

热处理温度对TiO₂结构性质及光催化活性的影响

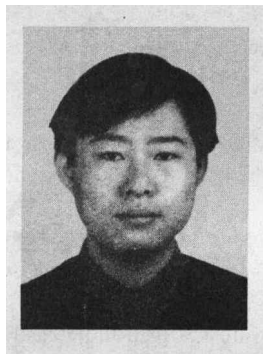
Effects of Thermal Treatment on Structural Properties and Photocatalytic Activity for TiO₂

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摘要 从晶相组成、晶格缺陷密度、表面羟基含量、晶粒尺寸以及比表面积等方面阐述了热处理温度与TiO₂光催化剂结构性质的关系, 并就混晶效应、载流子输运/俘获以及尺寸量子效应等对光催化活性的影响进行了讨论。此外, 还汇总了对水中典型有机污染物的光催化氧化研究以及相关的催化剂结构性质。

关键词: 热处理温度 二氧化钛 光催化活性



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1 前言

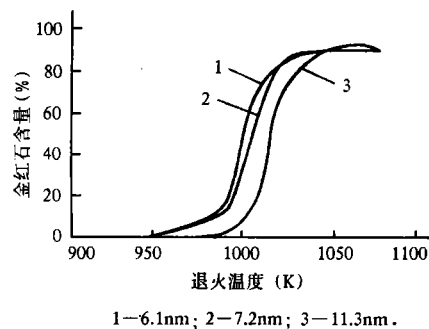
近年来, 半导体光催化氧化技术(Advanced Oxidation Process/Technology, AOP/AOT), 已成为环境领域的一个研究热点。在催化剂的研制方面, 许多制备和改性技术都是基于TiO₂进行的。研究表明, 不同温度、时间和氧化还原环境等热处理条件对催化剂的活性会产生很大影响。尤其是热处理温度, 它是决定催化剂晶体结构以及影响催化剂表面性质和尺度的重要因素^[1,2]。本文结合TiO₂的结构性质, 汇总和讨论了热处理过程中温度的影响及机理。

2 相变温度的影响因素

2.1 颗粒尺度的影响

郑茂盛等人^[3]运用Ising和Heisenberg模型建立了相变与颗粒尺度之间的定量关系。模拟结果表明, 催化剂的粒径越小, 相变的温度越低。Ding等人^[4]在溶胶-凝胶法制备TiO₂粉末试验中考察了纳米范

围内粒径对相变温度的影响, 由图1可见, 随着样品粒径减小, 相变温度曲线明显向低温方向偏移。他们在试验中采用低温热处理, 然后高温退火的方法, 在相对较低温度下制备了具有锐钛矿和金红石混晶的催化剂。



1—6.1nm; 2—7.2nm; 3—11.3nm.

图1 颗粒尺寸对相变温度的影响

Figure 1 Effect of particle size on temperature of phase transformation

2.2 金属氧化物掺杂的影响

许多催化剂的改性技术都涉及到金属或金属氧化物掺杂, 当这些物质介入时, TiO₂的晶格缺陷、晶粒尺寸、表面特性等都会发生较复杂的变化, 相变温度也会受到不同程度的影响。表1列出一些金属或金属氧化物掺杂对TiO₂相变温度的影响程度和对影响机理的解释。表1可以看出, 这种掺杂的影响较为复杂, 影响机理也尚无一定论。一些学者参照掺杂物质对Al₂O₃相变的影响, 基于液相烧结理论, 曾提出金属氧化物对TiO₂相变的影响主要与物质的熔点有关, 熔点低于TiO₂的物质对相变有促进作用, 并且熔点越低促进作用越明显, 反之, 则起抑制作用^[5]。

2.3 制备方法的影响

以氯化钛为前体的水热合成法的相变温度, 低于

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参加本试验工作的还有张彭义。

表 1 不同掺杂物质对相变的影响
Table 1 Effect of dopant on phase transformation

掺杂物质	相变温度的变化值	影响机理	文献
Ni	约降低 150℃	抑制了 TiO ₂ 晶粒的生长	[6]
V ₂ O ₅	约降低 15 ~ 100℃ ^[1] , 同处理温度下金红石含量增大	V 替代锐钛矿中的 Ti, 引起畸变, 促进金红石相的形成	[7]
ZnO	约降低 60℃	ZnO 熔化, 液相烧结, 促进相变 ZnO 弥散于晶粒之间, 充当成核中心	[8]
SnO	相变速率增大	与金红石微观结构相似, 充当金红石相的晶核	[5]
Fe ₃ O ₄ /Fe ₂ O ₃	相变速率增大	抑制了 TiO ₂ 晶粒的生长	[5, 9]
Nb ₂ O ₅	约升高 30℃	—	[10]
Ag ₂ O	约升高 200℃	—	[11]
Al ₂ O ₃	约升高 250℃	Al ₂ O ₃ 存在于 TiO ₂ 表面, 减少了金红石相的成核活性位	[12]
SiO ₂	约升高 100 ~ 300℃ (不同方法)	无定型 SiO ₂ 进入晶格, 增加了锐钛矿的热稳定性	[13, 14]

1) 根据文献中 XRD 谱图估计。

以钛醇盐为前体的溶胶-凝胶法^[15]。在水热合成法中, 使用 TiCl₃ 为前体的相变温度又低于 TiCl₄^[16]。Jung 等人^[13]的试验也表明, 溶胶-凝胶法中异丙醇钛前体制备的 TiO₂ 相变温度低于乙醇钛。此外, 溶胶-凝胶过程中一些有机物作为配位体使用, 也会在一定程度上影响相变温度。黄军华等人^[17]在溶胶-凝胶制备 TiO₂ 的过程中, 通过添加乙酸使相变温度降低约 100℃。Takahashi 等人^[18]也发现, 二乙醇胺能使锐钛矿转化为金红石的相变温度降低, 并且粉末 TiO₂ 降低的程度较薄膜 TiO₂ 显著。

3 热处理温度对晶格缺陷与表面羟基的影响

3.1 热处理对缺陷密度和羟基含量的影响

在通常的溶胶-凝胶制备过程中, 随着热处理温度的升高, 有机物质与水分逐渐被去除, 晶格缺陷密度增大, 表面羟基含量减少。其中, 晶格缺陷在很大程度上依赖于热处理的氧化还原条件, 氧化条件越充分, 晶格缺陷则越少。在空气下, 当温度继续提高到一定程度后, 晶格缺陷密度便会减小, 见表 2。

3.2 晶格缺陷、表面羟基与 TiO₂ 的活性

表 2 晶格缺陷和表面羟基随焙烧温度变化
Table 2 Changes of lattice defects and surface hydroxyl with calcination temperature

晶格缺陷		表面羟基	
温度 (℃)	Ti ³⁺ 浓度 (10 ¹⁸ spin/g)	温度 (℃)	化学吸附氧含量 (%)
130	1.16 ^[19]	320	26.09 ^[20]
450	322 ^[19]	480	19.86 ^[20]
		500	27.68 ^[21]
750	127 ^[19]	600	10.91 ^[21]

早期的研究认为, 晶格缺陷和表面羟基都有利于光催化反应的进行, 缺陷密度越大、表面羟基含量越高, 催化剂的活性也越高, 但具体的影响机理则有多种解释。Heller 等人^[22]认为, 晶格缺陷能够提高 TiO₂ 的 Fermi 能级, 增加表面能量壁垒, 使电子和空穴在表面的复合率降低。而 Salvador 等人^[23]则认为, Ti³⁺-Ti³⁺ 键距 (2.59Å) 小于 Ti⁴⁺-Ti⁴⁺ (4.59Å), 更容易与羟基自由基键合, 成为反应的活性中心。

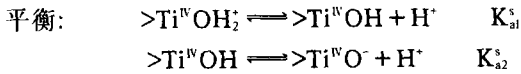
对于表面羟基在光催化反应中的作用, 看法则相对一致, 认为主要是俘获空穴, 形成羟基自由基·OH、超负氧离子 O₂⁻ 等活性物种, 间接实现有机物的氧化。

随着研究的深入, 人们开始认识到晶格缺陷和表面羟基, 对催化剂活性影响的双重作用, 即它们在俘获空穴以及形成氧化活性物种的同时, 也可能成为载流子复合的中心, 降低反应活性^[24]。最近, 又有学者提出, 在载流子的输运和俘获过程中, 表面羟基和晶格缺陷, 能同时起到俘获和复合的作用。其中羟基俘获空穴、缺陷, 同吸附的分子氧一起转移电子, 分别是主要作用。只有当它们比例合适时, 才会促进载流子的分离和界面电荷的转移。否则, 它们会体现出载流子复合中心的作用, 使光催化活性降低 (表 3)^[25]。

此外, 催化剂的表面羟基在水中有如下的酸碱

表 3 晶格缺陷与表面羟基在载流子输运过程中的作用
Table 3 Function of lattice defects and surface hydroxyl in course of transportation of carriers

主要作用	反应过程
表面羟基	h ⁺ 俘获 $>Ti^{IV}OH + h^+ \longrightarrow >Ti^{IV}OH^{\cdot}$
	复合 $>Ti^{IV}OH^{\cdot} + e^- \longrightarrow >Ti^{IV}OH$
晶格缺陷	e ⁻ 俘获 $>Ti^{\cdot} \cdots O_2 + e^- \longrightarrow >Ti^{\cdot} \cdots O_2^{\cdot} \longrightarrow >Ti^{\cdot} + O_2^{\cdot}$
	复合 $>Ti^{\cdot} \cdots O_2 + h^+ \longrightarrow >Ti^{\cdot} \cdots O_2$



热处理温度能够通过改变羟基含量,影响催化剂的表面电荷零点,从而使离子型反应物质在催化剂表面的吸附性能不同。

4 热处理温度对颗粒尺度与比表面积的影响

随着热处理温度的升高, TiO₂在晶化与相变过程中粒径不断增大,比表面积则由于微孔被烧结以及晶粒增大等因素而减小。对于一般的多相催化反应,这种变化会使催化剂的活性大大降低。但对于光催化反应来说,由于催化剂热处理中伴随着结晶度、晶格缺陷、表面羟基等变化,在反应过程中又涉及到光吸收以及载流子俘获/复合等因素,因此,催化剂的粒径和比表面积与活性之间,并不存在严格的相关关系。

热处理不充分的催化剂,往往具有较大的比表面积,这样的催化剂锐钛矿含量较少,同时由于表面键合或吸附羟基密度过高,载流子的复合过程可能成为主要作用,在一些非传质限制的反应体系中,经常会表现出低活性。只有结晶度、相组成、晶格缺陷密度以及表面羟基等相同或相近时,才能确认大比表面积的催化剂具有较高的活性。

粒径的改变会使催化剂的光、电化学性质发生很大变化。首先,粒径减小有利于载流子的分离。光激发产生的电子和空穴必须迁移到半导体的表面,才能与电子受体/给体作用,粒径的大小决定其迁移至表面所需要的时间。在粒径为 1 μm 的 TiO₂ 粒子中,电子从内部扩散到表面约需 100ns,而粒径为 10nm 的微粒只需 10ps,所以粒径越小,电子与空穴简单复合的几率越小,光催化活性也好。其次,当半导体的颗粒尺度进入

纳米范围内(<20nm)时,载流子限制在一个小尺寸的势阱中,导带和价带过渡为分立的能级,会产生尺寸量子效应,导致半导体的禁带变宽,吸收光谱蓝移。尺寸量子效应的出现有益于催化剂活性的改善(表 4)。

表 4 锐钛矿型 TiO₂ 的尺寸量子效应
Table 4 Size quantum effect of anatase TiO₂

粒径 (Å)	BET 表面积 (m ² /g)	最大激发波长 (nm)	带隙蓝移量 (ev)	量子产率 (%)
38	1068	371.5	0.156	0.0718
50	941	375	0.126	0.0702
65	609	380.5	0.079	0.0880
85	430	385	0.041	0.0242
110	312	387	0.024	0.0226
220	137	388	0.016	0.0113
530	26	389.9	0.000	0.00264

5 水中有机污染物的光催化降解

从目前研究现状看,尽管光催化技术在去除难降解有机物质方面有较大的应用潜力,但由于催化剂的活性尚不够高,无法大规模应用。如何进一步改善催化剂的性能已成为该技术的核心问题。表 5 结合热处理温度对催化剂结构性质的影响,总结了一些典型污染物光催化降解的研究结果。从表 5 可以看出,光催化氧化对多种有机物都具有较好的去除效果,且反应体系和催化剂制备方法不同,影响去除效果的主要结构因素也不一样,它们在很大程度上受热处理过程的影响。

6 结语

在 TiO₂ 半导体光催化剂的制备过程中,热处理是获得高活性的必要手段和有效途径。在不同温度下进

表 5 水中典型有机污染物的光催化氧化研究
Table 5 Study on photocatalytic oxidation of representative organic pollutant in water

有机污染物	去除效果描述	最佳热处理温度(℃)	影响去除效果的主要结构性质	文献
乙酸	远优于 DegussaP25	—	高晶化度和大比表面积	[26]
三氯乙烯	远优于 DegussaP25	700	涉及 Si 掺杂的影响	[27]
苯酚	大部分去除	650	颗粒尺度和比表面积	[28]
2-氯酚	大部分去除	Degussa P25	大比表面积	[29]
2,4-二氯酚			小粒径	
2,4,6-三氯酚			混晶效应	
活性艳红 X-3B	完全脱色	500	比表面积和晶相组成	[本研究组]
对氨基偶氮苯	完全脱色	650	晶型、晶粒度和比表面积	[30]
对羟基偶氮苯				
敌敌畏、久效磷	大部分去除	600	混晶效应	[31]
阿特拉津	几乎完全去除	650	—	[32]
地下水中 VOCs	几乎完全去除	—	美国 Matrix 系统	[33]

行热处理将会使催化剂的晶相组成、晶格缺陷密度、表面羟基含量、晶粒尺寸和比表面积等性质产生很大差异,进而呈现出不同的光催化反应活性。在催化剂的制备过程中,结合具体的制备方法和工艺条件,对影响活性的主要结构因素进行分析,选择合适的热处理温度,将会在很大程度上改善催化剂的活性。

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**Approach on General Conception of Implementing
River Valley Management Effectively**

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The fact that getting worse of water ecological environment in Yangtze River Valley has affected and restricted the sustainable development to the area along the river, especially to Shanghai. Although great efforts have been made by the provinces along the river, the deterioration of water quality has not been curbed. The main reason lies in the lack of joint efforts and effective control over the whole valley. Local governments should take their own responsibility, take comprehensive measures and cooperate with each other so as to establish a sustainable management module to make Yangtze River cleaner.

Key words: Yangtze River River valley management
urgency General conception

**Study on Effect of Regional Environmental Management
System in Jinqiao Export Processing Zone of Shanghai**

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Sciences, Shanghai 200233)

Taking ISO14001 Environmental Management System of Shanghai Jinqiao Export Processing Zone as a case study, the management modes of the development zone in China and the necessity of implementation ISO14001 in such zones have been discussed extensively. The major features, operating mechanisms and performance of regional EMS managed by the development company were expounded.

Key words: Regional environmental management system
Development zone Management mode
Management platform Treaty Demonstration
Shanghai Jinqiao

**Approach on Systematic Design and Scheme
of Automatic Monitoring**

Liu Hong Wang Yue
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Center, Shanghai 200030)

According to the current status of Shanghai environmental monitoring, this article proposed a suggestion on automatic monitoring and optimization of monitoring net from atmospheric environment, emission sources, water and wastewater etc., and the feasibility from fit out measure-

ment and management were also discussed

Key words: Environment Automatic monitoring
Systematic design Improved scheme

**Study on Establishment and Verification of Indicator
System for Strategic Environmental Assessment (SEA)**

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Discussion on the connotation of indicator system for SEA, and types of indicator, then proposed the methods on screening principles, process and weight definition of useful indicators were presented. Finally, a case study on SEA for China Energy Development and the proposed indicator system were also presented

Key words: Strategic environmental assessment (SEA)
Indicator system China energy Verification

**Study Progress on Exhaust Gas Emission
from Natural Gas Vehicles**

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Review on current development of exhaust emissions from natural gas vehicle and some advanced emission control techniques, and experimental emission data of CO, CO₂, NO_x, total hydrocarbons, and FTP toxic compounds etc. were presented. In comparison with the emission data of natural gas vehicle in China showed that the emission control technique of natural gas vehicle in China was just corresponding to 1970s level in the US.

Key words: Liquefied natural gas
Automobile exhaust gas
Purification of exhaust gas
Automobile exhaust catalytic cleaning cartridges

**Effects of Thermal Treatment on Structural Properties
and Photocatalytic Activity for TiO₂**

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Review on the relation between calcination temperature and structural properties of TiO₂ with aspects to the composition of phase, density of lattice defects, contents of surface hydroxyl, size of crystal particles and special surface area were presented. It also discussed some processes which influence the photocatalytic activity, such as two-phase ef-

fect, transportation/trap of carriers and size quantum effect. Furthermore, photocatalytic removal of representative organic pollutant in water and relevant characteristics of catalyst were summarized.

Key words: Thermal treatment temperature
Titanium dioxide Photocatalytic activity

Technical Progress on Detecting Dioxins in Environment

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Based on description of the structure, source and toxicity of dioxins, briefly presented the indispensability of monitoring dioxin-like compounds, such as PCDDs and PCDFs which could cause carcinoma, reproductive effects, immune response and dermatoses etc. Review on the current methods of measuring dioxin-like compounds, both instrumental chemical analysis and biomonitoring concerned, such as GC/MS and bioassay etc. at home or abroad were also presented.

Key words: Dioxins GC/MS Biomonitoring

Study Progress on Cyanobacterial Ecology

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Under proper environmental conditions, *cyanobacteria* often form waterblooms which will make harmful to human health. World widely, lots of researchers have been studied the *cyanobacterial* ecology. This paper deeply introduced the relationship between the growth and toxin production of *cyanobacteria* and environmental factors for nearly 20 years. Lots of studies showed that the growth and toxin production of *cyanobacteria* were effected by kinds of environmental factors, such as light, temperature, nitrogen, phosphorus, trace elements, dissolved oxygen, chlorophyll a, which collaboration led to the vibration of the degree of the growth and toxin production of *cyanobacteria* and the seasonal variation of predominant algae strains in water body.

Key words: *Cyanobacteria* Waterblooms
Cyanotoxin Environmental factors
Ecological study

Study Trends on Urban Motor Vehicle Pollution Dispersion Models

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There are two directions in research field of urban vehicle

exhaust dispersion, namely, street pollution dispersion in city-scale and street canyon dispersion in micro-scale. On this paper, advances in urban street canyon pollution dispersion were presented, the research background and the results of main methods were introduced.

Key words: Motor vehicle exhaust pollution
Wastegas Urban street Pollution diffusion
Pollution model

Experimental Study on Chemical Stabilization in Treating Fly Ash with Heavy Metal Chelating Agent

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Experimental study on the technological process and treatment efficiency of heavy metal chelating agent in treating fly ash from MSW incinerator, and compared with the results of inorganic chemical agents such as Na₂S and lime used in treating this kind of hazardous waste have been conducted. The results indicated that the heavy metal removal rate from the treated fly ash using this chelating agent was higher than 97%, and the amount of chelating agent used was much less than that of inorganic chemical agents to reach the same stabilization effect. Furthermore, the treated fly ash using this chelating agent didn't influence by the microorganism activity in the waste column.

Key words: Chemical stabilization
Heavy metal chelating agent
Municipal solid waste
Fly ash Heavy metal waste

Study on Regulating Verification of Water Supply and Demand in Central Area of Shanghai

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Through scientific study on water supply & demand in Xuhui District, an initial analysis on existing problems in water management in the central area of Shanghai was presented. From the angles of water saving, water quality, temporal-spatial features of water resource, city hydrologic effect, economic regulation & control and digital management, a preliminary approach on water resource management was also put forward. The aim of this article is to provide the research method of water supply & demand coordination in the city central area, and also to give an objective basis for leaders' policy decisions.

Key words: Shanghai city Center Xuhui District
Water resource
Analysis of water supply & demand
Regulation & control