# DESIGN OF UHV COMPATIBLE PERMEATION MEASUREMENT SYSTEM

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***Abstract:*** Ultra-high vacuum (UHV) is high degree vacuum order of pressure lower than about $10^{-7}$ pascal or $10^{-9}$ torr ($10^{-9}$ mbar). For UHV chamber gas load present are gas entering the volume through external and internal leaks, gas emanating from or passing through materials by diffusion and permeation. Permeation is a general phenomenon happens in the vacuum system where environmental gases permeates through the material. Knowledge of permeation is available for the standard materials, but it is not available to all the materials especially for the newly developed materials. Hence an experimental system is needed to be developed for the measurement of the permeation through various materials. The said system will also be useful for the gas load estimation in the compiles geometries and feed through the vacuum interfaces. Hence, an experimental system has been designed considering the UHV and material understanding and analyze against high pressure difference.

***Keywords* – Ultra-high vacuum, Permeation measurement system, Permeation coefficient, Diffusion flux.**

## I. INTRODUCTION

Ultra-high vacuum (UHV) is the high degree vacuum regime characterised by pressure lower than about $10^{-7}$ pascal or $10^{-9}$ torr ($10^{-9}$ mbar). UHV conditions are created by pumping the gas out of UHV chamber. The pressure of system is increase (degrade vacuum environment) with increase of gas load. The gas load is the rate by which gas enters the system volume. It includes gas entering the volume through external and internal leaks, gas emanating from or passing through materials by diffusion and permeation. Here, gas load due to other different reason is take care at initial stage but gas load due to permeation cannot reduce. Hence selection of the right material in Ultra High vacuum system with lower permeability coefficient is very mush essential. Knowledge of permeation is available for the standard materials, but it is not available to all the materials especially for the newly developed materials. Hence an experimental system is needed to be developed for the measurement of the permeation through various materials. The present system will also be useful for the gas load estimation in the compiles geometries and feed through the vacuum interfaces. Hence, an experimental system has to be designed considering the UHV and material understanding.

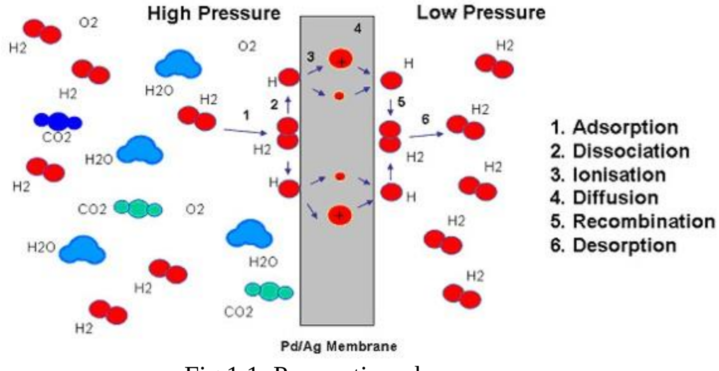


Fig.1.1. Permeation phenomenon

Hence many different types of experimental system are developed for the measurement of the permeation through various materials but there is lack of study for set-up where leakage can be identified and considered. A present paper includes design of experimental setup, design calculation and structural analysis. Categorically two different types of permeation measurement system are developed: (1) Measurement system which is used Self supported type membrane and (2) Measurement system which is used Composite type membrane are described in this review. The specific design and analysis, which satisfy the characteristics requirements in order to measure permeation parameters are discussed. The design for permeation measurement system is followed by general design procedure as shown below. We are developed concept for measurement system as describe.

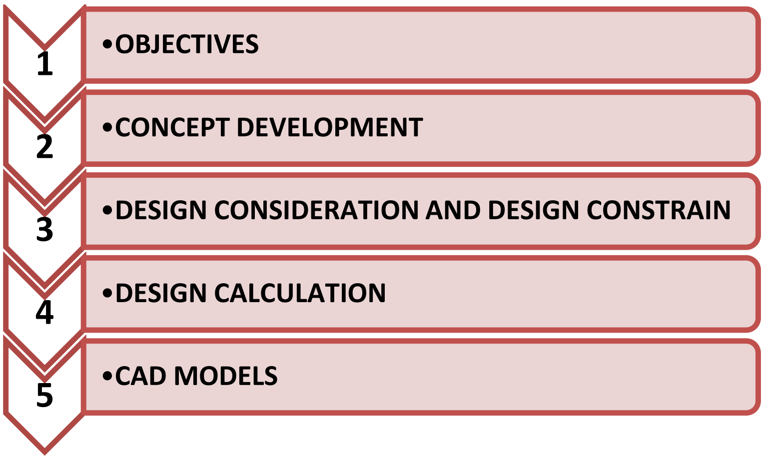


Fig.1.2. Design procedure

## II. SCHEMATIC DESIGN FOR PERMEATION MEASUREMENT SYSTEM

Experimental system is schematically shown for the measurement of the permeation through various material considering vacuum and temperature. The designed system is compatible with ultra-high vacuum ($10^{-9}$ torr) and high temperature (500°C).

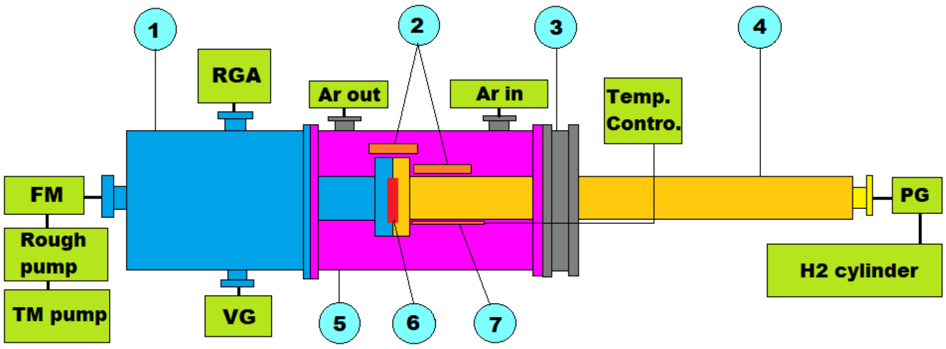


Fig.2.1. Schematic diagram for permeation measurement system for self-supporting type membrane

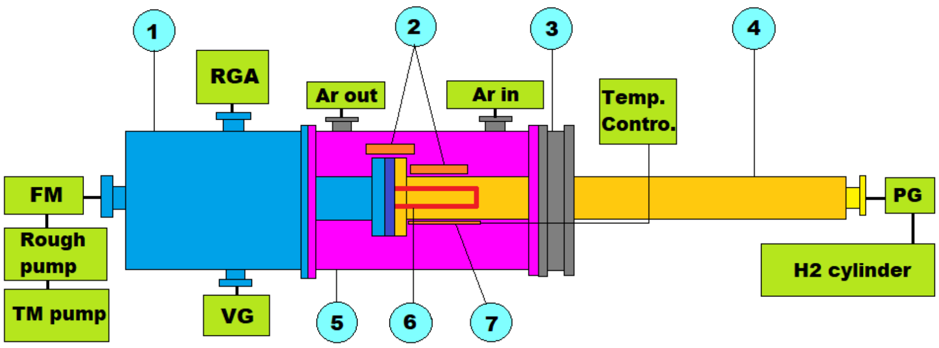


Fig.2.2. Schematic diagram for permeation measurement system for composite type membrane

Here,

| | | | |
|---|---|---|---|
| 1 - Vacuum chamber | 3- Bellows | 5 – Outer cylinder | 7 – Temperature sensor |
| 2 - Heater | 4 – Gas tube | 6 - Membrane | |
| FM: Flow meter | VG: Vacuum gauge | RGA: Residual gas analyzer | |

## III. DESIGN CONSTRAIN AND DESIGN CALCULATION

### 3.1 Design constrain

The meaning of constraint is a limitation or restriction. Constraint can define as “the state of being checked, restricted, or compelled to avoid or perform some action”. The keywords boundary, control, force, and restraint are suitable for the understanding of constraints in the context of conceptual design activity. There is three types of constrains,

Table 3.1. Types of constrain

| No | Types of Constraint | Constraint Sources |
|---|---|---|
| 1 | Functional, technology, time, material, motion, aesthetic, health and safety | Design options |
| 2 | Manufacturing, inspect ability, quality sustainability, life-cycle. | Conditions Imposed on the Development Process |
| 3 | Economic, environmental, social, legality, ethical | Standards and Regulations |

**3.2 Material Selection**

Materials for use in vacuum are need some extra important properties like low outgassing rate, low vapor pressure, low thermal expansion and non-porous.

Table 3.2. Material selection

| No | Name of Component | Required Material Properties | Suggested Material |
|---|---|---|---|
| 1 | Vacuum Cylinder & Tubes and Other Side Tubes | Low outgassing, Resistance to high pressure difference, can be sealed with welding to flange, Low vapor pressure, Non-porous and non-cracked, Low thermal expansion if possible | SS304 |
| 2 | Gasket | Prevent high vacuum leakage, withstand high temperature | Helicoflex Seal, Copper gaskets |
| 3 | Flange | Low outgassing, can be joined with tube or cylinder by joining process, Low thermal expansion if possible | SS304 |
| 4 | Sample material | As per requirement | Palladium |
| 5 | Porous material | Porous material (ceramic or porous SS) of pore size between 0.2 to 0.4 nm, Strength to withstand high pressure difference, Strength to withstand high temperature | Alumina |
| 6 | Base | Mechanical strength | Cast Iron |
| 7 | Nut-Bolt | Tensile strength and high temperature | Mild steel |
| 8 | Non-porous support tube | Low outgassing, can be deposit able with ceramic coating, Resistance to high pressure difference, Low vapor pressure, Non-porous and non-cracked, Low thermal expansion if possible | SS304 |
| 9 | Argon cylinder | Mechanical strength at normal and high temperature | SS304 |

## IV. DESIGN CALCULATION

### 4.1 Vacuum cylinder and tube

This is a combine component in which vacuum cylinder is join to flange through vacuum tube. So, from the Helicoflex data, groove size is as bellowed.

Table 4.1. Flange grooves details

| | |
|---|---|
| Groove ID: | 29.49 mm |
| Groove OD: | 36.91 mm |
| Depth of groove: | 1.95 mm |

The groove has been prepared on the Conflate type flange for membrane which also has standardize data as below:

Table 4.2. Flange details

| | |
|---|---|
| Nominal Tube Diameter | 1.50 in (38 mm) |
| Flange Outside Diameter | 2.75 in (70 mm) |
| Bolt Circle Diameter | 2.31 in (59 mm) |
| Thickness | 0.50 in (13 mm) |
| Number of Holes | 6 |
| Thru Holes Diameter | 0.265 in (6.7 mm) |

For the cylinder dimension calculation, we are followed ASME Boiler and Pressure Vessel Code VIII Rules for Construction of Pressure Vessels Division 1. As per section UG-28 THICKNESS OF SHELLS AND TUBES UNDER EXTERNAL PRESSURE we are followed step wise procedure. Here we are calculating cylinder thickness *(t)* for given cylinder length *L* and diameter as below:

- Cylinder length $L$: 150 mm.
- Cylinder diameter $D$: 120 mm.

Here, choose any thickness value so that ratio of $D/t$ is greater than 10 .

- Assumed Cylinder thickness: 2 mm.so that,

$$L/D=1.25 , D/t= 60$$

From above values, A (factor determined from Figure G in Subpart 3 of Section II, Part D)

$$A= 0.00291+(1.25-1)\left(\frac{0.00138-0.00291}{2-1}\right)$$

$$A=0.002527$$

We have to include factor for material and temperature behavior which is B (factor determined from the applicable material chart or table in Subpart 3 of Section II, Part D for maximum design metal temperature)

For the material SS304 and temperature above 500°C,

$$B= 0.427 \times 10^4 + (0.002527-0.0015)\left(\frac{0.590-0.427}{0.01-0.0015}\right)10^4$$

$$B= 4466.94$$

Using this value of B, calculate the value of the maximum allowable external working pressure $P_a$ using the following equation,

$$P_a=\left(\frac{4B}{3(D/t)}\right)$$

$$P_a= 4\times 4466.94\times 2/ 3\times 150$$

$$P_a= 79.412 \text{ Psi or } 5.47\times10^5 \text{ pascal}$$

For the Tube dimension calculation, we also follow above standard. Here we are calculating tube thickness ($t_2$) for given tube length $L_2$ and diameter as below:

- Cylinder length $L_2$: 50 mm.
- Cylinder diameter $D_2$: 25 mm.

Here, chooses any thickness value so that ratio of $D_2/t_2$ is greater than 10.

- Assumed Cylinder thickness: 2 mm. so that,

$L_2/D_2=2$ , $D_2/t_2= 12.5$

From above values, A (factor determined from Figure G in Subpart 3 of Section II, Part D) by interpolation method,

$$A_2= 0.0201+(12.5-10)\left(\frac{0.0109-0.0201}{15-10}\right)$$

$$A_2= 0.0155$$

As above factor for material and temperature behavior which is $B_2$ (factor determined from the applicable material chart or table in Subpart 3 of Section II, Part D for maximum design metal temperature)

For the material SS304 and temperature above 500°C,

$$B_2= 0.590 \times 10^4 + (0.0155-0.01)\left(\frac{0.720-0.590}{0.03-0.01}\right)10^4$$

$$B_2= 6257.5$$

Using this value of $B$, calculate the value of the maximum allowable external working pressure $P_a$ using the following equation,

$$P_{a2}=\left(\frac{4B}{3(D/t)}\right)$$

$$P_{a2}= 4\times 6257.5\times 2/ 3\times 25$$

$$P_{a2}= 667.466 \text{ Psi or } 46.02\times 10^5 \text{ pascal}$$

**4.2 Gas tube**

ASME Boiler and Pressure Vessel Code VIII Rules for Construction of Pressure Vessels Division 1. As per section UG-27 THICKNESS OF SHELLS UNDER INTERNAL PRESSURE is followed for calculating dimension of gas tube.

Here. We assumed outer radius for tube as 12.5 mm and thickness 2 mm. So now from UG 23 maximum allowable stress is calculated as below:

$$A=\left(\frac{0.125}{R_o/t}\right)$$

$A = 0.02$, And from UG 23,

$B_2= 0.590 \times 10^4 + (0.02-0.01)\left(\frac{0.720-0.590}{0.03-0.01}\right)10^4$, $B_2= 6550$, now from UG 27,

- Circumferential Stress $(P_c) = \frac{SEt}{R+0.6\,t}$

$$P_c= \frac{6550\times1\times2}{12.5+0.6\times2}$$

$$P_c= 956.20 \text{ Psi or } 6894.76 \text{ pascal}$$

- Longitudinal Stress $(P_L) = \frac{2SEt}{R-0.4\,t}$

$$P_L = \frac{2\times6550\times1\times2}{12.5-0.4\times2}$$

$$P_L = 2239.31 \text{ psi or } 154.39\times10^5 \text{ pascal}$$

In addition of this to support cylinder head, there is a manufactured thread on the tube at required portion. So for this threading allowance 4 mm extra thickness provide for threading and that threading details is as below:

Table 4.3. Thread details

| M32×1.5 | |
|---|---|
| Major diameter | 32 mm |
| Minor diameter | 30.5 mm |
| Pitch | 1.5 mm |

### 4.3 Self-supported type membrane

Force on self-supported type membrane can calculated by below calculation,

Force by external gas, $F_1 = PA = P\,(\pi r^2)$

Allowable force by membrane, $F_2 = \sigma_c\,(2\pi r t)$

So by equating, $P\,(\pi r^2) = \sigma_c\,(2\pi r t)$

so from above equation thickness is calculated as $t = \frac{Pr}{2\sigma_c}$

### 4.4 Composite type membrane

**1) Non-porous support tube:**

In this calculation we consider the pressure difference of gas and vacuum as above procedure. The flange dimension is same as mentioned in above section. Here we are calculating support tube thickness ($t_3$) for given tube length $L_3$ and diameter as below:

- Support tube length $L_3$: *100 mm.*
- Support tube diameter $D_3$: *10 mm.*

Here, chooses any thickness value so that ratio of $D_2/t_2$ is greater than 10.

- Assumed Cylinder thickness: 1 mm. so that,

$L_3/D_3 = 10$ , $D_3/t_3 = 10$

From above values, A (factor determined from Figure G in Subpart 3 of Section II, Part D)

$$A_3 = 0.0112$$

As above factor for material and temperature behavior which is $B_2$ (factor determined from the applicable material chart or table in Subpart 3 of Section II, Part D for maximum design metal temperature)

For the material SS304 and temperature above 500°C,

$$B_2 = 0.590 \times 10^4 + (0.0112-0.01)\left(\frac{0.720-0.590}{0.03-0.01}\right)10^4$$

$$B_2 = 5978$$

Using this value of B, calculate the value of the maximum allowable external working pressure $P_a$ using the following equation,

$$P_{a2} = \left(\frac{4B}{3(D/t)}\right)$$

$$P_{a2} = 4 \times 5978 \times 1/3 \times 10$$

$$P_{a2} = 797.066 \text{ Psi or } 54.95 \times 10^5 \text{Pascal}$$

**2) Porous coating dimension**

Porous coating dimension can be chosen as per our requirement, from literature review it can be generally taken as 1 mm.

**3) Sample coating dimension**

Sample thickness can be chosen as per our requirement in experiments. This coating thickness measure in microns.

### 4.5 Outer argon cylinder dimensions

The component argon cylinder does not have any high-pressure application. So it can be generally chosen as per experience as below:

- Argon cylinder thickness: *2 mm*.
- Argon cylinder diameter: *120 mm*.

Cylinder has collar on both side end for join purpose.

### 4.6 Cylinder head

It is used to close end of argon side cylinder and its dimension are as below:

- Cylinder head thickness: *10 mm*
- Cylinder head diameter: *152 mm*
- Cylinder head internal thread: *M32×1.5* (as same as on gas tube)

## V. CAD DESIGN OF MEASURMENT SYSTEM

The permeation system consists many components inside the argon chamber, so that final design is shown in below figure. The cad model can prepare with the help of software NX-10.

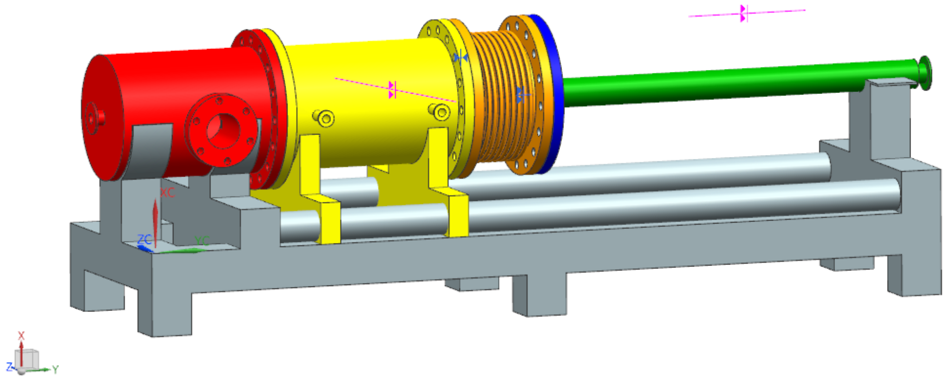

Fig 5.1. CAD model of permeation system (general)

### 5.1 Self-supported type membrane system

In this type of system, sample is sheet or blank type. This type of module chooses for experiment when thickness of membrane is large compare to coating type membrane. Due to enough thickness, membrane can withstand at high temperature and high-pressure difference. In this membrane can fix to flange with helicoflex seal or copper gasket.

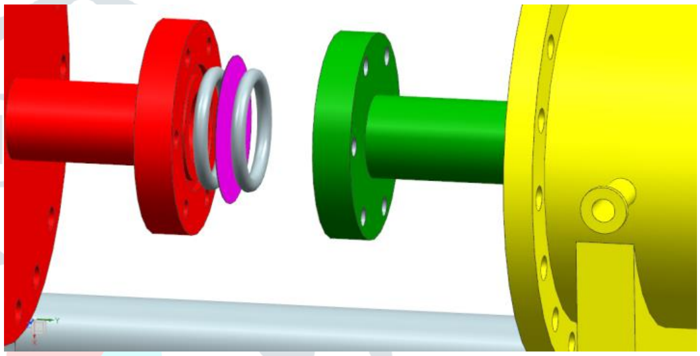

Fig 5.2. Self-supported membrane in measurement system

This group of membrane gives a very high selectivity for hydrogen, however shows low hydrogen fluxes due to small diffusion coefficients for gases in solids. Moreover, for mechanical stability of these membranes, higher thickness (in the 100 µm range) is needed which subsequently results in an expensive noncommercial membrane.

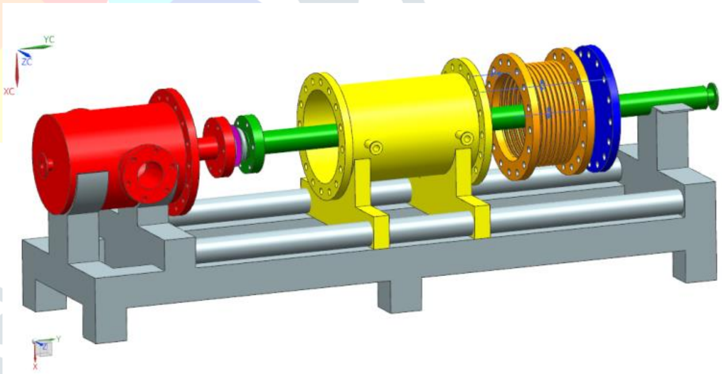

Fig 5.3. Self-supported membrane type measurement system

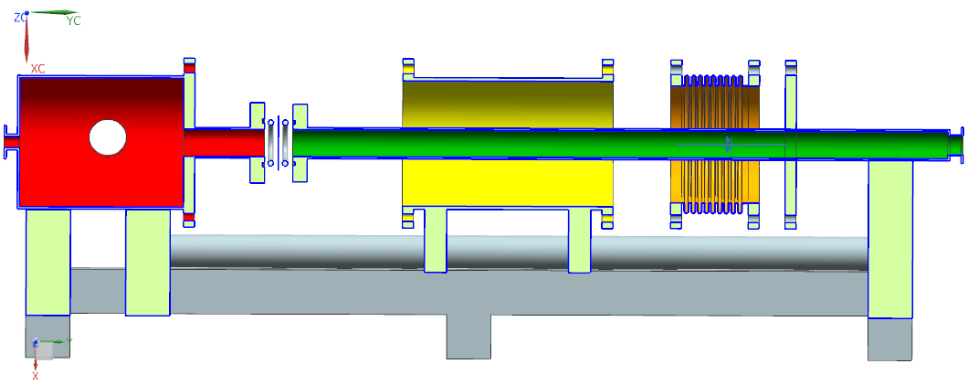

Fig 5.4. Cross-section view of permeation measurement system for self-supported type membrane

**5.2 Composite type membrane system**

This module is used when the film thickness is to small and not enough to withstand high temperature and high-pressure difference. In that case this module is used where support material is used to support the membrane film.

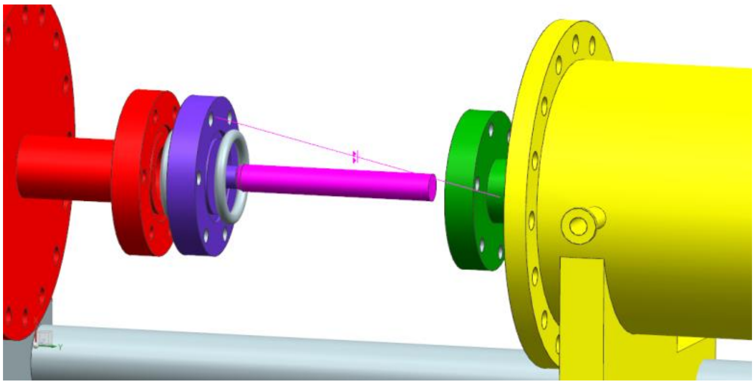

Fig 5.5. Composite membrane in measurement system

The main development for membrane technology was production of composite membranes made of a thin metallic film with thickness in the ranges of 1-10 μm deposited on porous material, Providing both high flux and mechanical strength of support. there is two major porous supports,

1. Porous ceramic supports
2. Porous metallic support

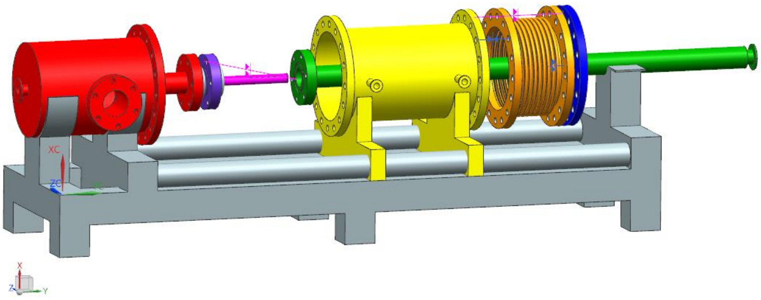

Fig 5.6. Composite membrane type measurement system

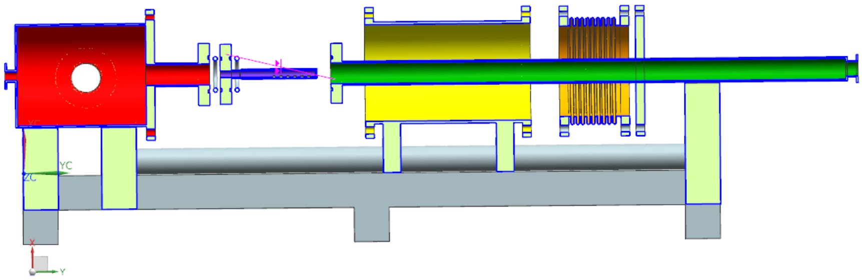

Fig 5.7. Cross-section view of permeation measurement system for composite type membrane

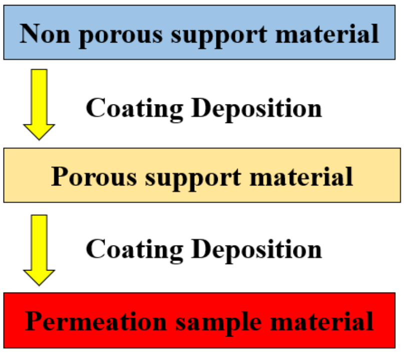


Fig 5.8. Fabrication process of composite membrane

Membrane tube made by coating of sample material on porous support material. It is made by coat the porous support material on SS316 stainless steel tube. This tube with porous material then coat with sample material. It is support tube manufactured with material SS316 Stainless steel. It is tube on which sample can be prepared. Here SS316 is used due to low outgassing properties of material.

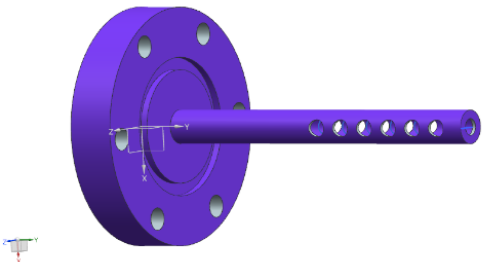

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Fig 5.9. Support tube

Porous material is material with high porosity. It is used as a support material for membrane film to withstand high temperature and high-pressure difference.

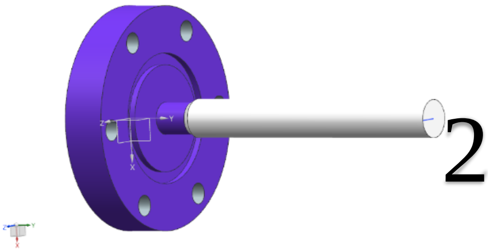

Fig 5.10. Support tube with porous coating

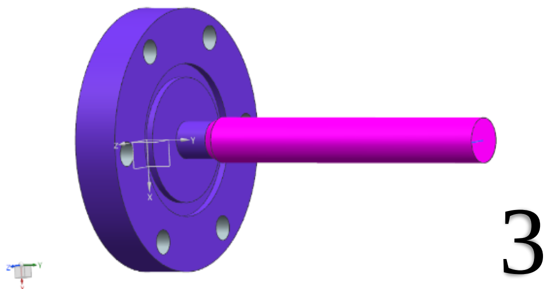

Fig 5.11. Support tube with sample coating on porous material

## VI. STRUCTURAL ANAYLYSIS

Using Siemens NX 10 software a CAD model was prepared, meshing was done and structural analysis has been done with vacuum and ambient pressure difference. Displacement diagram and Von mises stress diagram of all critical component are present as below.

### a) Vacuum chamber

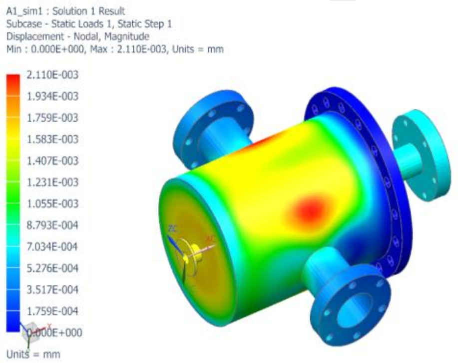


Fig.6.1(a) Displacement analysis

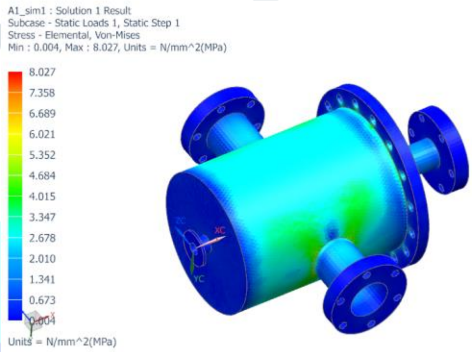


Fig.6.1(b) Von-mises stress analysis

### b) Gas pipe

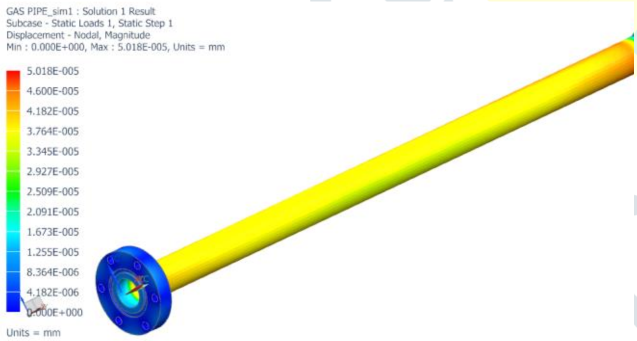


Fig.6.2(a) Displacement analysis

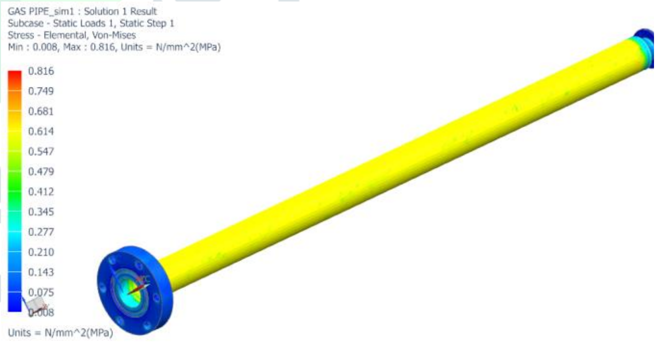


Fig.6.2(b) Von-mises stress analysis

### c) Self-supported membrane

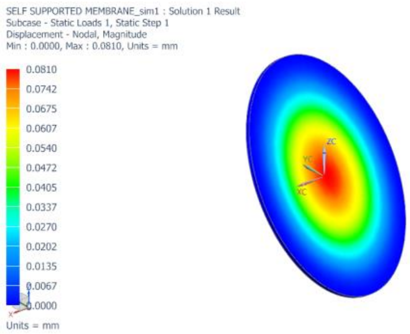


Fig.6.3(a) Displacement analysis

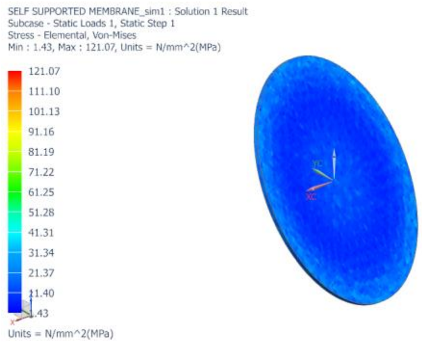


Fig.6.3(b) Von-mises stress analysis

**d) Outer cylinder**

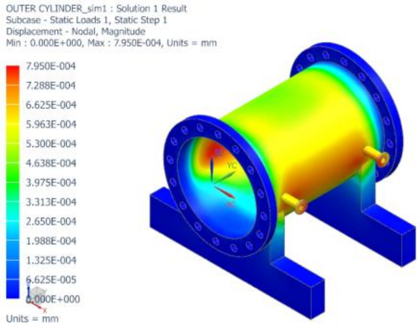


Fig.6.4(a) Displacement analysis

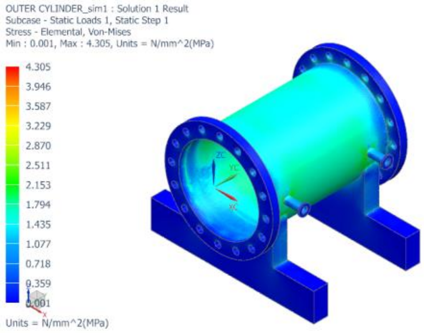


Fig.6.4(b) Von-mises stress analysis

**e) Bellows**

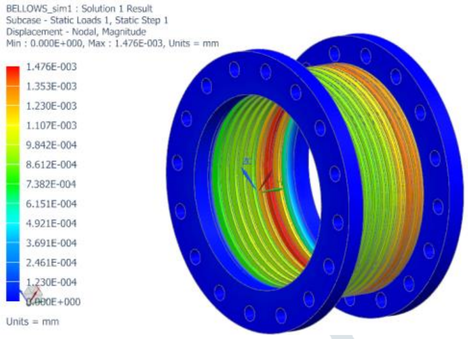


Fig.6.5(a) Displacement analysis

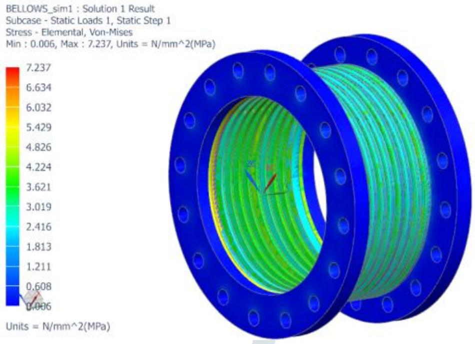


Fig.6.5(b) Von-mises stress analysis

**f) Head plate**

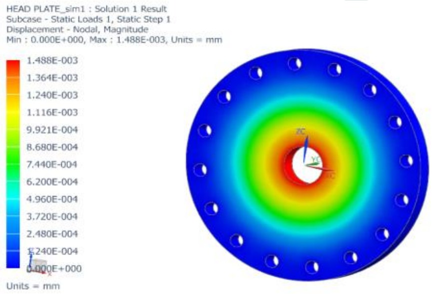


Fig.6.6(a) Displacement analysis

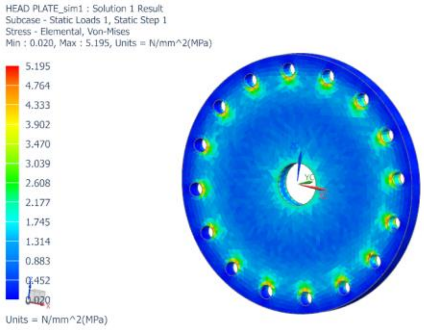


Fig.6.6(b) Von-mises stress analysis

## VI. CONCLUSION

In the paper, the design of a permeation measurement system has been presented, to analyze and tested for Ultra-high vacuum and high temperature. The system design has been checked by means of:

- An analytical and numerical design calculation done with ASME standards which ensure the legal, regulation and safety.
- A Structural analysis has been done, which guarantee that the measurement system satisfies the design against high temperature and high-pressure difference.
- This experiment set-up will help to calculate correct permeation data by including leakage consideration. The CFD analysis for permeation may be future scope of this study.

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