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A spectroscopic membrane permeation cell for *in-situ* infrared-reflection absorption spectroscopic analysis of membrane surfaces and simultaneous measurement of trans-

membrane gas permeation rates

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Abstract

In this work, we describe a spectroscopic membrane permeation cell that we have designed and fabricated to enable—for the first time—the surface of metal membranes to be analyzed by infrared-reflection absorption spectroscopy (IRAS) while simultaneously measuring the rate of hydrogen permeation across the membrane under realistic permeation conditions. As a proof-ofconcept, we demonstrate that the permeation cell can (1) accurately measure the rate of H_2 permeation across a 25 µm-thick Pd foil membrane, (2) detect sub-monolayer coverages of CO on the membrane surface in the 333 to 533 K temperature range, and (3) measure the rate of H_2 permeation across the membrane while simultaneously detecting surface-adsorbed CO during exposure to H_2/CO gas mixtures at 533 K. IRAS spectra recorded during exposure to H_2/CO gas mixtures at 533 K indicate that CO dissociates on the membrane surface, and C-H bonds are formed, which modify the surface adsorption properties of the membrane and result in irreversible losses in the H₂ flux across the membrane. With this spectroscopic membrane permeation cell, it is possible to correlate microscopic surface processes to macroscopic rates of

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