Guidelines for Engineering and Manufacturing for Vacuum

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Head of ESS Vacuum Section - STS

European Spallation Source ERIC

22/Oct/2019

Outline

- Vacuum Standards for vacuum
- Cleaning components
- Vacuum welding
- Materials for vacuum
- Good practices in the field

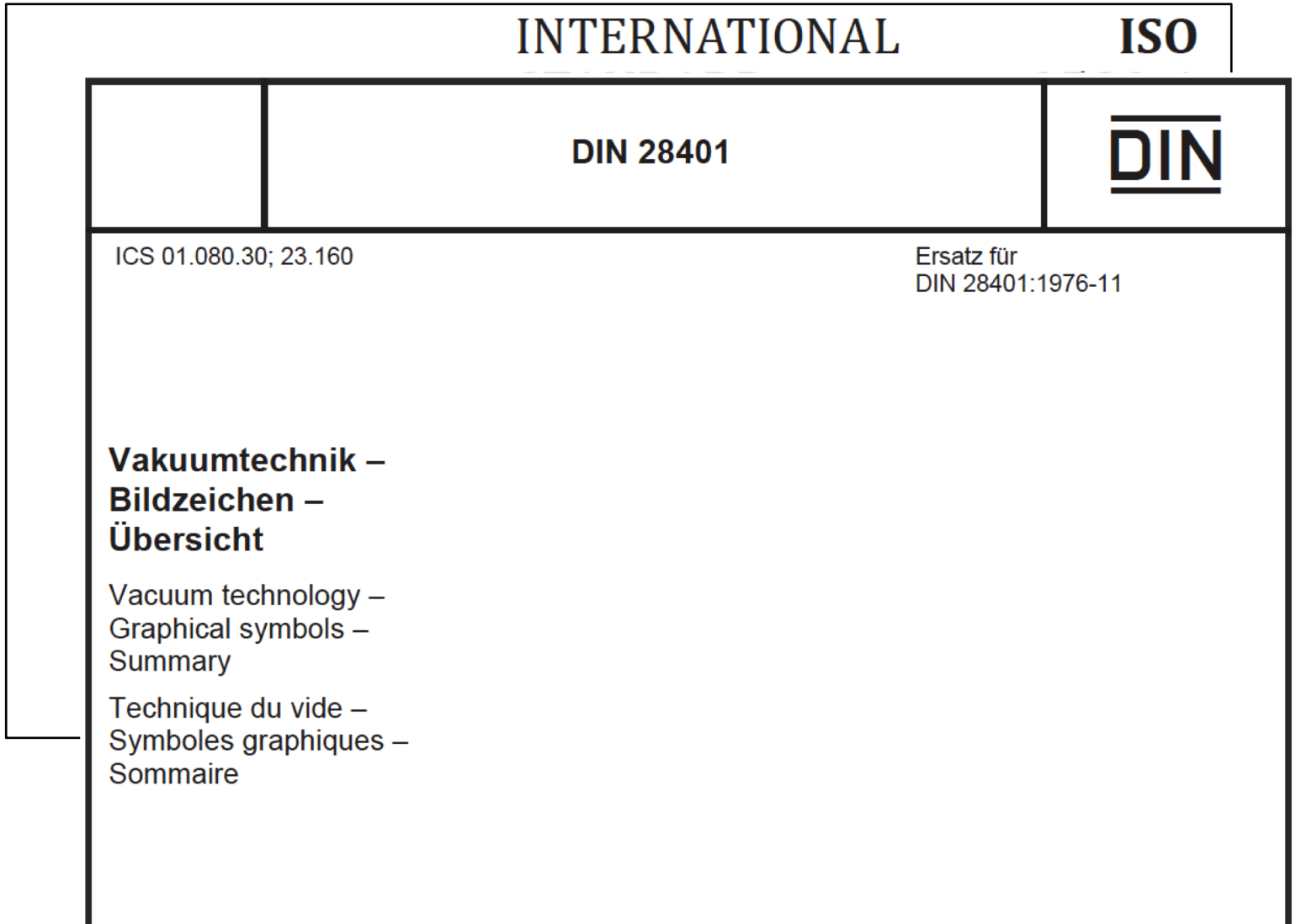
Vacuum Standards

Why vacuum standards?

Speaking the same language: Standard for vacuum vocabulary.

Vacuum symbols: no ISO!!!

Vacuum symbols: DIN



INTERNATIONAL ISO

DIN 28401

DIN

ICS 01.080.30; 23.160

Ersatz für
DIN 28401:1976-11

Vakuumtechnik – Bildzeichen – Übersicht

Vacuum technology – Graphical symbols – Summary

Technique du vide – Symboles graphiques – Sommaire

Vacuum Standards

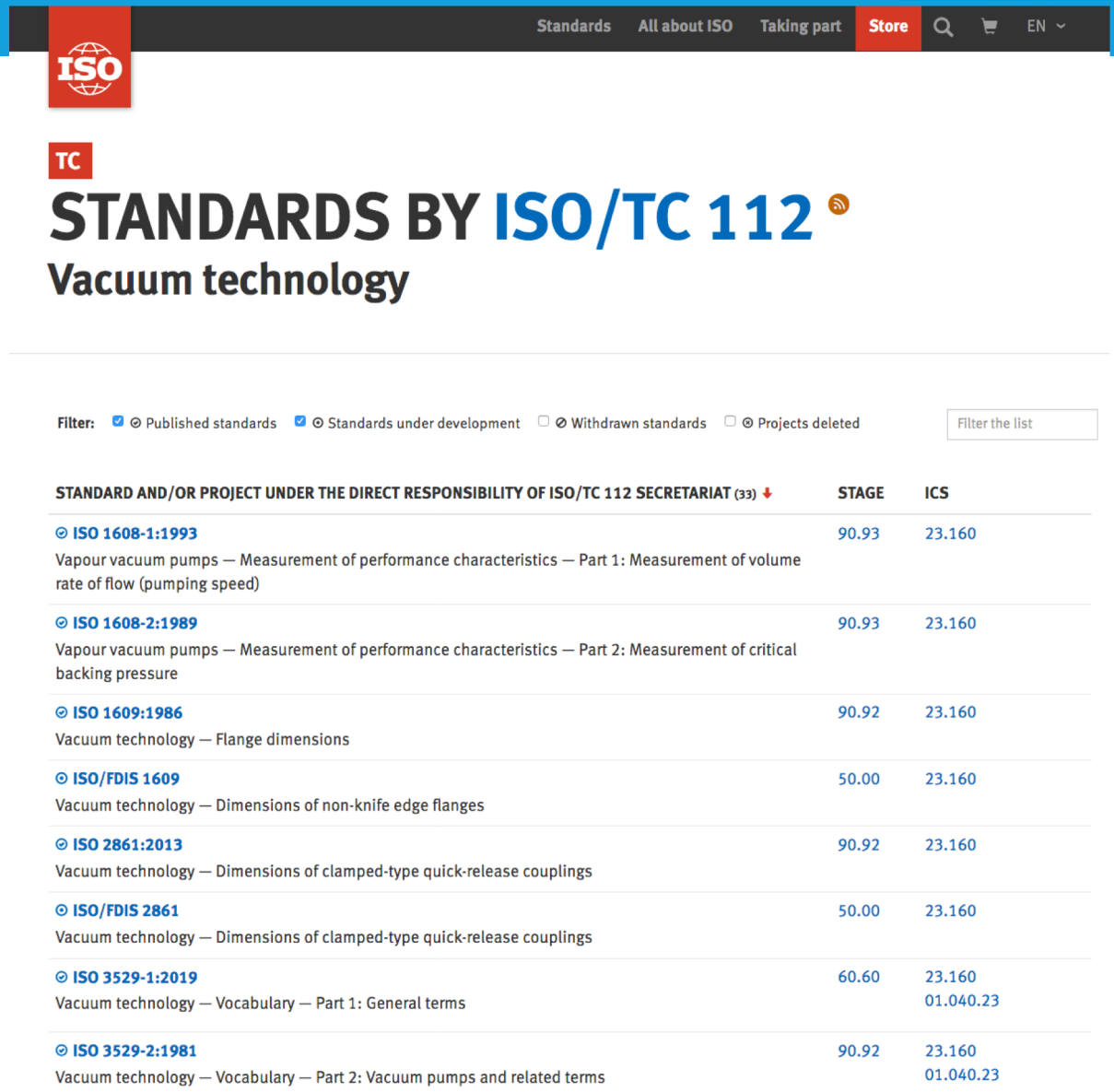
Vacuum systems are build only on vacuum standards?

Are there components for vacuum standardized?

Standards are on dimensions/shape, process and equipments.
NOT MATERIAL STANDARDS FOR VACUUM.

No vacuum standards for welding.
ONLY FOR LEAK TEST.

https://www.iso.org/committee/51654.html



ISO Standards All about ISO Taking part Store EN

TC

STANDARDS BY ISO/TC 112

Vacuum technology

Filter: Published standards Standards under development Withdrawn standards Projects deleted Filter the list

STANDARD AND/OR PROJECT UNDER THE DIRECT RESPONSIBILITY OF ISO/TC 112 SECRETARIAT (33)	STAGE	ICS
ISO 1608-1:1993 Vapour vacuum pumps — Measurement of performance characteristics — Part 1: Measurement of volume rate of flow (pumping speed)	90.93	23.160
ISO 1608-2:1989 Vapour vacuum pumps — Measurement of performance characteristics — Part 2: Measurement of critical backing pressure	90.93	23.160
ISO 1609:1986 Vacuum technology — Flange dimensions	90.92	23.160
ISO/FDIS 1609 Vacuum technology — Dimensions of non-knife edge flanges	50.00	23.160
ISO 2861:2013 Vacuum technology — Dimensions of clamped-type quick-release couplings	90.92	23.160
ISO/FDIS 2861 Vacuum technology — Dimensions of clamped-type quick-release couplings	50.00	23.160
ISO 3529-1:2019 Vacuum technology — Vocabulary — Part 1: General terms	60.60	23.160 01.040.23
ISO 3529-2:1981 Vacuum technology — Vocabulary — Part 2: Vacuum pumps and related terms	90.92	23.160 01.040.23

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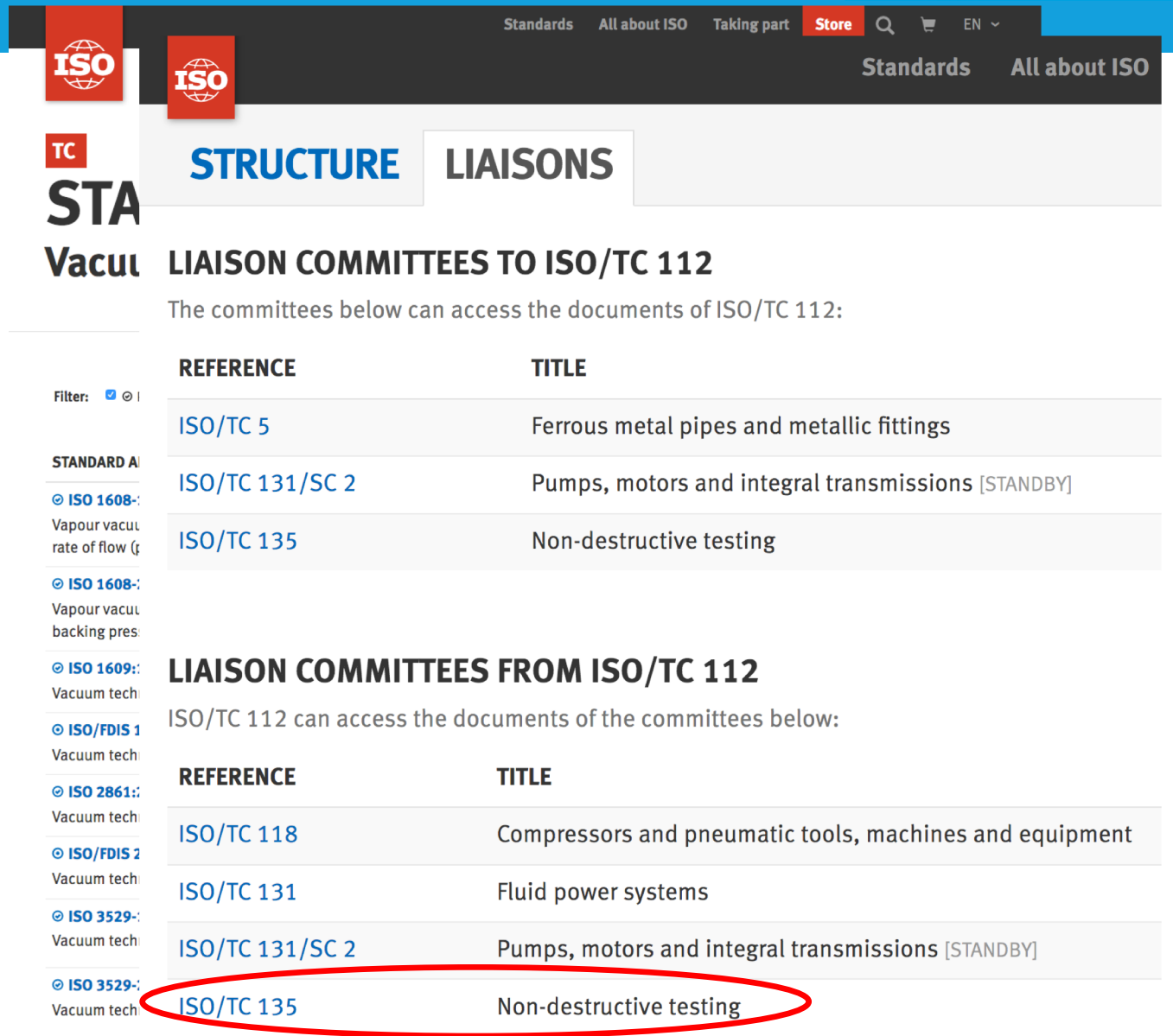
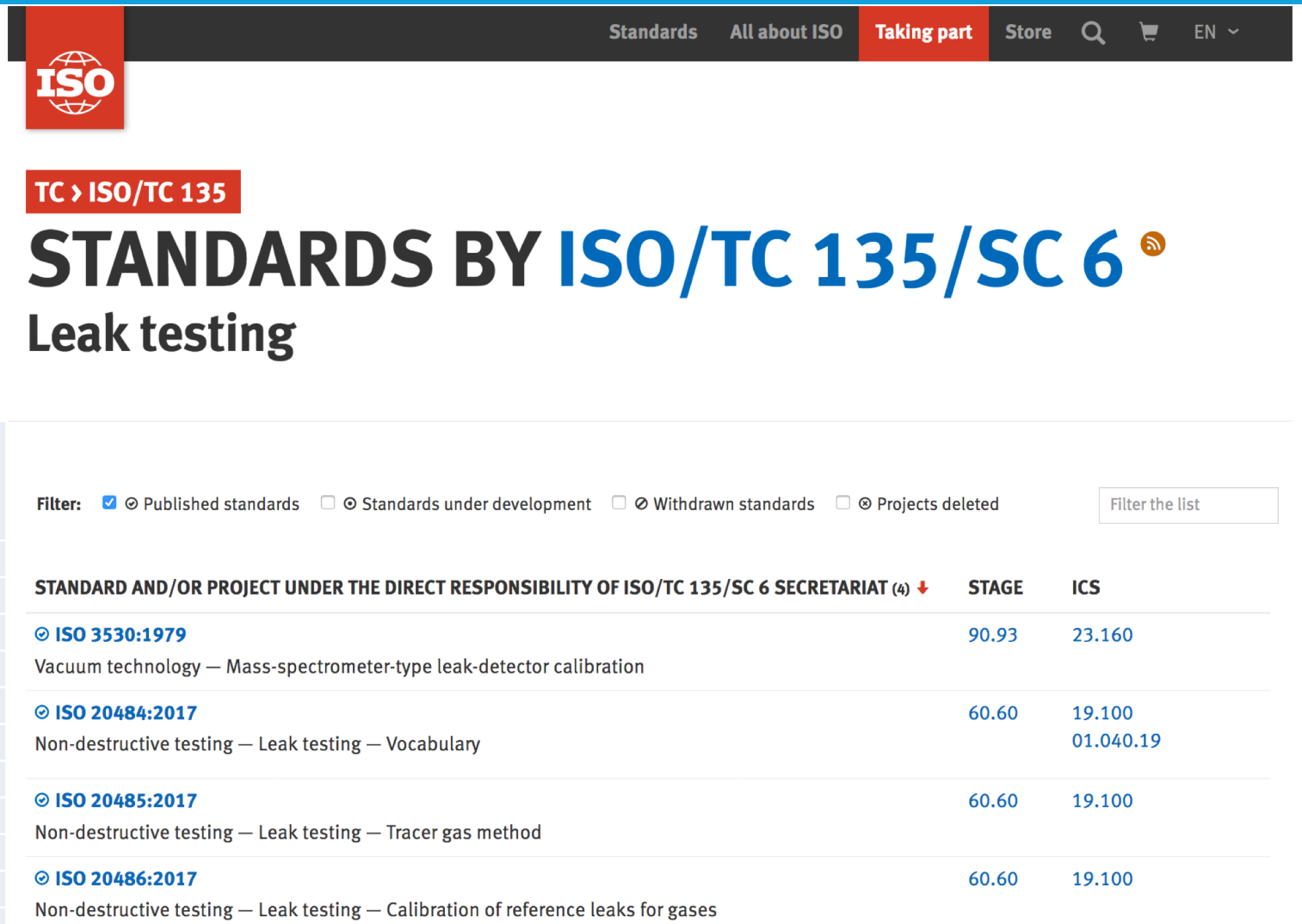


Vacuum Standards

Leak detection or Non-destructive testing.

Convert to:	**Multiply helium leak rate by factor:**
q of Hydrogen	2.23
q of Air	1.08
q of Water Vapour	2.09

mbar.l/s	time for one CC to leak		Time for one bubble to leak	
1,0E-01	10	sec	0,25	sec
1,0E-02	100	sec	2,5	sec
1,0E-03	16,7	min	25	sec
1,0E-04	2,8	h	4	min
1,0E-05	1,2	days	42	min
1,0E-06	11,6	days	7	h
1,0E-07	3,9	months	3	days
1,0E-08	3,2	years	1	month
1,0E-09	32,2	years	10	months
1,0E-10	321,5	years	8	years
1,0E-11	3215,0	years	80	years



Flanges types

Vacuum flanges types:

- Clamped-type Quick-release coupling, it is known as **KF**, several types of gaskets.

ISO 2861 only up to DN 50 flange size, under development by TC 112.

No standard for clamps, options by application.

KF CLAMPS

Stainless steel clamp

Amagnetic clamp

Bolt clamp

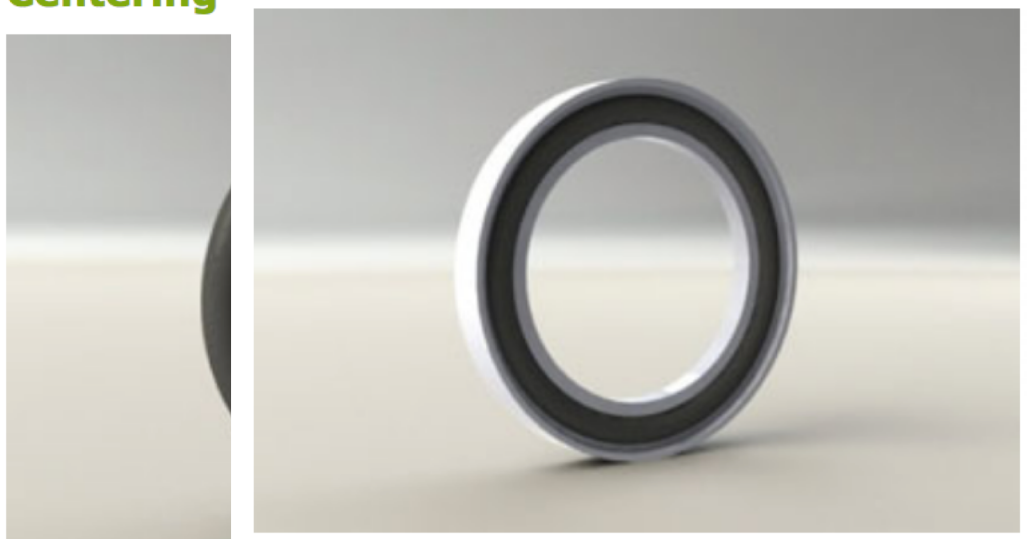
KF CENTERING RINGS

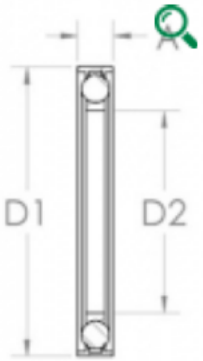
crew)

Alur

Centering

Trapped centering ring





- pres
- pressure rang
- temperature
- other o-rings

- 304L inner
- Aluminum outer
- seal : Viton (or other seal on request - ex : Nitrile, EPDM, ...)
- pressure range: 10^{-7} mbar to 10 bar
- temperature range : -10 to 200°C

Flanges types

Vacuum flanges types:

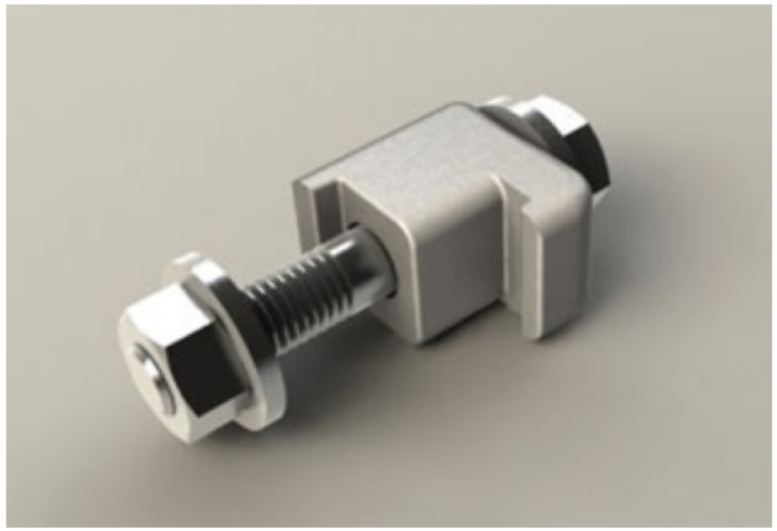
- ISO flanges: bolts and clamps, fixed or rotatebles types usual for sizes ≧ DN63.

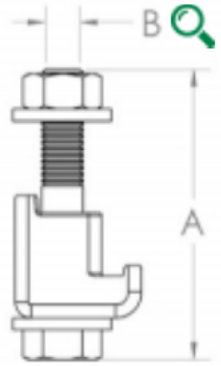
ISO 1609 non-knife edge flanges, standard under development.

Double claw - stainless steel

Double claw clamps - aluminum

Wall clamps, nut & washer - steel





- used with through bolt holes and centering ring
- also available with aluminum clamps on request

250	261	10	5	2,5	290	275	285
320	318	15	7,5	2,5	370	355	365
400	400	15	7,5	4	450	435	442
500	501	15	7,5	4	550	535	542
630	651	20	10	5	690	660	680

* See 3.2. It should be noted that the nominal bores recommended above 630 are: 800, 1 000, 1 250, 1 600, 2 000 and 2 500.

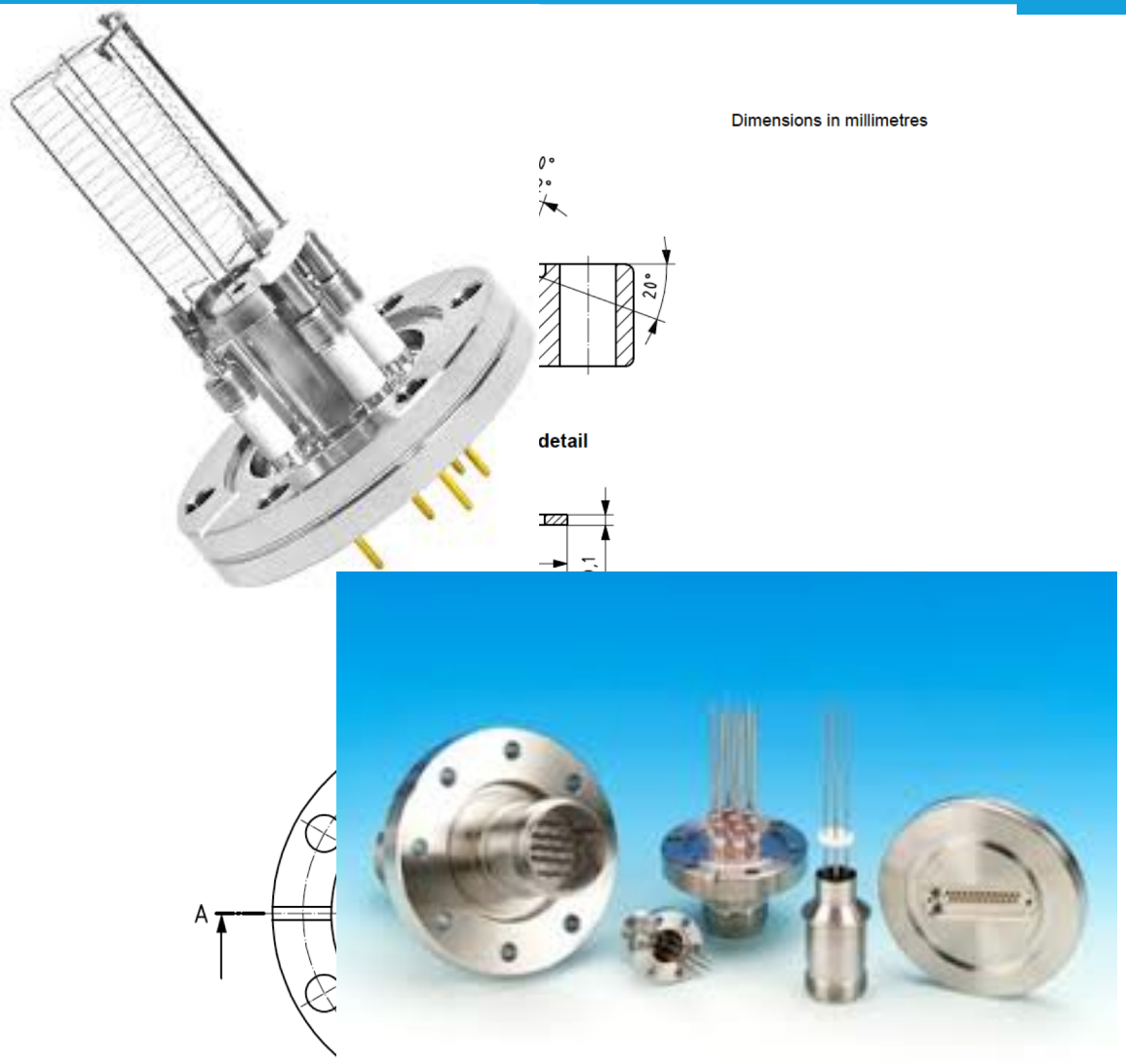
** See 3.5.2.

*** See 3.9.

Flanges types

Vacuum flanges types:

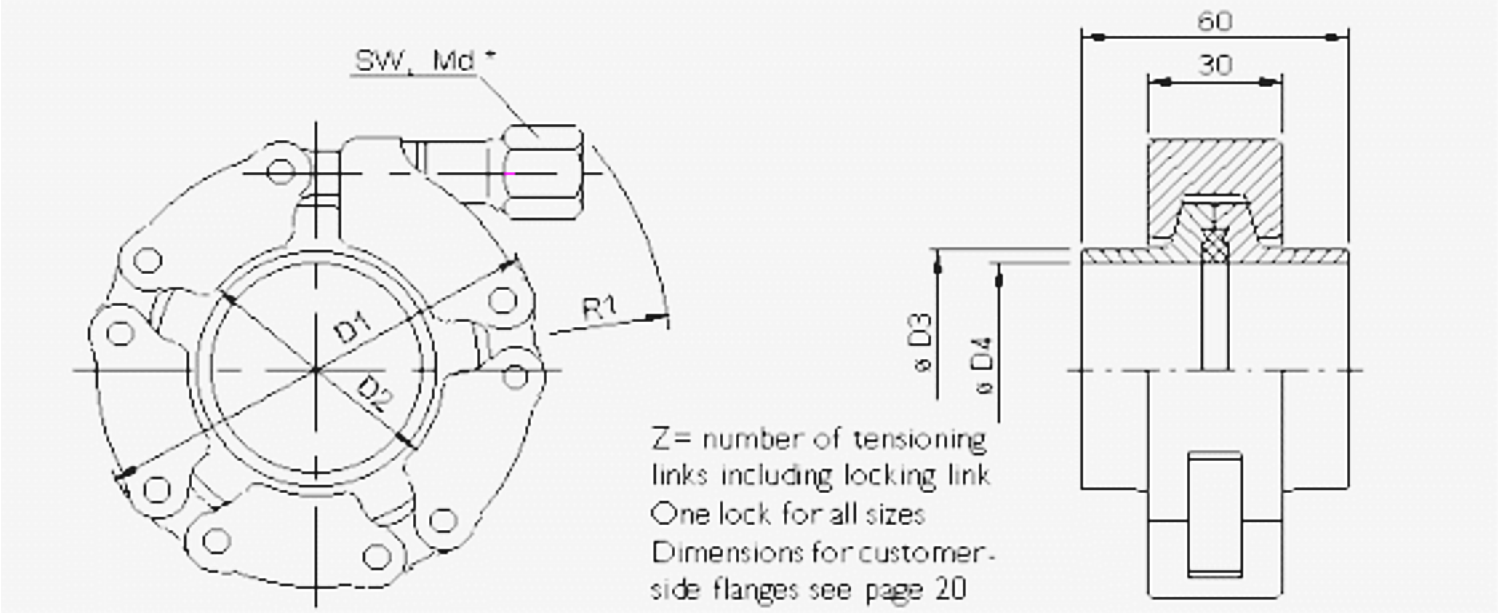
- ConFlat ™ (former Varian Inc.) knife-edge bakable flanges (UHV), it is known as **CF**.



Flanges types

Vacuum flanges types:

- Not standardized flanges: KF-CF, Cefix or CF quick-release.



Class “0” of vacuum : monolayer

CAS – CERN-94
A.G. Mathewson

How many molecules we have at the surface of a cube of 1 liter?

Place one molecule of **nitrogen** by side another, over the cube surface (definition of monolayers).

6 side = 0.010 x 0.010 x 6 = 0.06 m^2

$$\frac{1}{3.7 \cdot 10^{-10} \times 3.7 \cdot 10^{-10}} = 7.3 \cdot 10^{18} \text{ molecule/m}^2$$

$$0.06 \times 7.3 \cdot 10^{18} = \mathbf{4.4 \cdot 10^{17}\ molecules}$$

The molecular diameters are measured in Ångström (1 Å=10^{-10} m).
Diameter of **nitrogen** molecule : 3.7 Å

Class “0” of vacuum : pressure equivalent

What is one monolayer of gas as pressure equivalent?

Using the **ideal gas law** at **standard references** conditions:

$2.69 \cdot 10^{22}$ molecules in 1 liter.

$$\frac{\mathbf{4.38 \cdot 10^{17}} \times 101{,}325}{2.69 \cdot 10^{22}} = \mathbf{1.65\ Pa\ (1.65 \times 10^{-2}\ mbar)\ medium\ vacuum!!}$$

Class “0” of vacuum : gas in solid solution

EUROPEAN SPALLATION SOURCE

How much gas we have in solid solution on stailess steel (SS) 304?

Typical value (ASTM handbook) for **nitrogen** on austenitic phase is **150 ppm in weight**. SS304 density: $8 \cdot 10^3$ g/liter

$$150 \text{ ppm} = \frac{150}{10^6} \times 8 \cdot 10^3 = \mathbf{1.20 \text{ g/liter}}$$

Using the **ideal gas law** at **standard references** conditions:

$2.69 \cdot 10^{22}$ molecules in 1 liter => $4.77 \cdot 10^{-23} \times 2.69 \cdot 10^{22} = \mathbf{1.28 \text{ g}}$

≈ 150 ppm of nitrogen in SS304 is equivalent of 1 bar atmosphere!!

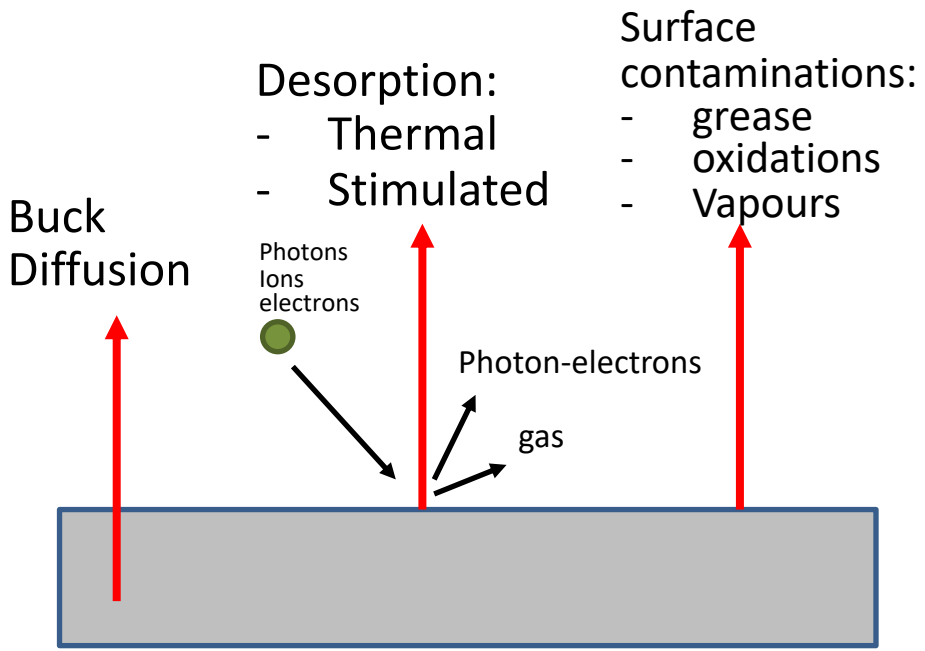
1 molecule of **nitrogen** weight = $4.77 \cdot 10^{-23}$ g

Cleaning components

K. Middleman
STFC/Daresbury

Contaminant: prevents vacuum system reaching required base pressure or introduces unwanted species into the residual gas.

Outgassing: The outgassing rate is the time-dependent rate at which gases and vapours are released under vacuum. This limits the ultimate pressure achievable and can introduce contamination into the vacuum system



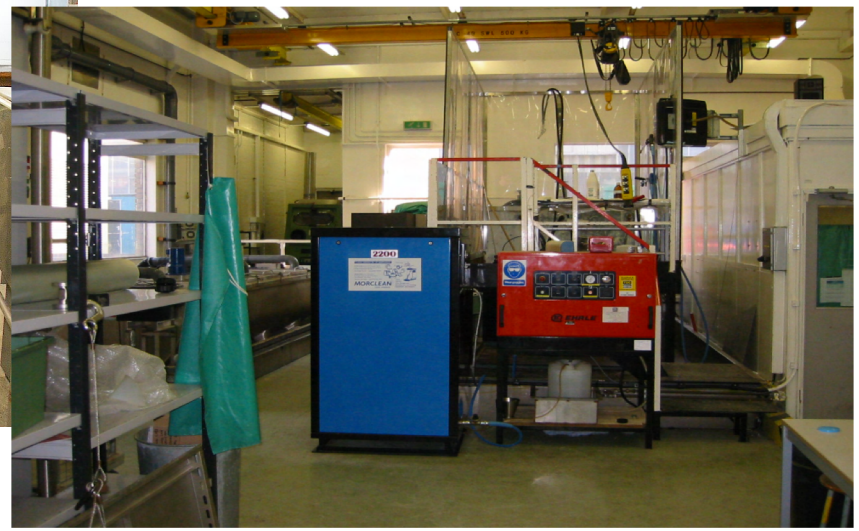
Cleaning methods

Chemical	Mechanical	Thermal	Surface
Wash (Solvent or detergent)	Bead blasting	Vacuum bakeout	Polishing
Water jet	Ultrasonic	Vacuum fire (950 C under vacuum)	Electro-polishing
Vapour (solvent)	CO_2 snow	Air bake (up to 400 C)	Plasma etch
Pickling		Vacuum remelt	Diamond turning
Plasma cleaning (H_2 or O_2)			

Cleaning “recipes”

Cleaning procedures are specific for each applications:

- ESS cleaning procedures are prepared for UHV describe at ESS Vacuum Handbook.



4.2.1 Aluminium alloy

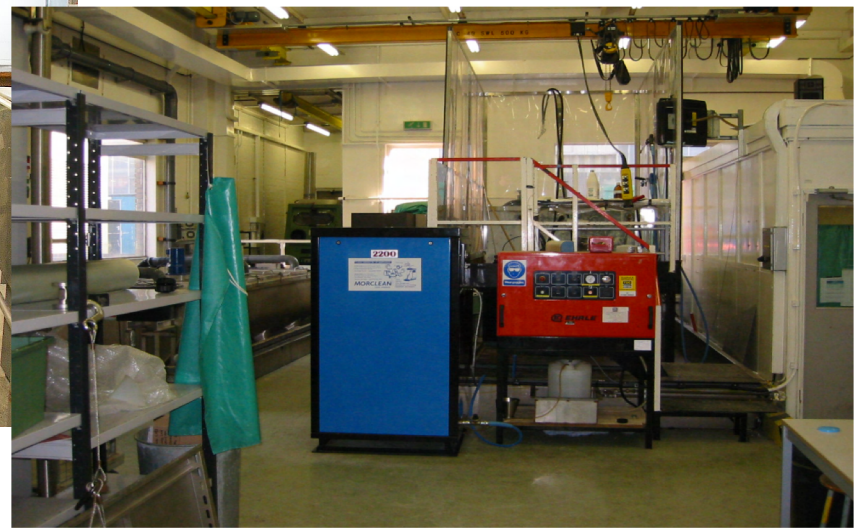
METHODOLOGY:

- Chemical degreasing with detergent and ultrasonic
    - o Formulation and operating parameters:
        - Detergent NGL 17.40 spec. ALU III: 10 g/l.
        - Temperature: 50°C.
        - Time: 30 – 60 minutes.
    - o Rinsing with water
- Pickling
    - o Formulation and operating parameters:
        - Caustic soda: 42 g/l
        - Temperature: 60°C.
        - Time: 10 – 30 seconds.
    - o Rinsing with water
- Neutralization with detergent and ultrasonic
    - o Formulation and operating parameters:
        - Nitric acid: 400 ml/l.
        - Hydrofluoric acid: 8.5 ml/l
        - Temperature: 20°C.
        - Time: 1 – 5 minutes.
    - o Rinsing with water.
- Rinsing with demineralised water
- Drying with clean compressed air and bake-out at 60°C

Cleaning “recipes”

Cleaning procedures are specific for each applications:

- ESS cleaning procedures are prepared for UHV describe at ESS Vacuum Handbook.

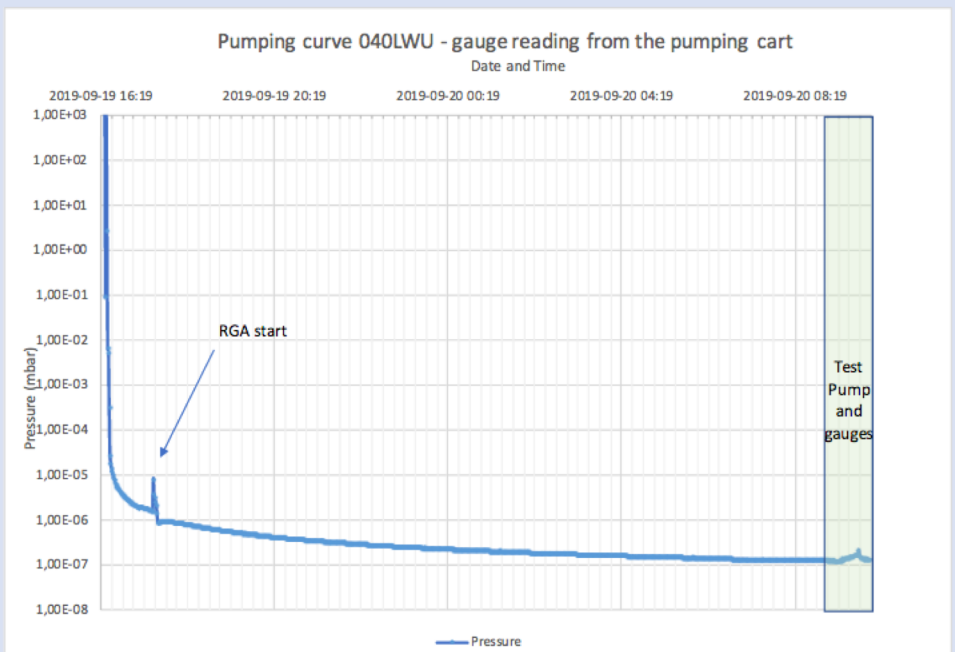


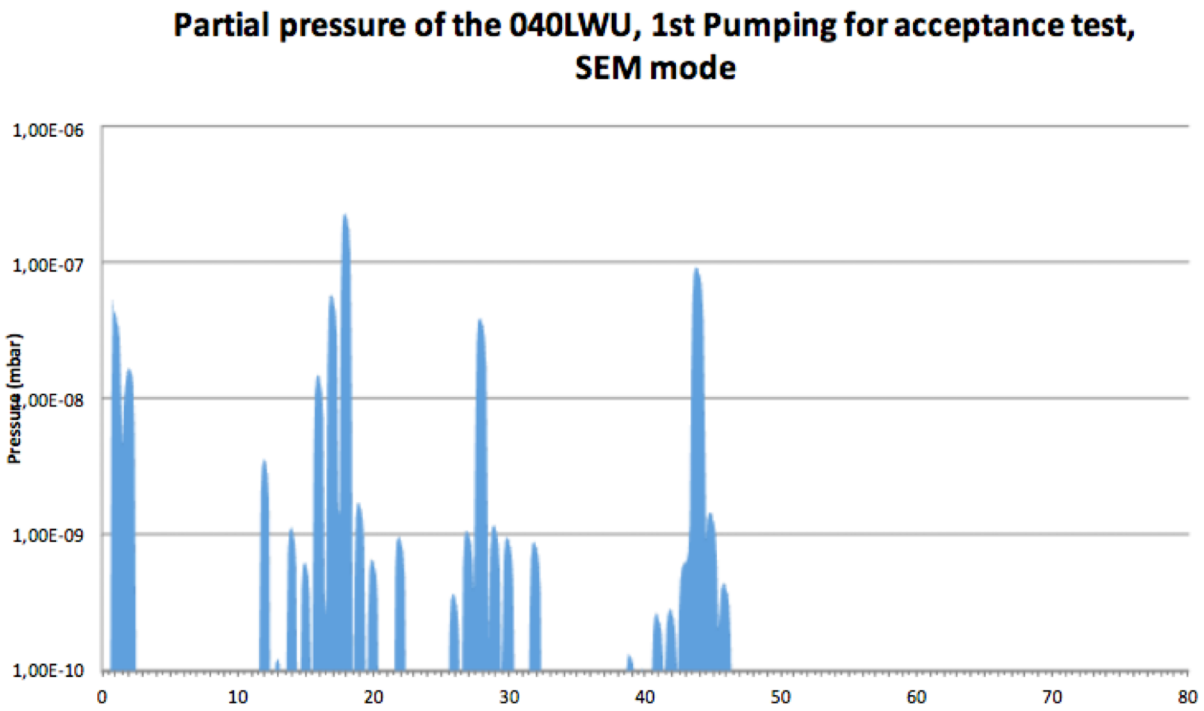
4.2.2 Stainless steel

METHODOLOGY:

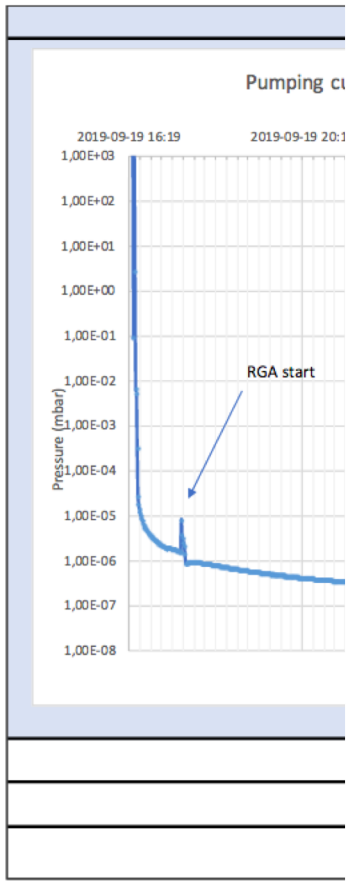
- Chemical degreasing with detergent and ultrasonic
  - o Formulation and operating parameters:
    - Detergent NGL 17.40 spec. ALU III: 10 g/l
    - Temperature: 50 – 60°C
    - Time: 30 – 60 minutes
  - o Rinsing with water
- Pickling
  - o Formulation and operating parameters:
    - Net inox (pure): HNO3 (~ 50 %) + HF (~ 3 %)
    - Temperature: 20°C
    - Time: 30 – 90 minutes
  - o Rinsing with water
- Neutralization with detergent and ultrasonic
  - o Formulation and operating parameters:
    - Detergent NGL 17.40 spec. ALU III: 10 g/l.
    - Temperature: 50 – 60°C.
    - Time: 5 – 10 minutes.
  - o Rinsing with water.
- Rinsing with demineralised water and alcohol
- Drying with clean compressed air and bake-out at 60°C

Cleaning results

Pressure curve

Action(s) taken : none
Action(s) to take : none
STATUS : ACCEPTED



Cleaning results



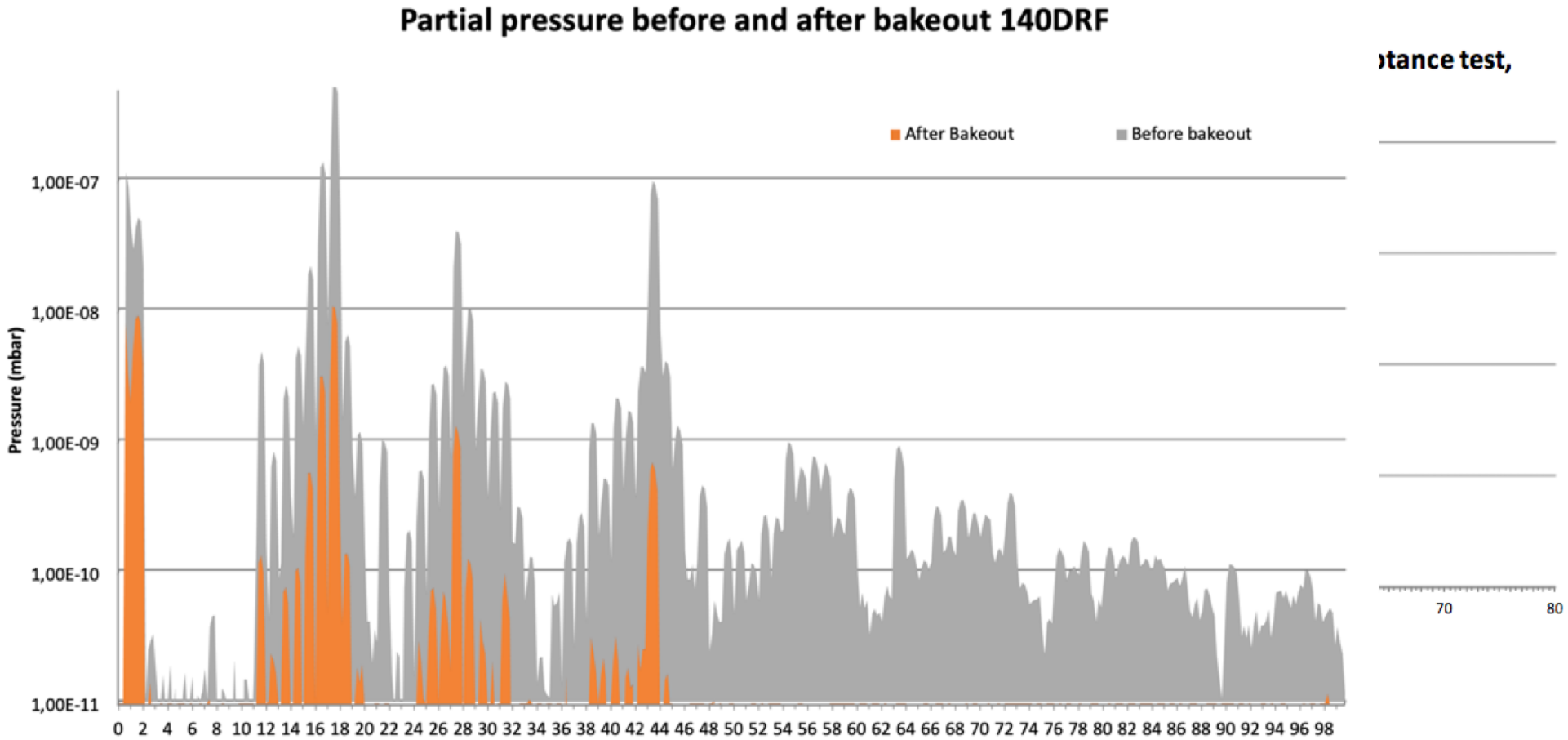
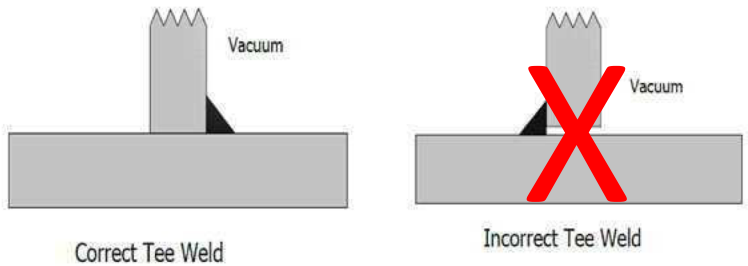


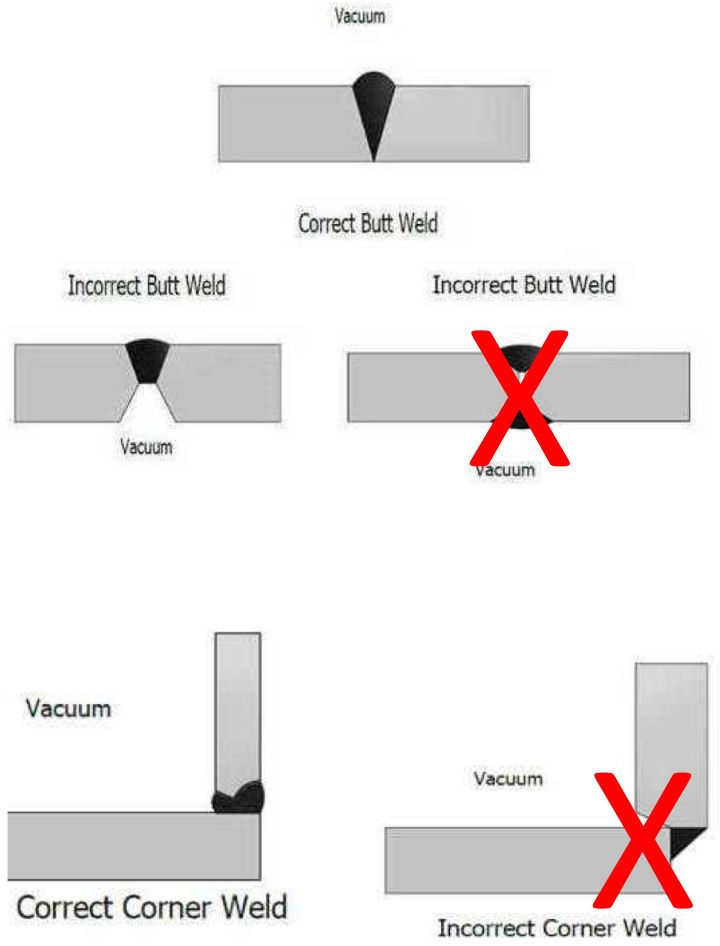
Fig : exemple of RGA before and after a bakeout at 150 °C during 16 hours

Vacuum Welding

When designing or constructing a vacuum system, the following points need to be observed:

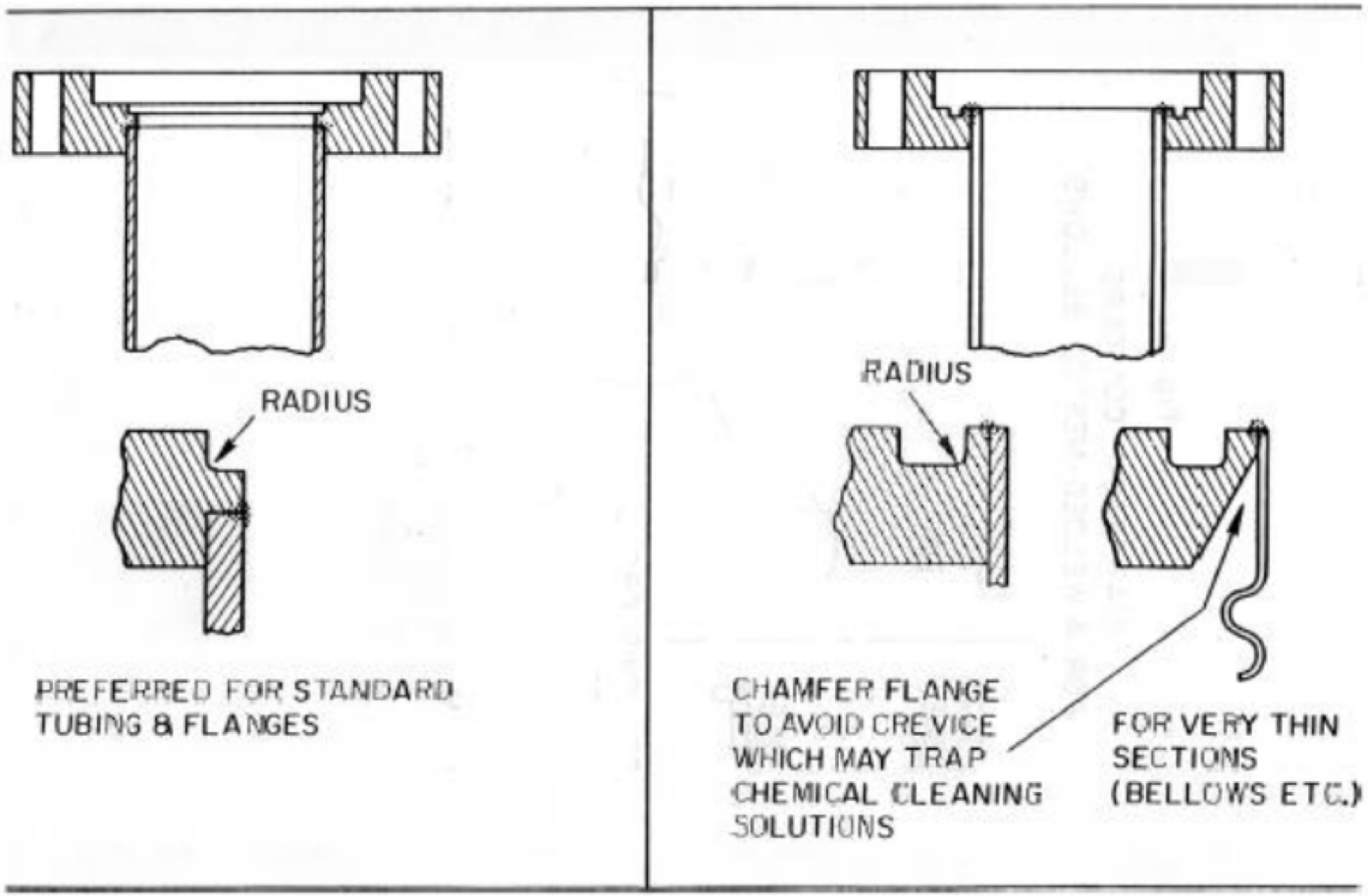
- Full penetration welds wherever possible to avoid pockets where volumes of gas or contaminants can be trapped.
- Single pass welds wherever possible to avoid trapped volumes that could be generated with multi-pass welds.
- Welds shall always be made on the vacuum side of the joint.
- If for structural reasons double welds are required, an easy path to flow gas from the joint shall be available. This could be in the form of a machined hole between the two welds or a discontinuous weld on the non-vacuum side.





Vacuum Welding

Preferred joint design for welding vacuum flanges.



Vacuum Welding

Welding area clean for vacuum weld.

Assembling area close to welding station.

Assembling already clean for UHV before welding!!!

Materials for vacuum

Materials used for vacuum systems and components of the accelerator and other systems exposed to vacuum and operating at <10^{-5} Pa (<10^{-7} mbar) shall be selected from the approved list of materials for UHV applications UNLESS specific approval.
It is important to ensure that the correct fabrication techniques (e.g. only the use of water-soluble machining lubricants for manufacture,) handling and cleaning procedures are used so as not to compromise the vacuum performance of the selected material.

ESS test all materials to be used at the vacuum system on a specific Outgassing Facility.

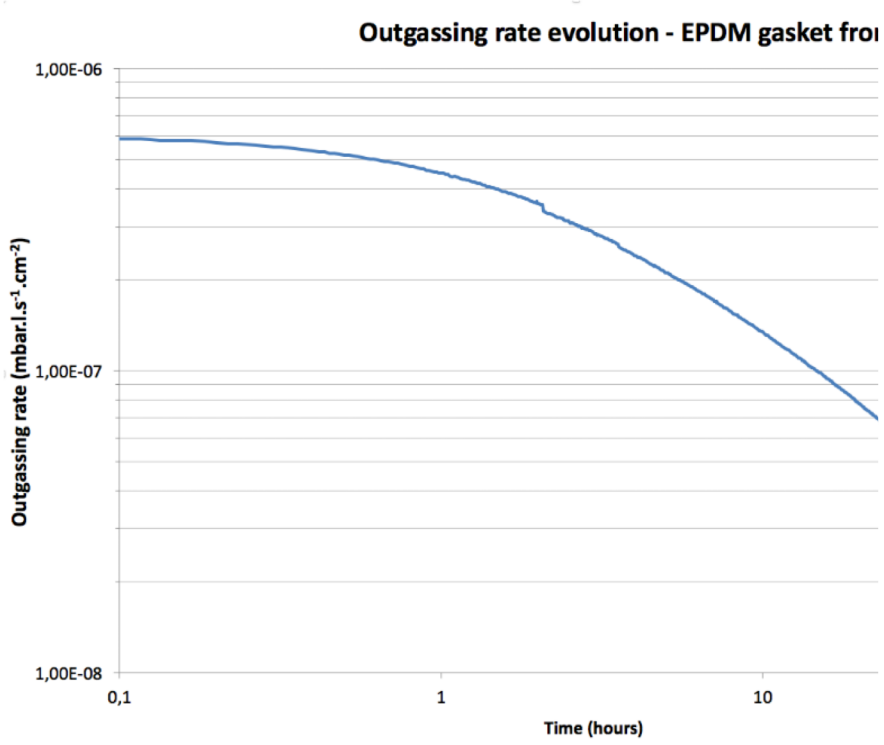
Approved UHV Materials List:
- Stainless Steel type 304 & 316 series
- Copper OFHC
- Aluminium and its alloys. Do not use cast components.
- Gold
- Silver
- Titanium
- Molybdenum
- Platinum
- Beryllium Copper
- Ceramic (as Al2O3) >90%
- Machinable glass (Macor)

Prohibited Materials List:
- Brass
- Soft Solder
- Standard Hard Solder
- Electrical Solder
- All Plastics
- All Glues
- Greases
- Silicon or Sulphur based machining lubricants
- Any material containing: Zinc, Cadmium, Phosphorus, Sodium, Selenium,
Potassium or Magnesium

Material Outgassing test

EUROPEAN SPALLATION SOURCE

EPDM Nichias



Date: 30/11/17 Ref. number: ESS-0194750	OUTGASSING TEST of EPDM sheet from Nichias	EUROPEAN SPALLATION SOURCE

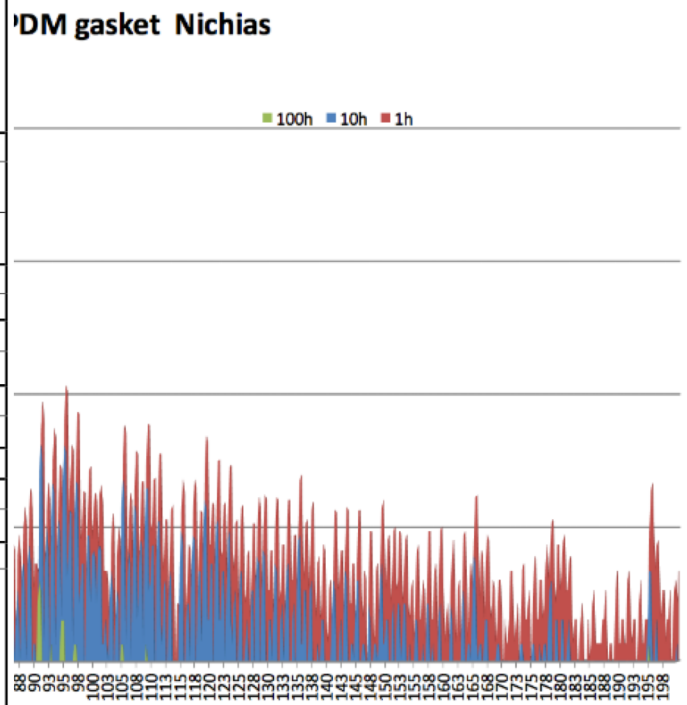
SAMPLE DESCRIPTION	
Material: EPDM	**Provider**: Nichias
Dimension [cm]: DN100	**Surface**: 44 cm^2

TEST FACILITY AND EQUIPMENT	
ESS outgassing test facility (See ANNEX 3). Test carried out according to Throughput Method of [REF 3].	
Gauges: IONIVAC IE514 N_2 calibrated according to [REF 1].	**RGA:** Hiden HALO 200 RC
PRE-TEST TREATMENTS	
Cleaning: None, used as received	**Bake-out**: NO
BLANK TEST	
Chamber Gas-Load: $5x10^{-8}$ mbar l/s	**RGA scan**: No anomalies (see ANNEX 4)
PREPUMPING IN THE LOADLOCK CHAMBER	
Time to reach <10^{-6} mbar: 50 min	**Total time in the loadlock :** 50 min
TEST PERFORMED AT: room temperature	

OUTGASSING DATA WITH BACKGROUND SUBTRACTED [mbar l/s/cm^2]		
OGR @1hour: $4x10^{-7}$	**OGR @10hours**: $1x10^{-7}$	**OGR @ 100hours**: $2x10^{-8}$

REFERENCES	
REF 1. ISO 3567:2011(E) -	Vacuum gauges, Calibration by direct comparison with a reference gauge.
REF 2. J. Vac. Sci. Technol. A 25(1) Jan/Feb 2007 -	Recommended practice for process sampling for for partial pressure analysis.
REF 3. AVS recommended practice -	Recommended practices for measuring and report outgassing data.

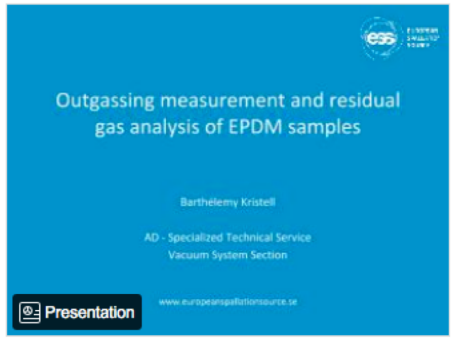
Test executor: K. Barthelemy	Checker: S.M. Scolari	Approver: M. J. Ferreira



Material Database for Vacuum

EPDM

Created by Kristell Barthelemy, last modified by Marcelo Juni Ferreira on Mar 19, 2019



Name (chess linked to their outgassing data report)	Type	Pumping time to reach <10-4 Pa	Outgassing rate (Pa.m3.s-1.m-2)			RGA	Comments
EPIDOR	gasket DN100	ABORTED					
PXL	gasket DN100	ABORTED					
Garlock	sheet	ABORTED					
DEMACO	gasket DN100	22h40	2x10-4	2x10-4	1x10-4	Bad	
DEMACO	sheet	12h15	2x10-4	7x10-4	3x10-4	Bad	
70 Perox Dichtomatik	gasket DN100	4h25	3x10-4	2x10-4	6x10-5	Intermediate	Pressure bumps probably due to gas traps inside the material
Nichias	gasket DN100	50 min	4x10-4	1x10-4	2x10-5	Bad	
EDPM shieldsield 663 from JW	sheet	1h10	2x10-4	2x10-4	4x10-5	Good	
Nichias	sheet	1h10	5x10-4	2x10-4	4x10-5	Intermediate	

Guideline for Vacuum

What is the Vacuum Guideline?

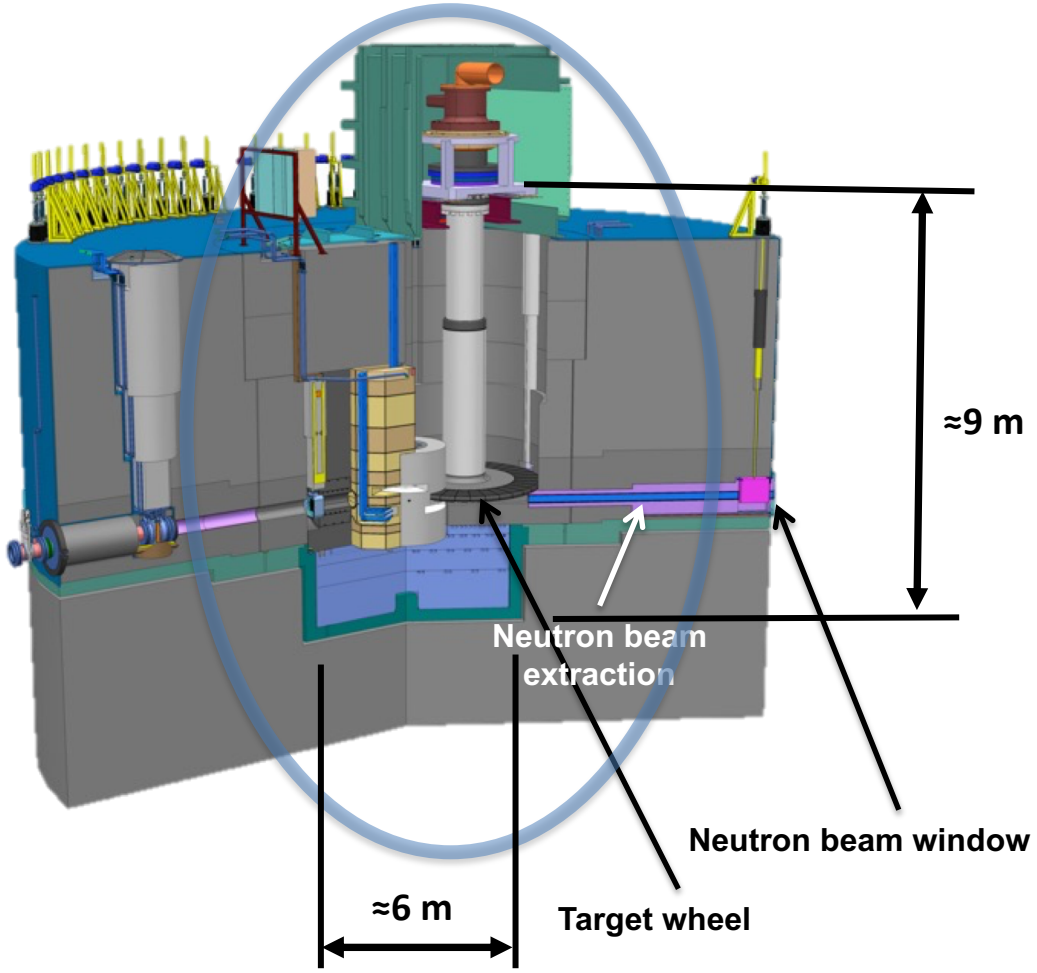
What is necessary for a vacuum project?

- Clear specifications of the vacuum necessary (life-time, maintenance, corrosion, cost & scheduling)

- Specifications can influence vacuum design or performance (gas sources, thermal/mechanical requirements, cooling, pressure, etc),

- Materials, design and fabrications processes can influence vacuum performance!!!

- Vacuum never forgive!!!!



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Thank you!
Tack!
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