

Advertisement for Incubation of Technology

Title of the technology	Composite Metal Membrane Reactor for Production/ Separation/ Recovery of High-Purity Hydrogen
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Current state of Technology

- ✓ Basic principles observed
- ✓ Technology concept formulated
- ✓ Experimental proof of concept
- ✓ Technology validated in lab
- ✓ Technology validated in relevant environment (industrially relevant environment in the case of key enabling technologies)
- ✓ Lab scale Demo system available

General Information

Metallic membrane is one of the most potential processes/technologies for separation of hydrogen from gaseous mixture, to a very-high level of purity (> 99.95%), because metals are permeable to only hydrogen. Supported metal membranes are one of the important classes of metal membranes that are commercially used. They have advantages of being of low cost while offering high flux due to low thickness of metal coatings provided on porous base supports.

BARC has developed a technology to produce supported metal membranes and membrane reactors, which are effective for separation, recovery and production of hydrogen. It has applications in separation of hydrogen in several thermochemical processes of water splitting, steam methane reforming for hydrogen production, H₂S and NH₃ decomposition of coke oven gas in steel industry for hydrogen production and recovery of hydrogen from vent gases of hydrocracker.

Features/Specification of system

The present technology describes a methodology to produce a composite metal membranes and membrane reactors in tubular configuration. This product is highly effective and almost indispensable for production/separation/recovery of high-purity hydrogen. Flux of hydrogen at 0.1 bar trans-membrane is 100 LMH with H₂-He selectivity >3000. Membrane Reactor, on the other hand, can enhance the equilibrium conversion beyond 85-90% for any equilibrium limited reactions, that are predominantly being carried out on industrial scale.

Characteristics of indigenous membrane

Support:

- Material: Clay-alumina.
- Method of fabrication: Extrusion.

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- Pore size: ~1.5 micron.
- Porosity: 30-35%.
- Surface roughness: 500-700 nm.

Metal layer:

- Material: Refractory and noble metals & their alloys (to be chosen, based on application environment)
- Method of fabrication: DC Magnetron Sputter coating technique (Indigenously designed for coating on tubular supports)
- Metal layer thickness: Tunable (from few nm to few micron)
- Structure: Dense
- Surface roughness: ~30 nm (for a metal layer of 3 micron thickness)

Performance

Specifications of Membrane Permeator:

- MOC of membrane housing: Hastelloy C-276/SS304/SS316 (As Applicable, based on process environment).
- Membrane Flux: 100 LMH at 0.1 bar trans membrane pressure.
- Gas Purity: > 99.95% (with H₂-He mixture).

Specification of Membrane Reactor:

- Configuration: Shell & Tube.
- MOC: Hastelloy/SS304/SS 316 (As Applicable, based on process environment).
- Design & fabrication capacity: 150 LPH.
- Tested Capacity: 50 LPH.
- Demonstration underway: 150 LPH.

Stability:

- Thermal stability: up to 600 °C.
- Mechanical stability: up to 5 bar (for porous ceramic support) and up to 30 bar (with porous metallic support).

Chemical stability: Top metal layer needs to be compatible with feed stream & type of metal can accordingly be selected.

Parameters for the current and Target systems:

Parameter	<i>For Current system System</i>	<i>For Target System</i>
Capacity	150 LPH	1000 LPH
Leak rate	10^{-3} mbar.lit/s	$\leq 10^{-4}$ mbar.lit/s

Temperature & pressure of operation	450 °C	600 °C
Feed stream components	H_2 , HI, I_2 , H_2O	CO, CO_2 , H_2S , HCN, NH_3 and light hydrocarbons

Working of the System (with schematic block diagram)

In the membrane reactor (MR), the hydrogen produced in the reaction zone can be removed from the reaction mixture *in-situ* by a hydrogen permselective membrane, thereby significantly improving the equilibrium conversion of the reaction in a single pass. MR is a state-of-the-art technology integrating reaction and separation steps into one unit, leading to high degree of process intensification. Application of metal membrane-based MRs for hydrogen production has been widely studied in literature, mainly for steam methane reforming and water gas shift reactions. , The experimental demonstration of refractory metal membrane-based MR in HI decomposition environment (Iodine-Sulphur Thermochemical Process), which involves highly corrosive (reducing) environment of HI- H_2O - I_2 at 700 K has been carried out at BARC. The demand of high purity ($\geq 99.95\%$) hydrogen is constantly increasing, mainly due to the advances in fuel cells for utilization of hydrogen, and thus metal membranes and MRs are the inevitable solution for providing high purity hydrogen.

Photograph(s) of the System



Base supports



Composite Metal Membrane



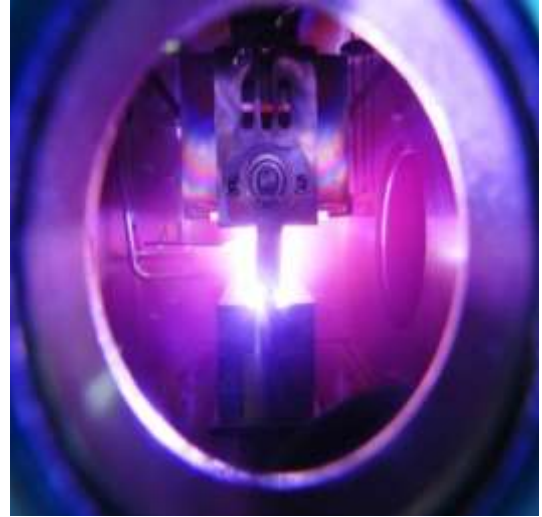
Permeator (single-tube)



Multi-tube membrane permeators/reactors



Indigenously developed Sputter Coating Machine for Metal Membrane Fabrication



Sputter deposition process (showing the argon gas plasma)

The membrane and membrane permeators (up to 500 mm length and 20 mm OD) have been successfully developed and tested in BARC.

Applications of the System

- Separation of hydrogen in several thermochemical processes of water splitting.
- Steam methane reforming for hydrogen production.
- H₂S and NH₃ decomposition of coke oven gas in steel industry for hydrogen production.
- Recovery of hydrogen from vent gases of hydrocracker.

Whether the parent product/ technology/ process is patented: Yes/No: Yes

If yes, provide the details

The patent title: “Multi-tube membrane reactor with provision for molten salt heating for hydrogen production using nuclear/solar heat”

Indian Patent Application Number: 202121046065; Dated: 8th Oct, 2021

The brief details of this technology may be found at following BARC weblink:

<https://barc.gov.in/technologies/cmm/index.html>

The technology will provide recipe, detailed specifications and fabrication of ceramic support, membrane fabrication parameters and detailed design and drawings of magnetron sputter coating machine, membrane reactor assembly (both single- and multi-tube membrane configurations).

Deliverables

Composite Metal Membrane Reactor for Production/Separation/Recovery of High-Purity Hydrogen

- Metal membrane reactor for industrial application (up to a capacity of 1000 LPH, at high temperature (up to 600 °C) and pressure (up to 10 bar).
- Certificate for Testing and endurance studies of the system in H₂S & NH₃ environment for industrial deployment.
- All source codes, drawings, full documentation of the final upgraded system.

Justification for Incubation

Following specifications are the need of the hour as per market/ industry requirement:-

- Design of scaled-up (multi-tube) membrane reactor (up to a capacity of 1000 LPH).
- Sealing arrangement for high temperature up to 600 °C) and high pressure (up to 10 bar).
- Fabrication & testing of multi-tube membrane reactor in intended environment (H₂S & NH₃ stream).
- Qualification of multi-tube membrane reactor in process environment with endurance testing.

Facility and Infrastructure requirements (to be provided by Incubatee and BARC)–

Title Head	To be provided by BARC	To be provided by Incubatee
Manpower/ expertise	Technical expertise and consultancy for the design, fabrication, QA/QC for integration, testing, qualification of membrane reactor.	Two Process engineering supervisors, 2 technicians, 1 draftsman, 1 mechanical design engineer.
Machinery and Equipment	Membrane supports, metal targets, DC magnetron sputtering assembly, vacuum furnace	Facility and tools for assembling of mechanical components, Heaters, Gas Analyzers, Gas Chromatograph/ Mass Spectrometer, Helium leak testing equipment, Mechanical fabrication, and theoretical (computational) facility for design.
Economic Viability:		
a. Investment and unit cost of production	<p>Consumable Cost (estimate only)</p> <ul style="list-style-type: none"> • Ceramic support tube (500 mm length, 12 mm OD): Rs. 1000/- per piece • Palladium-silver (75-25 wt%) alloy metal target (for coating about 500 tubes): Rs. 25 Lac <p>Capital investment Cost Estimate (excluding land & building cost):</p> <ul style="list-style-type: none"> • DC Magnetron Sputtering assembly for metal membrane 	

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	<p>fabrication: Rs. 1.0 Cr.</p> <ul style="list-style-type: none"> Membrane reactor (1000 LPH): Rs. 1.0 Cr. <p>Cost per membrane: Rs. 5000/- (area: 200 cm²) approximate</p>
b. Imported/indigenous market price of equivalent product, if available.	<p>Approx. Rs. 15,000/- per metal membrane of 100 cm² area;</p> <p>Approx. Rs. 20 Lac per single-tube membrane permeator/reactor</p>
Special Requirements:	
Any special requirements for plant, industry, location utilities, handling storage, safety etc.	<p>Hydrogen and other flammable/corrosive gases will be present in the test gas feed stream, so appropriate safety measures along with gas detectors and alarms required to be there in the Works of Incubatee.</p>

Note: As per in-house technology incubation policy, the incubatee should be a licensee of the existing technology. Alternatively, the applicant will be required to take the license of the existing technology before entering incubation agreement.

If interested in Incubation, kindly **download -> fill -> scan -> send** the application form to -

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