Project PAJ2: Dynamic Performance of Adhesively Bonded Joints
Report No1: August 1996

Working Draft for an International Standard
Adhesives - Methods for preparing bulk specimens.
Part 2: elevated-temperature curing, one part systems

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Performance of Adhesive Joints: Project 2
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WORKING DRAFT FOR AN INTERNATIONAL STANDARD

ISO/WD 15166-2: Adhesives - Methods for preparing bulk specimens. Part 2: elevated-temperature curing, one-part systems

1 INTRODUCTION

Accurate values for the mechanical properties of structural adhesives are needed in the design of bonded joints to enable calculations of the stress distribution in the loaded joint and to determine failure criteria for the adhesive material. 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 measurement 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 standardised 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.

2 SCOPE

In this international standard, methods are described for the preparation by moulding of bulk specimens of adhesives.

The procedures described are suitable for elevated-temperature curing, one-part adhesives such as epoxies. They are not suitable for adhesives whose cure requires the evaporation of solvents or the liberation of gas.

3 NORMATIVE REFERENCES

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

ISO 3167: 1983 Plastics - Preparation of multipurpose test specimens.

ISO 2818: 1980 Plastics - Preparation of test specimens by machining.

4 MOULD DESCRIPTION

The mould shall consist of two plates separated by a frame. For non free-flowing adhesives, a U-shaped frame may be used, see figure 1a. In the case of low-viscosity materials which would flow out of the open frame within the time taken to close the mould, it is necessary to use a continuous frame to avoid leakage, see figure 1b.

4.1 PLATES

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

It is necessary to ensure that the adhesive does not adhere to the metallic plates after cure.

NOTE 1: This can be achieved by applying on the plates a low surface energy material such as a polytetrafluoroethylene (PTFE) film or coating, or by spraying a release material on the surface of the plates.

Ensure that the low surface energy material used does not degrade at the cure temperature nor migrate into the adhesive.

4.2 FRAME

It shall be ensured that the adhesive does not adhere to the frame after cure.

NOTE 2: This can be achieved by applying on the frame a low surface energy material such as a PTFE film or coating, or by spraying a release material on the surface of the frame. Alternatively the frame 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%.

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

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.

Mechanical test specimens shall be machined according to ISO 2818, stamped or cut from the moulded plates.

Alternatively, pre-formed or shaped frames can be used to manufacture specimens of desired geometry.

5 DISPENSING THE ADHESIVE

5.1 PREPARATION OF THE ADHESIVE

Precautions shall be taken to minimise the volume of any entrapped air in the adhesive since air bubbles present in the material will lead to voids in the final bulk specimen. Entrapped air can be removed from the material by stirring the adhesive under 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.

5.2 DISPENSING

5.2.1 Non free-flowing adhesives

These adhesives shall be dispensed from cartridges with a nozzle. A continuous, steady flow of adhesive is required while dispensing.

The nozzle should remain within the cast of adhesive at all times to prevent the introduction of air (Figure 2).

The nozzle should be drawn slowly down the centre of the mould in a continuous action without lifting from the bottom of the mould or stopping the flow of adhesive (Figure 3).

NOTE 4: It is recommended that the nozzle is kept at an angle of about 30° to the plate (Figure 2).

The height of the dispensed adhesive shall be greater than the height of the frame.

5.2.2 Free-flowing adhesives

Where the adhesive is not supplied in a cartridge, the adhesive shall be degassed in a container with a lip to aid the pouring of the adhesive into the mould.

Dispense the adhesive carefully into the centre of the mould so as to avoid introducing air by turbulent flow. In this case, ensure that the volume of the adhesive is lower than the volume of the mould cavity.

If the adhesive is supplied in a cartridge, use the procedure described in clause 5.2.1 with a continuous frame and ensure that the volume of the adhesive is lower than the volume of the mould cavity.

6 MOULDING

Close the mould by placing the plate which constitutes the upper part of the mould over the frame. Where non free-flowing adhesives are being moulded with a U-shaped frame, place the upper plate over the bottom of the U-shaped frame, then slowly press down onto the adhesive using hand pressure to spread the adhesive in the mould until the plate rests on the frame. Apply sufficient clamping pressure between the upper and lower plates of the mould to prevent any excessive leakage of the adhesive during the curing process.

Place the mould into the curing oven such that it is inclined at an angle between 45° and 90° to the horizontal.

7 CURING

Cure the adhesive according to the manufacturer's instructions.

When curing adhesives that have a rapid exothermic reaction, it is important to ensure that the temperature rise does not exceed the maximum cure temperature specified by the adhesive manufacturer. In situations where the maximum temperature is exceeded, use the lower temperature of the cure temperature range specified by the manufacturer.

NOTE 5: It is unlikely that excessive temperatures will be experienced in mouldings with thickness ≤2 mm. Where there is a need for bulk specimens with a thickness greater than 3 mm, it is recommended that trial experiments are carried out using a mould in which calibrated temperature sensors are located in the mould cavity so as to measure the temperature in the centre of the moulding. If excessive temperatures are recorded, explore the use of lower cure temperatures and extended cure times. Specimens prepared this way shall be post-cured under recommended cure conditions.

8 TEST REPORT

The test report shall include the following:

a) a reference to this International Standard;

b) a full reference of the adhesive, including batch number, manufacturer, etc;

c) the date and time of casting;

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

e) details about any post-cure;

f) storage conditions after cure.

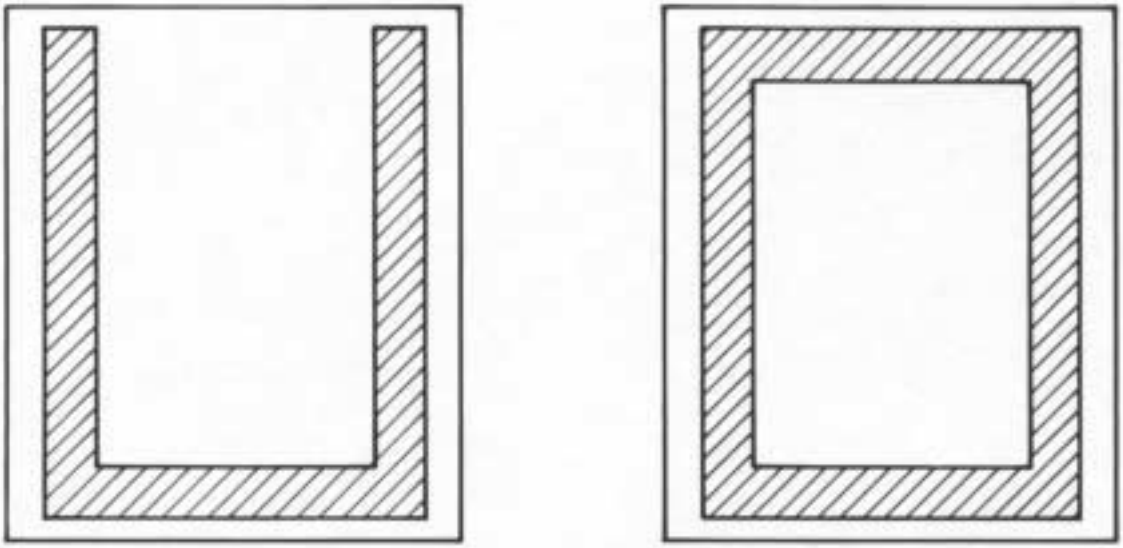

Figure 1 Example of suitable moulds with a) U-shaped frame for non free-flowing adhesives and b) continuous frame for free-flowing adhesives

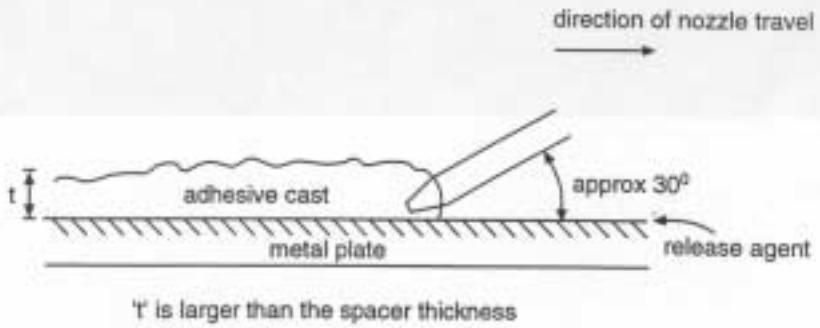

Figure 2. Casting the adhesive

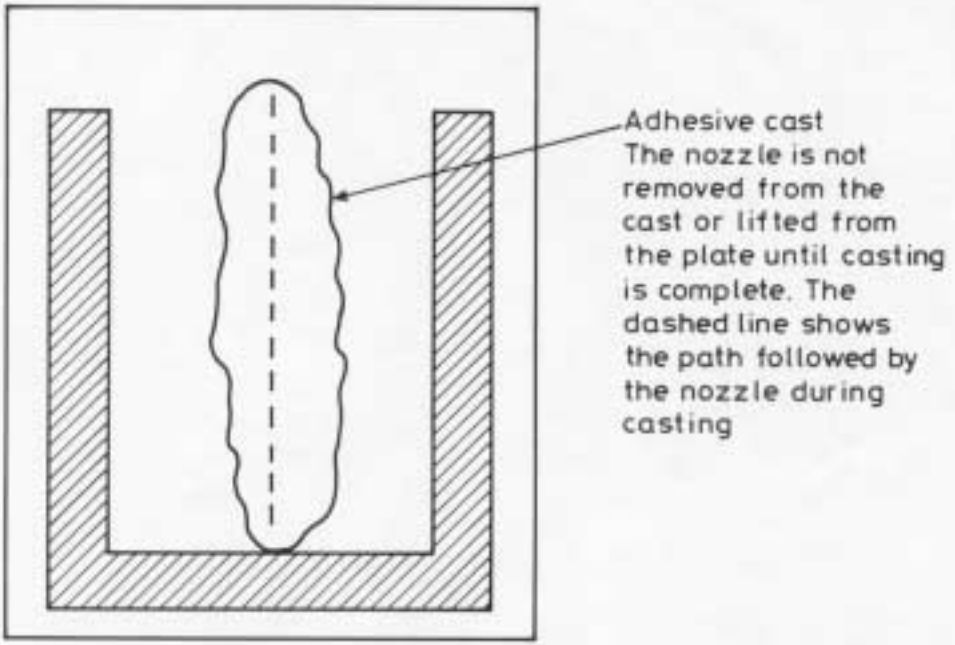

Figure 3 Adhesive cast prior to closing the mould