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热处理过程对湿化学法合成锂锰氧化物结构的影响

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摘要: 采用湿化学法在常压及温度低于100℃的水溶液中,使锂盐与锰盐发生化学反应生成 LiMn_2O_4 化合物,经过滤、洗涤后,在105℃时烘干制得无定型 LiMn_2O_4 化合物的先驱体.借助于X射线衍射及扫描电镜等现代测试手段研究了热处理温度及热处理时间对湿化学法合成 LiMn_2O_4 晶型结构及形貌的影响.结果表明,所得先驱体经适当的高温热处理可获得晶型结构完整的尖晶石型 LiMn_2O_4 ;热处理温度对 LiMn_2O_4 的晶体结构影响较大,随着热处理温度的升高,其X射线衍射峰的强度明显增大,晶体的结构趋于完整,且晶粒明显变粗;当热处理温度为500℃时,除 LiMn_2O_4 的衍射峰外,未见其它杂相的衍射峰,说明无定型的 LiMn_2O_4 已完全转变为尖晶石结构;在700℃时热处理4 h即可获得晶型结构完整、晶界分明、粒度均匀的 LiMn_2O_4 ;而热处理时间对 LiMn_2O_4 的晶型结构影响不大,相同温度下随热处理时间的延长,产物的X射线衍射峰的强度基本维持不变.

关键字: 锂锰氧化物;湿化学合成;晶体结构;热处理

Changes in the structure of spinel LiMn_2O_4 synthesized by wet chemical technique in the calcinating process

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Abstract: A LiMn_2O_4 compound was synthesized in aqueous solution from lithium compound which reacted with manganese compound by means of wet chemical technique at normal atmosphere and temperature of less than 100℃. After being filtered and washed with water, then dried at 105℃, the precursor of LiMn_2O_4 compound with an amorphous structure can be obtained. The changes in the crystal structure and the shape of LiMn_2O_4 samples with the calcinating temperature and time were detected by means of X-ray diffraction and scanning electronmicrograph. The results show that perfect spinel LiMn_2O_4 crystals can be obtained from the precursor of LiMn_2O_4 after being calcinated. It is concluded that the calcinating temperature has great influences on the crystal structure of LiMn_2O_4 . With the elevating of the temperature, the crystal structure becomes more perfect and the particle size gets larger. Only LiMn_2O_4 XRD patters can be observed when the sample is calcined at 500℃ and it is showed that the structure of LiMn_2O_4 compound is completely transformed from amorphous to spinel. A spinel type LiMn_2O_4 with regular crystal structure and well-distributed particlesize can be obtained after being calcinated for 4 h at 700℃. However, it is also observed that the calcinating

time has less effect on the perfection of the LiMn_2O_4 structure and the samples almost maintain the same XRD patterns when the calcinating time is prolonged at the same temperature.

Key words: lithiummanganese oxide; wet chemical synthesis; crystal structure; calcination

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