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A review on types, fabrication and support material of hydrogen separation membrane

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ABSTRACT

In recent decades, hydrogen has gained renewed and growing interest all over the world as a high-quality and renewable energy carrier, mostly due to advances in fuel cells and environmental issues including climate change. This review explains the hydrogen separation membranes with various fabrication methods as well as different membrane supports, and it also covers a different type of membrane for hydrogen separation. In recent, the researcher placed an interest in the study of the hydrogen selectivity and permeability of the membranes. It concluded that the composite palladium membrane developed by electroless pore plating fabrication technique on porous stainless steel has found a promising application for hydrogen-selective membranes. The use of the palladium membranes in industries has some associated problems such as high cost of pure Pd-based membrane, membrane fouling, and durability of the membrane.

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1. Introduction

The depletion of fossil fuel day-by-day increases the demand for alternative and clean energy to fulfill the energy requirements [1]. According to the EIA, fuel demand increases very rapidly in the late 2020s and it increases 0.1 mb/d each year. Increasing the automobile industry, climate change and air pollution very drastically increases due to the combustion of fuel [2]. As a result, alternative energy is needed to full fill world energy demand and environmentally friendly. Many renewable sources such as wind energy, geothermal, solar energy, and biofuels presently the solutions but these are not sufficient to meet the required energy demands [2]. Hydrogen based energy has been sustainable and effective fuel for fuel cells in the automobile industry [3]. To meet economical demands, hydrogen separation and purification have been required. Various technologies available for purification and extraction of hydrogen from other gasses such as wet scrubbing, dry scrubbing, and pressure swing adsorption, membrane separation, cryogenic distillation [4]. Among them, membrane technology has been found appealing due to the advantages like flexibility in operation, simplicity, energy efficiency, compactness, small foot-

print, eco-friendly, less operating cost and easy integration with established industrial processes [5]. Performance of membrane has been evaluated by its selectivity, thickness, thermal stability and permeation rate.

Several membrane materials, like polymer membrane (glassy and rubbery) and their composites, hydride membrane (metal-based membrane, silicon-based membrane), metal alloys, etc. have been used to prepare hydrogen-selective membrane [6]. In metal-based membranes, various metals used for preparing hydrogen-selective membranes such as vanadium, niobium, and tantalum but among them palladium has been an attractive material that provides high selectivity towards the hydrogen enabling the production of hydrogen [6]. Furthermore, the hydrogen separation process using dense palladium membrane has focused upon hydrogen atom being selectively transported through the dense metal sheet. Additionally, such type of membranes has been highly oxidation-resistant and had good thermal stability (up to ~550 °C) enabling their implementations at elevated temperatures and the essence of the permeation process that involves the higher value of hydrogen solubility and diffusivity into palladium/palladium alloy [7]. Such properties provide significant benefits of hydrogen (H_2) purification against other alternatives like polymeric membrane and its composite, zeolite membrane, other metallic membranes, carbon membrane. Polymeric membrane gives minimal

thermal resistance, permeability and selectivity, zeolite membrane has the main drawback as stated: *i*) difficulties occur to prevent shortcomings at a reasonable cost*, ii*) low selectivity. Other metallic membranes have a limitation that poor ability to integrate proper hydrogen (H_2) solubility with diffusion [7]. The main drawback of carbon membranes has been low selectivity. Hence, it can be summarised that metal based (palladium/palladium alloy) membranes show a relatively great potential towards the hydrogen selectivity with high permeate flux.

This work reviews about different fabrication methods, types of membrane and support used during the fabrication of hydrogen-selective membranes.

2. Type of membrane

2.1. Polymeric membrane

Polymeric membrane performs a major role in the separation of gases. These membranes are an Appling option for hydrogen separation due to their advantages such as minimal cost, higher energy efficiency, ease of use and lower ecological impact as compared to traditional separation techniques. High permeability, mechanical stability, high thermal, and high selectivity are the ideal properties for the selection of polymeric membrane material. The polymers that are usually highly permeable display low selectivity and vice versa most common polymers use polysulfone, polyimide, polyamide, etc. Gas separation properties of polymeric membrane strongly associated with material microstructure. The size distribution and free volume hole concentration have a significant impact on permeation property of membrane [8].

Polymer membranes could be categorized into a polymeric glass and rubbery membranes. When the operating temperature is lower than the glass transition temperature, the polymer act as a rigid glass. Under this situation, friction free volume decreases within polymer structure and large-scale polymer backbone cooperative activity was limited due to limited space. When an amorphous polymer was held above its transition temperature, the polymer was under a rubbery state.

The Glassy polymer was more practicable to commercialized purpose as compare to rubbery polymer membranes, because of their good gas selectivity and excellent mechanical property. The Commonly used glassy membrane was polysulphones, polycarbonates, and polyimides. In a glassy polymer, small size gas molecules (H_2) more permeable than large size molecules (CO_2, N_2). Therefore, these membranes suitable for hydrogen separation. Nonetheless, the polymeric membrane application is constrained by the relationship between permeability and selectivity. To enhance the performance of polymeric membrane a significant research effort has focused on adding inorganic material such as zeolite, silica, and carbon molecular sieves in the polymer. This type of membrane called mixed matrix membranes (MMMs). Sharma et al. create a highly hydrogen permeable and selective membrane by adding carbon nanotube within a polycarbonate (PC) matrix [3]. Khan et al., develop a polyether sulfone (PES) embed with Zeolite 5A [9]. Rezakazemi et al., investigated polydimethylsiloxane with zeolite nanoparticle membrane to improve the permeability and selectivity of hydrogen [10]. Cacho-Bailo et al. prepared a hydrogen separation membrane by using polysulfone and ZIF-8 [11]. Feng et al. investigated the composite membrane of poly(dimethyl siloxane) coated polysulfone hollow fiber for permselectivity to H_2/N_2 [12].

Metallic membranes are generally dense in nature which offers hydrogen permeability through proton and electrons. Metallic membrane offers high selectivity, High diffusivity and good thermal stability at elevated temperature [7]. Dense structure prohibits

the passage of large gases molecules due to this selectivity of H_2 molecule to the other gas (CO_2, N_2, CH_4, etc.) were very high. High selectivity denotes to ultra-purity of hydrogen. This is the main advantage of the metallic base membrane over other materials.

2.2. Metallic membrane

Metallic membranes are generally dense nature which offers hydrogen permeability through proton and electrons. Metallic membrane offers high selectivity, High diffusivity and good thermal stability at elevated temperature [13]. Dense structure prohibits the passage of large gases molecules due to this selectivity of H_2 molecule to the other gas (CO_2, N_2, CH_4, etc.) were very high. High selectivity denotes to ultra-purity of hydrogen. This is the main advantage of the metallic base membrane over other materials.

2.2.1. Non-palladium membranes

The biggest barrier to use palladium (Pd) membrane technology for broader applications is the high cost and volatility of palladium prices [1]. Hence, required to the development of membranes with less expensive metals. Group 5 metals such as tantalum, niobium (Nb), vanadium (V) with a body-centric cubic (BCC) structure promising alternative material to Pd due to their lower price and high permeability of hydrogen. Nevertheless, the palladium film needed to permeation of hydrogen. When the membrane was handled at an interdiffusion between Pd and V at more than 400 °C, the membranes start to degrade. The main issue with group V metals for membrane materials is their excessive solubility to hydrogen, which makes the membranes susceptible to extreme hydrogen embrittlement that can lead to pinholes and cracks being formed.

2.2.2. Palladium (Pd) membranes

Palladium membranes have recently gained great exposure due to its excellent permeability, high resistance to hydrogen flow and auto-catalyzing of the hydrogen dissociation. The significant aspect of Pd membrane has been its outstanding hydrogen embitterment tolerance and hydrogen recombining catalytic capability [13,14]. Pd unique existence makes the metal suitable for the purpose of separating hydrogen in the respect that it had the potential to disassociate molecular hydrogen into such a monoatomic shape ready for rapid diffusion by its lattice [16]. In addition, this metal can consume about 600 times its own volume for hydrogen while preserving its physical characteristics and structural components; a feature that Thomas Graham invented in 1866. Based on the metal content, pure Pd membrane exhibits phase change of α to β at pressure and temperature below 2 MPa and 300 °C [17]. This transformation contributes towards the lattice strain and hence distorts the metal lattice after a few periods as the β-phase lattice constant is 3% greater than for the α-phase lattice constant [18]. Palladium alloys, specifically silver alloys, minimize the critical temperature for such a defect, and increasing the permeability of hydrogen. Many technologies for the development of hydrogen use palladium-alloy membranes with stainless steel support [19]. The highest hydrogen permeability needs 23 wt% silver alloys [20].

Niobium and tantalum metals have 10 time's higher permeability as well as lower cost than palladium, but these metals have not catalytic activity for hydrogen dissociation and recombination. Also, inadequate resistance toward oxidation, hence, have oxide deposition on their surface. These limitations preclude the use of Niobium (Nb) and tantalum (Ta) as a hydrogen separation membrane operation [21].

Various Pd membranes prepared by the researcher. In the study of Jun & Lee, fabricated the Pd membrane by using CVD technique on mesoporous supports of Al_2O_3 and nickel-stainless steel (Ni-SUS). These membranes represent a selectivity 780 for on Al_2O_3

support and 100–200 on Ni-SUS support for H_2/N_2. Wei-Hsin Chen developed a Pd membrane and shows an effect of vacuum pressure (15 kPa–53 kPa) on the permeate side of the membrane and this study concludes that less vacuum pressure shows a significant effect on hydrogen permeation. A mechanism of hydrogen permeation through Pd-based films comprise various step such as *i*) on a palladium sheet, the molecule of hydrogen absorbs and then disassociates into the atoms of hydrogen. *ii*) The atoms of hydrogen then migrate through the Pd membrane whereas the electrons interact on an adjacent side with the metal lattice *iii*) Hydrogen atoms left the lattice structure and recombine to surface until they desorb as molecules of hydrogen. The palladium-based membrane frameworks are commonly classified into two types: unsupported palladium membranes, and supported palladium membranes, which also include pure Pd and Pd alloy materials. For unsupported membranes, Paglieri and Way developed a Pd/Pd alloy membrane by using tubular geometry having 20–100 µm thickness in the United States and the former Soviet Union [30,21]. These kinds of membranes produced ultra-high purity H_2 for use in the manufacturing of semiconductors or for the extraction of H_2 isotopes. Unfortunately, the high price of Pd material restricts economic practicability relative to other separation techniques [22]. However, the method of evaluating the material characteristic of pure Pd/Pd alloys remains important for smaller-scale production and for research purposes. These types of membrane associated some issues such as high cost, membrane fouling, the durability of the membrane due to these issues it not economical for industrial purposes. Table 1 shows a supported membrane prepared by many researchers. The Supported membranes could be developed with a much thin layer of palladium resulting in lower cost, so substantial efforts were made to improve preparation methods using supports. Supported palladium membranes investigated in this reviewed can be divided into three categories depending on the materials used: Vycor glass, ceramics, and stainless steel. Ceramic support is one of the non-metallic substrates that can be moulded into a variety of forms with 5–200 nm of controllable pore sizes. Alumina is the most commonly used ceramic materials for the pro-

duction of Pd composite membranes, since it is widely available in different formulations, provides better mechanical and thermal stability and it can be modified using intermediate layers [23]. Surface roughness in alumina support ensures maintained good mechanical strength of Pd alloys membrane and prevents the cracks creates during the operation [24]. Abate et al., developed Pd –Ag alloy membranes direct on an alumina substrate with an average pore size of 70 nm and several microns of surface roughness by using the ELP technique at 623 K. The permeability of hydrogen obtain was 1.2×10^{-6} molm^{-2} s^{-1} Pa^{-1} and selectivity (H_2/N_2) was 450.

Porous metal support one of the conductive substrates having an average pore size of 0.2–100 µm that can be moulded a different geometry. Alumina, Borosilicate glass, Steel, Stainless steel commonly used metal support for palladium membranes because of thermal expansion of these metals like the Pad metal. These are conventionally developed by powder sintering as well as electrochemical deposition and strong applicants for substances that support them. Raw surfaces with a large number of pores as well as the non-uniform distribution of pore size is responsible for membrane defect and creation of pinholes on the surface of palladium membranes [16]. Due to these reasons, for developing defect-free membrane on metal support creates a thick film as compared to ceramic support. Using metal support, needs a three-time thicker layer then pore diameter [18]. Ceramic support (alumina) gives excellent strength and creates thin film due to this permeability and selectivity of membrane improves. But in the case of ELP-PP metallic support (PSS) offers excellent selectivity of hydrogen towards the other gasses because the deposition of material on the pores due to this thickness of membrane reduces (Table 1).

3. Fabrication methods for hydrogen separation membrane

3.1. Chemical vapour deposition (CVD)

In CVD, produce high purity and excellent performance solid material. In this technique, the substrate is exposed to volatile pre-

Table 1
Palladium membrane: support, technique.

Material	Support	Thickness (µm)	Hydrogen permeability (10^{-8} mol·m^{-1}·s^{-1}·Pa$^{-0.5}$)	Selectivity (H_2/N_2)	Technique	Temperature (°C)	References
Pd/Al$_2$O$_3$	γ-Al$_2$O$_3$		1000 GPU	23	ALP	220	[13]
Pd	Ceramic	14	0.025–0.036	–	ELP	>350	[25]
Pd	PSS	20	13	170	ELP-PP	400	[14]
Pd/OXI-doped CeO$_2$	PSS	9.1	0.0000446–0.0000639	>10,000	ELP-PP	350–450	[32]
Pd	Ceramic	8	0.58-0.85	–	ELP-PP	>350	[25]
Pd	Vycor	15	22	7	ELP	200	[14]
Pd	Vycor	13	0.96	High	ELP	500	[33]
Pd	Alumina	1	3.34	5000	CVD	573	[16]
Pd	Alumina	6	0.67	–	ELP	673	[21]
Pd$_{97}$Cu$_7$	Alumina	–	–	>75	ELP	450	[17]
PdCu$_6$	Vycor	12	0.27	–	ELP	400	[6]
PdCu$_6$	Vycor	19	0.204	High	ELP	400	[33]
PdCu$_{19}$	Al$_2$O$_3$	11.6	1.73	–	ELP	500	[3]
PdCu$_{16}$	PSS/ZrO$_2$	5	0.21	High	ELP	400	[6]
Pd–Ni alloy	Alumina	1	2.06	317	CVD	450	[13]
PdAg$_{23}$	Alumina	12	1.2	450	ELP	350	[26]
PdAg$_{30}$Ru$_{15}$	Alumina	15	7.33	–	ELP	350	[21],
PdAg$_{30}$	Alumina	13	2	–	ELP	400	[21]
Pd/Sil-1	Alumina	5	1.8	1300	ELP	500	[34]
Pd	Alumina	2	3.34	5000	CVD	300	[35]
Pd/polymer	Alumina	5	3.33	∞	ELP	450	[36]
Pd/YSZ	PSS	11	0.97	–	ELP	500	[37]
Pd/WO$_3$	PSS	12	0.21	10.000	ELP	450	[38]
Pd$_{77}$Ag$_{23}$	Alumina	12	1.2	450	ELP	–	[23]
Pd	Alumina	1	2.06	780	CVD	450	[39]
Pd/TiO$_2$/Al$_2$O$_3$	PSS	4	8.78×10^8	1795	ELP	450	[18]

Table 2
Advantages and disadvantages of hydrogen separation membrane development techniques.

Technique	Advantages	Disadvantages	References
CVD	• Less wastage of deposited material • Deposited high purity solid material	• Low conformality • Low Uniformity • Required high temperature • Difficult to maintain constant temperature • Generate volatile by product • High capital and operating cost • Only inorganic material used	[1,40,15]
PVD	• Environmental friendly as compare to CVD • Inorganic and some organic material used.	• Rate of film deposition quite slow • Large amount of heat and vacuum required for deposition • High capital cost	[40]
ELP	• High uniformity • Excellent conformality • Very thick(several micron membrane can be prepared • low energy requirements • low operating cost	• Complex and indirectTechnique(at least two step required) • High manufacturing defect	[1,41]
ALD	• Good uniformity • Excellent conformality • Simple and direct technique • Membrane thickness quite thick • Thickness control (nanometre scale)	• High energy waste. • Time required for deposition of chemical on the surface. • High material waste	[1,26,27]
ELP-PP	• Possible to cover complex geometry • Ultra-thin membrane • High selectivity of hydrogen over other gasses (N_2, CO_2)	• High manufacturing defect	[17,31]

cursor which reacts and decomposes on the substrate surface at the same time co-deposition of material also occurs. The chemical reaction proceeds onto the hot surface due to this thin film deposit on the surface as well as chemical by a product that exhausted out from the chamber along with unreacted precursor gas. The advantage of CVD that the material deposits only in the heated surface due to this wastage of deposition material was less. Beside of this, it has some limitation (*i*) it required high temperature which degrade the property of material, (*ii*) to maintain constant temperature also very challenging, (*iii*) produce volatile by-product removed from the chamber in the form of gas [8] which is highly corrosive, toxic and explosive (*iv*) high capital and operating cost, (*v*) bad homogeneity of coating (*vi*) only inorganic material used (*vii*) property of the precursor.

3.2. Physical vapour deposition (PVD)

It is a vacuum deposition method which is used to deposit thin film and coating on the surface. This technique comprises a four step (*i*) evaporation, (*ii*) transportation, (*iii*) reaction, and (*iv*) deposition. This material goes to the condense phase to vapour phase and then back to form a thin film on the surface [8]. PVD has a number of advantages such as deposit material can be good properties as compare to substrate material, it more environmentally friendly as compare to CVD because it does not emit toxic gases, almost all type of inorganic material and some organic material used, PVD safer then CVD because no by-product gases remove in this process. But it has some disadvantages such as (*i*) high capital cost (*ii*) rate of film deposition quite slow, (*iii*) for deposition of solid material on the surface large amount of heat and vacuum required.

3.3. Electroless plating (ELP)

ELP based on autocatalysis homogenous deposition of metal on the target surface [25]. Thin metal film deposits on the substrate without using any external source such as electricity, electrodes by cause of this reduces the operating cost. ELP have the ability to create a uniform film on the complex structures [1,24]. Besides all advantages, during the manufacturing process of membrane

number of rejections is too high due to the presence of some defects significantly increases the overall cost of the membrane. So it needs an alternative technique to address this problem. Due to the auto catalyst nature of the ELP process. In the last decade, for this reason, ELP has been considering a potential technique, as it offers better uniformity of the films as compared to CVD and PVD counterparts [1].

3.4. Atomic layer deposition (ALD)

ALP process is CVD derive technique which allows preparing thin film on the large surface area with a high aspect ratio [1]. In this the chemical precursors introduced on the surface of the substrate where they direct chemically react to the surface due to self-saturating reaction with the surface group and form a self-limited layer by layer ultrathin films [26]. This technique comprised steps of adsorption and oxidation of metal precursors. This technique has various advantages such as Good uniformity, Excellent conformity, easily control membrane thickness (nanometre-scale), simple and direct technique [1,26]. Besides these advantages, this process not economically optimum due to having some reasons such as high material wastage, high energy requirement, intensive nature, nanoparticle emission [27]. This process also very time consuming because the time required for chemical reaction takes too much time (Table 2). Weber et al., reported for the first working prototype to developing a hydrogen selective membrane using ALD technique. In this work Pd in the pore of the y-Al_2O_3 top layer covering the inner side of the ceramic tubular support.

3.5. Electroless pore-plating (ELP-PP)

ELP-PP approach seeks to integrate the palladium within the pores, retaining the key advantages of ELP while at the same time offering specific advantages about the existing technology. The ELP process, firstly particle integrates on the surface of the membrane. They captivate more particles, which are loaded together until they built a bridge that progressively developed. After that, the palladium nanoparticles deposit onto the bridge until the whole pores covered due to this film thickness increases [28]. So, Pd-membrane thickness totally depends on the pore size of the sup-

port. The thickness of the file approximates three-time bigger then pores the size of the support surface [29]. However, the deposition of palladium nanoparticles on the surface area of the membrane together with the auto catalyst nature of the ELP process creates difficulties to make a thin film [13,30]. Trying to address this problem, the ELP-PP technique investigated to reduce generated defects on the membrane. This technique aims to, incorporates palladium nanoparticles inside the pores of support material and prepared a completely dense membrane [25]. Permeability and selectivity of hydrogen also improve (Table 2).

ELP-PP has various advantages over ELP (*i*) metallic material has only been inserted within pores that bind the retentive as well as the permeate side, thereby preventing or reducing the penetration of the metal into fully dense surfaces, thereby saving palladium, (*ii*) only coating solution and reducing agent are prevented, and the chemical reaction to expand the metal coating is thus allowed to stop. It means preventing an excessive increase in the metal thickness in whole dense area and maintaining reproducibility against any defect in the support, (*iii*) it is possible to obtain good thermal resistance to cycle of heating. It means that at elevated temperatures the creation of cracks and pinholes resulting from deformation and contraction of stable coating in the existence of hydrogen could be minimized. In addition, metal embedded within the pores of an optimal pore coating layer that partly creates a discontinuous layer that partially compensates for potential mechanisms [14].

Much researcher work on the ELP-PP technique for preparing highly hydrogen selective membrane. martinez-diaz et al investigates the use of Pd nuclei-doped CeO_2 particles to prepare the intermediate layer on porous support by using ELP-PP process rather than pure CeO_2 to improve the hydrogen permeability and selectivity of the membrane. Due to the use of uniformly doped ceria with Pd nuclei promote the reaction within the pores between both the Pd source and the hydrazine baths throughout the plating stage. Hence, it potentially affect both final layer thickness and H_2 permeability through the composite membrane. Tosto et al. investigated palladium-based membranes were prepared using the ELP-PP and consequently analyzed for water gas shift experiment in a fluidized bed membrane reactor and results show high hydrogen selectivity as well as high permeability.

4. Conclusion

This review comprises different fabrication methods of hydrogen separation membrane systems are ELP-PP, ALD, CVD, PVD, and ELP. The industry's future of Pd membranes seems very promising since many Pd-based membranes demonstrated strong thermally and mechanically stable with excellent gas properties. The membranes divided into two classes, supported membranes or unsupported. Unsupported membranes are typically dense to give physical strength, and due to high gas permeation resistance for dense materials, they exhibit superior gas separation characteristics as compared to supported membranes. Therefore, they are relevant for assessing the intrinsic properties of Pd membranes. Supported membranes developed mostly CVD, ELD and ELP-PP technique for requiring thin selective Pd layers with good H_2 selectivity because support provides structural strength. However, the uses of these methods for functional applications are limited. The CVD process needs volatile and thermally stable Pd precursors but this is restrictive because commercially available Pd precursors are costly or have very poor vapour pressures. ELP process needs conductive support and this is troubling because of this surface roughness as well as the non-uniform distribution of the available metal supports due to these limitations the thickness of the deposited film increases. For minimizing these drawbacks ELP-PP tech-

nique invented in this material deposited within pores due to this thickness of film reduces. In the ELP-PP technique, PSS offers good strength to the palladium membrane.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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