CHAPTER V

CONCLUSIONS AND SUGGUSTIONS

In order to extend the lifespan of the palladium membrane reactor used for the production of hydrogen gas, this study tested the efficacies in preventing intermetallic diffusion of four Cr-based films prepared by (1) direct oxidation of the stainless steel support, (2) oxidized Cr-electroplating, (3) oxidized Cr-sputtering and (4) Cr-sputtering in nitrogen atmosphere.

The most suitable condition for the oxidation step was at 600°C for 6 hours. 800°C was the upper limit of the temperature to choose from. Much more Cr was in unstable forms than in the stable Cr_2O_3 form across the range of incubation time examined. At 450°C, formation of thin film that consists almost exclusively of Cr_2O_3 can be achieved but such incubation time gave very low Cr content to be useful. Increasing the temperature to 600°C gave a better compromise between the Cr content and Cr_2O_3 content.

The film formed via direct oxidation, in spite of being extremely thin and not continuous, was more effective than the far thicker CrN film. Therefore, it can be concluded with confidence that Cr_2O_3 film is better for this function. The Cr_2O_3 film prepared by either (2) or (3) was much thicker and continuous and performed better in shielding the palladium layer.

With respect to the preparation methods of the Cr_2O_3 film, direct oxidation is not suitable due to the limited availability of Cr in the stainless steel. External source of Cr overcomes this issue. Electroplating and sputtering permitted the formation of continuous film with a range of thicknesses and the performance of the resulting films were comparable. Sputtering produced films with finer grain and was better at least in term of precision of resulting layer thickness. However, it requires expensive equipment as opposed to the general equipment setting of electroplating. To single out which one is better in term of performance of the resulting barrier, further studies that deal seriously with the barriers' thickness must be conducted.

The intermetallic diffusion barriers of all forms influence the deposition process of palladium atoms resulting in clumped, uneven surfaces of varying degrees and the properties of the membrane are, although not explicitly examined, definitely altered. Therefore, further experiments are required to fill this gap of knowledge since the best intermetallic diffusion barrier in term of shielding the palladium layer could make the membrane itself the worst in term of selectivity for hydrogen gas and hence useless.

5.1 Further Works

5.1.1 Adjust the time and temperature for the oxidation step of the Cr layer for further fine-tuning its performance.

5.1.2 Prepare Cr_2O_3 layer by Cr-reactive sputtering in oxygen atmosphere and compare its diffusion preventing efficacy with other methods.

5.1.3 Test if the selectivity of the palladium layer is altered by the intermetallic diffusion barriers.