中国科学院大学物理科学学院

奖学金申请表

学科专业	凝聚态物理	学生姓名	李傲雯
导师姓名	周武	本人联系方式	18133659336
就读年级	直博第 6 年		
研究方向	电子显微学	攻读学位	博士
此次申报奖学金名称		国家奖学金	

在学期间学习成绩、科研工作及取得的成果介绍

本人的博士课题研究方向为**面向纳米能源材料的单原子精度电子显微谱学分析技术开发**。在导师周武教授的指导下，于国际上首次实现了**具有化学键合灵敏度的单原子振动谱学测量**，这对于揭示缺陷处原子的成键构型和原子质量对局域晶格振动模式的影响提供具有重要意义，相关代表性研究论文已发表在材料领域顶级期刊 ***Nature Materials*** 上，本人是该论文的共同一作。本人还开发了单原子精度的**扫描透射电镜电子能量损失谱（STEM-EELS）芯损谱扩展边精细结构技术**，首次将材料局域配位环境的解析精度推进到了单个原子的新极限，相关结果已在第 20 届国际显微学大会（The 20th International Microscopy Congress（IMC））做口头报告，论文正在整理投稿。此外，本人还首次实现了**利用低电压 STEM-EELS 技术对负载于较重载体表面的较轻过渡金属物种进行单原子精度化学成像以及单原子尺度的化学价态谱学分析**，为能源催化材料的活性位点原子结构表征以及揭示材料构效关系提供有力手段。通过与国内外多个优秀课题组合作，本人已利用该低压 STEM-EELS 技术对多个催化材料体系的活性位点结构进行精细解析，为揭示材料的催化活性起源做出重要贡献，相关代表性研究论文已发表在 ***Journal of the American Chemical Society***（共同第一作者），***Nature Nanotechnology*** 和 ***Nature Communications***（共同第一作者）等高水平期刊上。基于上述电子显微方法学上的突出成果，本人成功入选 4 年一届的国际显微学大会的 IFSM Young Scientist（面向全球博士生和博士后进行评选，每届仅 50 人入选）。

此外，本人还参与在 ***Journal of Materials Chemistry A*** 和 ***Topics in Catalysis*** 上发表的阐述单原子分辨电子显微学在能源材料中的应用的综述论文以及 **Springer Handbook of Advanced Catalyst Characterization** 书籍中扫描透射电镜相关章节的撰写。

论文：

1. Xu, M.[#]; Bao, D.-L.[#]; **Li, A.[#]**; Gao, M.; Meng, D.; Li, A.; Du, S.; Su, G.; Pennycook, S. J.; Pantelides, S. T.[*]; Zhou, W.[*], Single-atom vibrational spectroscopy with chemical-bonding sensitivity. ***Nature Materials*** **22**, 612, 2023.（**共同第一作者**）

在该研究中，我们通过对商业化仪器稳定性的改进，进一步提高扫描透射电镜中振动谱的测量精度，成功地在石墨烯点缺陷结构处探测到由局域成键构型不同而导致的局域晶格振动特征的变化，并在原子尺度实现了化学键和原子质量对晶格振动的影响效应的解耦合，**在单原子精度上揭示了缺陷原子的成键构型和原子质量对局域特征晶格振动谱信号的影响**，为理解单个原子缺陷对局域晶格振动模式的影响提供了直接的实验依据，**从而将单原子精度振动谱学测量技术的探测灵敏度推进到了具有化学键合敏感的新极限**。在该工作中，本人作为共同第一作者，参与了本工作中 Nion HERMES-100 电镜的电子光路优化工作和暗场 EELS 振动谱学技术开发，关键实验数据的采集，发展了适用于处理单原子振动谱学数据解析的 python 代码，并参与了实验数据的解析及论文的撰写与修改。

2. Lin, L.[#]; Yu, Q.[#]; Peng, M.[#]; **Li, A.[#]**; Yao, S.; Tian, S.; Liu, X.; Li, A.; Jiang, Z.; Gao, R.[*]; Han, X.; Li, Y.-W.; Wen, X.-D.; Zhou, W.[*]; Ma, D.[*], Atomically dispersed Ni/α-MoC catalyst for hydrogen production from methanol/water. ***Journal of the American Chemical Society*** **143**, 309, 2021.（**共同第一作者**）

在该研究中，北京大学马丁教授团队通过将廉价的镍（Ni）原子级地分散在 α-MoC 上，开发出了经济高效的低温甲醇/水重整产氢 Ni/α-MoC 催化剂，有望显著降低催化产氢的生产和使用成本。然而，负载于较重载体的较轻元素物种无法通过常规电子显微成像进行观察，揭示其活性位点的原子结构极有挑战。**我们通过对电子光路、实验方法以及数据处理方法的优化，利用球差校正的低电压**

STEM-EELS 技术，首次实现对较重载体表面的较轻过渡金属元素的单原子精度化学成像和化学价态谱学分析，确认 Ni 以单原子的形式分散在 α-MoC 载体表面并且其电子态呈现出更偏金属的缺电子状态，表明高分散 Ni 原子与 α-MoC 载体之间存在少量电荷转移。上述电子显微谱学分析结果为揭示该高效产氢催化剂活性位点结构及其催化活性起源提供了原子尺度的精确结构信息。在该工作中，本人作为共同第一作者，发展了低电压 STEM-EELS 技术以及将之应用于催化剂样品的表征中，并且完成了电镜相关数据的分析和文章内容的撰写和修改。

3. Ge, Y.[#]; Qin, X.[#]; **Li, A.**[#]; Deng, Y.; Lin, L.; Zhang, M.; Yu, Q.; Li, S.; Peng, M.; Xu, Y.; Zhao, X.; Xu, M.; Zhou, W.[*]; Yao, S.[*]; Ma, D.[*], Maximizing the synergistic effect of CoNi catalyst on α-MoC for robust hydrogen production. ***Journal of the American Chemical Society*** **143**, 628, 2021.（共同第一作者）

在该研究中，北京大学马丁教授团队利用钴（Co）和镍（Ni）双原子的协同效应开发了一种能高效催化硼烷氨水相分解产氢的单原子分散的 CoNi/α-MoC 催化剂。由于负载于 α-MoC 载体上的 Co 和 Ni 无法通过常规电子显微成像技术进行观察和区分，**我们利用本课题组前期发展的球差校正的低压 STEM-原子分辨 EELS 技术对元素周期表上相邻的 Co 和 Ni 元素进行直接化学成像，确认 Co 和 Ni 以单原子的形式分散在 α-MoC 载体表面。同时，我们还发现载体上高密度的 Co 和 Ni 原子能够形成空间上近邻的 Co-Ni 原子对结构，这表明 Co 和 Ni 在催化反应中可以形成双金属活性中心从而协同催化硼烷氨水相分解反应。**上述电子显微学分析结果为揭示 CoNi/α-MoC 催化剂相较单金属催化剂的显著性能提升提供了原子尺度的结构信息。在该工作中，本人作为共同第一作者，完成了催化剂样品的单原子尺度 STEM-EELS 表征，并且完成了电镜相关数据的分析和文章内容的撰写和修改。

4. Xu, M.; **Li, A.**; Pennycook, S. J., Gao, S.-P.[*]; Zhou, W.[*], Probing a defect-site-specific electronic orbital in graphene with single-atom sensitivity. ***Physical Review Letters***, in press (2023).

在该研究中，我们利用无读取噪音的直接电子探测器结合多帧扫描的谱学成

像手段，成功地避免了单层样品中点缺陷处在 EELS 数据采集过程的结构损伤，**实现了石墨烯中四配位硅原子缺陷（Si-C_4）处单原子精度的电子能量损失谱近边精细结构（ELNES）的面分布分析**。通过解析碳原子的近边精细结构信号，我们发现 Si-C_4 缺陷附近的 C K 边信号中存在前置峰，结合理论计算发现实验所观察到的前置峰反映了碳原子中未占据的 p_z 轨道的信息。该成果展示了 **STEM-ELNES 能够在单原子尺度上对缺陷处的电子轨道信息进行解析，从而为理解化学键合对局域缺陷的性质的影响提供直接的实验依据**。在该工作中，本人参与了该原子级 STEM-ELNES 面分布实验的参数优化，并对文中关键数据解析及文章的修改提供意见。

5. Gao, Z.[#]; **Li, A.**[#]; Ma, D.[*]; Zhou, W.[*], Electron energy loss spectroscopy for single atom catalysis. ***Topics in Catalysis* 65**, 1609 (2022).（**共同第一作者**）

该综述总结了近年来本课题组及国内外其他课题组利用单原子电子能量损失谱技术揭示单原子催化剂活性位点结构及催化性能起源的前沿代表性工作，**系统阐述了单原子电子能量损失谱的发展历程和表征优势，展示了该技术在单原子催化剂研究中的巨大潜力**。我在其中承担了一半的写作工作。

专利：

无

书籍著作：

1. He, Q., **Li, A.**, Yao, B., Zhou, W., Kiely, C., STEM High Angle Annular Dark-Field Imaging. In *Springer Handbook of Advanced Catalyst Characterization*, Wachs, I. E.; Bañares, M. A., Eds. Springer International Publishing: Cham, pp 409-448 (2023).

该章节主要向具有催化领域研究背景的科研人员介绍球差校正 STEM-HAADF 技术的理论知识和其在催化研究中经典的应用案例，从而帮助非电镜专业人员建立对 HAADF 成像的基本成像原理、实验中的重要参数、图像中蕴含的催化剂结构信息以及如何正确解析图像数据有全面的认知。我在其中负责

HAADF 成像的理论知识，低压 STEM-HAADF 成像技术应用和 HAADF 成像在 WO_x/ZrO_2 催化剂中的应用三个部分的写作。

2. **Li, A.**, Xu, M., Ma, D., Zhou, W. Case Studies: Aberration Corrected High-Angle Annular Dark-Field (AC-HAADF) Microscopy. In *Springer Handbook of Advanced Catalyst Characterization*, Wachs, I. E.; Bañares, M. A., Eds. Springer International Publishing: Cham, pp 449-457 (2023).

该章节为上一章节的附属案例章节，旨在通过系统介绍 STEM-HAADF 成像技术在一系列金属负载 α-MoC 催化剂体系中的应用使读者对该技术在催化领域的应用有更加清晰的认识。我在其中承担了全文主要的写作工作。

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1. 三好学生荣誉称号（2020-2021 学年）

其他获奖情况：

1. 2022 年度中国科学院院长优秀奖（2022 年 7 月）
2. 第 20 届国际显微学大会（The 20th International Microscopy Congress）IFSM Young Scientist（韩国釜山，2023 年 9 月）

在学期间参加公共服务情况

1. 协助组内工程师老师管理 JEM-ARM300F2 电镜
2. 负责国科大电子显微学实验室**电镜原位加热/气氛样品杆**采购的技术论证、工厂测试等具体事务；

推荐意见：

李傲雯同学是我课题组 18 级直博生，她对学术有着极大的热情，在单原子精度的扫描透射电镜电子能量损失谱（STEM-EELS）技术开发和应用方面做出了非常优秀的成果，体现出极高的科研能力和学术潜力。她的一项重要工作是发展**具有原子分辨的 EELS 芯损谱扩展边精细结构技术（EXELFS）用于解析样品的局域配位环境，她通过对实验的优化，成功地将该技术的分析极限从纳米尺度推进到原子尺度，**为理解半导体和催化剂等功能材料的构效关系提供了新的实验手段。

该成果是我们实验室近年来最重要的研究成果之一，虽然论文尚未正式发表，但我在国际会议上介绍该成果时均得到最热烈的赞赏。此外，她还在我们课题组的**单原子尺度晶格振动谱技术**开发中发挥重要贡献，与课题组内其他成员合作**将该技术的探测灵敏度推进到化学键合的水平，从而成功揭示了缺陷原子的成键构型和原子质量对局域晶格振动模式的影响**，该工作今年年初发表于 ***Nature Materials*** 上，李傲雯同学是该论文的共同第一作者。基于在单原子尺度电子显微谱学研究的工作，李傲雯同学被选为今年**国际显微学大会的 IFSM Young scientist**，该项目面向全球所有参会的博士生和博士后进行评选，每届仅入选 50 人，是该 4 年一届的国际显微学大会给出的青年学者最高荣誉，其中今年中国大陆高校和科研院所中仅有 1 名博士生和 2 名博士后研究员入选，体现了李傲雯同学的科研成果在国际显微学届所受到的充分认可。李傲雯同学在自己的博士研究工作之外，还积极协助管理实验室的大型科研仪器设备，并承担组内的公共服务工作。

综上，李傲雯同学在学术上追求进步，勇于探索，积极参与学院和实验室的各项活动及公共服务，在日常学习生活中尊敬老师、友爱同学，是一位德智体全面发展的优秀博士生。因此，我强烈推荐她申报物理学院国家奖学金。

推荐人签名： 周武

日期：2023 年 9 月 26 日

学生郑重承诺以上陈述及提交的全部书面材料真实有效。

学生签字：李傲雯　　日期：2023 年 9 月 26 日

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Single-atom vibrational spectroscopy with chemical-bonding sensitivity

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Mingquan Xu[1,6], De-Liang Bao[2,6], Aowen Li[1,6], Meng Gao[1], Dongqian Meng[1], Ang Li[1], Shixuan Du[1,3], Gang Su[1,4], Stephen J. Pennycook[1], Sokrates T. Pantelides[1,2,5] & Wu Zhou[1]

Correlation of lattice vibrational properties with local atomic configurations in materials is essential for elucidating functionalities that involve phonon transport in solids. Recent developments in vibrational spectroscopy in a scanning transmission electron microscope have enabled direct measurements of local phonon modes at defects and interfaces by combining high spatial and energy resolution. However, pushing the ultimate limit of vibrational spectroscopy in a scanning transmission electron microscope to reveal the impact of chemical bonding on local phonon modes requires extreme sensitivity of the experiment at the chemical-bond level. Here we demonstrate that, with improved instrument stability and sensitivity, the specific vibrational signals of the same substitutional impurity and the neighbouring carbon atoms in monolayer graphene with different chemical-bonding configurations are clearly resolved, complementary with density functional theory calculations. The present work opens the door to the direct observation of local phonon modes with chemical-bonding sensitivity, and provides more insights into the defect-induced physics in graphene.

Structural inhomogeneity at the nanoscale or even atomic scale is usually not a demerit of materials. Instead, it serves as a crucial parameter for tuning the macroscopic properties[1–3]. Examples include tuning the electronic properties of silicon by dopants[4] and the emergence of Majorana bound states in iron-based superconductors upon chemical doping[5]. Understanding the local atomic vibrations at such structural defects is essential for controlling the material properties that are affected by the scattering of charge carriers or phonons, such as thermal transport[1,6,7], thermoelectric properties[8], superconductivity[9], phase transitions[10] and electron–phonon coupling[11]. The ability to probe the defect-induced local phonon modes is not only of fundamental importance in validating theoretical predictions, but also of practical importance, as the impact of defects on phonon-mediated properties becomes increasingly prominent in reduced-scale samples and devices[12]. However, the experimental measurement of local phonon modes at the atomic scale and correlation with the defect atomic configuration has long been a grand challenge because of the simultaneous requirement of millielectronvolt spectral resolution and atomic spatial resolution[13–15].

Recent advances in instrumentation have enabled the combination of sub-10 meV energy resolution in monochromated electron energy-loss spectroscopy (EELS) with atomic-scale spatial resolution and flexible electron optics in aberration-corrected scanning transmission electron microscopy[16,17] for nanoscale phonon measurements of bulk materials[18–27] and for probing the localized phonon modes at defects or interfaces[28–34]. Most notably, using inelastically

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Atomically Dispersed Ni/α-MoC Catalyst for Hydrogen Production from Methanol/Water

Lili Lin, Qiaolin Yu, Mi Peng, Aowen Li, Siyu Yao, Shuheng Tian, Xi Liu, Ang Li, Zheng Jiang, Rui Gao,* Xiaodong Han, Yong-wang Li, Xiao-dong Wen, Wu Zhou,* and Ding Ma*

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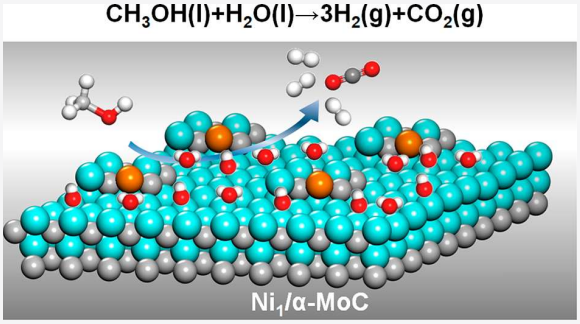
ABSTRACT: Methanol−water reforming is a promising solution for H_2 production/transportation in stationary and mobile hydrogen applications. Developing inexpensive catalysts with sufficiently high activity, selectivity, and stability remains challenging. In this paper, nickel-supported over face-centered cubic (fcc) phase α-MoC has been discovered to exhibit extraordinary hydrogen production activity in the aqueous-phase methanol reforming reaction. Under optimized condition, the hydrogen production rate of 2% Ni/α-MoC is about 6 times higher than that of conventional noble metal 2% Pt/Al_2O_3 catalyst. We demonstrate that Ni is atomically dispersed over α-MoC via carbon bridge bonds, forming a Ni_1-C_x motif on the carbide surface. Such Ni_1-C_x motifs can effectively stabilize the isolated Ni_1 sites over the α-MoC substrate, rendering maximized active site density and high structural stability. In addition, the synergy between Ni_1-C_x motif and α-MoC produces an active interfacial structure for water dissociation, methanol activation, and successive reforming processes with compatible activity.



■ INTRODUCTION

Hydrogen transportation, distribution, and storage at different scales in an efficient and reliable way remains technically and economically challenging,[1−3] which hinders the extensive utilization of polymer electrolyte membrane fuel cells (PEMFCs) in stationary and mobile applications.[4,5] It has been proposed that hydrogen stored in a chemical form and released *in situ* on demand through efficient catalytic processes provides a promising solution for hydrogen storage and application.[6−8] Compared with other liquid organic (such as quinoline and carbazole, etc.) and inorganic (such as NH_3 and borane, etc.) hydrogen carriers, methanol, an important inexpensive bulk chemical, is able to react with water and release 3-fold H_2 ($CH_3OH + H_2O{=}CO_2 + 3H_2$) under relatively mild condition, achieving a high H_2 gravimetric density of 18.8% by weight. The well-balanced high H_2 storage capacity and mild H_2 release condition make methanol one of the most attractive liquid forms of H_2 carrier candidates.[9,10] Methanol reforming in the aqueous phase can remove the tedious vaporization units from the hydrogen production device, thus improving economic efficiency. It can also take advantage of intensified water gas shift reaction (WGSR) to effectively suppress the selectivity toward byproduct CO.[11−13]

The pioneer works have demonstrated that hydrogen can be generated through aqueous-phase methanol reforming over platinum- and ruthenium-based noble metal catalysts.[9,13,14] Recently, we proposed that a new noble metal catalyst, Pt/α-MoC, is highly active for this reaction at low temperatures. The efficient C−H and O−H activation ability and the highly effective interfacial water gas shift activity of Pt/α-MoC render to the catalyst an excellent hydrogen evolution rate of 18 000 h^{-1}.[10] Despite the excellent activity, the high cost and limited reserve of noble metals are still the big obstacles for large-scale applications. Therefore, developing efficient non-noble-metal catalytic systems capable of working in an aqueous phase with high reactivity is highly desirable but challenging.

Herein, a novel Ni/α-MoC catalyst is reported to effectively catalyze the methanol reforming reaction with high activity and selectivity in aqueous solution below 240 °C. Nickel is atomically dispersed over an α-MoC surface, creating isolated Ni sites anchored over α-MoC host via carbon bridge bonds, forming Ni_1-C_x motifs with ultrahigh site density (up to 2 wt % loading of Ni). The 2% Ni/α-MoC catalyst shows a high hydrogen evolution activity of 171 $\mu mol_{H2}/g_{cat}/s$ (ATOF = 1805 h^{-1}) at 240 °C, which is around 6 times higher than that of the conventional 2% Pt/Al_2O_3 and the most active non-noble-metal catalyst reported so far.[13] The high activity is

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Maximizing the Synergistic Effect of CoNi Catalyst on α-MoC for Robust Hydrogen Production

Yuzhen Ge,[⊥] Xuetao Qin,[⊥] Aowen Li,[⊥] Yuchen Deng, Lili Lin, Mengtao Zhang, Qiaolin Yu, Siwei Li, Mi Peng, Yao Xu, Xueyao Zhao, Mingquan Xu, Wu Zhou,* Siyu Yao,* and Ding Ma*

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ABSTRACT: We report the syntheses of highly dispersed CoNi bimetallic catalysts on the surface of α-MoC based on the strong metal support interaction (SMSI) effect. The interaction between the nearly atomically dispersed Co and Ni atoms was observed for the first time by the real-space chemical mapping at the atomic level. Combined with the ability of α-MoC to split water at low temperatures, the as-synthesized CoNi/α-MoC catalysts exhibited robust and synergistic performance for the hydrogen production from hydrolysis of ammonia borane. The metal-normalized activity of the bimetallic 1.5Co1.5Ni/α-MoC catalyst reached 321.1 $mol_{H2}\cdot mol^{-1}{}_{CoNi}\cdot min^{-1}$ at 298 K, which surpasses all the noble metal-free catalysts ever reported and is four times higher than that of the commercial Pt/C catalyst.

The recent developments in the atomically dispersed metal catalysts or single-atom catalysts (SACs) have attracted considerable interest because of their maximized noble metal utilization efficiency and unique electronic and/or geometric characteristics.[1−4] However, SACs also have their own limitations. For example, when the activation of reacting molecules requires more than one metallic center, SACs generally exhibit limited activities because of the lack of suitable multicenter active sites. Lu et al. showed that the Pt dimers possess much higher activity for the hydrolysis of ammonia-borane ($NH_3\cdot BH_3$, AB) than that of the Pt SACs.[5] Therefore, for certain reactions such as the dehydrogenation of molecules that require diverse adsorption sites, the closely distributed under-coordinated metallic centers are more favorable for enhancing the catalytic performance.[6,7] The heterometal combinations in the highly dispersed metal domains may also result in a special synergistic effect that the SACs generally lacks, although this synergy has rarely been reported.

In addition to the geometric factors, modulation of the electronic properties of active centers that is induced by their interaction with the support materials has also been proven essential in determining the catalytic performance, especially for the catalysts with high or even atomic metal dispersion.[3,8] In our recent studies, we have demonstrated that both the geometric and electronic properties of Au and Pt can be tuned by the strong metal support interaction with α-MoC.[9] After combining the unique nature of α-MoC that is capable of activating water and maintaining a high surface coverage of hydroxyl species at a temperature of 303 K,[10,11] the as-synthesized catalysts showed extraordinary activity for the low-temperature water−gas shift and methanol reforming reactions. These unique properties of the α-MoC evoke us to explore the utility of its surface hydroxyl species in other catalytic reactions.

In this paper, we report the syntheses of highly dispersed CoNi bimetallic catalysts supported on α-MoC (CoNi/α-MoC) for the efficient hydrogen production from the hydrolysis of ammonia borane.[12−18] The metal-normalized activity of 1.5Co1.5Ni/α-MoC (α-MoC loaded with 1.5 wt % Co and 1.5 wt % Ni) reached 128.5 $mol_{H2}\cdot mol^{-1}{}_{CoNi}\cdot min^{-1}$ at 25 °C which is 3.2 and 3.6 times higher than that of the monometallic 3Co/α-MoC and 3Ni/α-MoC catalysts, respectively. The improved catalytic performance of 1.5Co1.5Ni/α-MoC is due to the synergistic effect between the nearly atomically dispersed Co and Ni atoms. Furthermore, by changing the reaction media from neutral to basic conditions, the optimum metal-normalized activity even reached 321.1 $mol_{H2}\cdot mol^{-1}{}_{CoNi}\cdot min^{-1}$, surpassing all the non-noble metal catalysts ever reported,[19−25] and achieving over four times higher activity than the commercial Pt/C catalyst.[26]

The α-MoC support was synthesized by following the stepwise temperature ammonization/carburization of MoO_3 at 973 K.[27] Further, the α-MoC supported Co/Ni catalysts were synthesized by the impregnation method. The structure of the catalysts was first investigated using X-ray diffraction (XRD) (Figure 1a). No obvious crystal diffraction peaks of Co and Ni were observed in any of the samples, demonstrating the high dispersion of Co and Ni on the surface of α-MoC.

The electronic structures of the α-MoC supported Co/Ni catalysts were studied by *ex situ* X-ray photoelectron spectroscopy (Figure 1b and 1c). The binding energy of 283.3 eV for C 1s (Figure S1) and 228.4 eV for Mo $3d_{5/2}$

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ARTICLE

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Electrochemical CO_2 reduction to ethylene by ultrathin CuO nanoplate arrays

Wei Liu[1], Pengbo Zhai[2], Aowen Li[3,4], Bo Wei[1], Kunpeng Si[1], Yi Wei[5,6], Xingguo Wang[1], Guangda Zhu[7], Qian Chen[1], Xiaokang Gu[1], Ruifeng Zhang[1], Wu Zhou[3,4,8] & Yongji Gong[1,9]✉

Electrochemical reduction of CO_2 to multi-carbon fuels and chemical feedstocks is an appealing approach to mitigate excessive CO_2 emissions. However, the reported catalysts always show either a low Faradaic efficiency of the C_{2+} product or poor long-term stability. Herein, we report a facile and scalable anodic corrosion method to synthesize oxygen-rich ultrathin CuO nanoplate arrays, which form Cu/Cu_2O heterogeneous interfaces through self-evolution during electrocatalysis. The catalyst exhibits a high C_2H_4 Faradaic efficiency of 84.5%, stable electrolysis for ~55 h in a flow cell using a neutral KCl electrolyte, and a full-cell ethylene energy efficiency of 27.6% at 200 mA cm^{-2} in a membrane electrode assembly electrolyzer. Mechanism analyses reveal that the stable nanostructures, stable Cu/Cu_2O interfaces, and enhanced adsorption of the *OCCOH intermediate preserve selective and prolonged C_2H_4 production. The robust and scalable produced catalyst coupled with mild electrolytic conditions facilitates the practical application of electrochemical CO_2 reduction.

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ARTICLES

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Synergizing metal–support interactions and spatial confinement boosts dynamics of atomic nickel for hydrogenations

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Atomically dispersed metal catalysts maximize atom efficiency and display unique catalytic properties compared with regular metal nanoparticles. However, achieving high reactivity while preserving high stability at appreciable loadings remains challenging. Here we solve the challenge by synergizing metal–support interactions and spatial confinement, which enables the fabrication of highly loaded atomic nickel (3.1 wt%) along with dense atomic copper grippers (8.1 wt%) on a graphitic carbon nitride support. For the semi-hydrogenation of acetylene in excess ethylene, the fabricated catalyst shows extraordinary catalytic performance in terms of activity, selectivity and stability—far superior to supported atomic nickel alone in the absence of a synergizing effect. Comprehensive characterization and theoretical calculations reveal that the active nickel site confined in two stable hydroxylated copper grippers dynamically changes by breaking the interfacial nickel–support bonds on reactant adsorption and making these bonds on product desorption. Such a dynamic effect confers high catalytic performance, providing an avenue to rationally design efficient, stable and highly loaded, yet atomically dispersed, catalysts.

Supported atomically dispersed catalysts (SADCs) have recently drawn great attention in numerous reactions for their maximized atom efficiency[1,2]. Exclusive site homogeneity, distinct energetics and spatial site confinement make SADCs very attractive in high selectivity and alleviated coke formation in the conversion of hydrocarbons[3–5], sharply distinct from the low selectivity and heavy coke formation on metal nanoparticle (NP) catalysts[6]. However, owing to drastically increased surface free energies, the stability of atomically dispersed metal species under reaction conditions, especially at high metal loadings, has been the key issue to substantially limit the overall performance, restraining their practical applications[7].

Realization of a highly loaded and stable SADC for hydrogenation reactions is particularly challenging, because atomically dispersed metal species often show higher mobility in reducing environments[8]. For instance, in the semi-hydrogenation of acetylene in excess ethylene, a crucial industrial process operated at a scale of billions of tons per annum to purify alkene streams[9,10], severe metal aggregations occurred even for low-loaded SADCs, which sequentially generated heavy coke formation—an even worse scenario to further accelerate catalyst deactivation to a shorter lifetime[11]. Increasing metal–support interactions (MSIs)[12,13] or confining active metal species in restrained spaces (for example, micropores)[14,15] are two widely adopted methodologies to achieve higher stability. However, excessively strong MSIs might over-stabilize the active sites and cause low activity[13,16]; confinement of metal species in micropores might also limit mass transfers[14,17]. Consequently, a seesaw relation between stability and activity has been often observed, obscuring efforts for catalytic performance optimizations[18–20].

In this work, we report that the challenge can be solved by synergizing MSIs and spatial confinement to break the activity–stability seesaw of SADCs at high metal loadings. Specifically, strong Ni–N/C interaction and spatial confinement by dense atomic Cu grippers (8.1 wt%) on a graphitic carbon nitride (g-C_3N_4) support are synergized to endow great structural flexibility of highly loaded (3.1 wt%) atomic Ni with reversible bond breakings and makings on reactant adsorption and product desorption, respectively. Such a dynamic effect not only facilitates acetylene adsorption for high activity but also ensures high thermal/chemical stability and unprecedented inhibition of coke formation.

Results

Synthesis and catalytic performance. As it contains periodic triangular cavities with six coordinately unsaturated pyridinic nitrogen (N_{py}) atoms, g-C_3N_4 provides a perfect platform for anchoring active and dense metal species[21,22]. Screening of metals among groups VIII and IB by density functional theory (DFT) calculations revealed that Ni and Cu atoms exhibit strong binding to g-C_3N_4 underneath

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ORIGINAL PAPER

Electron Energy Loss Spectroscopy for Single Atom Catalysis

Zirui Gao[1] · Aowen Li[2] · Ding Ma[1] · Wu Zhou[2,3]

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Abstract
Single atom catalysts (SACs) have attracted intense interest because of their ultimate metal utilization efficiency and outstanding catalytic performance. Unveiling the structural and chemistry characteristics of SACs at the single-atom level is crucial for understanding the structure–property relationship in SACs and the design of novel SACs for specific target reactions. Aberration corrected (scanning) transmission electron microscopy ((S)TEM) equipped with various advanced imaging detectors and spectrometers is regarded as a versatile platform for structural analysis of heterogeneous catalysts. In particular, electron energy-loss spectroscopy (EELS) on aberration-corrected STEM can offer chemical imaging at the atomic scale, taking advantage of the energy-loss occurring when the incident electrons interact inelastically with samples. In addition, a wide range of chemical and physical properties of SACs, such as valence state, bonding and coordination environment, as well as vibrations of absorbed molecules, can be readily revealed by STEM-EELS. In this review article, we use some recent representative studies of SACs to demonstrate the indispensable role and the general applicability of STEM-EELS. Furthermore, we share our insights on the future development of atomic scale EELS analysis, including the improvement of microscope and spectrometer hardware and signal denoising algorithm, as well as the combination with *in-situ* techniques.

Keywords Heterogeneous catalysis · Single atom catalysis · Electron microscopy · Electron energy loss spectroscopy

1 Introduction

Single atom catalysts (SACs) have become a fast-growing field in heterogenous catalysis [1, 2]. In an idealized picture, SACs contain exclusively isolated metal atoms anchored on the support surface, which, combining with their surrounding atoms, act as well-defined catalytic active sites and could serve as ideal model systems to probe the catalytic mechanisms for various chemical reactions [3]. The single-atom dispersion provides the ultimate metal utilization efficiency and is typically resulted from the strong interaction with supports, which also helps to tune the electronic structure of the metal sites and consequently enhance the intrinsic catalytic activity for many reactions. Because of these major benefits, SACs have attracted tremendous research interests, and have been widely considered as one of the most promising solutions for catalyst design [4].

The development of SACs has largely benefited from the availability of structural characterization tools with single-atom or single-site sensitivity. For example, infrared spectroscopy (IR) coupled with probe molecules (usually CO) adsorption [5, 6] has been widely applied to demonstrate the formation of single type of metal sites in catalysts; extended X-ray absorption fine structure (EXAFS) analysis is often used to demonstrate the atomic dispersion of metal species when the fitted metal–metal coordination vanishes [1]. More noticeably, the atomic number (Z) contrast images via high angle annular dark-field (HAADF) imaging on an aberration-corrected scanning transmission electron microscope (STEM) can provide direct real-spacing visualization of

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Single-atom electron microscopy for energy-related nanomaterials

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The development of the aberration correction technique has enabled atomic resolution imaging in scanning transmission electron microscopy (STEM) under low accelerating voltages and pushed the detection limit of STEM-based imaging and spectroscopy analysis down to the single-atom level. The so-called "single-atom electron microscopy" brings new opportunities to study the structure–property relationship in energy-related nanomaterials at the atomic scale. In this review, we will firstly summarize some recent advances in low-voltage STEM imaging and spectroscopy with single-atom sensitivity, including emerging techniques such as STEM-DPC imaging and *in situ* STEM, by using two-dimensional (2D) materials as model systems. We will then discuss the application of these single-atom electron microscopy techniques to more complex energy materials *via* specific examples. These powerful and comprehensive capabilities of aberration-corrected STEM with single-atom sensitivity have proven to be invaluable for unveiling the origins of functionalities of energy-related nanomaterials and guiding the design of novel materials with desired performance.

1. Introduction

Early in his famous 1959 lecture "There's plenty of room at the bottom", Richard Feynman proposed the conception of nanotechnology and a fascinating future in fundamental research of chemistry if "all one would have to do would be to look at it and see where the atoms are".[1] Over the past half century, nanotechnology has been successfully utilized to functionalize energy-related materials in numerous fields (energy sustainability, environmental protection, *etc.*). When the scale of materials decreases to nano-dimensions, structural heterogeneity at the atomic scale, such as the individual supported atoms in single-atom catalysts (SACs)[2] and single impurity atoms in semiconductor devices,[3] significantly tailors the functionalities of nanomaterials. Such structural features, however, exceed the detection limit of most conventional imaging/spectroscopic methods, which usually provide average structural information. Under such a background, aberration-corrected scanning transmission electron microscopy (STEM), which can constrict the detection volume by employing an atomic-sized electron probe,[4–9] has been under rapid

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STEM High Angle Annular Dark-Field Imaging

20

Qian He, Aowen Li, Bingqing Yao, Wu Zhou, and Christopher J. Kiely

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Abstract

High-angle annular dark field (HAADF) imaging in the aberration-corrected scanning transmission electron microscope (STEM) has become an invaluable technique for characterizing catalysts. Its powerful combination of good atomic number (Z) contrast and ultrahigh spatial resolution allows us to capture atomic resolution images whose intensity variations include important compositional information on the catalyst being examined. The physical principles and experimental limitations of the STEM-HAADF imaging technique are presented in this chapter, followed by some representative examples of applications to supported metals, supported oxides, and bulk mixed oxide catalyst systems. The challenges and progress in developing a "gentler" more benign form of STEM-HAADF imaging for analyzing highly beam-sensitive materials such as two dimensional (2D) materials and zeolites are then described. We also explain how three dimensional (3D) depth information from the catalyst can be extracted by quantifying STEM-HAADF image intensities, by performing electron tomography or through-focal imaging in HAADF mode. We will also introduce some applications of HAADF imaging of catalysts in a gaseous or liquid environment in order to understand their structural evolution under more realistic conditions. Finally, we stress the benefits of correlating HAADF imaging results with information obtained from complementary characterization methods.

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Case Studies: Aberration Corrected High-Angle Annular Dark-Field (AC-HAADF) Microscopy

21

Aowen Li, Mingquan Xu, Ding Ma, and Wu Zhou

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Abstract

As demonstrated in the previous chapter, scanning transmission electron microscopy-high-angle annular dark field (STEM-HAADF) imaging is widely applied to reveal the structural features of catalyst specimens at the atomic scale. This chapter will continue the topic of STEM-HAADF imaging by focusing on the application in α-MoC-supported metal catalysts, categorizing the catalysts by their supported metals. Owing to the robust atomic number (Z)-dependent contrast in STEM-HAADF imaging, the structure and distribution of the heavy noble metal species supported on the α-MoC support can be directly imaged. The collaboration of atomic-scale STEM-HAADF imaging with X-ray spectroscopies and theoretical calculations provided invaluable insights into the origins of the outstanding catalytic performance of these catalysts. In cases of α-MoC-supported light transition metal catalysts where STEM-HAADF imaging cannot provide direct visualization of the surface metal species, we show that the spectroscopy methods available in STEM are good supplements for imaging. It is noteworthy that the above discussion is based on results obtained from ex situ STEM experiments. It is necessary to extend the studies by employing in situ (S)TEM. Additionally, we point out that the newly emerging 4D-STEM techniques are promising to make a breakthrough in revealing the electronic structure at the level of individual catalytically active sites.

Keywords

Scanning transmission electron microscopy (STEM) · High-angle annular dark field (HAADF) imaging · Atomic number (Z)-dependent contrast · Molybdenum carbide-supported metal catalyst · Single atom catalyst (SAC)

Heterogeneous catalysis has been widely used in the chemical industry because of its excellent stability and easy separation process. Supported noble metal catalysts are one of the best examples in heterogeneous catalysis as they show outstanding performance in many important catalytic reactions, although their extensive use in practice is limited by their high cost. Thus, downsizing the supported metal particles is an effective strategy to reduce the cost and to tune the catalytic activity, with the maximum atom efficiency when achieving full dispersion of isolated atoms [1, 2]. As a new frontier of heterogeneous catalyst research [3, 4], single atom catalysts (SACs) consisting of solely individual metal atoms dispersed on substrates have been considered as an ideal model for exploring the underlying mechanisms of the catalytic reactions due to their structural simplicity.

As illustrated in the previous chapter, the recent development of aberration-corrected scanning transmission electron

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