

Novel *In Situ* Probes for Nanocatalysis

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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 sta-

bility, 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).² 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 ultrahigh-vacuum (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,² 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 so-called “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 application-relevant 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 under-coordinated 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

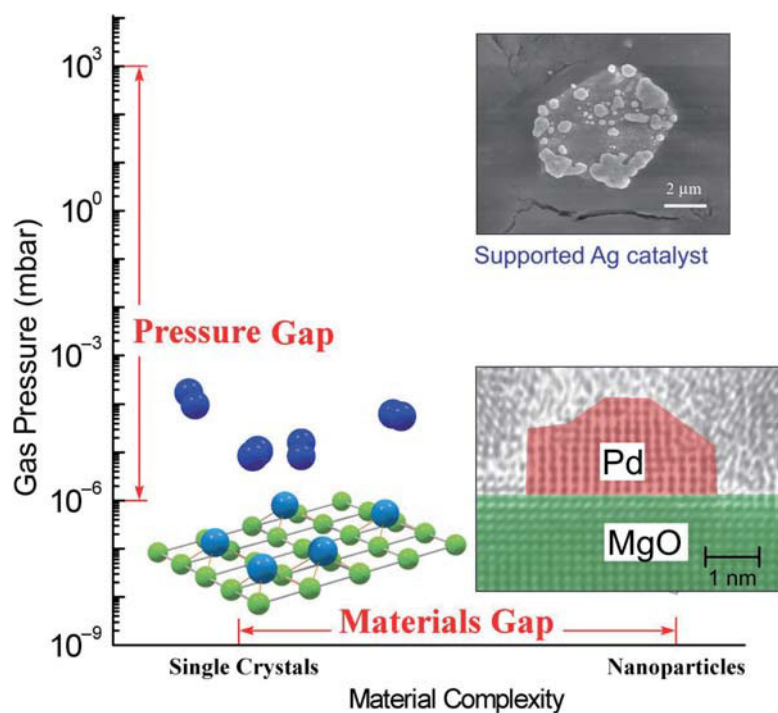


Figure 1. Pressure and materials gaps for chemical reactions: low-pressure studies on single crystals suffer from a lack of applicability to “real” systems. Novel experimental techniques, such as those discussed in this issue, bridge the enormous pressure gap of more than 9 orders of magnitude and the 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_2 response is detected by a mass spectrometer (10^{-6} mbar regime).¹⁰ Catalytic oxidation reactions at elevated pressures cannot be studied using these systems, because the ultrathin epitaxial oxide films are expected to be unstable under high-pressure 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 atomic-scale 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 single-crystal surface. X-ray photoelectron spectroscopy (XPS) is performed by using incident photons in the energy range $h\nu$ 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 (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 ($h\nu$ 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 ($h\nu'$ 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 grazing-incidence 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^{12–14} over a pressure range

Table I: Overview of Accessible Parameters for High-Pressure *In Situ* Probes.

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	...	5 cm ⁻¹ 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	...	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 ⁻¹ 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

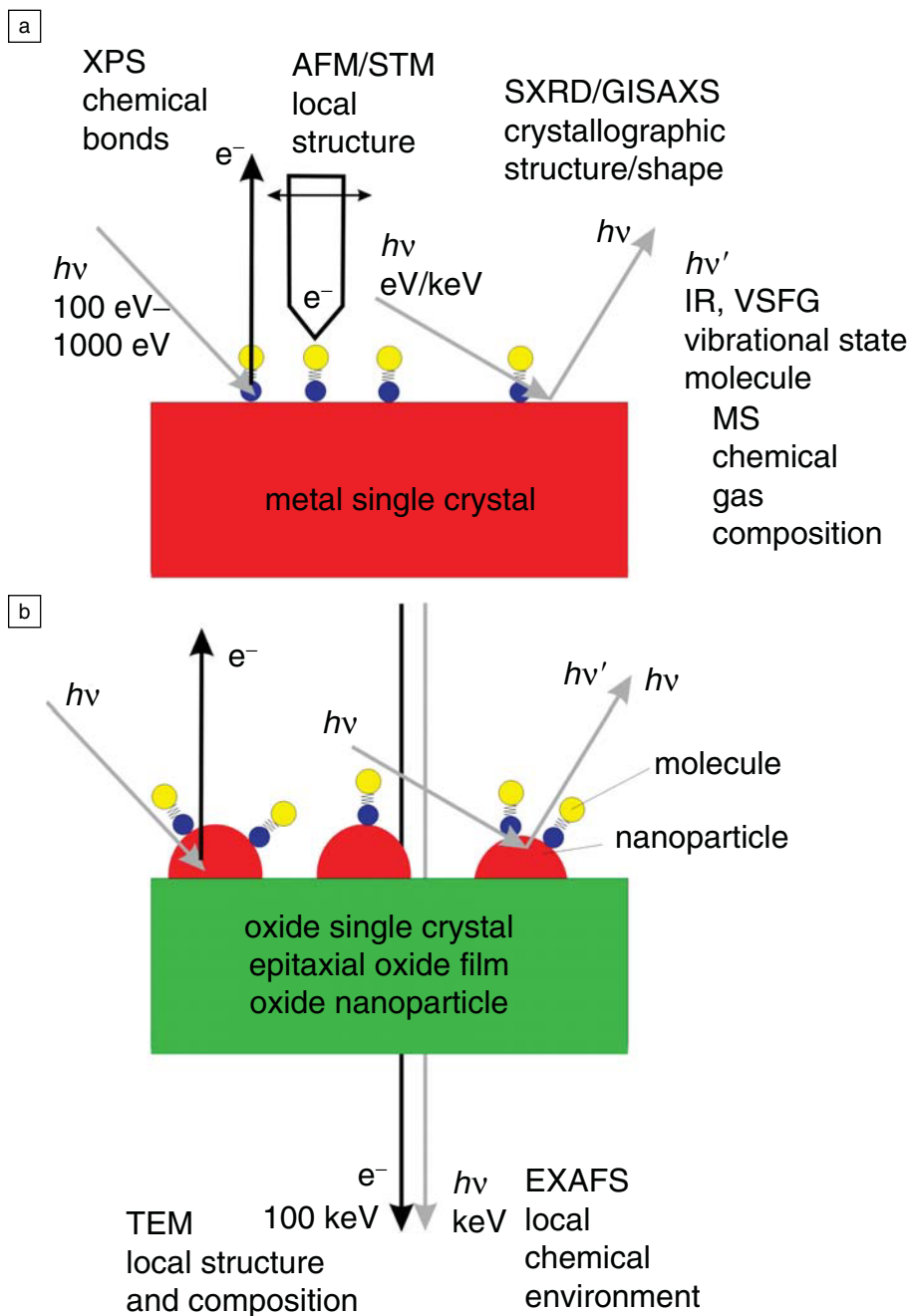


Figure 2. Novel *in situ*, pressure- and materials-gap-bridging techniques for studies on (a) single crystals and (b) supported nanoparticles. See text for discussion.

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.^{15–18} *In situ* STM is the topic of the article by Frenken and Hendriksen in this issue.

XPS using a differentially pumped spectrometer^{19,20} 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.^{21–24}

Information on the local structure and chemical environment of the nanoparticles can be obtained by EXAFS and near-edge 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^{25,26} (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.²⁷

Finally, a differentially pumped transmission electron microscope has been developed that operates at pressures up to 20 mbar and temperatures up to 900 K.^{28–30} 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 catalytic surface.

Conclusions

Using a combination of different *in situ* high-pressure- and high-temperature-compatible techniques, new atomistic insight into chemical reactions on nanoparticle systems under application- and 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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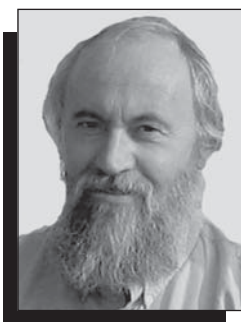
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