

Syntheses and Characterizations of Carbides and Lithiated Oxides of Group V and VI Metals

Student : Chun-Hsiung Chien

Advisor : Dr. Hsin-Tien Chiu

Institute of Applied Chemistry
National Chiao-Tung University

Abstract

The carbides and lithiated oxides of group V and VI metals were synthesized by means of homogenous solution-phase reaction and gas-solid reaction. In the solution-phase reaction, we explored three methods for the syntheses of LiTaO_3 , LiNbO_3 , LiMoO_2 and Mo_2C . Nanosized LiTaO_3 (50 – 100 nm) and LiNbO_3 (20 – 50 nm) powders can be obtained in liquid-phase reaction by heating the precursors generated between MCl_5 (M = Ta, Nb) and alkyllithiums in THF. LiMoO_2 powders were synthesized from a precursor prepared by reacting EtLi and $\text{MoOCl}_3(\text{THF})_2$ in the air for a week. Molybdenum carbide particles were prepared via carbothermal reduction of the precursors obtained from the reaction between $\text{MoOCl}_3(\text{THF})_2$ and $^n\text{BuLi}$ in the presence of THF. In the gas-solid reaction, we selected three compounds, 1-chlorobutane, hexachlorobenzene and carbon tetrachloride, as gas presursors for the reaction with Mo powders. $\text{Mo}_2\text{C}@a\text{-C}$ core-shell powders have been synthesized from the reactions between Mo metal powders with 1-chlorobutane or with hexachlorobenzene at 1173 K. Carbon hollow spheres (1 – 3 μm) and particles (200 – 300 nm) have been synthesized from CCl_4 and Mo at 873 K - 1173 K.

第五與六族金屬碳化物和鋰氧化物之合成及鑑定

學生：簡俊雄

指導教授：裘性天

國立交通大學應用化學研究所

摘要

第五與六族金屬碳化物和鋰氧化物已成功經由均勻液相反應及氣固相反應合成。在液相反應中，我們發展出合成三種鉭酸鋰 (LiTaO_3)、鈮酸鋰 (LiNbO_3)、鉬酸鋰 (LiMoO_2) 和碳化鉬 (Mo_2C) 的方法。奈米級鉭酸鋰 (直徑 50 – 100 nm) 和鈮酸鋰 (直徑 20 – 50 nm) 粉末可由 MCl_5 ($\text{M} = \text{Ta}, \text{Nb}$) 及有機鋰於四氫呋喃 (THF) 中反應所得之前驅物經鍛燒後而得；鉬酸鋰 (LiMoO_2) 粉末由 $\text{MoOCl}_3(\text{THF})_2$ 與乙基鋰於空氣中反應一星期所形成之前驅物經鍛燒後而得；碳化鉬 (Mo_2C) 顆粒可由 $\text{MoOCl}_3(\text{THF})_2$ 與正丁基鋰於四氫呋喃 (THF) 中反應所得之前驅物經鍛燒而得。

在氣固相反應中，我們選擇 1-氯丁烷、六氯化苯及四氯化碳當作氣相前驅物與固相的鉬金屬粉末反應生成碳化鉬 (Mo_2C) 粉末。 $\text{Mo}_2\text{C}@a\text{-C}$ 核殼粉末可由 1-氯丁烷、六氯化苯與鉬粉於 1173 K 反應合成；而四氯化碳與鉬粉的反應則可形成碳中空球與顆粒。

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