

2004

Development of Fluorapatite as a Waste Form: Progress Report 1-3

Boris E. Burakov

V.G. Khlopin Radium Institute – Research-Industrial Enterprise

Follow this and additional works at: https://digitalscholarship.unlv.edu/hrc_trp_separations



Part of the [Chemistry Commons](#), and the [Oil, Gas, and Energy Commons](#)

Repository Citation

Burakov, B. E. (2004). Development of Fluorapatite as a Waste Form: Progress Report 1-3. 1-4.

Available at: https://digitalscholarship.unlv.edu/hrc_trp_separations/65

This Report is protected by copyright and/or related rights. It has been brought to you by Digital Scholarship@UNLV with permission from the rights-holder(s). You are free to use this Report in any way that is permitted by the copyright and related rights legislation that applies to your use. For other uses you need to obtain permission from the rights-holder(s) directly, unless additional rights are indicated by a Creative Commons license in the record and/or on the work itself.

This Report has been accepted for inclusion in Separations Campaign (TRP) by an authorized administrator of Digital Scholarship@UNLV. For more information, please contact digitalscholarship@unlv.edu.

V.G. Khlopin Radium Institute (KRI)

UNLV – KRI-KIRSI Agreement 280204-1

Development of Fluorapatite as a Waste Form

Progress Report #1-3

Synthesis and study of fluorapatite doped with
fission product elements

Saint-Petersburg – 2005

1. Introduction

In the framework of this stage of research we had to carry out synthesis and study of fluorapatite doped with fission product elements including Cs, Sr, noble metals and Zr. During previous reporting period different samples of ceramics based on Sr-fluorapatite have been synthesized (Table 1). Sample of Cs-Nd-doped ceramic (Fig. 1) has been recognized as the most interesting for detailed examination by SEM and EMPA methods.

Table 1. Phase composition of ceramics based on Sr-fluorapatite

Sample	Desired formula	Phase yield from XRD, wt.%			
		apatite	SrHPO ₄	Sr ₂ P ₂ O ₇	Sr ₃ (PO ₄) ₂
precursor	–	50	50	–	–
#1	Sr ₁₀ (PO ₄) ₆ F ₂	50	–	40	10
#2	Sr ₁₀ (PO ₄) ₆ F ₂	65	–	10	25
#3	Sr ₈ CsNd(PO ₄) ₆ F _{2.3}	30	–	40	30

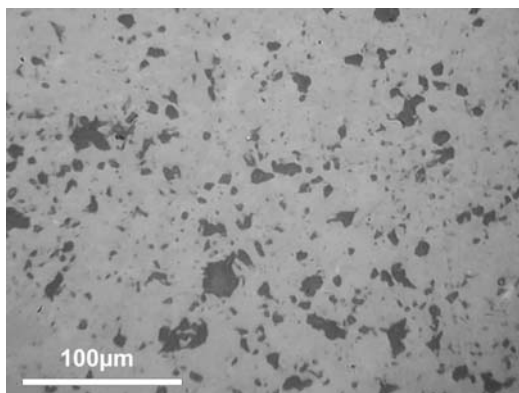


Figure 1. Optical microphotographs of ceramic based on Sr-fluorapatite doped with Cs and Nd (sample # 3). Reflected light images of polished cross-section. Black dots are void spaces (pores).

Also, we have decided to carry out synthesis of Ca-Sr-fluorapatite doped with Zr and Ag (as stimulant of noble metals).

2. SEM and EMPA of Cs-Nd-doped Sr-fluorapatite

The same polished pellet of sample #3 used for examination in optical microscope (Fig. 1) was applied for study by SEM and EMPA methods. Backscattered electron imaging (Fig. 2) allowed clear observation of two phases such as: apatite (light gray) and another Sr-phosphate (dark gray).

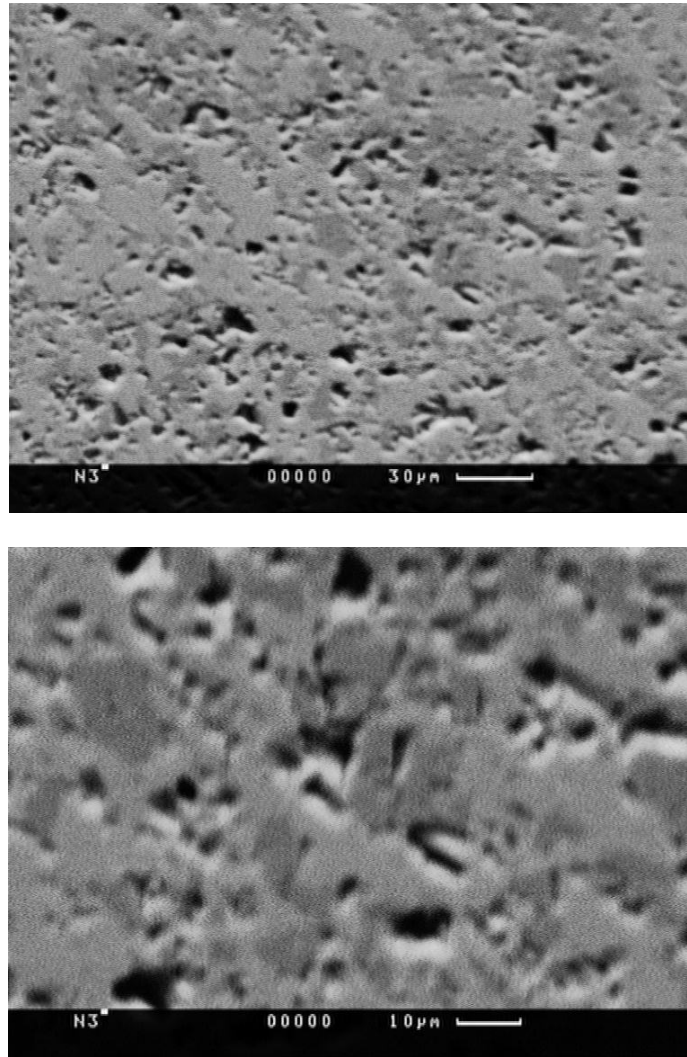


Figure 2. Backscattered electron images (at different magnification) of ceramic based on Sr-fluorapatite doped with Cs and Nd (sample #3). Light-gray is Sr-fluorapatite and dark-grey phase is Sr-phosphate.

Microprobe analyses of every phase was carried out first excluding fluorine and cesium. Then cesium content has been measured by EMPA using special WDS technique. The results obtained are summarized in Table 2.

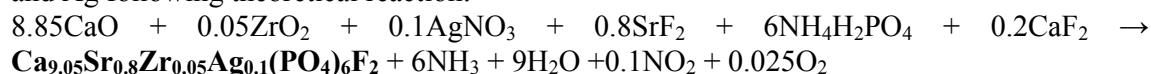
Table 2. Chemical composition of ceramics based on Sr-fluorapatite doped with Cs and Nd.

Element content, wt.%	Calculated		Measured by EMPA	
	apatite	other phosphates	apatite	other phosphates
Sr	58	50-58	57.5-58.9	48.5-55.1
Cs	1	-	0.1-0.6	0.1-0.6
Nd	1	-	0.7-0.8	-
P	12	18-14	11.8-13.7	14.6-17.2
O	25	32-28	No data	No data
F	3	-	No data	No data

It is important to note that Cs was accommodated by all phosphate phases although Nd admixture remained only in apatite. Cesium distribution is not homogeneous and it varies in a broad range from 0.1 to 0.6 wt.%. We assume that part of Cs volatilized during ceramic synthesis at 1250°C. This means that successful Cs incorporation into apatite structure requires development of more optimal synthesis conditions.

3. Synthesis of Ca-Sr-fluorapatite doped with Zr and Ag

Synthesis of fluorapatite doped with Zr and noble metals requires new approach in comparison with pure undoped apatite or Sr-Cs-Nd-doped samples. This is caused by necessity to innovate precursor preparation. Commonly used co-precipitation technique is not efficient for such doping elements as Zr and noble metals which have special chemical behavior. Possible formation of stable inert phases of Zr and Ag during precursor preparation might cause inhomogeneity of final ceramic and formation of separate inclusions of fission elements. We decided to carry out solid-phase synthesis of Ca-Sr-fluorapatite doped with Zr and Ag following theoretical reaction:



Some chemicals were used for the synthesis with small excess taking into account significant formation of gaseous products during reaction. In order to provide better homogeneity of all chemical compounds the starting precursor was set at temperature 350°C with excess of $\text{NH}_4\text{H}_2\text{PO}_4$ for 3 hours. Then starting precursor was sintered in air at 1150°C for one hour. Obtained material was ground in agate mortar, cold pressed into pellets (10 mm in diameter) and sintered again in the air at 1150°C for 4 hours.

At the moment we have started preparing samples for XRD and EMPA analysis.

Preliminary conclusions

- 1) No separate phases of Cs and Nd have been observed by XRD, SEM and EMPA in ceramic sample #3 with desired formula $\text{Sr}_8\text{CsNd}(\text{PO}_4)_6\text{F}_{2.3}$. Although, other phosphate phases such as: $\text{Sr}_2\text{P}_2\text{O}_7$ and $\text{Sr}_3(\text{PO}_4)_2$ have been identified in this sample, Nd was accommodated only by apatite.
- 2) Cesium behavior in sample #3 was completely different in comparison with Nd although cesium was considered as a charge compensator in apatite phase with desired formula $\text{Sr}_8\text{CsNd}(\text{PO}_4)_6\text{F}_{2.3}$. It was incorporated into all phosphates. Cesium distribution was not homogeneous and it varied in a broad range from 0.1 to 0.6 wt.%. It is assumed that part of Cs volatilized during ceramic synthesis at 1250°C. Therefore successful Cs incorporation into apatite structure requires development of more optimal synthesis conditions.
- 3) Solid phase synthesis of ceramic based on apatite with desired formula $\text{Ca}_{9.05}\text{Sr}_{0.8}\text{Zr}_{0.05}\text{Ag}_{0.1}(\text{PO}_4)_6\text{F}_2$ has been carried out using new method of precursor preparation. Study of sample obtained by XRD and EMPA methods is in progress.

Dr. Boris E. Burakov , Principal Investigator

