

Palladium-Based Alloy Membranes for Separation of High Purity Hydrogen from Hydrogen-Containing Gas Mixtures

doi:10.1595/147106711X540346

<http://www.platinummetalsreview.com/>

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Palladium-based alloys with yttrium, copper, ruthenium or indium additions were investigated. Their specific hydrogen permeability, strength, linear thermal expansion in hydrogen and corrosion resistance in a number of gas media were determined. This allowed effective membrane elements to be developed using membranes made from these alloys, which are used for the separation of high purity hydrogen from hydrogen-containing gas mixtures. Membrane elements with 93.5 wt% palladium-6 wt% indium-0.5 wt% ruthenium alloy membranes were developed by the authors' research group, and their technical characteristics are described.

Introduction

Hydrogen is an attractive alternative fuel to meet ever-growing energy requirements while reducing the reliance on oil and gas, with their attendant environmental and resource problems. By contrast, hydrogen is potentially an inexhaustible, clean and environmentally sustainable resource. One way to produce high purity hydrogen is by separation from hydrogen-containing gas mixtures produced by catalytic or chemical manufacturing processes.

Hydrogen production volumes vary widely, depending on the end use application. These can range from several cubic metres per hour ($\text{m}^3 \text{h}^{-1}$) for micro- and nanoelectronics and portable energy sources such as fuel cells, to tens or hundreds of $\text{m}^3 \text{h}^{-1}$ for transport and small stationary energy sources, tens of thousands of $\text{m}^3 \text{h}^{-1}$ for the chemical industry and metallurgical applications, and hundreds of thousands of $\text{m}^3 \text{h}^{-1}$ for large stationary energy installations and hydrogen production plants.

Recent progress in hydrogen power engineering means that the demand for high purity hydrogen

(>99.9999 vol%) is increasing rapidly. The pressure swing adsorption method is most frequently used in the USA (1). However, this method is energy intensive as high volumes of adsorbents are used, and some of the separated hydrogen is used for adsorbent regeneration, reducing the efficiency of the process for hydrogen purification. Therefore, the best method for the production of high purity hydrogen is currently by separation *via* selective diffusion through metallic membranes made from palladium-based alloys whose permeability for other gases is infinitely small.

Hydrogen separation installations using palladium-based alloy membranes are more expensive in terms of capital expenditure than installations using polymeric membranes. However, palladium-based membranes have a number of advantages, namely that high purity hydrogen can be prepared in a single step and that the process can be carried out at high temperatures. This allows the hydrogen separation process to be combined with the production of hydrogen-containing gas *via* the direct incorporation of membranes into membrane catalytic reactors (2–4).

There has been growing interest in the development of palladium-based alloy membranes for hydrogen separation. Analysis of published scientific and patent literature shows a large increase in the number of patent and non-patent publications related to palladium membranes in recent decades (see Figure 1). However, the analysis also shows that the commercial preparation of inexpensive high purity hydrogen remains problematic due to the lack of efficient palladium-based membrane alloys, of systematic data on the properties of membranes, and of original designs of membrane elements and modules.

This article reviews the work undertaken by the authors' research group to develop high-capacity palladium-based membrane alloys and to design efficient membrane elements and modules based on these alloys for the commercial production of high purity hydrogen.

Palladium-Based Alloy Membranes

Efficient hydrogen separation membrane alloys must have high hydrogen permeability, low expansion when saturated with hydrogen, good corrosion

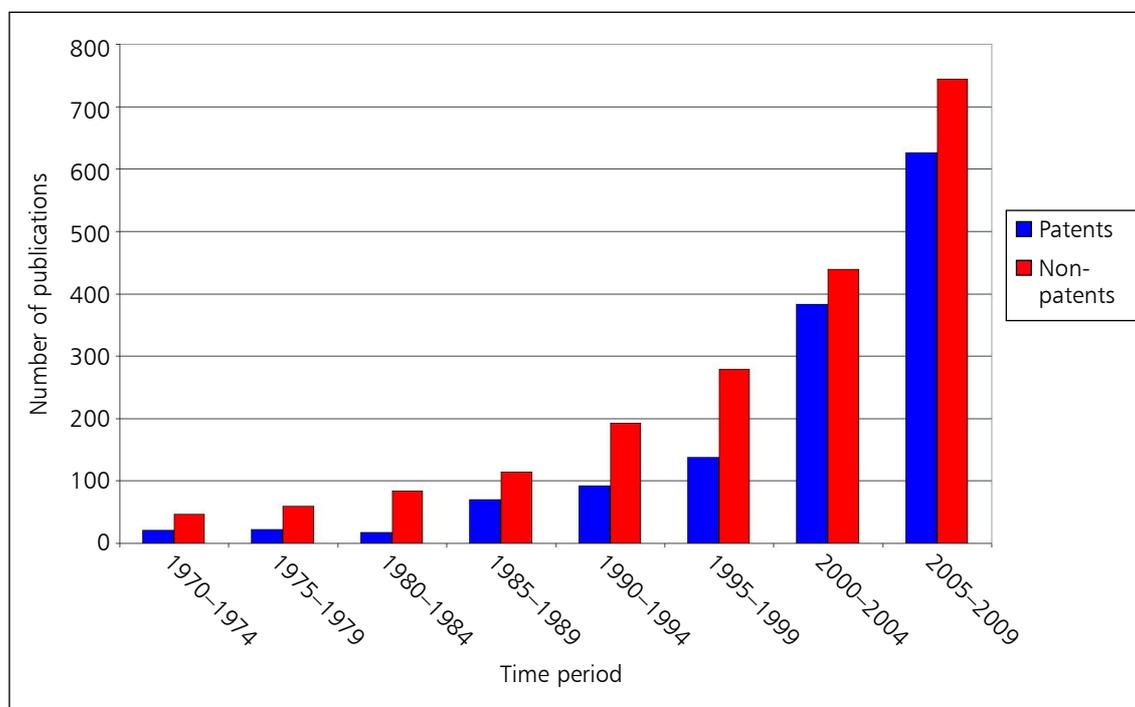


Fig. 1. Global trends in the number of publications in the patent and non-patent scientific literature relating to palladium membranes for hydrogen separation between 1970–2009

resistance and high plasticity and strength during operation at temperatures of 300–700°C. The main problems with membrane reliability are related to their corrosion resistance, changes in their hydrogen permeability and structural changes that occur during operation.

The search for effective alloys was limited to palladium-based solid solutions as these are the only materials which can demonstrate all of the required properties. Palladium alloys of high plasticity are required as they are used in the form of micron-sized foils and tubes. Palladium forms a wide range of solid solutions with many metals. Such palladium-based solid solutions are formed with 10–30 wt% of refractory metals (niobium, molybdenum, ruthenium, tantalum, tungsten, rhenium, vanadium, etc.) and low-melting metals (lithium, magnesium, indium, lead, tin, bismuth, etc.).

An interesting feature of palladium is the existence of wide solid solution regions (around 10–15 wt%) with all rare earth metals except lanthanum and neodymium, for which the regions are around 2 wt%. Palladium alloys with structurally isomorphous metals (iron, cobalt, nickel, copper, silver, gold, etc.) crystallise with the formation of continuous solid solutions. The wide range of palladium-based solid solutions observed in binary systems is also found in ternary systems of these metals.

The purity of palladium with respect to interstitial elements is of primary importance during alloy formation. To avoid the problems of internal oxidation and formation of complex impurity inclusions in the crystal lattice, both the chemical purity of the starting components and the preparation of the alloy must be carefully controlled. Arc-melted palladium alloys, which are pure with respect to interstitial impurities, have adequate plasticity (elongation before fracture, δ , of >20%). This allows micron-sized foils to be prepared by cold rolling with intermediate vacuum annealing (5, 6).

Palladium-Silver Alloy Membranes

Membrane alloys based on the palladium-silver system are well studied and have commercial applications (7). The 77 wt% Pd-23 wt% Ag composition is a plastic alloy with a specific hydrogen permeability of $3.4 \text{ Nm}^3 \text{ mm m}^{-2} \text{ h}^{-1} \text{ MPa}^{-0.5}$ at 600°C. However, the high silver content leads to a decrease in hydrogen purity above 450°C, as traces of oxygen are observed in the hydrogen output stream.

Multicomponent palladium-silver-based alloys referred to as 'B type' were developed in Russia during the 1970s. The 'B1' alloy, whose composition is 80.6 wt% Pd-15 wt% Ag-3 wt% Au-0.6 wt% Pt-0.6 wt% Ru-0.2 wt% Al, is used in industry (8–11) as it shows the optimum combination of membrane characteristics: it is strong, plastic and corrosion-resistant in hydrocarbon gas media. Its specific hydrogen permeability is $2.5 \text{ Nm}^3 \text{ mm m}^{-2} \text{ h}^{-1} \text{ MPa}^{-0.5}$ at 600°C. However, non-uniform distribution of alloying components can cause failure of capillaries and membranes made from this alloy. Moreover, palladium-silver alloys are poisoned irreversibly in gas mixtures containing even small amounts of hydrogen sulfide.

Silver-Free Palladium Alloy Membranes

Our studies were aimed at the development of silver-free alloys which are easy to manufacture and have a higher hydrogen permeability than that of pure palladium.

An analysis of our experimental results showed that variations in the hydrogen permeability caused by alloying are directly dependent on variations in the lattice parameters of palladium. In particular, alloying with ruthenium, indium, lead, and rare earth metals leads to an increase in the hydrogen permeability of palladium alloy foils, whereas tin slows down hydrogen diffusion. When alloying with copper, low levels of copper addition initially cause a drop in hydrogen permeability, but in palladium alloys with 38–42 wt% copper, which show solid-state ordering below 600°C (12), hydrogen permeability is increased. Changes in the hydrogen permeability of palladium-copper alloys have been studied by a number of investigators (13–15).

Hydrogen Permeability and Mechanical Properties

We studied the hydrogen permeability and strength of palladium-copper, palladium-indium, palladium-lead, palladium-yttrium-ruthenium, palladium-indium-ruthenium and palladium-rare earth metal alloys (16, 17). Based on our previous results, we selected the following compositions of membrane alloys for further investigation of their hydrogen permeability, strength, expansion and corrosion resistance:

- 93 wt% Pd-7 wt% Y;
- 60 wt% Pd-40 wt% Cu;
- 94 wt% Pd-6 wt% Ru;
- and 93.5 wt% Pd-6 wt% In-0.5 wt% Ru.

Preparation procedures and techniques for measuring the strength, hydrogen permeability and expansion are described in a previous paper by Burkhanov *et al.* (18).

Tables I and II show the mechanical properties and specific hydrogen permeability, respectively, of these alloys. It can be seen from the Tables that the 93 wt% Pd-7 wt% Y alloy has the highest hydrogen permeability and strength but the lowest plasticity of the alloys studied.

Effects of Temperature

Palladium alloys dissolve hydrogen to concentrations of around 0.3–0.6 at% H directly from the gas phase. Stresses resulting from the increase in alloy volume upon hydrogen dissolution are the principal cause of loss of membrane element sealing. Therefore, the

study of thermal expansion in the presence of hydrogen is of primary importance in designing membrane elements. Experimental values for linear thermal expansion ($\Delta L/L$, %) of the palladium alloys at a hydrogen pressure of 0.1 MPa are shown in Table III.

The thermal expansion must be kept below 1% and this determines the range of possible operating temperatures of the alloy membranes (see Table III). The lowest thermal expansion is observed for the 94 wt% Pd-6 wt% Ru alloy; the range of its operating temperatures is 150–500°C. The range of operating temperatures for the 60 wt% Pd-40 wt% Cu alloy is 200–450°C; above 450°C, the disordered face-centred cubic (fcc) phase appears and the hydrogen permeability decreases. The range of operating temperatures for the 93 wt% Pd-7 wt% Y and 93.5 wt% Pd-6 wt% In-0.5 wt% Ru alloys is 300–600°C.

Table I

Mechanical Properties of Palladium-Based Membrane Alloys in the Annealed State

Composition, wt%	Hardness, HV	Ultimate tensile strength, σ , kg mm ⁻²	Relative elongation, δ , %
100% Pd	40	30	28
93% Pd-7% Y	172	60	16
60% Pd-40% Cu	120	50	22
94% Pd-6% Ru	142	55	20
93.5% Pd-6% In-0.5% Ru	114	48	26

Table II

Specific Hydrogen Permeability of Palladium-Based Membrane Alloys at Different Temperatures^a

Composition, wt%	Temperature, °C								
	200	250	300	350	400	450	500	550	600
100% Pd	–	–	0.7	0.8	1.01	1.18	1.35	1.50	1.68
93% Pd-7% Y	–	2.6	3.1	3.6	4.13	4.59	5.18	5.57	5.7
60% Pd-40% Cu	–	1.1	1.3	1.5	1.65	1.75	–	–	–
94% Pd-6% Ru	0.3	0.4	0.55	0.7	0.9	1.1	1.3	1.5	1.8
93.5% Pd-6% In-0.5% Ru	–	0.7	0.85	1.0	1.2	1.5	1.7	1.9	2.2

^aSpecific permeability is given in Nm³ mm m⁻² h⁻¹ MPa^{-0.5}

Table III

Linear Thermal Expansion of Palladium-Based Membrane Alloys in Hydrogen^a

Composition, wt%	Temperature, °C										
	30	50	100	150	200	250	300	350	400	450	500
93% Pd-7% Y	3.2	2.8	2.37	2.00	0.96	0.76	0.46	0.30	0.23	0.19	0.11
60% Pd-40% Cu	3.1	2.7	2.2	1.8	0.57	0.50	0.43	0.28	0.20	0.14	–
94% Pd-6% Ru	2.74	2.42	1.7	0.25	0.15	0.09	0.07	0.06	0.05	0.04	0.04
93.5% Pd-6% In-0.5% Ru	4.1	3.5	3.2	2.3	0.76	0.63	0.5	0.32	0.25	0.19	0.13

^aThe linear thermal expansion is defined as $\Delta L/L$, %

Effects of Gas Composition

The stability of membranes and membrane elements depends on the hydrogen permeability. Heating palladium-based membrane elements from room temperature to ~300°C in hydrogen-containing gas mixtures and then cooling causes an increase in the membrane volume due to hydrogen absorption. Therefore the heating and cooling of membranes is typically carried out in either a vacuum or an inert gas atmosphere.

We studied the hydrogen permeability of membrane alloys during heating and cooling in the presence of carbon dioxide in synthesis gas obtained from hydrocarbon fuel conversion. The specific hydrogen permeability was measured at different temperatures during repeated heating and cooling of membranes 50 μm thick in vacuum, then in CO_2 , and again in vacuum. Within a selected temperature range of 200–600°C (200–400°C for the palladium-copper alloy), the specific hydrogen permeability was found to be independent of the pressure drop across the membrane (the difference between pressures before the membrane and after it).

With the exception of the 93 wt% Pd-7 wt% Y alloy, there was no change in the specific hydrogen permeability before and after heating and cooling of membranes in CO_2 . This indicates that there were no structural changes in the membranes under these conditions within this temperature range. The same results were observed after heating and cooling the membranes in a vacuum. Therefore, we conclude that membrane elements made from 60 wt% Pd-40 wt% Cu, 94 wt% Pd-6 wt% Ru and 93.5 wt% Pd-6 wt% In-0.5 wt% Ru alloys can be heated up to ~300°C in

a CO_2 atmosphere. This removes the need for a vacuum or an inert gas atmosphere and consequently allows the process to be simplified and costs to be reduced.

The hydrogen permeability of the 93 wt% Pd-7 wt% Y alloy decreases during heating and cooling in CO_2 . Therefore, the heating of this membrane should be carried out in a vacuum or an inert gas atmosphere (19).

Membrane characteristics of the 93 wt% Pd-7 wt% Y, 60 wt% Pd-40 wt% Cu, 94 wt% Pd-6 wt% Ru and 93.5 wt% Pd-6 wt% In-0.5 wt% Ru alloys were studied previously, and recommendations were made for their effective operation in selected gas media (20).

Behaviour of the Palladium Alloys

93 wt% Pd-7 wt% Y Alloy

Maximum hydrogen permeability and strength are observed for palladium alloys with rare earth metals, in particular with yttrium (21, 22). The optimum composition is 93 wt% Pd-7 wt% Y. At certain temperatures, its hydrogen permeability exceeds that of 77 wt% Pd-23 wt% Ag and B1 alloys by factors of two and three, respectively. Further work is being undertaken to optimise service conditions for this alloy in view of its instability in the presence of CO_2 .

Membranes made from the 93 wt% Pd-7 wt% Y alloy have good corrosion resistance and high productivity during operation in gas mixtures of argon and hydrogen, helium and hydrogen and nitrogen and hydrogen. Thus, the composition shows promise as a membrane alloy for the separation of high purity hydrogen from hydrogen-helium mixtures (purification of helium with respect to hydrogen isotopes),

ammonia dissociation products, and for liquid nitrogen production.

60 wt% Pd-40 wt% Cu Alloy

This alloy gives a substantial cost saving due to its high copper content. It is plastic and corrosion resistant during operation in hydrocarbon media. It operates stably at temperatures of 280–320°C for the separation of hydrogen from water vapour-methanol conversion products.

94 wt% Pd-6 wt% Ru Alloy

This alloy has the lowest hydrogen permeability and thermal expansion of all the alloys studied. It can operate at low temperatures (from 150°C) in hydrocarbon media and in waste streams from silicon production ($\text{H}_2 + \text{CH}_3\text{SiH}_3 + \text{SiCl}_4 + \text{HCl} + \text{C}_n\text{H}_n$).

93.5 wt% Pd-6 wt% In-0.5 wt% Ru Alloy

This alloy was found to have the optimum combination of strength, plasticity, hydrogen permeability and corrosion resistance. The addition of 0.5 wt% ruthenium to the palladium-indium matrix increased the strength of the alloy and stabilised its operation in a hydrogen-containing atmosphere without any changes to the composition of its surface, which were observed in the 94 wt% Pd-6 wt% In alloy. The ternary alloy withstands prolonged operation in a hydrogen atmosphere, and is stable during thermal cycling and in corrosive media (methane, carbon monoxide

(to 18 vol%), CO_2 , H_2S (to 1.5 vol%)) (19). Test reports demonstrating its performance in hydrogen-containing gas mixtures with up to 1.5 vol% H_2S are available on request by contacting the corresponding author Gennady S. Burkhanov at: genburkh@imet.ac.ru.

The ternary alloy may be used to separate high purity hydrogen from synthesis gas produced from hydrocarbon fuel and natural gas conversion (20).

Hydrogen Purification Modules

At present, increasing demand for high purity hydrogen means that effective, reliable and low-cost commercial modules for hydrogen production and purification are required. Membrane elements can be constructed in the form of either capillary tubes, flat plates or discs. In terms of ease of manufacture, service characteristics, efficiency and ease of repair it can be demonstrated that disc membranes are the most promising for commercial application (3, 23). The disc structure requires five to ten times fewer joints between membranes and structural elements than does the capillary tube structure, leading to improved reliability.

Figure 2 shows a hydrogen separation element and its components. The membranes, membrane elements and membrane modules are produced using cold forming and cutting techniques to ensure accuracy at each stage. Joints are sealed by diffusion and argon-arc welding in the absence of solders, pastes, etc., to ensure efficient sealing.

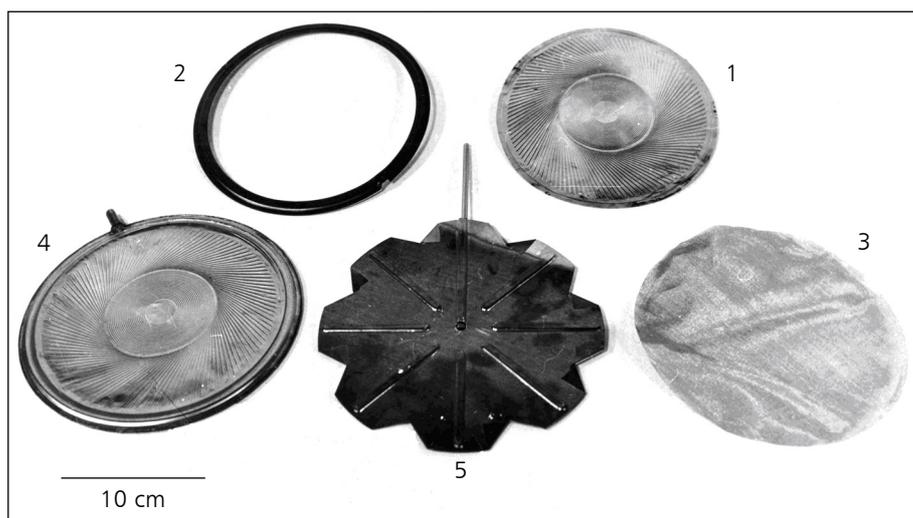


Fig. 2. Hydrogen separation element, its components, and gas distribution unit (deflector): 1 = palladium alloy membrane, 150 mm in diameter; 2 = ring frame; 3 = porous separator (wire netting); 4 = ready-assembled element; and 5 = deflector

The membrane filtration element is a 'sandwich-like' assemblage of two walls in the form of disc membranes either 50 mm or 150 mm in diameter, made from a palladium-based alloy foil 20–50 μm thick and welded onto a stainless steel ring frame by diffusion welding. A porous separator (wire netting) is placed between the walls. After that, the frames are welded circularly with a branch pipe for the output of high purity hydrogen.

Figure 3 shows the appearance of a hydrogen separation unit for the diffusion purification of hydrogen. Figure 4 shows a membrane module with a hydrogen separation unit containing seven membrane elements. This type of membrane unit can easily be introduced into a production process for the separation of hydrogen from hydrogen-containing gas mixtures or chemical product streams.

Technical characteristics of separation elements made from the 93.5 wt% Pd-6 wt% In-0.5 wt% Ru alloy with membranes 50 mm in diameter (FEL-50) and 150 mm in diameter (FEL-150) are given in Table IV.

The hydrogen separation capacity, Q , of the optimum hydrogen separation unit containing ten membrane elements with 93.5 wt% Pd-6 wt% In-0.5 wt% Ru membranes 150 mm in diameter was calculated for

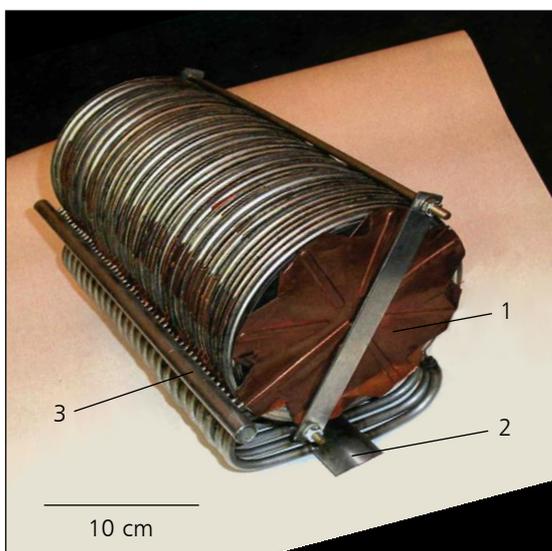


Fig. 3. Appearance of a hydrogen separation unit for the diffusion purification of hydrogen consisting of 50 diffusion elements with palladium-based alloy membrane 150 mm in diameter: 1 = deflector; 2 = high purity hydrogen outfall collector; and 3 = depleted mixture outfall collector

preset operating parameters: membrane thickness, operating temperature, input hydrogen pressure, output hydrogen pressure, and high purity hydrogen separation coefficient, η , (which is set as the ratio of the volume of separated pure hydrogen to the partial volume of hydrogen in the gas mixture). Results are given in Table V. The preset membrane area was 0.326 m^2 .

The hydrogen separation capacity of the membrane hydrogen separation units can easily be increased by increasing the differential pressure across the membrane, by increasing the number of membrane elements or by decreasing the membrane thickness. This hydrogen separation unit also allowed the precious metal loading to be reduced compared to that used in capillary tube elements (24).

Conclusions

The separation of high purity hydrogen from hydrogen-containing gas mixtures using palladium-based alloy membranes is an effective, environmentally sustainable and economical process for the commercial production of high purity hydrogen.

The membrane composition can be optimised for each gas mixture to ensure maximum stability and service life. During this study, it was found that disc membranes of diameter 150 mm made from 93.5 wt% Pd-6 wt% In-0.5 wt% Ru alloy are the most promising for commercial application for use with synthesis gas derived from hydrocarbon conversion. Modules were designed using this alloy and can be introduced

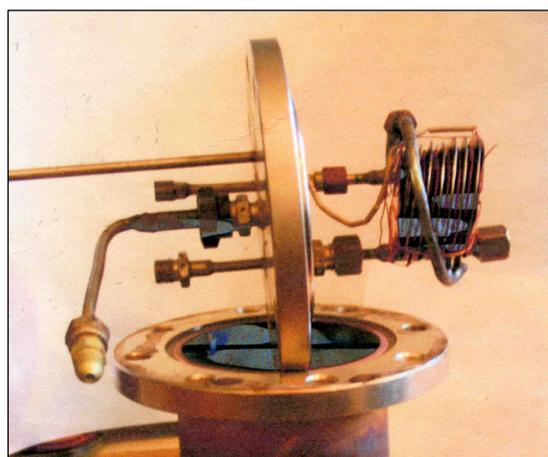


Fig. 4. Typical design of a membrane module with a hydrogen separation unit consisting of seven membrane elements with membranes 50 mm in diameter

Table IV

Technical Characteristics of Hydrogen Separation Elements FEL-50 and FEL-150

Property	Hydrogen Separation Element	
	FEL-50	FEL-150
Hydrogen separation capacity ^a , m ³ h ⁻¹	0.2–0.5	2.4–4.4
Weight of palladium alloy in the hydrogen separation element, g	2.4	22.0
Membrane thickness, μm	50	50
Hydrogen separation element diameter, mm	63	170
Maximum dimensions of optimum module (10 elements), mm	D = 70 H = 50	D = 200 H = 80
Hydrogen purity, vol%	>99.9999	>99.9999

^aOperating conditions: temperature = 600°C, input pressure = 1.0–5.0 MPa, partial pressure of pure hydrogen = 0.11 MPa

Table V

Calculated Optimum Hydrogen Separation Capacity of the Palladium-Indium-Ruthenium Alloy Membrane Unit

Membrane thickness, μm	Operating temperature, °C	Input hydrogen pressure, MPa	Output hydrogen pressure, MPa	Hydrogen separation capacity, Q, Nm ³ h ⁻¹		
				η = 0.6 ^a	η = 0.8 ^a	η = 0.9 ^a
30	350	1.8	0.15	15	14	11
30	600	5.0	0.15	65	60	48
50	600	5.0	0.15	39	36	29

^aHigh purity hydrogen separation coefficient, η, defined as the ratio of the volume of separated pure hydrogen to the partial volume of hydrogen in the gas mixture

into a production process to separate high purity hydrogen from a variety of hydrogen-containing gas mixtures.

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