INTERNATIONAL STANDARD

ISO 15166-1

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Adhesives — Methods of preparing bulk specimens —

Part 1:
Two-part systems

Adhésifs — Méthodes de préparation d'éprouvettes massiques —

Partie 1: Systèmes bicomposants

Reference number
ISO 15166-1:1998(E)

Contents

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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.

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.

International Standard ISO 15166-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

ISO 15166 consists of the following parts, under the general title *Adhesives — Methods of preparing bulk specimens:*

— *Part 1: Two-part systems*

— *Part 2: Elevated-temperature-curing one-part systems*

Introduction

Accurate values for the mechanical properties of adhesives are needed in the design of bonded joints to enable calculations of the stress distribution in the loaded joint to be made and failure criteria for the adhesive material to be determined. The use of test methods that employ specimens in the form of a bonded joint for the measurement of these properties is not ideal. This is because the thickness dimension of the bond constitutes the gauge length of the specimen for the determination of strain and, since the thickness is small (typically < 1 mm), strains cannot be routinely determined with high accuracy. Furthermore, the failure of a joint specimen usually arises under a multiaxial-stress state, the complexity of which depends upon many factors such as the geometry of the joint, the dimensions and properties of the adherends and the geometry of any fillets. The failure data derived from these specimens may be informative for design purposes, but additional data are needed corresponding to deformation and failure under more simple stress states such as shear and uniaxial tension.

The availability of bulk specimens with suitable dimensions will enable mechanical properties under different loading conditions and environments to be measured using methods developed and standardized for engineering plastics. These are both accurate and relatively inexpensive.

Procedures are described that will enable specimens to be obtained that have structures and properties comparable with those for the adhesive in a bonded joint. Additional tests on joint specimens will be necessary to give information relating to the performance of the bond between the adhesive and the adherend.

Adhesives — Methods of preparing bulk specimens —

Part 1:
Two-part systems

1 Scope

This part of ISO 15166 describes methods for the preparation of bulk specimens of adhesives.

The procedures described are suitable for two-part adhesives such as epoxies, polyurethanes, acrylics, etc.

The methods are not suitable for the preparation of bulk specimens of adhesives which require the evaporation of solvents or the liberation of gas for curing. They are also not suitable for pseudo-one-part adhesives that require an initiator to be applied to one of the surfaces to be bonded. In addition, the methods are not suitable for adhesives whose working life is very short, so that there is insufficient time to mix the components, dispense the adhesive and close the mould.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 15166. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 15166 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 472:1988, *Plastics — Vocabulary.*

ISO 2818:1994, *Plastics — Preparation of test specimens by machining.*

ISO 3167:1993, *Plastics — Multipurpose test specimens.*

3 Definition

For the purposes of this part of ISO 15166, the following definition, as given in ISO 472:1988, applies.

3.1 pot life; working life: The period of time during which an adhesive or resin, prepared for application, remains usable.

4 Mould description

4.1 General

The mould shall consist of two plates separated by spacers, a U-shaped frame or a continuous frame (see figure 1).

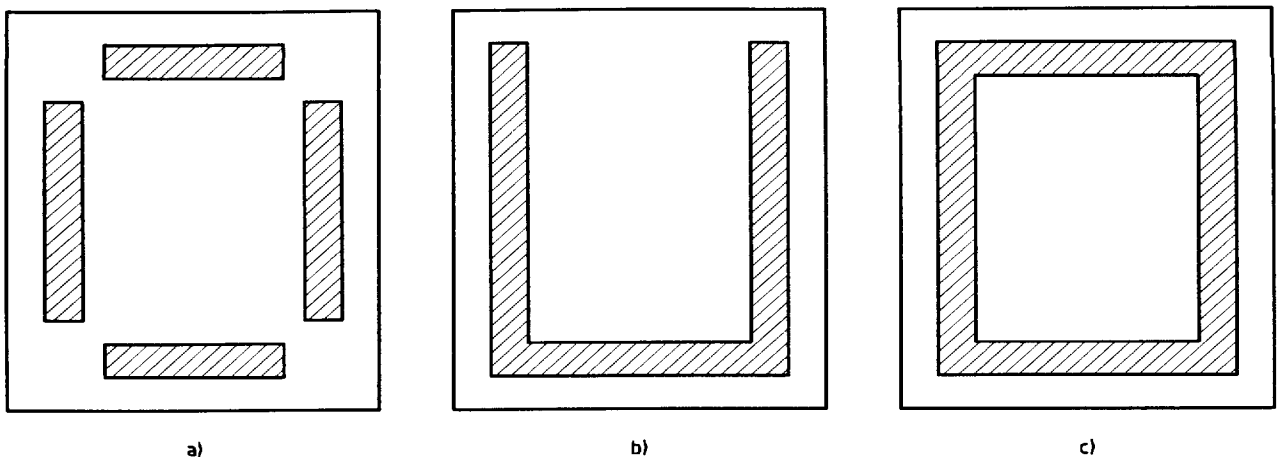


Figure 1 — Examples of suitable moulds with a) spacers, b) a U-shaped frame and c) a continuous frame

4.2 Plates

The two plates shall be made of metal and be at least 5 mm thick. This is both to provide a heat sink while curing and to avoid significant bowing of the plates under the forces experienced during moulding.

NOTE 1 Copper and brass are suggested as a plate material as they are good thermal conductors. However, when using certain components which are aggresive to copper, alternative metallic materials should be used.

Make sure that the adhesive does not adhere to the metal plates after cure.

NOTE 2 This can be achieved by applying on the plates a low-surface-energy material such as a polytetrafluoroethylene (PTFE) film, or by coating or spraying a release material on the surface of the plates. PTFE sprays need caution so as not to contaminate any other surfaces in the vicinity.

When preparing bulk specimens of adhesives that cure very rapidly at ambient temperature and where the evolution of large quantities of heat on curing is likely to lead to different properties of the adhesive, the plates in the mould shall be constructed to allow cooling water to pass through them.

4.3 Spacer or frame

A U-shaped frame as shown in figure 1b) is used for non-free-flowing adhesives. Spacers as shown in figure 1a) may be used for non-free-flowing adhesives cured at ambient temperature. A continuous frame as shown in figure 1c) shall be used for free-flowing adhesives.

Make sure that the adhesive does not adhere to the frame or spacers after cure.

NOTE 1 This can be achieved by applying on the frame or spacers a low-surface-energy material such as a PTFE film, or by coating or spraying a release material on the surface of the frame or spacers. PTFE sprays need caution so as not to contaminate any other surfaces in the vicinity. Alternatively the frame or spacers could be constructed from a low-surface-energy plastic such as PTFE.

The thickness of the frame determines the specimen thickness and shall be uniform to within ±0,5 %.

Ensure that the specimen thickness is sufficiently small that the temperature rise resulting from an exothermic cure reaction does not degrade the material (see clause 7).

NOTE 2 A thickness of 2 mm to 3 mm is suitable for most tests.

Where spacers are used to define the shape of the mould, precautions shall be taken to prevent relative movement of the spacers during casting or curing. Alternatively, a U-shaped frame may be used (see figure 1).

The dimensions of the mould shall be chosen to suit the type of test specimen required.

When bulk specimens are to be prepared having the standard multipurpose test specimen geometry specified in ISO 3167, the minimum length of the moulded material shall be greater than 150 mm.

Mechanically prepared test specimens shall be machined in accordance with ISO 2818, stamped or cut from the moulded plates.

Alternatively, pre-formed or shaped frames can be used to manufacture specimens of the desired geometry. The machine used for mixing the components may place a constraint on the mould volume (see 5.3).

5 Mixing and dispensing of the adhesive

5.1 Preparation of the individual components

Precautions shall be taken to minimize the volume of any entrapped air in the individual adhesive components, since air bubbles present in the materials before mixing can lead to voids in the final bulk specimen. Entrapped air can be removed from the materials by stirring the individual components under a vacuum. When there is a risk of losing volatile substances in this process, the operation shall be carried out at sufficiently low temperatures to avoid the removal of these substances.

NOTE — The adhesive manufacturer should preferably be consulted on whether vacuum degasssing is feasible.

Alternatively, and specifically for low-viscosity materials, the components shall be allowed to stand in the mixing apparatus until all visible air bubbles have risen to the surface. In this case, it shall be ensured that the final mixing operation will not introduce any further air.

5.2 Mixing

The components shall be mixed in the correct ratio in accordance with the manufacturer's instructions. Mixing is generally achieved by using static or dynamic mixers. Where a static mixer is used, the nozzle employed shall have a sufficient number of mixing elements to ensure thorough mixing of the components.

NOTE — When a static mixer is employed it is recommended that the mixer is evacuated during mixing to avoid entrapping air.

Where a free-flowing adhesive with a long working life is mixed under conditions which could introduce air into the mixture, the final product shall be degassed under a vacuum before moulding.

5.3 Mixture volume

When the mixing operation is not continuous and thus a limited volume of adequately mixed components is generated by the mixing machine, the mould volume shall be smaller than this discharged volume.

5.4 Dispensing

5.4.1 Non-free-flowing adhesives

These adhesives shall be dispensed from cartridges with a nozzle or from more sophisticated dispensing equipment.

A continuous, steady flow of adhesive is required while dispensing.

NOTE 1 Cartridges with static mixers are generally used.

Discard the first quantity of material from nozzles in case the "first-out" mix is not to the correct ratio with the mixer nozzles used.

The nozzle shall remain within the cast of adhesive at all times to prevent the introduction of air (see figure 2).

Draw the nozzle slowly down the centre of the mould using spacers [see figure 1a)] or a U-shaped frame [see figure 1b)] in a continuous action without lifting the nozzle from the bottom of the mould or stopping the flow of adhesive (see figure 3).

NOTE 2 It is recommended that the nozzle is kept at an angle of approximately 30° to the plate (see figure 2).

The height of the dispensed adhesive shall be greater than the spacer thickness.

5.4.2 Free-flowing adhesives

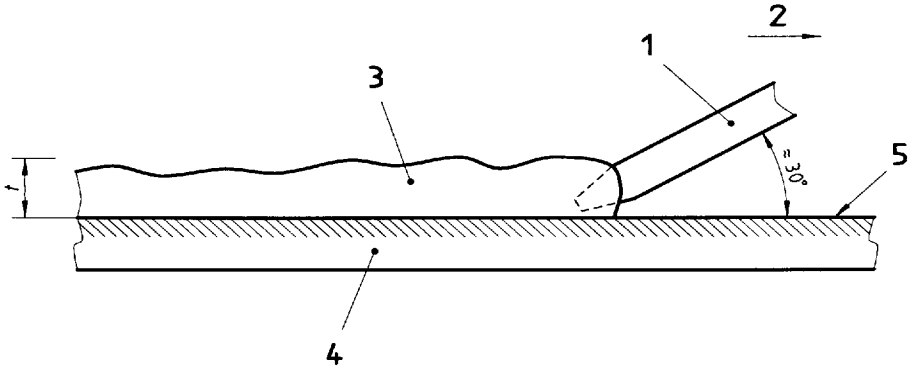
Where the components have been mixed by a static mixer or other mixing equipment, follow the procedure described in 5.4.1 but using a continuous-frame mould [see figure 1c)]. In this case, ensure that the volume of the adhesive is smaller than the volume of the mould cavity.

For long working life adhesives where the components have been mixed and subsequently vacuum degassed, the adhesive shall be degassed in a container with a lip to aid the pouring of the adhesive into the continuous-frame mould. Ensure that the volume of the adhesive is smaller than the volume of the mould cavity.

6 Moulding

To close the mould, place the plate which constitutes the upper part of the mould over one of the spacers or the bottom of the U-shaped frame and, using hand pressure, slowly press down onto the adhesive to spread the adhesive in the mould until the plate rests on the other spacers or sides of the frame.

When using the continuous frame (or the U-shaped frame in the specific case of curing at elevated temperature) apply sufficient clamping pressure between the upper and lower plates of the mould to prevent any excessive leakage of the adhesive during this curing process. Then incline the mould at an angle between 45° and 90° to the horizontal. When elevated-temperature cure is also required, then maintain the mould at this inclined orientation within the curing oven.

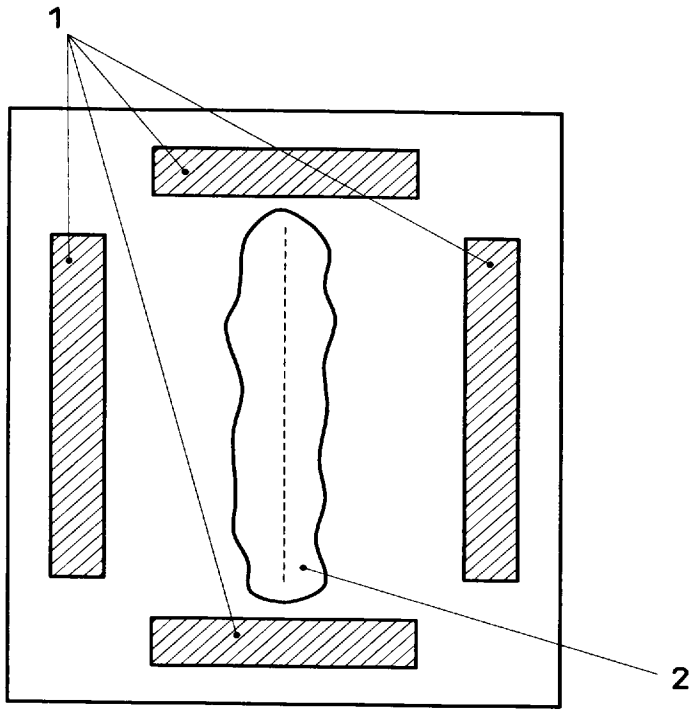


Key

1 Nozzle
2 Direction of nozzle travel
3 Adhesive cast
4 Metal baseplate
5 Release agent

t is greater than the spacer thickness.

Figure 2 — Casting the adhesive



Key

1 Four spacers of equal thickness firmly attached to the baseplate.

2 Adhesive cast. The nozzle is not removed from the cast or lifted from the baseplate until casting is complete. The dashed line shows the path followed by the nozzle during casting.

Figure 3 — Adhesive cast prior to closing the mould

7 Curing

Cure the adhesive in accordance with the manufacturer's instructions.

When moulding a large-thickness specimen of an adhesive having a rapid exothermic reaction, it is important to ensure that the temperature rise is not so excessive that changes in the properties of the cured adhesive might occur. In case of uncertainty, use a thermocouple, placed in the adhesive, to monitor the temperature. When it is excessive, explore the use of extended cure times and lower cure temperatures.

8 Test report

The test report shall include the following information:

a) a reference to this part of ISO 15166;

b) all details necessary for identification of the adhesive, including batch number, manufacturer, etc.;

c) the date and time of casting;

d) the moulding parameters, including curing temperature and time, mould details, use of release agents, etc.;

e) details of any post-cure;

f) the storage conditions after cure.

ICS 83.180

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