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Swift heavy ion irradiation of titanium phosphate and related materials

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Abstract

Amorphous and crystalline (both, α- and γ-forms) titanium phosphate, titanium molybdate and tungstate were irradiated with swift heavy ions of ^{84}Kr and ^{203}Bi at a fluence of 5×10^{10}–10^{14} ion cm^{-2}. The crystalline structure of the materials was characterized by XRD method before and after irradiation. Comparison of the powder diffraction patterns revealed that the irradiation had practically no effect on the materials with the exception of γ-crystalline structure. This structure was destroyed under the effect of radiation of ^{84}Kr ions and became amorphous above the fluence of 1×10^{12} ion cm^{-2}. In case of irradiation with ^{203}Bi ions it became amorphous at the lowest fluences applied.
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Keywords: Titanium phosphate; Titanium molybdate; Titanium tungstate; Swift heavy ion

1. Introduction

Among the investigations concerned with inorganic ion exchange materials the works belonging to titanium phosphate and related materials were done for a short time after the known results connected with zirconium phosphate. The crystalline α-titanium phosphate (hereafter TiP) have been obtained independently by various authors (Alberti et al., 1967; Alberti and Torracca, 1968; Weiss and Michel, 1966; Szirtes, 1987). The γ-crystalline form of TiP was synthesized later on by Alberti et al. (1979), then the structure of this material was investigated in more detail by Christensen et al. (1990). Later, the mixed zirconium–titanium and hafnium–titanium phosphates were studied (Shakshooki et al., 1991, 1992; Szirtes et al., 1993).

The titanium molybdate (TiMo) and tungstate were prepared and investigated earlier by us (Cziboly et al., 1971). These titanium salts have own interest in catalytic research, and especially in the nuclear chemistry practice (Szirtes, 1968; Szirtes et al., 1984). In connection with the earlier mentioned works we investigated the effect of γ-ray irradiation on these materials. Now, this was the first time that we investigated the radiation effect of heavy ions on the structure of titanium phosphate and some related materials.

The results of these experiments are collected in this paper.

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2. Experimental

2.1. Preparation

Amorphous TiP was prepared by mixing (at continuous stirring) the stoichiometric quantity of solutions of $TiCl_4$ in 1 M HCl and H_3PO_4 (85%) at temperature 353 K. The precipitate was filtered, washed with distilled water till pH = 4 and dried at room temperature in desiccator above P_2O_5. Both α- and γ-crystalline forms of titanium phosphate were prepared through fluoro-complex, whose methods were proposed first by Alberti (Albert and Costantino, 1974; Alberti et al., 1979). The TiMo was synthesized as follows: the aqueous solution of $(NH_4)Mo_6O_{24}\cdot 12H_2O$ whose pH was first adjusted to 1.9 with 6 M solution of HCl was added dropwise (at continuous vigorous stirring) to the stoichiometric amount of $TiCl_4$ in 1 M solution of HCl. The precipitate (amorphous gel) was held in contact with its mother liquid for 100 h at a temperature of 353 K. The product was filtered, washed with distilled water till pH = 4 and dried at room temperature in a vacuum desiccator over P_2O_5. The titanium tungstate (TiW) was prepared in the same way as described above with exception that $(NH_4)W_7O_{24}\cdot 12H_2O$ solution was used as starting material.

For the irradiation, pellets ($d = 13$ mm, $h = 3$ mm, $p = 3000$ Pa) were made from the above-synthesized materials.

2.2. Characterization

The samples were characterized before and after irradiation by the XRD method. The powder diffraction patterns were recorded using a computer-controlled powder diffractometer (DRON-2). The measurements were performed with a goniometer speed of $1^\circ\,min^{-1}$ in the range of $2\Theta = 3$–110°, using CoKα radiation ($\lambda = 1.7890$) at temperature 295 K. The powder diffraction patterns were evaluated by "EX-RAY" peak searching computer program (Klencsár, 1998) and the cell parameters were also fitted. All evaluations of non-irradiated samples were also controlled by modelling using the software of Kraus and Nolze (1999).

2.3. Irradiation

The irradiation of samples was carried out at room temperature with 246 MeV energy ^{84}Kr and 720 MeV energy ^{203}Bi ions with dose between 5×10^{10} and $1\times10^{14}\,ion\,cm^{-2}$ at the U-400 type cyclotron of the Laboratory of Nuclear Reactions of the JINR, Dubna. The ion current was $7\times10^{8}\,ion\,cm^{-2}\,min^{-1}$. The ion beam was scanned in order to achieve homogeneous irradiation. The irradiation was performed in vacuum (6×10^{-2} Pa). The temperature of samples was controlled during the irradiation. It did not exceed 293 K.

3. Results and discussion

The results are collected in Tables 1 and 2, and are shown in Figs. 1–4.

The amorphous TiP does not show any changes due to the irradiation both with ^{84}Kr and ^{203}Bi ions with a fluence of $5\times10^{13}\,ion\,cm^{-2}$. The material retained its initial value of specific surface area ($S = 31\,m^2\,g^{-1}$), and its original ion exchange capacity ($7.1\,meq\,g^{-1}$, at

Table 1
Cell parameters of the irradiated samples in comparison with those of the untreated samples

		α-TiP	α-TiP/1	α-TiP/2	α-TiP/3	γ-TiP	γ-TiP/1	γ-TiP/2	γ-TiP/3
Un-treated	a (nm)	0.804	—	—	—	0.518	—	—	—
	b (nm)	0.556	—	—	—	0.635	—	—	—
	c (nm)	1.541	—	—	—	1.188	—	—	—
	d_{002} (nm)	0.756	—	—	—	1.164	—	—	—
	β°	101.7	—	—	—	102.6	—	—	—
^{84}Kr	a (nm)	—	0.808	0.806	0.810	—	Amorphous		
	b (nm)	—	0.560	0.553	0.551	—			
	c (nm)	—	1.548	1.542	1.545	—			
	d_{002} (nm)	—	0.758	0.756	0.754	—			
	β°	—	101.2	101.5	101.5	—			
^{203}Bi	a (nm)	—	0.801	0.804	0.804	—	Amorphous		
	b (nm)	—	0.554	0.556	0.554	—			
	c (nm)	—	1.544	1.544	1.542	—			
	d_{002} (nm)	—	0.758	0.756	0.756	—			
	β°	—	101.2	101.2	101.3	—			

Note: $1 = 1\times10^{12}\,ion\,cm^{-2}$; $2 = 1\times10^{13}\,ion\,cm^{-2}$; $3 = 1\times10^{14}\,ion\,cm^{-2}$.

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