SYNTHESIS AND STRUCTURAL ANALYSIS OF $\text{Ba}_3\text{Ln}_2\text{O}_5\text{Cl}_2$ CRYSTALS

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Abstract

We aim to explore the composition and structure of the newly discovered crystal phase, $\text{Ba}_3\text{Ln}_2\text{O}_5\text{Cl}_2$ (Ln = La, Ce, ... Lu) to learn more about its properties and identify the optimum growth conditions for maximum yield. We created the $\text{Ba}_3\text{Ln}_2\text{O}_5\text{Cl}_2$ crystal phases using a molten barium metal flux method in which reactants are dissolved in molten barium so that they may form back together in the desired crystal phase upon cooling. The preparation that yielded the highest quantity and crystal purity was a barium flux heated to 1000°C in 10 hours, dwelled at 1000°C for 20 hours and then brought down to 750°C in 200 hours where it was centrifuged. The crystals were then verified using single crystal x-ray diffraction (SCXRD). Once verified full structural data was collected and the crystal structure was solved.

Introduction

Single crystals and crystalline materials have a wide variety of applications in many electronic devices from television screens to high precision lasers. The study of crystals and their properties is of growing interest as the demand for more efficient materials increases in correlation with the rapid expansion of computing and intelligent technology. Our lab aims to discover new crystal phases for the expansion of our knowledge in these unique materials and for their eventual incorporation into practical applications. The particular approach we used incorporates the dissolving of solutes into a molten metal solvent to create unique phases. After many failed attempts we have synthesized a new crystal phase never discovered before and which has proven to have many particular and interesting properties.

Procedure

1. Reactants are loaded into steel metal tubes of 8mm in diameter and approximately 100mm in length. Barium must be loaded in the tubes while inside an argon filled glove box to prevent oxidation.
2. Tubes are then welded shut using a TIG welder in argon atmosphere.
3. Once sealed, the steel tubes are then sealed inside quartz tubes under vacuum. This aids in both containment (if there is a leak in the steel tubes) and to limit thermal conductivity once the samples are removed from the ovens to be centrifuged. The steel tubes are sealed in quartz tubes also in order to prevent oxidation.
4. Samples are then placed in temperature-controlled box furnaces to achieve the desired heating profile.
5. After the samples have reached their final temperature they are removed from the ovens and immediately inverted and centrifuged. This allows the molten flux to be pushed to the opposite side of the crystals before it is able to cool completely and solidify.

6. Once the samples have returned to room temperature the tubes are opened to retrieve the crystals within. This is also done in an argon glove box incase the crystals are air sensitive.
7. Retrieved crystals are then run through the X-ray diffractometer to determine structural properties and access crystal purity.

Results

The optimal conditions for the formation of $\text{Ba}_3\text{Ln}_2\text{O}_5\text{Cl}_2$ crystals through the barium flux method were found. To synthesize the crystals: $\text{Ba}$, $\text{BaCl}_2$, $\text{BaO}$, and $\text{Ln}_2\text{O}_3$ were reacted in a molar ratio of 40:1:1:1. The crucibles were then heated to 1000°C in 10 hours, dwelled at 1000°C for 20 hours and brought down to 750°C in 200 hours.

Structural information about the $\text{Ba}_3\text{Ln}_2\text{O}_5\text{Cl}_2$ crystals were collected using SCXRD. The structure was refined to determine the atomic positions. The unit cell was found to be tetragonal with a space group of I4/mmm and cell parameters of $a = b = 4.40\text{Å}$; $c = 24.7\text{Å}$ with an overall volume of 476 Å$^3$.

Future & Acknowledgments

In the future we wish to explore the use of other Lanthanides and transition metals in the pursuit of higher yield and crystal purity. We also hope to investigate other crystal phases and the extent of their application to useful products.

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