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Operating procedure for chemical degreasing of parts for high-vacuum and ultra-high-vacuum applications

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Summary

This document describes the procedure for chemical degreasing in aqueous phase of stainless steel, copper and aluminium components to be used under high-vacuum and ultra-high-vacuum. This procedure is applicable for the CERN TE-VSC surface treatment workshop which has the required equipment and technical competence.

Any application of this procedure in external facilities will not be considered as sufficient to qualify it as a suitable cleaning process for high-vacuum and ultra-high vacuum components.

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INTRODUCTION

Mechanical components for Ultra-High-Vacuum (UHV) are designed for application in systems with a pressure $<10^{-9}$ mbar. The surface can be exposed to static vacuum or radiation from a particle beam in an accelerator (electron cloud, synchrotron radiation, ion bombardment). The cleanliness level is fundamental to reach and maintain such a low gas pressure.

Several elements and compounds can pollute the surface and have negative effects on the performance, such as:

- Hydrocarbons: outgassing, particle stimulated desorption.
- Zinc, cadmium: contamination of the surroundings due to the high vapour pressure.
- Halogens (Cl, F, Br) and S: corrosion.
- Sodium, potassium, calcium: salts retaining water.

More detailed information on the levels of cleanliness for such applications is given in the document EDMS n° 347564 “Codification of surface cleanliness levels”.

Chemical degreasing in aqueous phase

Degreasing in an alkaline solution eliminates greases, oils and fingerprint residues, as well as particles and dust. The degreasing process consists of various mechanisms:

- Mechanical action: this is due to the flushing of the surface and renewal of the liquid film on the surface of the component. This action can be enhanced by liquid circulation or ultrasonic agitation. Ultrasound waves at high frequency propagate in the cleaning fluid and create high local pressure differences with resulting cavitation bubbles. These cavitation bubbles act with a brushing action on the sample surface.
- Saponification: this reaction is limited to greases, animal and vegetal oils. Saponification is due to the reaction of hydroxide ions with glycerides, resulting in the formation of sodium stearate (soap) and glycerine which are both soluble in water.
- Emulsifying action: this consists in the dispersion of oils and greases in microscopic drops in the aqueous bath.
- Deflocculating action: solid particles included in greases are suspended in the aqueous solution and cannot be re-deposited on the surface to be cleaned.
- Wetting action: the surface tension of the solution is reduced by the detergent.

DEGREASING PROCEDURE

1. Reception of the components, unpacking and visual inspection of the surfaces. Communication of non-conformity, if any, to the client (e.g. damaged components).

2. Removal of scotch tape and ink traces with a cloth soaked in ethanol or acetone.

3. A suitable support (hook, basket, rod, etc.) shall be used to hold the components and to avoid contact between components to be cleaned which could result in surface damage.

4. Degreasing by immersion with ultrasonic agitation. Two dedicated detergents are used at CERN: NGL 17.40 spec. ALU III and Henkel P3 Almeco 18.

   4.1. Degreasing with the detergent NGL Cleaning Technology 17.40 spec. ALU III.

   This detergent can be used to clean components made of copper, copper alloys, aluminium, aluminium alloys and stainless steel.

   Set of parameters:

   - Concentration: 10 – 20 g/l.
   - Temperature: 45 - 55 °C.
   - Immersion time: 1 – 3 h (see point 6).
   - Ultrasound agitation time: 10 – 20 minutes.
   - Recommended ultrasound frequency: ≥ 40 KHz for Cu et Al, 25 KHz for stainless steel.

   4.2. Degreasing with detergent Henkel P3 Almeco 18.

   This detergent should only be used to clean components made of stainless steel. It should not be used for copper or its alloys to avoid oxidation. It should also not be used for aluminium or its alloys to avoid chemical attack of the surface.

   Set of parameters:

   - Concentration: 20 g/l.
   - Temperature: 60 - 70 °C.
   - Immersion time: 1 – 3 h (see point 6).
   - Ultrasonic agitation time: 10 – 20 minutes.
   - Recommended ultrasonic frequency: 25 KHz.

5. Rinsing with tap water jet and by immersion.

6. Wettability test with tap or demineralised water.

   This test is performed during rinsing, as a check of the effectiveness of the degreasing on the metallic component. This check is based on the visual inspection of the water spreading on the cleaned surface: after wetting the surface a uniform layer of water should remain on it. A rupture of the water film and the formation of drops on the surface indicates the presence of a residual hydrophobic contamination (oils or greases). In such cases a further cycle of degreasing starting from point 4 shall be performed.

7. Final rinsing with a demineralised water jet and by immersion (preferably with ultrasonic agitation). The water resistivity shall be above 1 MOhm.cm (conductivity ≤ 1 μS/cm).

8. Spraying with ethanol (analytical grade 99.8%, SCEM. 58.04.38.220.0 CERN store) to facilitate the subsequent drying.

9. Drying in nitrogen or filtered compressed air flow with particular attention to the zones which are prone to retain liquids (e.g. blind holes).

10. Bake by hot air flow:
    - Temperature: 60 °C.
    - Time: 10 - 60 minutes.

11. Parts shall be left in a clean place to cool down to room temperature.

12. Packing.

    Components shall only be handled with clean lint-free gloves (nitrile without powder). Aluminium foils (roll of aluminium food-quality, 0.02 mm thick, SCEM. 44.01.35.900.4 CERN store) or white « kraft » paper (40 g/m^2, SCEM. 54.31.10.014.5 CERN store) can be used to pack stainless steel and aluminium components. For copper components the « kraft » paper should be preferred. The parts packed in paper or in aluminium are further protected by a polyethylene bag.

    <u>Note.</u>

    Storage tests made in the laboratory (XPS-report 2986-TE-VSC-SCC-2014-01-28) have shown that clean copper samples packed in « kraft » paper foil and put in a plastic bag during 3 months, have a high carbon contamination (higher than the limit for UHV applications fixed at 44 at.%). Only aluminium foils allow storage of copper parts without carbon contamination.

    Aluminium foils should nevertheless in principle not be used for packing of copper parts in order to avoid galvanic corrosion which can occur when two dissimilar metals come into electrical contact in the presence of humidity. Aluminium foils could also damage or scratch the surface of the parts. However, for specific applications, where carbon contamination has to be avoided, packing in aluminium foil instead of « kraft » paper could be considered.