot of concrete drying out graph. Table 2 contains needed parameters for number 1 and 2 concretes and calculation results for temperatures 160 and 200 °C respectively.

Table 2. Mass transfer coefficient for concretes

№	Temperature, °C	Mass loss, %	Time, h	Mass transfer coefficient, m^2/s
1	160	0,15	2	$5{,}13 \cdot 10^{-12}$
1	200	0,34	3	$8{,}16 \cdot 10^{-12}$
2	160	0,044	3	$1{,}27 \cdot 10^{-12}$
2	200	0,069	4	$2{,}45 \cdot 10^{-12}$

For the verification of this drying out simulation a calculating algorithm, based on finite element method was suggested. The results were that water evacuation from opened pores needs about 2 hours and the following evaporation from closed pores needs about 8 hours. These results are not against experimental data.

A special attention needs chemically bonded water, which must be taken into consideration if temperature of the material (refractory concrete) is between 80 and 370 °C - the interval, where chemical compounds dissociation takes place [9]. It could be simply accounted by increasing water amount in all pores according to pore type part.

C. Percolation model usage for drying out simulation with chemical reaction consideration

The percolation structure model was used for gunning mass drying out simulation. Water transfer from different pore types was simulated using Fick's and Pouiseuille equations respectively.

According to table 1 data, the gunning mass is high porous material and main pores part is presented with percolation clusters. Also, gunning mass application needs significant amount of water (in the experimental case - about 23 mass %). So, about 94 % of added water (approximately 21,6 mass % of the sample) will appear inside percolation pore cluster and other 6 % of water (1,4 sample mass %) - inside closed pores [6].

Typically, gunning masses are applied to control lining layer, which has comparatively high surface temperature (about 80 °C - recommended and up to 150 °C - actual).

Gunning mass layer thickness commonly varies between 80 and 120 mm. Surely, the temperature field of gunning mass layer would be different in these two control layer temperature cases.

We prepared a special software for simulating at the same time heating of the gunning mass layer (for all three possible cases: from hot control layer or from the working surface or from both sides at the same time), water evacuation from channel percolating pores, water transfer from closed pores to opened channels and chemical reactions of oxides with water (for both calcium and magnesium oxide) or corresponding hydroxides dissociation, when temperature is high enough.

The following results, concerning gunning mass applying conditions were obtained:

1. Magnesium hydroxide synthesis occur only if starting temperature of control lining layer is above 90 °C, in the opposite situation the gunning mass layer can't receive the needed for hydration process temperature above 50 °C.

2. It is impossible to evacuate all free water from gunning mass layer, using only heat, generated by hot control layer. A significant amount of water remains in the layer even if the gunning mass was applied to the layer with 200 °C temperature and exposition time was 5 hours.

3. Nearly all possible amount of magnesium hydroxide would be synthesized while drying out with control layer heating in the zones, close to control layer if starting temperature is enough. The thickness of layer, containing magnesium hydroxide increase with starting temperature growth and it can reach 60 mm thickness, then control layer starting temperature is 200 °C.

The detailed analysis of these 3 results would be done below.

Free water evacuation can be simulated using both (1) and (2) equations. As it was mentioned above, equation (1) is used for simulating of water or vapor movement in channel pores and equation (2) is used for modeling of water movement from closed to opened pores. These processes take place one after another, because water vapor pressure in channel pores becomes a let for diffusion from closed voids.

The results of simulating are presented at fig. 6: humidity, referred to opened pores is marked by black columns, to closed - with red and their sum - with green. At the beginning of the process temperature and vapor pressure increase rapidly at the zone of gunning layer close to the control layer (high coordinate numbers at fig. 6). This leads to water evacuation through dry and porous control layer (commonly it is manufactured of porous heat insulating chamotte bricks) and partly some water is "pressed out" to the layers with less coordinate numbers (fig. 6), which are nearer to the working layer surface. Material in positions 3 and 4 at fig. 6 have humidity near to its starting level and position 5 has humidity, more than starting, this illustrates the mentioned above thesis of "pressing out". At the same time at position 7 humidity of opened pores is not significant, while in closed pores it is nearly equal to the starting value.

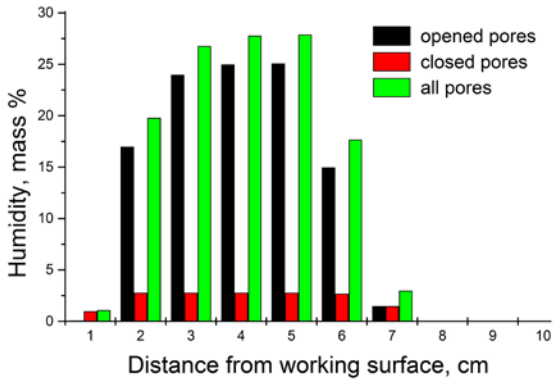

Fig. 6. Calculated humidity of lining working layer after 5 hours exposition (gunning mass was applied to the wall with surface temperature 200 °C).

The zone with low coordinate numbers (close to the working surface) also shows significant difference with actual and starting humidity, because it is situated close to surrounding atmosphere with low and constant water vapor pressure.

At the second stage of drying out, heating is provided from the working surface of gunning mass layer. So, the direction of humidity movement would change to opposite - it would be "pressed out" towards control layer. At the same time water vapor of the zones, close to working surface would be removed to atmosphere through working surface of the lining. High temperature of the control layer would discourage for significant water movement towards it.

For chemical reaction simulation, the reaction of magnesium oxide hydration-dehydration was chosen:

$$MgO+H_2O \leftrightarrow Mg(OH)_2 \quad (3)$$

This reaction bonds free water at relatively low temperature 50 - 100 °C and release it at temperature 270 °C, so this released water vapor becomes extremely dangerous because of high pressure. This is the only reaction taking place while gunning mass drying out. At the same time chemical processes while drying out of calcium aluminate concretes are complicated, due to dependence of material chemical composition on its hardening conditions [9].

Calculations approved that it is impossible to suggest such preheating schedule for gunning mass layer, which can prevent magnesium hydroxide synthesis - it would be formed in such quantity, as nearly all active MgO will participate in process. If gunning mass is applied to the control layer with temperature below 90 °C the reaction of $Mg(OH)_2$ formation take place only than preheating of the working surface takes place. At the case than control layer temperature is 200 °C, the zone, where this reaction is complete can reach 60 mm thick, other part of the layer would participate in reaction while working surface preheat.

The most dangerous stage of working layer preheating is period, than layer temperature reach 270 °C and dissociation of $Mg(OH)_2$ take place. The pressure, which can be reached

inside closed pores, while magnesium hydroxide decomposition depends on pores surface area and decomposition rate. We calculated time dependence of vapor pressure for pores structure, mentioned at line 3 of table 1 in assumption, that all closed pores are spherical with 30 μm diameter. For decomposition rate we took both maximum and minimum possible values [13] and result is presented at fig. 7.

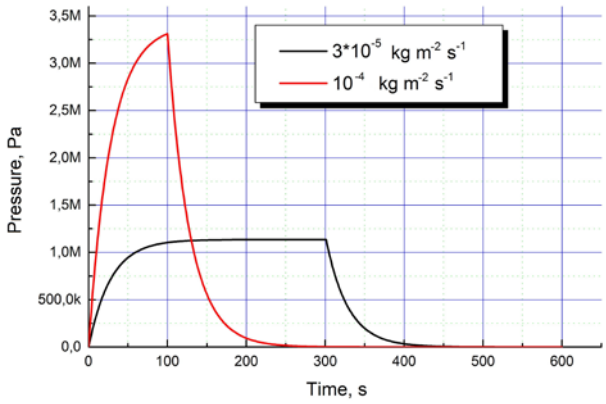

Fig. 7. Water vapor, formed of $Mg(OH)_2$ decomposition, pressure, depending on decomposition rate.

Obvious, that both decomposition rates are dangerous for material with tensile strength about 0,5 MPa. For real industrial installations, $Mg(OH)_2$ decomposition results in chipping of the working layer while preheating treatment and it is shown at fig. 8.

Fig. 8. Gunning mass chipping, while preheating process.

So, the computer simulation showed, that existing porous structure of gunning mass not allow creating a preheating schedule, which would be safe for the lining integrity. So, the way of preheating security increasing is in changing of the porous structure - for example, increasing of channel pores part or decreasing of closed pores share.

D. Percolation structure model usage for mechanical stress simulation

As it was mentioned before, the most dangerous pore structure of concretes is closed pore, which is loaded with inner vapor pressure and at the same time is a stress concentrator because of high curvature of surface. Such pore becomes stress concentrator for all types of stress, provided by thermal growth of material or by inner pressure.

As it was calculated before [3] spherical pores can multiply the mechanical stress of surrounding material with 1,46 multiplication coefficient, this value can be much more significant for the pores of non symmetrical form (crevice or thin disk, for example). Furthermore, such high-porous materials as concretes or gunning masses can suffer of greater stress multiplication, because of pores superposition, which have an effect, when pores are situated close enough to each other.

These factors were analyzed, using the ANSYS Mechanical software. A closed pore structure, presented at fig. 4 was used as a geometrical model for the calculation, typical elastic and thermal properties of high alumina refractory concrete were taken for material model. At fig. 9 at same time results of thermal growth stress modeling (left) and inner pressure (right) are presented.

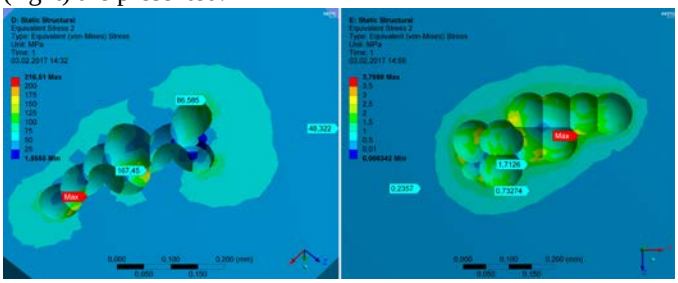

Fig. 9. Thermal growth and inner pressure stress inside closed pore of refractory concrete.

The left picture corresponds with temperature 160 - 200 °C, the stress relaxation effect (decreasing of thermal stress value with time, because of plastic deformation) was not taken into consideration, while it can be significant, specially at high temperature. The right picture concern to the inner pore pressure of 1 MPa (corresponds to the temperature 180 °C). So, we can see that thermal stresses near pores can be 3-5 times higher, than in surrounding material and can reach 200 MPa because of self thermal growth of material (this stress has compressive character), while material compressive strength is about 120 MPa in the considered conditions. The stress relaxation effect can reduce the calculated level of stress approximately 4 times, so it is probably, that such stress would not be dangerous for material (fig. 9, left).

At the same time tensile stress, produced by high inner vapor pressure, cannot be reduced by stress relaxation because vapor pressure does not depend on plastic deformations of solid phase (fig. 9, right). Typical tensile strength of refractory concrete is about 10 MPa (sometimes less) and stress concentration can reach 3 times or more. So, inner pressure level of 3 MPa, which correspond to temperature 230 °C is dangerous for the most lasting castables.

The pores superposition effect is illustrated at fig. 10. It shows, that additional stress growth if pores are situated close to each other can be significant, if distance between pores become less, than their own size. More close location of pores leads to more significant stress growth. According to results of structure modeling - the shortest average distance between pores in concrete matrix can be equal to 0,5 sizes of pore, so, it can cause 20-25% of addition stress growth.

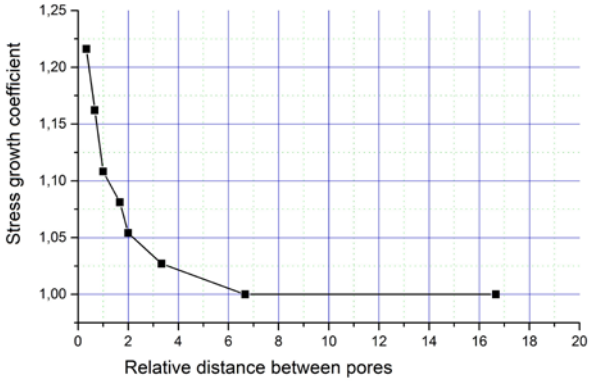

Fig. 10. Coefficient of stress growth dependence on relative distance between pores.

This need to be taken into consideration while stress simulating calculations of industrial concrete constructions or refractory linings, because standard software products give results without consideration of the porous structure features (singularity effects, causing mechanical stress growth at "solid - pore" border).

IV. Conclusions

A percolation theory was used for refractory concrete structure modeling. Random distribution of same size pores was chosen for the modeling. This structure model allowed to divide pores to such types: closed, opened and percolation clusters and it was shown that open porosity value (which is commonly measured while material testing) correlate with full porosity of material.

Mass transfer (water evacuation while drying out) in pores of different types proceeds with different mechanisms: viscous - in opened pores (Pouiselle equation can be used for it modeling) and diffusion for water evacuation from closed structures (Fick's equation can be used for it modeling). The model was completed with chemical transformation simulation, because processes of concrete (or gunning masses) hardening or drying out are commonly accompanied by hydrotation or dissociation reactions, changing humidity of material.

Water vapor inside pores can cause destruction of material, including explosion, at the moments, when vapor pressure exceeds material tensile strength. It was shown that vapor pressure inside closed pores can cause increasing of stress value, because of stress concentration at the high curved pore surfaces.

An experimental procedure for fixing of material's needed parameters was provided and verified at laboratory of "Magnezit Group".

The suggested model can be used for simulating of mass transfer in refractory castables or computer testing of their drying out schedule. The same approach could be used for mass transfer simulating in the case if pores in concrete are multi-sized spheres or even have non-spherical form: ellipsoids or long cylinders, for example.

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