

TECHNICAL NOTE

# UHV Guidelines for X-Ray Beam Transport Systems

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*M. Dommach*

*for X-Ray Optics and Beam Transport (WP73)*

*at the European XFEL*

European X-Ray Free-Electron Laser Facility GmbH

Albert-Einstein-Ring 19

22761 Hamburg

Germany



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# 1 General information

This document presents guidelines for ultrahigh vacuum (UHV) components for X-ray beam transport systems at the European XFEL facility.

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## 1.1 Introduction

The effective and reliable operation of the European XFEL X-ray beam transport systems and their connected instruments is strongly related to obtaining and keeping UHV conditions. These guidelines for UHV components at the European XFEL contain rules and important information on the design, manufacturing, cleaning, handling, and acceptance testing of UHV components to be installed in the European XFEL vacuum systems.

This document describes two levels of vacuum requirements: UHV systems and particle-free UHV systems. The requirements for particle-free UHV systems apply to vacuum components that are located in an area 30 m in front of or behind a mirror or grating. If you are unsure which requirements you were asked to comply with, ask your contact person at European XFEL.

Deviations from these guidelines must be clarified and accepted in advance by European XFEL.

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## 1.2 UHV-compatible design

Make sure that your UHV components meet the following requirements:

### 1.2.1 Flanges

For X-ray beam transport systems, use flanges of the “knife-edge” type (according to ISO 3669-2:2007) that are sealed by means of a flat circular gasket made of oxygen-free high conductivity (OFHC) copper. The gasket cannot be reused.

Flanges must be made of the electro slag remelted (ESR) version of the preferred stainless steel types.

For flange connections use unplated stainless steel (grade A2-70 or better) screws and CuNiSi (Copper-Nickel-Silicon alloy) nuts.

### 1.2.2 Designs

- **Choose designs that:**

- Avoid virtual leaks. For example, use vented screws or vent tapped holes.
- Allow easy cleaning. For example, avoid inaccessible volumes. This is of particular importance if a component has to be particle-free.

- **Rotatable flanges**

Unless otherwise stated all vacuum components will have one fixed and one rotatable flange for the “x-ray beam ports. The rotatable flange points downstream the x-ray beam.

- **Surface roughness**

The inner roughness of vacuum components should be  $R_a \leq 0.8 \mu\text{m}$ , except for welding seams.

- **Water connections**

Do not use brazed, welded, or other joints to separate UHV from water.

## 2 UHV-compatible materials

Only materials that are compatible with ultrahigh vacuum (UHV) may be used for the manufacturing of vacuum components. Table 1 lists materials that are compatible or incompatible with UHV, respectively. Use of materials other than those listed as UHV-compatible is permitted only after approval by European XFEL and before the beginning of production.

Generally, materials with high vapour pressure are not UHV compatible, in particular cadmium, lead, potassium, selenium, sodium, sulphur, and zinc, or an alloy containing one or more of the materials listed in Table 1.

**Table 1:** Material that are compatible or incompatible with UHV

Type	Compatible	Incompatible
Pure metals	Aluminium Beryllium Copper Indium Molybdenum Tantalum Titanium Tungsten	(e.g. Cadmium, Lead, Zinc)
Stainless steel	Preferred types: 304L and 316L for pipes, 316LN ESR for flanges	Steel containing incompatible materials (e.g. 303, 303S, 303Se)
Alloys	Aluminum (preferred types: EN AW-5086, EN AW-6061 T6, EN AW-6063 T6) Beryllium-copper DENSIMET <sup>®</sup> , GLIDKOP <sup>®</sup> INCONEL <sup>®</sup> 600, 718 Tin-bronze	Inappropriate Aluminium alloys (e.g. 7000 series) alloys containing Lead or Zinc (e.g. Brass)
Other	Aluminium ceramics Boron carbide (B <sub>4</sub> C) Diamond, Sapphire Macor <sup>®</sup> , PEEK, PI (Kapton <sup>®</sup> , Vespel <sup>®</sup> )	Organic materials Glue

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## 2.1 Electrical connections inside vacuum

Cables used inside vacuum have to be Kapton<sup>®</sup>- or ceramic-insulated. Other insulation materials (for example, PTFE or silicone) are prohibited. Voltages and currents carried by the cables may not exceed the manufacturer's ratings. Cable connections must be made by crimping or screwing. Soldered connections are prohibited.

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## 2.2 Feedthroughs

This section describes the requirements for feedthroughs.

### 2.2.1 Electrical feedthroughs

Feedthroughs used for electrical connections into the vacuum system must be of the ceramic-to-metal type. No glass-to-metal feedthroughs are permitted. The preferred installation method is feedthroughs that are mounted in a flange. Feedthroughs may also be welded directly to a chamber when a flange connection is not feasible. Care must be exercised to prevent the weld from putting undue stress on the ceramic. Voltages and currents carried by the feedthroughs must not exceed the manufacturer's ratings. Quality control of feedthroughs consists of visual and mechanical inspection of dimensions, as shown by the appropriate drawing and leak checking. External covers should be provided to protect the ceramics from damage after installation.

### 2.2.2 Mechanical feedthroughs

Bellow-type mechanical feedthroughs or magnetically coupled feedthroughs have to be used to impart a rotational or linear movement to the vacuum. O-ring-sealed feedthroughs are not permitted. Feedthroughs must adhere to all applicable sections of this specification with regard to materials, fabrication, cleaning, welding, leak tightness, bakeout capability, and so on. Installation of these feedthroughs must be by means of a "knife edge"-type flange. Feedthroughs that have bearings exposed to vacuum must be

evaluated regarding their UHV suitability. Feedthroughs containing parts that cannot be cleaned may not be used.

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## 3 Guidelines for UHV systems

The chapter includes guidelines for manufacturing, assembling, cleaning, testing, and packing ultrahigh vacuum (UHV) systems.

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### 3.1 Manufacturing and assembly

When manufacturing and assembling UHV systems, follow these guidelines:

■ **Cleaning and degreasing**

Clean and degrease all machined parts before welding.

For example, do the following:

— *Prewashing*

Use a high-pressure cleaner to remove coarse contaminations and then wash the parts with a suitable detergent.

— *Pickling*

Choose a pickling bath that is suited to the material and the pickling of UHV components. Immediately afterwards, rinse with deionized water. Do not pickle flanges.

Possible etching methods:

– *Aluminium*

Diluted NaOH, water rinse, neutralization (*Dekapierung*) with diluted nitric acid, water rinse, and drying.

– *Stainless steel*

Water-based mixtures of HF + HNO<sub>3</sub> or HF + H<sub>2</sub>SO<sub>4</sub>, water rinse, and drying.

## ■ **Welding**

Complete welds on the vacuum side of the vessel only. If the welds have to be done from the outside, fill the interior of the vessel with inert gas. Avoid spillage inside the vacuum vessel and sink holes in the welding seam as well as sagging. If necessary, cover the welds from inside and flush them with shielding gas. Perform the welds with full penetration.

Acceptable welding procedures:

- Electron beam
- Laser
- Metal inert-gas (MIG)
- Plasma
- Tungsten inert-gas (TIG)

Brush welds only with suitable brushes that are not contaminated by other materials.

## ■ **Brazing joints**

Braze joints only under a vacuum or shielding gas atmosphere without a fluxing agent. Before brazing, get approval from European XFEL of the brazing solders to be used.

## ■ **Cleaning parts**

Clean vacuum parts that will be assembled into larger units in advance. Clean the parts after mechanical manufacturing, taking into account the UHV cleaning requirements.

## ■ **Leak testing**

Before final assembly, leak-test smaller units and welds, which are not accessible separately after mounting.

## ■ **Marking**

If you are asked to mark the vacuum components (indicated on the technical drawing) follow the guidelines given in European XFEL drawing 4-12-5868-B-000, which can be found on page 19 of this document.

## ■ **Avoiding contamination**

Avoid contamination of the components during assembly and welding processes.

Therefore, do the following:

- Mount and weld in a clean room separated from the mechanical manufacturing.
- Wear clean, lint-free gloves during assembly.

■ **Surface treatments**

Such as glass-bead blasting or electro polishing, must be approved in advance by European XFEL.

■ **Machining**

Before performing necessary subsequent machining, get approval from European XFEL. For a cooling fluid, use isopropanol only. Use new cutting tools. Standard cooling lubricants and cutting oils are not allowed.

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## 3.2 Cleaning

Pre-cleaning must be applied to welding seams and holes. Wipe all in-vacuum welds with isopropanol. Holes and threaded holes shall be pre-cleaned with isopropanol using brushes or special sticks. After the general cleaning, check all holes and welds for residual contamination.

### 3.2.1 Cleaning of large items

When cleaning large UHV systems, like pipes, chambers or vessels, follow these guidelines (list of suitable cleaning companies is available on request):

- 1 Chemical mechanical precleaning (based on  $\text{HNO}_3$ )
- 2 Rinsing with water (resistivity  $\geq 1 \text{ M}\Omega\text{cm}^{-1}$  at  $25^\circ\text{C}$ )
- 3 Depending on the dimensions of the item to be cleaned, do one of the following:
  - Long pipe sections: Anodical cleaning (based on  $\text{H}_2\text{SO}_4$  and  $\text{H}_3\text{PO}_4$ )
  - Vacuum chambers, vessels or buffers: Pickling (based on HF and  $\text{HNO}_3$ )
- 4 Rinsing with water (resistivity  $\geq 1 \text{ M}\Omega\text{cm}^{-1}$  at  $25^\circ\text{C}$ )

- 5 Chemical passivation of the surface (based on  $\text{HNO}_3$ )
- 6 Rinsing with water (resistivity  $\geq 1 \text{ M}\Omega\text{cm}^{-1}$  at  $25^\circ\text{C}$ )
- 7 Final treatment (for particle-free cleaning, continue with Section 4.1, “Cleaning and assembly”)
  - Rinsing with  $80^\circ\text{C}$  DI water (resistivity  $\geq 15 \text{ M}\Omega\text{cm}^{-1}$  at  $25^\circ\text{C}$ )
  - Drying with Nitrogen (quality 5.0 or better)
  - Quality control (visual inspection, surface roughness)
  - Closing with clean plastic flange caps

### 3.2.2 Cleaning of small parts

Smaller UHV parts can be cleaned in a laboratory washing machine using Neodisher<sup>®</sup> detergent and DI water for rinsing.

Alternatively, you can use an ultrasonic bath with DI water and a suitable detergent (for example, Tickopur R33). Perform ultrasound operations (for example, 3 to 6 times for 5 min.), interrupted by short rinsing periods (at least 1 min.).

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## 3.3 Parts handling

Handle clean parts on all vacuum surfaces with clean, lint-free gloves only.

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## 3.4 Packing

When packing UHV systems, follow these guidelines:

### ■ Vacuum vessels

Fill vacuum vessels with dry nitrogen and seal them off with blank flanges or seal flanges with polyethylene plastic foil. Protect knife edges and sealing surfaces with clean plastic flange caps. Alternatively, wrap the evacuated vacuum vessel in a double layer of PE-foil.

### ■ Vacuum pipes

- Close vacuum pipes with clean plastic flange caps. Mark rotatable flanges of pipes longer than 2000 mm with an orange sticker (cleanroom-compatible tape) on the tube next to the flange
- Sleeved and sealed in a double layer of PE foil, inner foil filled with Nitrogen

### ■ Other components

Wrap and seal other components in a double layer of PE foil.

### ■ Transport

For transport, fix movable components (for example, bellows and rotating flanges).

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# 4 Particle-free UHV systems

To obtain ultrahigh vacuum (UHV) components that are particle free, follow the guidelines in this chapter **in addition** to the guidelines in Chapter 3, “Guidelines for UHV systems”.

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## 4.1 Cleaning and assembly

### 4.1.1 Cleaning of large items

#### ■ Final treatment in cleanroom ISO 14644-1 class 5 or better

After a UHV-compatible cleaning, described in Section 3.2, “Cleaning”, perform the following steps inside a cleanroom:

- Rinsing with 80°C DI-water (resistivity  $\geq 15 \text{ M}\Omega\text{cm}^{-1}$  at 25°C)
- Drying with Nitrogen (Quality 5.0 or better)
- Quality control (visual inspection, surface roughness)
- Closing with clean plastic flange caps

#### ■ Testing the absence of particles

The absence of particles must be verified using a suitable particle counter inside a cleanroom ISO 14644-1 class 5 or better. For the measurement, create a turbulent flow of particle-free (ionized) nitrogen inside the component and place the particle counter where most of the flow will come out. Run the measurement procedure for at least 5 minutes (longer for very large items or complex geometries). Determining factor is always the particle count for the smallest measurable particle size according to Table 2.

**Table 2:** Maximum number of particles for ISO 14644-1 class5

Particle size [ $\mu\text{m}$ ]	0,3	0,5	1,0	0,5
Particles/ $\text{m}^3$	10.200	3.520	832	29

#### ■ Getting approval

Due to the multitude of components to be analysed and their different geometries, there is no standardized test setup or specification of the procedure. Thus, a suitable positioning of the particle counter and the chosen blow process for the components must be approved by European XFEL.

#### 4.1.2 Cleaning of small parts

Use a laboratory washing machine operating with DI water and suitable detergents like Neodisher®. Unload in a room with ISO 14644-1 class 5 or better. When using ultrasonic bath for particle cleaning, it is mandatory to clean in several steps, changing the position of the parts (for example, 3 to 6 times for 5 min.).

As an alternative to a lab washer or an ultrasonic bath, you can place components that cannot be cleaned easily (for example, mirrors, detectors, and so on) in a cleanroom in a particle-free airflow (ISO 14644-1 class 5 or better) for several hours before assembly.

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## 4.2 Parts handling

Assemble components and join vacuum parts in an environment according to the specified cleanroom class. For assembly in cleanrooms, use only unlubricated and uncoated screws that do not cold-weld (for example, titanium screws in components made of stainless steel).

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## 4.3 Packing

When packing components for particle-free UHV systems, follow these guidelines:

### ■ Vacuum vessels

Fill vacuum vessels with dry particle-free nitrogen and seal them off with blank flanges or seal flanges with particle free PE foil. Protect knife edges and sealing surfaces with clean plastic flange caps. Alternatively, wrap the evacuated vacuum vessel in particle free PE foil. Mask gaps in flange connections on the outside with cleanroom-compatible adhesive tape.

### ■ Vacuum pipes

- Close vacuum pipes with clean plastic flange caps. Mark rotatable flanges of pipes longer than 2000 mm with a sticker (cleanroom compatible tape) on the tube next to the flange
- Sleeved and sealed in a double layer of particle free PE foil, inner foil filled with dry particle-free Nitrogen

### ■ Other components

Wrap and seal other components in a double layer of particle free PE foil.

### ■ Transport

For transport, fix movable components (for example, bellows and rotating flanges).

### ■ Aluminum foil

Aluminium foil is strictly forbidden for packing particle-free UHV components.

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# 5 Vacuum tests

This chapter includes guidelines for acceptance testing of UHV systems and particle-free UHV systems.

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## 5.1 Leak test

When performing leak tests, follow these guidelines:

- **Recommended procedures**

Follow the recommended leak-test procedures described in ASTM E498, “Standard Test Methods for Leaks Using the Mass Spectrometer Leak Detector or Residual Gas Analyser in the Tracer Probe Mode”.

- **Integral leak rate acceptance criterion**

Make sure that the integral leak rate (sum of all leaks) is  $\leq 1 \cdot 10^{-10}$  mbar·l·sec<sup>-1</sup>.

- **Inaccessible units and welds**

Before assembly, perform leak test on units and welds that are no longer accessible after assembly.

- **Equipment**

Perform the leak test using a standard leak detector or an RGA. Before performing the test, verify the required sensitivity and the correct function of the leak detector with a test leak.

- **Baked components**

Before and after the thermal treatment, leak test components at room temperature.

- **Vessel leak test**

For the final leak test of vessels, use metal gaskets.

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## 5.2 Residual gas analysis

The outgassing of all UHV components must be free of hydrocarbons. Perform the appropriate proofs using a sufficiently sensitive residual gas analyser (RGA), usually equipped with a secondary electron multiplier (SEM). The test procedure has to be performed and documented twice with the same parameters: the pumping system itself and with the component to be tested attached.

Components are considered to be clean when the respective conditions are fulfilled.

### 5.2.1 Acceptance criteria for unbaked vacuum systems

- The mass 18 peak of the leak-free system reaches a pressure below  $5 \cdot 10^{-8}$  mbar.
- After 15 hours of pumping the RGA spectra are recorded:
  - all mass peaks between mass 18 and 44 have to be 100 times lower than the mass 18 peak, except masses 28, 32 and 44
  - All mass peaks from mass 45 to at least mass 100 have to be 1000 times lower than the mass 18 peak.

### 5.2.2 Acceptance criteria for baked vacuum systems

- Leak-free system reaches a total pressure below  $10^{-7}$  mbar.
- Sum of the partial pressures of masses from mass 45 on to at least mass 100 is less than  $10^{-3}$  of the total pressure.

For documentation, a mass spectrum (at least Masses 1–100 amu, resolution  $1 \cdot 10^{-14}$  mbar)) of each component is needed, as well as a reference spectrum of the applied pumping system itself.

The integral specific desorption rate for baked components should be  $\leq 5 \cdot 10^{-12}$  mbar·l·sec<sup>-1</sup>·cm<sup>-2</sup>.

The method of verification must be approved by European XFEL.

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## **5.3 Pressure test of cooling water circuits**

Every water-cooled UHV device has to undergo a pressure test prior to installation. There should be no noticeable drop in pressure within eight hours after applying the 1.6 fold of the later used working pressure.

# A Appendix

## A.1 Instruction for marking vacuum parts

<sup>1</sup> Mechanically or laser engraved: *only in the main component of the pipe work, i.e. where  $L_{max}$  or  $D_{N_{max}}$*

XFEL EU "Doc. No." (check "text box" as highlighted)  
 "Vendor name" "Vendor product code" "Vendor serial No." (max 30 characters including blank)  
 Tube material: "Material according to EN10027-2" "Calendar week and year production ended"

Example:

XFEL EU	2-12-5862-A-000	Weight	0.000 kg
COMPANY-X	AC000 A0050123	Date	
Tube material:	14404 30/2012	Drawn	
		Approved	
		Drawing No.	
		Rev. A2	For 24.01.2012 Blatt 1 of 3

<sup>2</sup> Mechanically or laser engraved:

XFEL EU "Vendor name" "Vendor product code"  
 "Vendor serial No." "Material according to EN10027-2" "Calendar week and year production ended"

Example:

XFEL EU	COMPANY-X	62012
A0050100	14429	12/2012

\* A is equal to pipe-length/2 for less than 1 m long pipes

PIPE & FLANGE DIMENSION	A (mm)	B (mm)	C (mm)	D (mm)	E (mm)	HEIGHT FONT PIPE (mm)	LENGTH FONT PIPE (mm)	HEIGHT FONT FLANGE (mm)	LENGTH FONT FLANGE (mm)
DN16	90	60	8			2	2		
DN40	300	60	14	9	30	4	2	4	1
DN63	300	90	22	12	60	6	3	5	2
DN100	300	90	22	12	60	6	3	5	2
DN160	300	120	28	14	90	8	4	6	3
≥DN200	300	120	28	14	90	8	4	6	3

K-Zug-ID / C-DRAW-ID: SE000792122		K-Rev. / C-REV:		K-Status: 0-Available	
Projekt / PROJECT: Arbeitspaket / WORKPACKAGE		Gruppe / GROUP: WP73		Ers. für / REPLACES: Ers. durch / REPLACED BY:	
Gewicht / WEIGHT: 0.000 kg		Halbzeug / SEMIFINISHED PRODUCT		Werkstoff / MATERIAL: Format/SIZE:	
Allg. Toleranzen / ISO 2768 GENERAL TOLERANCES ISO 13020 Toleranzgrundsatz / FUNDAMENTAL TOLERANCING PRINCIPLE: ISO 8015 Oberflächenangaben / SURFACE TEXTURE: ISO 1302, 4297, 4298		Maßstab / SCALE: 1:2 Teil-ID / PART-ID: SE000565619 Datum / DATE: 03.12.2014 Name / NAME: Di Felice		Titel / TITLE: Engraving specifications Dokument-Nr. / DOCUMENT NO.: 4-12-5868-B-000 Blatt / SHEET: 1 von / OF: 1	
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