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

By Gennady S. Burkhanov*, Nelli B. Gorina, Natalia B. Kolchugina and Nataliya R. Roshan
Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences,Leninskii prospect 49, 119991 Moscow, Russia;
*E-mail: genburkh@imet.ac.ru

Dmitry I. Slovetsky and Evgeny M. Chistov
Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences,Leninskii prospect 29, 119991 Moscow, Russia

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 (m³ h⁻¹) for micro- and nanoelectronics and portable energy sources such as fuel cells, to tens or hundreds of m³ h⁻¹ for transport and small stationary energy sources, tens of thousands of m³ h⁻¹ for the chemical industry and metallurgical applications, and hundreds of thousands of m³ 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...
resistance and high plasticity and strength during operation at temperatures of 300–700ºC. 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, $\delta$, 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 $3.4 \text{ Nm}^3 \text{ mm}^{-2} \text{ h}^{-1} \text{ MPa}^{-0.5}$ at 600ºC. However, the high silver content leads to a decrease in hydrogen purity above 450ºC, 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 $2.5 \text{ Nm}^3 \text{ mm}^{-2} \text{ h}^{-1} \text{ MPa}^{-0.5}$ at 600ºC. 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 600ºC (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.

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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 150–500ºC. The range of operating temperatures for the 60 wt% Pd-40 wt% Cu alloy is 200–450ºC; above 450ºC, 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 300–600ºC.

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**Table I**

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

<table>
<thead>
<tr>
<th>Composition, wt%</th>
<th>Hardness, HV</th>
<th>Ultimate tensile strength, $\sigma$, kg mm$^{-2}$</th>
<th>Relative elongation, $\delta$, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% Pd</td>
<td>40</td>
<td>30</td>
<td>28</td>
</tr>
<tr>
<td>93% Pd-7% Y</td>
<td>172</td>
<td>60</td>
<td>16</td>
</tr>
<tr>
<td>60% Pd-40% Cu</td>
<td>120</td>
<td>50</td>
<td>22</td>
</tr>
<tr>
<td>94% Pd-6% Ru</td>
<td>142</td>
<td>55</td>
<td>20</td>
</tr>
<tr>
<td>93.5% Pd-6% In-0.5% Ru</td>
<td>114</td>
<td>48</td>
<td>26</td>
</tr>
</tbody>
</table>

**Table II**

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

<table>
<thead>
<tr>
<th>Composition, wt%</th>
<th>Temperature, ºC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>200</td>
</tr>
<tr>
<td>100% Pd</td>
<td>–</td>
</tr>
<tr>
<td>93% Pd-7% Y</td>
<td>–</td>
</tr>
<tr>
<td>60% Pd-40% Cu</td>
<td>–</td>
</tr>
<tr>
<td>94% Pd-6% Ru</td>
<td>0.3</td>
</tr>
<tr>
<td>93.5% Pd-6% In-0.5% Ru</td>
<td>–</td>
</tr>
</tbody>
</table>

$^a$Specific permeability is given in $\text{Nm}^3 \text{mm}^{-2} \text{h}^{-1} \text{MPa}^{-0.5}$.
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 CO₂, 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 CO₂. 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 CO₂ 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 CO₂. Therefore, the heating of this membrane should be carried out in a vacuum or an inert gas atmosphere (19).

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).

### Table III

<table>
<thead>
<tr>
<th>Composition, wt%</th>
<th>Temperature, ºC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>30</td>
</tr>
<tr>
<td>93% Pd-7% Y</td>
<td>3.2</td>
</tr>
<tr>
<td>60% Pd-40% Cu</td>
<td>3.1</td>
</tr>
<tr>
<td>94% Pd-6% Ru</td>
<td>2.74</td>
</tr>
<tr>
<td>93.5% Pd-6% In-0.5% Ru</td>
<td>4.1</td>
</tr>
</tbody>
</table>

*The linear thermal expansion is defined as ∆L/L, %

**Effects of Gas Composition**

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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 (H₂ + CH₃SiH₃ + SiCl₄ + HCl + CₙHₙ).

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%), CO₂, H₂S (to 1.5 vol%)) (19). Test reports demonstrating its performance in hydrogen-containing gas mixtures with up to 1.5 vol% H₂S 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.

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**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 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 m².

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

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**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

**Fig. 4.** Typical design of a membrane module with a hydrogen separation unit consisting of seven membrane elements with membranes 50 mm in diameter
into a production process to separate high purity hydrogen from a variety of hydrogen-containing gas mixtures.

References

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

<table>
<thead>
<tr>
<th>Property</th>
<th>FEL-50</th>
<th>FEL-150</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen separation capacity, m³ h⁻¹</td>
<td>0.2–0.5</td>
<td>2.4–4.4</td>
</tr>
<tr>
<td>Weight of palladium alloy in the hydrogen separation element, g</td>
<td>2.4</td>
<td>22.0</td>
</tr>
<tr>
<td>Membrane thickness, µm</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>Hydrogen separation element diameter, mm</td>
<td>63</td>
<td>170</td>
</tr>
<tr>
<td>Maximum dimensions of optimum module (10 elements), mm</td>
<td>D = 70</td>
<td>D = 200</td>
</tr>
<tr>
<td></td>
<td>H = 50</td>
<td>H = 80</td>
</tr>
<tr>
<td>Hydrogen purity, vol%</td>
<td>&gt;99.9999</td>
<td>&gt;99.9999</td>
</tr>
</tbody>
</table>

*Operating 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

<table>
<thead>
<tr>
<th>Membrane thickness, µm</th>
<th>Operating temperature, ºC</th>
<th>Input hydrogen pressure, MPa</th>
<th>Output hydrogen pressure, MPa</th>
<th>Hydrogen separation capacity, Q, Nm³ h⁻¹</th>
<th>η = 0.6*</th>
<th>η = 0.8*</th>
<th>η = 0.9*</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>350</td>
<td>1.8</td>
<td>0.15</td>
<td>15</td>
<td>14</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>600</td>
<td>5.0</td>
<td>0.15</td>
<td>65</td>
<td>60</td>
<td>48</td>
<td></td>
</tr>
<tr>
<td>50</td>
<td>600</td>
<td>5.0</td>
<td>0.15</td>
<td>39</td>
<td>36</td>
<td>29</td>
<td></td>
</tr>
</tbody>
</table>

*High 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
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The Authors

Gennady S. Burkhanov received a PhD from the Moscow Steel and Alloys Institute in 1961 and was appointed a lecturer in physics in 1975, Chief of Laboratory in 1984 and Professor in 1986. He received the USSR State Prize in 1968, 1986 and 1999 and was elected as corresponding member of the Russian Academy of Sciences in 2000. He has published over 380 papers, monographs and patents in the field of materials science of refractory and rare metals, single crystals, and superconductors.

Nataliya B. Kolchugina received her PhD from the Moscow Institute of Fine Chemical Technology in 1994 and is currently a lead researcher at the Russian Academy of Sciences. She has a number of scientific publications and patents to her name and is particularly interested in the science of high purity rare earth metals, alloys, single crystals and phase diagrams of palladium-rare earth metal systems.

Nelli B. Gorina was educated at the Moscow Steel and Alloys Institute and received her PhD in 1978. She is a senior researcher at the Russian Academy of Sciences and has contributed to many publications and patents, including the monograph “Physical Metallurgy of Platinum Metals” and the “Handbook of Precious Metals”. She is interested in the materials science of the platinum group metals, their single crystals, alloys for microelectronics, and hydrogen power engineering.

Nataliya R. Roshan is a graduate of the Bauman Moscow State Technical University and is a senior researcher at the Russian Academy of Sciences. She has contributed to over 100 scientific publications and patents, including the monograph “Physical Metallurgy of Platinum Metals”. Her interests are in the materials science of the platinum group metals, the development of advanced palladium-based materials for membrane diffusion elements, and the preparation of thin and ultra-thin membranes.

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Dmitry I. Slovetsky graduated from Lomonosov Moscow State University with a PhD in Physics in 1967 and was appointed as a lecturer in 1978 and Professor in 1986. He was a recipient of the USSR State Prize in 1989 and has published around 380 papers, monographs, reviews and patents. His interests lie in plasma chemistry and physics, membrane science and technology, hydrogen energy, reaction kinetics and mechanisms, and catalysis.

Evgeny M. Chistov was educated at the Moscow Physics and Engineering Institute and is a senior researcher at the Russian Academy of Sciences with more than 70 scientific publications and patents in hydrogen power engineering, fuel cells, reaction kinetics and mechanisms, catalysis, and construction of membrane elements and modules for separation of high purity hydrogen from hydrogen-containing gas mixtures.