



Molecular routes to group IV magnesium and calcium nanocrystalline ceramics.

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The effect of alkaline-earth-metal alkoxides on the protonolysis of $\text{Cp}_2\text{M}'\text{Cl}_2$ ($\text{M}' = \text{Ti}, \text{Zr}, \text{Hf}$; Cp = cyclopentadiene) was investigated. This approach enabled the design of compounds with well-defined molecular structures to generate high-purity binary metal oxides. Single-source molecular precursors with structures of $[\text{M}_2\text{M}'_2(\mu_3\text{-OEt})_2(\mu\text{-OEt})_4(\text{OEt})_6(\text{EtOH})_4]$ with $\text{M} = \text{Mg}$ and $\text{M}' = \text{Ti}$ (**1**), Zr (**2**), and Hf (**3**), $[\text{Ca}_6\text{Ti}_4(\mu_6\text{-O})_2(\mu_4\text{-O})_2(\mu_3\text{-OEt})_{12}(\text{OEt})_{12}(\text{EtOH})_6\text{Cl}_4]$ (**4**), and $[\text{M}_2\text{M}'_2(\mu_4\text{-O})(\mu\text{-OEt})_5(\text{OEt})_4(\text{EtOH})_4\text{Cl}]_n$ with $\text{M} = \text{Ca}$ and $\text{M}' = \text{Zr}$ (**5**) and Hf (**6**) were prepared via elimination of the cyclopentadienyl ring from $\text{Cp}_2\text{M}'\text{Cl}_2$ as CpH in the presence of $\text{M}(\text{OEt})_2$ and ethanol (EtOH) as a source of protons. Meanwhile, similar reactions involving the initial substitution of Cl ligands by OEt groups in $\text{Cp}_2\text{M}'\text{Cl}_2$ ($\text{M}' = \text{Ti}, \text{Zr}, \text{Hf}$) resulted in the formation of $[\text{M}_2\text{M}'_2(\mu_3\text{-OEt})_2(\mu\text{-OEt})_4(\text{OEt})_6(\text{EtOH})_4]$ with $\text{M} = \text{Ca}$ and $\text{M}' = \text{Ti}$ (**7**), Zr (**8**), and Hf (**9**). The precursors were characterized by elemental analysis, NMR spectroscopy, and single-crystal X-ray structural analysis. Magnesium compounds **1–3** decomposed at 750–850 °C to give MgTiO_3 along with small amounts of Mg_2TiO_4 , $\text{Mg}_2\text{Zr}_5\text{O}_{12}$, or $\text{Mg}_2\text{Hf}_5\text{O}_{12}$ binary metal oxides. The thermolysis of calcium compounds **4** and **7–9** led to highly pure CaTiO_3 , CaZrO_3 , or CaHfO_3 perovskite-like oxide particles with diameters of 20–30 nm.

Adres publiczny

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