化学学报 2003 61 (6): 869-877 ISSN: 0567-7351 CN: 31-1320/06

扩展功能

本文信息

Supporting info

PDF(0KB)

[HTML全文](0KB)

参考文献

服务与反馈

把本文推荐给朋友

加入我的书架

加入引用管理器

复制索引

Email Alert

文章反馈

浏览反馈信息

相关信息

本刊中 包含“水凝胶”的相关文章

本文作者相关文章

- 尹双凤
- 李莉
- 王恒秀
- 徐柏庆

碱液回流老化**ZrO(OH)_2**制备纳米晶**ZrO_2**的影响因素

尹双凤,李莉,王恒秀,徐柏庆

清华大学化学系

收稿日期 修回日期 网络版发布日期 接受日期

摘要 通过考察回流老化所用的碱液（NH_4OH, NaOH和KOH）介质和容器材质（玻璃 和Teflon）对ZrO(OH)_2凝胶及其焙烧产物ZrO_2的织构／结构和热稳定性的影响，
研究了杂质元素掺杂和凝胶溶解－再沉淀等因素在形成高表面积纳米晶ZrO(OH) _2/ZrO_2
过程中的作用。在Teflon容器中，以NH_4OH为介质（pH = 11.5）的回流 老化对ZrO(OH)_2/ZrO_2
的性质无明显影响。而使用玻璃容器则可显著提高ZrO(OH) _2/ZrO_2的表面积、孔容和抗烧结性质，并在800℃
获得小晶粒（5～7 nm）四方晶 相ZrO_2纳米晶材料；在DTA曲线上ZrO(OH)_2转变成ZrO_2
晶体的温度由回流老化前 的463℃提高到810～840℃。在以KOH和NaOH为介质(pH = 13)的实验中，使用玻璃
容器得到与经NH_4OH为介质时相类似的结果；但在Teflon容器中只形成低表面积和 较大尺寸（约20 nm）
以单斜相为主的混合晶相ZrO(OH)_2，其在800℃焙烧后形成 大晶粒（35 nm）
单斜相ZrO_2。样品的元素分析结果清楚地揭示出使用玻璃容器时 有SiO_2从器壁溶解掺杂进入ZrO(OH)_2
凝胶。样品的表面积和孔容与杂质Si~(4+) 含量之间有顺变关系，表明Si~(4+)掺杂是形成高表面积和大孔容ZrO(OH) _2/ZrO_2、提高ZrO_2晶化温度以及稳定小晶粒四方晶相ZrO_2的最主要因素。在不 发生Si~(4+)
掺杂前提下，K~+和Na~+的存在可促进ZrO(OH)_2形成结晶，但对高温 下ZrO_2
织构的稳定性影响不大。此外，ZrO(OH)_2凝胶的溶解－再沉淀和骨架网络 有序化也是回流老化影响ZrO(OH) _2/ZrO_2织构的重要因素。

关键词 水凝胶 氧化锆 纳米相材料 硅 掺杂 织构

分类号 0614

Effects of ZrO(OH)_2 Reflux-Digestion on the Preparation of ZrO_2 Nanocrystals

Yin Shuangfeng,Li Li,Wang Hengxiu,Xu Boqing

State Key Laboratory of C_1 Chemistry, & Technology, Department of Chemistry, Tsinghua University

Abstract The container materials (glass and Teflon) employed for reflux- digesting ZrO(OH)_2 gel in basic (NH_4OH: pH= 11.5, NaOH and KOH: pH = 13) solutions were shown to dramatically affect the texture, crystal phase and thermal stability of the ZrO(OH)2 and its calcined (600～800 °C) ZrO_2 products. Digestion in the glass container (G-preparation) led to prominent enhancement of the surface area, porosity and thermal stability of Zr0(OH)_2/ZrO_2 samples. And, the crystallization temperature on the sample DTA (differential thermal analysis) curves was shifted from ca. 460 t of the undigested ZrO(0H)_2 to 800 ～840 ° C of the digested ones. These effects were independent of the nature of the employed bases (NH_4OH, NaOH and KOH), and small tetragonal crystallites (5～7 nm) were always obtained after the calcination at 800 °C . In comparison, little effects were observed for the samples digested in the Teflon container (T-preparation). Elemental analysis revealed that incorporation into ZrO(OH)_2 gel of SiO_2 impurity from the glass container happened during the digestion. The surface area and pore volume of the ZrO(OH)_2 and ZrO_2 samples increased linearly with the amount of Si~(4+) -ions in the solids. The incorporated Si4+ - ions prevented the crystallization of ZrO(OH)_2 up to temperature higher than 600 °C , but they induced a stabilization of very small (5-7 nm) tetragonal crystallites after the calcination at 800 t. This role of the Si~(4+) -impurity was further confirmed with authentic SKVeontaining ZrO(OH)_2 gels prepared by coprecipitation of ZrOCl_2 and Si(OC_2H_5)_4 in the Teflon container (ZS-preparation) , When there was no incorporation of Si~(4+)-ions (i. e. in the T- preparation), the incorporation of alkali ions from NaOH or KOH solution could result in formation of low surface area (65～70 m /g) ZrO(OH)_2 crystallites (ca. 20 nm) with mixed monoclinic and tetragonal phases. But, the incorporated alkali ions showed little effect on the texture and crystal size of the calcined (600～800 X,) ZrO_2. In addition, dehydration and dissolution-reprecipitation of ZrO (OH)_2 gel during the digestion also affected the texture and crystal size of ZrO_2 but the effect was much less important than the incorporation of Si~(4+) -ions.

Key words HYDROGEL ZIRCONIUM OXIDE NANOPHASE MATERIALS SILICON DOPE TEXTURE

DOI:

通讯作者