Synthesis and Reduction of $K_5BiMo_4O_{16}$

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Abstract

The primary goal of our experimentation was to synthesize new strongly correlated materials. We synthesized the transition metal oxide Potassium Bismuth Molybdate ($K_5BiMo_4O_{16}$). Powder x-ray diffraction (XRD) was used for phase identification and characterization. Magnetization measurements were performed via a superconducting quantum interference device (SQUID).

Introduction[a]

Strongly correlated materials are known to display interesting electronic and magnetic properties. Some high temperature superconductors belong to this family of materials. For this study, K5BiMo4O16 was used as the starting material, the only known phase is a transparent colorless crystal with no magnetic properties. K5BiMo4O16 was annealed in order to reduce the molybdenum ions from 6+ to a 5+ or 4+ charge. At these oxidation states interesting magnetic properties are more likely. XRD and SQUID measurements were preformed to determine whether any significant changes to the crystal structure or magnetic properties had occurred.

Synthesis[b]

Direct solid state reaction produced single crystals of $K_5BiMo_4O_{16}$. The synthesis was performed in open atmosphere. The starting materials, K_2CO_3, Bi_2O_3, and MoO_2, were mixed in a 5 : 1 : 8 molar ratio using an agate mortar and pestle. This mixture was then pressed into a pellet at $3.5 \cdot 10^7$kPa. The pellet was heated in an alumina crucible as outlined in the temperature profile below:
T-profile: ramp to 1000˚C over 2h, hold at 1000˚C for 1h, cool to 690˚C over 10h, hold at 690˚C for 2h and finally cool to room temperature.
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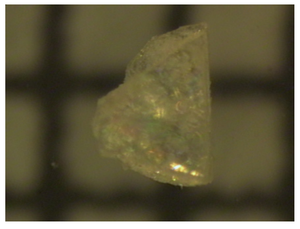
$K_5BiMo_4O_{16}$ was then annealed in a forming gas environment for 24h at 500˚C.
Annealing resulted in a color change from transparent/colorless to opaque/dark grey in both the single crystal and powder samples.



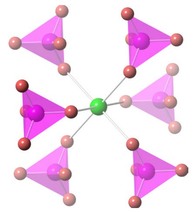
Structure[b]

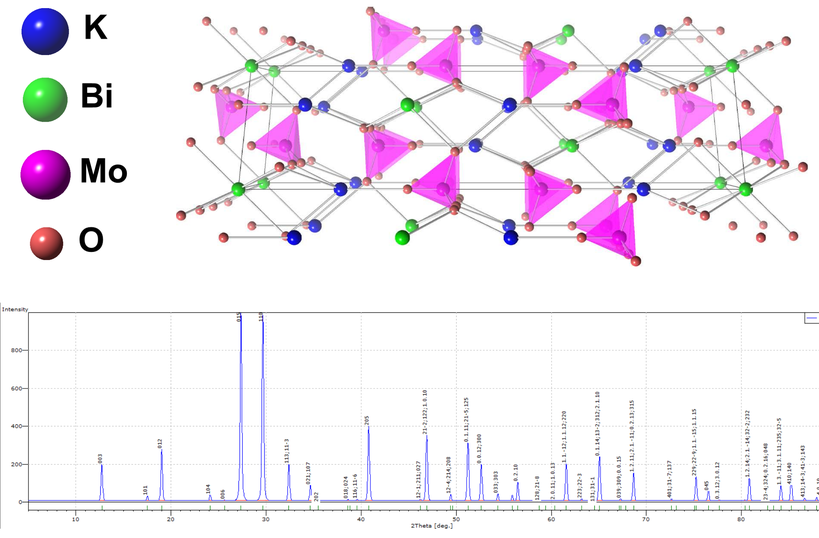
$K_5BiMo_4O_{16}$

Structure type: Trigonal

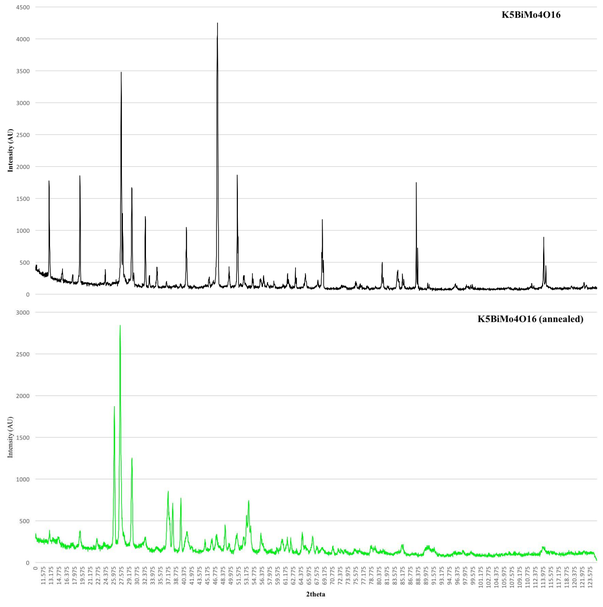
Symmetry group: R -3m

Lattice parameters: a=6.019 Å, c=20.85 Å.

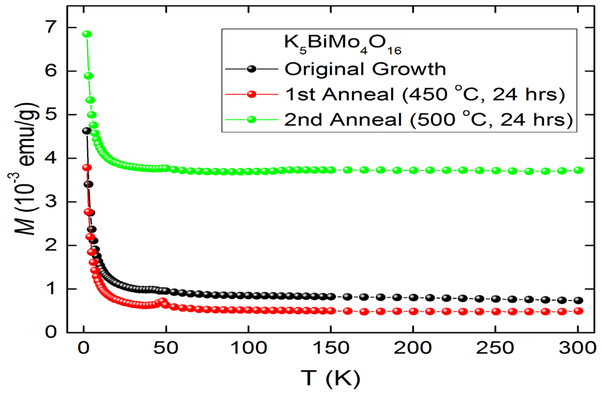




Analysis



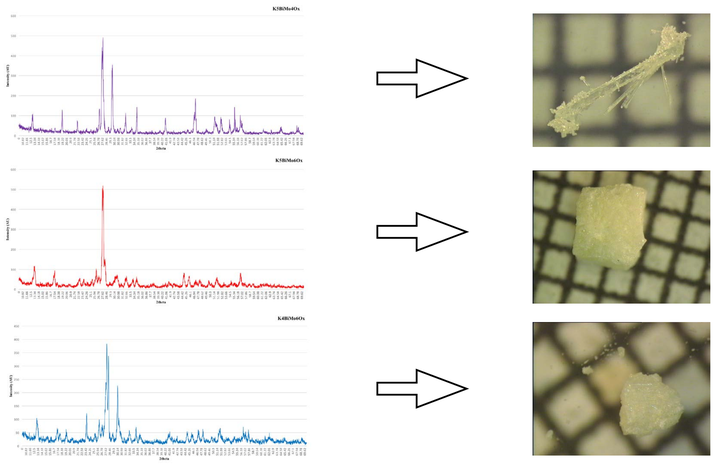
Powder x-ray diffraction patterns were taken of $K_5BiMo_4O_{16}$ before and after annealing. A comparison of the XRD patterns indicates that a change in structure has occurred.



Magnetization measurements were obtained before and after annealing. The data in the figure above indicates that there was an increase in magnetization due to annealing.

Future Work

Determination of the lattice parameters of the annealed compound must be calculated. The lattice parameters will indicate how the structure has changed and thus whether the molybdenum constituents were reduced.
Additionally, the XRD data of the successfully synthesized crystals containing differing stoichiometric ratios must be refined and analyzed to determine if new single phases have resulted. Finally if any new phases are identified, they too may be annealed and characterized for determination of unusal magnetic and electronic properties.



Acknowledgment & References

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a) Hizhnyi, Yu A.; Nedilko, S. G.; Chornii, V. P.; Slobodyanik, M. S.; Zatovsky, I. V.; Terebilenko, K. V. Electronic structures and origin of intrinsic luminescence in Bi-containing oxide crystals BiPO4, K3Bi5(PO4)(6), K2Bi(PO4)(MoO4), K2Bi(PO4)(WO4) and K5Bi(MoO4)(4), JOURNAL OF ALLOYS AND COMPOUNDS. 2014
b) Quintanilla, J.; Hooley, C. The Strong Correlation Puzzle. Physics World, Physics World Archive. 2009