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Highly sulfur-tolerant Pd composite membranes with a protective layer of MoS_2/γ-alumina

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Experimental Methods

Preparation of the Pd composite membranes

Pd membranes were deposited on the outside surface of porous ceramic tubes (o.d. = 12.5 mm, i.d. = 8 mm, length of Pd layer = 50 mm) by a modified electroless plating method, which has been described previously (J. Membr. Sci., 2007, 299, 130). Three Pd composite membranes were prepared for the experiments in this work, i.e., Pd-1, Pd-2 and Pd-3. The latter two tubes (Pd-2 and Pd-3) were coated with MoS_2/γ-alumina protective layer, as described in Table 1. Following the composite membranes were sealed at both ends with graphite O-rings.

Gas-permeation measurements

A shell-and-tube apparatus, as described in our previous work (J. Membr. Sci., 2007, 299, 130), was used to measure the permeation of H_2 with different concentrations of H_2S, i.e., 10 ppm H_2S/H_2 and 20 ppm H_2S/H_2. Pure H_2 and N_2 permeation across the composite membranes were also analyzed to check the performance of Pd composite membranes. All gases were introduced into the membrane tube from the outer stainless-steel reactor through mass flow controllers (MFC). The hydrogen permeation was investigated at 673 K, and the retentate pressure was set at 1 bar gauge pressure with a pressure controller while the permeate side was always kept at atmospheric pressure without using any sweep gas. The permeation rate was monitored by bubble flow

meters while the composition of the permeate gas was analyzed by an on-line gas chromatograph (Shimadzu GC-14C) equipped with a hayesep D column and a FPD detector.

Characterization

SEM/EDS

The morphologies of membrane samples were characterized by scanning electron microscopy on a high resolution scanning electron microscope (JSM-7800F), following the exposure to H_2S containing atmosphere.