IN-SITU AND OPERANDO CHARACTERIZATION OF MATERIALS

This special issue of the Journal of Materials Research contains articles that were accepted in response to an invitation for manuscripts.

Introduction

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Materials studies under in-situ and operando conditions in many cases provide key information towards understanding properties and function. Remarkable advances in analytical tools and data modeling have been made in recent years, with in-situ analysis continually developing to exploit the new capabilities. In-situ analysis relies on complex sample environment systems, necessitating careful development of experimental protocols to optimize accuracy and reliability of the results. Furthermore, in-situ studies typically generate large data sets that may be treated using new "parametric" approaches, wherein all individual datasets are considered as a single experimental observation. In some cases, control over temperature, pressure, and/or electric and magnetic fields, especially when combined with the introduction of reactive or process gasses, allows studies of active materials under operating conditions. Such operando studies probe the specimens in the true operating environment, often necessitating new and unique sample environment systems. This Focus Issue of the *Journal of Materials Research* (*JMR*) captures the most recent advances in in-situ analysis over a broad range of characterization tools.

The Focus Issue contains papers covering primarily diffraction and electron and probe microscopy, with invited papers capturing the state of the art for electron microscopy and AFM. A majority of the papers are aimed at tracking electrochemical responses in lithium ion batteries and fuel cells, with additional papers on electroactive materials and even MEMS systems, all topics of tremendous current interest. However, most of the techniques employed are equally or more applicable for studies of other ceramic, metal, polymeric and biological materials, and as such will appeal to a broader audience. Both laboratory experiments and synchrotron/neutron studies are represented in the issue, providing a flavor for the capabilities and limitations of each type of characterization tool and perhaps stimulating interest in the use of the large scale neutron and x-ray facilities around the world.

In addition to detailed descriptions of the remarkable experimental facilities available today, the papers in the issue cover advanced data analysis procedures and demonstrate extraction of meaningful models of materials that are not easily uncovered otherwise. Indeed, the issue contains reports of uniquely new information extracted from in-situ studies under, for example, electric field, where it is not physically possible to quench a sample for later study.

Taken together, the papers point to a bright future for in-situ/-operando systems, measurements, and analysis.

ON THE COVER: In-situ and operando investigations are crucial for assessing, understanding, and thereby ultimately improving the functionality, efficiency, and reliability of a wide range of devices. Examples include mapping the performance of solar cells under various conditions of illumination, sensors in the presence of various analytes, MEMS devices for various operating conditions, catalysis in various environments, fuel cells over various temperature ranges, etc. The cover figure (by Y. Kutes, University of Connecticut, B. A. Aguirre and D. Zubia, University of Texas El Paso, Jose L. Cruz-Campa, Sandia National Laboratories, and B. D. Huey, University of Connecticut) displays the three dimensional topography of a microstructured array of CdTe solar cells protruding through a dielectric film (12um x 12um x 600nm shown). Photocurrents resulting from illumination by ~1.6 suns were measured in-situ, overlain as color contrast indicating a range of approximately 30 pA between photoactive specimen regions and the surrounding dielectric. The contrast results from distinct grain orientations, grain boundaries, and current percolation pathways in the polycrystalline solar cell.

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