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Appendix 14 Passivation and Pickling

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ITER Vacuum Handbook
Appendix 14

Guide to Passivation & Pickling for the ITER Project

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14 Guide for the Pickling/passivation of Steels and Copper for the ITER Project

1.1 Scope of this Appendix

This Appendix specifies typical procedures and processes to be used when materials used for vacuum components for the ITER project need to be passivated.

It is intended that the *suppliers* using such processes should follow the guidance in this Appendix to achieve the requirements of the ITER Vacuum Handbook.

The *supplier* is at liberty to utilise other techniques not described in this Appendix provided that the components supplied comply with the requirements of the ITER Vacuum Handbook.

1.2 General Comments

Pickling is most frequently used to remove heavy scale from steels or a heavy, loose oxide layer from copper (or aluminium).

Pickling is rarely specified for vacuum components, normally only for those to be used in rough vacuum, since the process attacks the metal surface and the oxide layer, tending to leave residues which are difficult to remove.

Heavy scale on steel is best avoided by specifying that the plate produced in a rolling mill or a hot-forged blank is stripped with an air knife while still hot.

Light scale on steel may be removed with a wire brush. Loose oxide on a copper surface can also be brushed off.

Pickling often leaves the surface in an etched state with a matt finish, which may or may not be desirable.

Dimensional stability cannot be guaranteed during the pickling process, so it should normally be carried out on the material before manufacture.

If a vessel assembly is pickled, then final machining of vacuum sealing surfaces must be left until after the pickling/passivation process.

Pickling and passivation must always be followed immediately by an appropriate cleaning process, relevant to the Vacuum Classification of the component. (Refer to Appendix 13)

Pickling should always be followed by passivation. This is best carried out chemically, although native oxide layers can reform on exposure to atmosphere.

It should be noted that thermal outgassing from surfaces which have been pickled/passivated may well be greater than that from a native metal surface and may require additional baking to achieve the outgassing requirements of the ITER Vacuum Handbook.

1.3 Pickling and Passivation of Steels.

Steel manufacturers/suppliers will often have their own preferred method of pickling/passivation and may be unwilling to use any other method. Expert advice from

both a metallurgical and vacuum point of view shall be sought in this case. The vacuum person in this case will be the ITER Vacuum RO.

In no case, however, shall the use of glue in the pickling solution be permitted.

Note that the chemicals used in these processes are hazardous and all appropriate safety procedures must be followed

Table 14-1 below lists some of the acceptable pickling solutions for steels.

Material	**Solution**	**Concentration**	**Temperature (°C)**	**Comment**
Iron and steel	Sulphuric acid (SG 1.84)	10% solution	50-80	Until Scale visually removed
	Hydrochloric acid (SG 1.19)	10-20% solution	50-80	As above
Stainless steel	Nitric acid (SG 1.4) Hydrofluoric acid (52%)	$200gl^{-1}$ $40gl^{-1}$	55-65	As above
	Sulphuric acid (SG 1.84) Hydrofluoric acid (52%) Chromic acid - 60	$60gl^{-1}$ $60gl^{-1}$ $60gl^{-1}$	Room	As above
	Hydrochloric acid (SG 1.19) Nitric acid (SG 1.4)	$250gl^{-1}$ $22gl^{-1}$	60-70	Bright Finish

Table 14-1 – Pickling solutions for steels

Unless the pickling/passivation process is carried out on the raw material as part of the production process at the steel mill, the process to be used will typically be as follows -

- Gross contamination is removed by washing the material in a jet of hot (80°C) water.
- The material is allowed to dry.
- The material is thoroughly degreased using one of the methods specified in Appendix 13 of the ITER Vacuum Handbook
- The pickling baths should be checked visually to ensure that there are no visible signs of contamination, e.g. oils or greases floating on the surface. Ideally, clean pickling solutions in clean baths should be used.
- The material is lowered into the pickling solution for the specified time or until the process is complete.
- The material is washed in a jet of hot (80°C) water.
- The surface of the material is then passivated by lowering into a bath of dilute nitric or citric acid.

- The material is washed in a jet of hot (80°C) water and allowed to dry.

Note that there are alternative methods of pickling and passivation using spray and gel techniques. The use of such techniques is not prohibited but should only be used following *acceptance* of the proposal by the ITER Vacuum RO

1.4 Pickling and Passivation of Copper and Copper Alloys

The generalities and procedures of Section 1.3 above apply except where noted otherwise.

Pickling solutions for copper and copper alloys are given in Table 14-2 below

Material	Solution	Concentration	Temperature (°C)	Comment
Copper and copper alloys	Sulphuric acid (SG 1.84)	20% aqueous solution	65-75	
	Sulphuric acid (SG 1.84). Sodium dichromate	20% aqueous solution 75gl^{-1}	20-75	
	Citric acid	1% aqueous solution	Ambient	Also passivates the surface

Table 14-2 – Pickling solutions for copper

Following pickling, copper parts must be passivated immediately by dipping in a 1% aqueous solution of citric acid.

1.5 Standards

The following standard procedures may be used to inform the processes described in this Appendix

EN 2516:1997 – Passivation of corrosion resistant steels and decontamination of nickel bas alloys

ASTMA380 – Practice for Cleaning, Descaling and Passivation of Stainless Steel Parts, Equipment and Systems

ASTM A967 – Specification for Chemical Passivation Treatments for Stainless Steel Parts