Specification for Material Selection and the Cleaning of Components for use in the ISIS Synchrotron vacuum systems

1. Scope

This specification covers material selection and special cleaning methods required for components intended for use in the vacuum systems of the ISIS Synchrotron and related areas (eg Injector and EPB's). Vacuum acceptance tests for vessels and assemblies are also included.

In the context of this document "Component" means any of the following:

- a vacuum vessel
- any item forming all or part of the wall of a vacuum system
- any item totally immersed in a vacuum containment device

2. Contents

Specification for Material Selection and the Cleaning of Components for use in the ISIS Synchrotron vacuum systems

3. Introduction

3.1 General overview

3.1.1 In general the term UHV is applied to a vacuum with a pressure less than 10^{-7} mbar. Various sections of the ISIS Synchrotron fall into this category. Hence the system has been designed to meet UHV standards of cleanliness.

3.1.2 The UHV performance of the vacuum systems are highly dependent on the cleanliness of the internal surfaces. Therefore it is essential that all components which form part of, or are placed within this vacuum system conform to UHV standards of cleanliness as laid out in this specification.

3.1.3 The procedure for cleaning large or small components is essentially the same and relies on the effectiveness of soaking in a suitable detergent followed by repeated rinses in copious amounts of demineralised water, followed by air baking. Where possible smaller components may also benefit from cleaning in an Ultrasonic bath whilst immersed in isopropyl alcohol (IPA) or other similar solvent.

3.1.4 IMPORTANT: The methods contained in this specification have been chosen for their simplicity, effectiveness and availability to a broad range of engineering companies. Any deviation from these recommended process, must be discussed in advance with ISIS Vacuum Section Leader.

3.1.5 This specification DOES NOT apply to ISIS Target Station Beam Line Systems.

3.2 Health and Safety considerations

3.2.1 Many of the chemicals and processes described in this document are subject to control and regulation under various parts of Health and Safety Legislation or Regulations. Any persons or Companies implementing all or part of this specification must satisfy themselves that they are conversant with and adequately implementing any such Legislation or Regulations.

4. Material Selection

4.1 Materials permitted in or near UHV system construction

4.1.1 There are limitations on the materials which are allowed inside the vacuum space of a UHV system. The following materials are permissible with correct handling and cleaning:

Stainless Steel

Copper (Phosphorous De-oxidised grade should not be used)

Aluminium and its alloys (Do not use extruded or cast components)

Gold

Silver

Titanium

Molybdenum

Platinum

Beryllium Copper

Ceramic (formed using a vacuum sintering process)

Machinable glass

4.1.2 NOTE: They may also come in contact with UHV components in the form of jigs and fixtures etc.

4.2 Materials NOT permitted in or near UHV system production

4.2.1 The following materials MUST NOT be used in UHV or come in to contact with UHV components in the form of jigs, fixtures, tools, packing etc:

Brass

Soft Solder

Standard Hard Solder

Electrical Solder

All Plastics

All Glues

O-rings

Wet lubricants of any description (including cutting fluid residues from machining)

GE Varnish

Anodised surfaces or any mechanically polished components

Dirt

Finger prints

Any material containing:

Zinc Cadmium Phosphorus Sodium Selenium Potassium Magnesium

4.2.2 These lists are not exhaustive. Exceptions and additional limitations may apply. If in doubt please ask.

5. General Procedures

5.1 Mechanical operations on vacuum surfaces

5.1.1 Abrasive techniques to clean or attempt to improve the appearance of the surfaces of vacuum components must be kept to an absolute minimum and are preferably avoided. The use of grinding wheels, wire brushes, files, harsh abrasives, sand, shot or dry bead blasting, polishing pastes and the like is prohibited under normal circumstances and without the prior permission of STFC.

5.1.2 Permitted techniques are slurry blasting with alumina or glass beads in a water jet; gentle hand use of a dry fine stone or a fine stone lubricated with IPA or ethanol; hand polishing with fine mesh alumina in an IPA or ethanol carrier on a lint free cloth; hand polishing with ScotchBrite™ (Alumina loaded, Grade A).

Specification for Material Selection and the Cleaning of Components for use in the ISIS Synchrotron vacuum systems

5.2 Preparing components for welding

5.2.1 Pay attention to the cleanliness of fabricated parts that are to be welded. Dirt, oil and other contaminates in the vicinity of the weld prep area will lead to inclusions in the weld and out-gassing under vacuum. Therefore components MUST be cleaned to UHV standards, using the appropriate method described in this specification, BEFORE being welded together.

5.3 Treatment of weld burns

Although weld burns are sometimes removed by using acid pastes this is not suitable for UHV cleaning. In general such burns do not affect vacuum performance and are best left alone. Any scaling must be removed using the techniques outlined in section 5.1 above. If it is desired to remove burns, then slurry blasting with alumina in water or hand burnishing with alumina powder is a satisfactory alternative. Heavy abrading, grinding or wire brushing is prohibited. Hand finishing with ScotchBrite™ or a dry stone is also permissible.

5.4 Use of acids

5.4.1 Acid treatment of any sort is normally prohibited and may only be carried out with the specific prior agreement of STFC. Most acid treatments are for cosmetic purposes only and may result in degradation of vacuum performance.

5.4.2 Components must not be “pickled” in acid baths.

5.4.3 If, in exceptional circumstances, acids are permitted then exposure of the component must be kept to a minimum and must be followed by copious amounts of washing in hot (approx 80°C) demineralised water.

5.5 Marking and labelling

5.5.1 Marking out or marking for identification should be carried out with clean dry scribers or vibrating engravers only, never by acid etching or marker pen. Vacuum surfaces should only be marked if it essential to do so and never on sealing surfaces.

5.5.2 Identity labels should be tied to components or, in the case of small components, fixed to the packing bags. Self adhesive labels, tapes etc. should only be used if essential to do so and may only be fixed to non-vacuum surfaces. Ensure the adhesive used is soluble in acetone.

5.6 Handling and packing

5.6.1 Once components have been cleaned care should be taken that the vacuum surfaces are never touched by bare skin. Clean gloves should always be worn when handling components in order to avoid contamination. Polyethylene or natural vinyl are preferred. Coloured gloves should be tested to ensure that the dyes do not leach out when exposed to the solvents used. Powdered gloves of any description should be avoided since the powder can migrate into the components. A good solution is to use lint free gloves inside polyethylene gloves.

5.6.2 **Once components have started the cleaning process they should complete the cycle without a break.** If an unavoidable delay occurs between stages, then care must be exercised that the component is thoroughly dry before storage, and all seal faces and ports are covered as in 5.6.3 below. There must never be a break between any chemical cleaning stage and a subsequent water washing stage.

5.6.3 After the component has been cleaned and is thoroughly dry (air baked where possible) it must be packed carefully to ensure that it remains clean and free from damage. Protect all seal faces and/or knife edges with clean used metal gaskets where possible; cover all ports with strong clean new aluminium foil and protective covers. Small items should be wrapped in clean aluminium foil and sealed in a polyethylene bag.

6. UHV cleaning of small components and assemblies

6.1 General guidance

6.1.1 Where possible small components should be thoroughly degreased in a vapour degreaser or ultrasonic bath prior to detergent washing.

6.1.2 Once machined a trial assembly should be undertaken to ensure all components fit together correctly. Any machining, filing or welding operations should take place before cleaning. Only when UHV cleaning is complete should the final assembly take place. Refer to section 5.6 above for advice on handling and packing.

6.2 Machining of small components

6.2.1 The cleaning procedure for small components will remove normal machining fluids, therefore no special precautions or fluids are required.

6.3 Cleaning of small stainless steel components

Follow ALL of the steps in this sequence:

6.3.1 *Removal of loose surface scale and dirt.*
The component should be machined and any surface scale or dirt removed using ScotchBrite™ or emery paper. The use of a polishing paste is forbidden. If a polished finish is required then electropolishing should be considered. Alternatively use powder based polishing compounds such as Aluminium Oxide or Jewellers Rouge. Clean the component in an Ultrasonic bath after each polishing stage.

6.3.2 *Degrease heavily soiled components.*
If the component is heavily soiled with machining fluids, greases or oils then it should be cleaned in a vapour degreaser or agitated in an ultrasonic bath for at least 10 minutes whilst immersed in a suitable solvent. If there is still evidence of contamination, wipe clean with IPA until all traces are removed. Repeat the vapour wash, or ultrasonic clean, and from this point on, handle the component with clean plastic gloves and only rest on clean aluminium foil or fresh white absorbent paper roll.

6.3.3 *Hot water wash after optional vapour/ultrasonic clean.*
Wash component under a hot water tap to ensure all traces of solvent are removed. Allow to drip dry on clean white paper roll.

6.3.4 *Detergent wash*
Place components in an ultrasonic bath filled with hot alkali detergent, such as Decon 90 (approx 50ºC) and agitate for 60mins. If an ultrasonic bath is not available then components should be soaked for a minimum of 5 hours.

6.3.5 *Final hot water wash*
Repeat rinse with hot demineralised water (approx 80°C) two or three times to ensure all traces of detergent have been completely removed. Allow to drip dry on clean white paper roll.

6.3.6 *Air bake*
After the component has been fully cleaned and rinsed it must be air baked at 200°C for a period of 16 hours and allowed to cool gently to avoid thermal shocks. When removing the component from the oven observe the procedures laid out in section 5.6 'Handling and packing' to avoid any contamination.

6.4 Cleaning of small non-ferrous metal components

6.4.1 The procedure for cleaning small non-ferrous metal components is the same as for stainless steel except for the detergent wash stage. A cleaning agent specially formulated for cleaning non-ferrous metals, such as Decon Neutracon, must be used. Under no circumstances should acid or alkaline cleaning agents be used.

6.5 Cleaning of wires

6.5.1 *General guidance.*
Wires in UHV must be insulated with Ceramic.
Enamel insulation and GE varnish MUST NOT BE USED.
Electrical connections should be spot welded or crimped.

6.5.2 *Cleaning procedure.*
All wires should be cleaned by wiping the wire several times through a piece of white paper roll soaked in alcohol. Wipe the wire evenly on all sides and use a fresh piece of paper until it remains clean after wiping.

6.6 Cleaning of ceramic and machinable glass

6.6.1 *General* guidance
Use only demineralised water as a coolant when machining ceramics and machinable glass as cutting fluids will contaminate the components and render them unsuitable for UHV.

6.6.2 *Cleaning procedure*
Remove any surface contamination by wet slurry blasting with alumina powder, or by hand polishing with fine mesh alumina powder in an acetone, ethanol or isopropyl alcohol carrier. Continue to clean in the same manner as for small stainless steel components (from 6.3.4 onwards), including air bake.

7. UHV cleaning of large components and assemblies

7.1 General guidance

7.1.1 Any component or assembly requiring cleaning to UHV standards that is too large to fit into a vapour degreaser or ultrasonic bath must be cleaned as described below.

7.2 Cleaning of large stainless steel components

7.2.1 *Removal of loose scale and dirt.*
Use ScotchBrite™ or a stainless steel wire brush to clean heavily soiled areas prior to degreasing.

7.2.2 *Degrease using suitable solvent*

Where the component is too large to fit into a vapour degreaser or ultrasonic bath, swab with IPA or ethanol solvent on a lint free cloth. Change the cloth frequently.
NOTE: From now on observe the procedures laid out in section 5.6 'Handling and packing' to avoid any contamination.

7.2.3 *Alkaline solution wash*
Wash with a hot alkaline solution, such as Decon 90 (or Almeco 29, 5% concentration by weight). If the component is a vessel it should be filled with hot alkaline solution (approx 50ºC) and left to soak for a minimum of 5 hours.

7.2.4 *De-mineralised water wash*
Repeat rinse, two or three times, with hot de-mineralised water to remove all traces of detergent. As a final check flood the component with de-mineralised water over the entire surface. Any areas not covered in water indicate remaining grease which must be removed by starting the cleaning process again.

7.2.5 *Air bake*
After the component has been fully cleaned and rinsed it must be air baked at 200°C for a period of 16 hours and allowed to cool gently to avoid thermal shocks. Any components too large to fit into a drying oven should be hung up to dry. Ensure there is adequate signage to make everybody aware that it is a clean component and MUST NOT be touched. When removing the component from the oven observe the procedures laid out in section 5.6 'Handling and packing' to avoid any contamination.
NOTE: Any subsequent swabbing prior to sealing the vacuum should be done using IPA, lint free wipes and lint free gloves.

7.3 Cleaning of large non-ferrous metal components

7.3.1 For cleaning large non-ferrous metal components follow the procedure for large stainless steel components except for the Alkaline solution wash. Substitute this with a cleaning agent specially formulated for cleaning non-ferrous metals, such as Decon Neutracon. Components must not be cleaned with an acid or alkaline solution (see 5.4 Use of acids).

8. UHV cleaning of vacuum bellows

8.1 General guidance

8.1.1 Great care should be taken when cleaning thin walled metal bellows, particularly those of edge welded construction. Solvent residues trapped between the convolutions, either inside or out, can result in corrosion which will cause leaks to develop. Similarly if any particulates are deposited in the convolutions then mechanical puncturing may happen. Alkaline degreasing solutions are prone to particulate precipitation, and for this reason, MUST NOT be used for cleaning bellows assemblies.

8.2 Cleaning procedure

8.2.1 Fix the bellows in an extended position, if possible, and remove any visible traces of contamination with a jet of clean, dry air or nitrogen.

8.2.2 Immerse in an ultrasonically agitated bath containing isopropyl alcohol (IPA) or ethyl alcohol (ethanol) for typically 1 hour.

8.2.3 Vapour wash immediately in IPA or other suitable solvent.

8.2.4 Thoroughly dry the bellows, inside and out, using a jet of clean, dry air or nitrogen.

8.2.5 Rinse in acetone or ethanol and thoroughly dry with air or nitrogen.

8.2.6 Air bake at 200°C for 16 hrs.

8.2.7 When removing the component from the oven observe the procedures laid out in section 5.6 'Handling and packing' to avoid any contamination.

9. UHV cleaning of assemblies and sub-assemblies

9.1 General guidance

9.1.1 Many assemblies and sub-assemblies will contain components for which, as individual items, more than one of the above cleaning procedures would be applicable. Individual components should be cleaned according to the most appropriate procedure for that item. Assembly should then take place under clean conditions.

9.1.2 *Trial assembly*
Once machined a trial assembly should be undertaken to ensure all components fit together correctly. Any machining, filing or welding operations should take place before cleaning. Only when UHV cleaning is complete should the final assembly take place.

9.1.3 *Finger strip assembly*
All finger strip components, including rivets, must be UHV clean before assembly can begin. The assembly procedure must be carried out in clean conditions using clean tools. Refer to section 5.6 for advice on handling and packing.

10. UHV Vacuum acceptance tests for vessels and assemblies

10.1 Preparation for testing

10.1.1 The vacuum vessel or assembly must be cleaned and air baked by applicable STFC methods prior to testing.

10.1.2 A helium vacuum leak test should be carried out on all vessels and vacuum components being supplied. In the case of vacuum vessels the leak detector should be connector directly to the vessel itself or the turbo pump outlet used to pump-down the vessel. Under no circumstances should valves be used to restrict the flow of gas into the leak detector. Oil free (dry) pumps should only be used unless agreed otherwise.

10.1.3 A calibrated leak should be used to test the performance of the leak detector prior to commencing the leak test. A representative from ISIS Vacuum Section may be present to witness the calibration and leak testing process.

10.2 Leak testing

Specification for Material Selection and the Cleaning of Components for use in the ISIS Synchrotron vacuum systems

10.2.1 The acceptable leak rate for the vessel shall be $<1x10^{-8}$mbar/litre/sec, unless another value has been specified in the contract or has been agreed in writing with STFC.

10.2.2 The manufacturer is required to provide proof of vacuum test results. A print out from the vacuum test equipment is acceptable.

10.3 Outgassing rate

10.3.1 Vessel or assembly total outgassing rate shall be measured after 10 hours of pumping from atmosphere. The outgassing rate must not exceed $1x10^{-9}$mbar/litre/sec/cm^2.

10.3.2 The manufacturer is required to provide details of the specific outgassing test carried out.

10.3.3 An RGA scan (1-200amu), witnessed by a representative from ISIS Vacuum Section may also be required to assess cleanliness of a vessel or component.

For further information or queries regarding this specification please contact Sunil Patel, Vacuum Section Leader - ISIS (sunil.patel@stfc.ac.uk. Tel 01235 445519).