

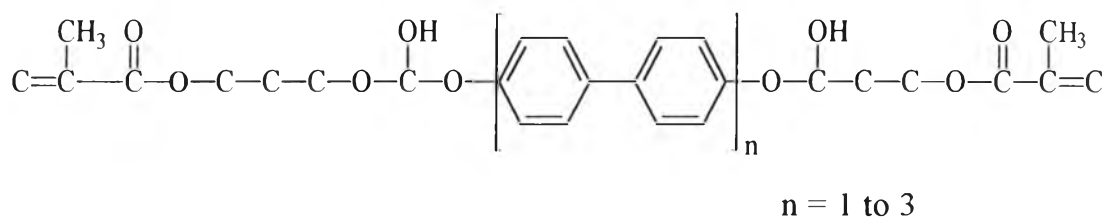
CHAPTER III EXPERIMENTAL

3.1. Materials and Coating Procedures

3.1.1 Materials

The quality of coating materials which the promising coating materials should be able to block chlorine, but allowed the passage of hydrogen to palladium wire was specified. Subsequently, the qualification was submit to coating material companies and asked for some suggestion about their materials for this purpose.

1. Derakane Resin (Epoxy vinyl ester resins)



Properties	Value
Density (g/cm ³)	1.045
Viscosity @ 25 °C (CPS)	350
Upper working temperature(°C)	99-104
Limiting oxygen index(%)	-
Tensile strength (MPa)	83.38

2. Lindron 31 (Acrylate Polymer) and Lindron 101 (Styrene Ethylhexylacrylate Copolymer)

Properties	Lindron 31	Lindron 101
Specific gravity @ 25/25 °C (water = 1.0)	0.99	0.87
Boiling point (°C)	157	157
Autoignition temperature (°C)	471	471
Flash point (°F)	100-115	100-115

3. Fluorodyn Caulk (made with Viton)

Properties	Value
Boiling point (°C)	77-82 @ 760 mmHg
Melting point (°C)	-87 @ 760 mmHg
Flash point (°C)	-9 to -5
Autoignition temperature(°C)	403-516
Vapor pressure	70-71 mmHg @20 °C 100 mmHg @ 25 °C

4. Boron Nitride Coating (Boron nitride , Water , Potassium Silicate , Acrylic, Diethylene Glycol Monobutyl Ether, and Graphite)

Boron Nitride (Active Ingredient)	12 %
Water (Carrier)	64 %
Potassium Silicate (Binder)	15 %
Acrylic	5 %
Diethylene Glycol Monobutyl Ether	3 %
Graphite	1 %

Properties	Value
Specific gravity	1.2 g/cc
Viscosity	~350
Boiling point ($^{\circ}$ C)	100
Vapor pressure (mmHg)	~10-20

3.1.2 Coating Procedures

1. Derakane Resin was mixed with MEKP as catalyst to form the polymer chain and coated on Teflon. It was allowed to dry for at least four days.
2. Lindron 31 and Lindron 101 were coated on Teflon and allowed to dry for two days.

3. Fluorodyn Caulk was coated on Teflon and allowed to dry for 48 hours or longer depending on the thickness. The sample was cured in the oven at 120 °C for 1 hour.
4. The boron nitride hardcoat coating procedure is similar to that for Lindron 31 or 101.

3.2. Apparatus

The experimental apparatus was assembled in two stages, as follows:

3.2.1 Apparatus for Determining H₂ Permeability

The apparatus for determining hydrogen permeability was shown in Figure 3.1. The apparatus was composed of two compartments. The first one was a test cell, which was a standard ASTM test cell purchased from A.A. Pesce Glass Company. It consisted of two sides; the first side was employed as the challenge side and the other side was the collection side. Between the two sides was the test material. The gaseous mixture which contained the gas to be tested was referred to as challenge gas flows continuously through the challenge side of the test cell. The collection gas flowed continuously through the collection side which carried any challenge gas that might have permeated through the test material. In this work, the collection gas used was Argon (Ar). The hydrogen concentration in the collection gas was determined with a gas chromatograph (Varian 3600).

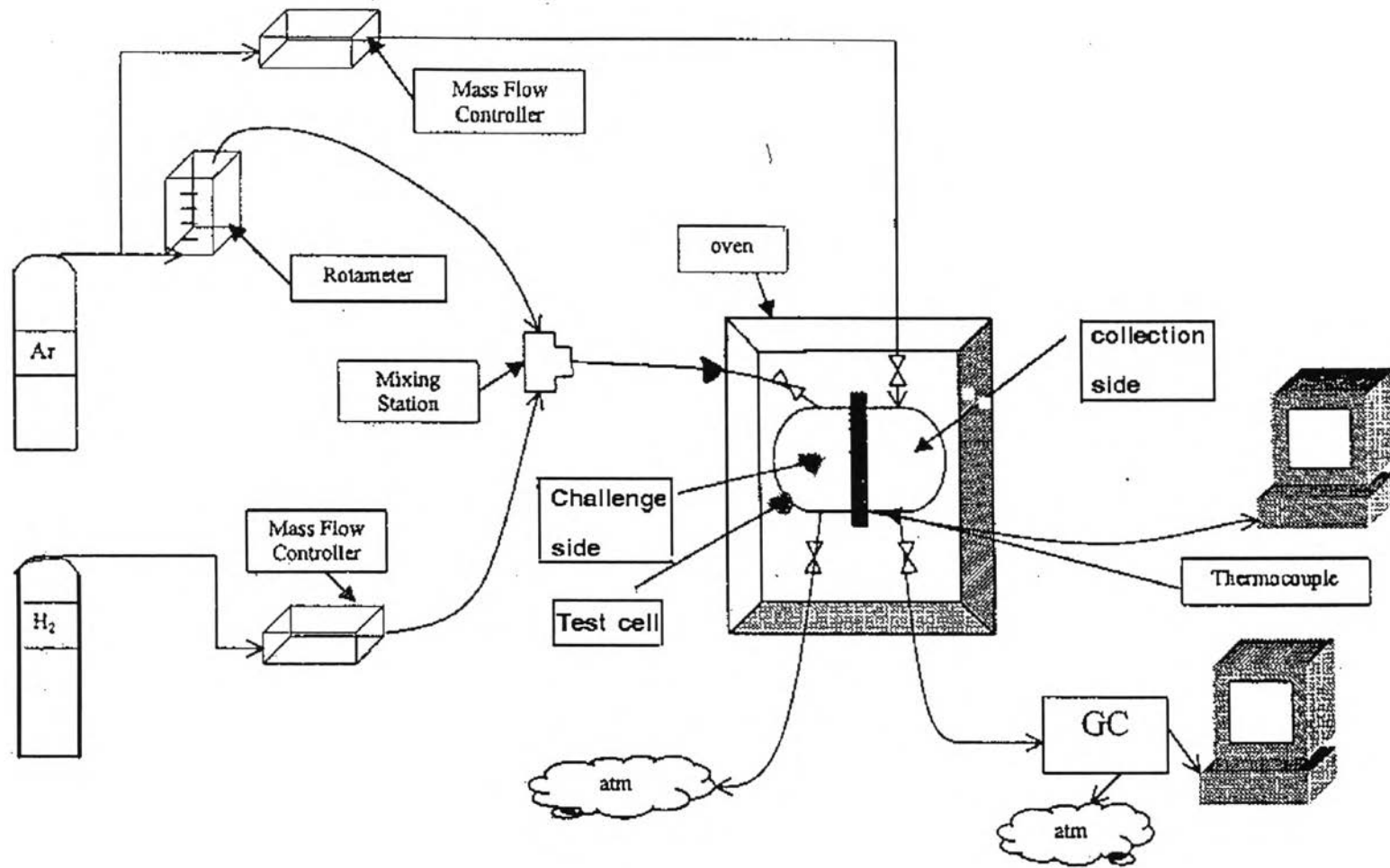


Figure 3.1 Apparatus for determination of hydrogen permeability through materials.

3.2.2 Apparatus for Determining Cl₂ Permeability

The apparatus for determining chlorine permeability was shown in Figure 3.2.

The test cell described previously in section 3.2.1. was also used for determining Cl₂ permeability. Detector tubes were used to detect and measure the concentration of chlorine in the collection gas. The method for using detector tubes in conjunction with ASTM F739-91 was described by Sarnier and Henry (1989). The color-forming reaction took place between chlorine gas and the chemicals in the detector tube. This allowed the measurement of the chlorine concentration as a function of time. The extent of tube material changing color (the stain length) gave an indication of the concentration of the collection gas. For the Dragger tube, the detection limit was 0-10 ppm .

Other standard equipment used in this experiment included mass flow controllers and a rotameter for the gas flow control, an oven to allow the tests to be conducted at elevated temperatures, thermocouples and an associated data acquisition system. Bubblers placed in a hot water bath (t ~ 70°C) were used to saturate the challenge gas stream with water at a controlled temperature (for humidified gas mixtures).

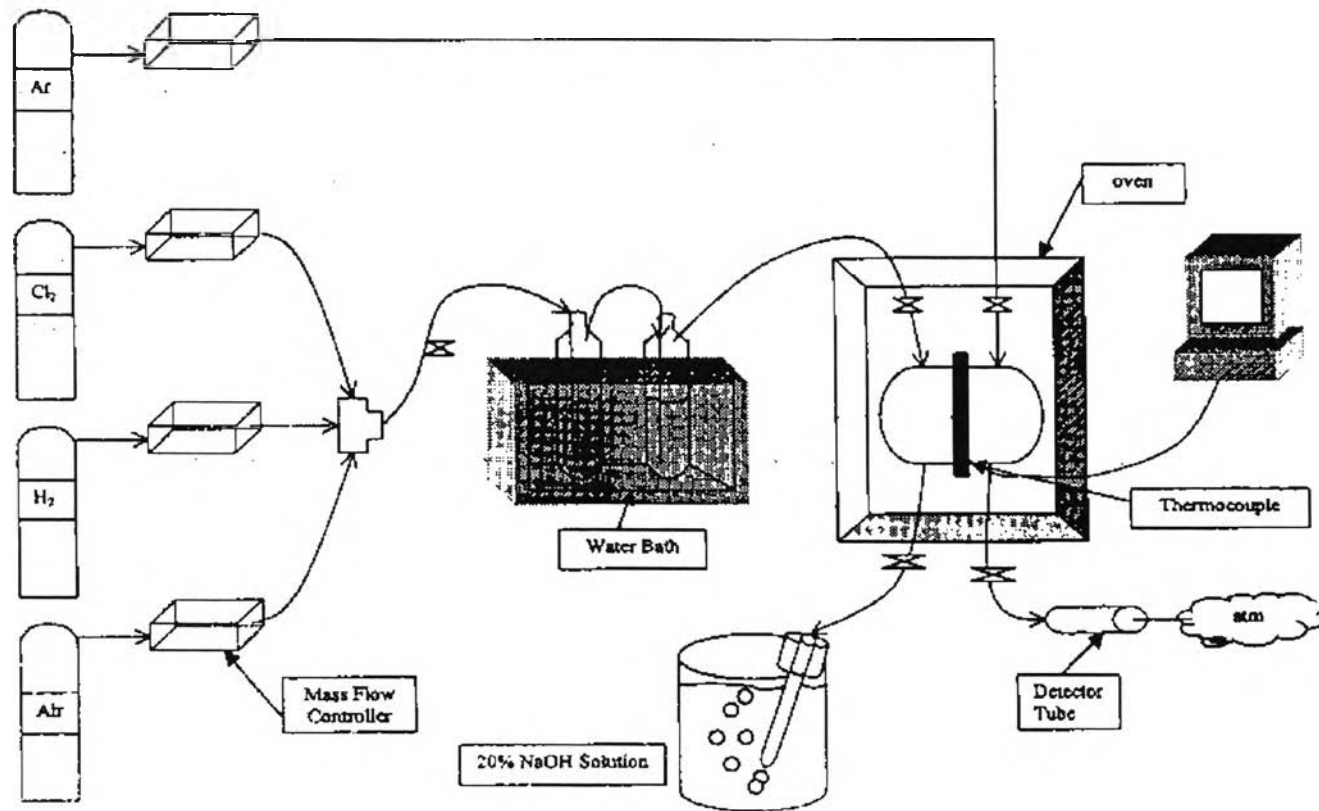


Figure 3.2 Apparatus for determination of chlorine permeability through

3.3. Methodology

3.3.1 Test Procedure with Dry Hydrogen Gas Mixtures

For gaseous chemical permeant, the procedure was as follows;

1. The test cell was set up with H₂/Ar mixture as the challenge gas.

Gas Mixture: 75 % Ar

25 % H₂

Collection Gas: Ar

2. The tested material was mounted in the test cell and assembled as Figure 3.1 shown hydrogen permeability coefficient.
3. The test cell was installed in constant temperature oven, held at 80 °C constant temperature.
4. The collection side was set up to include the Dragger tube (for Cl₂ permeability) or feed to GC (for H₂ permeability).
5. A collection gas was passed into a collection chamber continuously at the flow rate of 10 cm³ /min.
6. Sampling was initiated and analyzed of the collection gas at a frequency of 10 minutes by using GC (Varian 3600).
7. The base line values for subsequent analytical tests were established.
8. The challenge gas was allowed to flow into challenge chamber.
9. The challenge gas was passed at an equivalent of 5 chamber volumes (volume of chamber = 100 cm³) through the challenge chamber in 1 minute, as determined by the flow meter placed in the outlet stream of the chamber.
10. The timing of the test was started challenge gas flow rate to reduce 10 cm³ /min.

11. The concentration of the challenge gas (H_2) in the collection gas and the time which had elapsed between the initial gas contact with the tested material and withdrawal of the challenge gas were recorded.
12. The test was continued until constant concentration of H_2 in collection stream was achieved.

Calculation of the permeation rate

For Systems using a continuous flow fresh collection gas, the concentration of the permeant (H_2) in the collection gas at any time, t , was directly proportional to the permeation rate. Concentration was converted to permeation rate as follows:

$$P_i(t_i) = C \left[\frac{F}{A} \right] \quad (3.1)$$

where $P_i(t_i)$ = the permeation rate at any time t_i ($\text{cm}^3 H_2 \text{ cm}^{-2} \text{ min}^{-1}$)

C_i = the concentration of H_2 in the collection gas at time t_i ,
(ppm)

F = the flow rate of the fresh collection gas through the test
cell ($\text{cm}^3 \text{ Ar /min}$)

A = the area of the tested material contacted (cm^2)

3.3.2 Test for Materials with Dry Cl₂ Gas

The same procedure described in section 3.3.1 was followed. Test materials were tested by based on results from procedure described in section 3.3.1.

Gas Mixture : 99 % Cl₂
1 % H₂

3.3.3. Test of Materials with Water Saturated Cl₂ Gas

The same procedure described in 3.3.1 was followed. Test materials were tested by based on results from procedure described in section 3.3.1 and 3.3.2.

Gas Mixture : 99 % Cl₂
1 % H₂
Saturated mixture at 74 °C

3.3.4. Test of Materials with Representative Gas Stream

The representative gas stream was to be monitored for hydrogen typically contain 95 % Cl₂, 0.5-1.0 % H₂ with air making up the balance.

The same procedure as described in section 3.3.1 was followed. Test materials were tested by based on results from procedure described in section 3.3.1 and 3.3.2.

Gas Mixture : 95 % Cl₂
1 % H₂
4 % Ar
Saturated mixture at 74 °C