

## Preparation of a Thin Pd Membrane on a Modified Porous Stainless Steel by a Calcined Li–Al Layered Double Hydroxide Layer

Yen-Hsun Chi<sup>1,2</sup>, Meng-Chang Lin<sup>1</sup>, Yu-Li Lin<sup>1</sup>,  
Jun-Yen Uan<sup>3</sup> and Jin-Hua Huang<sup>2,\*</sup>

<sup>1</sup>Green Energy and Environment Research Lab., Industrial Technology Research Institute, Hsinchu, Taiwan, ROC

<sup>2</sup>Department of Materials Science and Engineering, National Tsing Hua University, Hsinchu, Taiwan, ROC

<sup>3</sup>Department of Materials Science and Engineering, National Chung Hsing University, Taichung, Taiwan, ROC

\*Corresponding author, E-mail address:  
[jihhuang@mx.nthu.edu.tw](mailto:jihhuang@mx.nthu.edu.tw)

Palladium (Pd) membranes have been widely used for hydrogen separation because of their high hydrogen permeability and selectivity [1]. One of the best choices for high hydrogen flux is a thin Pd membrane supported on porous substrate such as porous glass (PG) [2], porous ceramic (PC) [3], and porous stainless steel (PSS) [4], as this design increases hydrogen flux and decrease the membrane cost. In those substrates, PSS is the most frequently used due to the advantages of good mechanical strength, easy assembling, and similar thermal expansion coefficient to Pd [5]. However, PSS substrates have a large pore size, and a wide pore size distribution. Mardilovich *et al.* [6] showed that the minimum thickness of a dense Pd layer is approximately three times the size of the largest pore presented in the PSS surface. Accordingly, a modification layer is usually applied on the surface of PSS to achieve a reduced pore size and homogeneous pore distribution [7].

In this study, a calcined Li–Al layered double hydroxide layer (hereafter referred to as C-LDH layer) [8] is chosen to modify the PSS surface. The objective of the present work is to optimize the manufacturing process of a tube-shaped Pd membrane on PSS with high permeation flux using the C-LDH layer pretreatment for commercial applications.

The 0.5  $\mu\text{m}$  grade PSS tube, with 50 mm in length and 1.0 mm in diameter, were purchased from Pall Corporation. The PSS tube were welded to a nonporous stainless steel tube at one end and were sealed with a nonporous stainless steel cap at the other end. The PSS tubes were cleaned using an ultrasonic bath with an alkaline solution to remove surface contaminants such as dirt and grease, and dipped in an isopropanol solution and dried at 393 K overnight.

A C-LDH layer was applied to modify the pore size on the PSS tube surface. After the C-LDH modification, the PSS was placed in air in a furnace at 873 K for 12 hours. The details of the procedure can be found in literature [8].

Prior to Pd layer deposition, the modified PSS tubes were activated by successive immersions into stannous chloride ( $\text{SnCl}_2$ ), palladium chloride ( $\text{PdCl}_2$ ), HCl and deionized water with three cycles. Pd was then deposited onto the PSS tube by an electroless plating method. We repeated the activation-deposition steps until the membrane identified, which was determined by helium flux undetectable at room temperature under a pressure difference of 100 kPa. The Pd deposition process was based on the authors' previous work [7].

Fig. 1 shows the cross-sectional SEM image (back scattering electron image (BEI)) of the Pd/C-LDH/PSS composite membrane. The BEI was collected to reveal compositional information. The image color gradually gets more concentrated from Pd, to C-LDH to porous stainless steel (PSS). The highest hydrogen permeance of the composite membrane was  $68.24 \text{ Nm}^3/\text{m}^2\text{-hr-atm}^{0.5}$  at 673 K, with a selectivity coefficient ( $\text{H}_2/\text{He}$ ) of 255 under a pressure difference of 4 atm.

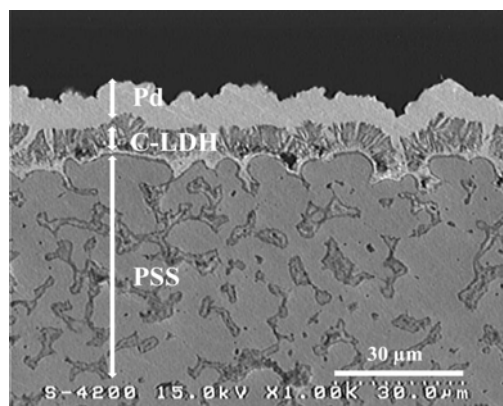


Fig. 1 Cross-sectional SEM image of the Pd/C-LDH/PSS composite membrane

### Acknowledgment

The authors would like to thank the financial support provided by Bureau of Energy, Ministry of Economic Affairs, Taiwan, ROC.

### References

- [1] J. Shu, B. P. A. Grandjean, S. Kaliaguine, *Can. J. Chem. Eng.*, **69** (1991) 1036.
- [2] S. Uemiya, N. Sato, H. Ando, Y. Kude, T. Matsuda, E. Kikuchi, *J. Membr. Sci.*, **56** (1991) 303.
- [3] S. Uemiya, T. Matsuda, E. Kikuchi, *J. Membr. Sci.*, **56** (1991) 315.
- [4] P. P. Mardilovich, Y. She, Y. H. Ma, M. H. Rei, *AIChE J.*, **44** (1998) 310.
- [5] Y. H. Ma, B. C. Akis, M. E. Ayturk, F. Guazzone, E. E. Engwall, I. P. Mardilovich, *Ind. Eng. Chem. Res.*, **43** (2004) 2936.
- [6] I. P. Mardilovich, E. Engwall, Y. H. Ma, *Desalination*, **144** (2002) 85.
- [7] Y. H. Chi, P. S. Yen, M. S. Jeng, S. T. Ko, T. C. Lee, *Int. J. Hydrogen Energy*, **35** (2010) 6303.
- [8] Z. L. Hsieh, M. C. Lin, J. Y. Uan, *J. Mater. Chem.*, **21** (2011) 1880.