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博士学位论文

纳米粒子形状控制合成功能分子在Pt单晶电极表面的吸附和反应特性：原子和分子层次研究

Study of adsorption and reaction of functional molecules on Pt single crystal electrodes at atomic and molecular level

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摘要

纳米材料形状控制合成是制备功能纳米材料、特别是高性能催化剂的重要途径。在纳米材料形状控制合成中，覆盖剂（Citrate、PVP）以及还原剂（抗坏血酸）等功能分子起着决定性的作用，但其调控作用机制亟需深入研究。本论文运用电化学循环伏安（CV）、电化学原位红外光谱（FTIR）和扫描隧道显微镜（STM）等技术研究了纳米材料形状控制合成功能分子在Pt(hk1)和Pd/Pt(hk1)单晶电极表面的吸脱附和反应过程；结合Pt纳米粒子合成，探讨其在纳米材料形状控制合成中的作用机制。主要研究结果如下：

1、研究表明柠檬酸根在Pt单晶电极表面发生特性吸附，在不同晶面上具有不同的吸附行为。在Pt(311)和Pt(100)电极上，柠檬酸根从0.30V左右开始从表面脱附，到0.20V已经完全脱附；而在Pt(111)电极上，柠檬酸根从0.40V开始脱附，随电位负移逐渐脱附，到0.20V左右完全脱附。在纳米粒子表面柠檬酸根的吸脱附行为与在Pt(hk1)单晶电极表面的行为类似。柠檬酸根会增强H在(100)位点吸脱附峰的强度这一现象可以用于表征纳米粒子表面(100)位点的比例。

2、研究发现PVP在Pt单晶电极表面吸附具有结构选择性，在Pt(100)电极表面发生强吸附，而在Pt(111)电极表面是弱吸附。PVP在Pt(100)电极上吸附会抑制(100)平台位和台阶位氢的吸脱附；PVP在Pt(111)电极上吸附对氢在(111)平台位的吸脱附没有影响，但会抑制硫酸根/硫酸氢根、氢氧根等阴离子在(111)平台位的吸脱附。

PVP在不同位点的吸附强弱具有以下的顺序

： $L(100) > S(100) \times (111) \approx S(111) \times (100) \approx S(111) \times (111) > L(111)$ 。STM研究结果

指出，PVP在Pt(100)和Pt(111)表面的吸附结构类似，在表面的吸附是以一个整体吸附在表面，每个聚合物分子占据 $2 \sim 4$ nm的表面位，而且在小范围内以六方密堆积形式在表面排列。电化学原位红外光谱从分子水平诠释了PVP在Pt(100)和

Pt(111)电极表面不同的吸附行为，在Pt(100)电极表面吸附的PVP羰基红外吸收峰位于 1663cm^{-1} ，而在Pt(111)电极上这个谱峰蓝移至 1671cm^{-1} ，证实了PVP在

Pt(100)电极上吸附更强。在Pt(100)电极上PVP的吸附使CO的Stark系数从 $21\text{cm}^{-1} \bullet \text{V}^{-1}$ 降到

$9\text{cm}^{-1} \bullet \text{V}^{-1}$ ，而对于Pt(111)电极表面则没有影响。电极表面吸附PVP对

电催化活性存在较大影响，可以通过piranha溶液除去电极表面吸附的PVP。

3、研究证实使用“force deposit”方法可在Pt单晶电极表面沉积单层至多层Pd。柠檬酸根在Pd/Pt(100)和Pd/Pt(111)电极上的行为与硫酸根类似。PVP在Pd/Pt(111)的吸附与Pd原子层数相关；而在Pd/Pt(100)电极表面的吸附会抑制Pd-H峰。PVP在Pd/Pt(100)电极表面的吸附强于在Pd/Pt(111)上的吸附，与在Pt(hkl)电极表面得到的结果一致。

4、研究揭示抗坏血酸电氧化是表面结构敏感的过程，且在手性晶面上具有手性选择性。台阶原子密度高的Pt(311)电极上在较低电位就开始解离生成CO，台阶原子密度低的表面在0.28V出现抗坏血酸的脱附峰。在两组手性晶面Pt(321)和Pt(643)电极的研究发现，表面为R型的Pt(321)R和Pt(643)R两个晶面对L-抗坏血酸和D-异抗坏血酸都具有较高的催化活性。

本论文系统研究了纳米材料形状控制功能分子在表面原子排列结构明确的单晶表面的吸附规律，同时运用原位红外反射光谱和扫描隧道显微镜从分子水平和原子尺度揭示其吸附反应机理。研究结果对于进一步认识功能分子在纳米合成中的机理以及理性地设计和制备高性能纳米催化剂具有重要意义。

关键词：柠檬酸根；PVP；Pt(hkl)；原位FTIR；STM

Abstract

Nanoparticles shape controlled synthesis is an important way to prepare functional material, especially for high performance catalyst. The functional molecules as capping agent (Citrate, PVP) and reducing agent (ascorbic acid) play a key role in the synthesis. However, the mechanism of functional molecules is still unclear. The adsorption of functional molecules on Pt(hkl) and Pd/Pt(hkl) electrodes were investigated by using cyclic voltammetry, STM and in situ FTIR spectroscopy, to understand the mechanism of functional molecules in nanoparticles shape controlled synthesis combined with the synthesis process. The main results are summarized below:

1. Citrate behaves as a simple, specifically adsorbing anion and is sensitive to surface structure. On Pt(311) and Pt(100), citrate starts to desorb from surfaces at 0.3V and complete at 0.2V; the desorption potential on Pt(111) is from 0.4V to 0.2V; which indicated the adsorption of citrate on Pt(111) is weaker than that on Pt(311) and Pt(100). Citrate can be used to test the Pt(100) terrace contributions on nanoparticles as the intensity increase and narrowing of H UPD features associated with the Pt(100) terrace in the solution containing citrate.

2. It is found that the adsorption of PVP on Pt single crystal is surface sensitive.

The adsorption of PVP on different Pt surface sites has the sequence:

$L(100) > S(100) \times (111) \approx S(111) \times (100) \approx S(111) \times (111) > L(111)$. PVP is bonded strongly to Pt(100), but weakly on Pt(111). The adsorption structure of PVP on Pt(100) and Pt(111) are similar in the STM images, the polymer acts as a big domain on the surface which is about 2-4nm. With in situ FTIRS, we can distinguish that the bond between PVP and Pt(100) is stronger than Pt(111), as the carbonyl band is red shift to 1663 cm^{-1} on Pt(100) compared with 1671 cm^{-1} on Pt(111). We also use CO as "probe molecule", and the result supports our conclusion. We have achieved the key of that why PVP can be served to control

the nanoparticles morphology.

3. We use “force deposit” method to achieve monolayer to multilayer Pd on Pt single crystal surfaces. The behavior of citrate on Pd/Pt(hkl) is similar to sulfate, as a simple, specifically adsorbing anion. PVP adsorbs strongly to Pd/Pt(100), but weakly to Pd/Pt(111).

4. The electro-oxidation of ascorbic acid depends strongly on Pt surface structure and is enantioselective to chiral surface. With long (100) terrace, L-ascorbic acid gives a peak at 0.28V on the 1st cycle CV curve, which is confirmed to be the desorption of ascorbate. L-ascorbic acid dissociates to produce CO on Pt(311) at lower potential which would poison the surface. The R type chiral surfaces Pt(321)R and Pt(643)R are more active to L-ascorbic acid and D-isoascorbic acid. We have studied the adsorption of functional molecules on Pt single crystal surfaces, combined with in situ FTIRS and STM to reveal the mechanism of these molecules in nanoparticles shape controlled synthesis. It's of importance to provide guidance on designing and preparing high performance nanocatalyst.

Keywords: citrate;PVP;Pt(hkl);in situ FTIR;STM

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