SMALL ANGLE NEUTRON SCATTERING BY U-9%Mo/Al DISPERSION FUEL IRRADIATED TO HIGH BURN-UPS

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ABSTRACT

In U-Mo/Al dispersion fuel under irradiation a $(U,Mo)Al_x$ interaction layer is formed along with the pores, which contain gas fission products. The gas pores are formed in fuel particles, the $(U,Mo)Al_x$ layer and the aluminum matrix, that is near the $(U,Mo)Al_x$ layer. A structure of pores is mainly studied by using optical and scanning electron microscopy to allow an examination of pores with sizes better than tenth parts of microns.
In this paper the influence of burn up and isochronal annealing at temperatures ranging from 150 to 580 ^{o}C on changes in the substructure characteristics of the U-Mo/Al dispersion fuel is investigated. The investigations were conducted by using the small angle neutron scattering that allows to study the substructure characteristics of the fuel in the range from 1 to 50 nm.

1. Introduction

In the U-Mo/Al dispersion fuel under irradiation a structure of the fuel material changes, new phases (in particular, a $(U,Mo)Al_x$ interaction layer between an Al matrix and fuel particles) as well as gas pores form. At certain fuel burn up values all these changes affect the behavior of the U-Mo dispersion fuel under reactor operation conditions and result in an increase of a fuel temperature and a undue onset of accelerated swelling in fuel elements at higher irradiation parameters.

The influence of the irradiation parameters on the changes in the structure of the U-Mo/Al dispersion fuel is given much attention by researchers. However, only traditional techniques (the optical metallography, SEM, X-ray spectrum microanalysis and neutron diffraction) are used to characterize the fuel materials. These techniques allow to study fuel imperfections of a size not better than tenth parts of micrometer, however, such structural imperfections like gas pores or new phase nucleation originate from imperfections of a much smaller size.

In this work the small-angle neutron scattering method was used to study the influence of a burn up of 33 to 97 % and isochronal annealing for 1 hour over the range of 150 to 580 ^{o}C on the changes in the substructure characteristics of irradiated U-Mo/Al dispersion fuel.

2. Materials and experimental methods

For the investigations the plates of size 40x8x1.3 mm were cut at different positions of the FEs of combined FAs designated as KM003 and KM004, which were tested in the IVV-2M reactor to reach a mean equivalent burn up of 40 and 60 %, respectively [1, 2]. Each plate had two fuel claddings of thickness 0.45 mm and one layer of fuel meat of thickness 0.45 mm. Three such plates made a specimen for the small-angle neutron scattering experiments. Thus, the specimens consisted of six fuel claddings and three layers of fuel meat.

Two specimens were cut from the KM003 fuel element: one (G13) was taken at the top and the other (G100) was taken at the center. Four specimens were cut from the KM004 fuel element, they were taken at the top (G96), at the center (G97), at the bottom (G99) and from the area directly adjacent to the bulging region in the cladding (G98). In the G98 fuel meat

individual gas pores and their clusters as well as small cracks of length 1.5 to 2 mm were observed. The characteristics of the specimens are given in Table 1.

Table 1. Characteristics of the specimens

Speci-men #	As-sembly #	Bu[1)], %	φ, 10^{14} fis/(cm^3·s)	ψ, 10^{21} fis/cm^3	T_{BOL}, °C	q_{BOL}, W/cm^2	IL[2)], μm	Volumetric fraction of phases in specimen, %			
								Al	U-Mo	IL	Al_{FP}[3)]
G13	KM003	32.9	2.6	2.1	43.5	48	1.2	87.0	12.1	0.9	13.4
G100	KM003	51.1	4.5	3.8	61.4	80	3.6	85.6	11.6	2.8	14.2
G96	KM004	55.1	3.1	4.1	48.6	66	3.5	85.7	11.6	2.7	14.2
G97	KM004	96.3	5.5	7.2	77.2	115	11.3	80.7	10.1	9.2	16.9
G98	KM004	96.9	5.7	7.3	85.3	118	11.2	80.8	10.1	9.1	16.8
G99	KM004	78.4	4.6	5.9	86.4	95	9.0	82.3	10.5	7.2	16.0

[1)] equivalent burn up;
[2)] (U,Mo)Al_x layer thickness;
[3)] aluminum matrix damaged by fission fragments.

The experiments on the small-angle neutron scattering were performed at the IVV-2M reactor (Zarechny, Russia). In the experiments two diffractometers "D6" and "D3" with the scattering vector intervals q=0.06-1 nm^{-1} and q=0.6-6 nm^{-1}, respectively, were used. The usage of two diffractometers permits to extend the size range of the investigated fuel substructure characteristics from 1 to 50 nm. The experiments were conducted in two stages. At the first stage the spectra of all the specimens were obtained. At the second stage three specimens (G96, G98 and G99) underwent stepwise annealings for 1 hour over the temperature range T_{an} from 150 to 580° C with a step equal to 50°°C. Neutron diffraction patterns were obtained after each annealing.

3. Experimental results

Influence of burn up. Figure 1 shows the dependence of the small-angle neutron scattering cross-sections $d\Sigma/d\Omega$ on the wave vector $\boldsymbol{q}$ obtained in the experiments for the specimens irradiated to different burn up values.

According to these data the curve $d\Sigma/d\Omega(q)$ changes its shape with burn up and that is proved by the following:

- for small values of q<0.5 nm^{-1} it is noticed that values $d\Sigma/d\Omega$ increase with burn up in the whole interval of values under study;
- at burn up less than Bu≥96% a peak with a maximum at $q_{max} \cong 0.8$ nm^{-1} is observed on the neutron scattering curves $d\Sigma/d\Omega(q)$,
- the values $d\Sigma/d\Omega$ corresponding to a maximum of the peak increase in the burn up range from 33 to 78 %;
- at Bu>78 % a value of the peak decreases;
- for the specimen G98 with Bu≅97 % a peak is absent.

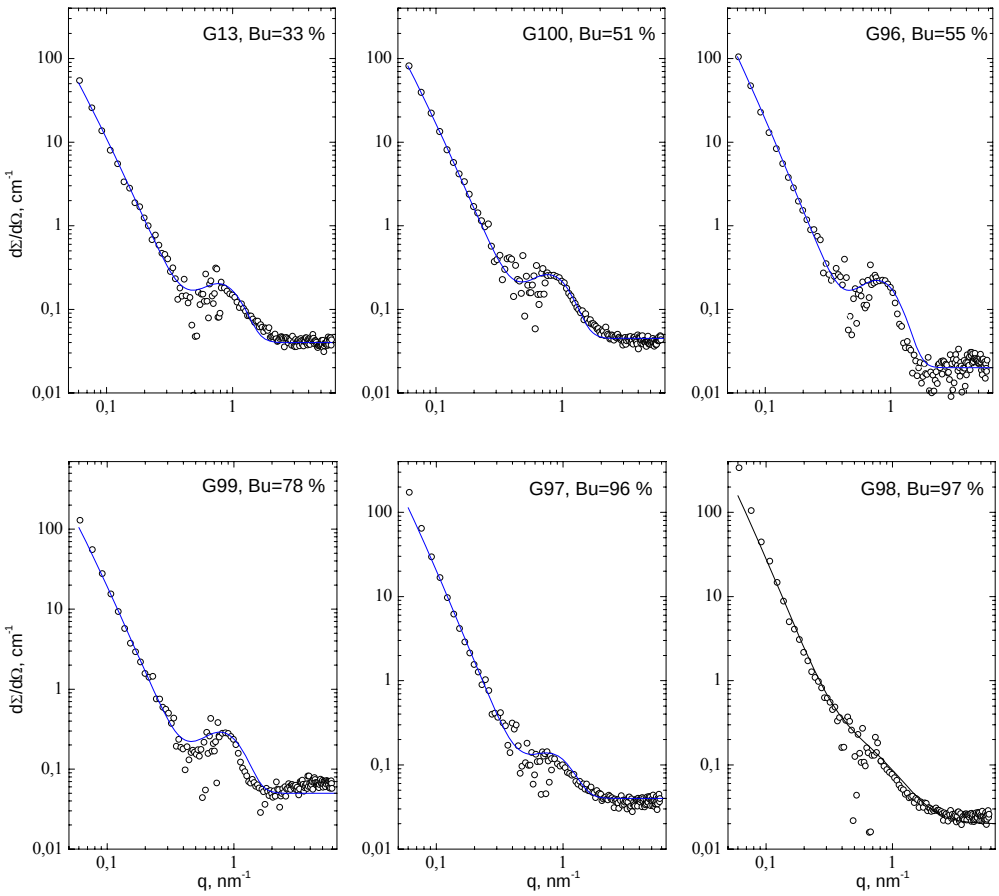

Fig. 1. Small-angle neutron scattering cross-sections versus wave vector for specimens irradiated to different burn up values

Influence of annealing temperature. Figures 2, 4 and 5 show neutron scattering curves $d\Sigma/d\Omega(q)$ versus annealing temperatures (typical dependencies on selected temperatures are given) for specimens G96, G98 and G99. A change in the shape of the curves $d\Sigma/d\Omega(q)$ with the annealing temperature is observed for all the specimens.

Specimen G96. It is observed that in G96 at $T_{an}\leq 300$ °C the values $d\Sigma/d\Omega$ corresponding to the scattering vectors $q<0.5$ nm^{-1} increase with the annealing temperature; but the peak position and the values of $d\Sigma/d\Omega$, which correspond to the maximum on the peak, don't change. A growth of annealing temperature to 350 ^{o}C leads to some decrease in the values of $d\Sigma/d\Omega$ over the range of small values of q and practically with no influence on the shape and the position of the peak. A further growth of T_{an} to 450 and 550 ^{o}C in the range of small values of q results in the increase of the values $d\Sigma/d\Omega$. Those $d\Sigma/d\Omega$ values, which correspond to the peak, begin to decrease at the T_{an} of 450 °C, and the peak itself completely disappears after annealing at 550 ^{o}C for 1 hour; and the shape of the curve $d\Sigma/d\Omega(q)$ changes in the range of the scattering vectors from 0.05 to 1.0 nm^{-1}, i.e. it becomes convex (Fig. 2).

In addition to the above changes pillowing of the cladding were found in the two plates of the specimen G96 after annealing at 550 ^{o}C (Fig. 3). The pillowing occurred on one side of the plates and had a semisphere shape. A pillowing on one specimen had a diameter of ~4 mm and its height above the cladding was ~1.3 mm, a pillowing on the other specimen had a diameter of ~1.5 mm and its height was ~0.3 mm.

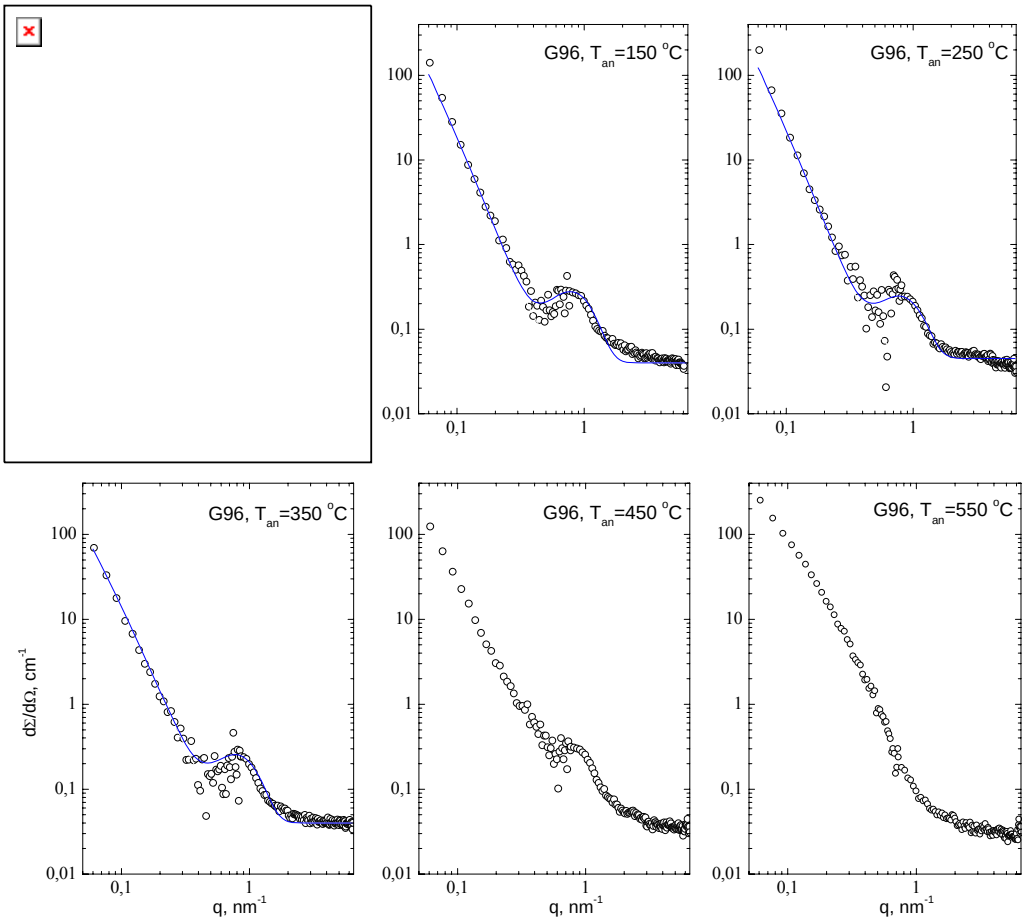

Fig. 2. Influence of annealing temperature of specimen G96 on shape of curves $d\Sigma/d\Omega(q)$

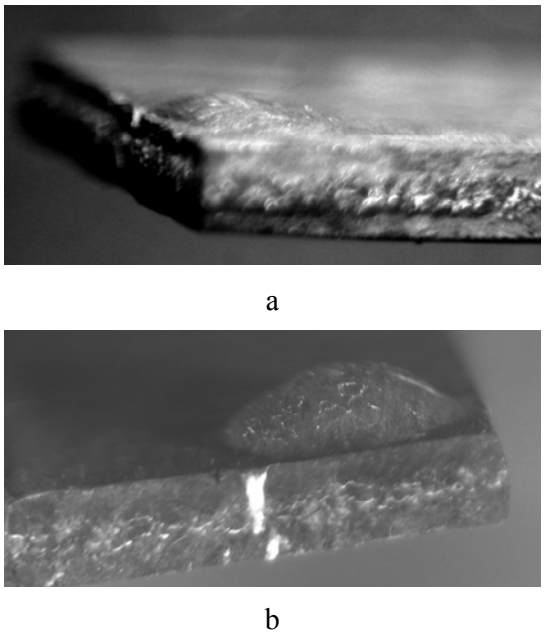

Fig. 3. Appearance of cladding bulges in plates G96/1 (*a*) and G96/2 (*b*), formed after annealing at 550 °C

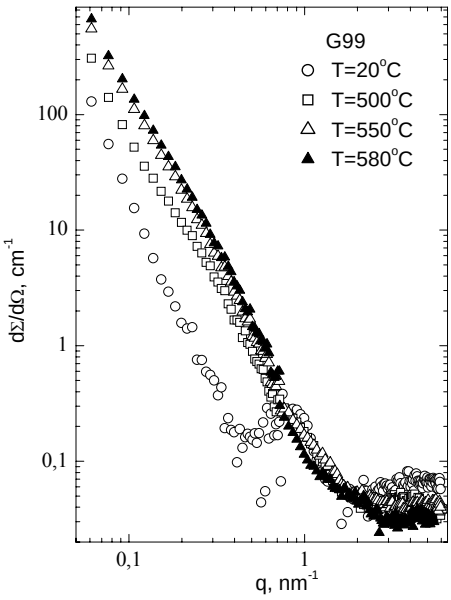

Fig. 4. Influence of annealing temperature on shape of curves $d\Sigma/d\Omega(q)$ of specimen G99

Specimen G99. It was assumed that the peak on the curve $d\Sigma/d\Omega(q)$ of the specimen G96 had disappeared at a temperature lower than 550 ^{o}C. Therefore it was decided to investigate the specimen G99, whose burn up value was ~1.5 times higher than that of G96 and also G99 has a peak on its curve in the region $q_{max} \cong 0.8$ nm^{-1} (Fig. 1). Actually after the annealing of G99 for 1 hour at 500 ^{o}C the peak disappeared from the curve $d\Sigma/d\Omega(q)$ and the shape of the curve in the scattering vector region q<1.0 nm^{-1} became also convex (Fig. 4). The increase in the values of $d\Sigma/d\Omega$ and convexity on the curve $d\Sigma/d\Omega(q)$ were observed at annealing temperatures of 550 and 580 ^{o}C (Fig. 4).

Specimen G98. The specimen G98 had no peak on the curve $d\Sigma/d\Omega(q)$ after irradiation and revealed the similar dependencies of changes in the values $d\Sigma/d\Omega$ and shape of the curve in the region of small values of q as the specimens G96 and G99 did (Fig 5).

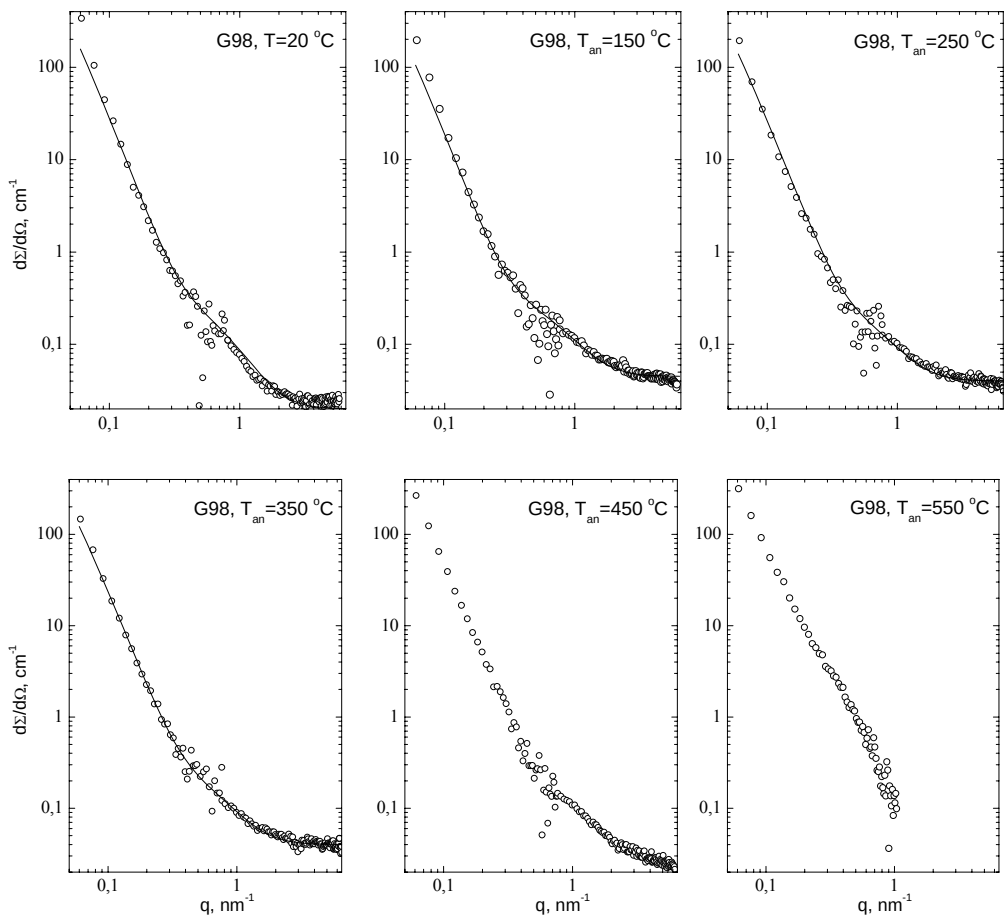

Fig. 5. Influence of annealing temperature of specimen G98 on shape of curves $d\Sigma/d\Omega(q)$

4. Discussion of experimental results

The analysis of the experimental data make evidence that there are two main types of dependencies $d\Sigma/d\Omega(q)$. The first type comprises relatively smooth curves observed for the specimen G98 as irradiated and annealed and those obtained for specimens G96 и G99 after annealing at temperatures higher than 500 ^{o}C. The second type includes the dependencies $d\Sigma/d\Omega(q)$ which are similar to those of the first type but with a peak in the scattering vector

region $q \cong 0.8$ nm^{-1}. These dependencies are observed practically for all the specimens (except G98) as irradiated and annealed at temperatures lower than 500 ^{o}C.

We used two models for an interpretation of the experimental data of the first type dependencies: the model of a random size-distribution of smooth spherical particles [3] and the empirical interpolation by the Debay-Porod formula [4], obtained to an assumption of a random distribution of solid (or gas) phase in space. The first model suggests that the size distribution of particles R is in the form $(R_{min}/R)^{3+\Delta}$ (where R_{min} is a minimal radius of particles). This leads to the power law of scattering $I \sim q^{-(4-\Delta)}$**,** which well describes the left part of the post irradiation curves and those of low temperature annealing (T_{an}<350 ^{o}C) for all the specimens (curves 2 and 5 in Fig. 6).

We obtain the power law of distribution $I \sim q^{-n}$ at $q \to \infty$ from the Debay-Porod formula and a deviation from this law at small angles of scattering (curve 2 in Fig. 6). The formula was used to describe the curves obtained for the specimens annealed at a temperature higher than 350 ^{o}C and it was obtained as the sum of two functions with different parameters. This testifies to the presence of two typical sizes of the particles in these specimens.

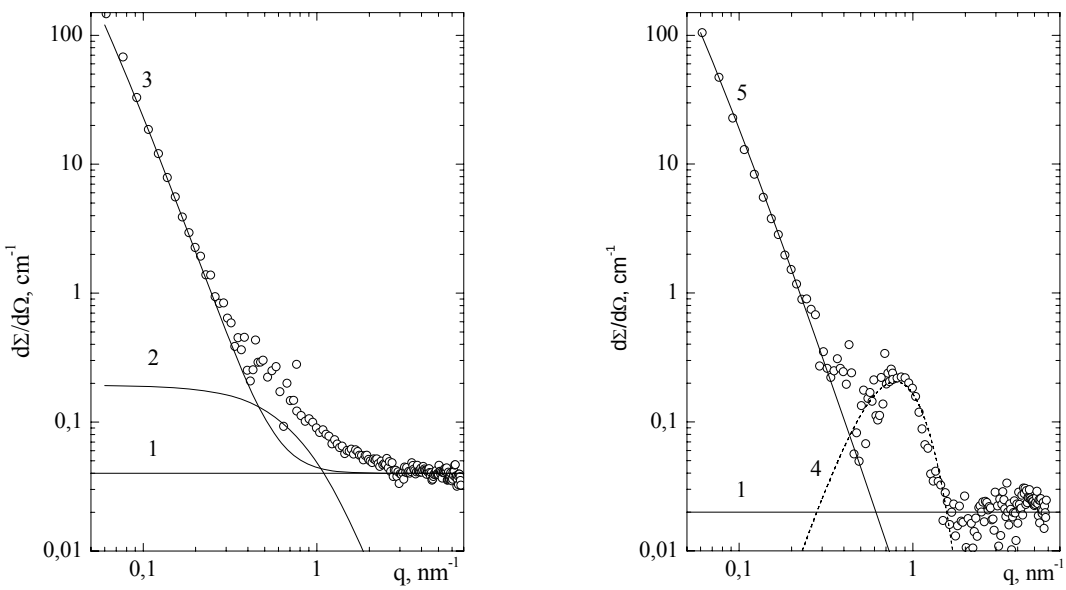

Fig. 6. Three components of model scattering cross-sections for specimen G98 annealed at 350 ^{o}C (*a*) and those for specimen G99 after irradiation (*b*): (*1*) - incoherent scattering cross - section: (*a*) 0.04cm $^{-1}$, (*b*) 0.02cm $^{-1}$; (*2*)- contribution from monodispersed particles of size 2 nm; (*3*) - scattering cross-sections from polydispersed particles with a mean size 7.8nm plus incoherent constituent (*1*); (*4*) - contribution from Genie -Preston zones; (*5*) - scattering cross-sections from polydispersed particles with a mean size 8.8 nm.
Dots designate the experimental data and lines designate the calculation data.

All the specimens show the incoherent background of the order of several hundredth parts of a reverse centimeter (curve 1 in Fig. 6).

We use a size and a concentration of particles, a power index *n* and a value of an incoherent constituent of a scattering cross-section as fitting parameters to describe the experimental data of the model functions.

We used the model of the Genie-Preston zones [5] to describe the peak at the small angles on the curves of the second type. As is known the Genie-Preston zones are formed at the initial stages of decomposition of supersaturated solid solutions [6]. When being heated or annealed during a long time, these zones disappear and stable phase precipitates occupy their place.

Let's explain the maximum at the small angles as a result of neutron scattering on the Genie-Preston zones. A great number of gas fission products (GFP) are generated during a fuel element operation, later they can make couples, triples and etc., and finally they form pores. It is assumed that porosity can be formed by the Genie-Preston zone mechanism. For certainty let's consider that this process takes place in an Al matrix. A kernel of the zone is a sphere of radius R_1, and it is rich in GFP atoms unlike the Al matrix, where a concentration of GFP is substantially lower. The kernel is surrounded by a spherical layer of radius $R_2>R_1$. A density of a GFP in this layer is lower than in the matrix. Since we do not know compositions of the zone kernel and of its environment let's set them tentatively provided these compositions permit us to describe quantitatively the observed neutron scattering.

Let's assume that Al atoms occupy all the sites in the zone cladding (i.e. a spherical layer between R_1 and R_2) and the zone kernel is rich in the GFP atoms and its composition is $Al_{0.75}(Kr,Xe)_{0.25}$. According to Ref. [6] amplitude of neutron scattering by «a complicated particle», which represents the Genie-Preston zone, is equal to

$$F = (4\pi R_2^3/3)(\rho_{sl} - \rho_{G\text{-}P}) [\exp(-q^2R_1^2/10) - \exp(-q^2R_2^2/10)]. \quad (1)$$

Then a differential cross-section of neutron scattering by a unit volume of a specimen, which contains $N_{G\text{-}P}$ zones, is equal to

$$d\Sigma/d\Omega = (N_{G\text{-}P}/V)F^2 = (4\pi R_2^3/3)\cdot C\cdot(\rho_{sl} - \rho_{G\text{-}P})^2 [\exp(-q^2R_1^2/10) - \exp(-q^2R_2^2/10)]^2 , \quad (2)$$

where $C=V_{G\text{-}P}\cdot N_{G\text{-}P}/V$ is the volume part occupied by the zones; $V_{G\text{-}P}$ is the effective volume of the zone, V is the volume of a specimen and q is the scattering vector, ρ_{sl} and $\rho_{G\text{-}P}$.- density of a spherical layer (ρ_{sl} is equal to ρ_{Al}) and the Genie-Preston zones accordingly.

As it follows from formula (2), the scattering cross-section is equal to zero at $q = 0$ and it is maximal at some value $q = q_{max}$, and then it decreases with a growing q.

Thus, the model function for the second type curves also is a sum of three components: the first is based on the Debay-Porod equation for a unimodal distribution of particles, the second is a contribution from the scattering on the Genie-Preston zones and the third one is a contribution from the incoherent scattering which is the same in the whole range of the vectors q (Fig. 6.*b)*.

We can estimate the size of the R_1 zone kernel from the condition $\partial(d\Sigma/d\Omega)/\partial q=0$ at $q=q_{max}$, if we assume that $2R_1 > R_2 > R_1$. This condition is typical for the observed cases of the scattering on the Genie-Preston zones [6]. For estimations we take $R_2=1.8R_1$. From the maximum cross-section value condition (2) at $q=q_{max}$ we obtain that

$$[R_2^2\exp(-q_{max}^2R_2^2/10) - R_1^2\exp(-q_{max}^2R_1^2/10)] = 0,$$

or

$$q_{max}^2 = 20\cdot\ln(R_2/R_1)[R_1^2(R_2^2/R_1^2 - 1)]^{-1}. \quad (3)$$

The position of the curves obtained for all the specimens under study (including those after annealing) was found for one and the same value of the wave vector q_{max} = 0.8 nm^{-1}. Thus, from (3) at q_{max}=0.8 nm^{-1} we calculate the sizes of the Genie-Preston zones R_1=2.9 nm, R_2=5.2 nm at R_2/R_1 = 1.8, that are practically equal for all the specimens.

The substructure characteristics of the specimens as irradiated and annealed in a temperature range from 150 to 580 ^{o}C are presented in Tables 2 to 5, where the following values: $2R_{min}$ and $2\overline{R}$ were used for a minimal and mean size of the polydispersed set of particles, respectively, c and C are the composition of particles and that of the Genie-Preston zones, respectively, $\Delta = 4-n$, n is the power index in the law of scattering, N is the concentration of particles, l is the diameter of the monodispersed set of particles and, D_s is the factor characterizing fractal structure of particles. For the analysis of the $d\Sigma/d\Omega$ curves it was assumed that:

- the main shape of the curves and their change with burn up and annealing temperature are determined by the presence of the particles of UAl_3 crystalline phase. The small quantity of this phase present on the specimens as irradiated (~0.5 %) was determined by the neutron diffraction method and this quantity was growing with the annealing temperature higher than 350°C.
- The gas pores and the Genie-Preston zones as predecessors of the pores formation are responsible for a presence or absence of the peak on the curve $d\Sigma/d\Omega$ q_{max}=0.8 nm^{-1}.

Table 2. Characteristics of UAl_3 particles and Genie-Preston zones in specimens as irradiated

Specimen #	Burn up, %	Characteristics of UAl_3 particles					Genie-Preston zones
		$2R_{min}$, nm	$2\overline{R}$, nm	c, %	3+Δ	N, cm^{-3}	C, %[1)]
G13	33	4	6.6	0.12	3.7	$1.2\cdot10^{15}$	6.0
G100	51	4	7.0	0.19	3.3	$1.2\cdot10^{15}$	8.0
G96	55	4	8.8	0.14	2.75	$2.2\cdot10^{14}$	8.0
G97	96	4	10.3	0.12	2.5	$1.0\cdot10^{14}$	3.5
G98	97	4	9.6	0.20	2.6	$1.1\cdot10^{15}$	0 (2.0[2)])
G99	78	4	8.6	0.17	2.8	$3.0\cdot10^{14}$	9.0

[1)] – content of Genie -Preston zones of sizes R_1=2.9 nm, R_2=5.2 nm
[2)] - content of gas pores of size l = 2.0 nm

According to the data in Table 2 there are the UAl_3 particles in the irradiated fuel structure; the particle content increases by ~2 times and sizes by ~1.5 times with burn up increasing from 33 to 97 %. However, a maximal content of UAl_3 particle is no more than 0.2 % of the fuel meat volume and a maximal size of the particles does not exceed 10.5 nm. A concentration N of UAl_3 particles is within $(1\text{-}3)\cdot10^{14}$ to $1.2\cdot10^{15}$ cm^{-3}. Besides, there are the Genie-Preston zones in the irradiated fuel structure, their content increases with burn up and reaches a maximum at Bu=78 %, then it decreases almost to zero at Bu=97 %.

The annealing of the 55% burn up specimen G96 over a temperature range of 150 to ~300 ^{o}C practically effects neither the size nor the content of the UAl_3 particles (Table 3). Initial changes in size are noticed with an annealing temperature increase to 350° ^{o}C and then the content of the UAl_3 particles grows dramatically from ~0.14 % at 350 ^{o}C to ~1.1 % at 450 ^{o}C. At the same time the occurrence of small particles of ~4.0 nm (c=0.6 %) and large ones of ~50 nm (c=0.5 %) is noticed. A further increase in the annealing temperature to 550 ^{o}C leads to a size-redistribution of the UAl_3 particles and growth of their content to ~2.3 %. This value is ~1/3 of the overall content of the $(U,Mo)Al_x$ interaction layer in the fuel meat of G96, this content is equal to 2.7·3=8.1 % (Table 1). Thus, one may believe that only ~1/3 of the content of the $(U,Mo)Al_x$ layer has transformed into the UAl_3 phase.

Table 3. Substructure characteristics of specimen G96 as irradiated and annealed at temperatures lower than 350 °C

Temperature, °C	Characteristics of UAl_3 particles					Genie - Preston zones
	$2R_{min}$, nm	$2\overline{R}$, nm	c, %	3+Δ	N, cm^{-3}	C, %[1)]
20	4	8.8	0.14	2.75	$2.2\cdot10^{14}$	8.0
150	4	8.8	0.14	2.75	$2.2\cdot10^{14}$	9.0
250	4	8.8	0.19	2.75	$3.0\cdot10^{14}$	7.5
300	4	8.8	0.14	2.75	$2.2\cdot10^{14}$	10.0
350	4	6.8	0.14	3.4	$1.1\cdot10^{15}$	8.0
[1)] – content of Genie-Preston zones of sizes R_1=2.9 nm, R_2=5.2 nm						

Table 4. Substructure characteristics of specimen G98 as irradiated and annealed at temperatures lower than 350 °C

Temperature, °C	Characteristics of UAl_3 particles					Gas pores
	$2R_{min}$, nm	$2\overline{R}$, nm	c, %	3+Δ	N, cm^{-3}	C, %[1)]
20	4	9.6	0.20	2.6	$2.0\cdot10^{14}$	2.0
150	4	9.6	0.16	2.6	$1.5\cdot10^{14}$	1.7
250	4	8.6	0.17	2.8	$3.0\cdot10^{14}$	2.0
300	4	8.6	0.25	2.8	$4.5\cdot10^{14}$	1.2
350	4	7.8	0.25	3.0	$7.5\cdot10^{14}$	1.7
[1)] – content of gas pores of size l=2.0 nm						

Table 5. Substructure characteristics of specimens G96, G98 and G99 as annealed at temperatures higher than 350 °C

Specimen #	Temperature, °C	Characteristics of UAl_3 particles						Genie-Preston zones
		Small particles			Large particles			
		l, nm	c, %	D_s	l, nm	c, %	D_s	C, %[1)]
G96	450	4	0.6	2.0	50	0.5	2.0	9.0
	550	14	1.5	2.0	30	0.8	2.3	-
G98	400	9.5	0.3	2.0	70	0.6	2.0	-
	450	7	0.1	2.0	50	1.0	2.0	-
	550	6	1.0	2.0	100	2.0	2.4	-
G99	500	9.5	1.2	2.0	100	1.5	2.0	3.5
	550	8	1.7	2.0	50	2.0	2.2	-
	580	3.6	2.0	2.0	100	7.0	2.8	-
[1)] – content of Genie-Preston zones with sizes R_1=2.9 nm. R_2=5.2 nm								

In contrast to the UAl_3 particles, the size and content of the Genie-Preston zones in G96 remain intact until they experienced temperatures up to 450 °C (Table 5). The annealing of G96 at 550 °C resulted in the disappearance of the Genie-Preston zones, which had formed in the specimen under irradiation and annealing temperatures <550 °C. A destruction of the zones leads to the formation of gas pores of size ~2.0 nm.

In the 97% burn up specimen G98 after irradiation the content of the UAl_3 particles was larger by ~1.5 times than that of G96 and the particles had a larger size (Table 2). The sizes

and the content of the particles do not practically change before reaching a temperature of ~300 ^{o}C. Then the content of the UAl_3 particles increases and the sizes decrease with a simultaneous growth of UAl_3 particles content by ~2.5 times. A further increase of the temperature to 400, 450 and 550 ^{o}C leads to a size-redistribution of the particles and an increase of their content to ~3.0 % at an annealing temperature of 550 ^{o}C. It should be noted that this value is ~1/9 of the content of the $(U,Mo)Al_x$ layer in the fuel meat of G98 (Table 1). The Genie-Preston zones have not been found in the structure of G98 after annealing over a temperature range from 150 to 550 ^{o}C. The absence of the zones in G98 as irradiated suggests that the damage of the zones has already occurred during the irradiation of the specimen.

The 78 % burn up specimen G99 reveals the same thermally activated processes of UAl_3 particle formation as in G96 and G98. Besides, for G99 the temperature, at which the content of the Genie-Preston zones started to decrease, was detected. Thus, the Genie -Preston zones content after annealing at 500 ^{o}C decreased from 9 % after irradiation to 3.5 % after annealing and the zones completely disappeared after annealing at 550 ^{o}C.

5. Conclusions

It was found that the structure of the irradiated U-9 % Mo fuel is characterized by the presence of the UAl_3 particles and inhomogeneities of the type of Genie-Preston zones, which are the predecessors of the gas pores formation in the aluminum matrix.

It was shown that the content and size of the UAl_3 particles increase with burn up. At the annealing temperature to ~300 ^{o}C the content and the size of the UAl_3 particles practically does not change and at annealing temperature higher than 350 ^{o}C the content of the particles increases along with the redistribution of the sizes.

It was found that the Genie-Preston zones are stable formations up to ~500 ^{o}C and at the temperatures higher than that they are damaged to form pores.

6. References

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