doi:10.1017/S1431927613003978
Microsc. Microanal. 19 (Suppl 2), 2013

In-Situ Environmental Atom Probe Tomography for Studying Gas-Solid Reactions In Extreme Environments: Instrumentation and Results

S. Dumpala[1], S.R. Broderick[1], P.A.J. Bagot[2] and K. Rajan[1]

[1.] Department of Materials Science and Engineering and Institute for Combinatorial Discovery, Iowa State University, 2220 Hoover Hall, Iowa State University, Ames, USA
[2.] Department of Materials, University of Oxford, Parks Road, Oxford OX13PH, UK

Quantitative characterization of the first few monolayers of gas-metal interaction is necessary to understand the degradation of materials and surface reaction mechanisms. Atom probe tomography (APT) offers advantages over other techniques with its unique capability to simultaneously compute 3D spatial information and chemical identity individual atoms. Extending the capabilities of APT beyond any other technique, we have designed and built an environmental cell for in-situ gas-solid reactions. We describe this challenge with the design and operational details of an environmental cell coupled to a local electrode atom probe (LEAP) to link surface and sub-surface chemistry/structure to gas-solid reactions, present results from this new APT system and demonstrate what is gained with this new analytical approach.

The current environmental cell is a development from a previous prototype designed for studying catalytic reactions on an earlier generation 3D Atom Probe [1-2]. In a similar set-up to this earlier design, the new unit is integrated directly onto the existing vacuum storage chamber of a LEAP instrument to minimize surface contamination of treated samples. By attaching the new cell onto a laser-pulsed atom probe, we have the potential to now study a much wider range of treatment conditions including thick oxide layers, while the cell is engineered to operate at higher temperature/pressures to greatly broaden its applications to materials science problems. Fig. 1 shows the detached cell (a) then as installed on the LEAP (b).

This cell will lead to new studies of 3D imaging of nanoscale oxides and quantification of the oxide layers formed. As an example, we demonstrate a study of the aluminum-oxygen interaction by pulsed laser APT. The diffusion of oxygen into aluminum following exposure at 723 K and $2x10^{-3}$ Torr for 10 minutes, as evident by the presence of oxygen and various aluminum oxides on the aluminum tip surface can be seen in the 3D APT data of Fig. 2(a). In addition to chemically-mapped atomic scale imaging of the material, the spatial and chemical information obtained from the APT is processed to identify the preferred stoichiometry of aluminum oxides as a function of the distance from the aluminum-oxygen interface (Fig. 2(b)), thus quantifying the microstructure of the contaminant layer formed due to aluminum-oxygen interaction. These diffusion profiles provide information about the metal-gas reaction kinetics at the nanoscale that can be extrapolated to quantify the material characteristics. This demonstrated ability of the APT to simultaneously image and chemically quantify gas-metal interactions at atomic level enables us to systematically quantify these interactions as a function of material chemistries, crystallographic orientations and important microstructural features.

References:

[1] P.A.J. Bagot *et al*, Surface Science **600** (2006) 3028.
[2] P.A.J. Bagot *et al*, Surface Science **601** (2007) 2245.
[3] The authors acknowledge the support from Air Force Office of Scientific Research grants: FA9550-10-1-0256, FA9550-11-1-0158 and FA9550-12-0496; NSF grants: ARI Program CMMI-09-389018 and PHY CDI-09-41576; and Defense Advanced Research Projects Agency grant N66001-10-1-4004.

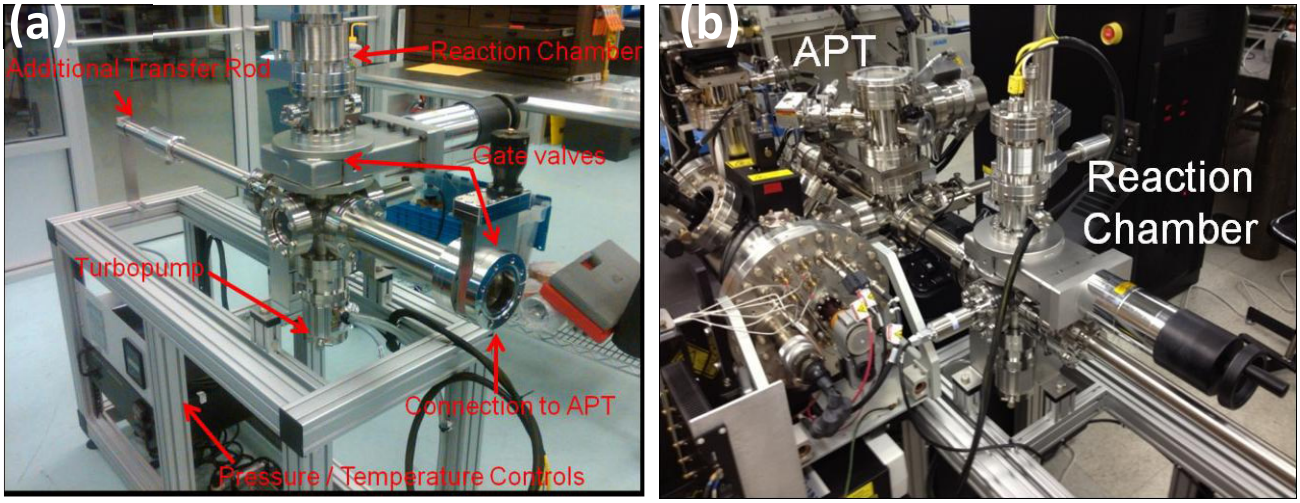

Figure 1. (a) The complete environmental cell design (with all the parts named). The sample transfer, pumping and gas inlet to this chamber have no effect on the atom probe or its standard operation, (b) APT with the environmental cell connected.

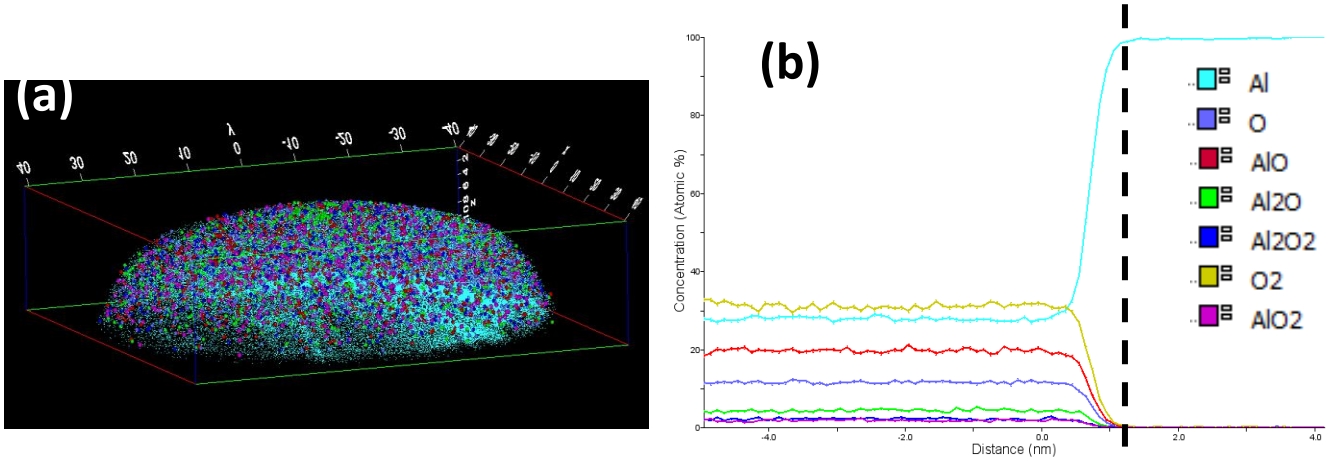

Figure 2. (a) 3D image of nanoscale oxide layer formed on Al following exposure to 723 K and $2x10^{-3}$ torr for 10 minutes (each sphere corresponds to an ion detected in APT), (b) Compositional variation of Al, O and various oxides of aluminum as a function of the distance from the Al-O interface (dashed line)

https://doi.org/10.1017/S1431927613003978 Published online by Cambridge University Press