

PHASE FORMATION AND CHARACTERISTICS OF DYSPROSIUM TITANATE PELLETS PREPARED BY SOLID STATE SYNTHESIS

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Dysprosium titanate have been developed and used as absorbing material for control rods of thermal neutron nuclear reactors. The study results of phase formation kinetics in the pellets prepared from powder mixtures composition 1 (50 mol.% TiO_2 + 50 mol.% Dy_2O_3) and composition 2 (56 mol.% TiO_2 + 44 mol.% Dy_2O_3) after sintering at temperatures of 1250...1650 °C in an air are presented. It is shown that initial mixture composition affects the phase composition of sintering dysprosium titanate pellets. Also, it was revealed the formation of high-temperature radiation-resistant fluorite phase of Dy_2TiO_5 using composition 1 powder mixture. Two-stage sintering with partial synthesis allows obtaining fine-grained dysprosium titanate pellets with high density of 7.1 g/cm³ and low open porosity.

INTRODUCTION

Radiation tests and post-radiation studies of absorbing material have revealed that ceramics based on lanthanide oxides have high radiation damage resistance [1, 2].

Dysprosium titanate ($\text{Dy}_2\text{O}_3 \cdot \text{TiO}_2$) as a material of this class of ceramics got sufficiently wide usage. Since 1995, after preliminary tests in reactor MIR, it has been successfully applied as a vibrocompacted powder in absorbing elements of the rod cluster control assembly (RCCA) of the WWER-1000 reactor [3–5].

Except Russia, investigations of properties of dysprosium oxide based absorbing materials are also performed in such countries as India, South Korea and China [5–7].

In general, compositions with 78...82 wt.% Dy_2O_3 content are considered. One of the ways to improve the absorbing elements physical efficiency is a transition to a pellet type of the absorbing core that provides density increasing of $\text{Dy}_2\text{O}_3 \cdot \text{TiO}_2$ from 4.9 to 6.2 g/cm³ and, accordingly, increases both initial efficiency of RCCA and their exploitation terms [5].

It is of interest to investigate phase composition and properties of dysprosium titanate based materials obtained by high-temperature solid-phase synthesis of native oxides in air.

Therefore the aim of this work is to study the phase composition and characteristics of dysprosium titanate based absorbent material after sintering at temperatures in the range between 1250 and 1650 °C.

1. MATERIALS AND EXPERIMENTAL PROCEDURES

According to the phase equilibrium diagram (Fig. 1) two type of compounds are crystallized in the Dy_2O_3 - TiO_2 system: dysprosium titanates, $\text{Dy}_2\text{Ti}_2\text{O}_7$ and Dy_2TiO_5 [8].

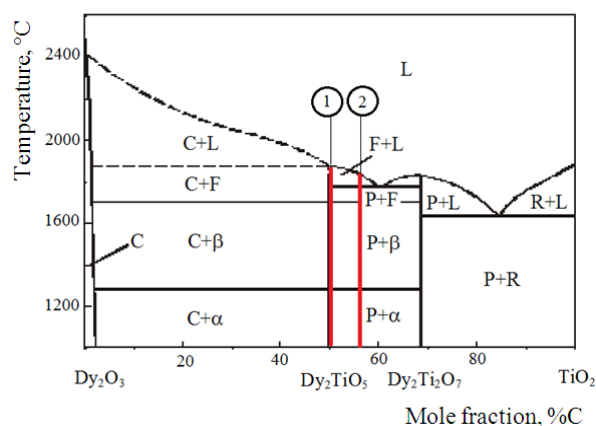


Fig. 1. Phase diagram of Dy_2O_3 - TiO_2 system:

F – fluorite Dy_2TiO_5 ; P – pyrochlore $\text{Dy}_2\text{Ti}_2\text{O}_7$;
α – orthorhombic Dy_2TiO_5 ; β – hexagonal Dy_2TiO_5 ;
R – rutile TiO_2 ; C – solid solution based on Dy_2O_3 ;
L – liquid

Investigations made on powders obtained by dysprosium and titanium hydroxides sediments coprecipitation from hydrochloric acid solutions with ammonium hydroxide testify that dysprosium titanate $\text{Dy}_2\text{Ti}_2\text{O}_7$ crystallizes at 700 °C in a pyrochlore structure and melts congruently at 1850 °C [9]. $\text{Dy}_2\text{Ti}_2\text{O}_7$ compound has FCC lattice (pyrochlore structure type) with parameter $a = 1.0132$ nm. A specific feature of pyrochlore-type phase is non-stoichiometry, which appears by partial substitution of Ti^{4+} ions with Dy^{3+} ions (at the same time additional vacancies are formed in the anion sublattice) [1].

Dysprosium titanate Dy_2TiO_5 crystallizes at 800 °C and has three polymorphs depending on temperature. At low temperatures it exists in orthorhombic modification, above 1350 °C it transforms to hexagonal one, which at 1680 °C changes to fluorite solid solution melting at 1870 °C.

The liquidus curve of the system is characterized by two eutectics at 15 and 40 mol.% Dy_2O_3 , which melt at 1650 and 1750 °C, respectively.

Unit cell parameters of Dy_2TiO_5 polymorphs are given in Tabl. 1.

Table 1
Unit cell parameters of Dy_2TiO_5 polymorphs

Structure	Lattice parameters, nm		
	a	b	c
Orthorhombic	1.049	1.126	0.370
Hexagonal	0.3632	–	1.1837
Cubic	0.5200	–	–

The investigation of phase formation in Dy_2O_3 - TiO_2 system under solid-phase synthesis carried out on two compositions of powder mixtures:

– composition 1: 50 mol.% TiO_2 + 50 mol.% Dy_2O_3 (17.6 wt.% TiO_2 + 82.4 wt.% Dy_2O_3);

– composition 2: 56 mol.% TiO_2 + 44 mol.% Dy_2O_3 (21.4 wt.% TiO_2 + 78.6 wt.% Dy_2O_3) (see Fig. 1).

Titanium oxide (ОСЧ 7-3, purity 99.9%, ТУ 6-09-3811-79) and dysprosium oxide (ДнО-М, purity 99.5%, ТУ 48-4-524-90) were used as initial powders.

Pellets were prepared from powder mixtures according with two schemes. The first one includes the following steps: mixing titanium and dysprosium oxides, blending with a binder, compacting and sintering pellets. The second one includes additional operations to the scheme 1 such as: grinding pellets after preliminary sintering, adding binding material, compacting and final sintering.

Grinding and mixing of powder mixtures was performed in a ball mill “Pulverisette-6” at 300 rpm for 3 hours in ethanol at weight balls-powder ratio 3:1. Pellets forming was performed by uniaxial pressing in a steel press-form Ø 8.4 mm at 200 MPa. Polyethylene

glycol was used as a binder material. Pellets were sintered in air at 1250...1650 °C for 3 hours. Pre-annealing of pellets was carried out at 1350 °C in a case of scheme 2.

Phase analysis of pellets was studied by X-ray diffraction using DRON-2.0 diffractometer in $\text{Co-K}\alpha$ radiation.

Microstructure investigations were performed using scanning electron microscope JSM-7001F (JEOL, Japan). Sample composition analysis was performed by energy-dispersive X-ray microanalysis (EDXMA) using INCA Penta FETx3 analyzer (Oxford Instruments, Great Britain) with a detection limit not less than 0.3 wt.%.

Density and open porosity of pellets were measured in accordance with the GOST [10]. The microhardness and microstructure of pellets were investigated with the LECO metallographic equipment.

2. RESULTS

2.1. PHASE FORMATION KINETICS

The results of investigation of sintering effect in the temperature range of 1250...1650 °C on the phase content of samples with compositions 1 and 2 are presented in Fig. 2 and Tabl. 2. After sintering at 1250...1350 °C it was observed the formation of low-temperature orthorhombic phase $\text{Dy}_2\text{TiO}_5\text{-o}$ and pyrochlore $\text{Dy}_2\text{Ti}_2\text{O}_7\text{-p}$ in pellets for both compositions, which corresponds to the Dy_2O_3 - TiO_2 equilibrium phase diagram (see Fig. 1). Moreover, in composition 1 pellets, sintered at 1350 °C, fraction of these phases have similar values (38.8 and 33.8 wt.%), and in composition 2 pellets the formation of $\text{Dy}_2\text{TiO}_5\text{-o}$ phase run more intensively (52.8 against 37.4 wt.%) (see Tabl. 2).

In both cases remaining dysprosium oxide was found.

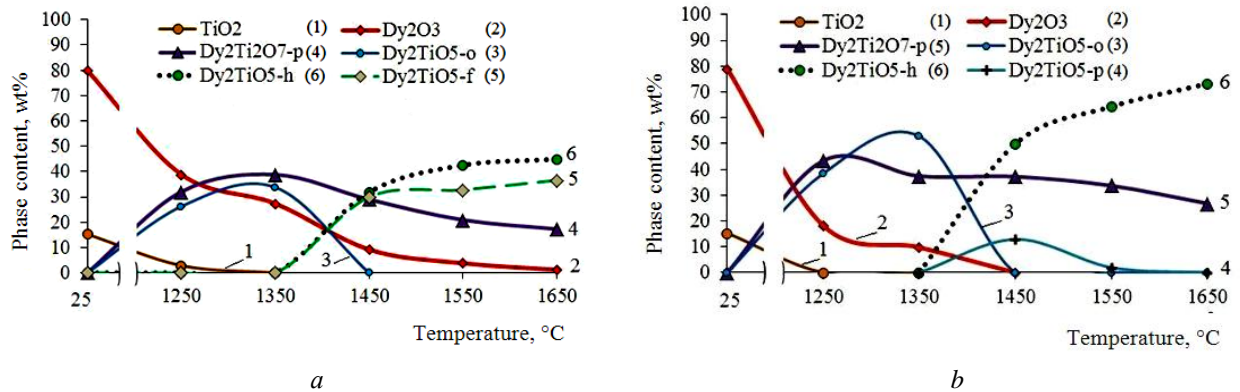


Fig. 2. Phase composition of pellets, sintered for 3 hours with compositions 1 (a) and 2 (b) at temperatures in the range between 1250 and 1650 °C

Phase composition and lattice parameters depending on sintering temperatures

Sintering temperature, °C	Phase	Composition No 1		Composition No 2	
		Fraction, wt. %	Lattice parameters, nm	Fraction, wt. %	Lattice parameters, nm
1250	Dy ₂ O ₃	38.9	a = 1.066	18.2	a = 1.0657
	TiO ₂ -ru	2.9	a = 0.4588; c = 0.2963	–	–
	Dy ₂ Ti ₂ O ₇ -p	32.0	a = 1.0124	43.4	a = 1.0121
	Dy ₂ TiO ₅ -o	26.2	a = 1.0368; b = 1.1219; c = 0.3720	38.4	a = 1.0362; b = 1.1221; c = 0.3717
1350	Dy ₂ O ₃	27.4	a = 1.0664	9.8	a = 1.0659
	Dy ₂ Ti ₂ O ₇ -p	38.8	a = 1.0128	37.4	a = 1.0124
	Dy ₂ TiO ₅ -o	33.8	a = 1.0372; b = 1.1228; c = 0.3721	52.8	a = 1.0365; b = 1.1229; c = 0.3718
1450	Dy ₂ O ₃	9.2	a = 1.0661	–	–
	Dy ₂ Ti ₂ O ₇ -p	29.0	a = 1.0131	33.7	a = 1.0143
	Dy ₂ TiO ₅ -h	31.9	a = 0.3632; c = 1.1910	64.3	a = 0.3631; c = 1.1876;
	Dy ₂ TiO ₅ -f	29.9	a = 0.5211	–	–
	Dy ₂ TiO ₅ -p	–	–	12.9	a = 1.0418
1550	Dy ₂ O ₃	3.8	a = 1.0661	–	–
	Dy ₂ Ti ₂ O ₇ -p	20.9	a = 1.0142	33.7	a = 1.0143
	Dy ₂ TiO ₅ -h	42.5	a = 0.3631; c = 1.1894	64.3	a = 0.3631; c = 1.1876
	Dy ₂ TiO ₅ -f	32.8	a = 0.5200	–	–
	Dy ₂ TiO ₅ -p	–	–	2	a = 1.0410
1650	Dy ₂ O ₃	1.2	a = 1.0669	–	–
	Dy ₂ Ti ₂ O ₇ -p	17.3	a = 1.0167	26.8	a = 1.0161
	Dy ₂ TiO ₅ -h	44.9	a = 0.3633; c = 1.1891	73.2	a = 0.3630; c = 1.1860
	Dy ₂ TiO ₅ -f	36.6	a = 0.5188	–	–

Increasing the sintering temperature up to 1450 °C provides formation of high-temperature pyrochlore phase Dy₂Ti₂O₇-p, hexagonal Dy₂TiO₅-h and fluorite Dy₂TiO₅-f polymorphs for composition 1 approximately in equal fractions.

Also, it was observed ~ 9 wt.% of unreacted Dy₂O₃. In composition 2 the formation of three phases was observed. The main phase of the sample was Dy₂TiO₅-h with hexagonal structure. The other two phases, Dy₂Ti₂O₇-p and Dy₂TiO₅-p, had pyrochlore structure.

As sintering temperature increases up to 1650 °C, the slow decreasing of Dy₂Ti₂O₇-p phase and increasing of Dy₂TiO₅-h phase in pellets for both compositions is observed. A distinguishing feature of the composition 1 was the presence of fluorite phase Dy₂TiO₅-f (36.6 wt.%). Also, about 1 wt.% of unreacted Dy₂O₃ was found too. After sintering at 1350 °C the composition 2 material becomes two-phased one and consists of 73.2 wt.% of Dy₂TiO₅-h phase and 26.8 wt.% of pyrochlore phase Dy₂Ti₂O₇-p (see Tabl. 2).

The application of scheme 2, which comprise pre-sintering at 1250 °C and final sintering at 1650 °C for 3 hours, provides formation of two-phase structure, hexagonal Dy₂TiO₅-h and fluorite Dy₂TiO₅-f ones, in amounts of 83 and 17 wt.%, respectively. Unreacted titanium and dysprosium oxides were not observed in these samples.

2.2. DENSITY AND STRUCTURE

Dependencies of the pellets density and open porosity on the sintering temperature in the range between 1250 and 1650 °C using two schemes of dysprosium titanate pellet production (composition 1) are shown in Fig. 3, pellet structures are shown in Fig. 4.

These data show that the density of pellets produced by scheme 1 increases with the temperature increasing from 4.2 to 5.7 g/cm³ approximately in linear manner (see Fig. 3,a). The microstructure of pellet is characterized by the irregular distribution of ~ 30 μm pores with irregular geometric shape (see Fig. 4,a). The open porosity after sintering at 1250 °C was 32% and reduced up to 8% after sintering at 1650 °C (see Fig. 3,b). The microhardness of pellet material was equal to (2380±137) MPa.

The employment of the second scheme allows to obtain pellets with higher density in comparison with the first one. Pellets density increased from 4.5 g/cm³ in the initial state to 7.1 g/cm³ after sintering for 3 hours at 1650 °C (see Fig. 3,a). The open porosity was decreased from 20% down to 1...2% (see Fig. 3,b).

The microstructure of pellets with the density of 7.1 g/cm³ is characterized by uniform distribution of pores with predominantly spherical shape. Most of these pores had size less than 3 μm (see Fig. 4,b).

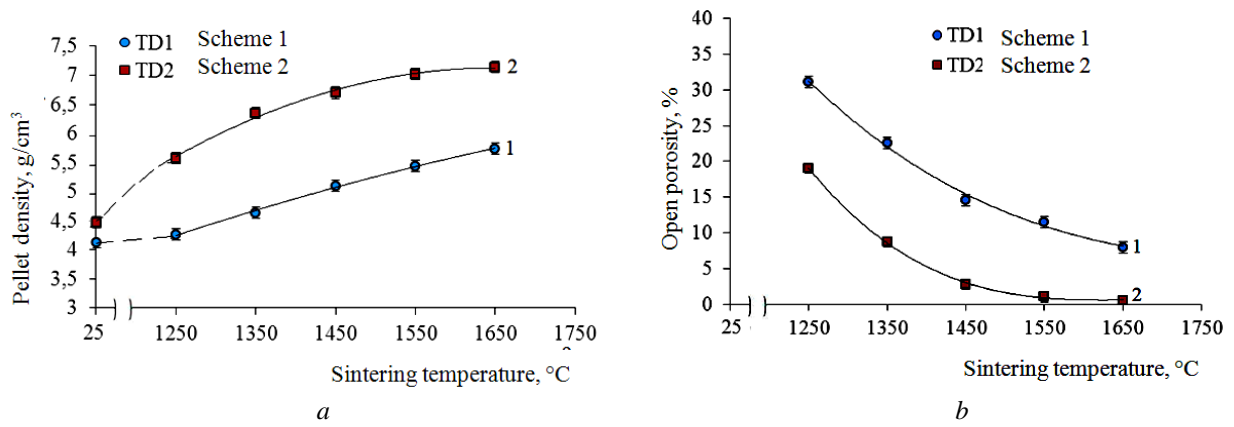


Fig. 3. The density (a) and open porosity (b) of pellets (composition 1) depending on the sintering temperature: 1 – scheme 1; 2 – scheme 2

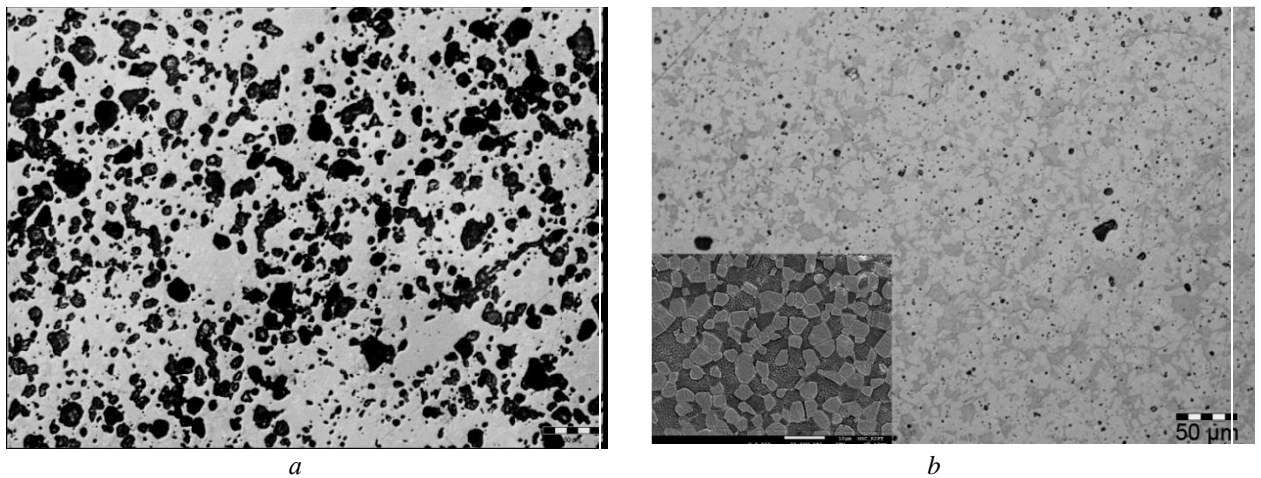


Fig. 4. Microstructure of dysprosium titanate pellets (magn. $\times 500$): a – scheme 1 ($\rho = 5.7 \text{ g/cm}^3$); b – scheme 2 ($\rho = 7.1 \text{ g/cm}^3$)

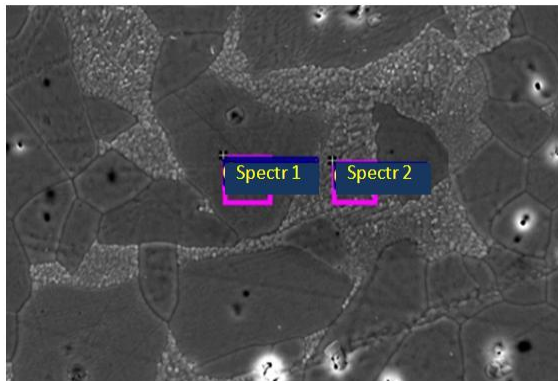


Fig. 5. Microstructure of dysprosium titanate pellets (composition 1, scheme 2, $\rho = 7.1 \text{ g/cm}^3$)

According to the electron microscopy results, the pellet structure consist of two grain types. Most of them had non-uniform shapes and well-defined edges.

Pores are concentrated both on the edges and in the grains volume (Fig. 5). Average grain size is $5.3 \mu\text{m}$. Grains of the second type have subgrain structure with characteristic size up to $1 \mu\text{m}$ and represented by high-light areas. The microhardness of pellet material was $(9015 \pm 450) \text{ MPa}$.

Concentrations of major elements (Dy, Ti, and O) in the different parts of polished sections of pellets, produced by scheme 2, are presented in Tabl. 3.

Table 3

Element content in pellets, produced according to scheme 2 ($\rho = 7.1 \text{ g/cm}^3$)

Element	Element content, % by weight (see Fig. 5)	
	Spectrum 1	Spectrum 2
Dy	71.97	69.90
Ti	9.15	10.42
O	18.88	19.68

3. DISCUSSION

Sintering of composition 1 (50 mol.% TiO_2 + 50 mol.% Dy_2O_3) and composition 2 (56 mol.% TiO_2 + 44 mol.% Dy_2O_3) at temperatures $\geq 1250 \text{ }^\circ\text{C}$ results in formation of low-temperature phases: pyrochlore $\text{Dy}_2\text{Ti}_2\text{O}_7\text{-p}$ and orthorhombic $\text{Dy}_2\text{TiO}_5\text{-o}$ phase, according to the $\text{Dy}_2\text{O}_3\text{-TiO}_2$ phase diagram.

In agreement with data given in works [1, 9], at the first sintering stage dysprosium titanate $\text{Dy}_2\text{Ti}_2\text{O}_7$ is forming. Its synthesis temperature and formation kinetics significantly depends on crystal structure of the initial oxides. For example, the usage of TiO_2 in the form of anatase instead of rutile modification allows to decrease the formation temperature down to $700 \dots 800 \text{ }^\circ\text{C}$ [1]. The process of interaction occurs predominantly via one-way diffusion of titanium and oxygen ions to dysprosium oxide [11]. Therefore, the formation of com-

pounds with a crystal structure similar to dysprosium oxide is more energetically favourable. $\text{Dy}_2\text{Ti}_2\text{O}_7$ and Dy_2O_3 are related to cubic crystal and have comparatively equal lattice parameters. It is deemed [12] that formation of $\text{Dy}_2\text{Ti}_2\text{O}_7$ occurs in two stages: 1) chemical interaction of the initial oxides; 2) diffusion of titanium and oxygen ions into dysprosium oxide.

At sintering temperatures $\leq 1350^\circ\text{C}$ the speeds of formation of $\text{Dy}_2\text{Ti}_2\text{O}_7$ -p and Dy_2TiO_5 -o phases differs depending on the mixture composition. For example, fraction of these phases in composition 1 samples, sintered at 1350°C , have similar values (38.8 and 33.8 wt.%). But, in composition 2, containing smaller amount of Dy_2O_3 , the process of Dy_2TiO_5 -o phase formation is more intensively (52.8 compared to 37.4 wt.%).

Above 1450°C the transformation of the orthorhombic Dy_2TiO_5 -o phase into the hexagonal Dy_2TiO_5 -h one occurs. The quantity of Dy_2TiO_5 -h phase increases with the temperature increasing, and, accordingly, decreasing of the $\text{Dy}_2\text{Ti}_2\text{O}_7$ -p fraction is observed. This confirmed the fact of dysprosium titanate Dy_2TiO_5 formation according to the reaction: $\text{Dy}_2\text{Ti}_2\text{O}_7 + \text{Dy}_2\text{O}_3 \rightarrow 2\text{Dy}_2\text{TiO}_5$ [6]. The speed of this reaction slows down by the presence of pyrochlore $\text{Dy}_2\text{Ti}_2\text{O}_7$ -p layer, which is a barrier for diffusing titanium and oxygen ions [11, 12]. Therefore, to obtain a single-phase Dy_2TiO_5 structure throughout the material volume prolonged sintering time or higher temperatures are needed.

Noteworthy is the fact of the radiation-resistant fluorite phase, Dy_2TiO_5 -f, formation in composition 1 samples. According to the phase diagram (see Fig. 1), this phase should be formed at temperatures above 1680°C .

Harnessing scheme 2 provides production of dysprosium titanate pellets with density about 7.1 g/cm^3 and with fine-grained structure and low level of open porosity. Also, in comparison with scheme 1 (one-stage process) a decrease in Dy_2TiO_5 -f content took place (from 36.6 to 17.0 wt.%) for pellets sintered at the same temperature of 1650°C .

CONCLUSIONS

1. The kinetics of phase formation in dysprosium titanate pellets having composition 1 (50 mol.% TiO_2 + 50 mol.% Dy_2O_3) and composition 2 (56 mol.% TiO_2 + 44 mol.% Dy_2O_3) after sintering in air at temperature range of $1250\text{--}1650^\circ\text{C}$ was investigated.

2. The initial composition of powder mixture (TiO_2 - Dy_2O_3 ratio) affects to the phase composition of the synthesized materials.

3. At temperatures above 1450°C in dysprosium titanate pellets with composition 1 (50 mol.% TiO_2 + 50 mol.% Dy_2O_3) radiation-resistant high-temperature fluorite phase, Dy_2TiO_5 -f, is formed, the amount of which increased with increasing sintering temperature.

4. Harnessing scheme 2 (two-stage sintering) provides production of dysprosium titanate pellets with

density $\sim 7.1\text{ g/cm}^3$, low level of open porosity (1...2%) and fine-grained structure.

5. The probable mechanisms of dysprosium titanate formation at solid-phase synthesis in air were considered.

REFERENCES

1. V.D. Risovany, A.V. Zakharov, V.B. Ponomarenko, E.P. Klochkov, and E.M. Muraliova. Dysprosium in Nuclear Technology. $^{162}\text{Dy}_{66}$. Dimitrovgrad: JSC "SSC RIAR", 2011, 224 p.
2. L.G. Scherbakova, L.G. Mamsurova, and G.E. Sukhanova. Titanates of rare earth elements // *Uspekhi khimii*. 1979, v. 48, issue 3, p. 423-447 (in Russian)
3. V.D. Risovany, E.E. Varlashova, and S.R. Fridman. Absorbing materials and control rod clusters of WWER-1000 and PWR reactors: a review // *Conditions, problems and solutions*. Dimitrovgrad: JSC "SSC RIAR", 1998, 54 p.
4. N.N. Belash, A.V. Kushtym, V.R. Tatarinov, and I.A. Chernov. Analysis of the constructions and materials engineering for rod cluster control assemblies with improve performance // *Nuclear and radiation technologies*. 2007, v. 7, N 3-4, p. 18-28.
5. V.D. Risovany, A.V. Zakharov, and E.M. Muraliova. New promising materials for thermal neutron reactors. // *Problems of Atomic Science and Technology. Series "Physics of Radiation Effects and Radiation Materials Science"* (86). 2005, N 3, p. 87-93.
6. A. Sinha, B.P. Sharma. Development of Dysprosium Titanate Based Ceramics for Control Rod Applications // *Journal of the American Ceramic Society*. 2015, v. 88, N 4, p. 1061-1066.
7. Choong-Hwan Jung, Chan-Joong Kim, and Sang-Jin Lee. Synthesis and sintering studies on Dy_2TiO_5 prepared by polymer carrier chemical process // *Journal of Nuclear Materials*. 2006, v. 354, p. 137-142.
8. V.D. Risovany, E.E. Varshalova, and D.N. Suslov. Dysprosium titanate as absorber material for control rods // *Journal of Nuclear Materials*. 2000, v. 281, p. 84-89.
9. G.V. Shamrai, R.L. Magunov, I.V. Stasenko, and A.P. Zhirnova. Dy_2O_3 - TiO_2 System // *Inorganic Materials*. 1989, v. 25, N 2, p. 273-275.
10. GOST 2409-2014 Refractories. *Method for determination of bulk density, apparent and true porosity, water absorption*.
11. V.B. Glushkova, P.V. Sazonova, N.M. Sidorova, and L.G. Scherbakova. The chemistry of rare earth element titanates. *The Chemistry of Silicates and Oxides*. Leningrad: "Nauka", 1982, p. 170-211.
12. G.M. Griaznov, B.G. Arabei, A.Ya. Beliakov, et al. Investigation of kinetics of $\text{Gd}_2\text{Ti}_2\text{O}_7$ and $\text{Dy}_2\text{Ti}_2\text{O}_7$ syntheses // *Inorganic Materials*. 1979, v. 15, N 11, p. 1986-1989.

ФАЗООБРАЗОВАНИЕ И ХАРАКТЕРИСТИКИ ТАБЛЕТОК НА ОСНОВЕ ТИТАНАТА ДИСПРОЗИЯ ПРИ ТВЕРДОФАЗНОМ СИНТЕЗЕ

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Разработан титанат диспрозия, который используется в качестве поглощающего материала для регулирующих стержней ядерных реакторов на тепловых нейтронах. Представлены результаты исследования кинетики фазообразования в материале таблеток из порошковых смесей состава 1 (50 мол.% TiO_2 + 50 мол.% Dy_2O_3) и состава 2 (56 мол.% TiO_2 + 44 мол.% Dy_2O_3) после спекания при температурах 1250...1650 °С в воздушной атмосфере. Показано, что состав исходной смеси влияет на фазовый состав и соотношение фаз в синтезируемом материале. Зафиксировано образование радиационно стойкой высокотемпературной фазы со структурой флюорита (Dy_2TiO_5 -f) при использовании смеси состава 1. Применение схемы двухстадийного спекания с частичным синтезом материала на первой стадии обеспечивает получение таблеток из Dy_2TiO_5 плотностью 7,1 г/см³ с мелкозернистой структурой и низким значением открытой пористости.

ФАЗОУТВОРЕННЯ І ХАРАКТЕРИСТИКИ ТАБЛЕТОК НА ОСНОВІ ТИТАНАТУ ДИСПРОЗІЮ ПРИ ТВЕРДОФАЗОВОМУ СИНТЕЗІ

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Розроблено титанат диспрозію, який використовується в якості поглинаючого матеріалу для регулюючих стрижнів ядерних реакторів на теплових нейтронах. Наведено результати дослідження кінетики фазоутворення в матеріалі таблеток із порошкових сумішей складу 1 (50 мол.% TiO_2 + 50 мол.% Dy_2O_3) та складу 2 (56 мол.% TiO_2 + 44 мол.% Dy_2O_3) після спікання за температур 1250...1650 °С в повітряній атмосфері. Показано, що склад вихідної суміші впливає на фазовий склад і співвідношення фаз у матеріалі, який синтезується. Зафіксовано утворення радіаційно стійкої високотемпературної фази зі структурою флюориту (Dy_2TiO_5 -f) при використанні суміші складу 1. Використання схеми двостадійного спікання з частковим синтезом матеріалу на першій стадії забезпечує одержання таблеток із Dy_2TiO_5 щільністю 7,1 г/см³ з дрібнозернистою структурою та низьким значенням відкритої пористості.