

## Steel Corrosion Mechanisms during Pipeline Operation: *In Situ* Characterization

Katherine L. Jungjohann<sup>1</sup>, Steven C. Hayden<sup>2</sup>, Jeremy T. O'Brien<sup>2</sup>, Timothy J. Kucharski<sup>2</sup>, William M. Mook<sup>1</sup>, Claire Chisholm<sup>1</sup>, Anastasia Ilgen<sup>3</sup>, Dan Bufford<sup>4</sup>, Khalid Hattar<sup>4</sup>, and Michele L. Ostraat<sup>2</sup>

<sup>1</sup> Sandia National Laboratories, Center for Integrated Nanotechnologies, Albuquerque, USA

<sup>2</sup> Aramco Services Company, Aramco Research Center, Boston, USA

<sup>3</sup> Sandia National Laboratories, Geochemistry, Albuquerque, USA

<sup>4</sup> Sandia National Laboratories, Radiation-Solid Interactions, Albuquerque, USA

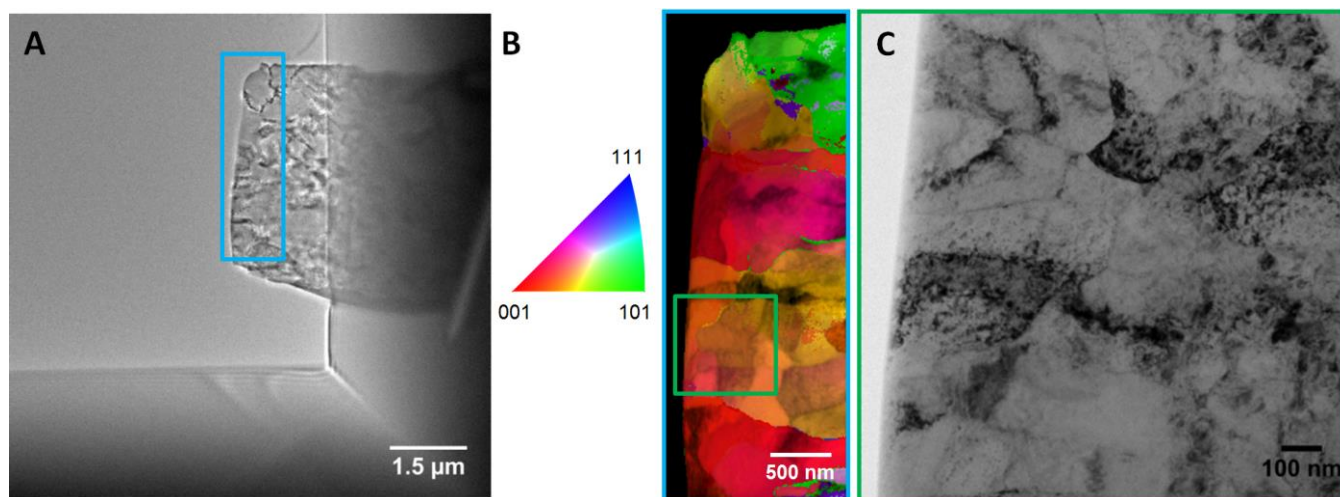
Pipeline corrosion continues to present challenges to the oil and gas industry, with billions of dollars being spent annually to remediate, repair, and replace steel piping. The corrosion processes of this piping are not well understood, due in part to the seemingly endless environmental and industrial parameters involved in corrosion of the steel. We have applied a nanoscale approach to investigate the initial oilfield corrosion pathways, formation products, and susceptible grain structures of low-alloy carbon steel through the course of corrosion by hydrogen sulfide (H<sub>2</sub>S) and carbon dioxide (CO<sub>2</sub>). The goal of the project is to develop a mechanistic understanding of the early onset of corrosion under well-controlled environmental conditions in order to develop future strategies to reduce corrosion-related losses. The most aggressive component in these operations is caused by CO<sub>2</sub> attack on the steel surface, where an aqueous phase present provides the electrolyte needed for corrosion to occur [1]. Though it is postulated that grain boundaries, material defects, and nanoscale features are likely sites for this mesa-type corrosion, predictive relationships have not been adequately demonstrated.

Pipeline steel coupons (1018 low-carbon steel) were sectioned for initial characterization, after which lift-out TEM samples were fashioned by cutting out thin foils using a focused ion-beam (FIB). The samples were then characterized using grain orientation mapping (Figure 1b) and EDS to determine the texture of the grains and to generate elemental compositional maps. Comparison to bulk surface characterization identified direct correlation in the chemical composition, although the thinning process did lead to a thin (1-2 nm) amorphous surface layer in the thin FIB foil samples. The outer thinned section of the samples was placed onto the edge of a SiN membrane window (Figure 1a), and corrosion analysis was undertaken using a microfluidic liquid-cell holder by Hummingbird Scientific [2-4]. Thin-film sections were initially mapped in vacuum to identify the locations of defect regions or interesting nanoscale features where corrosion processes were expected to occur (Figure 1c).

The pre-selected regions of interest were monitored during the in-situ TEM exposure of the low-carbon steel thin-film when exposed to varied concentrations of H<sub>2</sub>S and CO<sub>2</sub> in organic and aqueous mixtures of solvent and with flow rates of ~2 μL/min. Images were acquired sparingly to prevent beam induced processes that may promote corrosion. Beam currents of < 10 pA were used for imaging in low-dose STEM mode. Several series of images were acquired from pre-selected regions to allow for *in situ* observation of the local nanoscale corrosion process at specific regions of interest. Comparison of the in-situ TEM and ex-situ bulk scale corrosion rates/products will be described to provide details of the mechanism and rates under varied solvent conditions. Local nanoscale morphologies that exhibit susceptibility to corrosion as well as the critical parameters that influence corrosion will be described in order to advance predictive capabilities for modeling low-carbon steel corrosion by CO<sub>2</sub> and H<sub>2</sub>S exposure in aqueous environments.

## References:

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**Figure 1.** FIB lift-out thin-film of 1018 low-carbon steel, prior to *in situ* exposure to the corrosive environment. A) TEM image at the corner of a SiN membrane window. B) Precession electron diffraction mapping of the grain orientation of highlighted region in (a). C) Nanoscale features at the center-edge in sample of highlighted region in (b).