SLOVENSKI STANDARD
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Konstrukcijska lepila – Navodila za pripravo kovinskih in polimernih površin za lepljenje

Structural Adhesives - Guidelines for surface preparation of metals and plastics prior to adhesive bonding

Strukturklebstoffe - Leitlinien für die Oberflächenvorbehandlung von Metallen und Kunststoffen vor dem Kleben

Adhésifs structuraux - Guide pour la préparation des surfaces des métaux et des plastiques avant le collage par adhésif

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Structural Adhesives - Guidelines for surface preparation of metals and plastics prior to adhesive bonding

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This European Standard was approved by CEN on 10 July 2003.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 13887:2003) has been prepared by Technical Committee CEN /TC 193, "Adhesives", the secretariat of which is held by AENOR.

This. European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2004, and conflicting national standards shall be withdrawn at the latest by February 2004.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

Historically, surface preparation has required the use of solvents and reactive chemicals. However, it should be noted that the use of such materials is now generally deprecated and individual identified substances are becoming progressively banned throughout the EU. For these reasons, traditional procedures using, for example, chromic acid are – with one exception - not provided in this standard. In addition, such chlorinated solvents, as may be allowed by the Montreal Protocol and National laws, should only be used in enclosed systems. Even so, the use of these solvents is not generally supported by this standard. See also EU Regulation (EC) No 2037/2000 [1].

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

— the removal of contaminants;

— physically induced modification of the surface;

— chemical treatment.

However, legislative pressure is driving development and with that the introduction of new methods. In these latter, the more hazardous oxidative treatments, and their associated disposal problems, are avoided. The corresponding introduction of less aggressive oxidising techniques, or the use of coupling agents, often allows the separate steps of the foregoing sequence to be advantageously combined. These new processes are largely proprietary and, where used, the coupling agents tend to be based upon silane chemistry. However, whatever the process, it is important to realise that while the effectiveness of the new techniques (presented here) is well established, general utility cannot be assumed. Consequently, it is necessary to demonstrate that any method selected is appropriate for the intended purpose and that the optimal conditions for the production process itself have been correctly established.

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, its function (joining, sealing, etc.) and the environment in which it has to perform.

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 engineering metals, or their alloys, can be bonded satisfactorily. However, the optimisation of the durability of a metal-based joint usually requires the introduction of progressively more complex and specific treatments.

The majority of both thermoset and thermoplastics materials can be prepared by commonly applicable techniques. Were it not for contamination and residual mould release agents, the thermoset plastics (e.g. polyepoxy and polyester composites) would bond well without any preparation at all. By contrast, most thermoplastics require careful preparation because of their low surface energy. However, while the techniques required are generally applicable there will often be differences in the process detail.

Some paints - especially the cataphoretic, water-based primers used by the vehicle industries, can provide an excellent surface for bonding. Nevertheless, a check of the stability of the interface below the paint is recommended. Sometimes the surface of the paint, 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 at all prior to bonding.

The various methods provided for cleaning and modifying surfaces are drawn from current and developing practice. The techniques described can be used in a variety of combinations to create the basis of a practical, preparative process that will provide enhanced environmental durability.

This standard does not provide a means of identifying the most appropriate method of preparation. This can only be determined, in particular instances, with regard to the manufacturing process and the eventual use of the bonded items. However, in any event, the method used, should be both compatible with evolving National legislation and EC regulations and represent the least hazardous means of achieving the performance required.

A variety of process options are described for commonly encountered metals and plastics in clause 7.

1 Scope

This European Standard specifies the usual procedures for the preparation of component surfaces prior to bonding for either laboratory evaluation or the process of construction.

This European Standard is applicable to metal and plastic surfaces that are commonly encountered.

These comprise the following metal and plastic families – the latter including filled versions and suitable paints :

METALS: aluminium, chromium, copper, magnesium, nickel, steel (mild), steel (stainless), tin, titanium, zinc.

PAINTS: cataphoretic, polyalkyd, polyester, polyepoxide, polyurethane.

PLASTICS:

thermoplastic: Acrylonitrile/butadiene/styrene plastic (ABS plastic), Polyacetal plastic, Polyacrylic plastic, Polyamide plastic (PA plastic), Poly(butylene terephthalate) (PBT), Polycarbonate plastic (PC plastic), Polyester plastic, Polyetheretherketone (PEEK), Polyethersulfone (PES), Polyethylene plastic (PE plastic), Polyimide, Poly(methyl methacrylate) plastic (PMMA plastic), Poly(phenylene oxide) (PPO), Polypropylene plastic, Polystyrene plastic (PS plastic), Polytetrafluoroethylene (PTFE), Poly(vinyl chloride) plastic (PVC plastic).

thermoset: Allylphthalate plastic, Aminoplastic, Cellulose esters, Epoxy plastic, Polyester plastic, Phenolic plastic, Polyurethane (PUR).

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 923:1998, *Adhesives - Terms and definitions*.

EN 2243-5, *Aerospace Series – Structural adhesives – Test methods - Part 5: Ageing tests*

EN ISO 472:2001, *Plastics – Vocabulary (ISO 472:1999).*

3 Terms and definitions

For the purposes of this European Standard, the terms and definitions given in EN 923:1998 and in EN ISO 472:2001 and the following apply.

3.1
plastic (including paint)
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 Elastomeric materials, which also are shaped by flow, are not considered as plastics.

NOTE 2 In some countries, particularly in the United Kingdom, it is a permitted option to use the term "plastics" as the singular form as well as the plural form.

NOTE 3 Adapted from EN ISO 472

4 Safety and environment legislation

The users of this standard shall be familiar with normal laboratory practice and the principles of good industrial hygiene.

This standard does not purport to address all safety problems, if any, associated with its use. It is the responsibility of the user to establish safety and health practices and to ensure compliance with any European and national regulatory conditions.

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

Concentrated acids, alkalis and oxidising agents are 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. Do not allow any cleaning materials to contact the skin. Abuse can lead to dermatitis.

Attention is drawn to the identification of solvents in accordance with EU Directive 89/655/EEC [2].

Wherever possible use propan-2-ol as a solvent, preferably in a closed system. Otherwise, if there is no practical alternative, a ketone can be used though it shall be employed in a closed system. Take care! All solvent-associated, handling processes shall take into account the flammability of alcohols and ketones - particularly ketones – and the fact that all such materials are narcotic in concentration. Ensure that appropriate precautions are taken to eliminate all sources of solvent vapour and ignition – particularly electro-static sparking and to prevent operators from being exposed to solvent vapour during any part of the work cycle.

Waste and spent materials shall be disposed of in accordance with EU and national legislative requirements through the services of an authorised disposal organisation - 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, personal 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.

It is useful to mention that only a few paint surfaces are capable of supporting anything other than purely nominal loads. The exceptions are typically those based upon the aqueous, electrochemical paints - such as those used in the automotive industries and the epoxy and polyester based paints used during the preparation of 'pre-coated' metal sheet.

NOTE 1 Elastomeric materials, which are also shaped by flow, are not considered as plastics.

NOTE 2 In some countries, the use of the term 'plastics' as the singular form as well as the plural form is permitted (as in the English language version).

5.2 Handling

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

5.3 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 they cannot only remove hydrocarbons but the more aggressive, stronger versions can also remove metallic soaps and salts. However, aluminium shall not be prepared with either a sodium hydroxide based cleaner or other alkaline material.

Some proprietary mixtures are used hot, while others utilise either anodic or cathodic currents. Whichever cleaning agent is used, components shall always be rinsed thoroughly (see 6.3 d)) and dried in a stream of clean, warm, oil free and dry 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 a ketone (see clause 4). Solvents can severely damage some thermoplastic materials by either dissolving them or initiating stress cracking. Polycarbonate, polymethyl methacrylate and ABS based plastics are particularly susceptible in this latter regard.

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

Vapour baths are very effective and are recommended for the preparation of titanium and its alloys. However, they shall be based on a closed system. See clause 4.

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, fume, and vapour 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 performed in separate areas where such contamination can be avoided.

5.4 Storing

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 should be bonded as soon as possible after preparation and, before bonding, shall always be kept under defined conditions such that contamination shall be impossible.

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 who 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 General

Scarification, the shallow roughening of both metallic and plastic (see 3.1) surfaces, using either abrasion or blasting, almost invariably improves the performance of the final bonded joint.

Sometimes abrasion or blasting is required to smooth a coarse, rough surface, such as can be found on a casting, or simply to remove corrosion or other forms of contamination.

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

6.1.2 Abrasion

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

The following sequence is recommended:

a) abrade straight across in a convenient direction until all the surface has been lightly and uniformly scarified then ;

b) abrade similarly, at right angles until all traces (from a) above) have been obliterated then ;

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

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

e) then either bond or implement a further surface modification process.

If parts are to be bonded, then they shall be dry and shall be bonded as soon as practical (see 5.4). Drying can be speeded by the use of a clean, warm, oil free and dry air stream at a temperature not exceeding 60 °C.

NOTE 1 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 e) - the 'Water break test' - a procedure that demonstrates that a component's surface is free of contamination.

6.1.3 Blasting

Dry blasting is usually reserved for metallic components. However, when used carefully – to avoid excess erosion - the less aggressive processes can be effective when used on the more robust plastics. Proprietary processes are available. These encompass such specialised grits 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. 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 6.1.2 d) and 6.1.2 e) shall be implemented and the notes observed.

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 (see 3.1) by physically induced, oxidative processes. Some of these processes also introduce other chemical elements during the modification of the surface being treated. Some processes can also remove modest levels of contamination.

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

The following comments can 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 method is particularly useful for preparing strongly formed, three-dimensional components.

However, it shall be noted that careful, automated control of the process parameters is essential and that these latter need to be selected in relation to the polymer to be treated and the component's design.

To obtain consistent results particular attention shall be paid to:

— the gaseous fuel used;

— the residual oxygen content of the flame;

— the distance of the flame from the surface;

— the rate at which the flame moves across the surface;

— the standardisation of the time lapse prior to bonding ;

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 performance maintenance can be difficult 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 optimised 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, according to 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 5.4). All processes shall be standardised and, wherever practical, should be automated.

6.3 Chemical

Until recently, the usual object of the wet, chemical treatments was to oxidise a surface. However, as oxidation usually requires the use and disposal of powerful oxidising 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. See also clause 4.

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

a) the water used shall be either distilled or de-ionised 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 ; with a limit deviation of ± 1 %) and maintained by means of periodic sampling, analysis and relevant documentation;

c) waste and spent materials, chemicals and solutions shall be disposed of in accordance with legal requirements and through the services of an authorised disposal organisation - whose advice shall always be sought. Whenever solutions need to be made up, or utilised, then, with the possible exception of the sodium