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Preparation of nanocrystalline metal oxides and intermetallic phases by controlled thermolysis of organometallic coordination polymers

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Abstract

Organometallic coordination polymers of the *super*-Prussian blue type $[(Me_3Sn)_nM(CN)_6]$ ($Me = CH_3$; $n = 3$, 4; M = Fe, Co, Ru) were subjected to thermolysis in different atmospheres (air, argon, hydrogen/nitrogen). In air, oxides were found: Fe_2O_3/SnO_2 (crystalline and nanocrystalline), Co_2SnO_4 and RuO_2. In argon and in hydrogen, the intermetallic phases $FeSn_2$, $CoSn_2$, Ru_3Sn_7 and Fe_3SnC were obtained. A detailed mechanistic study was carried out using thermogravimetry (TG), X-ray diffraction (XRD), X-ray absorption spectroscopy (EXAFS) at Fe, Co, Ru and Sn K-edges, infrared spectroscopy (IR) and elemental analysis. Below 250°C, Me_3SnCN and $(CN)_2$ are released, whereas above 250°C oxidation or pyrolysis leads to the corresponding oxides or intermetallic phases. Polymeric cyanides containing at least two metals have turned out to be suitable precursors to prepare well-defined oxides and intermetallic phases at comparatively low temperature.

Keywords: Thermolysis; Polymeric metal cyanides; Metal oxides; Intermetallic phases; Thermal analysis; Solid state reactions; EXAFS