# **Novel** *In Situ* **Probes for Nanocatalysis**

Andreas Stierle and Alfons M. Molenbroek, Guest Editors

## Abstract

During the past few years, substantial effort has been devoted to developing new experimental techniques capable of delivering atomic-scale information on surfaces and nanoparticles under catalytic reaction conditions. Since the advent of surface science, pioneering experiments under highly idealized conditions have been performed (at very low gas pressures, <10-6 mbar), and idealized model material systems (e.g., single crystals) have been investigated. However, understanding chemical reactions on single-crystal surfaces close to ultrahigh vacuum does not always enable prediction of the performance of nanoparticles operating at gas pressures near or above atmospheric pressure. Therefore, this *MRS Bulletin* issue focuses on the capabilities of atomic-scale-resolution, high-gas-pressure- and high-temperature-compatible *in situ* probes sensitive to the structure, chemical composition, and dynamical properties of nanomaterials. It will be demonstrated how novel *in situ* techniques enable one to bridge the combined pressure and materials gaps from ultrahigh vacuum to atmospheric pressures and from metal single-crystal surfaces to nanoparticles or oxides.

### Introduction

The investigation of the atomic structure and reactivity of nanomaterials under operational conditions is of utmost importance for the enhanced performance of catalysts involved in applications ranging from fuel cells and chemical production to electronic sensors for automotive and environmental monitoring applications.1 A heterogeneous catalyst is typically composed of active metal nanoparticles supported by a metal oxide substrate, such as that shown in the upper-right inset of Figure 1, although sulfide or oxide active phases are also quite common. The activity of the catalyst to promote chemical reactions and its selectivity toward the desired reaction depend strongly on the microscopic details of these composed metal oxide systems. These microscopic details include the shape of the metal nanoparticles and adsorption behavior on different facets or defect sites, adhesion to the oxide substrate, and the variation of electronic properties because of the reduced dimensionality of the particles. Also, unwanted deactivation processes of the catalyst, such as sintering of the nanoparticles, are strongly influenced by these parameters. In addition, chemical reactions under ambient conditions, such as oxidation, determine the stability, functionality, and long-term performance of metallic nanoparticles (e.g., nanomagnets for magnetic storage devices) in their working environment.

Substantial effort has been devoted during the past few years to developing new experimental techniques capable of delivering atomic-scale information on surfaces and nanoparticles in situ under catalytic, industrially relevant reaction conditions. The development of such techniques is crucial, if one desires to tailor chemical reactions on nanoparticles for industrial applications, particularly catalytic reactions, and to increase the nanoparticle lifetime. In addition, these new techniques allow a direct, critical comparison with results from ab initio density functional theory (a novel approach that connects density functional theory results obtained at T = 0 K, p = 0 bar to applied conditions at elevated temperatures and pressures).<sup>2</sup> Results of kinetic Monte Carlo simulations, which simulate kinetic steady-state equilibrium situations during catalytic reactions, can also be compared directly.3

# The Pressure and Materials Gaps

Since the advent of surface science in the 1960s, pioneering experiments under

highly idealized conditions have been performed at very low gas pressures (<10-6 mbar), because many standard ultrahighvacuum (UHV) surface analysis methods are electron- and ion-beam-based tools that interact strongly with gas atmospheres at elevated pressures. In addition, fast degradation of classical UHV equipment due to interaction of oxygen with the hot filaments takes place at elevated pressures and temperatures. However, understanding chemical reactions on single-crystal surfaces close to ultrahigh vacuum does not necessarily enable prediction of the performance of a catalyst made of nanoparticles operating at near-atmospheric gas pressures or higher. For some systems, successful experiments have been performed at low temperatures (100-200 K) and low, close to UHV pressures, which is thermodynamically equivalent to working at high temperatures and near-atmospheric pressures because of the equivalent oxygen chemical potential,<sup>2</sup> neglecting entropic energy contributions. This approach is valid only if no kinetic barriers exist at lower temperatures for the processes involved, such as dissociative gas adsorption and metal atom diffusion, both taking place during the adsorbate-induced surface structure formation process. For selected model systems, results obtained at low temperatures and low pressures can be extrapolated to high temperatures and high pressures, thereby bridging the socalled "pressure gap."4-6 In general, kinetic barriers are so huge at low temperatures and low pressures that the results obtained cannot be extrapolated to "real-world" conditions. To bridge the pressure gap (see Figure 1), novel techniques have been developed in the past few years that allow new, atomic-scale insight into reaction processes on surfaces under applicationrelevant conditions.

Finally, in the low-p/T experiments, idealized model systems have been investigated, such as single-crystal surfaces, that exhibit properties similar to those of facets of regularly shaped nanoparticles. The difference between the reactivities of nanoparticles and single-crystal surfaces is manifested by the presence of undercoordinated atoms, having fewer nearest neighbors than in the bulk. They are located at the intersections of the different facets and close to the oxide support. This "materials gap" has been overcome partially by the investigation of metal nanoparticles deposited on ultrathin epitaxial oxide films on metallic substrates.7-9 These systems can be investigated by molecular beams at low gas pressures in titration-type experiments, during which a pre-oxidized surface is exposed to CO

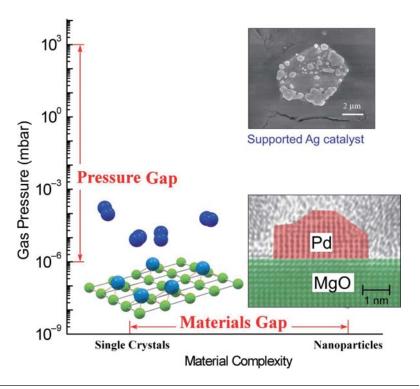


Figure 1. Pressure and materials gaps for chemical reactions: low-pressure studies on single crystals suffer from a lack of applicability to "real" systems. Novel experimental techniques, such as those discussed in this issue, bridge the enormous pressure gap of more than 9 orders of magnitude and the materials gap between single crystals and supported (oxide) nanoparticle systems, thus enabling atomically resolved experiments under application relevant conditions. The inset at upper right shows a typical, heterogeneous catalyst composed of active metal nanoparticles supported by a metal oxide substrate. The inset at lower right shows an artificial-color TEM image of a Pd nanoparticle on MgO.

gas pulses, and the resulting CO<sub>2</sub> response is detected by a mass spectrometer (10-6 mbar regime).<sup>10</sup> Catalytic oxidation reactions at elevated pressures cannot be studied using these systems, because the ultrathin epitaxial oxide films are expected to be unstable under highpressure conditions. By deposition of metal nanoparticles onto single-crystal oxide supports or oxide nanoparticles, model systems can be prepared that can be investigated at near-atmospheric or higher pressures using in situ techniques presented in this MRS Bulletin issue, thereby bridging both the pressure and materials gaps. 11,12 With respect to the materials gap, there is almost a continuous range of systems with increasing complexity starting from low-index single-crystal surfaces, via vicinal surfaces with geometrically well-defined regular steps and nanoparticles deposited on single crystals, to industrial catalysts consisting of catalytically active metallic or oxide nanoparticles on a high-surface-area metal oxide support. The use of model systems presents the clear advantages of controlled metal–support interactions, the absence of heat- and mass-transfer limitations, and a much higher control of surface chemistry and structure.

# Novel High-Resolution *In Situ* Probes

To overcome the combined pressure and materials gap and to reveal atomicscale information of the underlying microscopic processes and chemical reactions under applied conditions, novel experimental techniques have been developed.

Various properties need to be characterized under reaction conditions:

- structure and shape of the nanoparticle
- structure of the oxide support
- metal/support interface
- local surface morphology
- chemical state of the active surface under reaction conditions
- adsorption sites of reacting gases and gas composition under reaction conditions.

To obtain these parameters, a combination of different techniques is used. Table I

summarizes the characteristic parameters of the different *in situ* techniques. Figure 2 illustrates the use of these techniques for studies on single crystals and supported nanoparticles.

In Figure 2a, two atom molecules (such as CO, represented by blue and yellow circles) are adsorbed on a metal singlecrystal surface. X-ray photoelectron spectroscopy (XPS) is performed by using incident photons in the energy range hv from 100 eV to 1000 eV to probe the chemical state of the surface atoms; the outgoing photoelectrons are detected by an electron energy analyzer. Raster probe techniques such as atomic force microscopy and scanning tunneling microscopy (AFM/STM) can be applied to study the local surface structure and morphology. Using hard x-rays (photon energy > 10 keV), diffraction experiments can be performed, providing information on the crystallographic structure of the surface. Using low-energy photons (hv in the range of eV), internal excitations of the molecules can be probed (vibrations, rotations). The outgoing photons are detected with a different photon energy (hv' in this case). Mass spectrometry (MS) is used to analyze the residual gas composition during the reactions. In the case of oxide supported metal nanoparticles (represented by red half-spheres in Figure 2b), additional information about the nanoparticle shape can be obtained by grazingincidence small-angle x-ray scattering (GISAXS) using hard x-rays.31 The local structure and composition of the nanoparticles can be analyzed by transmission electron microscopy (TEM) and surface x-ray diffraction (SXRD). For the TEM analysis, specially prepared specimens are required that allow transmission of the high-energy electrons (>100 keV). The local chemical environment of the metal atoms inside the nanoparticles can be investigated by extended x-ray absorption fine structure (EXAFS) using hard x-rays in transmission or fluorescence mode.

Photon-in/photon-out techniques are applicable up to the bar pressure regime and higher; electron-out techniques are limited to an upper pressure of 20–30 mbar (the current state-of-the-art limit, to the best of the authors' knowledge), because of the necessary differential pumping of the electron detection system and the mean free path of the electrons.

# In This Issue

Atomic-scale structural information can be obtained using surface x-ray diffraction (SXRD), which can be applied both to single crystals and epitaxial nanoparticles on oxide supports<sup>12–14</sup> over a pressure range

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Technique	Abbreviation	Pressure Range	Temperature Range	Absolute/ Relative Spatial Resolution	Energy Resolution/ Chemical Sensitivity	Time Resolution	Principle Information	Sample Restrictions
Atomic force microscopy and scanning tunneling microscopy	AFM/STM	<1.5 bar	<450 K	0.01 nm	limited	0.05 s	surface structure and morphology	conducting, single crystal
(Surface) x-ray diffraction	(S)XRD	no limitation	no limitation	0.001 nm	a few eV anomalous scattering	seconds to nanoseconds in pump-probe experiments	crystallographic structure and morphology	(single crystal) none
Extended x-ray absorption fine structure	EXAFS	no limitation	no limitation	0.003 nm	0.1 eV yes	milliseconds	local structure, chemical environment	none
Vibrational sum frequency generation spectroscopy	VSFG	<1 bar	<500 K	<u>:</u>	5 cm <sup>-1</sup> yes	seconds to picoseconds in pump-probe experiments	vibrational state of adsorbates	none
(Scanning) transmission electron microscopy	(S)TEM	<50 mbar	<1400 K	0.1 nm	3 eV yes	seconds	local structure, morphology, and chemical composition	none, sample preparation
X-ray photoelectron spectroscopy	XPS	<13 mbar	<900 K	<u>:</u>	0.05–0.3 eV yes	seconds	chemical bonding state	conducting material
Mass spectrometry	MS	no limitation	no limitation	÷	atomic mass selection	0.1 s	gas composition	none
Raman spectroscopy	÷	no limitation	no limitation	÷	0.02 cm <sup>-1</sup> yes	seconds to picoseconds in pump-probe experiments	vibrational state of adsorbates	none
(Grazing-incidence) small-angle x-ray scattering	(GI)SAXS	no limitation	no limitation	1–100 nm	a few eV anomalous scattering	seconds to ns in pump-probe experiments	particle size, shape, porosity	optically flat surface
Scanning electron microscopy	SEM	<1 bar	300 K	1 nm to 1 mm	limited	0.05 s	surface morphology and chemical composition	conducting material

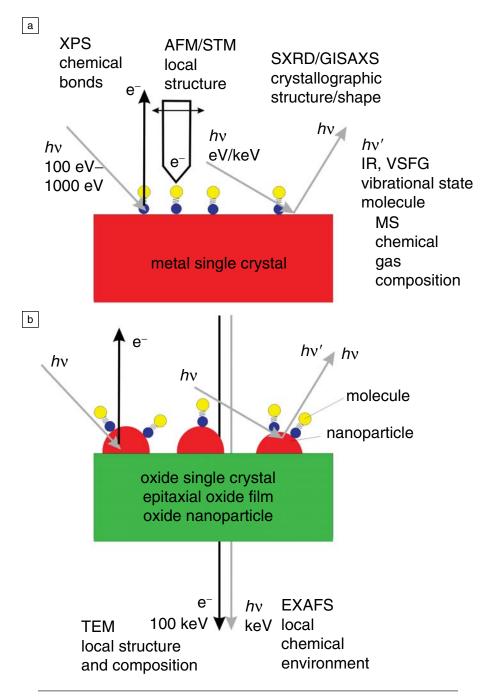


Figure 2. Novel *in situ*, pressure- and materials-gap-bridging techniques for studies on (a) single crystals and (b) supported nanoparticles. See text for discussion.

from UHV to several bar (limited by the currently available equipment) and a temperature range from low temperatures to >1600 K. *In situ* SXRD is covered in the article by Ferrer et al. in this issue.

Complementary real-space information on the surface periodicity and morphology can be obtained by *in situ* STM on metal single crystals, which is operative

up to p = 1.5 bar and T = 470 K.<sup>15–18</sup> In situ STM is the topic of the article by Frenken and Hendriksen in this issue.

XPS using a differentially pumped spectrometer<sup>19,20</sup> allows binding-energy studies of the adsorbed gas species and the metal near-surface atoms up to 10 mbar and 900 K. Bluhm et al. discuss *in situ* XPS in their article in this issue.

Using vibrational spectroscopy, namely, vibrational sum frequency generation (VSFG) spectroscopy (see the article by Rupprechter in this issue), the adsorption-site-dependent energy shift of the vibrational modes of the adsorbed gas species can be characterized.<sup>21–24</sup>

Information on the local structure and chemical environment of the nanoparticles can be obtained by EXAFS and nearedge x-ray absorption fine structure (NEXAFS) measurements, which can be performed under nearly true industrial conditions up to several hundred bar and temperatures well above 1300 K<sup>25,26</sup> (see the article by Evans et al. in this issue). Today's state-of-the art EXAFS data analysis allows one to obtain precise atomic-scale information on the particle nanostructure for the absorbing element of interest.<sup>27</sup>

Finally, a differentially pumped transmission electron microscope has been developed that operates at pressures up to 20 mbar and temperatures up to 900 K.<sup>28–30</sup> This instrument can be used to image the shape and shape changes of supported nanoparticles under reaction conditions. In combination with spatially resolved electron energy-loss spectroscopy (EELS), the composition and oxidation state of the nanoparticles and the support can be studied (see the article by Gai et al. in this issue).

The different *in situ* techniques provide time resolutions ranging from milliseconds (energy-dispersive EXAFS) to seconds (XPS). All techniques are accompanied by *in situ* mass spectrometry inside the reaction chamber to monitor the chemical reactions and to enable correlations of changes in the reaction rates with structural, morphological, or chemical states of the catalyst surface.

# **Conclusions**

Using a combination of different in situ high-pressure- and high-temperaturecompatible techniques, new atomistic insight into chemical reactions on nanoparticle systems under applicationand industry-relevant conditions can be obtained. Both the pressure gap from ultrahigh-vacuum to atmospheric pressures and the materials gap from metal single crystals to supported (oxide) nanoparticles can be overcome by novel in situ techniques sensitive to the structure, morphology, and chemical composition of nanomaterials under reaction conditions. The results from experiments performed with such novel in situ techniques are expected to have a strong impact on the design of novel catalysts and ambient-resistant nanomaterials. In addition, they allow a direct, critical comparison with results from the above-mentioned theoretical investigations.

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Andreas Stierle



Alfons M. Molenbroek

Andreas Stierle, Guest Editor for this issue of MRS Bulletin, is a senior scientist at the Max Planck Institute for Metals Research in Stuttgart, Germany. After he obtained his PhD degree in physics from the Ruhr-Universität Bochum, Stierle joined the European Synchrotron Radiation Facility as a postdoctoral fellow.

Currently, he leads a research group working on nanoscale oxidation and corrosion, and he is the operation manager of the Max Planck beam-

line for *in situ* synchrotron studies at the Angstromquelle Karlsruhe. Stierle also teaches as a lecturer at the University of Stuttgart. He received the first Günter Petzow Award in 2006 for his work in the field of nanocorrosion and oxidation of alloys and noble metals.

Stierle can be reached at Max Planck Institute for Metals Research, Heisenbergstr. 3, D-70569 Stuttgart, Germany; tel. +49 711 689 1842; e-mail stierle@mf.mpg.de. Alfons M. Molenbroek, Guest Editor for this issue of MRS Bulletin, is manager of the Characterization Department at Haldor Topsøe A/S in Lyngby, Denmark.

His main research interests are in heterogeneous catalysis and characterization under *in situ* conditions. At Haldor Topsøe A/S, he is responsible for resources, safety, and characterization competences in the fields of microscopy, x-ray techniques, spectroscopy, rheology, thermal techniques, and high-performance computing.

Molenbroek performed his PhD work on surface melting at the FOM–Institute for Atomic and Molecular Physics (AMOLF) in Amsterdam and obtained his PhD degree in physics and mathematics from Leiden University in the Netherlands. He then joined the Physics Department at the Technical University of Denmark in Lyngby as an assistant research professor, working at the Center for Atomic-Scale Material Physics (CAMP) and as a consultant at Haldor Topsøe A/S. Molenbroek joined the staff of Haldor Topsøe A/S in 1997.

Molenbroek can be reached at Haldor Topsøe A/S, Characterization Department, R&D Division, Nymøllevej 55, DK-2800 Kgs. Lyngby, Denmark; tel. +45 4527 2483; e-mail am@topsoe.dk.

M.D. Ackermann is a PhD student assigned to both the group of professor J.W.M. Frenken at Leiden University, the Netherlands, and at the ID03 beamline of the European Synchrotron Radiation Facility in Grenoble, France.

His research is focused on the correlation between surface structure and reactivity in model catalysts under realistic reaction conditions, using both high-pressure surface x-ray diffraction and high-pressure scanning tunneling microscopy.

Ackermann received the ECOSS Prize in 2006 for his work on CO oxidation on Pd(001), performed with B.L.M. Hendriksen.

Ackermann can be reached at LION, Leiden Institute of Physics, Niels Bohrweg 2, 2333 CA Leiden, the Netherlands; tel. +31 71 527 5627; e-mail ackermann@ physics.leidenuniv.nl.

Hendrik Bluhm is a staff scientist in the Chemical Sciences Division at Lawrence Berkeley National Laboratory. He obtained his MSc degree in crystallography from the University of Leipzig, Germany, and his PhD degree in physics from the University of Hamburg.



M.D. Ackermann



Hendrik Bluhm



Edward D. Boyes



John Evans



S. Ferrer

Bluhm's current research interests include the study of water adsorption on environmentally relevant surfaces under ambient conditions, as well as the interaction of atmospheric trace gases with ice surfaces.

As a postdoctoral researcher in the group of Miquel Salmeron at LBNL, Bluhm participated in the development of synchrotronbased high-pressure x-ray photoelectron spectroscopy. He continued this work at the Fritz Haber Institute of the Max Planck Society in Berlin. In 2004, Bluhm received the Klaus Halbach Award for the development of innovative instrumentation at the Advanced Light Source

Bluhm can be reached at Lawrence Berkeley National Laboratory, Chemical Sciences Division, MS 6R2100, Berkeley, CA 94720, USA; tel. +1-510-486-5431; email hbluhm@lbl.gov.

Edward D. Boyes is a professor in the Departments of Physics and Electronics at the University of York and is co-director of the York JEOL Nanocentre. The laboratory's work is centered on aberration-corrected electron microscopy for dynamic

in situ experiments. His research interests are focused on novel instrumentation for application-driven analytical methods using various microscopy techniques and related chemical and crystallographic analyses.

Boyes received his PhD degree in materials science from Cambridge University and was a postdoctoral researcher in Cambridge (Cavendish Lab) and Oxford, which led to a faculty position in the Oxford Materials Department and a fellowship at Wolfson College. Before joining the University of York, he was a senior research fellow in the Central Research and Development Department of DuPont in Wilmington, Delaware.

As a member of the Technical Advisory Group (TAG) for the President's Council of Advisors on Science and Technology (PCAST), Boyes is involved in reviews of the National Nanotechnology Initiative.

Boyes can be reached at tel. +44 1904 328407 and by e-mail at eb520@york.ac.uk.

**John Evans** is head of the School of Chemistry at the University of Southampton. He received his BSc and PhD degrees in chemistry from Imperial College London and the University of Cambridge, respectively. Evans was a postdoctoral researcher at Princeton University and at Cambridge.

For five years, Evans was the science program advisor at the Diamond Light Source. His research interests are centered on structure–function relationships in transition-metal catalysts. He has received a Royal Society Pickering Research Fellowship and the Meldola and Tilden Medals of the Royal Society of Chemistry.

Evans can be reached at the University of Southampton, School of Chemistry, Southampton, S017 1BJ, UK; tel +44 23 8059 3307; e-mail je@soton.ac.uk.

**S. Ferrer** is the director of the Experiments Division of the Spanish Synchrotron project ALBA in Barcelona, which will be open to users in 2010. Ferrer received his physics degree at Barcelona University and his PhD degree at the Autonomous University of Madrid (UAM). Ferrer held a postdoctoral research position at the University of California, Berkeley. Afterward, he obtained a position as an



Stig Helveg

assistant professor at UAM.

In 1988, Ferrer was employed at the European Synchrotron Radiation Facility (ESRF) as a scientist in charge of the surface diffraction beamline. Ferrer stayed at ESRF until 2004, where he headed the Surface Science Group during his later years there. His research interests are focused on surface physics and chemistry, as well as on surface magnetism.

Ferrer can be reached at CELLS, Ap. 68, 08193 Bellaterra, Spain; tel. +34 93 592 43 06; e-mail ferrer@cells.es.

Joost Frenken is a full professor at the Kamerlingh Onnes Laboratory within Leiden University in the Netherlands. After his PhD degree research on ion scattering, performed at the FOM— Institute AMOLF in



Bas Hendriksen

Amsterdam (1986), he was a postdoctoral researcher at the University of Göttingen, Germany, and at IBM in Yorktown Heights, N.Y. Frenken then formed his research group, first at AMOLF and later at Leiden.

Recently, Frenken was appointed scientific director of the Dutch national consortium "Nano-Imaging under Industrial Conditions." Central to Frenken's research is exploring the dynamic aspects of surfaces. His interests include surface diffusion, crystal growth, surface phase transitions, model catalysis, nanotribology, and biomembranes. Frenken's research group has developed a variety of special-purpose scanning probe microscopes for tailor-made measurements in each of these areas.

Frenken has received the Nottingham Prize, an



Joost Frenken



Pratibha L. Gai



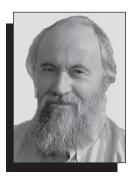
Suzanne Giorgio



Poul L. Hansen



Michael Hävecker



Claude Henry



Maya Kiskinova

NWO pioneer grant, the IUVSTA Prize in Science, and the Jacob Kistemaker Prize.

Frenken can be reached at the Kamerlingh Onnes Laboratory, Leiden University, PO Box 9504, 2300 RA Leiden, The Netherlands; tel. +31 71 527 5603; e-mail frenken@physics. leidenuniv.nl.

Pratibha L. Gai is JEOL Professor and Yorkshire Forward Chair of Electron Microscopy and Nanotechnology, a professor in the Department of Chemistry, a professor in the Department of Physics, and co-director of the York JEOL Nanocentre, all at the University of York. Gai graduated with a PhD degree in physics from Cavendish Laboratory, University of Cambridge. After postdoctoral work in the Inorganic Chemistry Department

at the University of Oxford, Gai became a group leader of the Surface Reactions and In Situ Microscopy Group at the Department of Materials, Oxford, and a fellow of Wolfson College. She also was a research fellow in the Central Research and Development Department of DuPont in Wilmington, Delaware, and jointly served as an adjunct professor of materials science at the University of Delaware.

Her research interests include nanomaterials, catalysts, semiconductors, superconductors, and in situ electron microscopy under controlled environments, including dynamic aberration-corrected electron microscopy. Gai is a fellow of the Institute of Physics, a chartered engineer of the Institute of Materials, and a recipient of awards for her

work on *in situ* electron microscopy of catalytic materials. She has published nine books and more than 200 scientific papers and holds numerous patents in catalysis and nanocoatings. Her patented co-invention of a nanocoating process for plastic materials has been commercialized by DuPont.

Gai can be reached by e-mail at pgb500@york. ac.uk.

Suzanne Giorgio is a professor in the ESIL Department of Materials at the University of the Mediterranean Aix-Marseille II in France.

Giorgio earned her habilitation from the University Aix-Marseille III in 1987 and was a postdoctoral researcher at the Fritz Haber Institute in Berlin in 1988. From 1983 through 2004, Giorgio was also a researcher at CNRS.

Giorgio can be reached at CRMCN-CNRS, Campus de Luminy, Case 913, 13288 Marseille Cedex 9, France; tel. +33 6 62 90 38 28; e-mail giorgio@crmcn.univmrs.fr.

Poul L. Hansen is project and department manager in the R&D Division at Haldor Topsøe A/S. He received his PhD degree in technical physics from the Technical University of Denmark in Lyngby in 1990. Hansen has a wide range of experience in catalyst characterization, with a special interest in advanced transmission electron microscopy techniques. His current work focuses on the research and development of hydrotreating catalysts for the oil refining industry.

Hansen can be reached at Haldor Topsøe A/S, Nymøllevej 55, DK-2800 Kgs. Lyngby, Denmark; tel. +45 4527 4598; e-mail plh@topsoe.dk.

Michael Hävecker is a postdoctoral researcher in the Department of Inorganic Chemistry at the Fritz Haber Institute (FHI) of the Max Planck Society in Berlin. He received his MSc degree in physics from the University of Hamburg and his PhD degree in physics from the Technical University of Berlin. During his studies at FHI, Hävecker participated in the development of a highpressure soft x-ray absorption apparatus for the characterization of catalysts; the technique of synchrotronbased high-pressure x-ray photoelectron spectroscopy; and the optical layout, construction, and operation of the FHI beamline for catalyst research (ISISS)

at the synchrotron source BESSY in Berlin.

Currently, his research interests focus on studying the dynamic response of catalyst surfaces in ambient conditions.

Hävecker can be reached at Fritz Haber Institute, Max Planck Society, Department of Inorganic Chemistry, Faradayweg 4–6, D-14195 Berlin, Germany; tel. +49 30 8413 4421 and e-mail mh@fhi-berlin.mpg.de.

Stig Helveg is a research scientist in the R&D Division at Haldor Topsøe A/S. He received his PhD degree in physics from the University of Aarhus, Denmark, in 2000 on model catalyst studies using STM. He joined Haldor Topsøe A/S in 2000 and is responsible for catalyst characterization by means of transmission electron microscopy techniques. His research interests focus on environmental transmission electron microscopy for in situ studies of the atomic-scale structure and reactivity of heterogeneous catalysts and related materials.

Helveg can be reached at Haldor Topsøe A/S, Nymøllevej 55, DK-2800 Kgs. Lyngby, Denmark; tel. +45 4527 2823; e-mail sth@topsoe.dk.

Bas Hendriksen is a postdoctoral fellow at Lawrence Berkeley National Laboratory. He earned his MS degree in applied physics in 1997 from the University of Twente, the Netherlands. In 2003, Hendriksen received his PhD degree in physics from Leiden University for high-pressure scanning tunneling microscopy studies of catalytic surfaces in the group of Joost Frenken. After earning his degree, Hendriksen continued his work in Frenken's group as a postdoctoral fellow. For his thesis, Hendriksen was awarded the Christiaan Huygens prize by the Royal Netherlands Academy of Arts and Sciences. His current research uses scanning probe microscopy to study the electronic and mechanical properties of molecular electronics.

Hendriksen can be reached at Lawrence Berkeley National Laboratory, Materials Sciences Division, 1 Cyclotron Road, MS 67R2206, Berkeley, CA 94720, USA; tel. 510-495-2375; e-mail blmhendriksen@lbl.gov.

Claude Henry is director of the Centre de Recherche en Matière Condensée et Nanosciences at the Centre National de la Recherche Scientifique (CRMCN-CNRS). He is also a research director at CNRS and the head of a "cluster group" at CNRS. His PhD studies in 1983 focused on the nucleation of supported clusters. Afterward, he was a postdoctoral researcher at IBM-Almaden from 1986 to 1987. Henry is a recognized expert on supported model catalysts. He is a member



Axel Knop-Gericke

of the editorial board of *Surface Science*, chair of four international conferences, and has authored 150 publications. Henry also received the Humboldt–Gay Lussac research award in 2001.

Henry can be reached at CRMCN-CNRS, Campus de Luminy, Case 913, 13288 Marseille Cedex 9, France; tel. +33 662 92 28 32; e-mail henry@crmcn.univ-mrs.fr.

Maya Kiskinova is head of the microscopy section at the Synchrotron Light Laboratory Elettra in Italy, where she has been a senior scientist since 1990.

She received her PhD degree in 1977, and her ScD degree in 1989, both from the Bulgarian Academy of Sciences in Sofia. She held research positions at the Academy from 1977 to 1989. She also was a visiting scientist at U.S. National Bureau of Standards in 1980, an Alexander von Humboldt fellow at Forschungszentrum Jülich from 1982 to 1983 and at the University of Pittsburgh from 1987 to

Her research interests are focused on the characterization of complex materials and processes at surfaces and interfaces using a variety of spectroscopic and microscopic methods. She was awarded with Italian



E. Lundgren

citizenship for scientific merit (2002) and is a recipient of the Alexander von Humboldt Distinguished Research Award (2004).

Kiskinova can be reached at Sincrotrone Trieste, 34012 Trieste, Italy; tel. +39 040 375 8549; e-mail kiskinova@ elettra.trieste.it.

Axel Knop-Gericke leads the surface analysis group of the Department of Inorganic Chemistry at the Fritz Haber Institute (FHI) in Berlin. He obtained his diploma in physics and his PhD degree from the Technical University of Berlin.

He was a postdoctoral researcher in the group of E. Rühl at the Free University of Berlin, and in the group of R. Schlögl at FHI. During this time, he developed new techniques for the investigation of free clusters by synchrotron radiation and high-pressure x-ray absorption spectroscopy in the soft energy range, respectively.

His current research focuses on the investigation of real catalysts under reaction conditions by photoelectron spectroscopy in the soft x-ray range.

Knop-Gericke can be reached at the Fritz Haber Institute, Department of Inorganic Chemistry, Faradayweg 4–6, 14195 Berlin,



Anna Puig-Molina

Germany; tel. +49 30 8413 4422; e-mail knop@fhi-berlin.mpg.de.

E. Lundgren is a special researcher funded by the Swedish Research Council, placed at the Department of Synchrotron Radiation Research at Lund University in Sweden. Lundgren earned his PhD degree in 1996 at Lund University.

Before his research appointment at the university, Lundgren was a postdoctoral researcher at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, and was a European Commission Marie Curie fellow at the TU–Wien in Vienna, Austria.

His research is focused on fundamental studies of structures at surfaces and nanostructures on the atomic scale.

Lundgren can be reached at the Department of Synchrotron Radiation Research, Institute of Physics, Lund University, Sweden; tel. +46 46 222 4154; e-mail edvin. lundgren@sljus.lu.se.

Anna Puig-Molina is research scientist at Haldor Topsøe A/S, where she is responsible for catalyst characterization with synchrotron x-ray techniques. She obtained her PhD degree



Günther Rupprechter

in crystallography at the Autonomous University of Barcelona, and did several years of postdoctoral research at the European Synchrotron Radiation Facility (ESRF) before joining Haldor Topsøe A/S in 2001.

Her interests are centered on structure—activity relationships in heterogeneous catalysis and *in situ* techniques applied to heterogeneous catalyst characterization.

Puig-Molina can be reached at Haldor Topsøe A/S, Materials Characterization, Research, and Development, Nymøllevej 55, DK-2800 Kgs. Lyngby, Denmark; tel. +45 4527 2658; e-mail apm@ topsoe.dk.

Günther Rupprechter is the chair of surface and interface chemistry at the Vienna University of Technology's Institute of Materials Chemistry. He also is head of the Austrian Catalysis Society.

After receiving his PhD degree in 1996 from the University of Innsbruck, Rupprechter served as a postdoctoral fellow at the University of California, Berkeley, until 1998. From 1999 to 2005, he was the group leader for laser spectroscopy and catalysis at the Fritz Haber Institute of the Max Planck Society.

Rupprechter has published more than 90



Miquel Salmeron

papers on surface chemistry and catalysis, focusing on nanoparticle characterization, *in situ* vibrational spectroscopy, and structure–activity correlations in heterogeneous catalysis. In 2005, he received the Jochen Block Award of the German Catalysis Society for his work on the application of surface science methods to heterogeneous catalysis.

Rupprechter can be reached at Veterinärplatz 1, 1210 Vienna, Austria; tel. +43 1 25077 3813; e-mail grupp@ imc.tuwien.ac.at.

Miquel Salmeron is a senior scientist at Lawrence Berkeley National Laboratory and adjunct professor in the Materials Science and



Robert Schlögl

Engineering Department at the University of California, Berkeley. He also is director of the Imaging and Manipulation Facility of the Molecular Foundry, the Department of Energy Nanoscience Institute at Berkeley.

Salmeron received his BA degree in physics from the University of Barcelona and his PhD degree from the **Autonomous University** of Madrid. In 1984, he moved to LBNL as a divisional fellow, becoming a senior scientist in 1996. Salmeron's research focuses on atomic-scale structure and properties of surfaces and nanomaterials for applications in electronics, catalysis, tribology, and environmental science.



**Moniek Tromp** 

He was elected fellow of the American Physical Society in 1996, and of the American Vacuum Society in 2003. He received the Outstanding Research Award (1996) and the Outstanding Scientific Accomplishment Award in Materials Chemistry (1995) from the U.S. Department of Energy. In 2004, he received the Klaus Halbach Award for the development of innovative instrumentation at the Advanced Light Source.

Salmeron has published 360 journal articles and book chapters.
Salmeron can be reached at Lawrence Berkeley National Laboratory, Materials Sciences
Division, MS 67R2111,
Berkeley, CA 94720, USA;

tel. +1-510-486-5431; e-mail mbsalmeron@ lbl.gov.

Robert Schlögl is a director at the Fritz Haber Institute (FHI) of the Max Planck Society in Berlin, where he has been since 1994. The Department of Inorganic Chemistry at FHI focuses on understanding of heterogeneous catalysts. The mission of the department is to bridge the gap between nonfunctional model systems for demanding reactions and technical heterogeneous catalysts by in situ spectroscopy and controlled synthetic chemistry.

Schlögl studied inorganic chemistry at the Ludwig Maximilian University of Munich and was a postdoctoral researcher at Cambridge and Basel with a focus in solid-state science.

He earned his *habilitation* at the Technical University of Berlin in 1989. From 1989 through 1994, he was a full professor at Frankfurt University.

Schlögl can be reached at the Fritz Haber Institute, Max Planck Society, Faradayweg 4–6, D-14195 Berlin, Germany; tel. +49 30 8413 4400; e-mail acsek@fhi-berlin.mpg.de; Web site www.fhi-berlin. mpg.de.

Moniek Tromp is currently an Engineering and Physical Sciences Research Council (EPSRC) advanced research fellow at the University of Southampton.

She obtained her PhD degree in chemistry (cum laude) at Utrecht University, the Netherlands. Afterward, she was a postdoctoral research assistant at the University of Southampton. Her interests are centered on the development of x-ray absorption spectroscopy methods and related characterization techniques for their application in homogeneous and heterogeneous catalysis, toward obtaining more insights into their structural- and electronicperformance relationships.

Tromp can be reached at the University of Southampton, School of Chemistry, Southampton, S017 1BJ, UK; tel. +44 23 8059 6877; e-mail m.tromp@ soton.ac.uk.

