

# 진공 재료 (종류, 특성, 제작)

박종도  
포항가속기연구소

# 진공재료 선택

## 재료 선택시 고려할 내용

- 얻고자 하는 진공도는?

## 관련 항목

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

# 진공재료 선택

## 재료 선택시 고려할 내용

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

## 관련 항목

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

# 진공재료 선택

## 재료 선택시 고려할 내용

- 사용온도와 수명은?
- 투자율은?
- 전기전도도는?
- 제작 비용은?
- 재료 수급성은?

## 관련 항목

- 금속 재료 또는 플라스틱?
- 극저온 또는 고온?
- 높은 내열피로성
- 전자기 특성에 영향
- 전도체 또는 부도체
- 국내 또는 국외 수입
- 구입 기간

# Vacuum Materials

Steels

Stainless Steel

Aluminum (alloy)

Copper (alloy)

Other metals

Ceramics

Plastics

# Steels



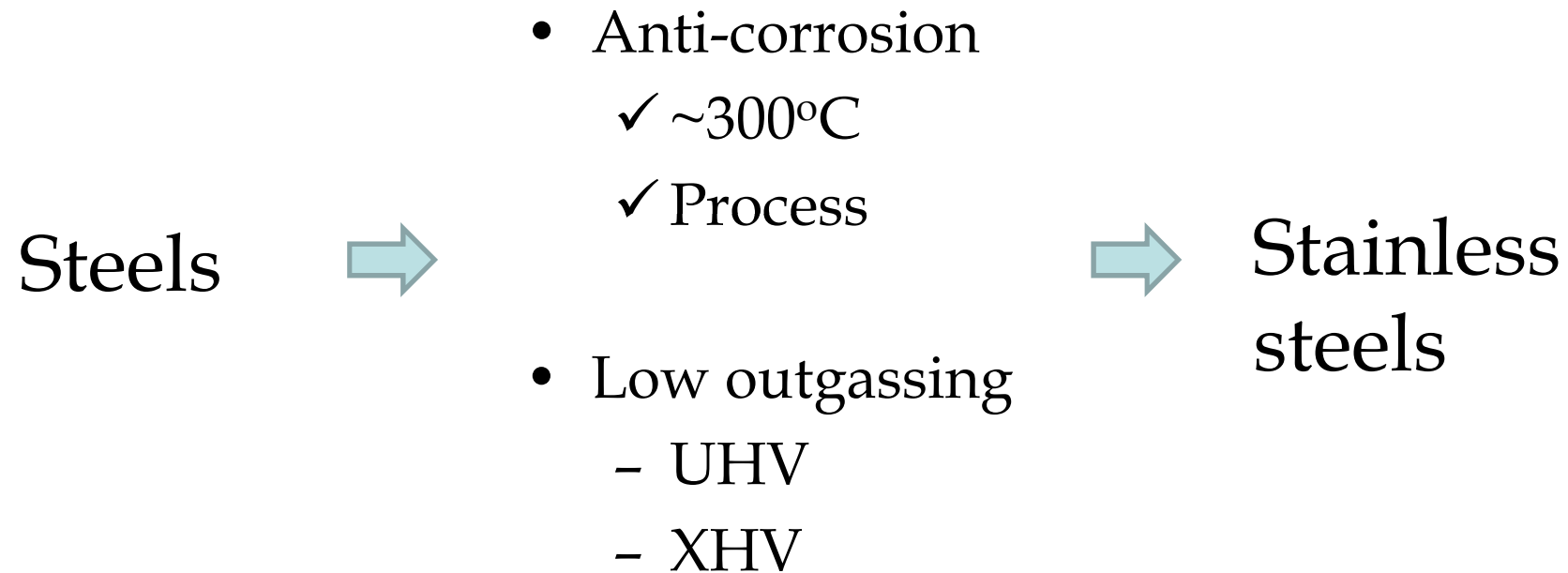
# Steels

- Mild/structural steels
  - ✓ Carbon ~ 0.2 %
  - ✓ higher outgassing rate
    - $q > (20\sim 200) \times$  Stainless steels
    - HV compatible ( $10^{-6}$  mbar)
    - Contains contaminants of C, P, S etc.
    - endless emission of CO
  - ✓ Weldable
  - ✓ Easy to corrode
    - ✓ Needs anti-corroding coating
  - ✓ Magnetic
    - ✓ Shielding material for magnetic field

# Steels

- Mild/structural steels
  - ✓ S235, S355, S20C
  - ✓ HV compatible
    - plate, pipe, rod
    - $\sim 10^{-7}$  mbar
    - $5 \times 10^{-10}$  (mbar l/scm<sup>2</sup>) after bake
  - ✓ MV, RV compatible
    - Cast parts; pump and valve housing
    - $\sim 10^{-3}$  mbar

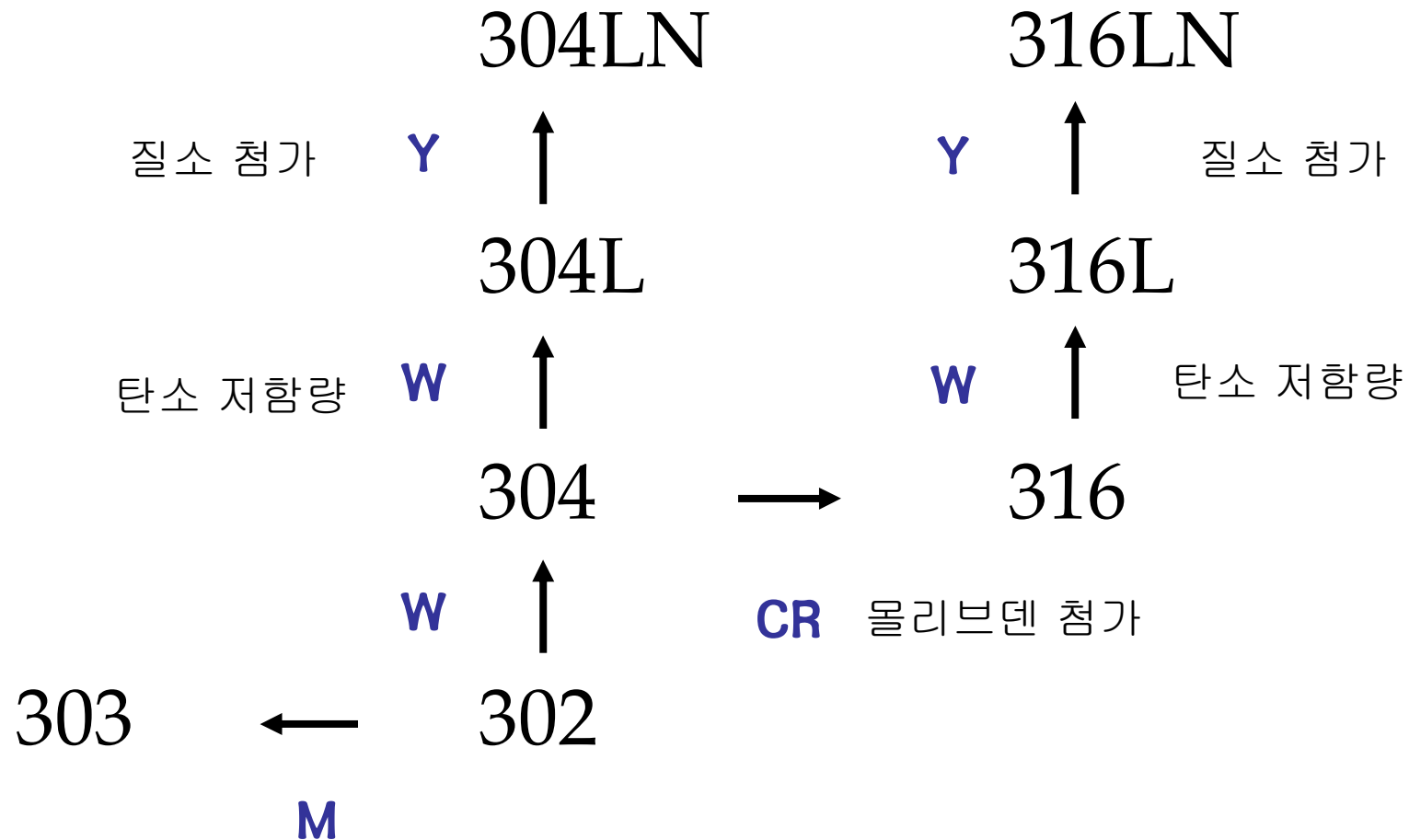




# Stainless steel

- AISI 304/316
  - ✓ Austenitic
    - High strength
    - Non-magnetic (but, not entirely)
    - Good weldability
  - ✓ Corrosion resistance
    - During vacuum processing and bakeout ( $\sim 300^{\circ}\text{C}$ )
  - ✓ UHV/XHV compatible
    - plate, pipe, rod
    - $\sim 10^{-11}$  /  $< 10^{-12}$  < mbar
    - $(2\sim6) \times 10^{-12}$  (mbar l/scm<sup>2</sup>) after bake
    - $< 1 \times 10^{-13}$  (mbar l/scm<sup>2</sup>) 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 outgassing rate of 303 stainless can be lowered to  $10^{-13}$  mbarl/sec cm<sup>2</sup> by a combination of fabrication and post treatments such as bakeout.)
  - ✓ Welding for vacuum systems 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
  - ✓ Less carbide precipitation
  - ✓ Cleaner machining and better welds than 304
- 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

# Stainless steel

## ■ 316L

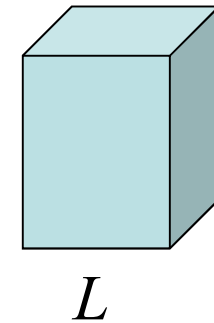
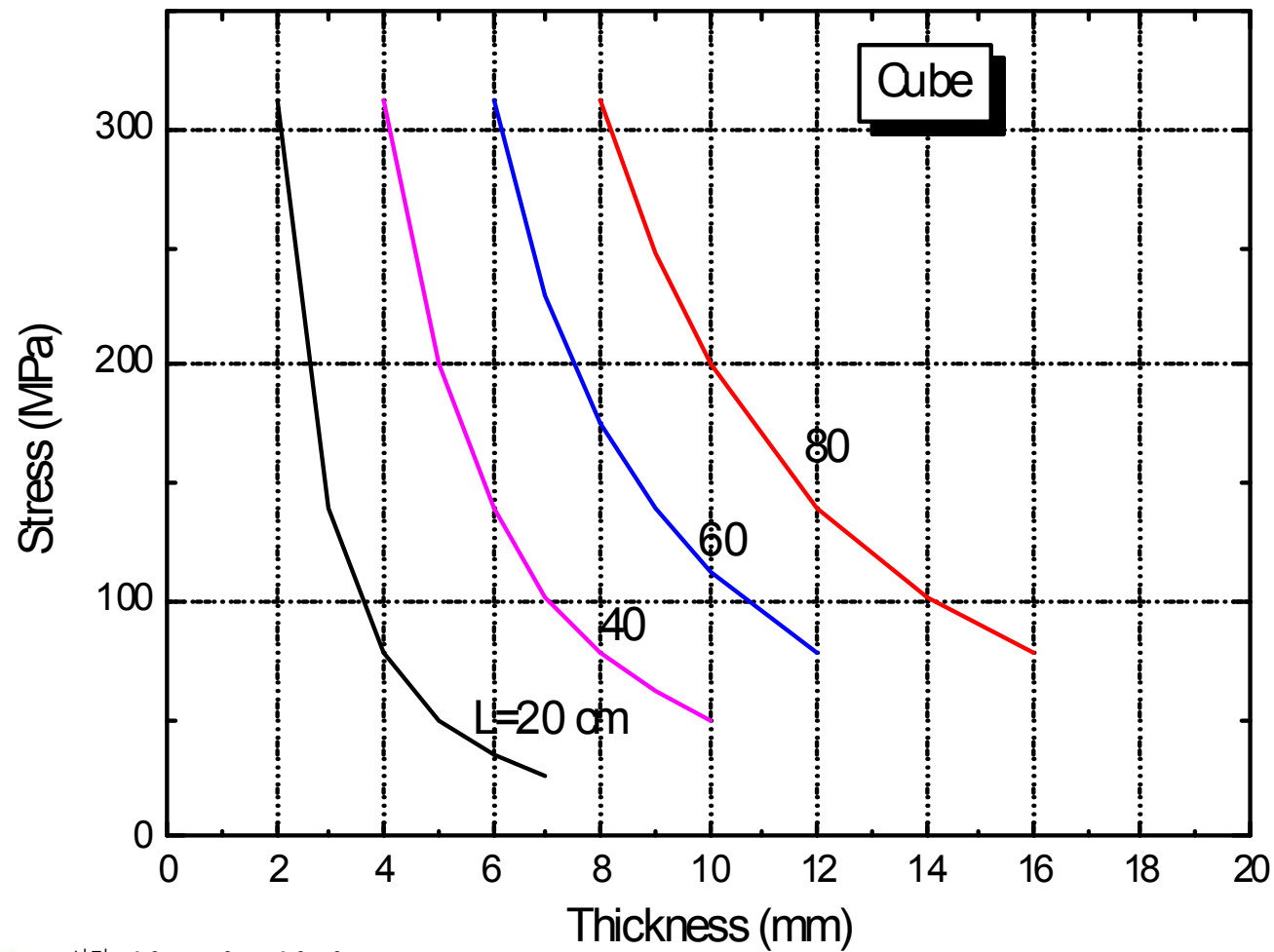
- ✓ 18% Cr, 14% Ni, 0.03% C, 3% Mo
- ✓ Stabilized with molybdenum to prevent carbide precipitation in the weld zone
- ✓ Lower outgassing rates than 304(L)
  - $\sim 2 \times 10^{-12}$  (mbar l/scm<sup>2</sup>) after bake
  - $< 5 \times 10^{-14}$  (mbar l/scm<sup>2</sup>) after special treatments
- ✓ 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

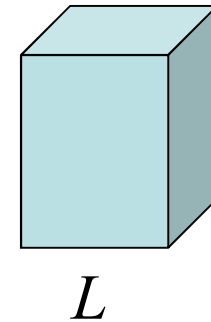
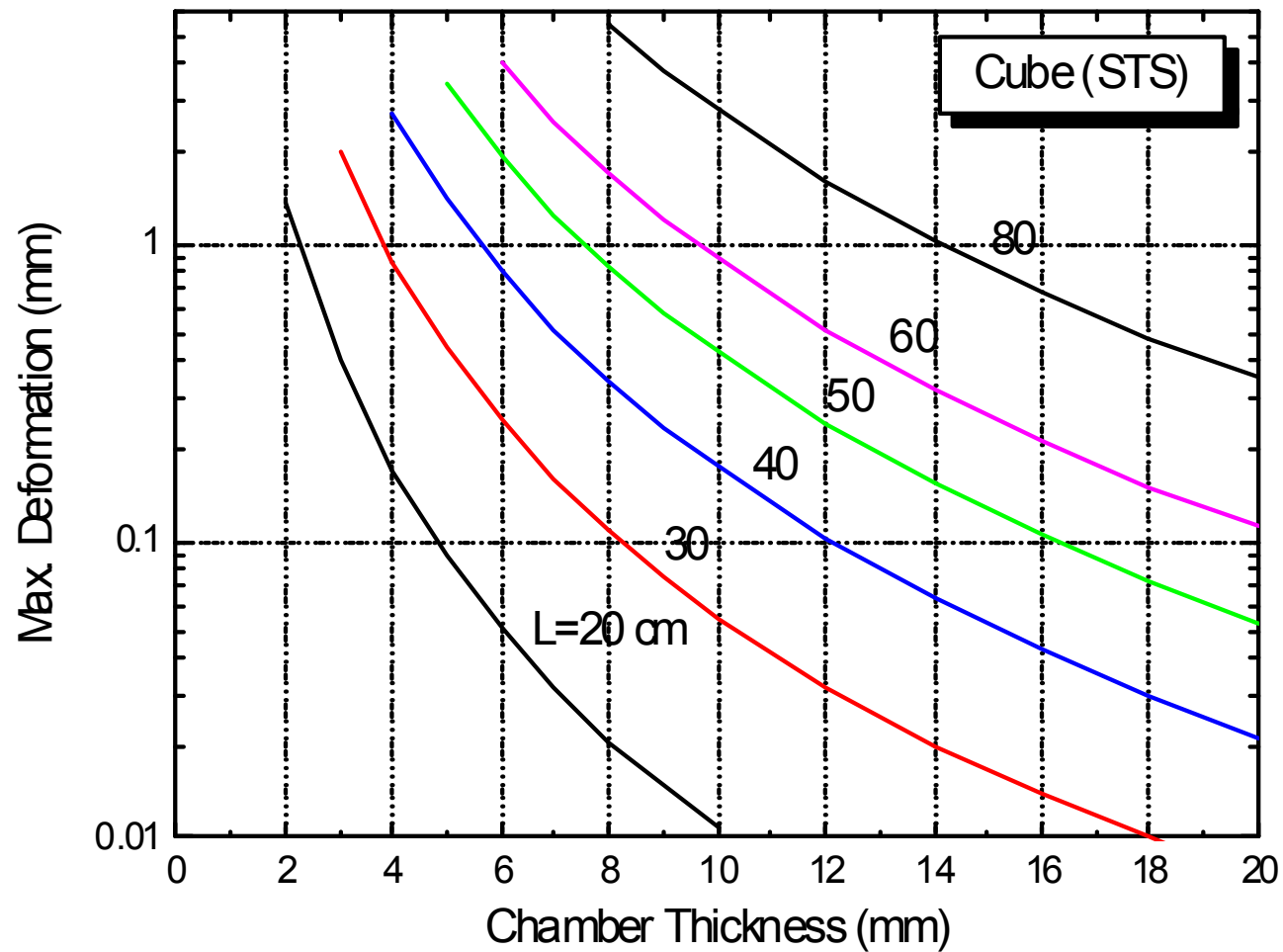
	Yield strength (항복강도, 내력, 내구력)		Tensile strength (인장강도)	
	Kgf/mm <sup>2</sup>	MPa	Kgf/mm <sup>2</sup>	MPa
304, 316	21	205.8	53	519.4
304L, 316L	18	176.4	49	480.2
A5083	7.24	71.0		
A6061, 6063	7.03	68.9		



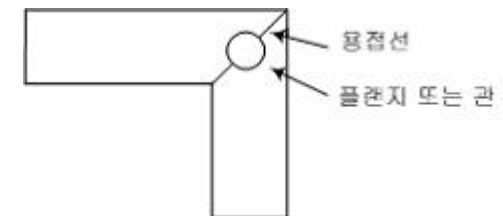
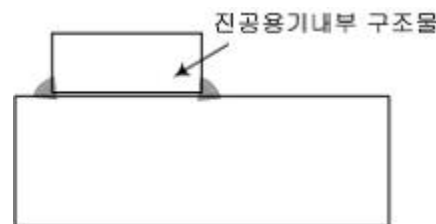
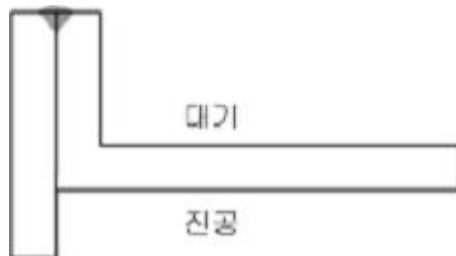
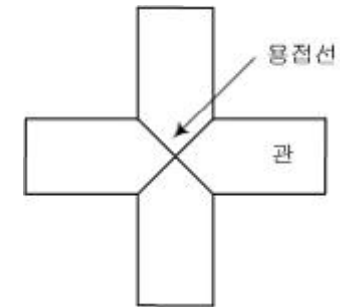
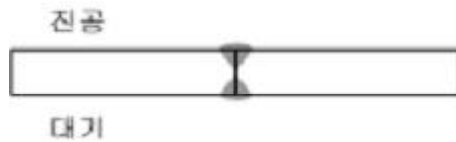
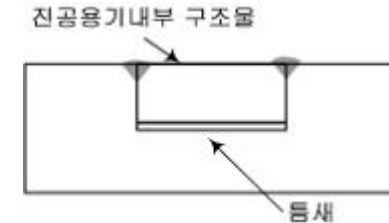
# Stress : Cube



# Deformation : Stainless steel



# 피해야 하는 용접



→ “초고진공 용접핸드북(KRISS)”

# Aluminum (alloys)



# Aluminum (alloys)

- 1xxx pure aluminum
  - ✓ > 99% Al by weight
  - ✓ A1050; suitable for metal gaskets
  - ✓ can be work hardened
- 2xxx copper alloys
  - ✓ can be precipitation hardened
  - ✓ 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
  - ✓ can be work hardened.
  - ✓ A3004; suitable for vacuum bellows
  
- 4xxx            silicon alloys
  - ✓ known as Silumin

# Aluminum (alloys)

- 5xxx            magnesium alloys
  - ✓ easy to machine, higher strength, good weldability
  - ✓ A5083; used in accelerators
  
- 6xxx            magnesium and silicon alloys
  - ✓ Easy to machine
  - ✓ can be precipitation hardened  
(but not to the high strengths that 2xxx and 7xxx can reach)
  - ✓ A6063; most common materials in vacuum technology
  - ✓ A6061; one of the most vacuum materials

# Aluminum (alloys)

- 7xxx          zinc alloys
  - ✓ with can be precipitation hardened  
(to the highest strengths of any aluminum alloy)
- 8xxx          lithium alloys



# 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

# Temper designation

- -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
  - -T1 Cooled from hot working and naturally aged (at room temperature)
  - -T2 Cooled from hot working, cold-worked, and naturally aged
  - -T3 Solution heat treated and cold worked
  - -T4 Solution heat treated and naturally aged
  - -T5 Cooled from hot working and artificially aged (at elevated temperature)
  - -T51 Stress relieved by stretching
  - -T510 No further straightening after stretching
  - -T511 Minor straightening after stretching
  - -T52 Stress relieved by thermal treatment
  - -T6 Solution heat treated and artificially aged
  - -T7 Solution heat treated and stabilized
  - -T8 Solution heat treated, cold worked, and artificially aged
  - -T9 Solution heat treated, artificially aged, and cold worked
  - -T10 Cooled from hot working, cold-worked, and artificially aged
- -W Solution heat treated only.

# Aluminum (alloys)

- A6063-T5(T6)
  - ✓ The most widely used aluminum alloy
  - ✓ The most common materials in vacuum technology
  - ✓ Low outgassing rates
    - $< 5 \times 10^{-12}$  (mbar l/scm<sup>2</sup>) after bake at 150°C
  - ✓ Easy to forming using *extrusion*, machining and welding
  - ✓ Alloy retains its strength after welding
  - ✓ ConFlat<sup>®</sup> flanges are made from A2219 with knife edge coated with TiC (TiN, CrN).
    - ✓ With A1050 metal gaskets

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

# Aluminum (alloys)

- A6061-T5(T6)
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  - ✓ Easy to forming using *machining* and welding
  - ✓ Alloy retains its strength after welding
  - ✓ ConFlat<sup>®</sup> 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)

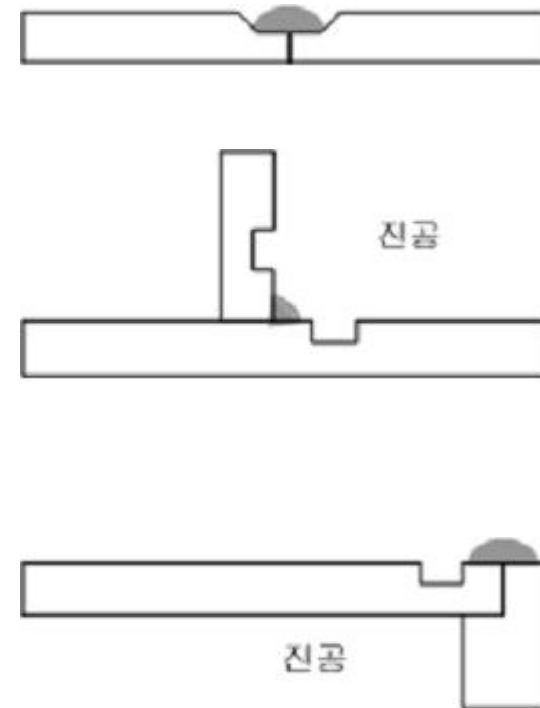
- Initial outgassing rate is higher( $\sim 5x$ ) than that of SST.
  - ✓ The desorption rate of water vapor from the surface of aluminum is slower than stainless steels which gives it different initial pump down characteristics.
- Anodizing
  - ✓ a common surface treatment.
  - ✓ produces hard inert surface, but outgassing rate is degraded by  $\sim x10$ .
  - ✓ Not suitable for UHV applications
- ✓ Surface 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)

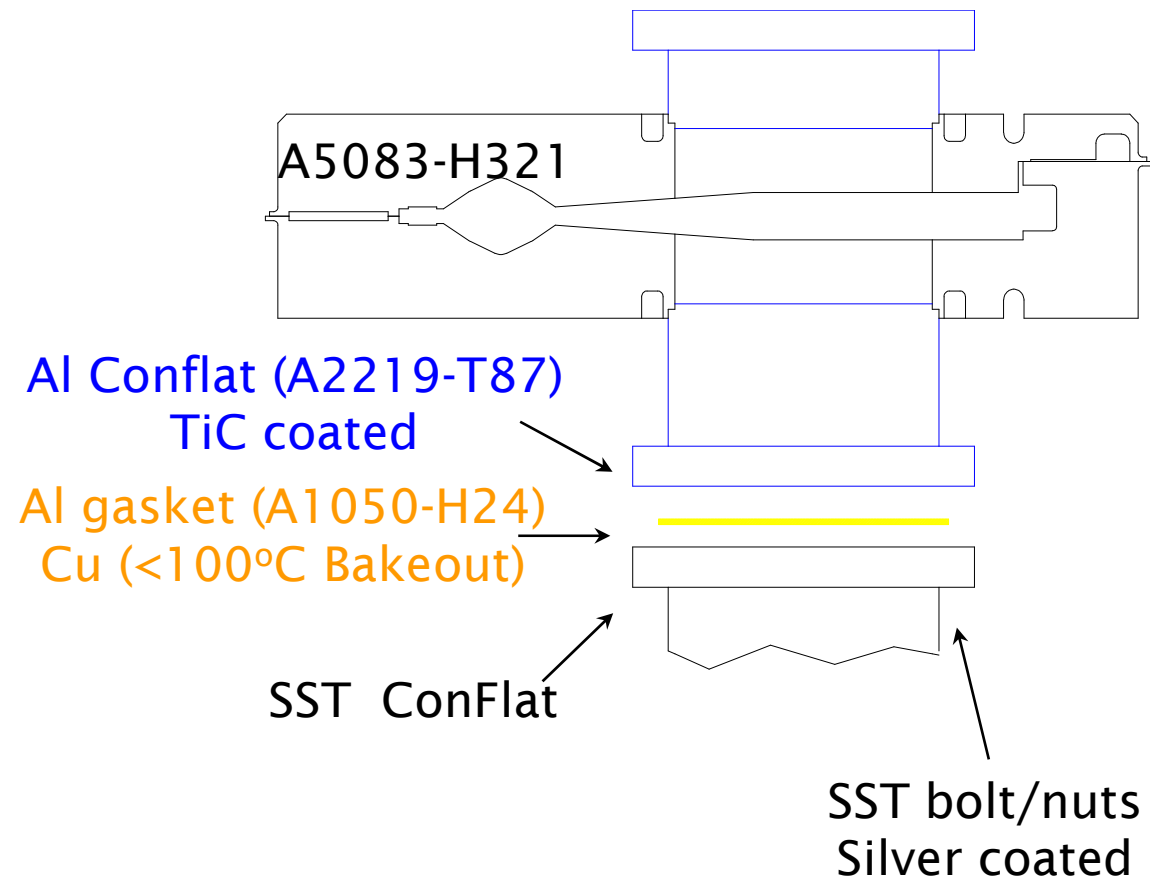


→ “초고진공 용접핸드북  
(KRISSE)”

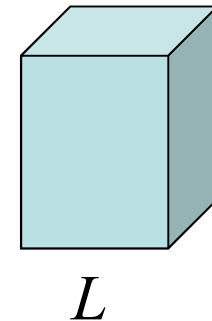
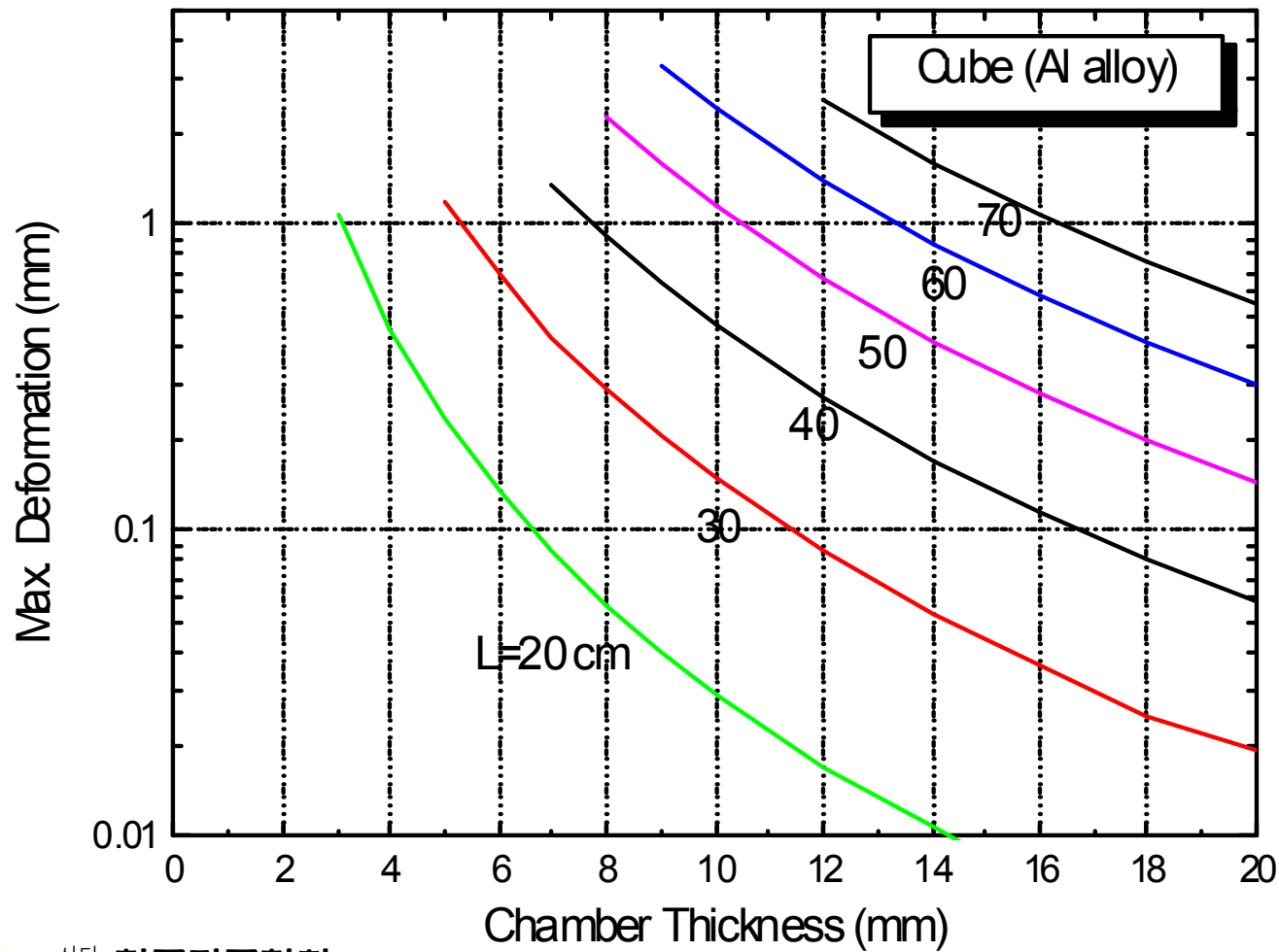


# Vacuum Seals

- Al/SST hybrid ConFlat system



# Deformation : Al alloy



$t \ll L$   
 $\delta < t$

## SST - Al 비교

		Stainless steel	Aluminum alloy
Vacuum characteristics	Outgassing rate	depends on surface treatments	
	Preinstallation bakeout	950℃	< 210℃
	<i>In situ</i> 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	
Other characteristics	Quality of demountable seals	better	
	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.
- Bakout; Up to 300°C in vacuum
- Cold welding; OHFC gasket for ConFlat flanges
- Joining techniques; brazing, soldering, welding
- Outgassing rate;  $\sim 10^{-9}$  mbar liter/sec cm<sup>2</sup>

# Copper alloy

## Brass and Tombac

- Tin or Zinc alloys
  - High vapor pressure at high temperatures  
(Used in vacuum systems where temperatures above 100°C are not found)
  - 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<sup>2</sup>

# Copper alloy

- To increase its strength
  - OFC +  $\text{Al}_2\text{O}_3$ (0.1-0.5%) GlidCop
    - Yield strength(at 0.2% offset) > 200 Mpa  
(OFC ~ 100 MPa)
  - OFC + Ag or  $\text{Au}_{0.2\%}$  Expensive (x 4)
  - OFC + Zr High outgassing rate

# Outgassing rates

Q(Torrl/scm <sup>2</sup> )	@ 10 hour	Baked
Aluminum	$5 \times 10^{-8}$	$5 \times 10^{-13}$
Aluminum (anodized)	$3 \times 10^{-7}$	$5 \times 10^{-10}$
Stainless steel	$1 \times 10^{-8}$	$2 \times 10^{-12}$
Mild Steel	$2 \times 10^{-7}$	$5 \times 10^{-10}$
1018 Steel (Ni plated)	$5 \times 10^{-7}$	
Brass	$6 \times 10^{-7}$	
Copper	$5 \times 10^{-9}$	$1 \times 10^{-12}$
Copper (OFHC)	$2 \times 10^{-9}$	$1 \times 10^{-12}$



# 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  $> 150^\circ C$ 
  - ✓ Weld should be done with inert gas environment
- Used as metalizing materials for brazing
- TSP(Titium sublimation pump)
  - ✓ Sublimation at  $\sim 1,350^\circ C$
  - ✓  $\sim 5 \mu m$  coating for 1 hr
  - ✓ High oxygen permeation rate through silver at high temperature.
- Ion pump
  - ✓ Cathode material

# Indium

- Melting point; 156°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, Zirconia, Beryllium oxide,...
  - ✓ Alumina ( $\text{Al}_2\text{O}_3$ )
    - Mostly used ceramics
    - Max temperature;  $1,800^\circ\text{C}$
    - $> 92\%$  in vacuum technology
    - Can be brazed
    - Mainly used as electrical feedthroughs
      - Balable upto  $350^\circ\text{C}$
    - Tensile strength 25 kpsi (96% density)
  - ✓ Sapphire (monocrystalline  $\text{Al}_2\text{O}_3$ )
    - UV and IR transparent
    - Used as vacuum window

# Ceramics

- Silicate ceramics

- ✓ Steatite ( $\text{MgO-SiO}_2$ )

- Max temperature 1,000°C
    - Tensile strength 15 kpsi

- Glass-ceramics

- ✓ Crystalline ceramic

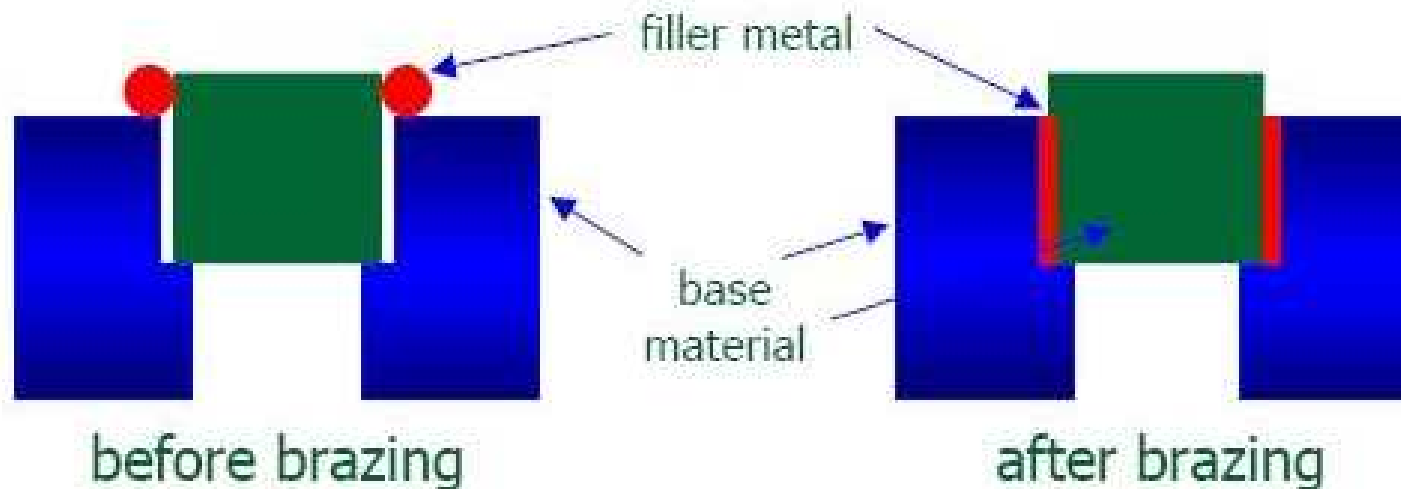
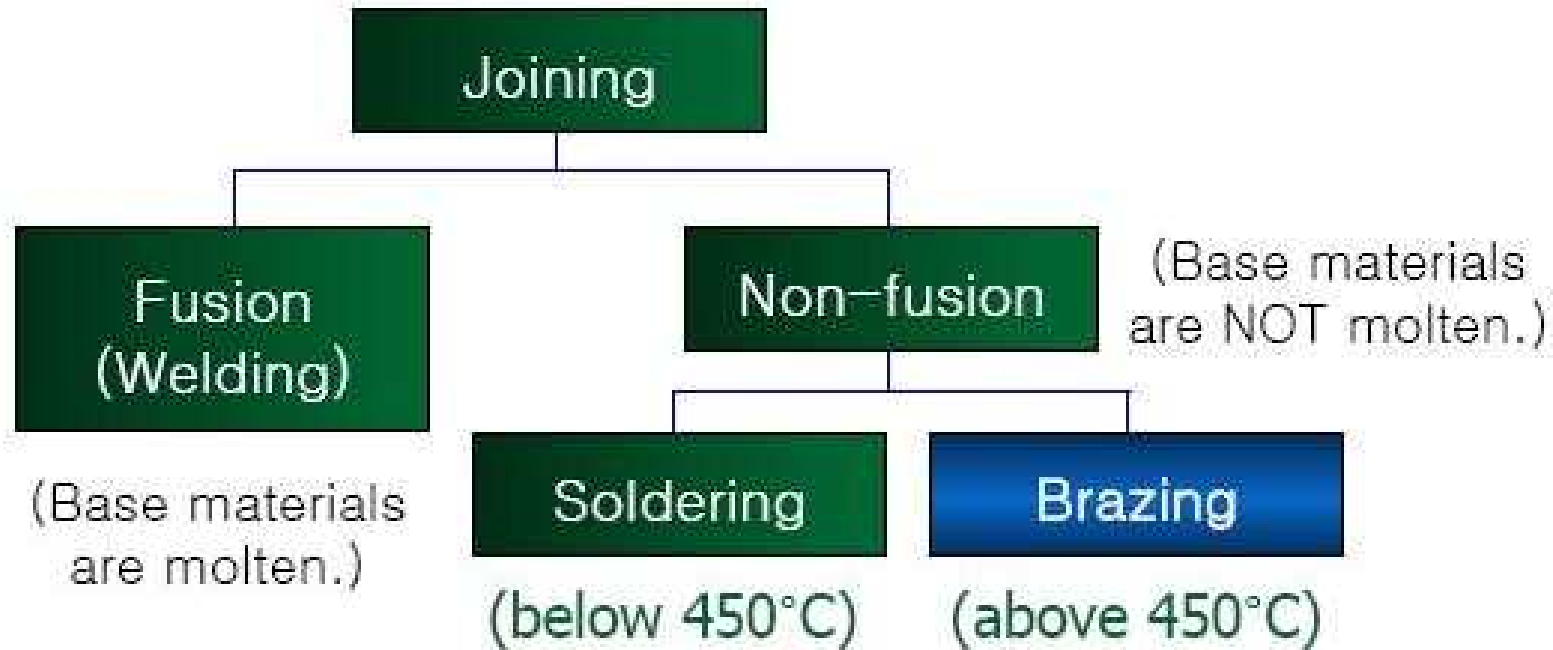
- Can be machined with standard tools
    - Macor®, Corning 9658



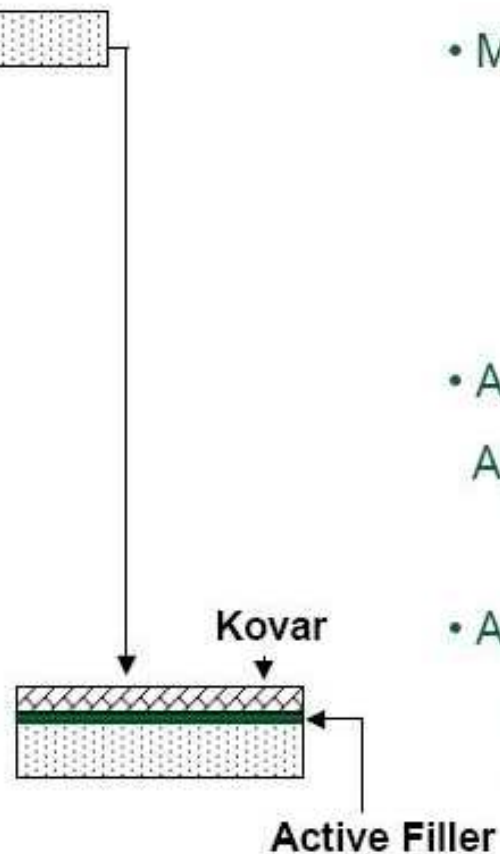
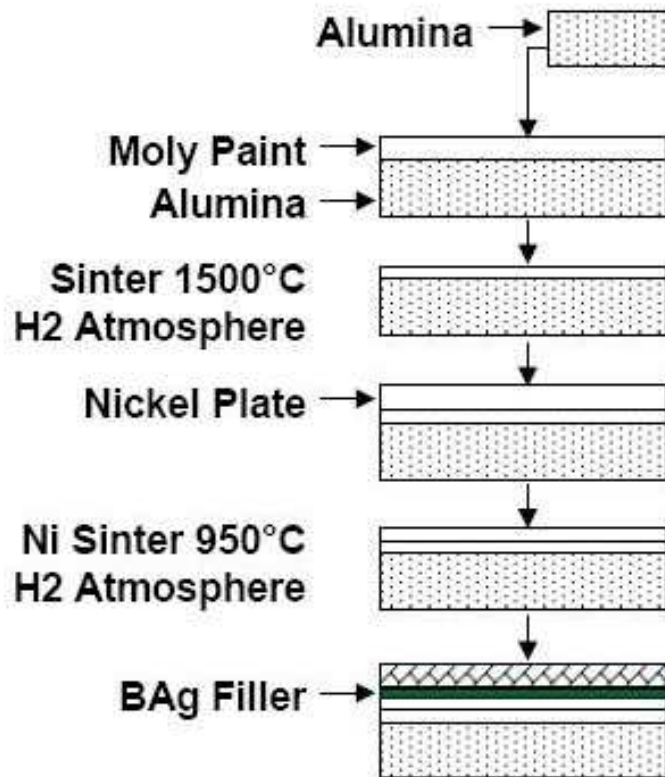
# Ceramics

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

# Permanent vacuum joints



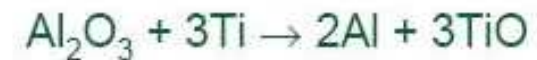
# Brazing



- Metallization Method

- Mo/Mn Paint Sintering
- Vapor Deposition

- Active Brazing



- 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

# Plastics



# Plastics

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

# Outgassing rates

	Outgassing rate (Torrl/scm <sup>2</sup> )	
	@ 10 h	Baked
Viton	$5 \times 10^{-8}$	$5 \times 10^{-10}$
Buna N	$2 \times 10^{-6}$	$4 \times 10^{-8}$
Silicon rubber	$8 \times 10^{-7}$	$6 \times 10^{-10}$
Epoxy (Shell Epon)	$1 \times 10^{-6}$	$8 \times 10^{-8}$
Teflon (poly'fluoro'lene)	$8 \times 10^{-8}$	$8 \times 10^{-9}$
Mylar	$1 \times 10^{-7}$	$2 \times 10^{-9}$
Nylon (polyamide)	$3 \times 10^{-7}$	$6 \times 10^{-9}$
PVC	$3 \times 10^{-7}$	$8 \times 10^{-8}$

# O-ring seal

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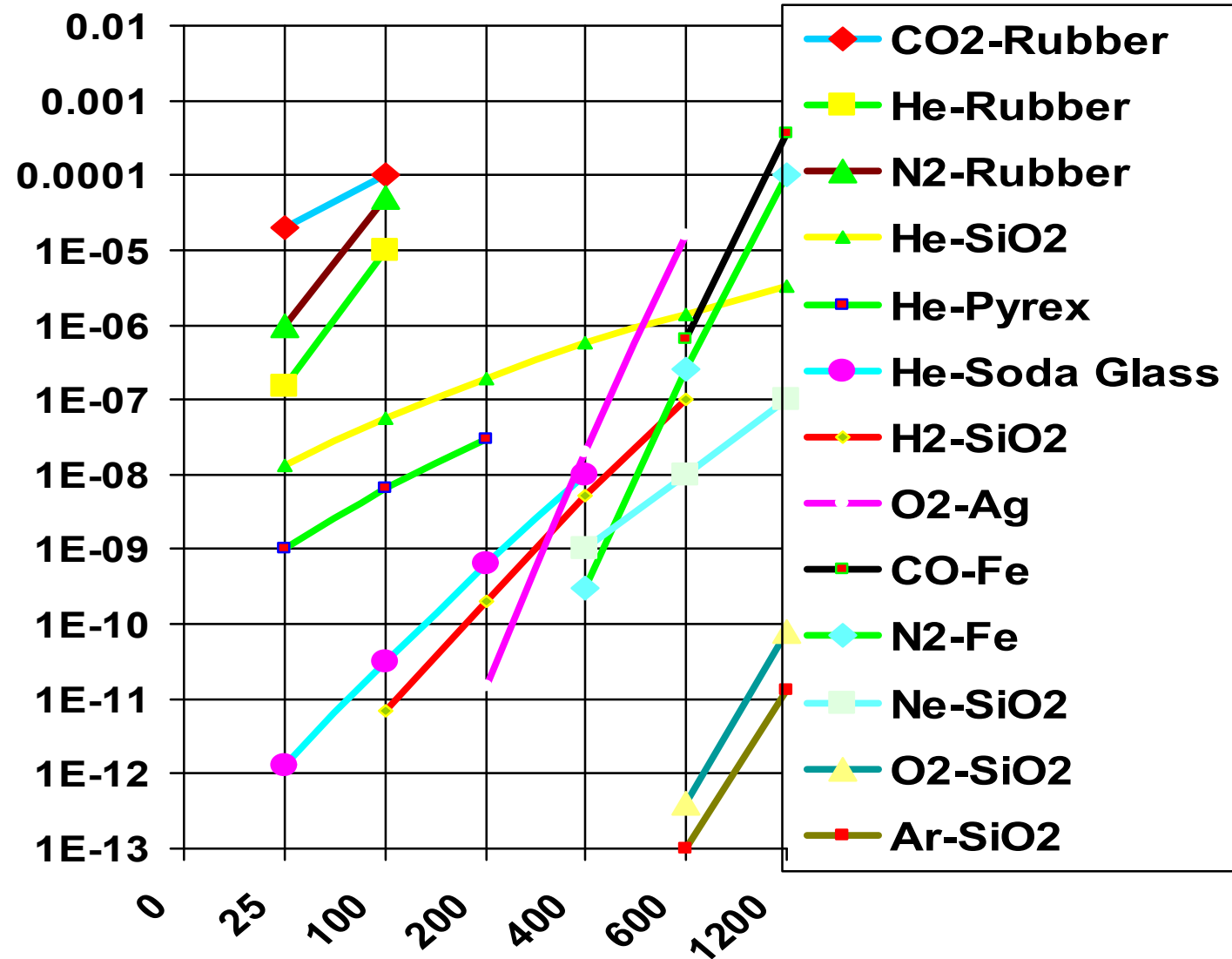




# Permeation

- 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



# Permeation

- Permeation is a strong function of temperature.
- Permeation gives additional gas loads.
  - Modifies the chamber environment(residual gases)
  - effecting 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 <sup>2</sup> (x10 <sup>-7</sup> ) at 1 atm	
	25°C	150°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.