FLUORAPATITE SYNTHESIS USING HIGH TEMPERATURE SOLID STATE REACTION

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ABSTRACT

Apatite is a group of minerals which include fluorapatite (FAp), chlorapatite (ClAp) and hydroxylapatite. This mineral is important, as it is similar in composition to teeth and bones. In this paper we report on the synthesis of fluorapatite through solid state reaction. The fluorapatite was prepared by heating a mixture of calcium orthophosphate and calcium fluoride at 1200°C for 2 hours. X-ray diffraction measurements show that an apatite structure was formed with unit cell dimensions of $a=9.36\ \text{Å}$ and $c=6.85\ \text{Å}$.

Introduction

Fluorapatite is a member of the apatite mineral group. Its chemical formula is $\text{Ca}_3\text{F}((\text{PO}_4)_3$. Other members of the apatite group include hydroxylapatite and chlorapatite. The chemical formula of chlorapatite is similar to fluorapatite except that the fluorine atoms are replaced with chlorine atoms. Hydroxylapatite is an important mineral since the composition of bones is similar to this mineral.

Fluorapatite is an important mineral since it is a promising candidate for near infra red laser when doped with transition metal ions such as Mn$^{+5}$ [4]. These ions substitute for phosphorous, which is situated in a tetrahedral coordination within the apatite structure. In this paper, we report the synthesis of fluorapatite via solid state reaction at high temperatures.
**Experimental method**

The fluorapatite was prepared via the reaction [1]

\[ 3\text{Ca}_3(\text{PO}_4)_2 + \text{CaF}_2 \rightarrow 2\text{Ca}_5(\text{PO}_4)_3\text{F} \]

Appropriate amounts of calcium orthophosphate and calcium fluoride were weighed and mixed thoroughly together. The mixture was placed in an alumina crucible and heated in air to 1200°C for 2 hours. Samples doped with small amounts of manganese was also prepared. The amount of manganese dopant was less than 0.2 wt.% MnO₂.

**Results and discussions**

The apatites were prepared well below its melting point of about 1620°C. X-ray diffraction patterns of the resultant samples are shown in figure 1. Major peaks obtained coincide well with that from fluorapatite diffraction pattern calculated from standard data [2]. This indicates that heat treatment at 1200°C of a mixture of calcium orthophosphate and calcium fluoride results in the formation of fluorapatite. The positions of the (300) and (002) diffraction peaks, which are located at 2θ = 33.17° and 26.02° respectively, were used to obtain the unit cell parameters. Unit cell values obtained were a = 9.36 Å and c = 6.85 Å. This compares well with the standard values of a = 9.368 Å and c = 6.884 Å obtained from reference [2].

Some samples doped with manganese were also prepared. Diffraction pattern of sample doped with 0.2 wt.% MnO₂ is shown in figure 1. The doping level is small and it does not have major effect on the fluorapatite structure. In certain materials doped with manganese ions, emission spectra originating from the manganese ions are observed [3]. However, we do not observe such emission in the visible region from our samples. It was reported that manganese emit at about 1.2 μm in fluorapatite [4]. This was attributed to emission from Mn⁴⁺ ions in tetrahedral sites.
Figure 1. XRD pattern of (a) undoped fluorapatite and (b) doped with 0.2 wt.% MnO₂.

Conclusions

Fluorapatite can be synthesised easily by solid state reaction of calcium orthophosphate and calcium fluoride at 1200 °C. Doping with manganese up to 0.2 wt.% MnO₂ did not affect the apatite structure.

References

2. ASTM X-ray diffraction pattern no 16-146