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$KTa_{0.6}Nb_{0.4}O_3$粉体溶剂热和水热法合成的对比研究

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摘要 以Nb_2O_5和 Ta_2O_5为前驱反应物, KOH为矿化剂, 采用水热法和溶剂热法两种合成工艺制备了$KTa_{1-x}Nb_xO_3$ (KTN)陶瓷粉体.实验结果表明, 反应溶剂(水/异丙醇)和矿化剂KOH的摩尔浓度是影响KTN粉体结构和形貌的关键因素. 采用水热工艺制备的KTN粉体, 当KOH浓度达到3mol/L、反应温度为523K、反应时间8h时, 开始出现以焦绿石结构为主的KTN粉体; 随着KOH的浓度和反应温度的增加, 粉体中的钙钛矿结构成分随之增加, 而焦绿石结构则逐渐减少, 但始终难以完全消除. 采用溶剂热法, 在KOH浓度1~2mol/L、反应温度523K、反应时间8h的条件下, 合成了立方相钙钛矿结构$KTa_{0.6}Nb_{0.4}O_3$陶瓷粉体, KTN晶粒形状呈规则的立方体, 尺寸约为30~50nm; 最后对溶剂热法合成纳米粉体的机理进行了分析讨论.

关键词 钽铌酸钾 纳米粉体 溶剂热 水热 合成

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Hydrothermal and Solvothermal Preparation of Nanocrystalline KTN Powders

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Abstract

By using Nb_2O_5 and Ta_2O_5 as precursors, nanocrystalline potassium tantalite niobate $KTa_{0.6}Nb_{0.4}O_3$ (KTN) powders were prepared by hydrothermal and solvothermal (isopropyl alcohol) synthesis processing. The relations of crystal structure and the synthesis conditions of KTN powders were investigated. All results of measurement on samples obtained demonstrate that the microstructure and morphology of nanocrystal powders depend on the reaction temperature, pressure and pH values of solvents. The reaction solvents are very important factors. The KTN with pyrochlore structure fabricated by the hydrothermal method shows although the amounts of pyrochlore phase in samples decrease gradually as the pH values of solvents increasing, pure perovskite phase will be gained first and last. KTN ceramic powders with perovskite structure were successfully synthesized via solvothermal processing with 1--2mol/L of KOH at 523K for 8h. The SEM images of powders show that the shape of particles is mainly cubic and the size of particles is 30--50nm.

Key words KTN nanopowders solvothermal hydrothermal preparation

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