진공재료의 종류와 부품의 특성 및 제조 관리

2019.06.21

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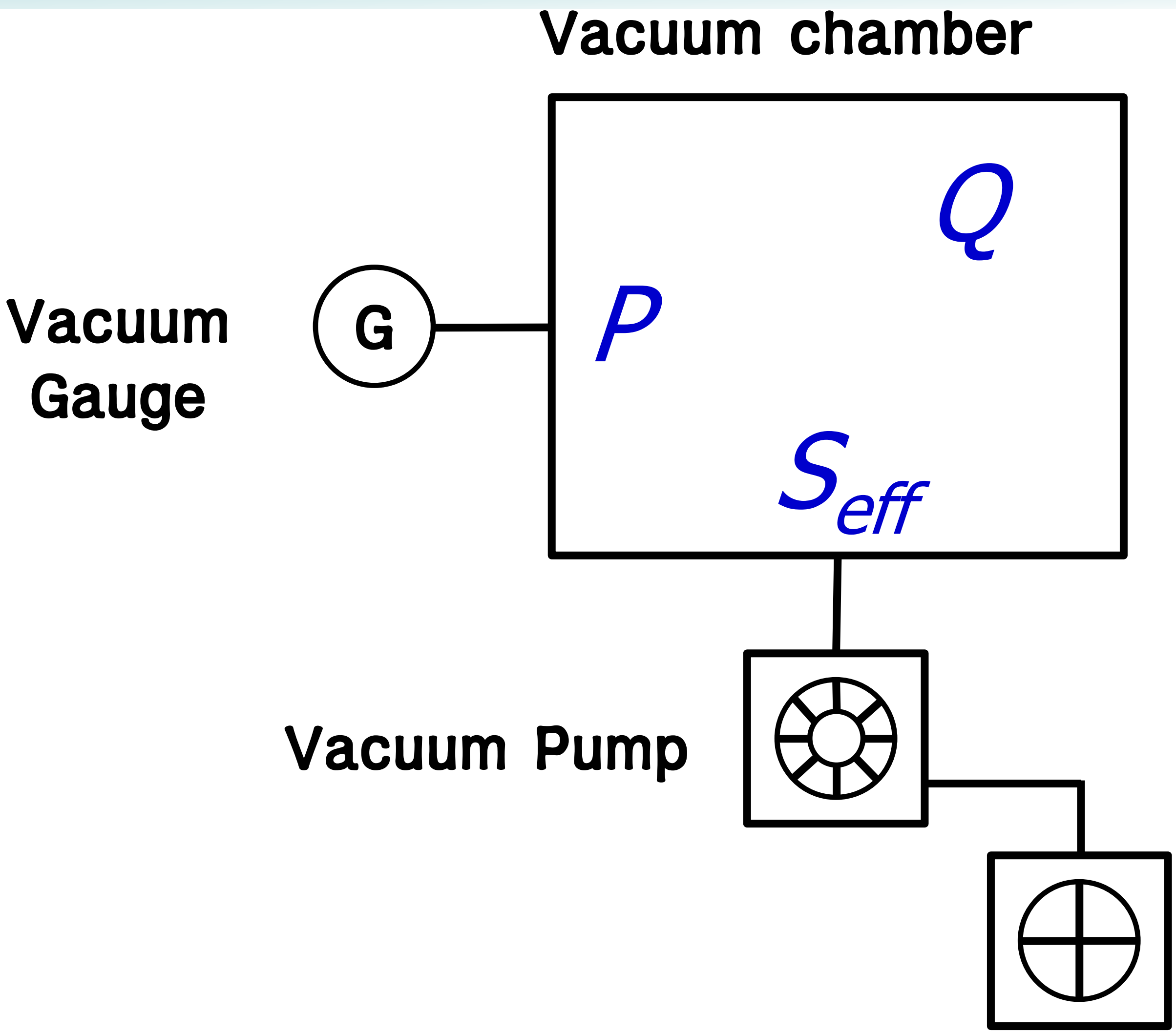
포항가속기연구소

1. 진공 재료의 특성 이해

2. 진공 재료의 성능 향상


Vacuum chamber
Q
Vacuum
Gauge
G
P
S_{eff}
Vacuum Pump

왜 재료가 중요한 가?

System base pressure

$$P \text{ (mbar)} = \frac{Q}{S_{eff}} \quad \frac{(\text{mbar} \quad \ell/\text{s})}{(\ell/\text{s})}$$

$$\frac{1}{S_{eff}} = \frac{1}{S} + \frac{1}{C}$$

목표진공도에 따라 재료가 달라짐

1013 mbar	Rough	**Absolute pressure** Viscous (gas-gas interaction) Volume gas pumping	Vacuum cleaner
1	Medium	**Gas density** Transition Gas conduction	Vacuum packing Vacuum drying
E-3	High	**Mean free path > chamber size** Molecular (gas-surface interaction) Surface pumping	Vacuum tube Thin film deposition
E-7	Ultra high	**Monolayer formation time > min**	Surface science
E-11	Extreme high	**Mean free time > min**	Atomic manipulation

Materials

for

Vacuum technology

진공재료 선택

재료 선택시 고려할 내용 | 관련 항목

- 얻고자 하는 진공도는?

- 적절한 기체방출률
  - ✓ 전처리 필요여부
  - ✓ 세척 방법
- 고유 증기압
- 적절한 녹는점과 끓는점
- 재료의 누출률
- 적절한 투과율
- 불순물 기체 함유량
- 깨끗한 표면

진공재료 선택

재료 선택시 고려할 내용 / **관련 항목**

- 강도는 충분한가?
- 제작은 용이한가?
- 기밀방법은?
- 사용환경과 수명은?

- **1 기압차 유지**
  - ✓ **허용 응력과 허용 변형**
- **기계가공성, 성형성**
- **적절한 접합법**
- **금속 또는 엘라스토머**
- **내부식성(내화학성)**
- **내방사성**
- **열변형(적절한 열팽창 거동)**

진공재료 선택

재료 선택시 고려할 내용	관련 항목
• 사용온도와 수명은?	• 금속 재료 또는 플라스틱? • 극저온 또는 고온? • 높은 내열피로성
• 투자율은?	• 전자기 특성에 영향
• 전기전도도는?	• 전도체 또는 부도체
• 제작 비용은?	
• 재료 수급성은?	• 국내 또는 국외 수입 • 구입 기간

Vacuum Materials

- *Steels*
- *Stainless Steel*
- *Aluminum (alloy)*
- *Copper (alloy)*
- *Other metals*
- *Ceramics*
- *Glass*
- *Plastics*

Steels

Steels

- Mild/structural steels
  - ✓ Carbon < 0.3 %
  - ✓ outgassing rate
    - ~~q > (20~200)x STS~~
    - → q_{rd} > (20~200)x STS, $q_{H2} \lesssim$ STS
    - ~~HV compatible (10^{-6} mbar), endless emission of CO~~
    - → UHV compatible, $RegGas_{ST} \sim RegGas_{STS}$
  - ✓ Weldable
  - ✓ Easy to corrode
    - ✓ Needs anti-corroding coating
  - ✓ Magnetic
    - ✓ Shielding material for magnetic field

[C.D. Park et al., JVSTA **34**, 021601 (2016)]

Steels

- Mild/structural steels
  - ✓ S235, S355, S20C
  - ✓ UHV compatible
    - plate, pipe, rod
    - ~ 10^{-11} mbar
    - ≲ $5x10^{-12}$ (mbar l/s cm^2) after bake
  - ✓ MV, RV compatible
    - Cast parts; pump and valve housing
    - ~ 10^{-3} mbar

Steels ⇨

- Anti-corrosion
  - ✓ ~300^{o}C
  - ✓ Process

- ~~Low *q*~~
  - ~~UHV~~
  - ~~XHV~~

⇨ Stainless steels

Stainless steel

- AISI 304/316
  - ✓ Austenitic
    - High strength
    - Non-magnetic (but, not entirely)
    - Good weldability
  - ✓ Corrosion resistance
    - During vacuum processing and bakeout (~ 300^{o}C)
  - ✓ UHV/XHV compatible
    - < 10^{-11} mbar
    - (2~6) x10^{-12} (mbar l/scm^2) after bake
    - < 1x10^{-13} (mbar l/scm^2) after special treatment

Stainless steel

18-8 Steel Family

Stainless steel

- Role of ingredients
  - Cr(10%) Resistance to oxidation
  - Ni(8%) Austenitic structure/ Anticorrosion
  - Mo Accelerates passivating film formation
  - W Mechanical resistance at high temperature
  - Ti During welding and cycles stabilizes the austenitic structure
  - N Mechanical characteristics

Stainless steel

- 303
  - ✓ 19% Cr, 10% Ni, 0.15% C, 0.15% S
  - ✓ A free machining stainless
  - ✓ Not suitable for UHV applications
    - ✓ Emission of sulfur at higher temperature
    - ✓ (The q of 303 can be lowered to 10^{-13} mbar l/s·cm^2 by a combi nation of fabrication and post treatments such as bakeout.)
  - ✓ Welding is a problem due to the evolution of sulfur during welding causing porosity.

Stainless steel

- 304
  - ✓ 18% Cr, 8% Ni, 0.08% C
  - ✓ Most common materials used in vacuum technology

- 304L
  - ✓ 18% Cr, 8% Ni, 0.03% C
  - ✓ One of common steels used in vacuum technology
  - ✓ Less carbide precipitation
  - ✓ Cleaner machining and better welds than 304
  - ✓ Lower mechanical properties than 304

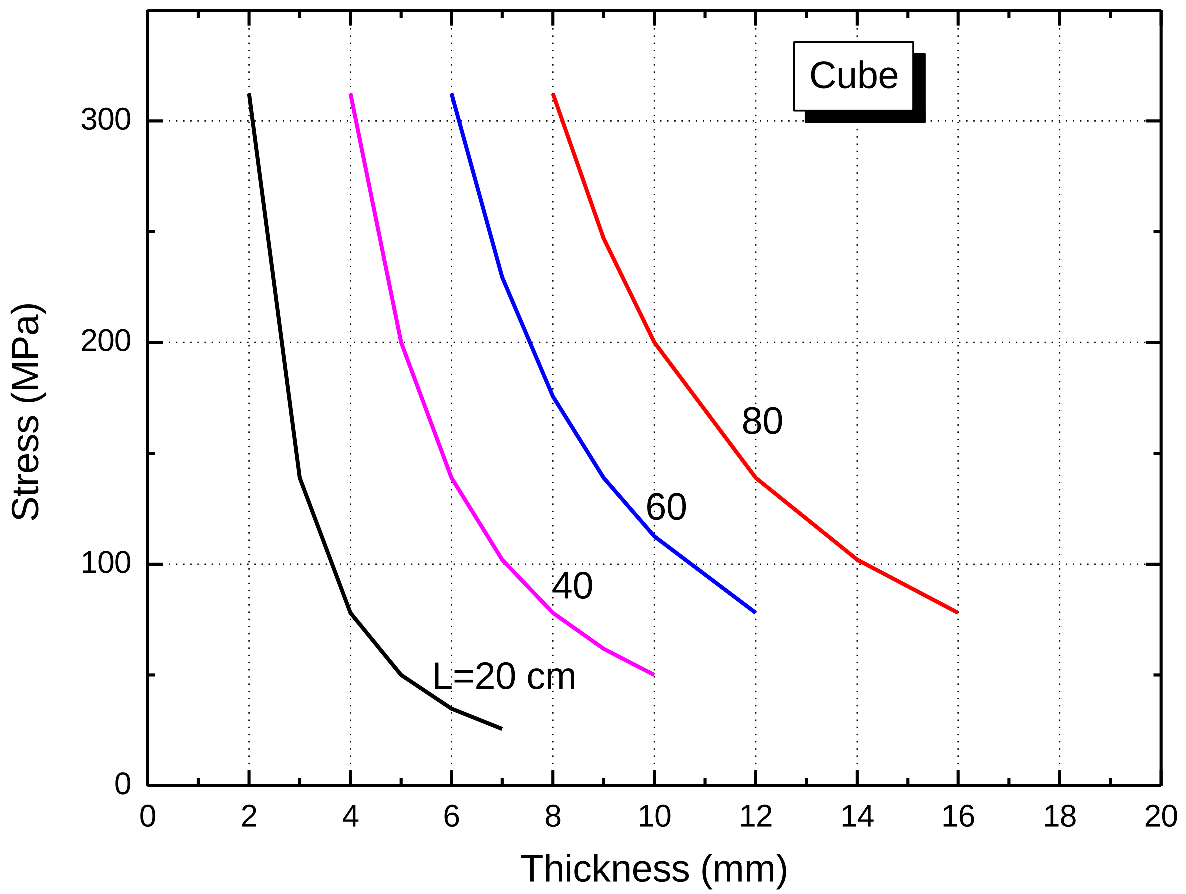
Stainless steel

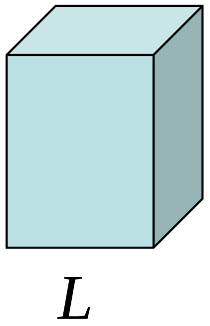
- 316L
  - ✓ 18% Cr, 14% Ni, 0.03% C, 3% Mo
  - ✓ Stabilized with molybdenum to prevent carbide preci pitation in the weld zone
  - ✓ Lower *q* than 304(L)
    - ~2 $\times 10^{-12}$ (mbar l/scm^2) after bake
    - < 5×10^{-14} (mbar l/scm^2) after special treatments
  - ✓ Used where chemical compatibility is a concern.
  - ✓ Low-magnetic stainless steels ($\mu_r < 1.02$)
    - Suitable for analyzers, accelerators
      - Heat treatment; (750 ~ 1050°C)

Mechanical properties

	Yield strength (0.2%)	Tensile strength
	MPa	MPa
316	206.8	517.1
316L	172.4	482.6
A6061-T6	241	289.4
A6063-T5 - T6	110	152

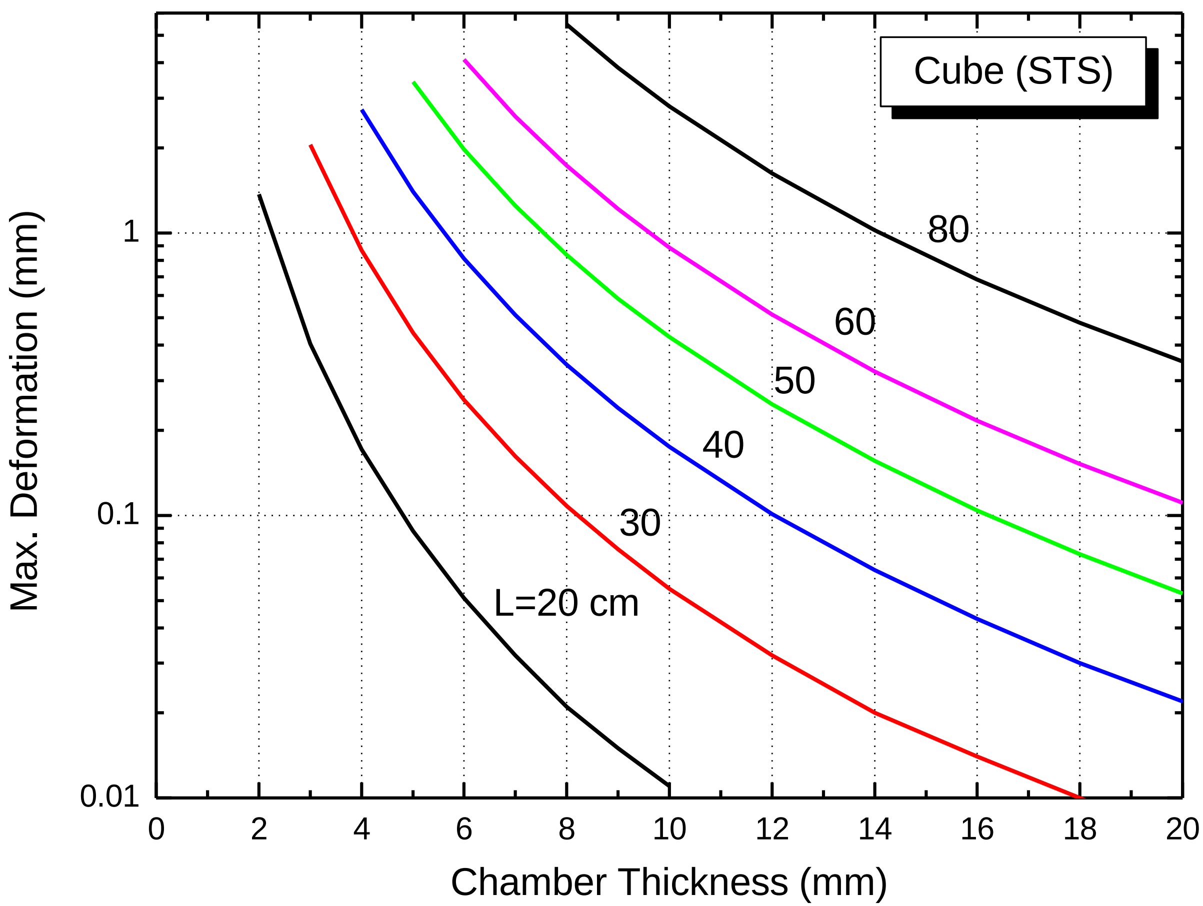
Stress : Cube

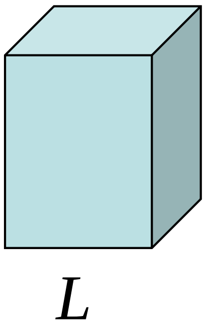




→ "진공공학(인상렬 외)"

Deformation : Stainless steel





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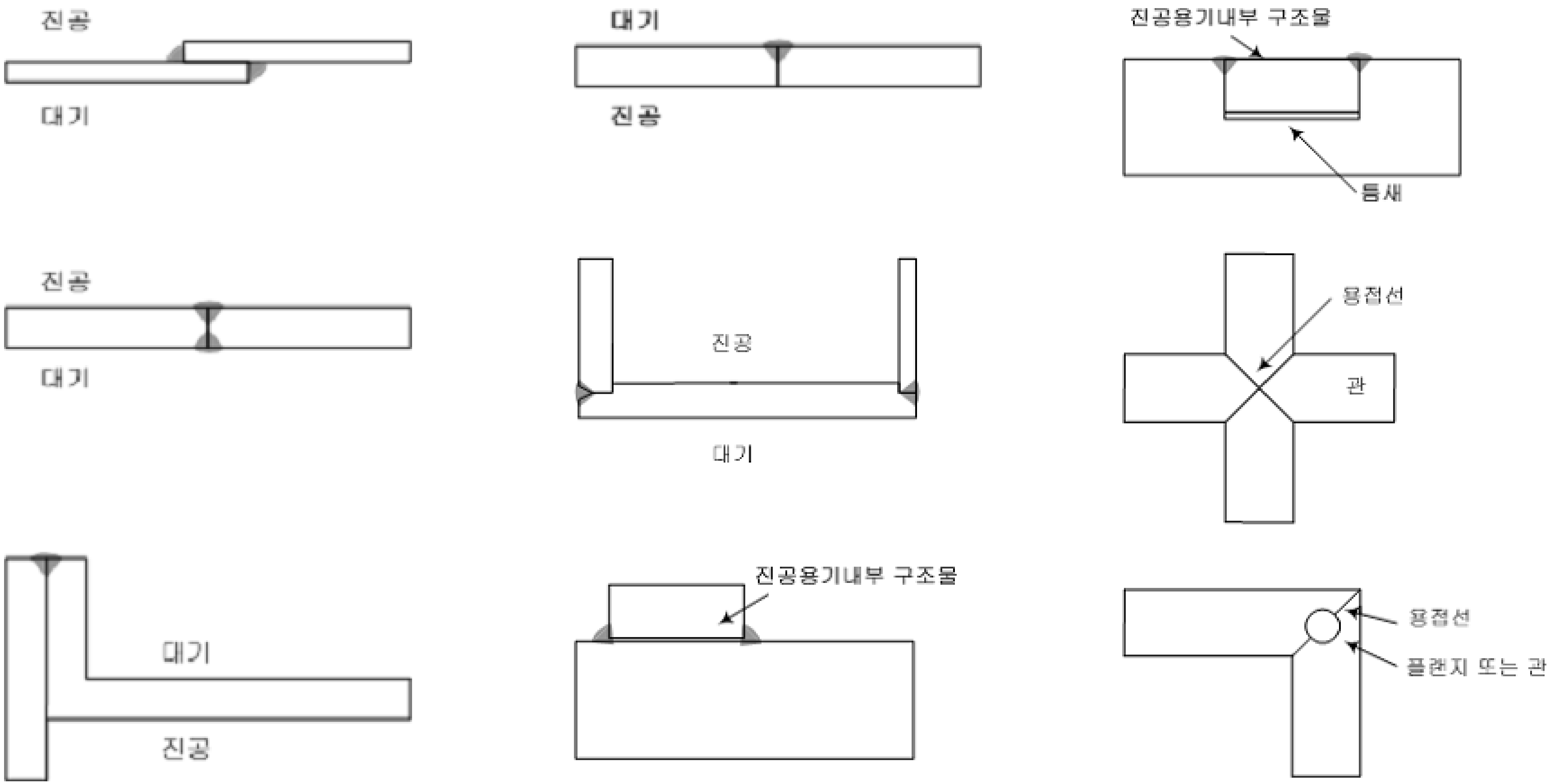
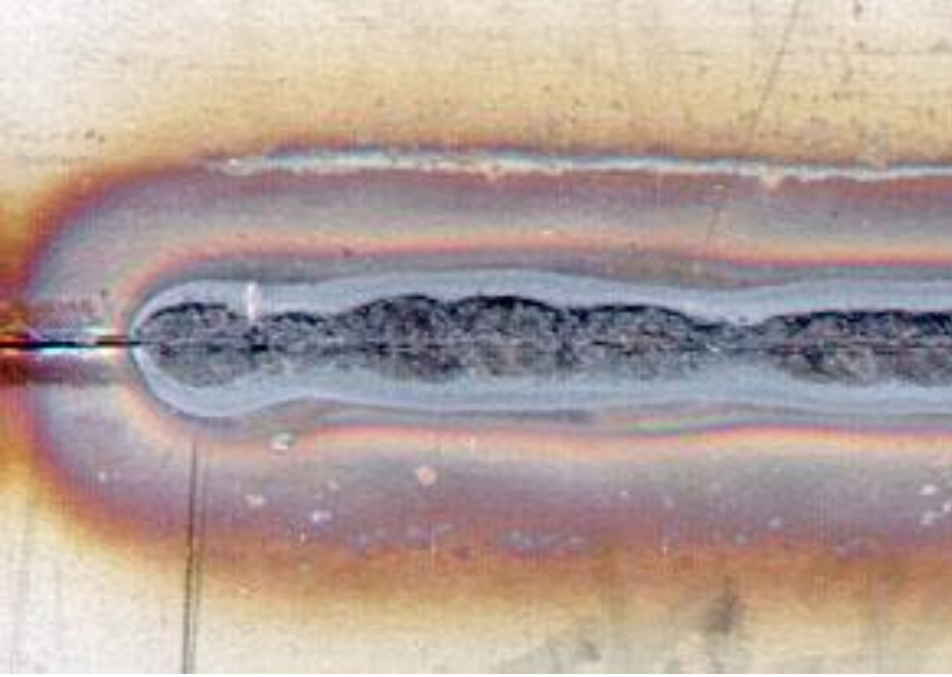
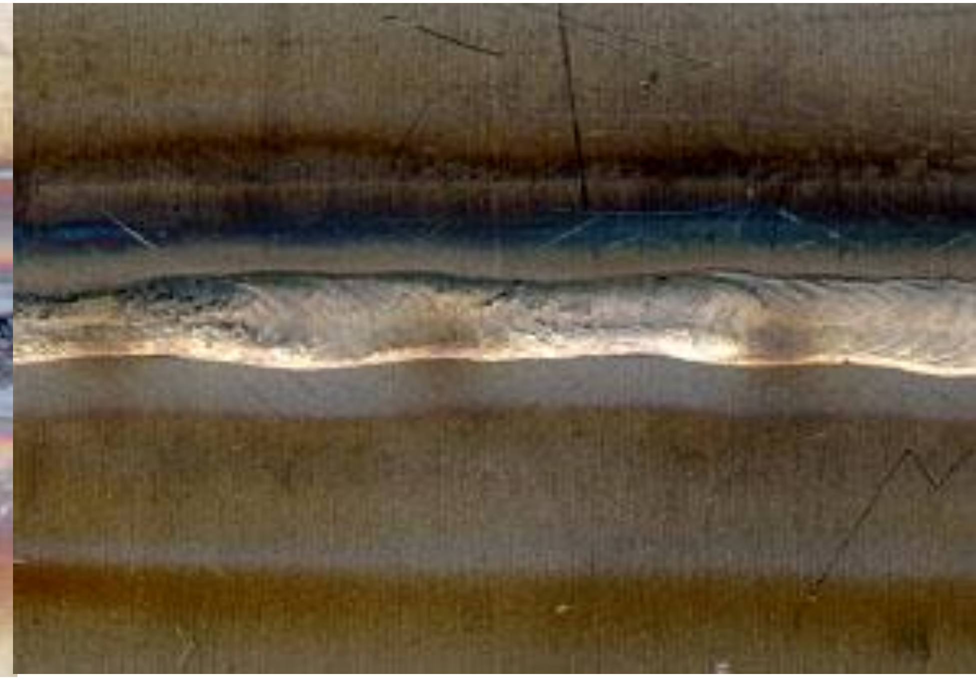
피해야 하는 용접

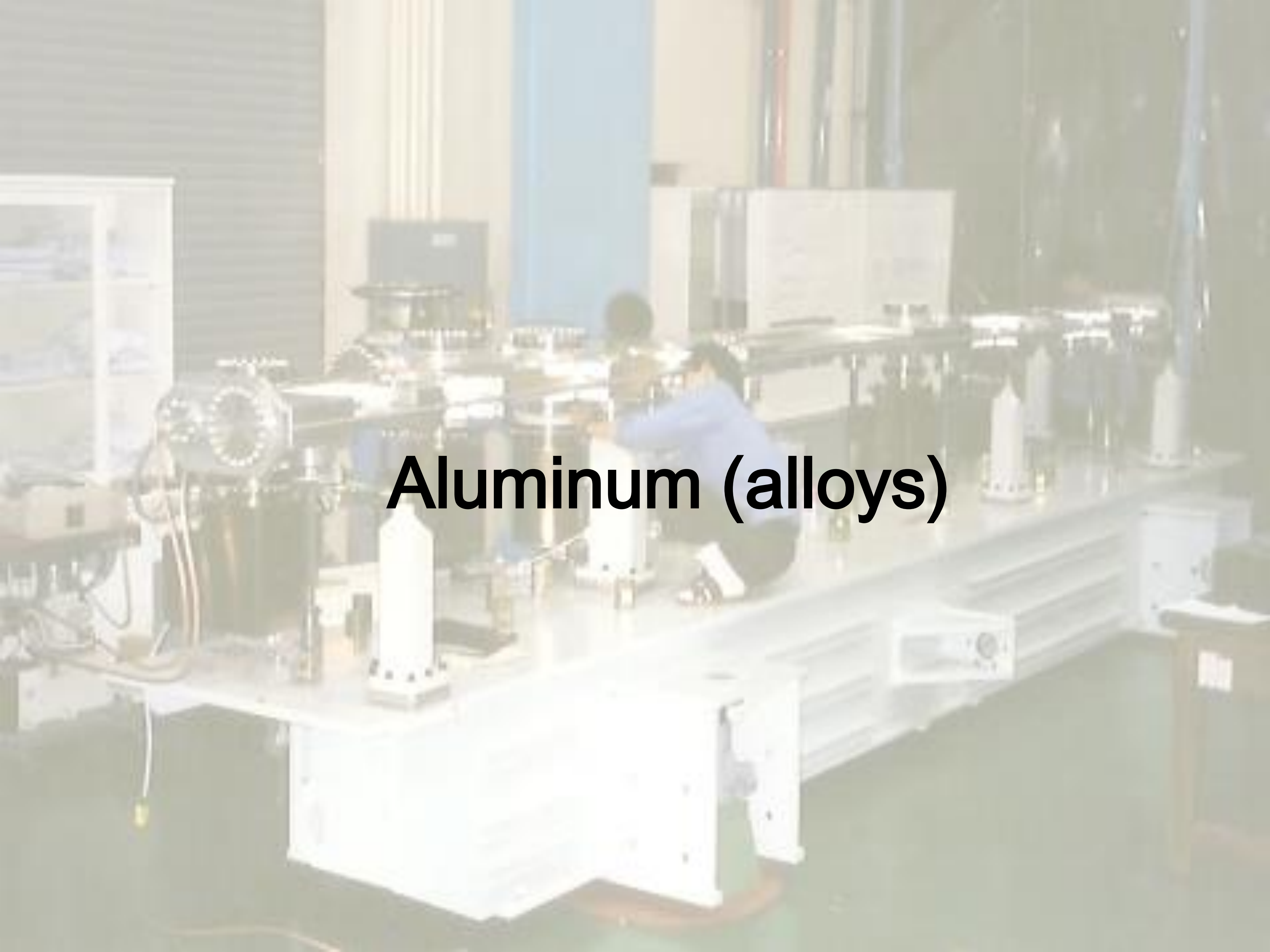


→ "초고진공 용접핸드북(KRISS)"

용접 품질







Aluminum (alloys)

Aluminum (alloys)

- 1xxx pure aluminum
  - ✓ > 99% Al by weight
  - ✓ A1050; suitable for metal gaskets

- 2xxx copper alloys
  - ✓ Duralumin; once the most common aerospace alloys (they were susceptible to stress corrosion cracking and are increasingly replaced by 7000 series in new designs.)
  - ✓ A2219; suitable for Conflat flange (weldable)

Aluminum (alloys)

- 3xxx manganese alloys
  - ✓ A3004; suitable for vacuum bellows
- 5xxx magnesium alloys
  - ✓ easy to machine, higher strength, good weldibility
  - ✓ A5083; for a large scale chamber
- 6xxx magnesium and silicon alloys
  - ✓ Easy to machine and extrude
  - ✓ A6063; most common materials in vacuum technology
  - ✓ A6061; one of the most vacuum materials
  - ✓ A6060; extrusion

Aluminum (alloys)(진공용)

재 료	처 리	주요 합금성분 (%)		용 도
2219	T87, T852	Cu 5.8-6.8	Mn 0.2-0.4	플랜지
3004		Mn 1-1.5	Fe 0.25	벨로우즈
5052		Mg 2.2-2.8	Fe 0.4	진공용기, 벨로우즈
5083	H321	Mg 4-4.9	Mn 0.4-1	진공용기
6061	T5, T6	Mg 0.8-1.2	Si 0.4-0.8	진공용기, 벨로우즈
6063	T5, T6	Mg 0.45-0.9	Si 0.2-0.6	진공용기
6263		Mg	Si	진공부품 (티, 크로스, 엘보)
6951	T6	Mg 0.4-0.8	Si 0.2-0.5	벨로우즈

Tempering

- -F As fabricated
- -H Strain hardened (cold worked) with or without thermal treatment
  - -H1 Strain hardened without thermal treatment
  - -H2 Strain hardened and partially annealed
  - -H3 Strain hardened and stabilized by low temperature heating
- Second digit A second digit denotes the degree of hardness
  - -HX2 = 1/4 hard
  - -HX4 = 1/2 hard
  - -HX6 = 3/4 hard
  - -HX8 = full hard
  - -HX9 = extra hard
- -O Full soft (annealed)

- -T Heat treated to produce stable tempers

  -T5 Cooled from hot working and artificially aged (at elevated temperature)

  A6061 160°C 18 h

  (ex) 160°C for 18 h -> ambient cooling

  170°C for 12 h -> ambient cooling

  175°C for 8 h -> ambient cooling

  205°C for 3 h -> ambient cooling (1.5 h)

  -T6 Solution heat treated and artificially aged

  (ex) ~ 180°C – 220°C for 6 h – 1h -> water cooling

- -W Solution heat treated only.

Aluminum (alloys)

- A6063-T5(T6)(T0)
  - ✓ The most widely used aluminum alloy
  - ✓ The most common materials in vacuum technology
  - ✓ Low outgassing rates
    - < 5×10^{-13} (mbar l/scm^2) after bake (< 180^{o}C)
  - ✓ Easy to forming using ***extrusion***, machining and welding
  - ✓ Alloy retains its strength after welding
  - ✓ ConFlat® flanges are made from A2219 with knife edge coated with TiC (TiN, CrN).
    - ✓ With A1050 metal gaskets

- Mainly used in UHV and forelines
- Bakeout temperature
  - Max 180^{o}C
  - In-general 100 – 150^{o}C
- Melting point; 660^{o}C (P_{vapor}=10^{-8} mbar)
- Entirely non-magnetic
- High thermal and electrical conductivity

Aluminum (alloys)

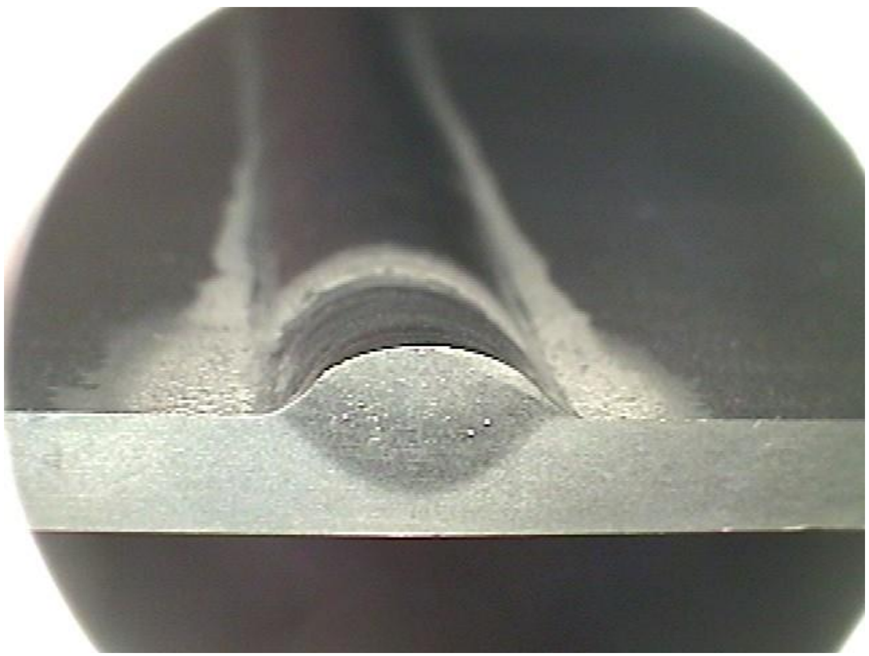
- A6061-T5(T6)
  - ✓ The most widely used aluminum alloy
  - ✓ Low outgassing rates
    - ✓ $< 5 \times 10^{-13}$ (mbar l/scm^2) after bake ($< 180^{o}C$)
  - ✓ Easy to forming using *machining* and welding
  - ✓ Alloy retains its strength after welding
  - ✓ ConFlat® flanges are made from A2219 with knife edge coated with TiC (TiN, CrN).
  - ✓ Large chambers which would be difficult to heat treat in to T6 condition are often made from 5083.

Aluminum (alloys)

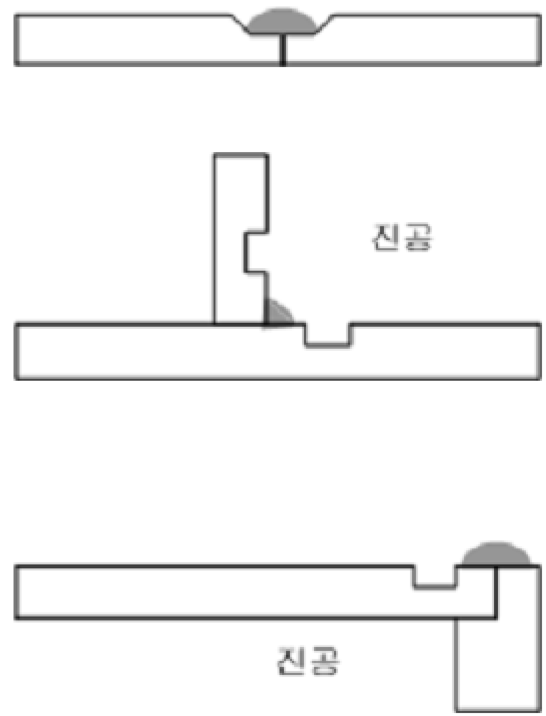
- Initial outgassing rate is higher(~ 5x) than that of SST.
  - ✓ The desorption rate of water vapor from the surface of aluminum is slower than stainless steels which gives it different initial pump down characteristics.
- Anodizing
  - ✓ a common surface treatment.
  - ✓ produces hard inert surface, but q becomes higer, ~ x10.
  - ✓ Not suitable for UHV applications
- ✓ Suface oxidation
  - ✓ Suitable for UHV and XHV applications

알루미늄 용접

- Al alloys require special attention to both weld design and weld technique.



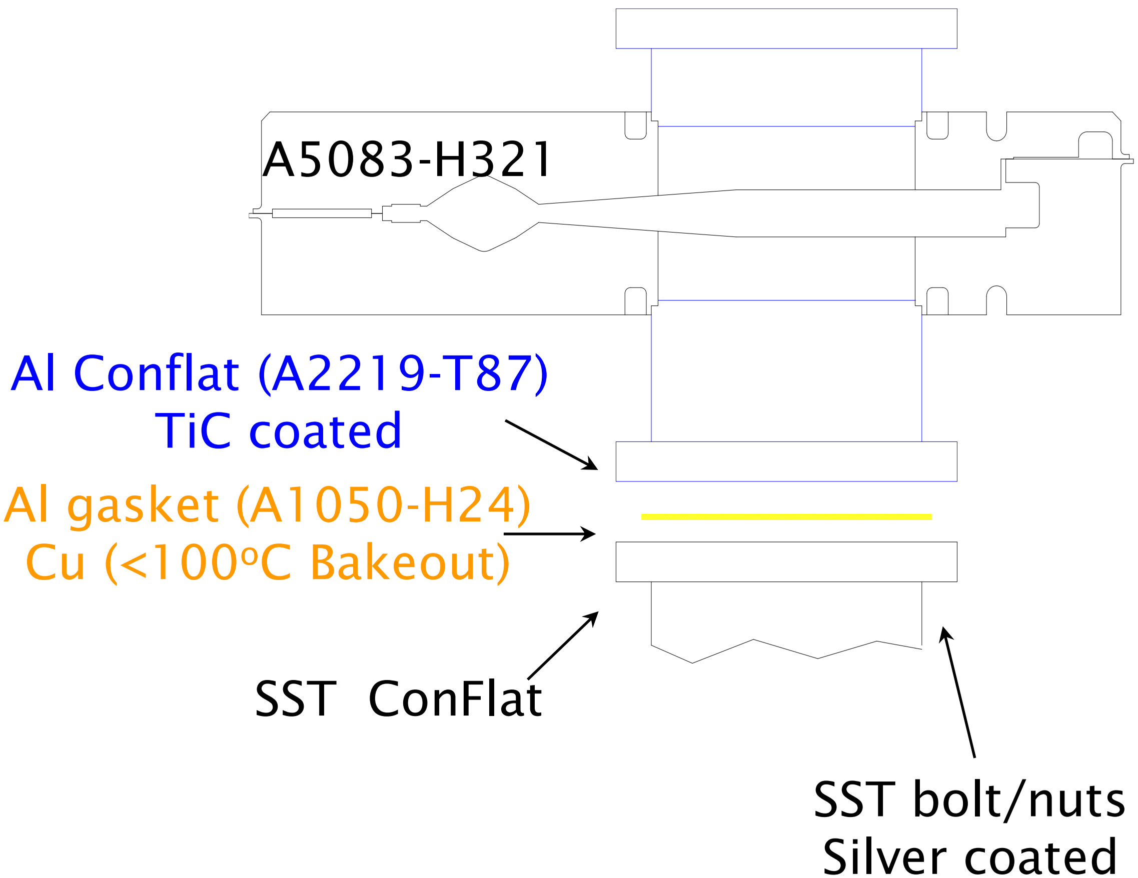
ac-TIG welding
(with filler metal)



→ "초고진공 용접핸드북 (KRISS)"

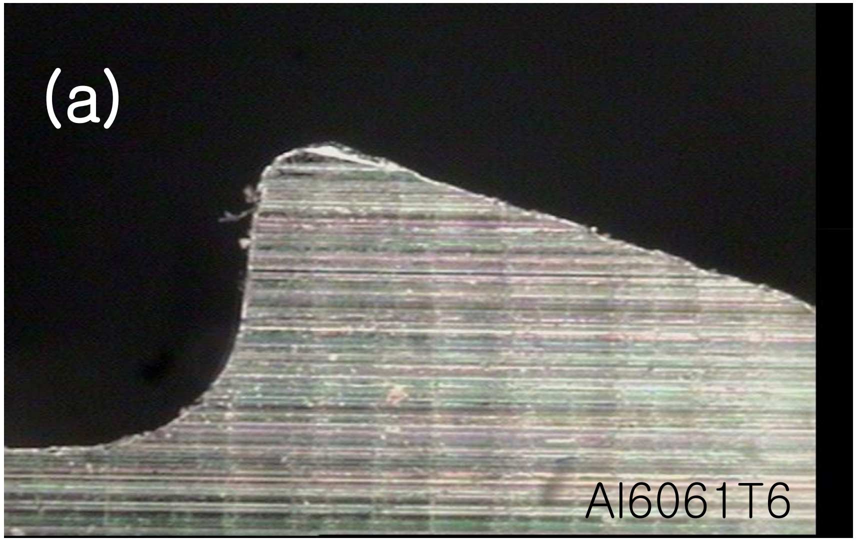
Vacuum Seals

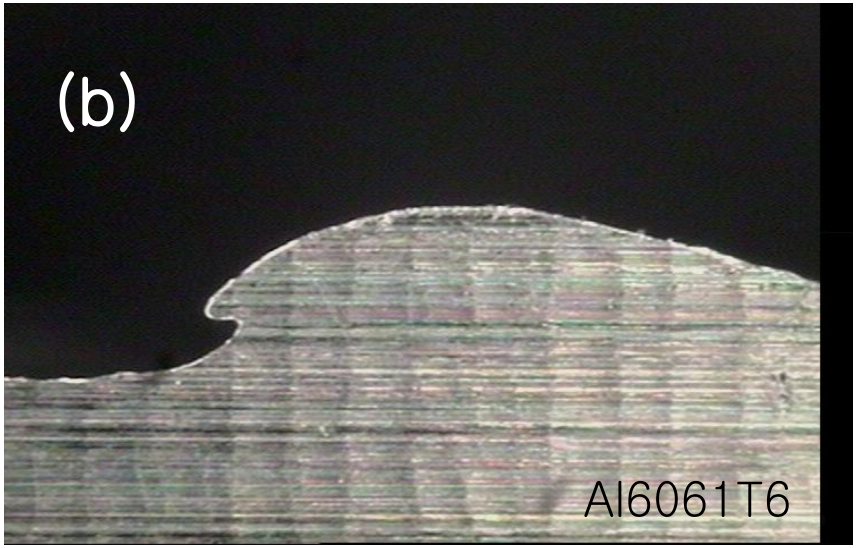
- Al/SST hybrid ConFlat system



Vacuum Seals

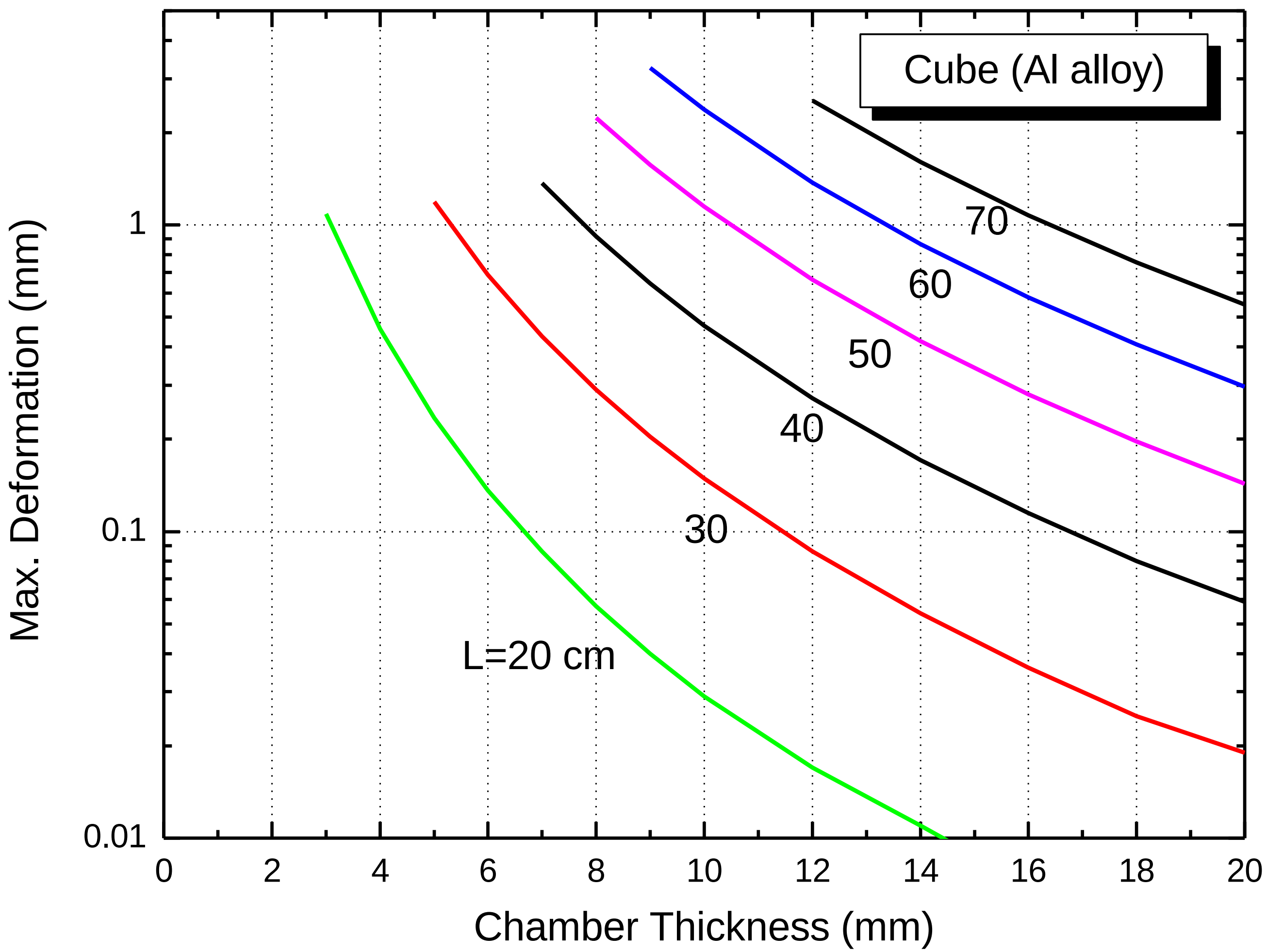
- Al/SST hybrid ConFlat system

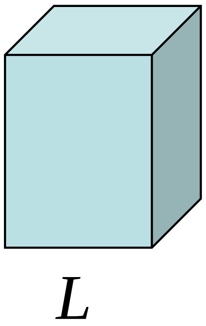




Knife edge of Al flange (before use (a) and after use (100 times) (b)]

Deformation : Al alloy

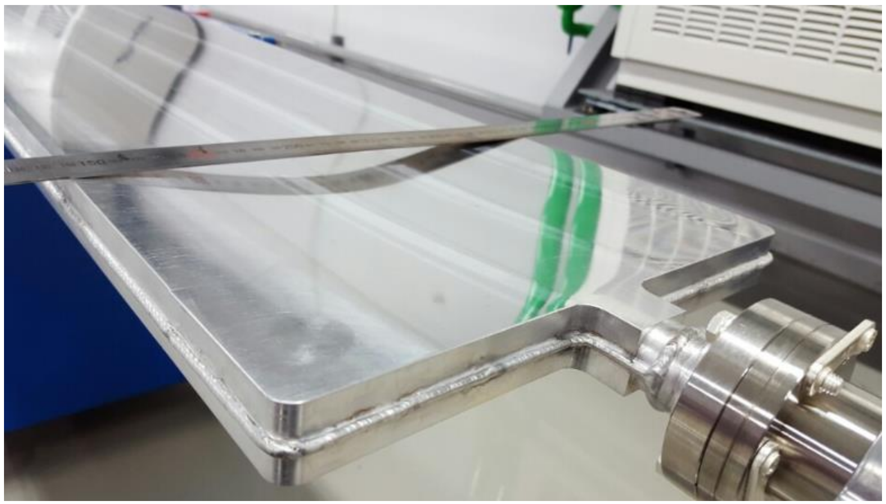




$t << L$

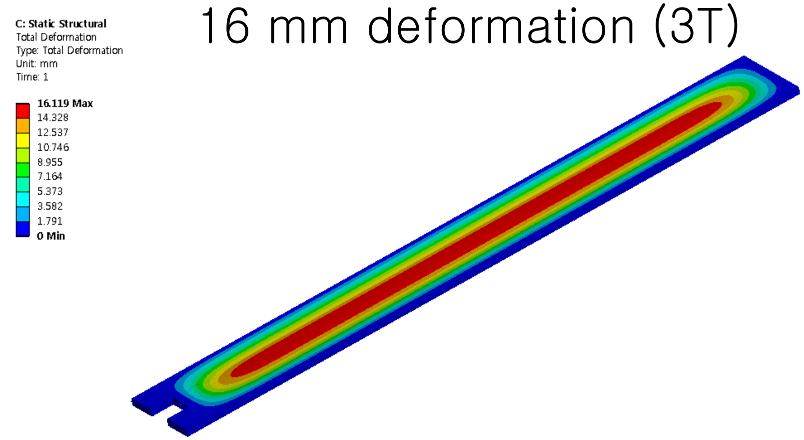
$\delta < t$

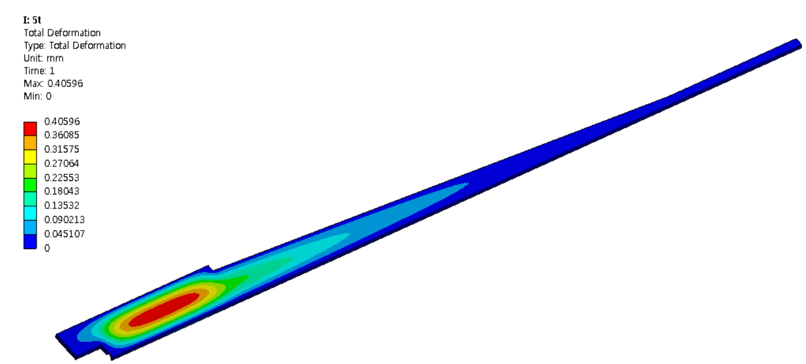
Deformation : Al alloy



(Thickness=3 mm, width = 270 mm)

16 mm deformation (3T)



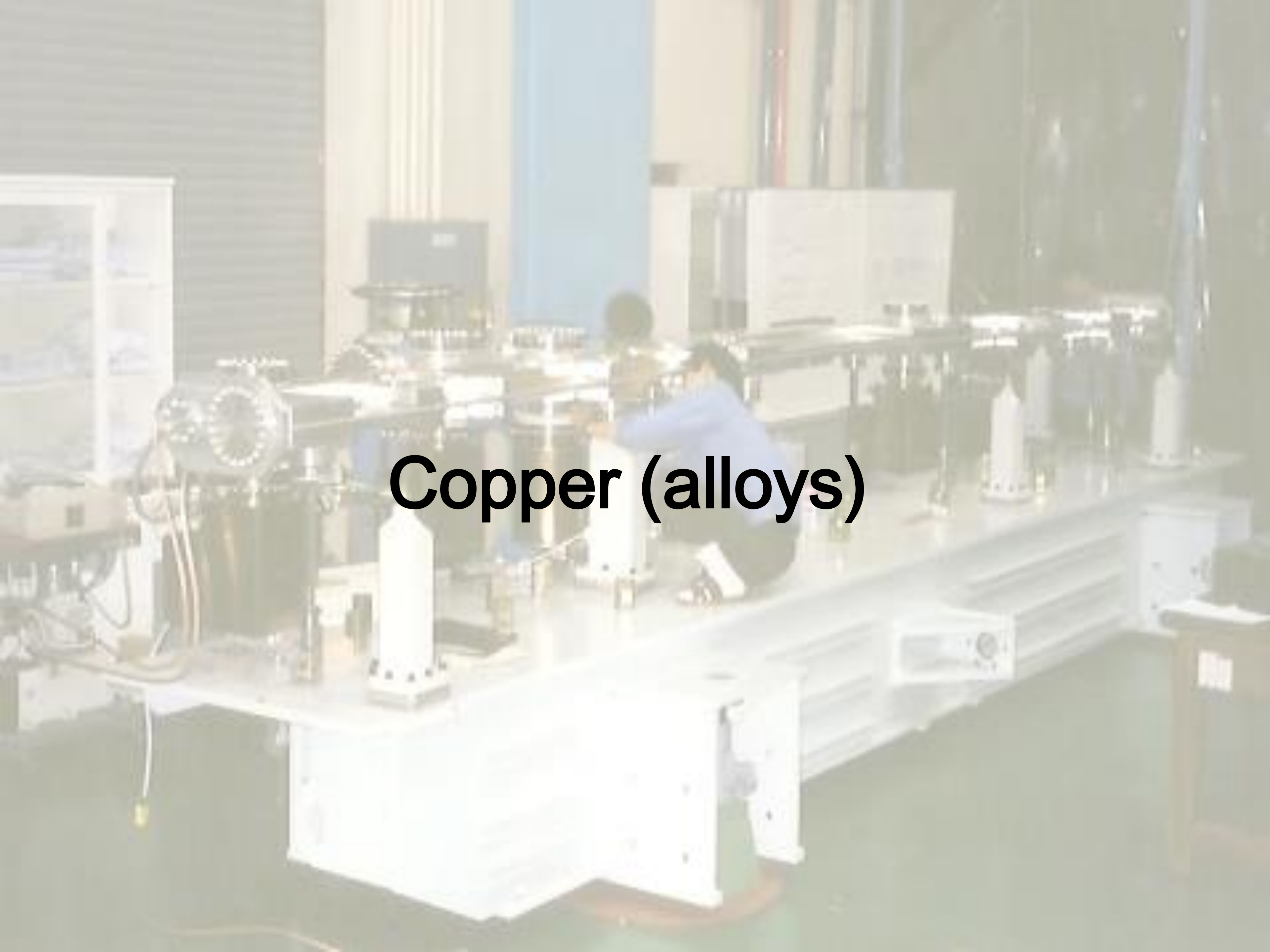


0.4 mm deformation (4T)

SST – Al 비교

		Stainless steel	Aluminum alloy
Vacuum characteristics	Outgassing rate	depends on surface treatments	
	Preinstallation bakeout	950℃	< 210℃
	In situ bakeout	150 - 450℃	< 150℃
Mechanical characteristics	Mechanical strength	higher (×1.5)	
	Thermal expansion coefficient		higher (×2)
	Thermal conductivity		higher (×15)
	Shaping by machining		easier
	Shaping by extrusion		easier
	Shaping by welding	easier	
	Quality of demountable seals	better	
Other characteristics	Magnetic property	not entirely non-magnetic	completely non-magnetic
	Residual radioactivity		lower

Copper (alloys)



Copper (alloy)

- High thermal and electrical conductivity
  - Suitable for electrical feedthrough
  - Suitable for thermal/radiation absorber
  - Suitable for cryogenic applications
- Hydrogen embrittlement
  - For HV and UHV, coppers(alloys) with oxygen free or reduced oxygen contents are required.
- Bakeout; Up to 300^{o}C in vacuum
- Cold welding; OFHC gasket for ConFlat flanges
- Joining techniques; brazing, soldering, welding
- Outgassing rate; ~ 10^{-9} mbar liter/sec cm^2

Copper alloy

Brass(Zinc alloy) and Bronze(Tin alloy)

- High vapor pressure at high temperatures
  (Used in vacuum systems where $T < 100^{o}C$)
  - Easy to machine
  - Cheap
- Commonly used in rough and high vacuum chambers and fixtures.
- Common joining techniques: Soldering
- Outgassing rate
  $\sim 10^{-7}$ mbar liter/sec cm^2

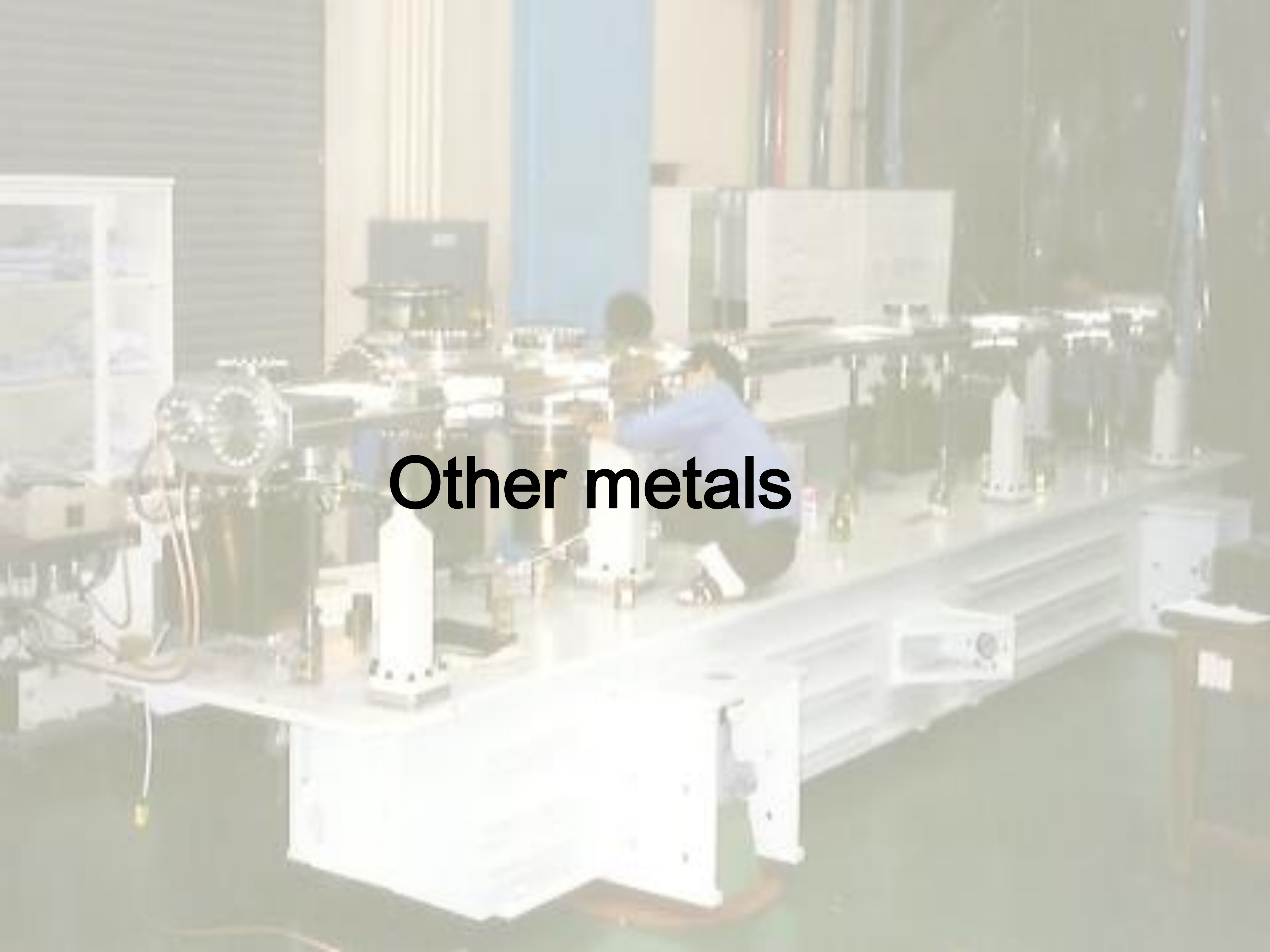
Copper alloy

- To increase its strength
  - OFC + Al_2O_3(0.1-0.5%) GlidCop
    - Yield strength(at 0.2% offset) > 200 MPa (OFC < 100 MPa)
  - OFC + Ag or $Au_{0.2\%}$ Expensive (x 4)
  - OFC + Zr High outgassing rate
  - OFC + Be Brazing(X), EBW(O)
  - OFC + Cr

Outgassing rates

q(mbar l/s cm^2)	@ 10 hour	Baked
Aluminum alloy	$5x10^{-8}$	$< 5x10^{-13}$
Aluminum(anodized)	$3x10^{-7}$	$5x10^{-10}$
Stainless steel	$1x10^{-8}$	$(2-6)x10^{-12}$
Mild Steel	$2x10^{-7}$	$< 1x10^{-12}$
Brass	$6x10^{-7}$	
Copper	$5x10^{-9}$	$1x10^{-12}$
Copper (OFHC)	$2x10^{-9}$	$<1x10^{-12}$

Other metals



Gold and Silver

- Gold
  - ✓ Very low vapor pressure
  - ✓ Used as metal gaskets, surface seals in valves
    as coating for electrical conductors
  - ✓ Used as brazing filler alloys
    - ✓ Cu/Au or Cu/Au/Pd
- Silver
  - ✓ Very low vapor pressure
  - ✓ Silver plated bolts/nuts to reduce friction/cold weld.
  - ✓ High oxygen permeation rate through silver at high temperature.

Titanium

- Very active metal
- Easily react with O_2, N_2 at > 150^{o}C
  - ✓ Weld should be done with inert gas environment
- Used as *metalizing materials* for brazing
- *TSP*(Titium sublimation pump)
  - ✓ Sublimation at ~ 1,350^{o}C
  - ✓ ~ 5 um coating for 1 hr
- *Ion pump*: Cathode material
- *NEG alloy (Ti-Zr-Fe)*
- *Chamber materials:* Very low outgassing rate

Indium

- Melting point; 156^{o}C
  - Not suitable for bakeable UHV applications
- Very low vapor pressure
- Very soft
- High thermal conductivity

- Thus indium may be used as
  - ✓ *vacuum seal* for UHV at cryogenic applications
  - ✓ *thermal conductors* between two different metals

Ceramics

Ceramics

- Ceramics
  - ✓ Non-metal, inorganic materials
  - ✓ Mainly used as insulators in vacuum technology
- Three types of ceramics
  - ✓ Pure-oxide ceramics
  - ✓ Silicate ceramics
  - ✓ Glass-ceramics

Ceramics

- Pure oxide ceramics
  - ✓ Alumina, Zrconia, Beryllium oxide,…
  - ✓ Alumina (Al_2O_3)
    - Mostly used ceramics
    - Max temperature; 1,800°C
    - > 92% in vacuum technology
    - Can be brazed
    - Mainly used as electrical *feedthroughs/insulator*
      - Bakable upto 350-550°C
    - Tensile strength 25 kpsi (96% density)
  - ✓ Sapphire (monocrystalline Al_2O_3)
    - UV and IR transparent
    - Used as *vacuum window*

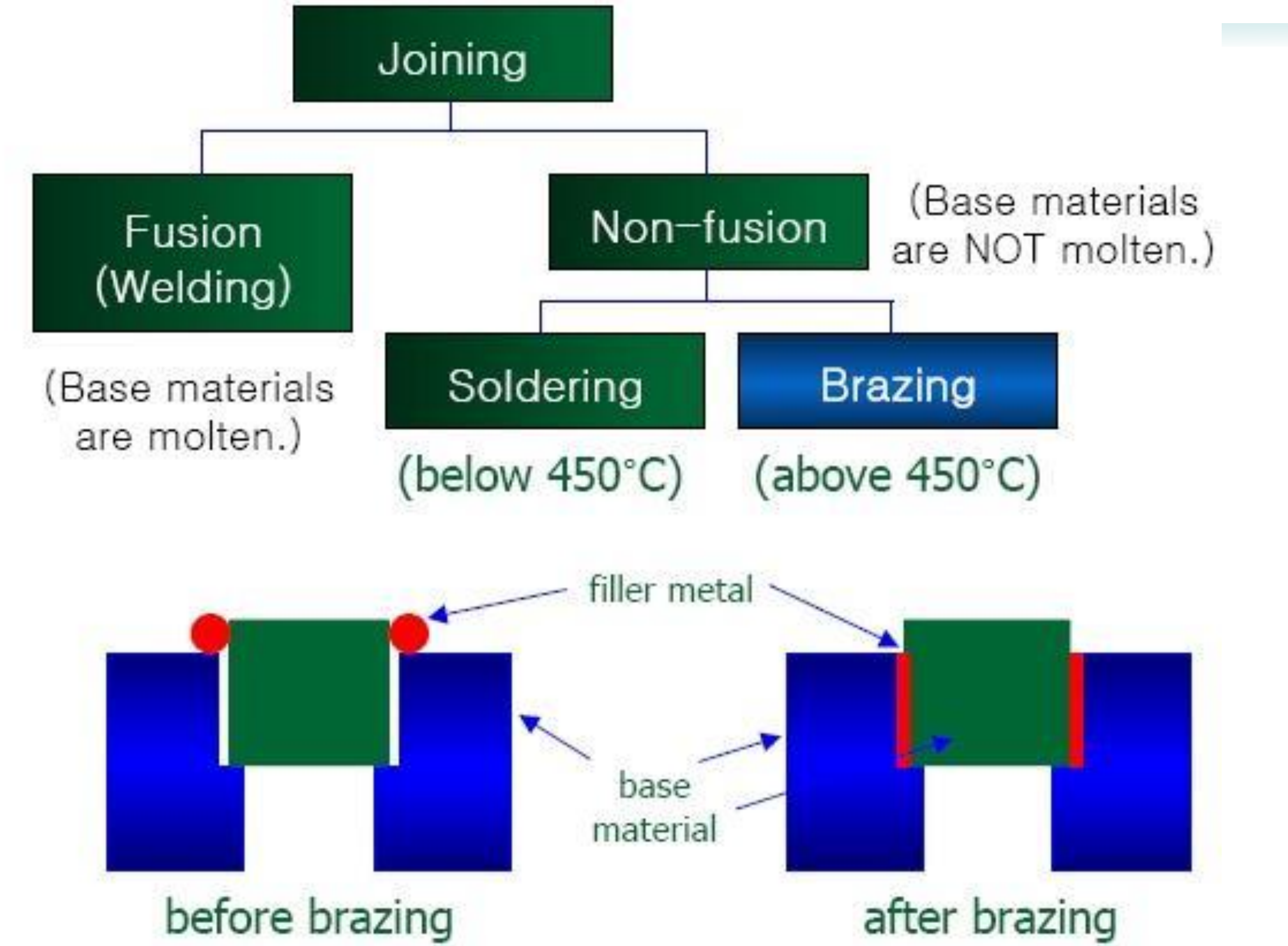
Ceramics

- Silicate ceramics
  - ✓ Steatite ($MgO\text{-}SiO_2$)
    - Max temperature 1,000^{o}C
    - Tensile strength 15 kpsi
- Glass-ceramics
  - ✓ Crystalline ceramic
    - Can be machined with standard tools
    - Macor®, Corning 9658

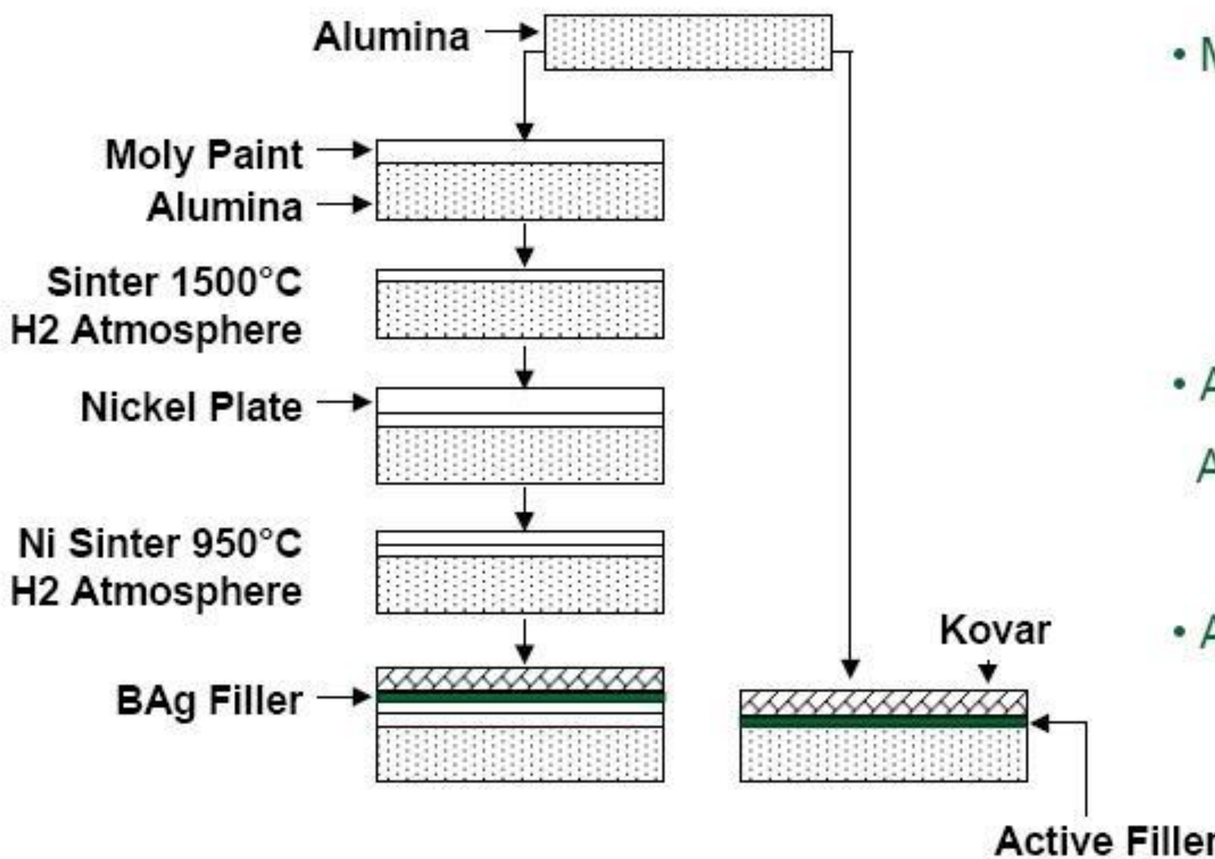
Ceramics

- Kovar
  - ✓ Fe-Ni-Co alloy (thermal expansion ~ glass)
  - ✓ Magnetic
  - ✓ Intermediate material between ceramic and metal for brazing joint

Permanent vacuum joints



Brazing



- Metallization Method
  - Mo/Mn Paint Sintering
  - Vapor Deposition
- Active Brazing

  $Al_2O_3 + 3Ti \rightarrow 2Al + 3TiO$
- Active Soldering
  - Brushing, Vibration required
  - No Spreading

Zeolite

- Very porous alumina silicates with alkali metals
- Mainly used as drying agents
- Mostly used as vapor adsorbing agents in vacuum
- *Sorption pump*
  - Liquid nitrogen cooled
  - Regeneration by heating

Glass

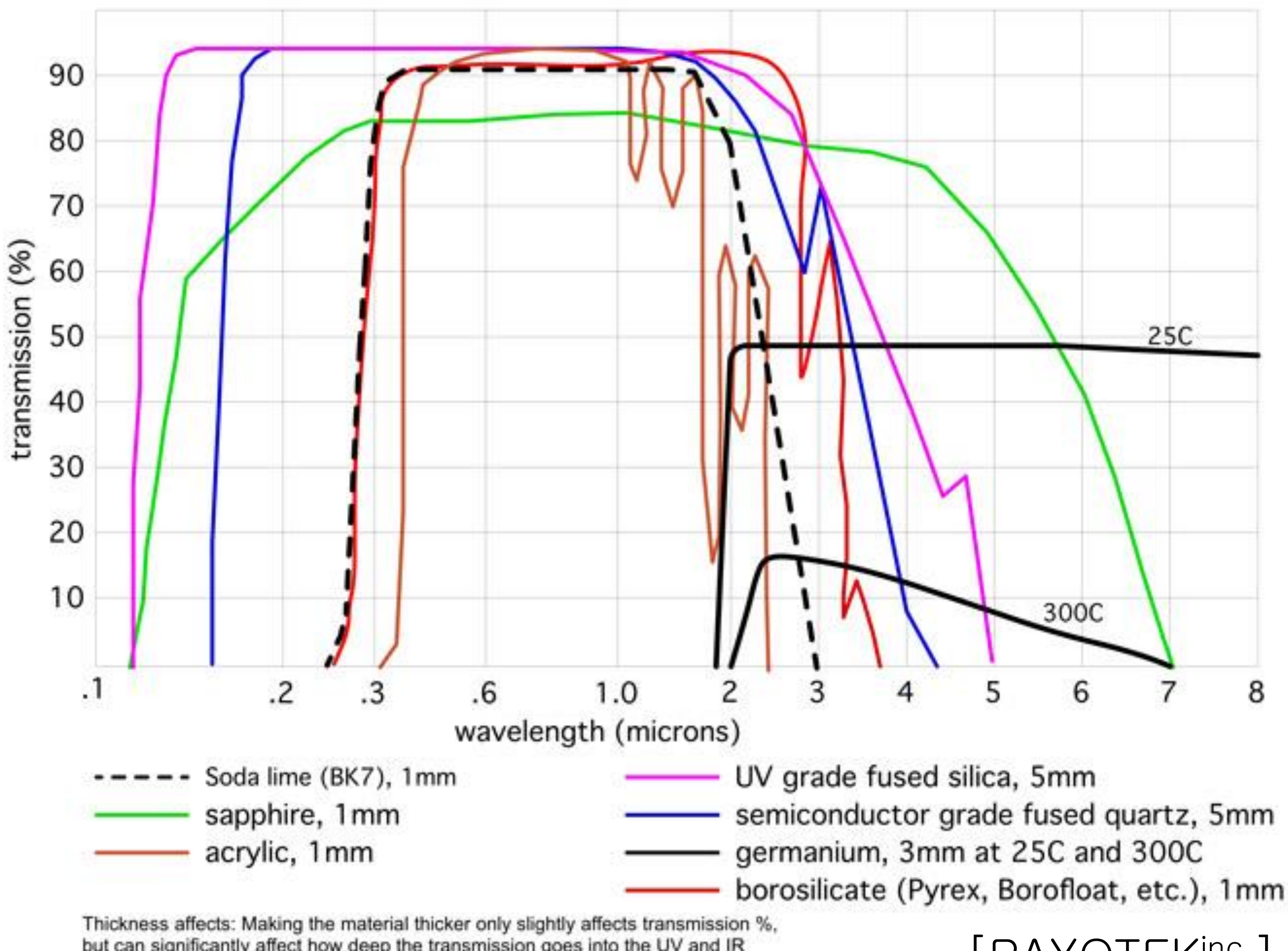
Glass

- Glasses
  - ✓ Non-metal, inorganic materials
  - ✓ Mainly used as *vacuum windows*
    - ✓ Also used in helium permeation leaks
- Three types of glasses
  - ✓ Soft glass: $60 \cdot 10^{-7}\ K^{-1} – 120 \cdot 10^{-7}\ K^{-1}$
  - ✓ Hard glass: $<50 \cdot 10^{-7}\ K^{-1}$
  - ✓ Quartz glass: $\sim 5 \cdot 10^{-7}\ K^{-1}$

Glass

- Soft glass(SiO_2 +Alkali)
  - 65–70% SiO_2, 2.5–15% Na_2O, 5–15% CaO
- Hard glass (SiO_2 > 70% + Boron)
  - Corning 7056, Duran, Pyrex
  - Most common glass in vacuum tech.
- Quartz (SiO_2 100%)
  - Used as optical vacuum window

Optical transmission



[RAYOTEKinc.]

Plastics

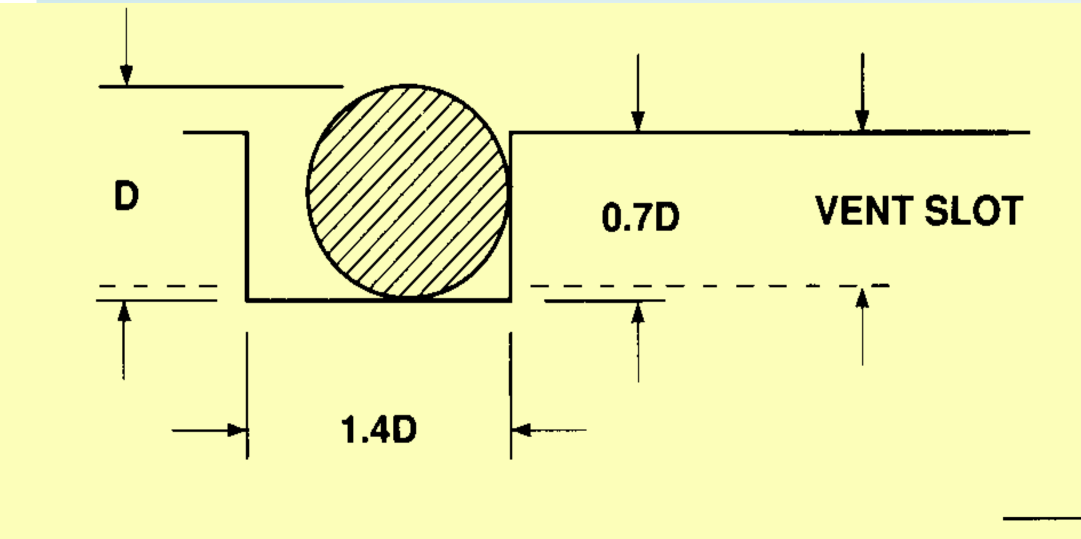
Plastics

- Elastomers
  - Elastic , Rubber-like materials
  - Used as reusable vacuum gaskets for RV, MV, HV, or (UHV)
  - Buna-N, Viton, Kalez, …
- Thermoplastics
  - Thermally reversible
- Duroplastics
  - Thermally irreversible
  - Epoxy
  - Good adhesion with metals, glasses, ceramics

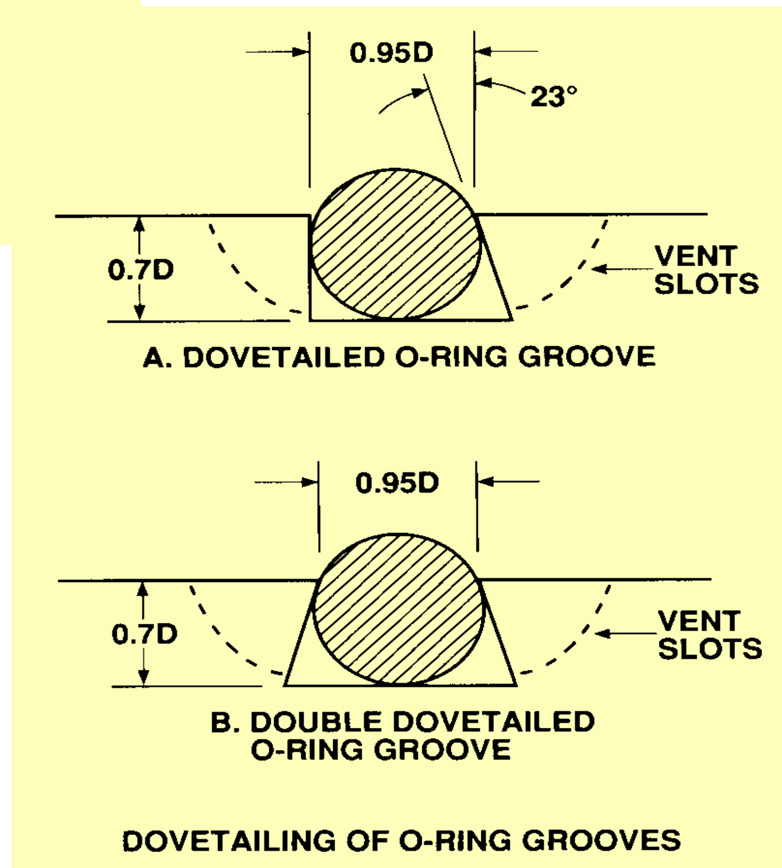
Outgassing rates

Outgassing rate ($TorrLs^{-1}cm^{-2}$)	@ 10 h	Baked
Viton (FKM)	$5x10^{-8}$	$5x10^{-10}$
Buna N (NBR)	$2x10^{-6}$	$4x10^{-8}$
Epoxy (Shell Epon)	$1x10^{-6}$	$8x10^{-8}$
Teflon (poly'fluoro'lene/PTFE)	$8x10^{-8}$	$8x10^{-9}$
Nylon (polyamide)	$3x10^{-7}$	$6x10^{-9}$
PVC	$3x10^{-7}$	$8x10^{-8}$

O-ring seal



Retaining the O-ring

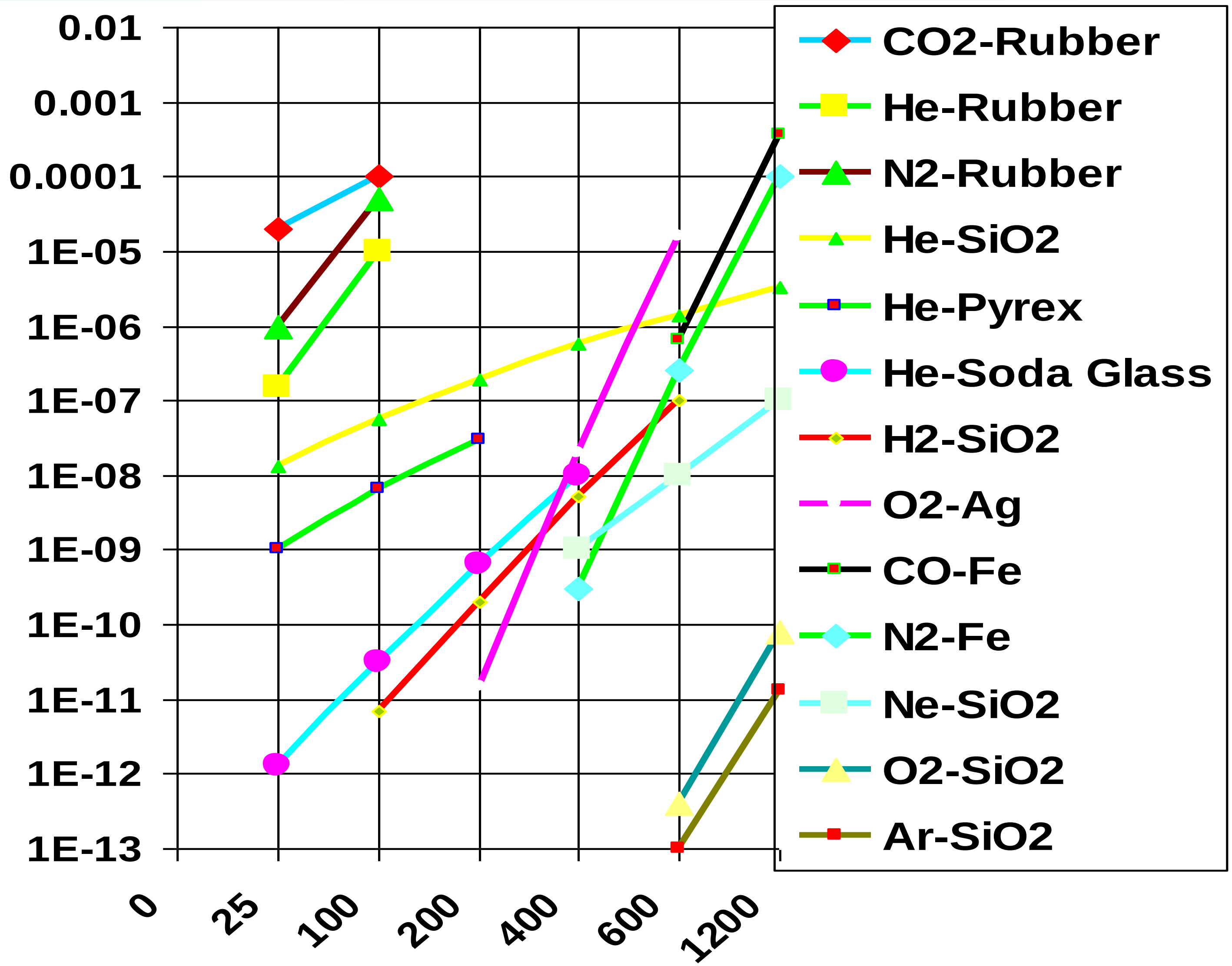


Permeation

- Permeation through solid materials involves
  - sorption, diffusion and desorption.

- Materials have permeation rates for different gases specific to that material.
  - steels have higher permeation rates with higher carbon content;
  - copper has low permeation for all gases;
  - aluminum has low permeation for hydrogen;
  - silver has high permeation rate for oxygen at high temperature
  - palladium has high permeation rate for hydrogen
  - polymers are permeable to all gases.

Permeation


0.01
0.001
0.0001
1E-05
1E-06
1E-07
1E-08
1E-09
1E-10
1E-11
1E-12
1E-13
0
25
100
200
400
600
1200
CO2-Rubber
He-Rubber
N2-Rubber
He-SiO2
He-Pyrex
He-Soda Glass
H2-SiO2
O2-Ag
CO-Fe
N2-Fe
Ne-SiO2
O2-SiO2
Ar-SiO2

Permeation

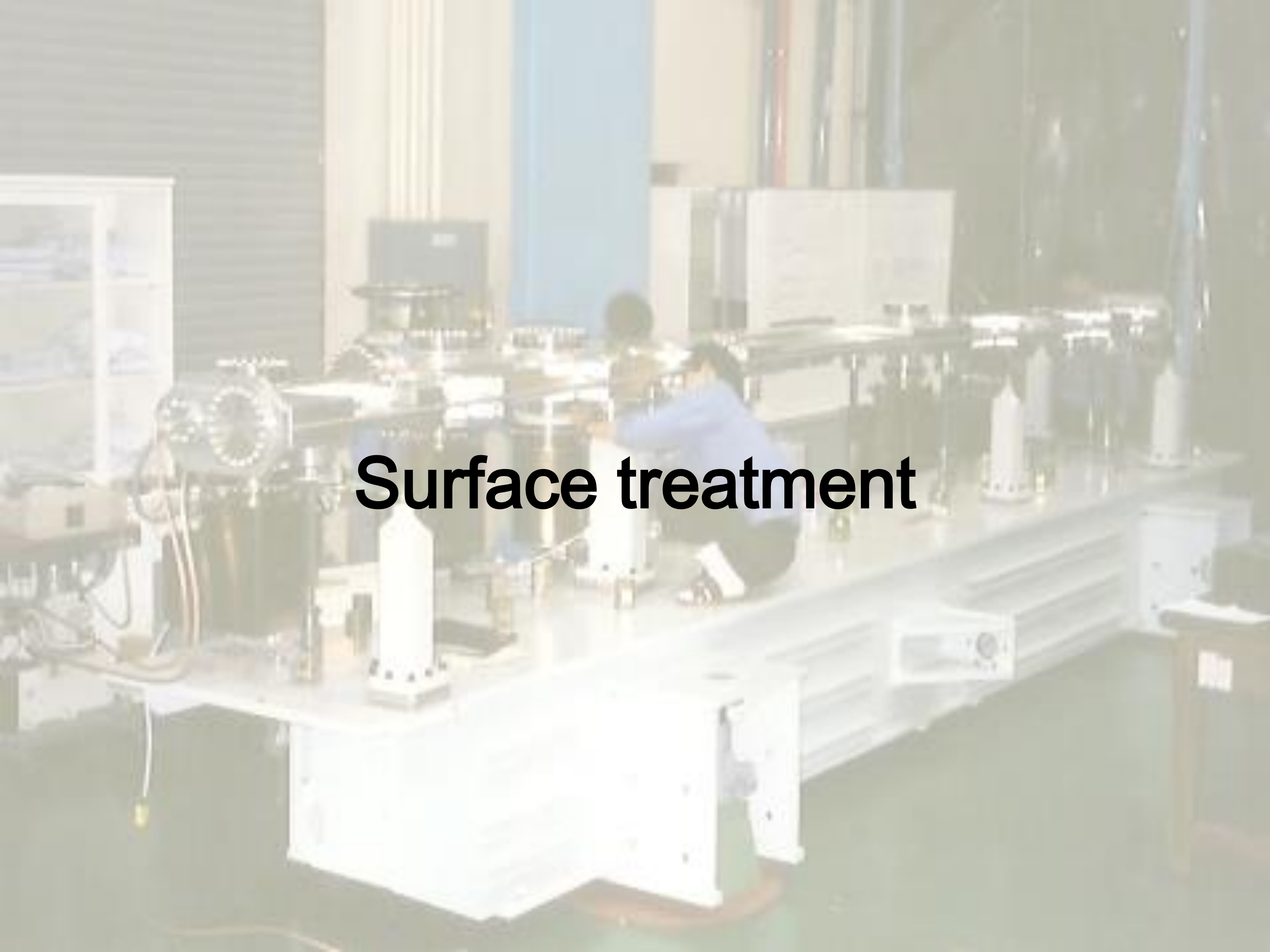
- Permeation is a strong function of temperature.
- Permeation gives additional gas loads.
  - Modifies the chamber environment(residual gases)
  - Affecting chemistry in vacuum process
  - Limits the ability to reach ultra high vacuum.
- Calibrated leaks
  - helium permeation through pyrex or quartz.

Helium permeation through elastomers

Polymer	Permeation rates	
	std-cc/s cm^2 (x10^{-7}) at 1 atm	
	25^{o}C	150^{o}C
Viton	1.3	49
Buna N	0.8	25.2

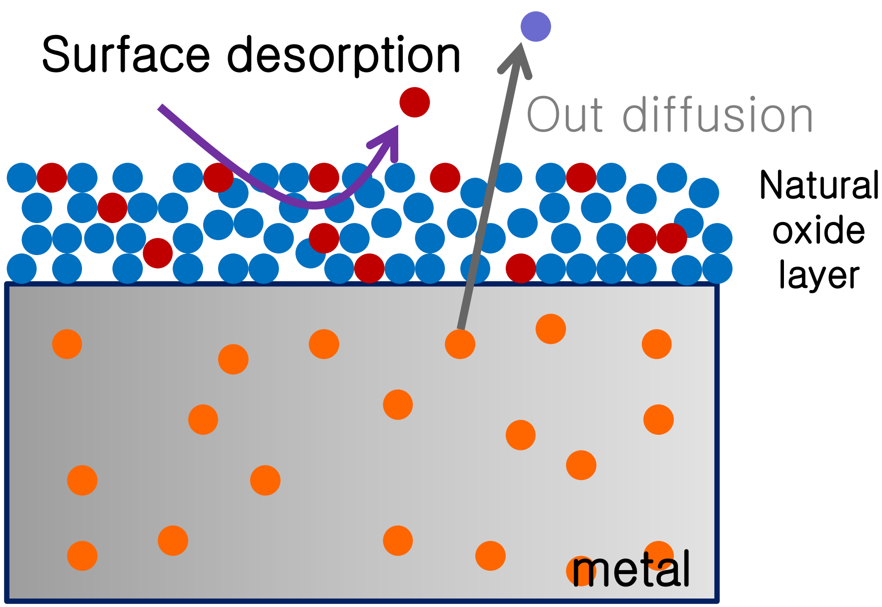
After several minutes helium will begin to permeate through the elastomer O-ring and show up as a leak.

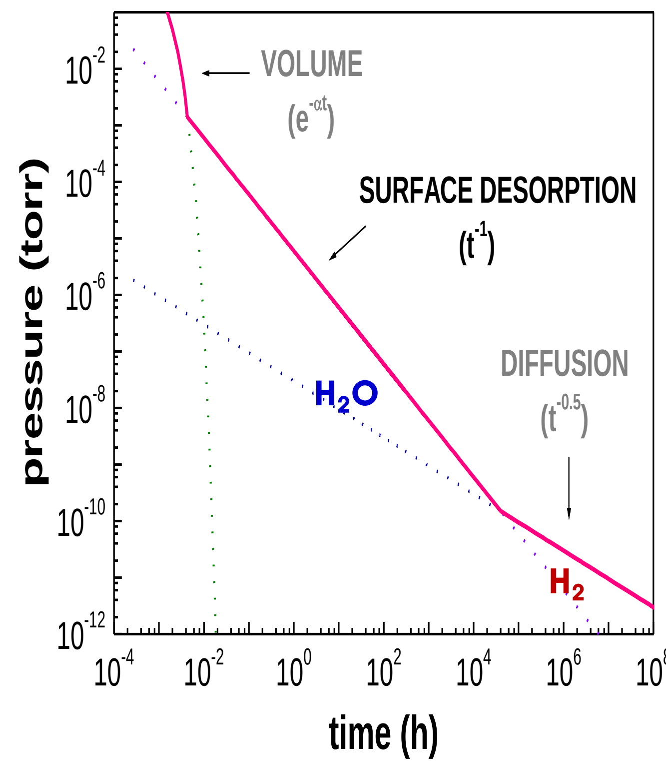
Surface treatment



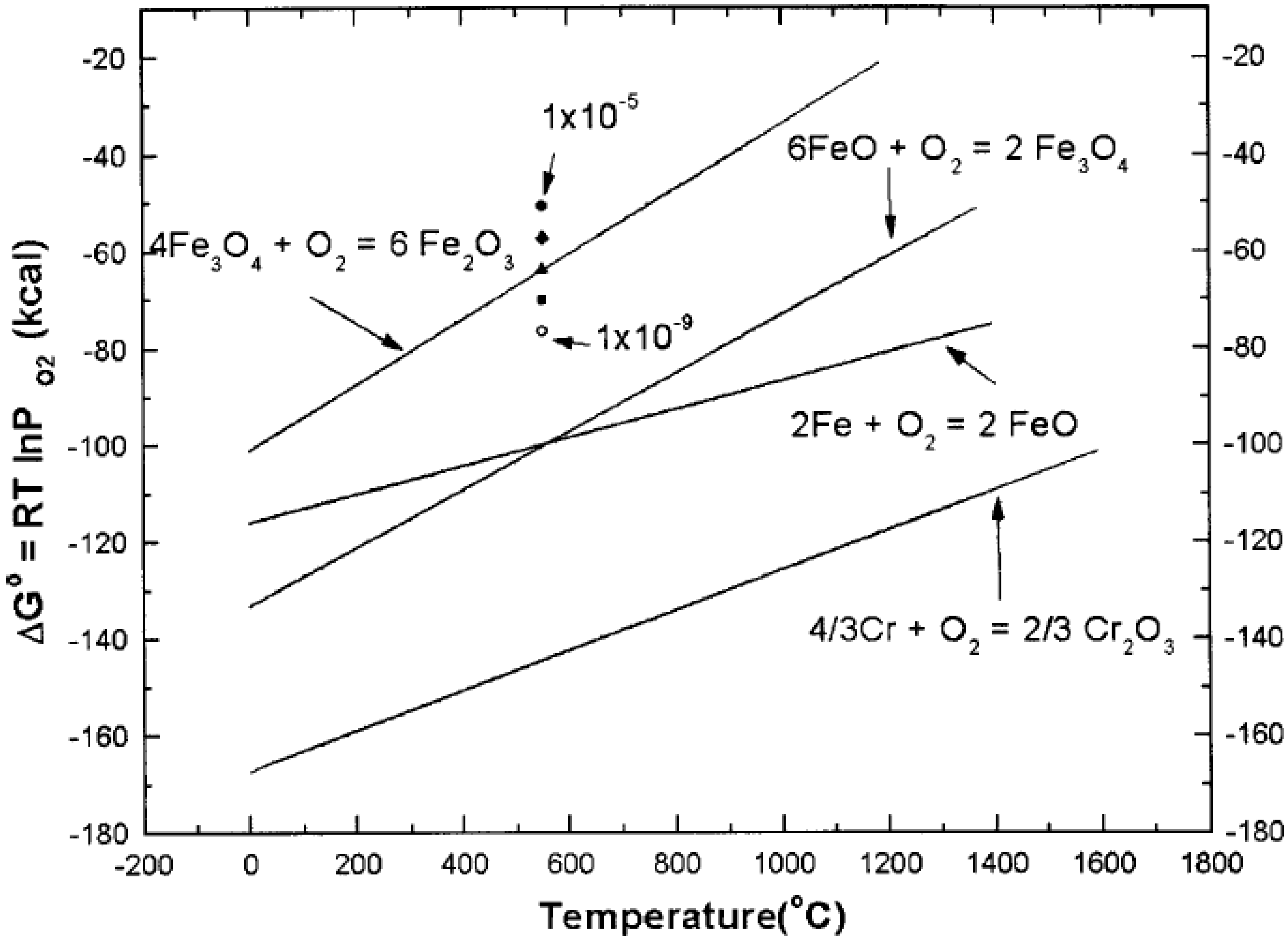
일반 스테인리스강의 진공특성

- ✓ 일반 금속(스테인리스강)의 표면은 다공질의 산화막이 형성되어 있음.
- ✓ 공기 노출 시 다공질 표면에 물과 같이 흡착성이 강한 입자가 다량으로 흡착됨.
- ✓ 진공 배기하면 흡착된 물 분자가 서서히 방출되므로 압력은 시간의 역수에 비례하며 ($p \propto 1/t$) 느리게 떨어짐.





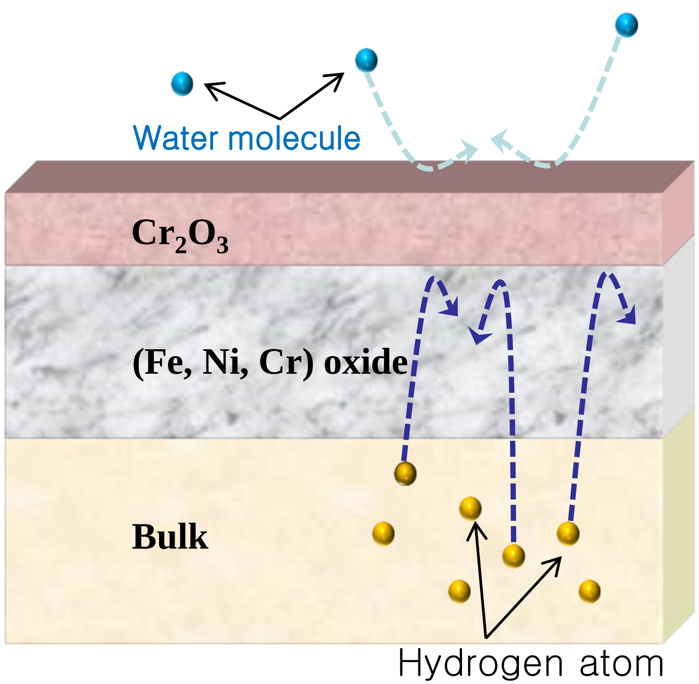
Ellingham Diagrams

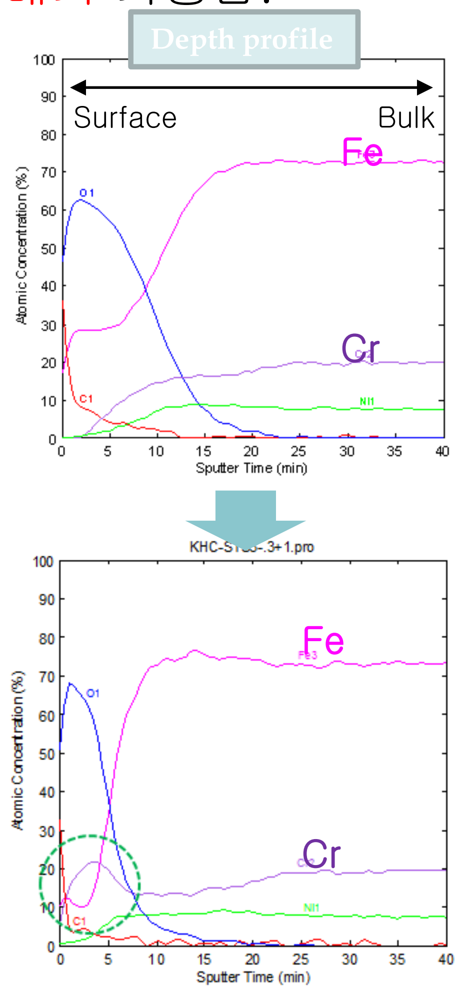


[S. Moon, Sur. Rev. Lett. 9, 285 (2002)]

Vacuum Thermal Oxidation (VTO)

- ✓ VTO 표면 처리를 하면 다공질 산화막 위에 순수한 크롬산화막이 형성됨.
- ✓ 크롬산화막에 의해 공기 노출 시 물 분자의 흡착량이 크게 감소하고 흡착된 물 분자도 비교적 쉽게 떨어져나가므로 급속 배기 가능함.



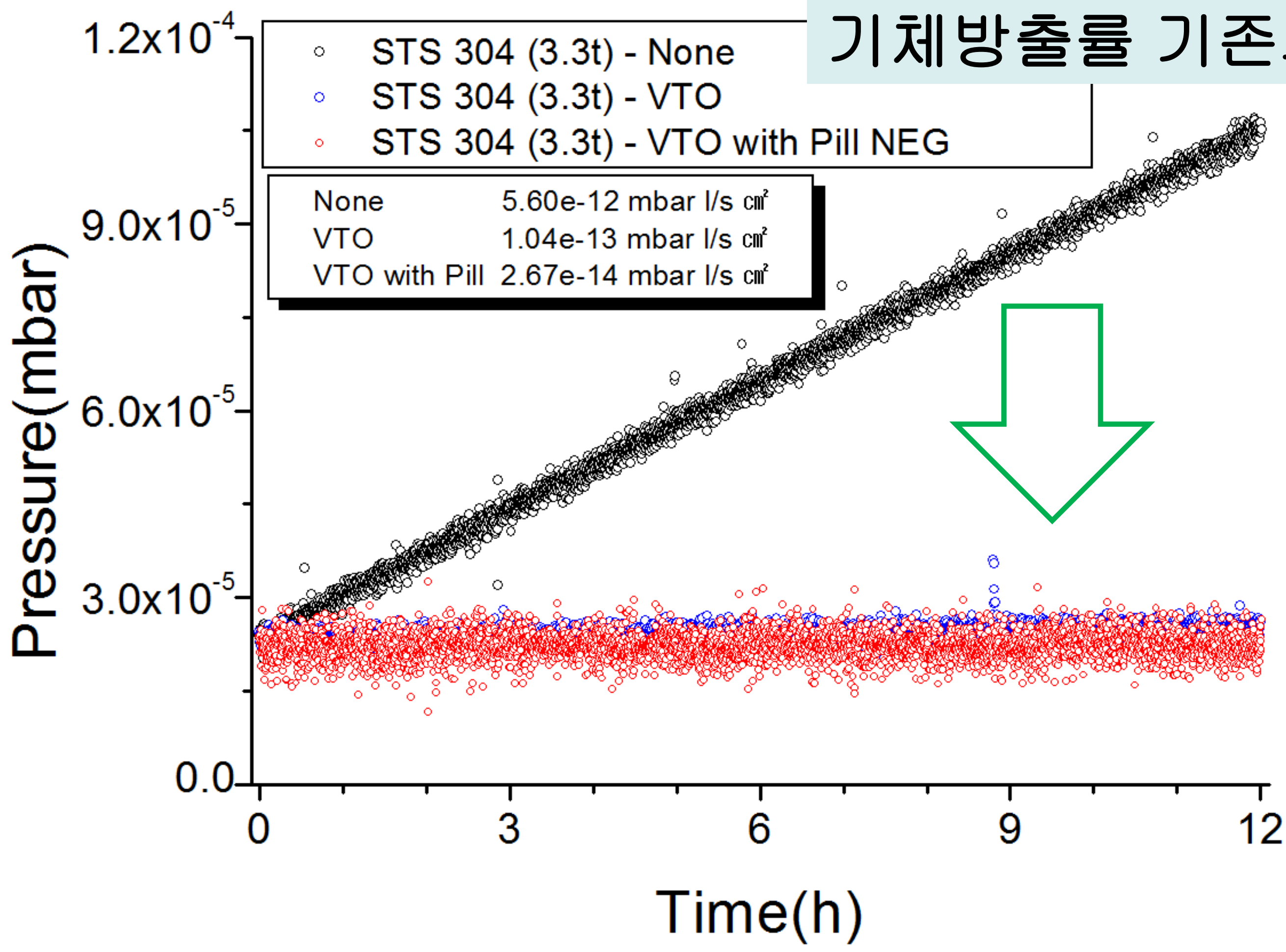


일반 (STS304)
:표면에 철산화물 많음

VTO (STS304)
:표면이 순수한 크롬산화물

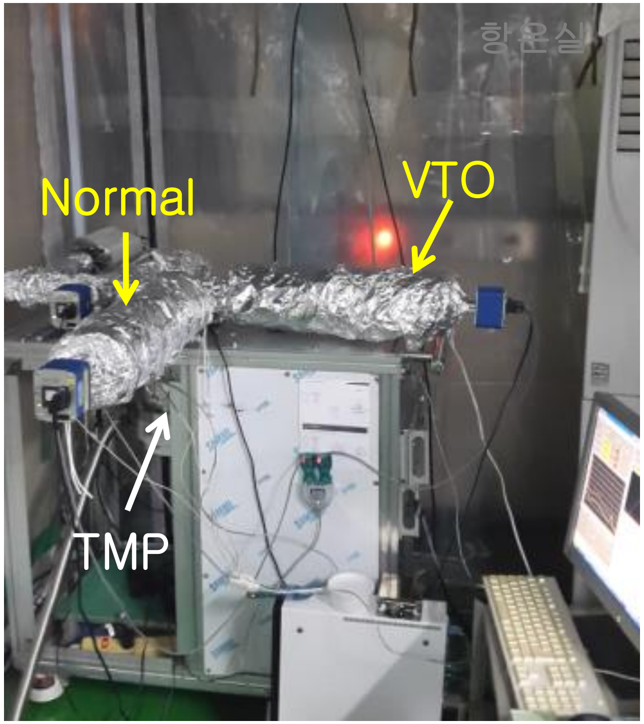
기체방출률 비교

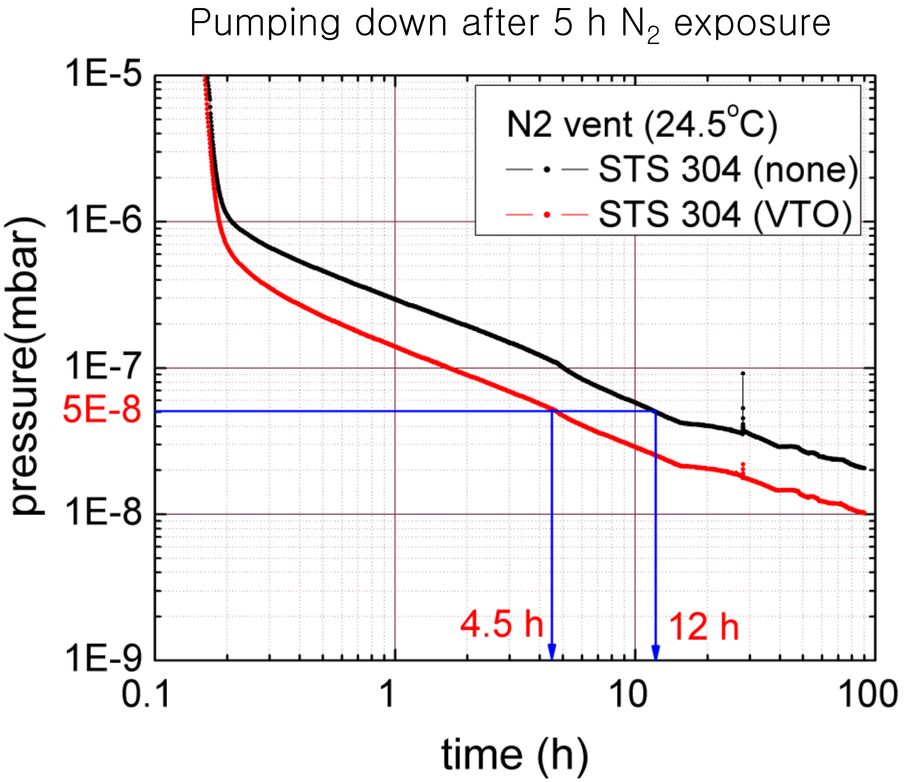
기체방출률 기존의 1/300



배기 시간 비교

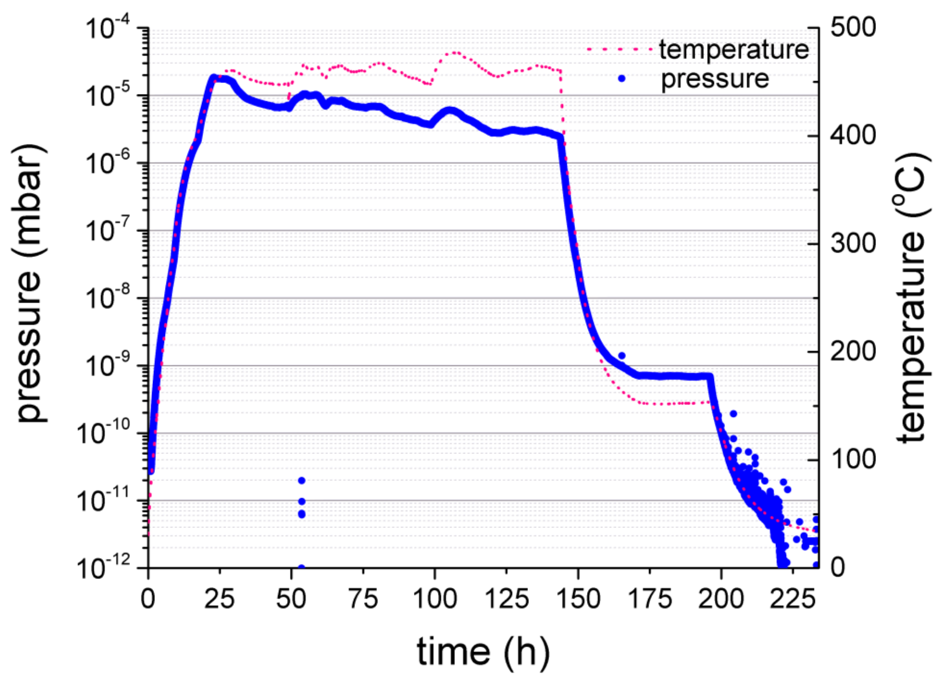
- ✓ 동일한 STS304 진공용기(6 리터) 2 set 제작하여 1 set는 VTO 표면처리 함.
- ✓ 동시에 같은 조건으로 baking 후 질소 벤팅하여 5시간 유지한 이후 진공배기 함.
- ✓ 일반 STS304 진공용기와 VTO 처리한 진공용기의 배기시간 비교
  : 고진공 (5E-8 mbar) 도달 시간이 일반 STS의 1/3





극고진공 챔버

- 1000 리터 급 극고진공 시스템: 상용 진공게이지의 측정 하한 도달.



- Literature survey
  - Do not believe those data
- Test and evaluation
- Make your own data