

TECHNICAL NOTE

HV Guidelines for Scientific Instruments

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Contents

1	General information	3
1.1	Introduction.....	3
1.2	HV-compatible design	4
1.2.1	Flanges.....	4
1.2.2	Designs	4
2	HV-compatible materials	5
2.1	Electrical connections inside vacuum	5
2.2	Feedthroughs	6
2.2.1	Electrical feedthroughs.....	6
2.2.2	Mechanical feedthroughs	6
3	Guidelines for HV systems.....	7
3.1	Manufacturing and assembly	7
3.2	Cleaning	9
3.2.1	Cleaning of small parts.....	9
3.2.2	Cleaning of large items	9
3.3	Parts handling	10
3.3.1	Packing.....	10
4	Vacuum tests	11
4.1	Leak test.....	11
4.2	Residual gas analysis	12
4.2.1	Acceptance criteria for unbaked vacuum systems.....	12
4.2.2	Acceptance criteria for baked vacuum systems.....	12
4.3	Pressure test of cooling water circuits	13
A	Recommendation for marking vacuum parts.....	14

1 General information

This document presents guidelines for high vacuum (HV) components for X-ray beam experiments at the European XFEL facility. HV is vacuum in the pressure range of 10^{-3} to 10^{-7} mbar.

1.1 Introduction

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

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

1.2 HV-compatible design

Make sure that your HV components meet the following requirements.

1.2.1 Flanges

For flanges, KF, CF, ISO-K, and ISO-F types can be used. For the O-rings, a Viton or FKM elastomer is prescribed.

For flange connections, it is recommended to use unplated stainless steel screws (grade A2-70 or better) and CuNiSi nuts (copper–nickel–silicon alloy).

1.2.2 Designs

■ Leaks and cleaning

Choose designs that:

- Avoid virtual leaks. For example, use vented screws or vent tapped holes.
- Allow easy cleaning. For example, avoid inaccessible volumes.

■ Rotatable flanges

Unless otherwise stated, all vacuum components will have one fixed and one rotatable flange.

■ Surface roughness

The inner roughness of vacuum components should be $Ra \leq 3.2 \mu\text{m}$ for raw surfaces and $Ra \leq 1.6 \mu\text{m}$ for machined surfaces, except for welding seams. Sealing surfaces for O-rings should be $Ra \leq 0.8 \mu\text{m}$.

■ Water connections

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

2 HV-compatible materials

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

Generally, materials with high vapour pressure are not HV-compatible, in particular cadmium, lead, potassium, selenium, sodium, sculpture, 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 HV

Type	Compatible	Incompatible
Pure metals	Aluminium, beryllium, copper, indium, molybdenum, tantalum, titanium, tungsten	(e.g. cadmium, lead, zinc)
Stainless steel	304, 304L, 316, and 316L	Steel containing incompatible materials (e.g. 303, 303S, 303Se)
Alloys	Aluminum (5000 and 6000 series), beryllium-copper DENSIMET [®] , GLIDKOP [®] INCONEL [®] 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 ₄ C), diamond, sapphire Macor [®] , PEEK, PI (Kapton [®] , Vespel [®])	Organic materials, glue

2.1 Electrical connections inside vacuum

Cables used inside vacuum have to be Kapton[®]- or ceramic-insulated. Other insulation materials which contain silicone or halogen are prohibited. Cables isolated by polytetrafluoroethylene (PTFE) must be approved in advanced by European XFEL. 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.

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 must be approved in advance by the representative experts. Feedthroughs must adhere to all applicable sections of this specification with regard to materials, fabrication, cleaning, welding, leak tightness, bakeout capability, and so on. Feedthroughs that have bearings exposed to vacuum must be evaluated regarding their HV suitability. Feedthroughs containing parts that cannot be cleaned may not be used.

3 Guidelines for HV systems

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

3.1 Manufacturing and assembly

When manufacturing and assembling HV 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 HV components. Immediately afterwards, rinse with deionized (DI) water. Do not pickle flanges or sealing surfaces.

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 + HNO₃ or HF + H₂SO₄, water rinse, and drying.

■ **Welding**

Apply given standards (e.g. DIN EN ISO 3834, AD-2000). If full penetration is not possible, assure a sealing pass on the vacuum side of the vessel.

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 for 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 HV cleaning requirements.

■ **Leak testing**

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

■ **Marking / traceability**

To facilitate traceability, each part has to be marked with a unique serial number or description. The marking must be carried out so that it is visible throughout the entire production process and cannot be removed by the production or cleaning processes. A list with all parts and their unique serial number or description with reference to the used material and material certificate is to be provided by the contractor.

■ **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.

— Assure that no residuals of prior weld jobs can contaminate the HV components.

— Wear clean, lint-free gloves during assembly.

■ **Surface treatments**

Surface treatments (e.g. glass-bead blasting or electro polishing) or other treatments must be approved in advance by European XFEL.

■ **Machining**

For a cooling fluid, use isopropanol or fresh mixed standard cooling lubricants. Use new cutting tools only.

3.2 Cleaning

To obtain HV components that are clean, the cleaning and assembling environment needs to be certified as a Cleanroom ISO 14644-1 Class 7 or better.

3.2.1 Cleaning of small parts

Use a laboratory washing machine operating with deionized (DI) water and suitable detergents like Neodisher®. Alternatively, you can use an ultrasonic bath with DI water and a suitable detergent (e.g. Tickopur R33). Perform ultrasound operations (e.g. 3 to 6 times for 5 min.), interrupted by short rinsing periods (at least 1 min.), followed by rinsing with DI water to a resistivity $\geq 1 \text{ M}\Omega\text{cm}^{-1}$ at 25°C.

3.2.2 Cleaning of large items

When cleaning large HV components that cannot be cleaned in an ultrasonic bath or washing machine (e.g. long pipes, chambers, or vessels), the cleaning method must be approved in advance by a European XFEL representative.

3.3 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 (e.g. titanium screws in components made of stainless steel).

3.3.1 Packing

When packing HV 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 polyethylene (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.
- Sleeve and seal in a double layer of PE foil, with the 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 (e.g. bellows and rotating flanges).

- **Aluminum foil**

Aluminium foil is strictly forbidden for packing HV components.

4 Vacuum tests

This chapter includes guidelines for acceptance testing HV systems.

4.1 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”, or DIN EN 13185:2001, “Non-destructive testing – Leak testing – Tracer gas method”.

- **Integral leak rate acceptance criterion**

Make sure that the integral leak rate (sum of all leaks) is $\leq 1 \cdot 10^{-8}$ mbar·l·sec⁻¹.

- **Inaccessible units and welds**

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

- **Equipment**

Perform the leak test using a standard leak detector or a residual gas analyzer (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 or unused O-rings

4.2 Residual gas analysis

The outgassing of all HV components must be free of hydrocarbons. Perform the appropriate proofs using a sufficiently sensitive residual gas analyzer (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.

4.2.1 Acceptance criteria for unbaked vacuum systems

The acceptance criteria for unbaked vacuum systems include the following:

- Mass 18 peak of the leak-free system reaches a pressure below $1 \cdot 10^{-6}$ 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.

4.2.2 Acceptance criteria for baked vacuum systems

The acceptance criteria for baked vacuum systems include the following:

- 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 description rate for baked components should be $\leq 5 \cdot 10^{-12} \text{ mbar} \cdot \text{l} \cdot \text{sec}^{-1} \cdot \text{cm}^{-2}$.

The method of verification must be approved by European XFEL.

4.3 Pressure test of cooling water circuits

Every water-cooled HV 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 Recommendation for marking vacuum parts

1 Mechanically or laser engraved: *only in the main component of the pipe work, i.e. where Lmax or DNmax!*

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
 COMPANY-X AC000 A0050123
 Tube material: 14404 30/2012

2 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 B2012
 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

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