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Adhesives — Preparation of metal surfaces for adhesive bonding

Adhésifs — Préparation des surfaces métalliques pour le collage par adhésif



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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4588 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Annexes A to D of this International Standard are for information only.

Adhesives — Preparation of metal surfaces for adhesive bonding

1 Scope

This International Standard describes the usual procedures for the laboratory preparation of metal surfaces for adhesive bonding and testing. Prebond surface treatments for the following materials are given :

- a) aluminium and aluminium alloys;
- b) stainless steel;
- c) carbon steel;
- d) copper and copper alloys.

Mechanical treatments such as grit blasting are widely applicable, but it is not possible to present one chemical method that is applicable to all metals. Several methods are listed so that the user will have a greater choice in selecting a treatment appropriate to a particular metal.

The surface treatments described comprise various solvent cleaning and degreasing methods, mechanical cleaning (abrasion) and chemical cleaning (etching). When used together, these treatments should normally be used in the following order: solvent cleaning to remove gross contamination, then abrasion and finally etching to achieve the optimum surface for bonding. Alternative sequences may, however, be used.

WARNING — The procedures described do not necessarily detail all the precautions necessary for health and safety in a particular country. Because of the hazardous nature of some of the chemicals used, national and local legislative requirements for safe handling and disposal of these materials shall be observed.

2 Handling and storage

2.1 Handling

All parts shall be handled as little as possible during and after processing and before bonding, and the use of bare hands shall be avoided. Use clean hand coverings that will not contaminate the surface, such as lint-free cotton or nylon fabric gloves.

2.2 Storage conditions

Prepared surfaces shall be stored at $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ and $50\% \pm 5\%$ R.H., except for metals such as carbon steels which cannot sustain these conditions without prejudicial oxidation. If necessary, the air shall be filtered to remove dust.

The time between, and conditions of, surface preparation and application of the adhesive primer and bonding are critical. For sensitive surfaces, the storage time shall not exceed 8 h and parts shall be covered with or enveloped in unbleached kraft paper or other suitable coverings. When possible, prepared surfaces should be stored in a conditioned container without surface contact.

3 Initial preparation

3.1 Cleanliness

Do not carry out contaminating operations in the working area. Paint or other spraying operations, processes using powdered materials, oil vapours from pumps and other machinery, spraying of mould release agents, etc., are especially detrimental.

3.2 Preliminary cleaning

Remove ink markings or printed identifications from the metals by wiping with a cloth wetted with an appropriate solvent such as acetone, methyl ethyl ketone (butan-2-one), hydrocarbon mixture or 1,1,1-trichloroethane.

3.3 Organic solvent degreasing

3.3.1 When vapour degreasing equipment is available, this is the preferred treatment method; the vapour degreasing solution shall be a stabilized chlorinated solvent or similar. The pH value or acid content of the vapour degreasing solution, or both, shall be determined.

3.3.2 If vapour degreasing is not possible, use 1,1,1-trichloroethane or another suitable solvent. Parts shall be at room temperature. Pour the solvent on to a clean lint-free cloth and wipe the parts vigorously, changing the position of the cloth repeatedly, and dry the surface with a clean lint-free cloth. Repeat the operation if necessary. Use a new cloth for each test specimen.

3.3.3 As a last resort, degreasing may be carried out by immersing the parts in a suitable solvent and wiping as in 3.3.2. Renew the immersion solvent frequently, as it easily becomes contaminated.

The procedures of 3.3.2 and 3.3.3 are applicable to small parts or test specimens.

3.4 Alkaline degreasing

This method is intended primarily for final degreasing prior to etching. Proprietary materials are generally used, but care shall be taken to ensure that these are free from sodium hydroxide or any other strong alkali; silicates or carbonates may be included, as these have the advantage of rendering the surface inactive to corrosion. A 25 % (m/m) solution of sodium metasilicate may also be used.

3.5 Phosphate degreasing

This treatment may be employed when appropriate.

3.6 Ultrasonic degreasing

This treatment may be employed when appropriate.

4 Abrasion

Abrasion may be used as the principal surface treatment, or prior to etching, to remove oxide and to roughen the surface. Abrasion may not be acceptable for parts that require environmental stability during the operation.

4.1 Preliminary treatment

Degrease the surface using one of the procedures described in 3.2 and 3.3 to remove gross oily contamination.

4.2 Method 1 — Dry blasting

4.2.1 Dry blast with an appropriate abrasive such as clean, dry, sharp-edged alumina, silicon carbide, sand or similar abrasive of particle size 106 µm to 45 µm. Do not use steel shot or steel-contaminated abrasive to blast-clean aluminium, copper or stainless steel. The blasted surface shall be visually uniform. Measure the surface roughness; the method of measurement and the results shall be mentioned in the test report.

4.2.2 Blow-clean parts with clean, dry, filtered air or nitrogen to remove particulate matter.

4.3 Method 2 — Wet blasting

Vapour blast with water or steam containing suspended grit particles of mesh size 1 000 or smaller. The angle of spray shall be 90°.

NOTE — This can be very effective and should not warp the parts if done carefully. Vapour blasting is recommended for small items only.

Wet blasting equipment usually contains flow modifiers and corrosion-resistance additives. Rinse the wet-blasted parts thoroughly in water and clean solvent, or vapour degrease, prior to drying and bonding.

4.4 Method 3 — Hand abrasion

4.4.1 Abrade the surface to be bonded with abrasive paper or cloth, using alumina, silicon carbide, emery or other suitable material of particle size 106 µm to 45 µm. This abrasion may be carried out wet or dry.

Use the following sequence of abrasive operations :

- a) straight across the part in a direction parallel to any one side;
- b) at right angles to the first direction until all signs of the first abrasion operation have been removed;
- c) circular motion of diameter approximately 80 mm to 100 mm, until a pattern has been produced consisting only of circular abrasion marks superimposed one upon the other and the surface appears visually uniform.

4.4.2 Blow-clean parts with clean, dry, filtered air or nitrogen, or brush with a clean, dry brush to remove particulate matter.

If wet abrasion is used, wipe the surface with a cloth dampened with an appropriate solvent, or brush with a clean, dry cloth.

5 Etching

5.1 General

Etching may be preceded by abrasion (see clause 4) and/or by a chemical deoxidizing treatment.

5.2 Apparatus, materials and procedures

5.2.1 Apparatus

All containers and other equipment shall be resistant to the etching solutions used.

5.2.2 Water

Water for preparing etching solutions, such as distilled water or demineralized water, shall not contain more than 50 mg/kg of solids and shall have a pH value between 5,5 and 7,5 and a conductance of less than 20 µS.

5.2.3 Monitoring of etching solutions

The useful life of the solutions depends upon the number and dimensions of parts being prepared. Solutions shall be sampled periodically and analysed for materials pertinent to the particular treatment, such as hexavalent chromium (CrO₃), iron, chlorides, aluminium. Compliance with the purity specifications shall be established for each batch and each container of solution.

Records shall indicate the date of preparation of each solution, any change in concentration with time, and the amounts of contaminants present.

Solutions may be made up from technical or reagent grade chemicals and, if not otherwise specified, with water as specified in 5.2.2. Except for water, all constituents shall be weighed to within $\pm 1\%$.

5.2.4 Rinsing

Rinsing may be done by spraying or by dipping in a tank of water (5.2.2) which is being circulated and constantly renewed by an overflow method.

Rinsing shall be sufficiently long and vigorous to ensure the removal of all traces of chemicals, smut or other particulate matter. Mechanical wiping with a clean cloth or brush should be avoided if possible. Any temperature limit specified for the rinsing water shall be respected.

After rinsing, the water-break test is commonly used to determine whether the surface of the metal is clean. The surface under test is completely immersed in water and then withdrawn; if the water film is continuous and does not break up into droplets within 30 s, then the surface may be assumed to be free of contamination.

If the water film is discontinuous, this indicates that the cleaning treatment was unsatisfactory, and it shall therefore be repeated. The number of times that cleaning may be repeated depends on the amount of metal removed in relation to the tolerance requirements for the part and whether clad or bare metal is involved. Generally, not more than two repeat treatments are permitted.

NOTE — Even if the cleaned metal surface is wetted by water, this gives no information as to the likely strength of an adhesive bond made with the metal surface. At best, it is a necessary, but not a sufficient, requirement for the achievement of high bond strength.

6 Procedures for use with various types of metal

6.1 Stainless steel

6.1.1 Organic solvent degreasing

See clause 3.3.

6.1.2 Abrasion

See clause 4.

6.1.3 Etching

6.1.3.1 Equipment and materials

For general procedures, see 5.1 and 5.2.

6.1.3.2 Suggested etching compositions

Alternative chemical treatments are listed in annex A. These are given to show the wide range of treatments now being used and are intended as a guide.

6.1.4 Desmutting

The etching treatments listed in annex A may leave smut on the surface of the steel. Smut may be removed by wiping with a clean cloth or by brushing or preferably by dipping in a solution containing 300 g of sulfuric acid and 30 g of sodium dichromate per litre at a temperature of 60 °C for about 5 min, followed by rinsing and drying.

6.1.5 Storage

Stainless steel shall be bonded after 24 h drying. Storage in a desiccator helps to preserve the surface.

6.2 Copper and copper alloys

6.2.1 Organic solvent degreasing

See clause 3.3.

6.2.2 Abrasion

See clause 4.

6.2.3 Etching

6.2.3.1 Equipment and materials

For general procedures, see 5.1 and 5.2.

6.2.3.2 Suggested etching compositions

Alternative chemical treatments are listed in annex B. These are given to show the wide range of treatments now being used and are intended as a guide.

6.2.4 Storage

Copper and copper alloys oxidize easily and shall be bonded within 2 h of cleaning. They shall be stored dry. Storage in a desiccator helps to preserve the surface.

6.3 Carbon steel

6.3.1 Organic solvent degreasing

See clause 3.3.

6.3.2 Abrasion

See clause 4.

6.3.3 Etching**6.3.3.1 Equipment and materials**

For general procedures, see 5.1 and 5.2.

6.3.3.2 Initial preparation

Clean the surface in accordance with 3.3.

6.3.3.3 Suggested etching compositions

A chemical system is given in annex C. Alternatively, the method for aluminium in annex D may be used.

6.3.4 Storage

Carbon steels oxidize easily. They shall be stored dry and bonded within 1 h after cleaning. Storage in a desiccator helps preserve the surface.

6.4 Aluminium and aluminium alloys**6.4.1 Organic solvent degreasing**

See clause 3.3.

6.4.2 Abrasion

See clause 4.

6.4.3 Etching**6.4.3.1 Equipment and materials**

For general procedures, see 5.1 and 5.2.

6.4.3.2 Suggested etching compositions

See annex D.

Solutions shall be discarded when pitting of the parts occurs or when solid material begins to separate out on cooling the solution to room temperature. The latter can be expected when the density of the solution exceeds 1,36 g/ml.

Annex A (informative)

Alternative chemical treatments for stainless steel

Materials	Parts by mass						
	A	B	C	D	E	F	G
H ₂ SO ₄ (technical) (ρ 1,84 g/ml)	10			200		100	
HCl (ρ 1,2 g/ml)		30	45		200		50
Oxalic acid [(COOH) ₂ ·2H ₂ O]	10						
Na ₂ Cr ₂ O ₇				3,5		30 (saturated solution)	
H ₂ O	80	70	33	3,5			45
HF [35,35 % (m/m)] (ρ 1,15 g/ml)					10		
H ₂ O ₂ [30 % (m/m) to 35 % (m/m)]			16				2
H ₃ PO ₄ (ρ 1,8 g/ml)					30		
Formaldehyde [40 % (m/m) aqueous solution]			9				10
Etching temperature, °C	60 ± 3	room temperature (min. 20 °C)	room temperature (min. 20 °C)	74 ± 3	boiling water bath	63 ± 3	60 to 65
Time, min	30	15	10 to 30	10 to 20	2	15	10
Rinse liquid	H ₂ O	H ₂ O	H ₂ O	H ₂ O	H ₂ O	H ₂ O	H ₂ O
Rinse temperature	steam	room temperature (min. 20 °C)	room temperature (min. 20 °C)	room temperature (min. 20 °C)	room temperature (min. 20 °C)	room temperature (min. 20 °C)	room temperature (min. 20 °C)
Maximum drying temperature, °C	93	93	93	95	93	93	