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BIBLIOGRAPHY — JOURNAL ARTICLES

Title	Authors	Citation	Description
NiAl Oxidation Reaction Processes Studied In Situ Using MEMS-Based Closed-Cell Gas Reaction Transmission Electron Microscopy	Kinga A. Unocic, Dongwon Shin, Raymond R. Unocic, Lawrence F. Allard	*Oxid. Met.*, v87, pp1, 2017	The in situ closed-cell gas reaction STEM characterization method presented in this study offers the potential to understand the fundamental high-temperature oxidation mechanisms and kinetics for a wide range of alloys.
Quantitative and Atomic Scale View of CO-Induced Pt Nanoparticle Surface Reconstruction at Saturation Coverage via DFT Calculations Coupled with in-situ TEM and IR	Talin Avanesian, Sheng Dai, Matthew J. Kale, George W. Graham, Xiaoqing Pan, and Phillip Christopher	*J. Am. Chem. Soc.*, v139, pp4551, 2017	This approach is expected to be useful for identifying the exposed surface structure on various supported metal catalysts under reactions conditions, and have impacts on active site elucidation for structurally dynamic catalytic systems.
In situ Atomic-Scale Observation of 2D $Co(OH)_2$ Transition at Atmospheric Pressure	Xiaochen Shen, Sheng Dai , Changlin Zhang , Shuyi Zhang, Stephen M. Sharkey, George W. Graham, Xiaoqing Pan, and Zhenmeng Peng	*Chem. Mater.*, v29, pp4572, 2017	The in situ techniques demonstrated in this study can also be extended to many other material transition systems, which offer a novel opportunity to get insightful understanding of 2D materials and develop ideal materials with excellent performance.
In Situ Observation of Rh-$CaTiO_3$ Catalysts during Reduction and Oxidation Treatments by Transmission Electron Microscopy	Sheng Dai, Shuyi Zhang, Michael B. Katz, George W. Graham, and Xiaoqing Pan	*ACS Catal.*, v7, pp1579, 2017	In situ oxidation and reduction experiments at 1 atm pressure revealed working mechanism of intellegent catalyst nanoparticles.
The application of in situ analytical transmission electron microscopy to the study of preferential intergranular oxidation in Alloy 600	M.G. Burke, G. Bertali, E. Prestat, F. Scenini, S.J. Haigh	*Ultramicroscopy,* v176, pp46, 2016	Electropolished sample was exposed to H2-H2O vapor for replicate nuclear power systems corrosion environments. Combination of images, EDS and ex-situ verifications revealed the degradation mechanism and effect of grain boundary.
High-temperature electron microscopy study of ThO_2 microspheres sintering	G.I. Nkou Boualaa, N. Claviera, J. Léchelleb, J. Monniera, Ch. Ricolleaud, N. Dacheuxa, R. Podora	*J. Eur. Ceram. Soc.*, v37, pp727, 2016	The recommendations and findings in this work back up the innovative ways ofpreparation currently under investigation for the fabrication ofnuclear fuels for the next generation of reactors.
Dynamical Observation and Detailed Description of Catalysts under Strong Metal−Support Interaction	Shuyi Zhang, Philipp N. Plessow, Joshua J. Willis, Sheng Dai, Mingjie Xu, George W. Graham, Matteo Cargnello, Frank Abild-Pedersen, and Xiaoqing Pan	*Nano Lett.*, v16, pp4528, 2016	Catalytic behavior of TiO_2 supported Pd nanoparticles is studied under sequential reducing and oxidizing environments. The experiments were carried out under 1atm pressure and at elevated temperatures of up to 500 °C. It was observed that TiO_2 layer forms, first as a monolayer and then double layer, during the reducing step. Formation for of the double layer resulted in a round-to-faceted transformation of the Pd nanoparticles. High resolution HAADF and ABF STEM imaging, EELS and DFT calculations revealed unprecedented details toward understanding strong metal support interaction of catalysts.
Defects do Catalysis: CO Monolayer Oxidation and Oxygen Reduction Reaction on Hollow PtNi/C Nanoparticles	Laetitia Dubau, Jaysen Nelayah, Simona Moldovan, Ovidiu Ersen, Pierre Bordet, Jakub Drnec, Tristan Asset, Raphaël Chattot, Frédéric Maillard	*ACS Catal.*, v6, pp4673, 2016	Hollow PtNi nanoparticles supported on carbon were thermally treated under different types of gases to controllably heal the structural defects. Aberration-corrected HRTEM imaging and EDS were utilized to investigate structural evolution as a function of gas and temperature. In situ TEM annealing along with XRD and electrochemical measurements shed fundamental light on the effect of structural defect on the catalytic performance of bimetallic nanomaterials.
Determination of the initial oxidation behavior of Zircaloy-4 by in-situ TEM	Wayne Harlow, Hessam Ghassemi, Mitra L. Taheri	*J. Nucl. Mater.,* v474, pp126, 2016	Zirconium-based alloys (Zircaloy) are important materials in nuclear fuel cladding, as they are corrosion resistant and exhibit a low neutron cross section. However, the oxidation behavior is not well understood. Researchers at Drexel University sought to better understand the oxidation of Zircaloys usingused the Atmosphere gas cell, focused ion beam (FIB) sample preparation and precession diffraction analysis. They found that in situ analysis compared well with ex situ analysis techniques, confirming the viability of in situ analysis for this material. Changes in grain structure and including texture and boundaries are reported.
Improved Thermal Stability and Methane-Oxidation Activity of Pd/ Al2O3 Catalysts by Atomic Layer Deposition of ZrO2	Tzia Ming Onn, Shuyi Zhang, Lisandra Arroyo-Ramirez, Yu-Chieh Chung, George W. Graham, Xiaoqing Pan, Raymond J. Gorte	*ACS Catal.,* v5, pp5696, 2015	Nanoparticle catalysts for automotive exhaust applications must remain thermally stable for optimal efficiency. This paper introduces a method to thermally stabilize Pt nanoparticles on alumna using a thin layer of zirconia deposited via ALD. In situ TEM measurements with Atmosphere were used to confirm the thermal stability under typical calcination conditions. The authors found that the thin zirconia layers form nanoparticles at surprisingly low temperatures, and confirmed the behavior seen in ex situ analysis.
Preparation and Loading Process of Single Crystalline Samples into a Gas Environmental Cell Holder for In Situ Atomic Resolution Scanning Transmission Electron Microscopic Observation	Rainer Straubinger, Andreas Beyer, Kerstin Volt	*Microsc. and Microanal.,* v22, pp515, 2016	Focused ion beam (FIB) sample preparation is an important technique for TEM analysis of bulk materials, including single crystal samples. Closed cell holders are single tilt, so the ability to tilt to a zone axis is limited. A method to create FIB samples compatible with the Atmosphere gas cell is described, including a method to precisely orient single crystal samples, so high resolution STEM images are achieved.

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Revealing particle growth mechanisms by combining high-surface-area catalysts made with monodisperse particles and electron microscopy conducted at atmospheric pressure	Shuyi Zhang, Matteo Cargnello, Wei Cai, Christopher B. Murray, George W. Graham, Xiaoqing Pan	*J. Catal.,* v337, pp240, 2016	Nanoparticle catalysts are becoming increasingly important across a variety of applications. They offer high surface area, so less material is required, which reduces costs. However, the thermal stability of nanoparticle catalysts is problematic because nanoparticles tend to agglomerate into larger particles reducing their effectiveness. They usually agglomerate in one of two ways: particle migration and Oswald ripening. To better understand the agglomeration behavior, researchers used Atmosphere to study Pt nanoparticles of various sizes supported on alumina under reducing conditions and high temperature. They found that both agglomeration processes can be limited using highly mono disperse nanoparticles.
In Situ Catalyst Analysis at the Nanoscale Using Protochips Atmosphere 200 Gas E-cell System	Benjamin W Jacobs	*Microscopical Society of Canada Bulletin,* v43, 2015	Catalysts are critical in a variety of industries including automobiles, oil and gas and chemical and materials synthesis. Catalyst reactions happen on the surface of materials, and to understand these reactions and how catalyst materials evolve over time, researchers often want to see samples at the nano and atomic scales. The TEM is often the tool of choice for small scale analysis, however, it operates under high vacuum. Atmosphere allows catalyst researchers to probe materials under realistic reaction conditions, including up to 1 atm and 1000 deg C. In the paper, the Atmosphere system is described in detail, and two catalyst applications are described.
Correlation of morphology with catalytic performance of CrOx/Ce 0.2 Zr 0.8 O 2 catalysts for NO oxidation via in situ STEM	Cai, Wei, et al.	*Chem. Eng. J.,* v288, pp238, 2015	The drying conditions during synthesis of Ce0.2Zr0.8O2 was investigated and correlated to the the NO oxidation efficiency. The authors used in situ TEM with additional techniques such as XRD and XPS, to evaluate the resulting material structure as a function of drying rate. They concluded that a slow drying rate and the nature of the solvent increases surface area, and increases the number of oxygen vacancies leading to better oxygen adsorption thus higher NO oxidation activity.
Dynamic structural evolution of supported palladium–ceria core–shell catalysts revealed by in situ electron microscopy	Zhang, S., Chen, C., Cargnello, M., Fornasiero, P., Gorte, R. J., Graham, G. W. and Pan, X.	*Nat. Commun.,* v6, pp1, 2015	Report on use of advanced ex situ and in situ electron microscopy with atomic resolution to show that the modular palladium–ceria core–shell subunits undergo structural evolution over a wide temperature range. In situ observations performed in an atmospheric gas cell within this temperature range provide real-time evidence that the palladium and ceria nanoparticle constituents of the palladium–ceria core–shell participate in a dynamical process that leads to the formation of an unanticipated structure comprised of an intimate mixture of palladium, cerium, silicon and oxygen, with very high dispersion.
In situ Analytical TEM of Ilmenite Reduction in Hydrogen	Janssen, Arne, et al.	*Microsc. and Microanal.,* v21, pp565, 2015	Extended abstract of a paper presented Ilmenite (FeTiO3) is an important mineral, formed in igneous and metamorphic rocks, and is the main feedstock for the titanium industry. Ilmenite is also an attractive and inexpensive oxygen carrier for chemical-looping combustion (CLC), one of the most promising technologies to accomplish CO2 capture in power plants.
XEDS Performance of Atmospheric Membrane Holders in the AEM	Zaluzec, Nestor J.	*Microsc. and Microanal.,* v21, pp733, 2015	Extended abstract of a paper presented the use of SiNx windows has been well established as a technology which can be readily employed to observe microstructural evolution during in situ studies in the TEM/STEM as an alternative to experiments using a differentially pumped environmental TEMs.
In situ Observation of Annealing Effects in Ga (NAsP) Multi Quantum Well Structures	Straubinger, Rainer, et al.	*Microsc. and Microanal.,* v21, pp957, 2015	Extended abstract of a paper presented the epitaxial growth of multi component semiconductor layers such as Ga(NAsP) enables the improvement of laser and transistor devices due to the individually tunable band gap and lattice constant. Furthermore, the material system shows great potential for realizing optical light sources on Si substrate.
In Situ Environmental Transmission Electron Microscopy Study of Oxidation of Two-Dimensional Ti3C2 and formation of Carbon-Supported TiO2.	Ghassemi, H., W. Harlow, O. Mashtalir, M. Beigdahi, M.R. Lukatskaya,Y. Gogotsi and M.L. Taheri	*J. Mater. Chem.,* v2, pp14339, 2014	The oxidation in air of two-dimensional Ti3C2 (MXene) using two different oxidizing regimes with the intent of producing carbon-supported TiO2. The researchers observed that by controlling time, temperature and heating rate with in situ TEM and Raman spectroscopy, either anatase nanoparticles or planar rutile nanocrystals would result depending on the conditions.
Synthesis of Mesoporous Palladium with Tunable Porosity and Demonstration of Its Thermal Stability by In Situ Heating and Environmental Transmission Electron Microscopy	Cappillino, Patrick J, Khalid M. Hattar, Blythe G. Clark, Ryan J. Hartnett, Vitalie Stavila, Michelle A. Hekmaty and David B. Robinson	*J. Mater. Chem.,* v1, pp602, 2013	A technique for creating mesoporous Pd powder with pore diameters of 7 or 13 nm for the purposes of high-performance hydrogen storage as well as chromatographic separation of hydrogen isotopes, electrocatalysis and catalysis. The researchers used heated-stage TEM to observe the materials in vacuum and in the presence of H2 gas in situ, thereby showing that pore diameter and the surface's chemical state are essential to determining thermal stability. Improved stability enables the preparation of scalable quantities of regularly mesoporous PD that retain their porosity at the conditions necessary for application to hydrogen charge, discharge and catalysis.
Novel Closed Cell Gas-Reaction Holder Allows Characterization of Behavior of Bimetallic Nanoparticles at Elevated Temperatures and Gas Pressures	Allard, L. F., J.E. Wittig, M. Chi, K.L. More, W.C. Bigelow, J. Damiano and D.P. Nackashi	*Microsc. and Microanal.,* v19, pp1474-1475, 2013	The gas composition and type can have a dramatic effect on the temperature of the heating membrane inside the gas cell. New work presented here show the effect, and how much the temperature changes in different environments.
Understanding Behavior of Self-Regenerative Catalysts by Transmission Electron Microscopy and Density Functional Theory	Pan, X., M.B. Katz, B. Li, X. Du, Y. Duan, L.F. Allard, G.W. Graham, et al.	*Microsc. and Microanal.,* v19, pp1694, 2013	The gas cell is used to study the behavior of metal/perovskite materials for 3-way automotive catalysts. The in situ studies revealed a more complex interplay between the metal and support than had been previously realized.

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Novel MEMS-Based Gas Cell/Heating Specimen Holder Provides Advanced Imaging Capabilities for In Situ Reaction Studies	Allard, L. F., S.H. Overbury, W.C. Bigelow, M.B. Katz, D.P. Nackashi and J. Damiano	*Microsc. and Microanal.,* v18, pp656, 2012	The development of a new specimen holder that adapts the Aduro device into a "closed-cell" configuration. The adapted holder is designed to function within the 2 mm-thick pole piece gap of an aberration-corrected JEOL 2200FS STEM/TEM's objective lens. It can also expose specimens to gases at pressures up to 1 atm. The researchers present the results of early tests using the holder at elevated temperatures and pressures up to one atmosphere. The resulting imaging retains the microscope's atomic resolution performance in both high-angle annular dark-field and bright field modes.
In Situ TEM Ion Irradiation and Atmospheric Heating of Cladding Materials	Hattar, K., S. Rajasekhara, and B. G. Clark	*MRS Proceedings,* v1383, 2012	A description of the new in situ ion irradiation TEM and in situ gas cell capabilities developed at Sandia National Laboratories, as well as demonstration of their use. The researchers used both systems to investigate potential Generation IV cladding materials' radiation tolerance as well as Zr-based claddings' degradation mechanisms.
In Situ Observation of the Evolution of Pt Particles in a Perovskite-Based Catalyst During Redox Cycling at High Temperature and Atmospheric Pressure with Atomic Resolution	Katz, M. B., Y. Duan, G.W. Graham, X. Pan and L.F. Allard	*Microsc. and Microanal.,* v18, pp1120, 2012	Perovskite materials are a promising material for new 3-way automotive catalysts. The authors use the Atmosphere system to better understand the cycling process in Pt doped calcium titanate. Using high temperature reduction and oxidation, they obverse Pt agglomeration and re-dispersion.
Hydride Formation in Cladding Materials Studied via In Situ Environmental Heating Transmission Electron Microscopy	Rajasekhara, Shreyas, K. Hattar, V. Tikare, R. Dingreville and B. G. Clark	*Sandia National Laboratories,* SAND2012-6468C, 2012	The study of hydride formation in Zirlo™ cladding material using a Protochips environmental in-situ heating TEM stage. Two Si-based chips with 5 nm-thick SiNx viewing windows (one of which was patterned with a resistive heater) comprised the gas cell held in the stage tip. This contrivance allowed the researchers to observe in-situ the material's microstructural transformation as it was exposed to temperatures up to 1200° C and pressures up to 1 atm. The results show the disappearance of microstructural features and the formation of a new grain, demonstrating the possibilities of using in situ observation to verify and validate predictive material models.
Development of a Novel Environmental Cell for In Situ Gas Reaction Experiments via Aberration-Corrected STEM Imaging	Allard, L. F., W.C. Bigelow, S.H. Overbury, D.P. Nackashi and J. Damiano	*Microsc. and Microanal.,* v16, pp296, 2010	Latest results using the Protochips in situ gas cell are discussed, including high resolution images of Au nanoparticles on an iron oxide support at 2 Torr and temperatures of 500 deg C.
A Novel Heating Technology for Ultra-High Resolution Imaging in Electron Microscopes	Allard, Lawrence F., Wilbur C. Bigelow, Steven A. Bradley and Jingyue "Jimmy" Liu	*Micros. Today,* v17, pp50, 2009	The development of a new technology for in situ heating experiments that can record images at high temperatures without compromising the microscope's resolution. Previously developed devices have proved to be limited by the sample's speed at reaching a stable operation temperature. They also lack the required stability for sub-Ångström imaging. In creating their new device, the researchers seek to solve both these and other problems currently faced by those using present technology.