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Structural adhesives — Guidelines for the surface preparation of metals and plastics prior to adhesive bonding

Adhésifs structuraux — Lignes directrices pour la préparation de surface de métaux et de plastiques avant le collage par adhésif

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17212 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

This second edition cancels and replaces the first edition (ISO 17212:2004), which has been technically revised.

Introduction

Some materials will bond far better than others and some will not bond at all without special treatment. The suitability of a surface for bonding depends upon the degree of surface preparation, the joint's design, the function it has to perform (joining, sealing, etc.) and the environment it has to perform in.

Prior to bonding, some degree of surface preparation is required for most adhesives — but not all. Material surfaces that are particularly prone to weak or loose surface layers, stress cracking or solvent attack usually require special treatment.

Following appropriate preparation, most common metals and their alloys can be bonded satisfactorily. Were it not for contamination and residual mould release agents, thermoset plastics (e.g. polyepoxy and polyester composites) would bond well without any preparation. By contrast, most thermoplastics require careful preparation because of their low surface energy.

Some paints — especially the cataphoretic, water-based primers used by the vehicle industries — can provide an excellent surface for bonding. However, the stability of the interface below the paint should be checked. The surface of the paint itself, even if fresh, can require treatment in order to raise its free energy and thus facilitate wetting.

Certain adhesives possess the ability to dissolve light oils and some polymeric materials. Consequently, for joints that are not "safety-critical" some surfaces do not require any preparation prior to bonding.

In order to achieve the optimum environmental durability from a bonded joint, the traditional preparative approach usually, though not necessarily, consisted of three sequential steps:

- the removal of contaminants;
- physically induced modification of the surface to be bonded;
- chemical treatment.

However, legislative pressure is driving development and the introduction of new methods. Consequently, the separate steps of the foregoing sequence are being combined and the more hazardous chemicals are being progressively eliminated.

The majority of both thermoset and thermoplastics materials can be prepared by commonly applicable techniques — though there will often be detailed differences. By contrast, metal and metal-alloy surfaces to be bonded generally require individual treatment. The optimization of the durability of a metal-based joint usually requires the introduction of progressively more complex and specific treatments.

Such process options are described for a number of metals and their alloys, and some plastics (see Clause 7).

Structural adhesives — Guidelines for the surface preparation of metals and plastics prior to adhesive bonding

1 Scope

1.1 General

This International Standard provides and describes the usual procedures for the preparation of component surfaces prior to bonding for either laboratory evaluation or the process of construction. This International Standard is applicable to metal and plastic surfaces that are commonly encountered.

1.2 Surfaces

These comprise the following metal, metal-alloy and plastic families — the last-mentioned including filled versions and suitable paints:

Metals and metal alloys

- aluminium
- chromium
- copper
- magnesium
- nickel
- steel (mild)
- steel (stainless)
- tin
- titanium
- zinc

Paints

- cataphoretic (water-based)
- poly-alkyd
  - ester
  - epoxide
  - urethane

Plastics

Thermoplastic

- acrylonitrile-butadiene-styrene copolymer
- poly-acetal
    - acrylate
    - amide
    - butylene terephthalate
    - carbonate
    - ester
    - ether ether ketone
    - ethylene
    - imide
    - methyl methacrylate
    - phenylene oxide
    - propylene
    - styrene
    - sulfone
    - tetrafluoroethylene
    - vinyl chloride

Thermoset

- cellulose-based esters
- poly-alkyd
    - allyl phthalate
    - amino
    - epoxide
    - ester
    - phenolic
    - urethane
    - urea-based (see poly-amino above)

1.3 Methods

The various techniques described for cleaning and modifying surfaces are drawn from the best of current practice. The methods can be used in a variety of combinations to create the most effective preparative process conducive with the environmental durability required of the bonded joint.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

EN 923, *Adhesives — Terms and definitions*

EN 2243-5, *Aerospace series — Non-metallic materials — Structural adhesives — Test methods — Part 5: Ageing tests*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 472 and EN 923, together with the following, apply.

3.1
plastic
material which contains, as an essential ingredient, a high polymer and which, at some stage in its processing into finished products, can be shaped by flow

NOTE 1 For the specific requirements of this International Standard, “plastic” also includes paint. In this latter context, it needs be realized that only a few paint surfaces are capable of supporting anything other than purely nominal loads. The exceptions are typically those based upon aqueous, electrochemical paints — such as those used in the automotive industries — and the acrylic-, epoxy- and polyester-based paints used during the preparation of “pre-coated” metal sheet.

NOTE 2 In some countries, the use of the term “plastics” as the singular form as well as the plural form is permitted.

3.2
scarification
shallow roughening of both metallic and plastic (including paint) surfaces, using either abrasion or blasting, almost invariably improving the performance of the final bonded joint

4 Safety

Users of this International Standard shall be familiar with normal laboratory practice and the principles of good industrial hygiene.

Users shall be aware that this International Standard does not purport to address all safety problems, and it is their responsibility to establish practices which are compliant with relevant national health, safety and environmental legislation.

Concentrated acids, alkalis and oxidizing agents (e.g. chromium trioxide, dichromates and chrome-based solutions) are all highly corrosive chemicals. Splashes can cause severe damage to both skin and eyes and will damage normal clothing. Protective clothing (e.g. overalls, gloves and goggles or visors) shall always be worn when using these chemicals.

Similarly, appropriate precautions shall be taken when using solvents. At a minimum, eye protection and gloves (or appropriate barrier cream) shall be worn.

Wherever possible, use propan-2-ol as a solvent. Otherwise, a ketone (acetone or methyl ethyl ketone) or, though deprecated, a halogenated solvent, that meets the requirements of the Montreal Protocol and national legislative requirements, can be used. Alcohols and ketones are flammable — particularly ketones. All such materials are narcotic when concentrated. Ventilate properly, take account of vapour density and draw fumes away from the operator.

Do not allow any cleaning materials to contact the skin. Abuse can lead to dermatitis.

Some of the methods given below employ dangerous techniques, materials and proprietary chemicals. It is essential, therefore, that the supplier's instructions be followed, health and safety data studied, and appropriate safety procedures established.

Waste and spent materials shall be disposed of in accordance with national legislative requirements through the services of an authorized disposal organization — whose advice shall always be sought.

WARNING — When making up solutions:

— NEVER POUR WATER INTO ACID.

— ALWAYS ADD ACID IN A SLOW, STEADY STREAM TO A STIRRED SOLUTION.

An exothermic reaction can heat the resulting mixture. If this occurs, contamination hazards will be intensified. TAKE GREAT CARE.

5 Initial preparative techniques

5.1 General

When safety-critical structures are being bonded, optimum treatment is always necessary. This requires the use of appropriate techniques to both clean and modify the surface, which itself can be either an inorganic or an organic coating — or even a combination of both. By contrast, when joints are only to be lightly or nominally loaded, the use of adhesives capable of dissolving light oils can allow minimal, or even no, surface preparation. The manufacturer's advice shall always be sought.

5.2 Handling, cleaning and storing

5.2.1 Handling

Component areas which are to be bonded shall be handled as little as possible prior to preparation. After preparation, such areas should not be handled directly at all. However, if this is unavoidable then clean, lint-free cotton or nylon gloves shall be worn.

5.2.2 Cleaning

Remove oil- and grease-based residues using aqueous materials if possible. Non-ionic detergents give good results. Proprietary alkaline cleaners are particularly good for metals because not only can they remove hydrocarbons but the more aggressive, stronger, versions can also remove metallic soaps and salts. However, the latter shall not be used on aluminium, and care shall be taken to ensure that this metal is not exposed to cleaners based on sodium hydroxide or other alkaline materials. Some proprietary mixtures are used hot while others utilize either anodic or a cathodic currents. Whichever cleaning agent is used, components shall always be rinsed thoroughly and dried in a stream of warm, clean, dry, oil-free air for about 10 min at 60 °C.

If solvents need to be used to remove identification marks, or paint, then propan-2-ol shall be used wherever possible. Alternatively, use acetone, methyl ethyl ketone or another permitted solvent (see Clause 4). Solvents can severely damage some thermoplastic materials by either dissolving them or initiating stress cracking. Polycarbonate, poly(methyl methacrylate) and acrylonitrile-butadiene-styrene-based plastics are particularly susceptible in this latter regard.

Ultrasonic cleaning can prove acceptable for the preparation of smaller components.

The use of a vapour bath is generally deprecated, but is recommended for the preparation of titanium and its alloys. However, chlorinated solvents shall not be used in this instance.

It shall not be forgotten that some industrial processes can, and do, have a damaging effect on surfaces, both during and after their preparation. The use of equipment often releases deleterious dust, fumes and vapours into the air. Oil vapour, mould release agent sprays and the atmosphere of a plating shop are particularly detrimental. Consequently, surface preparation (and bonding) shall be carried out in separate areas where such contamination can be avoided.

5.2.3 Storage

A distinction needs to be made between laboratory storage and the delays incurred during industrial production. The former implies performance qualification of either surface or adhesive. Where qualification is required, storage shall be maintained in an ambient atmosphere of (23 ± 2) °C with a relative humidity of (50 ± 5) %. The components shall be used within 8 h, except for those materials, such as mild steel, which are still liable to prejudicial oxidation. Such surfaces shall be bonded as soon as possible after preparation and, prior to bonding, shall always be maintained in a dry atmosphere. Wherever practical, parts shall not be touched and shall be kept in a closed container or under a suitable non-contaminating cover, such as unbleached Kraft paper.

Industrial production requires that minimum performance standards be maintained. To this end, procedures shall be established such that the integrity of a prepared surface is not unacceptably prejudiced prior to assembly. Particular regard shall be paid to the possibility of damage occurring through oxidation, condensation and contamination — particularly by release agents, which shall never be used in the same building. Ideally, parts shall be bonded immediately after preparation and only exceptionally after 4 h.

6 Surface modification

6.1 Physical: Mechanical (scarification)

6.1.1 Abrasion

Abrasion can be carried out either wet or dry, using either a water-resisting, coated paper (45 µm to 106 µm grit) or a non-woven abrasive fabric.

NOTE 1 Scarification can be inappropriate for use on thin (≤ 2 mm) light alloys that are likely to be highly stressed in use, because of the possibility of inducing surface stress (eigenstress).

The following sequence shall be employed:

a) Abrade straight across in a convenient direction until all the surface has been lightly and uniformly scarified.

b) Then abrade, similarly, at right angles until all traces produced in operation a) have been obliterated.

c) Then abrade by means of a circular (≤ 100 mm diameter) motion until, again, all traces of the foregoing operation b) have been obliterated and the surface appears uniform.

d) Remove debris. If dry-abraded, and if practical, use a vacuum. Otherwise, blow-clean in a suitably ventilated enclosure with clean, dry, oil-free air. If wet-abraded, solvent-wipe using a clean, lint-free cloth and allow to dry.

e) Then either bond or carry out a further surface modification process.

If parts are to be bonded, then they shall be dry and shall be bonded as soon as practical, preferably within one minute (see 5.2.3). Drying can be speeded by the use of a warm, clean, dry, oil-free air stream at a temperature not exceeding 60 °C.

Care should be exercised to ensure that abrasives do not become clogged and that contaminants are not being transferred from step to step through the above sequence.

NOTE 2 See 6.3.2 e) — the “water-break test” — a procedure that demonstrates that a component’s surface is free of contamination.

6.1.2 Blasting

Dry blasting is usually reserved for metallic components. However, when used carefully — to avoid excessive erosion — the less aggressive processes can be effective when used on the more robust plastics. Proprietary processes are available. These encompass specialized grits such as particulate carbon dioxide and shredded nut husks. However, in the main, metallic components are usually prepared by dry blasting with 45 µm to 106 µm abrasive grit until the surface is uniform in appearance. Neither iron- nor steel-based grits shall be used on aluminium, copper, stainless-steel or titanium parts.

Wet blasting at an angle less than normal to the surface, using ≤ 20 µm grit suspended in either water or steam, can be particularly effective on small metallic parts. Note that proprietary systems usually contain water-soluble additives. For this reason, the manufacturer's advice shall be sought in order to prevent further contamination of the surface.

Wet blasting is not yet recommended for titanium.

Whichever technique is used, steps 6.1.1 d) and 6.1.1 e) shall be implemented.

6.2 Physical: Non-mechanical

A number of processes have been developed whose purpose is to modify a surface without using either mechanical abrasion or liquid-based chemical techniques. Mainly, these are dedicated to bringing about a beneficial chemical modification of the surfaces of the plastics by physically induced, oxidative processes. Some of these processes can also remove modest levels of contamination.

The two major examples of these specialized techniques are the plasma discharge and flame treatments. As optimum conditions need to be developed for both, it is suggested that appropriate techniques be devised in conjunction with an equipment supplier and a recognized investigative laboratory.

The following comments could be helpful:

a) Surface modification induced by an oxidative gas flame is a relatively simple, fast, effective and economic means of improving the surfaces of a wide variety of plastics. The process has the very useful advantage of being able to cope with rapid changes in component topography.

b) Similarly, plasma discharge at ambient pressure — often called corona discharge — is fast, effective and economic. However, the technique has a restricted ability to cope with a varying component topography. Consequently, equipment can be troublesome to adjust and it can be difficult to maintain performance unless components are simple in shape and essentially flat.

c) Low-pressure plasma discharge processes can be considered to be more versatile in their nature than the use of flame oxidation. Complex shapes generally present no problems, and surface modification can be optimized by the use of different gas combinations in the discharge chamber. However, the attractiveness of the technique is diminished by the high capital cost of equipment and the fact that, unlike gas flame and corona-based methods — which can be run continuously — plasma chambers require a batch-based process.

d) Lasers have been used to prepare both plastic and metal surfaces. However, as the technique is not yet considered to be sufficiently well developed, it should only be considered when there are no alternatives.

e) None of the foregoing methods involves liquids. Therefore, the need to dry treated surfaces is avoided. However, depending on the process used, the nature of the surface itself and the ambient environment, the manner of surface deterioration will vary. Some combinations of the foregoing variants can be very tolerant but, in principle, all surfaces shall be bonded as soon as practical after treatment (see 5.2.3).

6.3 Chemical

6.3.1 Background

The usual object of chemical treatment is to oxidize a surface that has been cleaned in accordance with 5.2.2 and scarified in accordance with 6.1.1 or 6.1.2. However, as oxidation usually requires the use and disposal of powerful oxidizing agents, alternative approaches using coupling agents have been, and are still being, developed. To date, they have tended to be based upon silane chemistry. These processes, which are largely proprietary, are discussed separately in 6.4.

Table 1 — Basis of preparative methods for metals and their alloys

Metals and alloys	Procedure	Notes
Aluminium and alloys	De-grease (5.2.2) and scarify (6.1), then bond after etching as required in 7.2.1.1.	Alternatively, coupling agents can be used (see 6.4).
Anodized:		For all anodized surfaces, preferably bond within 4 h.
Normal (prepared by either the chromic or the sulfuric acid processes)	De-grease (5.2.2) and abrade lightly (6.1.1), then bond.	
Hard-anodized	Blast (6.1.2) and bond after etching as required by 7.2.1.2.	This surface shall not be bonded without the further treatment described here. Hence, the requirement to strip the surface. Coupling agents can also be used (see 6.4).
Phosphoric acid anodized	Proprietary.	Follow proprietary process before bonding.
Chromium	De-grease (5.2.2), then bond following either scarification (6.1) or etching as required in 7.2.1.3.	
Copper (including brass and bronze)	De-grease (5.2.2), then bond following either scarification (6.1) or etching as required in 7.2.1.4.	Any one of the three etch solutions described may be used.
Magnesium	De-grease (5.2.2), then bond after proceeding as required in 7.2.1.5.	UNDER NO CIRCUMSTANCES SHALL THIS METAL BE SCARIFIED, ABRADED OR BLASTED. DO NOT EXPOSE TO THE ATMOSPHERE OF A VAPOUR BATH.
Nickel	De-grease (5.2.2), then bond following either scarification (6.1) or etching as required in 7.2.1.6.	Any one of the three etch solutions described may be used.
Steel (mild)	De-grease (5.2.2), then bond following either scarification (6.1) or etching as required in 7.2.1.7.	Coupling agents can also be used and are the preferred method (see 6.4).
Steel (stainless)	De-grease (5.2.2), then bond following either scarification (6.1) or etching as required in 7.2.1.8.	Coupling agents may also be used and are the preferred method (see 6.4).
Tin	De-grease (5.2.2), then bond following either abrasion (6.1.1) or dry blasting (6.1.2).	See also 7.2.1.9.
Titanium	Vapour de-grease (5.2.2), then bond following the special techniques set out in method 1, 2 or 3 of 7.2.1.10.	This metal is usually used for safety-critical aerospace purposes. The provisions set out in 7.2.1.10 shall be considered.
Zinc	De-grease (5.2.2), then bond following either scarification (6.1) or etching as required in 7.2.1.11.	Coupling agents can also be used and are the preferred method (see 6.4).

Table 2 — Basis of method for plastics, including paints

Plastics, including paints	Procedure	Notes
Paints: Cataphoretic Poly-alkyd ester epoxide urethane	All are usually prepared by cleaning and scarifying (see 5.2.2 and 6.1).	Some polyester paints have proved difficult to bond and some formulations could well benefit from flame- or plasma-based techniques (see 6.2).
Thermoplastic plastics: Acrylonitrile-butadiene-styrene copolymer Poly-acetal acrylate amide butylene terephthalate carbonate ester ether ether ketone ethylene imide methyl methacrylate phenylene oxide propylene styrene sulfone tetrafluoroethylene vinyl chloride	Those plastics in this group that do not dissolve readily in common solvents can prove difficult to bond — even with acrylic-based adhesives. Usually, performance will be substantially improved following treatment with flame- or plasma-based techniques (see 6.2).	The chemical treatments cited below have also been developed for the following thermoplastic materials, and their use could prove beneficial if difficulties are encountered with other methods: Acrylonitrile-butadiene-styrene copolymer see 7.2.2.1 Poly-acetal see 7.2.2.2 amide see 7.2.2.3 butylene terephthlalate see 7.2.2.4 ethylene see 7.2.2.5 propylene see 7.2.2.6 tetrafluoroethylene see 7.2.2.7
Thermoset plastics: Cellulose-based esters (see Note 1) Poly-alkyd allyl phthalate amino epoxide (see Note 2) ester (see Notes 2 and 3) phenolic urethane urea-based (see poly-amino above)	All are usually prepared by cleaning and scarifying (see 7.2.2 and 6.1).	The durability of joints based on these materials is likely to be improved by one or more of the combined techniques described in 6.4. Note also: 1) If bonding with epoxy-based adhesive, heat for 1 h at 93 °C prior to bonding and bond while still warm. Avoid premature curing of hot adhesive. 2) If bonding hot, ensure that surface water has been expelled before bonding. 3) Use lower-modulus adhesives to improve load distribution on brittle gel-coat surfaces.

6.3.2 Chemical reagents and solutions

The effectiveness of chemical-based surface modification processes depends upon the maintenance of the integrity of the individual process. Professional advice could prove advantageous, especially in relation to the maintenance of etch solutions. The points expressed in the following requirements shall be observed.

Requirements:

a) The water used shall be either distilled or de-ionized and shall not contain more than 50 mg/kg of solids. Its pH-value shall lie between 6,5 and 8,5 and its conductance shall be less than 20 µS.

b) Solutions shall be made up accurately (technical or reagent grade materials, to an accuracy of ±1 %) and maintained by means of periodic sampling, analysis and relevant documentation. Whenever solutions need to be made up, or utilized, then only equipment made from polyethylene, polypropylene or tetrafluoroethylene shall be used.

c) Waste and spent materials, chemicals and other solutions shall be disposed of in accordance with legal requirements and through the services of an authorized disposal organization, whose advice shall always be sought.

d) Rinsing shall be conducted by either spraying or dipping in a constantly refreshed water tank — see requirement a). In both cases, the action shall be sufficiently vigorous to ensure thorough removal of all residues.

e) The “water-break” test will indicate whether the surface is clean and is conducted by immersing the surface in water — see requirement a). Following withdrawal, the ensuing water film shall remain continuous and unbroken for 30 s. If this condition is met, the surface can be assumed to be clean and in an optimum condition for bonding. However, although long-term durability will be enhanced, improvements in bond strength should not be anticipated.

6.4 Combined procedures

6.4.1 Introduction

A number of preparative techniques have been developed in which scarification is combined with a chemical treatment that is usually, though not necessarily, associated with the use of a silane-based coupling agent.

6.4.2 Dry techniques

6.4.2.1 Background

Dry techniques are applicable to both metal and plastic surfaces. The two methods available are based on proprietary processes. The manufacturer or supplier shall be consulted with regard to the selection of a specific process for a chosen surface.

6.4.2.2 Silane-coated abrasive

The surface is blasted with a silane-coated abrasive — usually corundum — following, if required, cleaning and scarification (see 5.2.2 and 6.1). Dependent upon the proprietary method actually used, it could be necessary to follow this treatment with a process-specific primer prior to bonding.

6.4.2.3 Flame treatment

Following the manufacturer’s instructions, the appropriately prepared surface (see 5.2.2 and 6.1) is chemically modified by exposing it to a flame comprising the combustion products of a proprietary material.

6.4.3 Wet techniques

6.4.3.1 Background

The two wet techniques described are similar in principle, but can differ widely in the materials used — though often these will still be based on silane, or related, coupling agents. Both techniques are suitable for metals and plastics and both rely on proprietary materials. Consequently, the manufacturer or supplier shall be consulted with regard to the suitability of a specific process for a chosen surface.

6.4.3.2 Modification during abrasion

Here, following initial cleaning (see 5.2.2), the surface is abraded wet, in accordance with the requirements of 6.1.1 up to and including 6.1.1 c). Thereafter, the proprietary process is introduced and the manufacturer's instructions followed to the commencement of the bonding process itself.

Some of the essentially aqueous solutions available will also indicate whether a surface is actually in a condition suitable for bonding. This is achieved by matching the surface tension of the liquid to that of the adhesive to be used.

6.4.3.3 Modification after abrasion

Here, following cleaning and scarification in accordance with 5.2.2 and 6.1, the proprietary process is introduced, as required by the manufacturer, prior to bonding.

7 Preparative procedures

7.1 General

Table 1 (metals) and Table 2 (plastics) summarize the preparative procedures available for the more commonly used materials. It will be seen that, for the optimization of joint durability, all metals require a specific, individual treatment. Plastics, by comparison, do not, and similar, if not identical, methods can be used to prepare several different polymer types. In addition, most plastics can be prepared without recourse to a hazardous wet-chemical process.

Most thermoset plastic surfaces are usually, and adequately, treated by degreasing and scarifying using appropriate and convenient processes chosen from 5.2.2, 6.1 and, particularly, 6.2.

The majority of the low surface energy, thermoplastic materials can usually be prepared by employing either of the two methods — plasma or flame — described in 6.2. Unless their surfaces are contaminated with mould release agent, or have become soiled, freshly made parts will not require cleaning, as both treatments can cope with minor levels of contamination. If cleaning is necessary, then care needs to be exercised to ensure that the plastic materials are not damaged. If at all possible, solvents should be avoided, as these can have a particularly deleterious effect, e.g. stress cracking and partial dissolution (see 5.2.2). Scarifying will not normally be required.

However, some specific chemical treatments have been developed for individual thermoplastics, and these can prove helpful in cases of difficulty.

The following subclauses shall be read in conjunction with the requirements given in 6.3.2.

7.2 Specific

7.2.1 Metals

7.2.1.1 Aluminium and alloys

7.2.1.1.1 General

The four methods presented below increase progressively in complexity and, with that, the probability that they will enhance the durability of the ensuing joint. Method 1 is the minimum preparation that shall be considered and is best considered only for indoor applications. Method 2 is effective and can be considered for noncritical, temperate-zone applications. Method 3 has been found to be generally effective. Method 4 is currently used in the construction of aircraft and associated structures.

7.2.1.1.2 Methods

a) Method 1:
   - Remove oil or grease contamination (see 5.2.2).
   - Scarify by abrading or blasting with alumina grit (see 6.1.1 and 6.1.2).
   - Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

b) Method 2:
   - Remove oil or grease contamination (see 5.2.2).
   - Following scarification with an alumina-based grit (see 6.1), use a proprietary etch in accordance with the manufacturer’s instructions.
   - Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

c) Method 3:
   - Remove oil or grease contamination (see 5.2.2).
   - Either in conjunction with, or following, scarification with an alumina-based grit (see 6.1), use a proprietary coupling agent in accordance with 6.4.2 or, more usually, 6.4.3.
   - Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

d) Method 4:
   - Remove oil or grease contamination (see 5.2.2).
   - Scarify if appropriate, by abrading or blasting with alumina grit (see 6.1.1 and 6.1.2).
   - Pre-heat the item to (80 ± 2) °C.
   - Immerse the item for (60 ± 10) s at (80 ± 2) °C in the etch solution (7.2.1.1.3).
   - Remove the item and rinse thoroughly in cold distilled or de-ionized water.
   - Conduct the “water-break” test [see 6.3.2 e)].
   - Drain for 15 min.
   - Dry in a warm, clean, dry, oil-free air stream for about 10 min at a temperature not exceeding 60 °C.

- Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

Some special variations of this etching procedure will produce surfaces that can be successfully bonded after both oiling and forming. However, it should be generally assumed that conventionally treated surfaces are readily damaged and should not be touched, even when gloves are worn, if very high performance, safety-critical structures are being created.

7.2.1.1.3 Etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition of the etch solution is as follows:

Table 3 — Composition of the etch solution for aluminium and its alloys (non-anodized)

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Phosphate-based, aluminium-specific cleaning agent [a]	3,0
Sodium nitrite	3,0
Distilled/de-ionized water	100,0

[a] Typically, such a reagent would be based on a powdered mixture of alkaline borate, carbonate and phosphate salts used in conjunction with a non-ionic detergent.

Prepare the solution as follows:

With constant stirring, dissolve the sodium nitrite and the phosphate-based cleaning agent in the water and heat to (80 ± 2) °C.

It is essential to maintain the bath's temperature. Hence, the requirement to preheat the items to be treated when using this etch solution (i.e. method 4).

7.2.1.2 Aluminium and alloys (anodized)

7.2.1.2.1 General

For general-purpose, non-critical bonding, the only preparation normally required for unsealed, sulfuric acid anodized surfaces is de-greasing followed by light abrasion (see 5.2.2 and 6.1.1). However, surfaces shall be bonded as quickly as possible after anodizing and preferably within 4 h. For critical structural applications, the oxide shall be removed by blasting (see 5.2.2 and 6.1.2), after which the fresh surface shall be re-anodized and bonded as rapidly as possible.

By contrast, surfaces which have been "hard"-anodized shall not be bonded because the surface oxide is unsuitable and shall be removed — preferably by blasting (see 5.2.2 and 6.1.2). Following this, any of the methods given in 7.2.1.1.2 can be used.

7.2.1.2.2 Method

- Remove oil or grease contamination (see 5.2.2).
- Scarify by abrading or blasting with alumina grit (see 6.1.1 and 6.1.2).
- Immerse the item to be etched for (20 ± 5) min at (62 ± 2) °C in the etch solution (7.2.1.2.3). Immerse the item in the electrolyte (7.2.1.2.4) of an anodizing bath set at a temperature of (90 ± 2) °C. Raise the voltage stepwise to 40 V, in increments not greater than 5 V, over a period of 10 min. Maintain this voltage for 20 min. Then raise the voltage progressively to 50 V over the next 5 min. Maintain the voltage at 50 V for a further 5 min.
- Remove the item, wash with tap water and rinse thoroughly in cold distilled or de-ionized water.

- — Conduct the “water-break” test.
- — Drain for 15 min.
- — Dry with warm, clean, dry, oil-free air for about 10 min at a temperature not exceeding 60 °C.
- — Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

7.2.1.2.3 Etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition of the etch solution is as follows:

Table 4 — Composition of the etch solution for aluminium and its alloys (anodized)

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Potassium dichromate or sodium dichromate	1,0
Concentrated sulfuric acid (ρ = 1,84 g/ml)	5,5
Distilled/de-ionized water	21,5

Prepare the solution as follows:

With constant stirring, cautiously add the acid to 60 % of the water and then add the dichromate. Stir until dissolution is complete and then add the remaining water.

7.2.1.2.4 Electrolyte

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The initial electrolyte has the proportion of 50 g of chromium trioxide made up into a 1 litre solution with water. As the chrome is consumed during the anodizing process, its concentration is maintained by the addition of more chromium trioxide. The solution strength shall be maintained at between 30 g and 100 g of chromium trioxide per litre of solution.

7.2.1.3 Chrome plate (irrespective of base material)

7.2.1.3.1 Method

- — Remove oil or grease contamination (see 5.2.2).
- — Either scarify (see 6.1) and bond (see below) or immerse the item for 1 min to 5 min at (93 ± 2) °C in the etch solution (7.2.1.3.2).
- — Remove the item and rinse thoroughly in cold distilled or de-ionized water.
- — Dry in a warm, clean, dry, oil-free air stream for about 10 min at a temperature not exceeding 60 °C.
- — Bond as soon as possible after drying and preferably within 4 h.

7.2.1.3.2 Etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition of the etch solution is as follows:

Table 5 — Composition of the etch solution for chrome plate

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Concentrated hydrochloric acid ($\rho = 1{,}18$ g/ml)	1,0
Distilled/de-ionized water	1,0

Prepare the solution as follows:

With constant stirring, cautiously add the acid to the water.

7.2.1.4 Copper and copper alloys (includes brass and bronze)

7.2.1.4.1 Method

- Remove oil or grease contamination (see 5.2.2).
- Either scarify with an alumina-based grit (6.1) and bond (see below) or immerse the item at room temperature for the appropriate, specified time in the preferred of the three etch solutions (see 7.2.1.4.2). Note that, for certain alloys, it could be necessary to vary the time required.
- Remove the item and rinse thoroughly in cold distilled or de-ionized water.
- Dry using cold, clean, dry, oil-free pressurized air. Hot air will discolour the surface.
- Bond as soon as possible after drying and preferably within 4 h.

7.2.1.4.2 Etch solutions

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

a) Ammonium persulfate

The composition is as follows:

Table 6 — Composition of etch solution a) for copper and copper alloys

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Ammonium persulfate	1,0
Water	3,0

Prepare the solution as follows:

With constant stirring, dissolve the ammonium persulfate in the water. Immerse the items for 1 min.

b) Iron(III) chloride

The composition is as follows:

Table 7 — Composition of etch solution b) for copper and copper alloys

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Aqueous iron(III) chloride (with a mass fraction of 42 % $FeCl_3$)	15,0
Concentrated nitric acid (ρ = 1,42 g/ml)	30,0
Distilled/de-ionized water	195,0

Prepare the solution as follows:

Add the iron(III) chloride solution to the water and then cautiously add the acid, stirring continuously. Immerse the items for 1 min to 2 min.

c) Nitric acid

The composition is as follows:

Table 8 — Composition of etch solution c) for copper and copper alloys

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Concentrated nitric acid (ρ = 1,42 g/ml)	7,0
Distilled/de-ionized water	15,0

Prepare the solution as follows:

While stirring continuously, add the acid cautiously to the water. Immerse the items for 30 s.

7.2.1.5 Magnesium and its alloys

7.2.1.5.1 General

This metal is dangerous and its potential to catch fire shall be considered.

WARNING:

DO NOT ABRADE MAGNESIUM OR ITS ALLOYS.

DO NOT EXPOSE COMPONENT SURFACES TO THE ATMOSPHERE OR A HOT VAPOUR BATH.

Unfortunately, while the use of the traditional chromium-based process is deprecated, there is as yet no suitable alternative. Consequently, it is suggested that method 1 be evaluated for the alloy in question and, if found unsuitable, then, and only then, shall the chrome-based process, method 2, be invoked.

7.2.1.5.2 Methods

a) Method 1:

- — **Bearing in mind the above warning**, remove oil or grease contamination (see 5.2.2).
- — Rinse thoroughly in cold distilled or de-ionized water.
- — Etch, at room temperature, in concentrated (density 1,75 g/ml) orthophosphoric acid for (10 ± 5) min.
- — Remove the item and rinse thoroughly in cold distilled or de-ionized water.

- Dry in a warm, clean, dry, oil-free air stream for about 10 min at a temperature not exceeding 60 °C.
- Bond immediately to reduce surface oxidation.

b) Method 2:

- **Bearing in mind the above warning**, remove oil or grease contamination (see 5.2.2).
- Immerse the item in the sodium hydroxide solution (7.2.1.5.3) for 10 min at (70 ± 5) °C. Wash in cold tap water.
- Immerse the item in the etch solution (7.2.1.5.4) for about 10 min at room temperature.
- Wash with cold tap water. Wash again in distilled or de-ionized water.
- Dry in a warm, clean, dry, oil-free air stream for about 10 min at a temperature not exceeding 60 °C.

7.2.1.5.3 Sodium hydroxide solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition is as follows:

Table 9 — Sodium hydroxide solution

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Sodium hydroxide	1,0
Distilled/de-ionized water	12,0

Prepare the solution as follows:

Add the sodium hydroxide to the water and stir until dissolution is complete.

7.2.1.5.4 Etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition of the etch solution is as follows:

Table 10 — Composition of the etch solution for magnesium and its alloys

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Sodium sulfate (anhydrous)	1,8
Calcium nitrate	2,2
Chromium trioxide	24,0
Distilled/de-ionized water	122,0

Prepare the solution as follows:

Add the reagents to the water in the order given above and stir until dissolution is complete.

7.2.1.6 Nickel and its alloys (including plate)

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

- Remove oil or grease contamination (see 5.2.2).

- — Either scarify (see 6.1) and bond or etch by immersing the item for 5 s at room temperature in concentrated nitric acid ($\rho = 1{,}42$ g/ml). Note that, for certain alloys, it might be necessary to vary the time required. Alternatively, etching may be carried out by using either solution 7.2.1.4.2 a) or 7.2.1.4.2 b) given under the preparation for copper (see 7.2.1.4).
- — Remove the item and rinse thoroughly in cold distilled or de-ionized water.
- — Dry in a warm, clean, dry, oil-free air stream for about 10 min at a temperature not exceeding 60 °C.
- — Bond as soon as possible after drying and preferably within 4 h.

7.2.1.7 Steel (mild) and iron, but excluding stainless steel

7.2.1.7.1 General

The four methods presented below increase progressively in complexity and, with that, the probability that they will enhance the durability of the ensuing joint.

7.2.1.7.2 Methods

a) Method 1:
    - — Remove oil or grease contamination (see 5.2.2).
    - — Scarify by abrading or blasting (see 6.1.1 and 6.1.2).
    - — Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

b) Method 2:
    - — Remove oil or grease contamination (see 5.2.2).
    - — Following scarification (see 6.1), use a proprietary etch in accordance with the manufacturer’s instructions.
    - — Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

c) Method 3:
    - — Remove oil or grease contamination (see 5.2.2).
    - — Either in conjunction with, or following, scarification (see 6.1), use a proprietary coupling agent in accordance with 6.4.2.
    - — Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

d) Method 4:
    - — Remove oil or grease contamination (see 5.2.2).
    - — Scarify by abrading or blasting (see 6.1.1 and 6.1.2).
    - — Immerse for 10 min at (60 ± 2) °C in the etch solution (7.2.1.7.3).
    - — Brush off the black deposit with a stiff nylon brush under clean, cold, running water.
    - — Blow-dry with clean, dry, oil-free air.
    - — Wipe over with propan-2-ol and allow the surface to dry.

- — Then heat for 1 h at (120 ± 3) °C.
- — Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

Oily (not lanolin-based) steel can be bonded very well with certain types of adhesive. However, for safety-critical structures, preparation should be thorough and use either method 3 or method 4 above.

7.2.1.7.3 Etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition of the etch solution is as follows:

Table 11 — Composition of the etch solution for steel

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Industrial methylated spirit	1,0
Phosphoric acid ($\rho = 1{,}7$ g/ml)	1,0

Prepare the solution as follows:

With constant stirring, mix the acid into the methylated spirit.

7.2.1.8 Steel (stainless)

7.2.1.8.1 General

The three methods presented below increase progressively in complexity and, with that, the probability that they will enhance the durability of the ensuing joint.

7.2.1.8.2 Methods

a) Method 1:

- — Remove oil or grease contamination (see 5.2.2).
- — Scarify by abrading or blasting with alumina grit (see 6.1.1 and 6.1.2).
- — Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

b) Method 2:

- — Remove oil or grease contamination (see 5.2.2).
- — Either in conjunction with, or following, scarification with an alumina-based grit (see 6.1), use a proprietary coupling agent in accordance with 6.4.
- — Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

c) Method 3:

- — Remove oil or grease contamination (see 5.2.2).
- — Scarify by abrading or blasting with alumina grit (see 6.1.1 and 6.1.2).
- — Wash in an alkaline detergent solution (7.2.1.8.3) (see also 5.2.2) for 10 min at (75 ± 5) °C.

- Rinse in distilled or de-ionized water.
- Immerse the item in the oxalic acid etch solution (7.2.1.8.4) for 5 min to 10 min at a temperature of (62 ± 2) °C.
- Rinse in cold distilled or de-ionized water while removing the black deposit formed during the oxalic etching with a stiff nylon brush.
- Dry. Use a hot, clean, dry, oil free-air stream, an oven or infrared lamps for about 10 min at a temperature not exceeding 95 °C.
- Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

7.2.1.8.3 Detergent solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition is as follows:

Table 12 — Composition of detergent solution

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Anionic alkylarylsulfonate detergent	0,5
Tetrasodium pyrophosphate	1,5
Sodium hydroxide	1,5
Sodium metasilicate	3,0
Distilled/de-ionized water	133,5

Prepare as follows:

Add the reagents to the water in the order given and stir to dissolve.

7.2.1.8.4 Oxalic acid etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition is as follows:

Table 13 — Composition of oxalic acid etch solution for steel (stainless)

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Oxalic acid	1,0
Concentrated sulfuric acid ($\rho = 1{,}84$ g/ml)	6,0
Distilled/de-ionized water	7,0

Prepare the solution as follows:

Add the sulfuric acid very cautiously to the water with continuous stirring. Then, with further stirring, add and dissolve the oxalic acid at the etching temperature of (62 ± 2) °C.

7.2.1.9 Tin

- Remove oil or grease contamination (see 5.2.2).
- Scarify by abrading or blasting (see 6.1.1 and 6.1.2).

- Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

7.2.1.10 Titanium

7.2.1.10.1 General

This metal and its alloys are usually used for critical aerospace applications. Consequently, the following observations shall be borne in mind.

In order to achieve maximum strength and durability, a rough but coherent layer of rutile titanium oxide shall be formed on the metal surface. This is achieved by using an etching process in which the rate of reaction of the alloy phases differs considerably. However, depending on the alloy being treated, etching gives rise to different surface structures. Consequently, the precise nature of the most appropriate treatment needs to be investigated for any newly introduced alloy. Further, titanium alloys are subject to hydrogen embrittlement, and consequently processes have been developed that minimize the uptake of hydrogen. Three processes are provided here., They can be considered appropriate for further development with a particular alloy.

7.2.1.10.2 Methods

a) Method 1:
    - Remove oil or grease contamination by using a vapour bath (see 5.2.2).
    - Scarify by abrading or blasting with alumina grit (see 6.1.1 and 6.1.2). Note that only alumina may be used and wet blasting is not yet recommended for this metal.
    - Wash in the detergent solution (7.2.1.10.3).
    - Etch the item with the alkaline etch solution (7.2.1.10.4) at (65 ± 2) °C until its surfaces have darkened uniformly — approximately 10 min to 20 min.
    - Wash for 10 min in cold [(23 ± 2) °C] distilled or de-ionized water.
    - Dry in a warm, clean, dry, oil-free air stream for about 10 min at a temperature not exceeding 60 °C.
    - Bond as soon as possible after drying and preferably within 4 h. Alternatively, immediately coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

b) Method 2:
    - Remove oil or grease contamination by using a vapour bath (see 5.2.2).
    - Scarify by abrading or blasting with alumina grit (see 6.1.1 and 6.1.2). Note that only alumina may be used and wet blasting is not yet recommended for this metal.
    - Wash in the detergent solution (7.2.1.10.3).
    - Wash for about 10 min in cold [(23 ± 2) °C] distilled or de-ionized water.
    - Etch the item by immersing for about 30 min at (75 ± 2) °C in the sodium tartrate etch solution (7.2.1.10.5).
    - Remove the item and rinse thoroughly in distilled or de-ionized water at room temperature.
    - Immerse in nitric acid (5 M) for about 10 min at room temperature to remove EDTA residues.
    - Remove the item and rinse thoroughly in distilled or de-ionized water at room temperature.
    - Dry in a warm, clean, dry, oil-free air stream for about 10 min at a temperature not exceeding 60 °C.
    - Bond as soon as possible after drying and preferably within 4 h. Alternatively, immediately coat the surface with an adhesive compatible primer — consult the adhesive manufacturer.

c) Method 3:

- — Remove oil or grease contamination by using a vapour bath (see 5.2.2).
- — Scarify by abrading or blasting with alumina grit (see 6.1.1 and 6.1.2). Note that only alumina may be used and wet blasting is not yet recommended for this metal.
- — Wash in the detergent solution (7.2.1.10.3).
- — Wash for about 10 min in cold [(23 ± 2) °C] distilled or de-ionized water.
- — Immerse the item in the warm [(30 ± 2) °C] sodium tartrate anodizing solution (7.2.1.10.5) in an anodizing bath and apply a DC voltage of 10 V for 15 min.
- — Remove the item and rinse thoroughly in distilled or de-ionized water at room temperature.
- — Dry in a warm, clean, dry, oil-free air stream for about 10 min at a temperature not exceeding 60 °C.
- — Bond as soon as possible after drying and preferably within 4 h. Alternatively, immediately coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

7.2.1.10.3 Detergent solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition is as follows:

Table 14 — Composition of the detergent solution for titanium

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Anionic alkylarylsulfonate detergent	0,5
Tetrasodium pyrophosphate	1,5
Sodium hydroxide	1,5
Sodium metasilicate	3,0
Distilled/de-ionized water	133,5

Prepare the solution as follows:

Add the reagents to the water in the order given and stir to dissolve.

7.2.1.10.4 Alkaline etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition is as follows:

Table 15 — Composition of the alkaline etch solution for titanium

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Sodium hydroxide	20,0
Hydrogen peroxide (30 %)	20,0
Distilled/de-ionized water	960,0

Prepare the solution as follows:

Dissolve the sodium hydroxide in the water with continuous stirring. Having done so, raise the temperature to 65 °C. Then, immediately before the items are to be treated, cautiously and with further stirring add the hydrogen peroxide.

7.2.1.10.5 Sodium tartrate etching and anodizing solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition is as follows:

Table 16 — Sodium tartrate etching and anodizing solution

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Sodium hydroxide	240,0
Sodium tartrate	75,0
Ethylenediaminotetraacetic acid (EDTA)	30,0
Sodium metasilicate	6,0
Distilled/de-ionized water	810,0

Prepare the solution as follows:

Add the sodium hydroxide and sodium tartrate to a proportion of the water and stir to dissolve. When dissolution is complete, add the EDTA and again stir to dissolve. Then, with stirring, add the sodium metasilicate to the remaining water. When it has dissolved, add the resulting solution to the previously prepared solution of sodium hydroxide, sodium tartrate and EDTA.

7.2.1.11 Zinc and its alloys, including hot-dip-galvanized steel, electro-galvanized steel and iron/zinc-alloy coated steel

7.2.1.11.1 General

The four methods presented below increase progressively in complexity and, with that, the probability that they will enhance the durability of the ensuing joint.

The load-bearing capacity of all the surfaces produced by the following methods will almost certainly be enhanced by the use of an adhesive with the lowest modulus practical. This is because such an adhesive will reduce the stresses imposed on what is likely to be a weak surface layer.

7.2.1.11.2 Methods

a) Method 1:

   The standard anti-corrosion and pre-painting passivation processes provide a bonding surface that will suffice for most non-structural bonding applications. However, care needs to be taken to ensure that the passivated layer is not too thick (a dark yellow or greenish appearance is an indication of this) and therefore weak and friable. Similarly, ensure it is not covered in a loose residue from a poor post-passivation washing process. If the surface is questionable, consider any of the following three methods.

b) Method 2:

   — Remove oil or grease contamination (see 5.2.2).

   — Scarify by abrading or blasting (see 6.1.1 and 6.1.2).

   — Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

c) Method 3:

- Remove oil or grease contamination (see 5.2.2).
- Following scarification (see 6.1), use a proprietary etch solution in accordance with the manufacturer's instructions.
- Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

d) Method 4:

- Remove oil or grease contamination (see 5.2.2).
- Either in conjunction with, or following, scarification (see 6.1), use a proprietary coupling agent in accordance with 6.4.
- Bond as soon as possible after drying and preferably within 4 h. Alternatively, coat the surface with an adhesive-compatible primer — consult the adhesive manufacturer.

7.2.2 Plastics

7.2.2.1 Acrylonitrile-butadiene-styrene copolymer

7.2.2.1.1 General

Acrylic- and solvent-based adhesives will normally bond this plastic without preparation. However, care needs to be exercised to ensure that stress cracking is not induced. It is recommended that plasma and flame treatments be considered (see 6.2) before using 7.2.2.1.2.

7.2.2.1.2 Method

- Remove oil or grease contamination, taking care to avoid stress cracking (see 5.2.2).
- Using a proprietary silane-coated abrasive, modify the surface in accordance with the manufacturer's instruction (see 6.4.2.2).
- Bond as directed.

7.2.2.2 Polyacetal

7.2.2.2.1 General

Components shall be stress-free before being prepared further. It is recommended that plasma and flame treatments be considered (see 6.2) before using the sulfonic-acid-based etch as described in 7.2.2.2.2.

7.2.2.2.2 Method

- Remove oil or grease contamination, taking care to avoid stress cracking (see 5.2.2).
- Immerse the item in the etch solution (7.2.2.2.3) at (94 ± 5) °C for (8 ± 2) s.
- Heat in an oven at (110 ± 10) °C for (45 ± 15) s.
- Remove the item and rinse thoroughly in water at (55 ± 5) °C.
- Dry in a warm, clean, dry, oil-free air stream at a maximum of 60 °C.
- Bond as soon as possible after drying and preferably within 4 h.

7.2.2.2.3 Etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition is as follows:

Table 17 — Composition of the etch solution for polyacetal

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Perchloroethylene	96,0
Dioxane	3,7
p-Toluenesulfonic acid	0,3

Prepare the solution as follows:

Mix the dioxane and perchloroethylene. Stir in the acid until completely dissolved.

7.2.2.3 Polyamide

7.2.2.3.1 General

Polyamide-based plastics are hygroscopic. Consequently, if heat-cured adhesives are to be used, absorbed water shall be removed from the body of the component, by heating if necessary, before bonding begins. Otherwise, released vapour can cause the adhesive to foam during the curing process.

It is recommended that plasma and flame treatments be considered (see 6.2) before using 7.2.2.3.2.

Prior to bonding, always ensure that absorbed water is removed by heating if necessary.

7.2.2.3.2 Method

- Remove oil or grease contamination, taking care to avoid stress cracking (see 5.2.2).
- Immerse the item in the etch solution (7.2.2.3.3) for (8 ± 2) s at room temperature.
- Dry in a well-ventilated atmosphere at (23 ± 2) °C for not more than 30 min.
- Bond as soon as possible after drying and preferably within 4 h.

7.2.2.3.3 Etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2.

The composition is as follows:

Table 18 — Composition of the etch solution for polyamide

Reagent (technical or reagent grade)	Parts by mass (±1 %)
Ethyl acetate	91,0
Resorcinol	9,0

Prepare the solution as follows:

Stir the resorcinol into the ethyl acetate until dissolution is complete.

7.2.2.4 Poly(butylene terephthalate)

It is recommended that the flame and plasma treatments be considered before using the newer laser treatment (see 6.2) or 7.2.2.1.2, viz

- Remove oil or grease contamination, taking care to avoid stress cracking (see 5.2.2).
- Using a proprietary silane-coated abrasive, modify the surface in accordance with the manufacturer's instructions (see 6.4.2.2).
- Bond as directed.

7.2.2.5 Polyethylene

7.2.2.5.1 General

It is recommended that the flame and plasma treatments be considered before using the newer laser treatment (see 6.2) or 7.2.2.5.2.

7.2.2.5.2 Methods

a) Method 1:
   - Remove oil or grease contamination, taking care to avoid stress cracking (see 5.2.2).
   - Using a proprietary silane-coated abrasive, modify the surface in accordance with the manufacturer's instructions (see 6.4.2.2).
   - Bond as directed.

b) Method 2:
   - Remove oil or grease contamination, taking care to avoid stress cracking (see 5.2.2).
   - Etch by immersing the item in a proprietary solution of sodium naphthalide (7.2.2.5.3) for between 5 s and 10 s.
   - Wash with water, acetone and water, in that sequence, unless otherwise required.
   - Dry in a warm, clean, dry, oil-free air stream at a maximum of 60 °C.
   - Bond as soon as possible after drying and preferably within 4 h.

7.2.2.5.3 Etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2. Prepare the solution in accordance with the manufacturer's instructions.

7.2.2.6 Polypropylene

It is recommended that the flame and plasma treatments be considered before using the newer laser treatment (see 6.2) or one of the methods in 7.2.2.5.2.

7.2.2.7 PTFE (polytetrafluoroethylene)

7.2.2.7.1 General

If the flame or plasma techniques (see 6.2) are to be investigated, and used, then care shall be taken to ensure that any fumes, resulting from the heating of the plastic, are suitably dealt with because they are likely to be highly corrosive.

An alternative, material-specific, preparative method (see 7.2.2.7.2) is based on a solution of sodium naphthalide.

7.2.2.7.2 Method

- Remove oil or grease contamination, taking care to avoid stress cracking (see 5.2.2).
- Etch, by immersion, in a proprietary solution of sodium naphthalide (see 7.2.2.7.3) for 5 s to 10 s.
- Wash with water, acetone and water, in that sequence, unless otherwise required.
- Dry in a warm, clean, dry, oil-free air stream for about 10 min at a temperature not exceeding 60 °C.
- Bond as soon as possible after drying and preferably within 4 h.

7.2.2.7.3 Etch solution

Before commencing, see Clause 4 and requirements a) to e) of 6.3.2. Prepare the sodium naphthalide solution, using recommended equipment, in accordance with the manufacturer's instructions.

8 Evaluation of durability

The purpose of surface preparation is to enhance both the reliability of the bonded joint and its long-term durability — especially when it is exposed to harsh environments.

It has been shown that accelerated ageing can be used successfully to rank the improvements provided by the various preparative techniques available, though it is generally agreed that laboratory evaluation does not equate to actual reality — that latter usually being less demanding. However, for safety-critical structures, the requirements of EN 2243-5 may be used as a guide to the performance levels that need to be achieved.

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