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Development of Fluorapatite as a Waste Form: Progress Report 1-2

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V.G. Khlopin Radium Institute (KRI)

UNLV – KRI-KIRSI Agreement 280204-1

Development of Fluorapatite as a Waste Form

Progress Report #1-2

Examination of samples obtained using
different methods

Saint-Petersburg – 2004

1. Introduction

In the framework of this stage of research the samples of Sr-fluorapatite synthesized earlier had to be studied using different methods. These are samples of the following desired compositions:

- 1) $\text{Sr}_{10}(\text{PO}_4)_6\text{F}_2$ from precipitated precursor;
- 2) $\text{Sr}_{10}(\text{PO}_4)_6\text{F}_2$ from precipitated precursor and adding $\text{Sr}(\text{NO}_3)_2$;
- 3) $\text{Sr}_8\text{CsNd}(\text{PO}_4)_6\text{F}_{2.3}$;
- 4) Initial precursor for the synthesis of un-doped Sr-fluorapatite.

2. Optical microscopy

Single pellet of each sample #1-3 was cut by diamond blade on two similar parts. Then a half of each pellet was placed into acrylic resin and polished in order to obtain cross-section specimen for optical examination. The same samples will be used for SEM and microprobe analysis.

No cracks were observed in all ceramic matrices (Fig. 1-2). Both samples #1 and #2 of undoped Sr-fluorapatite are characterized by relatively homogeneous matrices, although sample #1 has a higher porosity level (Fig. 1a).

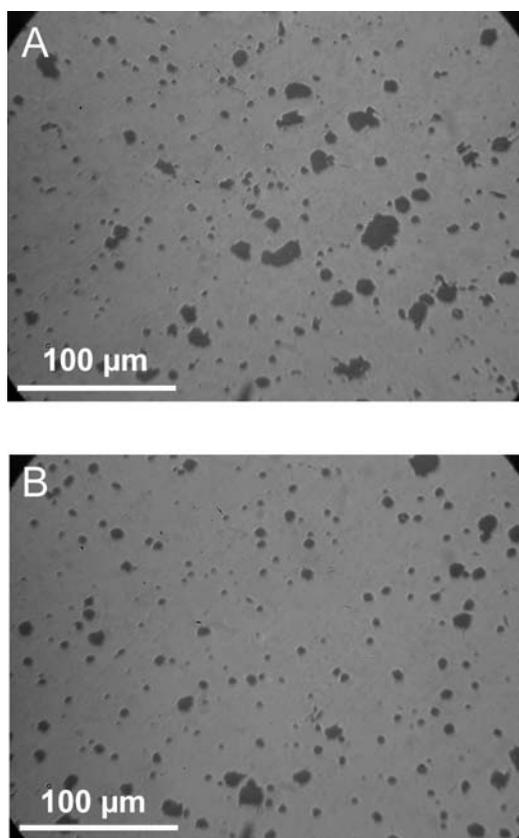


Figure 1. Optical microphotographs of ceramic based on undoped Sr-fluorapatite. Reflected light images of polished cross-sections. Black dots are void spaces (pores). Both samples #1 (A) and #2 (B) were obtained from the same co-precipitated precursor but adding of $\text{Sr}(\text{NO}_3)_2$ in starting material of sample #2 has been done before final sintering.

Ceramic based on Sr-fluorapatite doped with Cs and Nd (Sample #3) is more porous in comparison with undoped samples (Fig. 2). Double phase composition (phases with dark-gray and light-gray contrast) was clear observed in optical microscope (Fig. 2).

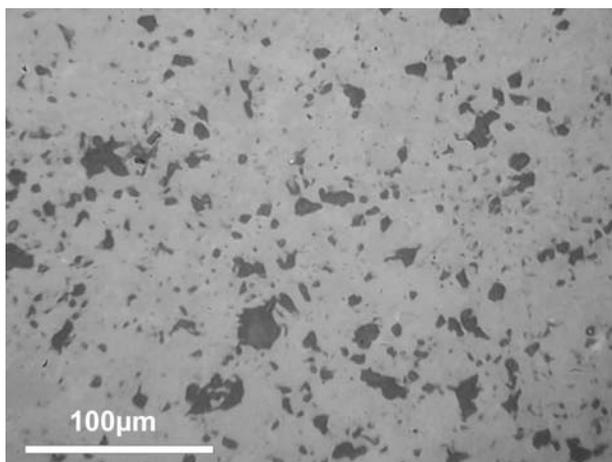


Figure 2. Optical microphotographs of ceramic based on Sr-fluorapatite doped with Cs and Nd (sample # 3). Reflected light images of polished cross-section. Black dots are void spaces (pores). At least two phases with dark-gray and light-gray contrast might be observed in ceramic matrix.

3. XRD analysis

Second half of the pellet of each sample #1-3 was ground and used for X-ray diffraction quantitative analysis. The results obtained (Fig. 3, Table 1) demonstrated that formation of Sr-fluorapatite phase took place already during preparation of starting precursor. This is similar to Ca-fluorapatite obtained in previous experiments.

The highest yield of apatite phase (about 65 wt.%) has been observed in sample #2, when additional amount of Sr in the form of $\text{Sr}(\text{NO}_3)_2$ was added into precipitated and calcined powder. This confirmed our expectation that during co-precipitation under excess of H_3PO_4 (in order to provide complete Sr precipitation) it is difficult to secure stoichiometry of final starting material. Although, we found the way to provide essential increase of apatite yield, obtaining single-phase ceramic will require solving a problem of fluorine lack.

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

Sample	Desired formula	Phase yield from XRD, wt. %			
		apatite	SrHPO_4	$\text{Sr}_2\text{P}_2\text{O}_7$	$\text{Sr}_3(\text{PO}_4)_2$
precursor	–	50	50	–	–
#1	$\text{Sr}_{10}(\text{PO}_4)_6\text{F}_2$	50	–	40	10
#2	$\text{Sr}_{10}(\text{PO}_4)_6\text{F}_2$	65	–	10	25
#3	$\text{Sr}_8\text{CsNd}(\text{PO}_4)_6\text{F}_{2.3}$	30	–	40	30

Very promising result was obtained from sample of Sr-fluorapatite (sample #3) doped with Cs and Sr. The yield of apatite phase in this ceramic was even less than in starting un-doped precursor, however, no separate phases of Cs and Nd have been observed. Detailed investigation of Cs and Nd incorporation into crystalline structure of Sr-fluorapatite (or other

Sr-phosphate phases) will be carried out by SEM and TEM methods during next reporting period.

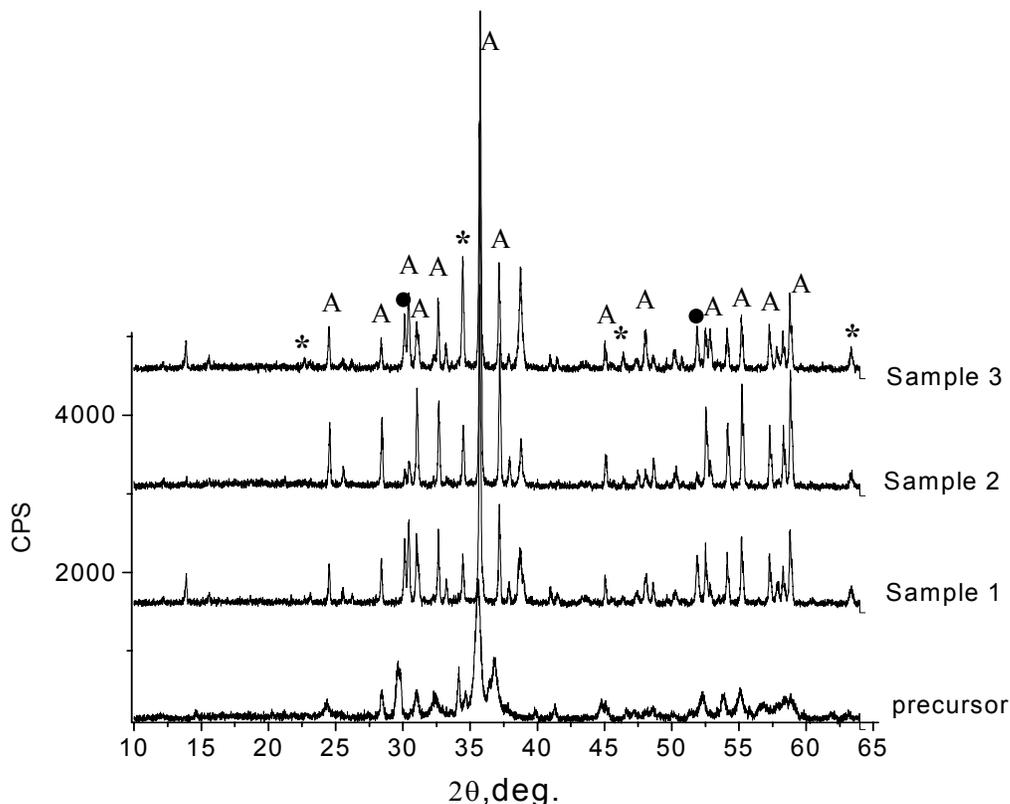


Figure 3. X-ray powder diffraction analysis of starting precursor and ceramic based on Sr-fluorapatite. Phases are marked by: apatite -“A”; $\text{Sr}_3(\text{PO}_4)_2$ - “*” and $\text{Sr}_2\text{P}_2\text{O}_7$ - “■”.

Preliminary conclusions

- 1) The Sr-fluorapatite demonstrated similar features with Ca-fluorapatite. Essential yield up to 50 wt.% of Sr-fluorapatite takes place during precursor fabrication;
- 2) Co-precipitation of “raw” Sr-fluorapatite under excess of H_3PO_4 is accompanied with the change of stoichiometry and as a result – formation of $\text{Sr}_2\text{P}_2\text{O}_7$ and $\text{Sr}_3(\text{PO}_4)_2$ phases. Adding of $\text{Sr}(\text{NO}_3)_2$ into co-precipitated material allows essential increasing apatite yield. However, obtaining single-phase ceramic will require solving a problem of fluorine lack.
- 3) No separate phases of Cs and Nd have been observed by XRD analysis in ceramic sample with desired formula $\text{Sr}_8\text{CsNd}(\text{PO}_4)_6\text{F}_{2.3}$. Study of this sample will be continued using SEM and TEM methods.

