Seeing is Believing – New Horizons for In Situ Microscopy

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New innovations are transforming the Transmission Electron Microscope (TEM) from a simple high-resolution image acquisition tool into a nanoscale materials research and development laboratory. Researchers can now better understand material behavior by analyzing samples in real-world gas or liquid environments, at high temperature and with ultra-low noise electrochemical and electrical characterization techniques. With the new *in situ* tools from Protochips, materials research occurs in highly controlled environments at high resolution without sacrificing the analytical capabilities of the TEM such as EDS. Applications for these tools include heterogeneous catalysis, semiconductor fabrication, lithium-ion batteries, energy materials, memory storage, and more. In this presentation, we show some of the most recent in situ results and findings from leading researchers in fields spanning the entire scientific discipline from life science to material science.

The recently released Atmosphere 210 gas cell delivers in situ sample heating up to 1000ºC with comprehensive environmental control of the gas environment. Arbitrary gas mixtures can be created at pressures ranging from 0.3 – 760.0 Torr and can be controlled at flow rates ranging from 0.005 – 1.000 mL/min, allowing realistic application conditions to be reproduced within the EM. Coupled with Mass Spectrometry (MS) compatibility, Atmosphere 210 enables comprehensive analysis of catalyst mateirals undergoing complex reactions with their surrounding environment [1]. Results featuring these and other analytical capabilities will be presented.

Fusion offers accurate and precise thermal and electrical characterization of samples ranging from nanowires and thin films to FIB Lamellae and 2D materials. FIB-optimized E-chips utilize a unique design that complements the conventional liftout workflow to streamline and improve sample preparation for samples ranging from metals to seminconductors. The chemically inert silicon carbide heating membrane delivers accurate, uniform heating which enables atomic resolution imaging of nucleation and growth phenomenon of nanoscale materials and much more.

The Poseidon Select liquid cell surrounds samples in a self-contained, fully hydrated, hermetically sealed chamber directly within the TEM. It features in situ electrochemistry capabilities, which enables the observation and characterization of electrochemical reactions in realistic reaction environments in real-time, and now offers liquid heating, for experiments in growth and reaction kinetics and temperature sensitive samples. Recently released EDS-optimized E-chips, which when combined with a low penumbra in situ results in a six-fold improvement in the counts per second obtained by the EDS detector and enables EDS maps to be obtained without tilting the specimen and/or at low beam currents [2].

References:
[1] K.A. Unocic *et al*, Microsc. Microanal. (2018) p. 286-287.
[2] J Moering *et al*, Microsc. Microanal. (2018) p. 304-305.

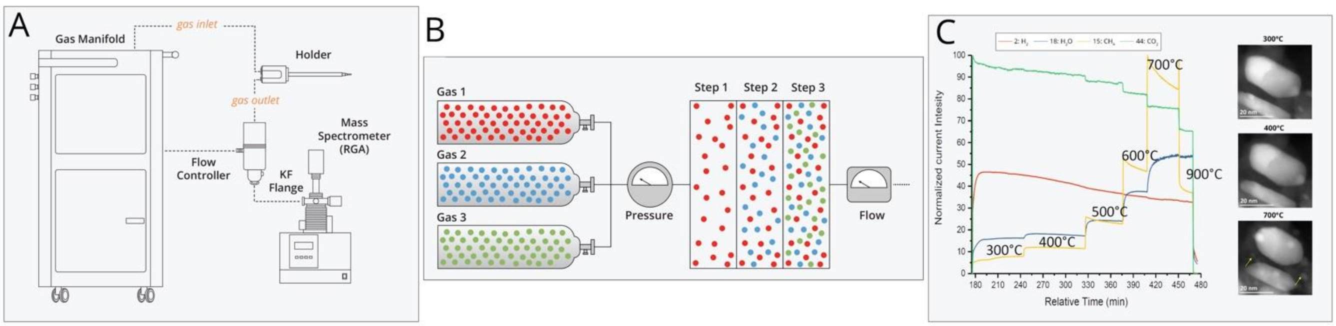

Figure 1. A) Schematic showing how a MS can be connected to the Atmosphere 210 system. B) Schematic of a three-component mixture being created with Atmosphere 210. C) MS monitoring of a NiO catalyst showing the consumption of CO_2 and H_2 in the production of CH_4 and H_2O at various temperatures. Courtesy Mounib Bahr institut de Physique et Chimie des Materiaux de Strasbourg.

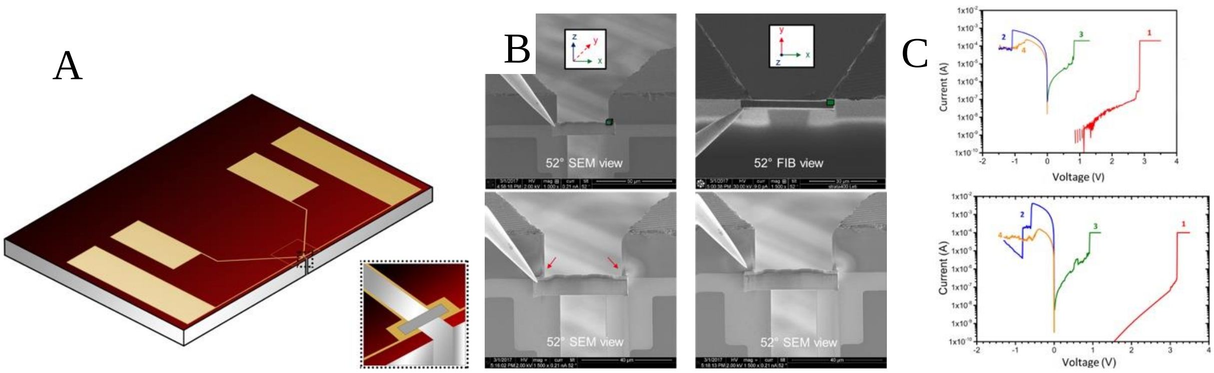

Figure 2. A) Overview of FIB-Optimized E-chip with inset showing lamellae placement. B) Attaching lamellae to the electrical contacts similar to traditional FIB sample preparation. C) In Situ electrical characterization of semiconductor lamellae (top) aligning well with test results on a bulk sample (bottom).

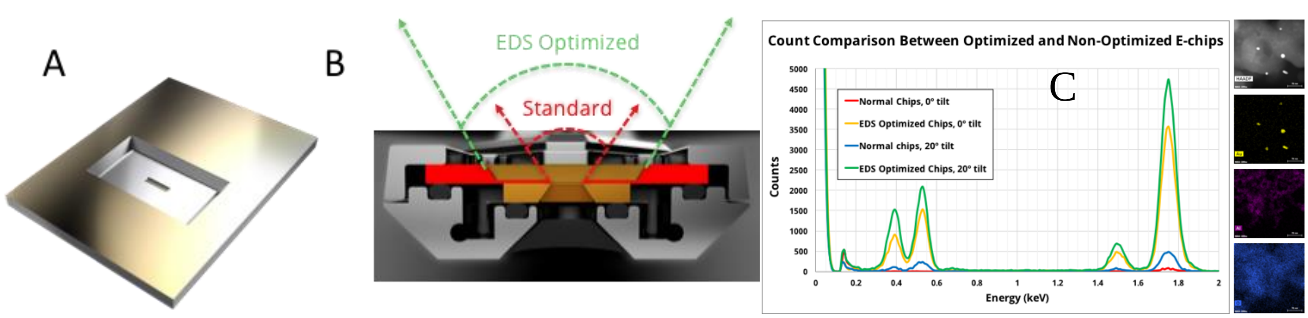

Figure 3. A) Back view of the optimized EDS E-chip for the Poseidon Select liquid cell. B) Cross section of the x-ray exit angle for the EDS optimized E-chip (green) and standard E-chip (red). C) EDS spectra of gold nanoparticles and aluminum obtained using both standard and EDS optimized E-chips, taken at 0° and 20° holder tilt, at a beam current of 60pA, and with a 5 minute acquisition time.