UDC 539.21

NATURE OF FORMATION OF NANOSIZED PORE CHANNELS OF LAVA-LIKE FUEL-CONTAINING MATERIALS AT THE «SHELTER» OBJECT

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The evolution of the components of the brown and black ceramics' pore space of the lava-like fuel-containing materials from the "Shelter" Object, which generated during heat treatment, was being investigated. With increasing of the temperature, a part of the nanoscale pore channels are closed at 100…150 °C, some of cracks – at 200 °C also at 400 °C, the nanoscale channels are fully closed at 400 °C, the cracks – at 530 °C, and the nanopores – at 600 °C. Open nanoscale channels are the result of combining at least a part of α-particle tracks, formed by α-decay of transuranic elements at the self-irradiation, during the long period. Nanoscale pore channels and cracks were being formed as a result of processes taking place in the materials well their "preparing" after, during the accident.

Nuclear, radiation and environmental safety of the "Shelter" Object is determined by the lava-like fuel-containing mass (LFCM) due to the fact that they contain the majority of radionuclides from the beyond control nuclear reactor of Unit 4 of the Chernobyl Nuclear Power Plant. LFCM structure and its characteristics are necessary to be researched for the purpose of a long-term forecasting of their degradation. In the decades after the accident, there were sufficiently studied not only crystal inclusions, but also silica glass matrix of LFCM. There were received data on the phase composition, content and size of crystal inclusions, the distribution of uranium between the crystal inclusions and silica glass phase, elemental composition of silica glass phase and parameters of open porosity [1–3].

Research conducted recently [4, 5] had shown, that along with the gas pores and cracks previously known, LFCM also contain nanoscale pore channels. It is shown that nanochannels connected gas pores to each other and with the environment in whole and also provided with the formation of that open porosity of material, which is responsible for the ingress of air and water into the LFCM and thus makes a significant effect on the degradation of their structure and properties.

The data previously received [5], induce us to suppose, that nanosized pore channels in LFCM are the result of accumulation of the radiation defects and the basic mechanism of their formation - interaction with the material of α-particles formed at the α-decay of radionuclides. To deepen our image about LFCM it is necessary to find out the nature of the formation of nanosized pore channels. Familiarity with the nature of the formation of each components from all available LFCM pore space, would allow to reconsider the model of structure degradation and physical-mechanical properties of LFCM at the higher level approach, notable to identify definitely the stages of material degradation, find their sequence, to evaluate their duration, etc. That would allow considerably improving the reliability of predicting the LFCM behavior and create conditions for choosing the ways of LFCM treatment in future on the subsequent stages of work at the "Shelter" Object.

The purpose of the investigation was to determine the nature of the formation of nanosized pore channels for improvement of the model of LFCM degradation at the "Shelter" Object by means of study of changes of the characteristics of all the components of the LFCM pore space as a result of heat treatment.

EXPERIMENTAL METHODS

The brown and black ceramics samples, which are typical for the steam distribution corridor and rooms 305/2, 304/2, were investigated, respectively.

During the study it was being conducted the method for determination of volumes of each the group of open pore channels, from available into the porous body, on working fluid disposal at evaporation [6, 7]. Samples of LFCM as plates with dimensions (3…3.5) x (20…30) x (30…40) mm were placed into a vacuum chamber. Air was pumped out from the pore channels to a pressure of $6 \cdot 10^{-2}$ mm of mercury column to filled them with working fluid - distilled water. Then it was carried out the controlled drying to remove the working fluid during its evaporation and determined the time dependence on mass of the system at a constant temperature.

Each of the LFCM samples was being heat-treated serially at constant temperature during 5 hours into air laboratory oven. Subsequent heat treatment was carried out at a constant temperature, higher than the previous one. Increasing and reduction of the temperature was less than 2.5…4.5 °C/min. After each heat treatment there were determined the volumes of all open pore channels and the solid phase volume. Heat treatments were carried out in the interval of temperatures 70…800 °C.

The apparent density of the LFCM samples was determined by means of hydrostatic weighing method and pycnometric - by water pycnometry method.

EXPERIMENTAL RESULTS

Specimens of brown ceramics (№1 and №2) had an apparent density of 2.70 /cm^3 and 2.81 g/cm^3 and an open porosity of 6.9 % and 7.2 % respectively. Specimens of black ceramics had an apparent density of 2.16 g/cm^3 and an open porosity of 9.8 %.

Time dependence on mass of system the 'brown ceramics (initial sample № 1) – water' at a temperature of controlled drying 36…37 °C is represented in Fig. 1.

The mass of system decreases with the course of time. Dependence contains 5 linear sections: 4 from which are oblique and 1 that parallel to the abscissa axis. The straight lines, which extend the linear segments up to the intersection with the ordinate axis, intercept on the above mentioned the values of the mass, difference of which (m_0 - m_1), (m_1 - m_2), (m_2 - m_3) and (m_3 - m_4) are numerically equal to volume of 4 group of pore channels of different diameter. The relative volumes of the pore channels values of the duration of water evaporation stage are represented in Table.

It was issued earlier [5], that stages I, II, III and IV of water removal from the pore channels of brown ceramics correspond to large size gas pores, small size gas pores, cracks and nanosized pore channels, respectively. Stage V corresponds to solid stage.

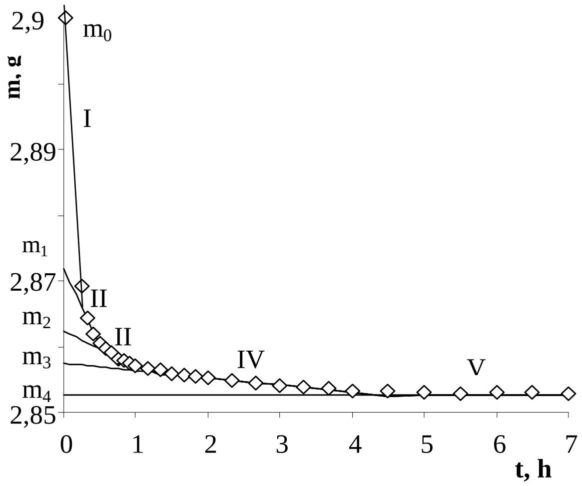

Fig. 1. Mass of system 'brown ceramics – water' dependence on time of drying

The relative volumes of the open pore channels, stage durations of removing of working fluid from them and volumes of solids phase in the brown ceramics (specimens №1) of lava-like fuel-containing materials before and after heat treatment

Stages		I	II	III	IV	V
Component of structure		large size gas pore	small size gas pore	cracks	nano-channels	solid stage
Relative volumes before and after heat treatment, vol. %	initial	5,43	0,61	0,39	0,44	93,13
	300 °C	5,35	0,66	0,32	0,14	93,52
	530 °C	6,03		0,0	0,0	93,97
error, ± %		0,10	0,08	0,05	0,05	0,10
Stage durations before and after heat treatment, h	initial	0,24	0,54	1,2	4,5	-
	300 °C	0,24	0,50	1,0	3,5	-
	530 °C	0,20	0,36	-	-	-
error, ± h		0,014	0,03	0,04	0,4	-

Notes: temperature of controlled drying was 36…- 37 ºC.

As a result of the heat treatment at 300 °C specimen mass decrease from 2.8526 g to 2.8492 g by detaching from him a few particulates. Heat treatment at 530 °C resulted to the destruction of the sample on seven large and many small size fragments. Weight of seven large fragments was 2.0276 g.

Heat treatment at 300 °C did not result to relative volume change of the large size and small size gas pores. But cracks volume decreased by 0.07 % (in 1.2 times), and the volume of nanoscale channels by 0.3% (in 3.1 times). The solid phase volume increased by 0.39 %. This, points to the closure of some part of cracks and considerable part of nanoscale pore channels. The durations of the water removal from large size and small size gas pores has stayed about the same, and from the cracks and nanoscale pore channels decreased (table).

Heat treatment at 530 °C also did not result to change of relative volume of the large size and small size gas pores. Their total volume did not change. The dependence of mass of system 'brown ceramics – water' don't have stages III and IV, corresponding to cracks and nanochannels. The solid phase volume has increased by 0.45 %. These results indicate the closure of all nanochannels and the predominant part of cracks. Durations of removing water from the large size and small size gas pores reduced slightly by increasing of the specimen surface due to the increase of their side surface under destruction into several fragments (table).

For a more comprehensive study of the behavior of pore space components of LFCM while heat treatment, it was conducted the heat treatment of the specimen № 2 of brown ceramics sequentially with duration for 5 hours, under temperatures of 150, 200, 250, 300, 350, 400 and 450 °C. Mass of the specimen with reduced heat treatment temperature increases (Fig. 2). In the temperature range 20…300 °C specimen weight decreases by 0.002…0.005 g every 50 °C, in connection with the detaching from the specimen for a few particles of material with diameter of 0.1…0.3 mm. At temperatures of 350, 400 and 450 °C, weight reduced more significantly, for 0.13…0.4 g every 50 °C, due to more intensive detaching of material particulates at the destruction of the sample into 4, 6 and 8 parts, respectively.

Dependences of the relative volumes of open pore channels and the solid phase of brown ceramics (specimen 2) on the heat treatment temperature are represented in the Fig. 3. The temperature-controlled drying was 35…37 °C. In the temperature range 20…300 °C the solid phase volume does not change and makes at average 92,8 % (see Fig. 3, curv. 1). With increasing temperature up to 400 °C and 450 °C it increases to 93.04…93.12%.

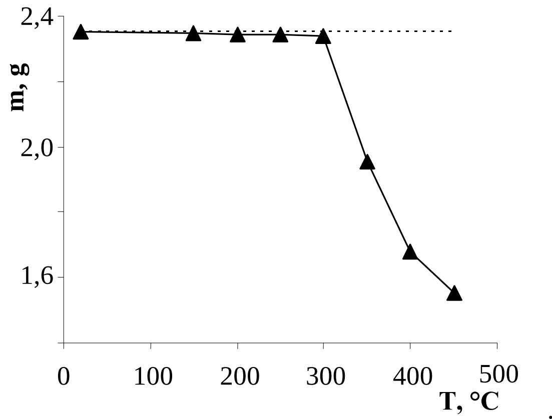

Fig. 2. Dependence of the mass of the LFCM brown ceramics specimens №2 on heat treatment temperature

The relative volumes of the both large and small size gas pores are independent on the heat treatment temperature (see Fig. 3, curv. 2 and 3). After heat treatment at 400 and 450 °C their total volume also is not changed (see Fig. 3, curv. 4).

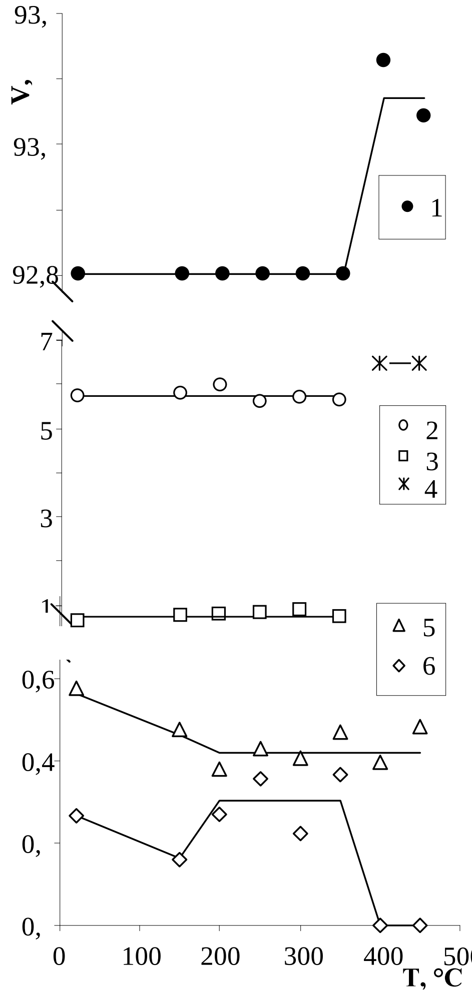

Fig. 3. Dependences of relative volumes of open pore channels and the solid phase of the LFCM brown ceramics specimens 2 on the heat treatment temperature (1 – solid phase, gas pores: 2 – large; 3 – small; 4 – large and small (sum) 5 – cracks; 6 – nanoscale pore channels)

With the temperature of heat treatment increasing the relative volume of cracks is decreased at first, into the temperature range 20…200 °C, and then reached to the constant with an average value of 0.5% (see Fig. 3, curv. 5). The relative volume of nanoscale pore channels when increasing temperature up to 150 °C is reduced, then increased (at 200 °C) and stayed practically unchanged up to 350 °C and at 400 and 450 °C became zero (curv. 6).

The durations of stages during water removing from large size gas pores, small size gas pores and cracks stayed practically unchanged into the temperature range 20…350° C (Fig. 4, curv. 2, 3 and 5). With increasing of the temperature up to 400 and 450 °C, the stage durations are reduced, apparently due to the increasing of the sample surface because of its destruction onto several pieces. The duration of the water removal stages from nanoscale channels with elevation of the heat treatment temperature to 150 °C is increased to 3.1 hours, and then into the temperature range 150…350 °C is significantly reduced to 1.86 hours (see Fig. 4, curv. 5). This indicates that at 150 °C a reduction in the average pore diameter of nanoscale channels occurs, and at further elevating of the heat treatment temperature – it increases.

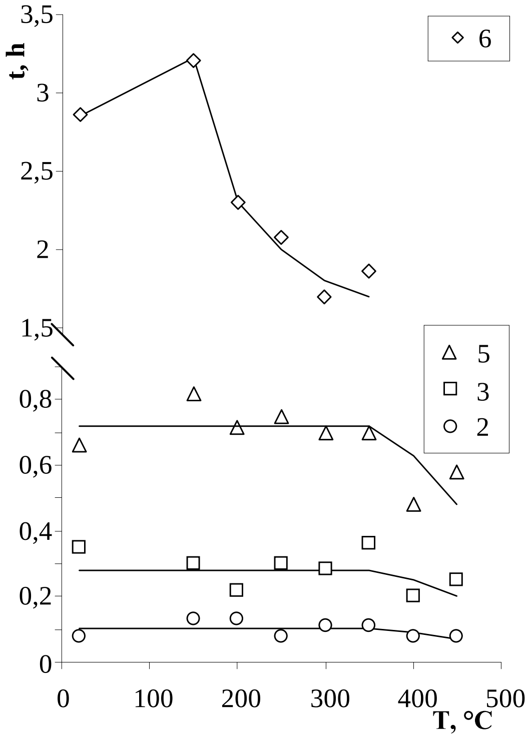

Fig. 4. Dependences of the water removing stages duration from the open pore channels of the LFCM brown ceramics specimen 2 on the heat treatment temperature (for notation see Fig. 3)

The reduction of the relative volume of cracks into the temperature range 20…150 °C when duration of water removing from them is practically unchanged indicates the closing of the part of cracks. A reduction in the volume of nanoscale pore channels into this temperature range with increasing of duration of the water removing stage (notably, decreasing their average diameter) definitely indicates about closure of some part of those channels. The subsequent rise in temperature up to 200 °C results to an increasing of their volume, notably to the opening of previously closed down part of nanoscale pore channels. Constancy of the nanoscale pore channels' volume and shortening of the duration stages of water removing into the temperature range 200…350 °C indicates that it causes an increase in their average diameter, which at 400 °C leads to complete closure.

Dependence of the mass of system the 'black ceramics – water' on time at temperature of 68 °C has also one parallel to x-axis and 4 inclined linear sections and such dependence is equal to similar one as system of 'brown ceramics – water' (see Fig. 1). Analysis of the data in this article and the previous results [5] showed that stages I, II, III and IV of water removing from the pore channels of black ceramics of LFCM corresponds to gas pores, cracks, nanoscale pore channels and nanopores respectively. Stage V corresponds to the solid phase.

For the purpose of study the behavior of the components of the pore space of LFCM during the heat treatment it was sequentially conducted a heat treatment of the LFCM sample of black ceramic, with a duration of 5 hours at temperatures of 70, 100, 150, 210, 260, 300, 350, 400, 500, 600, 700 and 800 °C. Mass of the specimen into the temperature range 20…300 °C was practically unchanged (4,276±0,005) g (Fig. 5). Into the temperature range 300…500 °C, the mass was reduced by 0.12…0.2 g every 50 °C in connection with the detaching from the specimen the particulates of material after its breaking into several parts.

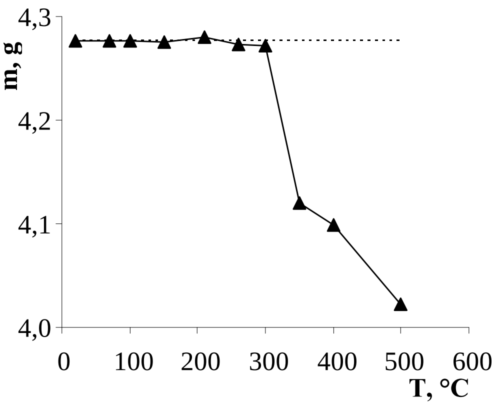

Fig. 5. Dependence of mass of the specimen of LFCM black ceramics on the heat treatment temperature

Depending of relative volumes of open pore channels and the solid phase of black ceramics on the heat treatment is represented in Fig. 6. The temperature-controlled drying was 68…70 °C. With elevation of the heat treatment temperature to 70 °C, the volumes of solid phase increased from 90.2 to 90.83% and then, in the range of 70…300 °C, it stayed constant. With elevating of the temperature up to 400°C it had increased to 91.6% and then into the range of 400…800 °C it stayed constant (curv. 1). The relative volume of gas pores volume has no dependence on the heat treatment temperature and was 7.5% (curv. 2). The relative volume of cracks into the temperature range 20…300 °C does not depend on the heat treatment temperature and equal to 0.93%. With elevating of the temperature up to 400 °C it decreased to 0.35% and stayed so until a temperature rose up to 800 °C (curv. 3). With elevation of the temperature to 100 °C, the volume fraction of nanoscale pore channels had reduced rapidly from 1.09 to 0.23%, with elevation of the temperature to 150 °C had increased sharply to 0.72%, and then with the elevation of the temperature to 400 °C had redused slightly up to 0.42% and till 800 °C had not been changing (curv. 4). The relative volume of the nanopores into the temperature range 20…500 °C does not depend on the heat treatment temperature and was 0.19%. With elevating of the temperature up to 600 °C, it had redused to zero and was staying so until the temperature of 800 °C (curv. 5).

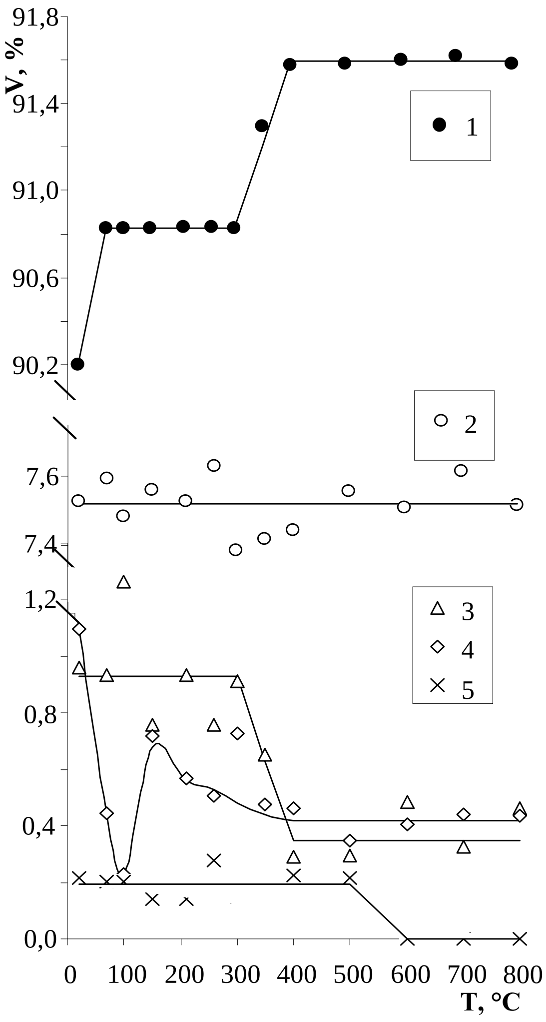

Fig. 6. The dependences of the volumes of open pore channels and the solid phase on the heat treatment temperature of the specimen of LFCM black ceramic (1 – solid phase; 2 – gas pores; 3 – cracks; 4 – nanoscale pore channels; 5 – nanopores)

The durations of stages of water removing from gas pores, cracks, nanoscale pore channels and nanopores do not depend on the heat treatment temperature (Fig. 7, curv. 2–5).

Reduces in relative volume of nanoscale pore channels with elevation of the heat treatment temperature up to 100 °C in combination with an increase in relative volume of the solid phase indicates the closing of the significant part of those channels (see Fig. 6, curv. 4 and curv. 1). Increased volume of nanoscale pore channels with a consequent elevation of the temperature indicates the opening of substantial part of them (60%). Crack volumes reduce into the temperature range 300…400 °C points to the closing of the greater part of cracks (curv. 3). Reduction of the nanopore volume to zero into the temperature range 500…600 °C indicates the closing all of the nanopores (curv. 5).

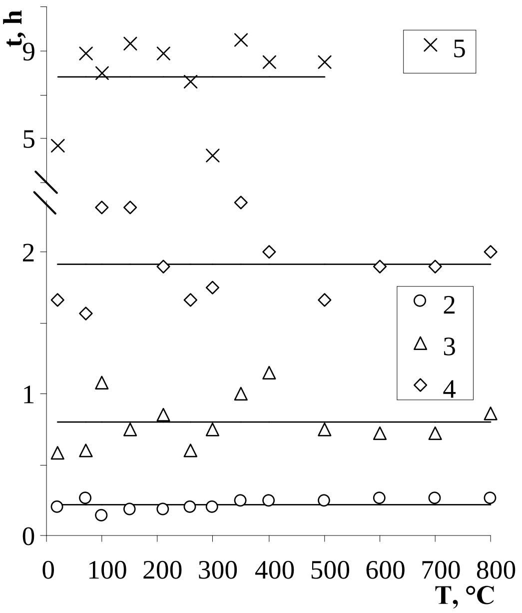

Fig. 7. Dependence of the stages duration of water removing from the open pore channels of the sample of LFCM black ceramics on the heat treatment temperature (for notation see Fig. 6)

RESULT DISCUSSION

Analysis of our previous results [5] shows that black ceramics stage II corresponds to the cracks, and stage III - to nanoscale pore channels. Taking into account, that volume estimation of open pore channels was performed under close temperatures (36…37 °C and 41…42 °C, Tables 1 and 2), we can compare the durations of stages of water removing from the pore canals. Evidently that the duration of stage II of black ceramics (3.2 h) is close to the duration of stage III of brown ceramics (3 h) and the stage III of black ceramics (7.2 h) is close to the duration of stage IV of brown ceramics (6,94 h).

The accumulated experience of using the method to determine the volumes of pore channels on removing of working fluid from them [7, 8] allows us to estimate the average diameter of the nanopores in black ceramics as 15…35 nm. The formation of nanopores apparently occurred when LFCM cooled during the accident that was caused by the difference in linear thermal expansion coefficient of the basic mass of glass phase and its regions, possibly formed as a result of silicate melt segregation.

As a result of consecutive heat treatments of the specimens of brown and black ceramics (see Fig. 2 and 5) the destruction of the material was observed. Heat treatment at temperatures less than 300 °C it was observed a detaching of particulates of the material from specimen. Heat treatment at higher temperatures leads to destruction of the specimen into several parts. This is caused by the development of several cracks into the specimen when elevating and decreasing of the temperatures of materials.

Experimental data show that when elevating of the heat treatment temperature, the pore space of LFCM evolves (see Figs. 3 and 6). Heat treatment at 100…150 °C leads to the closure of the part of nanoscale pore channels as specimens of brown (see Fig. 3), and black ceramics (see Fig. 6). The subsequent elevating of the temperature up to 150…200 °C leads to a complete opening (in brown ceramics) or to the opening of their most (in black ceramics). Closing of some cracks in brown ceramic occurs into the temperature range 20…200 °C. Closing of cracks in black ceramics is observed when elevating the temperature up to 300…400 °C. Completely closed cracks in brown ceramics are observed at 530 °C temperature. In black ceramics cracks could not close completely till the temperature of 800 °C. Nanopores of black ceramics completely got closed into the temperature range 500…600 °C. Volumes of gas pores both brown and black ceramics do not depend on the heat treatment temperature.

Changes in the volumes and average diameters of the LFCM pore space components under the heat treatment (see Figs. 3 and 6) confirms once again the compliance of stages of the water removal from the pore channels, stage III in brown ceramics and stage II in black – with cracks, and stage IV in brown ceramics and stage III in black – with nanoscale pore channels.

Behavior of nanoscale pore channels clearly indicates that they are the result of combining at least some of α-particle tracks, available in the material. It is known [9] that at the use of method of solid-state nuclear detectors for measuring of the α-active materials activity, the plates of silica glass are applied. After keeping the test samples on the plates and chemical etching at their surface, the number of α-particle tracks is calculated. For subsequent measurements, researchers try to achieve the α-tracks removal from the glass surface by means of heat treatment at 150 °C during several hours.

An estimate of surface value of all the components of the pore space of initial (non-heat-treated) black ceramics – gas pores, cracks, nanoscale channels and nanopores using data on their volume (see Table and Fig. 3 and 6) and an average diameters (or thicknesses) [5] indicates that their surfaces per 1 cm^3 of LFCM are respectively equal to 62, 90, 1700, and 76 cm^2. Pore space components of the specific surface area among the decreasing row are arranged as follows: nanoscale pore channels, cracks, nanopores, gas pores. This correlates with the temperatures when the decrease in volumes of the pore space components is observed due to increasing of the heat treatment temperature. The base temperature of volume reducing of nanoscale channels has a minimum value, 100…150 °C, in comparison with those of other components of the pore space, for the reason, that the nanoscale channels have the largest specific surface. It should also be noted, that reduce in volume at rather low heat treatment temperatures is observed in the components of pore space with a large surface-to-volume ratio – in the nanoscale channels and cracks. They were formed later as a result of internal processes that took place in the materials. Such components of the structure could not be formed at high temperatures, typical for LFCM formation during the accident, such as gas pores. Gas pores having a nearly spherical shape and a relatively large average diameter of a few unity or tens of microns [4, 5] and they do not evolve in the temperature range of heat treatment surveyed.

According to the design-theoretical assessments [10] among the radiation damage in the LFCM caused by all possible emitters of radiation under conditions of the "Shelter" Object (α-, β-particles, γ-quanta, neutrons, etc.), the main contribution to the formation of structure defects is made by damages caused by the α-particles and heavy recoil nucleus. It is assumed, that 90% of all possible radiation defects are provided by heavy recoil nuclei [10]. However, only open nanoscale pore channels, among all components of the pore space are structural defects caused by the self-irradiation of LFCM. They were formed by combining at least a part of α-particle tracks available in the material.

Gas pores formed upon cooling of LFCM by gas release from the melt at LFCM cooling due to the decreasing of their solubility [1–3]. Gas pores in the glass almost always are closed [11]. But in 11 – 12 years after the accident it has been established [12, 13], that LFCM have an open porosity represented by two physically significantly different size of pores: macro- and ultramicroscopic. Apparently, at that time, the nanosized pore channels had already been started to form, which have connected close gas pores with the environment. Already at that time, it has been suggested that the formation of ultramicropore was associated with radiation-induced defect formation [13].

Cracks have formed later, in approximately 2004–2011, at the expense of increased crystalline inclusions of uranium oxides during their oxidation, as evidenced by significant reductions in the mechanical properties of LFCM [14].

CONCLUSIONS

Studies conducted have shown that the components of the pore space of brown and black ceramics of LFCM inside the "Shelter" Object could evolve as a result of heat treatment. When elevating of the temperature the partial closure of nanoscale pore channels takes place at 100…150 °C in brown and black ceramics, of cracks at 200 °C in brown and at 400 °C in black ceramics, complete closure of nanoscale pore channels at 400 °C and of cracks at 530 °C in brown ceramics and of nanopores at 600 °C in black ceramics. The driving force behind the process of reducing the volumes of pore space is tendency to reducing the surface energy by reducing their specific surface area.

The Nature of formation of nanoscale open pore channels in LFCM installed. Nanochannels are the result of combining at least a part of α-particle tracks formed in LFCM at the expense of α-decay of transuranic elements during the self-irradiation for a long period (about ten years). Behavior during heat treatment (partial closure) of nanoscale pore channels in LFCM coincides with the behavior of the α-particles tracks in the silicate glass used for measuring the activity of specimen containing transuranic radionuclides in the method of solid-state nuclear detectors.

It is shown that nanoscale pore channels and cracks are the components of LFCM pore space with a large ratio of surface-to-volume. This clearly indicates that cracks inside the material (not on the surface) and nanoscale channels could not be a consequence of "preparing" of LFCM during the accident but formed later as a result of internal processes, as previously held and undergoing right now inside the LFCM .

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Article received 20.01.2015

ПРИРОДА ФОРМИРОВАНИЯ НАНОРАЗМЕРНЫХ ПОРОВЫХ КАНАЛОВ ЛАВООБРАЗНЫХ ТОПЛИВОСОДЕРЖАЩИХ МАТЕРИАЛОВ ОБЪЕКТА «УКРЫТИЕ»

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Исследована эволюция составляющих порового пространства – коричневой и черной керамик лавообразных топливосодержащих материалов объекта «Укрытие» при термообработке. С повышением температуры часть наноразмерных поровых каналов закрывается при 100…150 °С, часть трещин – при 200 и 400 °С, полностью закрываются наноразмерные каналы при 400 °С, трещины – при 530 °С, а нанопоры – при 600 °С. Открытые наноразмерные каналы являются результатом объединения, по крайней мере, части треков α-частиц, образовавшихся за счет α-распада трансурановых радионуклидов при самооблучении в течение длительного периода времени. Наноразмерные поровые каналы и трещины сформировались в результате процессов, проходящих в материалах значительно позднее их «приготовления» во время аварии.

ПРИРОДА ФОРМУВАННЯ НАНОРОЗМІРНИХ ПОРОВИХ КАНАЛІВ ЛАВОПОДІБНИХ ПАЛИВОВМІСНИХ МАТЕРІАЛІВ ОБ'ЄКТУ «УКРИТТЯ»

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Досліджено еволюцію складових порового простору коричневої та чорної керамік лавоподібних паливовмісних матеріалів об'єкту «Укриття» при термообробці. З підвищенням температури частина нанорозмірних порових каналів закривається при 100…150 °С, частина тріщин – при 200 і 400 °С, повністю закриваються нанорозмірні канали при 400 °С, тріщини – при 530 °С, а нанопори – при 600 °С. Відкриті нанорозмірні канали є результатом об'єднання принаймні частини треків α-часток, що утворилися за рахунок α-розпаду трансуранових радіонуклідів при самоопроміненні протягом тривалого періоду часу. Нанорозмірні порові канали та тріщини сформувалися в результаті процесів, що проходять в матеріалах значно пізніше їх «приготування» під час аварії.