INTERNATIONAL STANDARD

ISO 17212

Second edition
2012-02-15

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

Reference number
ISO 17212:2012(E)

Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

