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Manufacture of Quality Specimens

Lucas F.M. da Silva, Stefanos Giannis, Robert D. Adams, Edoardo Nicoli, Jean-Yves Cognard, Romain Créac'hcadec, Bamber R.K. Blackman, Hitendra K. Singh, Charles E. Frazier, Laurent Sohier, and Bernard Gineste

1.1
Preparing Bulk Specimens by Hydrostatic Pressure

Lucas F.M. da Silva

1.1.1
Introduction

There are many test methods for the determination of failure strength data. Basically, they can be divided into two main categories: tests on neat resin or bulk specimens and tests in a joint or *in situ*. Tests in the bulk form are easy to perform and follow the standards for plastic materials. However, the thickness used should be as low as possible to represent the thin adhesive layer present in adhesive joints. Tests conducted on *in situ* joints more closely represent reality, but there are some difficulties associated with accurately measuring the very small adhesive displacements of thin adhesive layers. There has been intense debate about the most appropriate method and whether the two methods (bulk and *in situ*) can be related. Some argue that the properties in the bulk form may not be the same as those in a joint because the cure in the bulk form and the cure in a joint (thin film) may not be identical. In effect, the adherends remove the heat produced by the exothermic reaction associated with cure and prevent overheating. To minimize this problem, cure schedules should be selected to ensure that the thermal histories of the materials are similar in each case. Dynamic mechanical thermal analysis (DMTA) or differential scanning calorimetry (DSC) measurements can be made to compare the final state of cure of the materials (Section 6.1).

Bulk specimens are usually manufactured by pouring or injecting the adhesive in a mold with the final shape (Section 1.2), or by pressure between plates. The first method is suited to one-part adhesives that are relatively liquid. The mold can be open but can also be a closed cavity, in which case the adhesive needs to be injected. When the adhesive is viscous, in the form of a film or of two components, the

Testing Adhesive Joints: Best Practices, First Edition.
Edited by Lucas F.M. da Silva, David A. Dillard, Bamber Blackman, and Robert D. Adams.

second method generally gives better results. If the adhesive is viscous or in film form, the pouring (or injection) phase is difficult or impossible. On the other hand, the mixing of two-part adhesives can introduce voids. If the adhesive is liquid, the air bubbles can be removed by vacuum. da Silva and Adams [1] have used an ''open'' vacuum release technique to produce void-free specimens with limited success. If the adhesive is viscous, recent sophisticated machines in which the mixing is done at high speed under vacuum can ensure that the adhesive is void free. If the voids have been removed properly, the adhesive can be manufactured by pouring or injection, taking care not to introduce voids during this operation. A number of simple techniques have been used to reduce void incorporation during mixing and dispensing, such as mixing in a sealed bag by kneading and then snipping off a corner to dispense or preparing the adhesive in a syringe to minimize air entrainment. If not, the voids can be removed by high pressures and an excess of adhesive to compensate for the voids.

Although voids and other defects in bulk specimens may have minimal effects on averaged properties, such as modulus and other constitutive properties, they can have a significant effect on failure properties such as various strength metrics and strain at break. In reducing voids, it is important to understand where their source is. Voids can result from outgassing of the adhesive during cure, from air entrained in the adhesive during mixing or dispensing, and from absorbed water that evaporated at the elevated cure temperatures. Although some adhesives intentionally outgas to create foams, most adhesives do not outgas significantly. Especially when the cure temperature exceeds 100 °C, absorbed water in the adhesive can be vaporized, leaving the adhesive riddled with voids. Adhesive components that are known to absorb water may need to be dried before mixing to reduce voiding due to this latter mechanism.

1.1.2 Principle

The technique described in the French standard **NF T 76-142** works particularly well for producing plate specimens without porosity [1, 2]. It provides a technique for curing plates of adhesive in a mold with a silicon rubber frame under high pressure (2 MPa or 20 atm). The pressure is calculated using the external dimensions of the silicone rubber frame. The technique, shown schematically in Figure 1.1, consists of placing in the center part of the mold a quantity of adhesive slightly greater (5% in volume) than the volume corresponding to the internal part of the silicone rubber frame. There is a gap, at the beginning of the cure, between the adhesive and the silicone rubber frame. This gap enables, at the moment of application of the pressure, the adhesive to flow (until the mold is completely filled) and to avoid gas entrapment. Note that there is an external metallic frame to keep the silicone rubber frame in place. If the adhesive is a film, the lid is placed on top of the layers, but the load is applied only when the adhesive has the lowest viscosity to ease the adhesive flow. If the adhesive is a paste, the pressure is applied right from the beginning of the cure. However, an internal metallic frame is necessary to keep

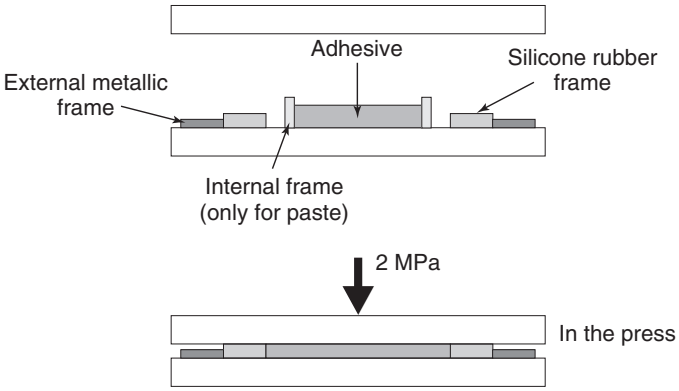


Figure 1.1 Adhesive plate manufacture according to **NF T 76-142**.

the adhesive at the center of the mold when being poured to guarantee that there is a gap between the adhesive and the silicone rubber frame. This frame is, of course, removed before the application of pressure. This technique is suitable for any type of adhesive, that is, liquid, paste, or film. Standard **ISO 15166** describes a similar method (Figure 1.2) of producing bulk plate specimens. However, this standard does not include a silicone rubber frame and just uses spacers to control the adhesive thickness. The bulk specimens obtained have a poor surface finish and contain voids, especially for two-part adhesives.

1.1.3 Metallic Mold

Standard **NF T 76-142** recommends using a metallic frame with an area of $150 \times 150\ mm^2$ and a silicone frame with a width of 50 mm and a thickness of 2 mm to produce an adhesive plate of $100 \times 100 \times 2\ mm^3$. A pressure of 2 MPa is applied on the external dimensions of the silicone frame, that is, in this case, 20 kN. The thickness of the silicone rubber frame gives the final thickness to the adhesive plate. However, a thin metallic frame may deform easily under pressure or due to an incorrect use, which must not occur if perfectly plane plates are to be obtained. Therefore, a more robust metallic support to keep the silicone frame in place is advised. For example, the mold represented in Figure 1.3 can be used. It consists of a base and a lid with a working area of $195 \times 90\ mm^2$ (external dimension of the silicone frame) for the application of the 2 MPa pressure (35.1 kN). That area is sufficient to machine two dogbone specimens for tensile testing. However, smaller or larger areas can be used. Four metallic pieces are put around the base and bolted to form a metallic box that fits the base and the lid. The metallic box keeps the silicone frame in place and also enables excess of adhesive to escape. Figure 1.4 shows an exploded view of the mold with the metallic frame, the silicone rubber frame, and the adhesive plate. All the pieces of the mold have a good finish (ground), especially the base and the lid, because they will dictate the surface finish

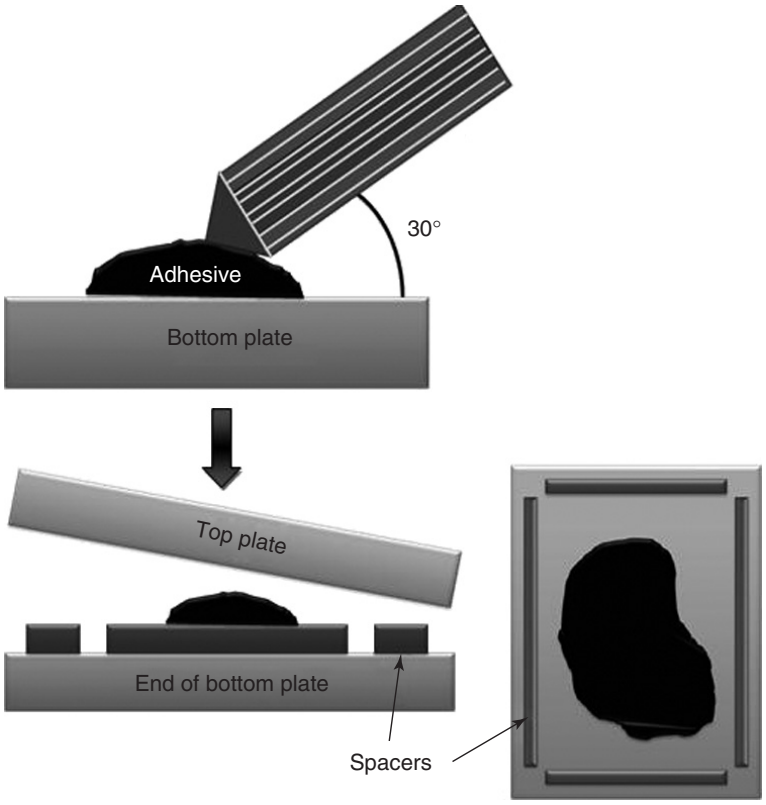


Figure 1.2 Adhesive plate manufacture according to **ISO 15166**.

of the adhesive plate. The metal used to build the metallic mold can be carbon steel (e.g., 0.45% C) in the annealed condition. It is cheap, easy to machine, and guarantees good heat dissipation.

1.1.4 Silicone Frame

The silicone rubber frame seals the adhesive very tightly enabling the application of hydrostatic pressure to the adhesive. The silicone rubber frame also serves as a thickness control since the thickness of the adhesive plate is equal to that of the silicone frame because of the incompressibility of the silicone. Generally, a thickness of 2 mm is used. Larger thicknesses can be used, but the exothermic reaction during cure can cause adhesive burning for some adhesives. Thicknesses of up to 16 mm have been produced with the mold presented in Figure 1.4. Thick plates can be used to produce round specimens. The silicone used is a room temperature vulcanizing (RTV) silicone. The hardness of the silicone is not critical, and soft (20 shore A) to hard silicones (70 shore A) can be used. In general, a silicone

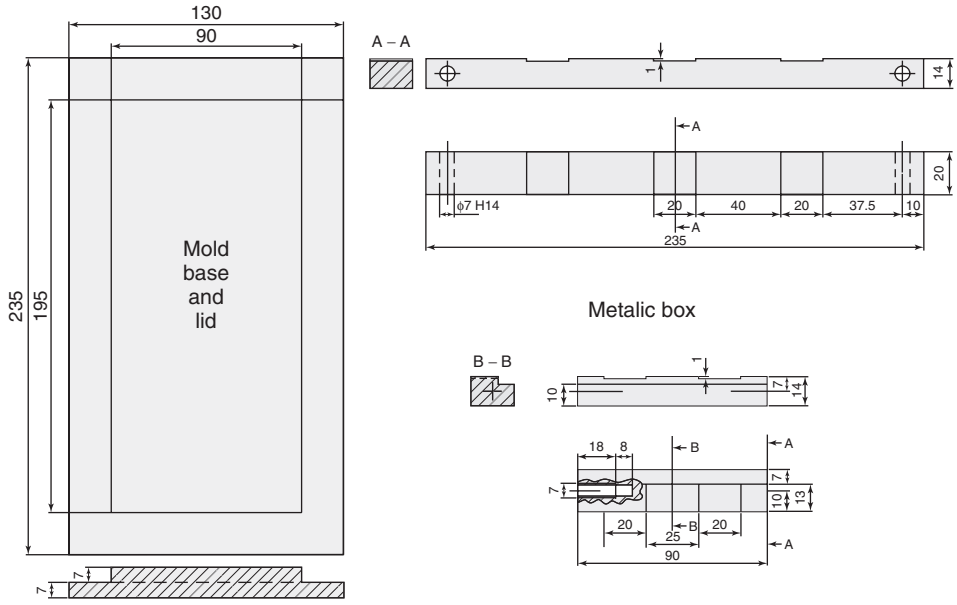


Figure 1.3 Dimensions in mm of the metallic mold.

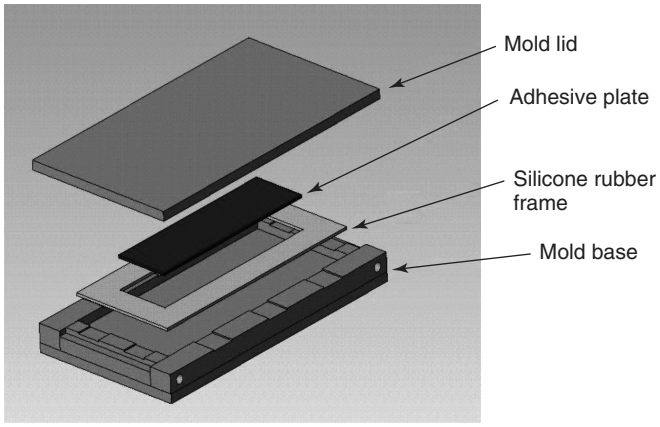


Figure 1.4 Exploded view of the mold to produce plate specimens under hydrostatic pressure.

with 50 shore hardness is used. Sheets of silicone rubber can be purchased easily in drugstores. Alternatively, a sheet of silicone can be manufactured with uncured silicone using the metallic mold described above. The silicone can be cut to the final dimensions with a cutter. The width of the silicone rubber used for the mold presented above is 22.5 mm, which means that the internal space of the silicone rubber frame (also the dimensions of the adhesive plate) is $140 \times 45 \times 2$ mm^3. The width of the silicone rubber frame should not be less than 15 mm.

1.1.5
Adhesive Application

Before the adhesive application, the release agent must be applied to the metallic mold. It is not necessary to apply the release agent to the silicone rubber frame because most adhesives do not usually bond well to silicone (unless silicone is used). The release agent should be well cured before the adhesive is applied to avoid interaction of the release agent with the adhesive. It is very important to precisely control the quantity of adhesive to deposit in the mold. An adhesive in excess of 5% in volume should be applied. The amount of adhesive to apply is calculated from the volume of the adhesive plate ($140 \times 45 \times 2\ mm^3$) plus 5% and the adhesive density. The adhesive is weighted carefully in a precision scale and then transferred to the mold (Figure 1.5a,b). In the case of two-component adhesives, the mixture of the resin and hardener introduce voids, but this technique eliminates most of these voids. To reduce even further the quantity of voids, a toothpick can be used to burst the air bubbles before the application of pressure. If the adhesive is a liquid, the adhesive can be degassed with vacuum.

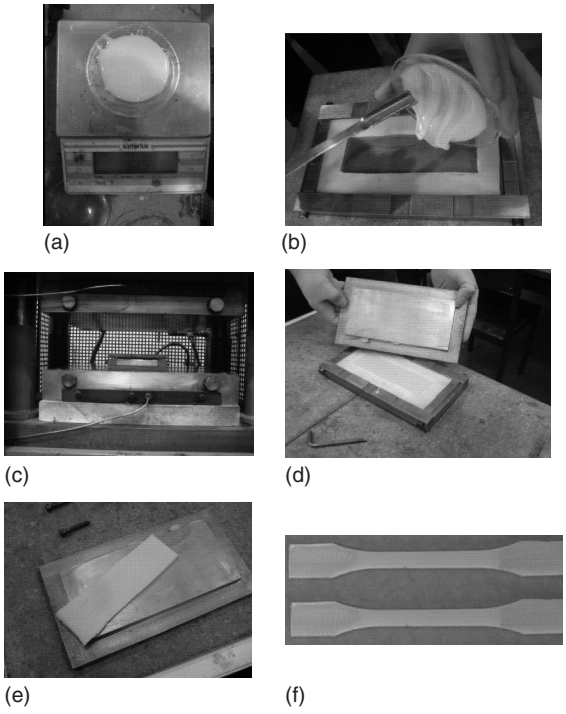


Figure 1.5 Manufacture of bulk specimens under hydrostatic pressure. (a) Weighing of the adhesive, (b) adhesive application in the mold, (c) cure in a hot press, (d) adhesive plate removal from the mold, (e) final adhesive plate, and (f) dogbone specimens obtained machined from the adhesive plate.

1.1.6
Cure

The high pressure is best applied using a hot press (Figure 1.5c). In case the adhesive cures with temperature, the hot press is also the most practical equipment to apply temperature in a short time. However, it is advised to use a thermocouple close to the adhesive and to count the cure time from the moment the adhesive reaches the cure temperature.

After the adhesive cure, the cooling rate should be slow to guarantee a uniform temperature in the mold and avoid residual stresses. Also, the plate should not be removed before the mold has reached room temperature; otherwise, the residual stresses can deform the plate permanently.

After cure, the lateral parts of the mold are unbolted and the adhesive plate is easily removed (Figure 1.5d). The surface finish of the adhesive plate is excellent because of the high pressure, as can be seen in Figure 1.5e.

1.1.7
Specimen Machining

The plates are then machined according to the dimensions used for mechanical or physical testing with any type of geometry (Figure 1.5f) (see Sections 2.1, 2.3, 2.4, 2.5, 3.1, 3.5, 4.4, 5.1, 5.4, 6.1 and 6.6). The standard **ISO 2818** gives details on how to machine specimens from adhesive plates. The use of coolants should be avoided because they can diffuse into the adhesive and influence the adhesive's mechanical behavior. In that case, the adhesive should be dried before being tested. It is better to use an air coolant. Machining might not be possible with very flexible adhesives. In that case, sharp dies can be used to cut out dogbone or other shapes from flexible adhesive sheets. Alternatively, injection or pouring techniques can be used. The specimens should be conditioned under controlled temperature and humidity because these factors influence the mechanical properties of the adhesive.

1.1.8
Results

The strain to failure is highly dependent on the presence of defects such as voids and microcracks. In tension, once a crack is triggered next to a void, the specimen often fails there because of the high stress concentration. Generally, the strain to failure can vary widely unless the manufacture is very well controlled. Figure 1.6 shows tensile stress–strain curves of a two-part epoxy adhesive that was manufactured with the technique described above. This adhesive is relatively brittle and therefore, particularly sensitive to voids. Four curves are presented, and the difference between them is barely perceptible, even in terms of strain to failure, which shows that the hydrostatic pressure technique is well suited to manufacture bulk specimens.

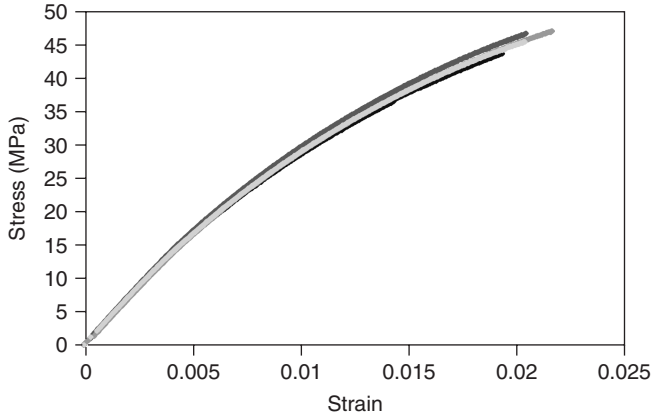


Figure 1.6 Tensile stress–strain curve of a two-part epoxy adhesive manufactured with the hydrostatic pressure technique.

1.2
Preparing Bulk Specimens by Injection

Stefanos Giannis

1.2.1
Introduction

During the process of manufacturing of bulk specimens of adhesives and/or sealants for mechanical characterization, and to use for modeling adhesive joints, important issues can arise, and they are well addressed in the literature [3]. These are mainly voids that result from the inclusion of air, especially when mixing two-part components. Curing conditions can also introduce some issues, since curing of most adhesives is exothermic and, in thick layers such as those used for bulk specimens, additional heat may be present, which results in a different curing temperature compared to that of a joint configuration where the adhesive is present in a very thin layer and the effect of the additional heat is negligible.

Techniques for manufacturing sheets of bulk material are presented in Section 1.1. This method was found to work very well for a number of paste and film adhesives, by producing consistent void-free flat sheets of cured material from which tensile specimens can be cut. However, for tensile dumbbell-shaped specimens, an adequate way of cutting them out of the sheets has to be used. When manufacturing flat sheets of sealant materials, because the fully cured sealant is very soft, machining to the right dimensions is not a feasible option. In this case, cutting specimens out of the flat sheet, by means of a die having the appropriate geometry, is the main option. Nevertheless, potential edge effects due to die cutting could possibly affect the test results. An alternative method would be to mold the specimens in the desired shape. Some researchers used

a centrifuging technique to manufacture bulk specimens of viscous cold-cure adhesive [4]. Although they addressed all the issues associated with void formation during mixing of their adhesives and managed to create a smooth paste, free of air bubbles, by putting the adhesive into syringes and centrifuging at 3500 rpm for 10 min; they did not take into account void formation while injecting the material into the molds. An alternative methodology would involve injecting the material into the molds and centrifuging the whole mold. This technique was used in Ref. [5] and is presented here.

1.2.2 Mold

The metallic mold shown in Figure 1.7 can be used. It consists of three individual parts: (i) the base part, which is used as the support and is attached to the centrifuge, (ii) the middle part, which determines the shape and the thickness of the specimen, and (iii) the top part, which seals the mold. To ensure that the liquid material (e.g., adhesive or sealant) does not stick to the mold during curing, it is recommended to apply three layers of a release agent (e.g., Frekote 55-NC, Henkel) before the injection of the uncured material. For the application of the release agent, the metallic molds can be heated to 80 °C. The three parts are then fitted together with bolts, which are tightened to 10 Nm. The uncured material is injected through a 2.5 mm threaded hole, as shown in Figure 1.7, at one side of the mold, while a 1.5 mm release hole at the other side is used to ensure that the interior is filled with material. When the mold is filled, both these holes are closed with screws.

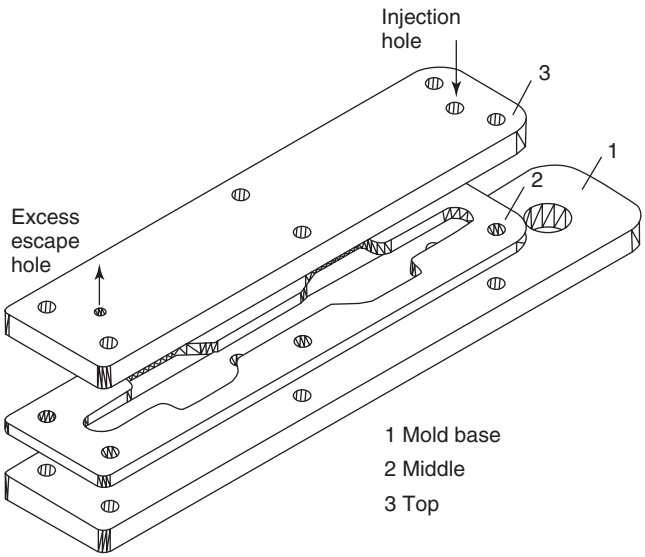


Figure 1.7 Schematic representation of the mold used to manufacture the tensile specimens.

One- or two-part paste adhesives can be injected, after mixing, using a syringe. However, there are a number of material systems that are packaged in special plastic cartridge assemblies, which store, mix, and apply multiple-component adhesives, sealants, and other materials. The plastic cartridges assure accurate proportioning of the materials since the premeasured components are stored in separate compartments within the cartridges. These cartridges can be fitted in an air gun, and the mixed material can be injected directly from the mixing nozzle into the sealed steel mold under pressure (usually 2 bars).

1.2.3
Centrifuge

For the centrifuging process, six metallic molds are placed in a radial configuration (Figure 1.8) on a centrifuge, and they are centrifuged at 1500 rpm for ~30 min. An enclosed lathe can be used as a centrifuge. It is difficult to estimate a universal required time for centrifuging for all material systems, since this would depend on a number of factors such as the void size, the viscosity of the material, the temperature of the centrifuge, the cure temperature and kinetics, and so on. Therefore, the centrifuge time needs to be evaluated by trial and error, judging from the quality of the specimens, in terms of the voids present, at the end of the process. During the centrifuging, the uncured material, under the action of the centrifugal force, moves toward the outer end of the mold and the entrapped air toward the inner end. Following the end of the centrifuging process, the molds are placed in a vertical position for the adhesive or sealant to cure. The manufacturer's common practice is followed during this phase with regard to choosing the appropriate temperature and humidity levels.

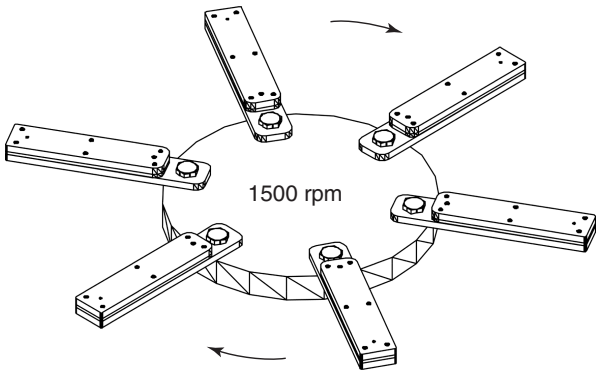


Figure 1.8 Schematic representation of the centrifuging configuration used to manufacture tensile specimens of sealants.

1.2.4
Cure

For adhesives that cure at room temperature, the mold should be placed in an air circulating oven at ambient conditions. Some control of the humidity level within the oven is desirable, as the cure of a number of adhesives can be affected by moisture in the environment. In the case that curing takes place at temperatures higher than the ambient room temperature, this should be monitored with a thermocouple placed as close as possible to the adhesive layer. One of the threaded holes on the top part of the mold can be used for this reason. Actual curing time should be measured from the moment that the adhesive reaches the curing temperature. Cooling should be slow, and the specimens should be allowed to reach ambient conditions before being removed from the mold. Most sealants cure at room temperature, so it is essential to control the humidity levels of the environment where curing takes place. Most laboratories have constant temperature and humidity levels, but using an oven at ambient temperature and humidity is recommended.

1.2.5
Final Specimen Preparation and Testing

At the end of the curing process and after demolding, the edges of the specimens are cut using a sharp knife. The resultant specimens used for tensile testing are shown in Figure 1.9. Any shape, following the principles of different international standards, can be produced by changing the middle part of the molds. The sealant specimens manufactured by this technique in Ref. [5] were 3.5 mm in thickness.

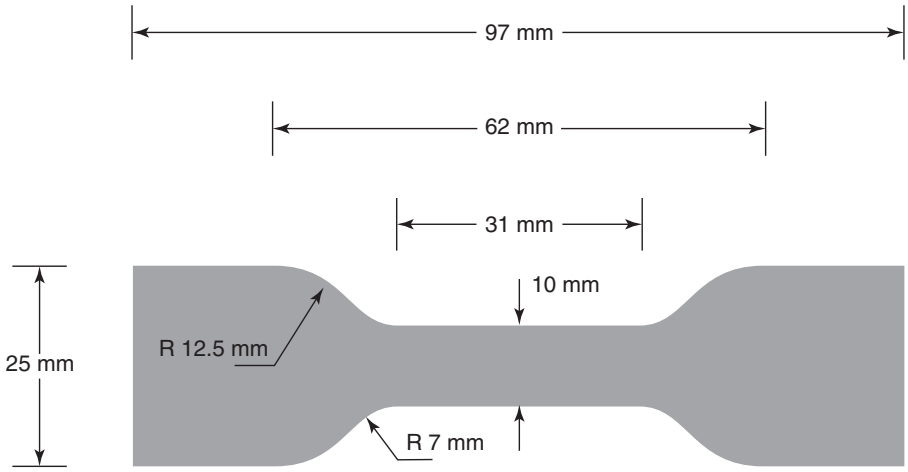


Figure 1.9 Geometry of the specimens for tensile testing of sealants based on **ASTM D412**.

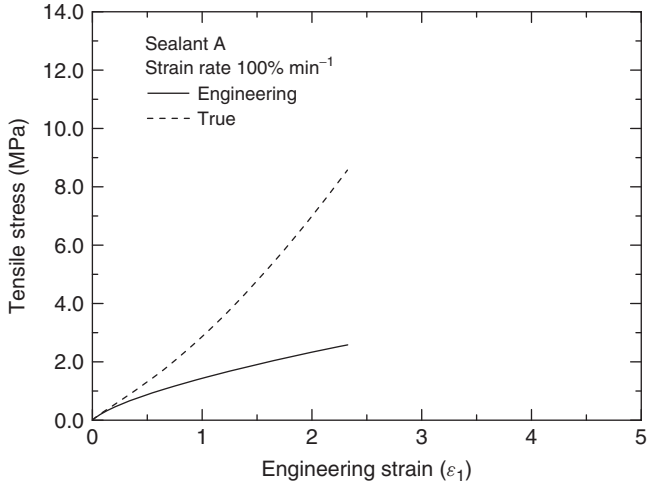


Figure 1.10 Experimental tensile stress–strain curves for a sealant material.

Dumbbell-shaped specimens can then be tested for tension following common practices (Section 2.1). In Figure 1.10, the experimental results for a rubbery sealant material tested in tension are presented. Specimens were produced by following the principles described in this Section 1.2. Experimental results are presented as the tensile stress, both engineering and true stress, as a function of the measured engineering strain. Strain in this case was measured using a noncontacting laser extensometer. Very good reproducibility of results was found between the specimens manufactured with this technique.

1.3
Preparing Bulk Specimens by Pouring

Robert D. Adams

1.3.1
Introduction

It is usually necessary to understand the mechanical and physical properties of adhesives so that they can be used successfully in adhesive joints. The preparation of these adhesives in a useful form for testing is not easy if the properties are to resemble those of the actual joints, which are commonly in a thin film between two sheets of metal or composite. Using algebraic or numerical mathematical tools (such as finite element analysis), modulus, strength, and ductility values are necessary if predictions of joint strength are to be made. These values are measured on bulk specimens of such a size that is suitable for insertion into a test machine and in which the strains or displacement can be measured. Physical constants needed for diffusion mechanisms or for measuring the glass-transition

temperature (T_g) are also usually measured on specimens whose dimensions are in the order of millimetres rather than micrometres. The need is therefore to be able to make specimens which give reliable values.

It has long been realized that the properties of bulk specimens may not be exactly the same as those in the thin film form. There are two main reasons for this. First, there is often a physicochemical action between the adherend surface and the adhesive closely adjacent thereto. The nature of any such action depends on the surface and the adhesive, but it will be more important in very thin films. Second, the chemical cure of many adhesives results in an exotherm. In thin films between metallic sheets, the heat produced by this exotherm is conducted away. However, in bulk specimens, the heat cannot be conducted away so easily and the temperature of the adhesive can rise to much higher than the nominal cure temperature.

The objective of this note is to explain how bulk specimens can be made (by pouring) and cured, bearing in mind the above cautions, for structural adhesives such as epoxies, acrylics, and so on. But experimenters should bear in mind the comments of Gillham [6] who used an impregnated braid as a torsion pendulum to study the cure of adhesives. He made it quite clear that it is not generally possible to achieve the same cure state by slow cure as by quick cure.

1.3.2
Nature of Adhesives Supplied

Adhesives come in many forms. They may be in a single part, which will be cured by heating to some temperature, or they may be in two parts, which must be mixed in agreed proportions and applied within some specified time, with or without the application of heat. Single-part adhesives can also be supplied as a thin film, often containing some supporting medium such as a woven web. If there is no supporting web, these films can be melted at an elevated temperature and poured. Adhesives are supplied in a variety of forms; they may be low-viscosity liquids or highly viscous pastes. When heated, most adhesive systems become less viscous for a time, but they cure and begin to gel, thus increasing in viscosity. However, with care and experience, it is possible to get most adhesives in a form that can be poured.

1.3.3
Mixing

One-part adhesives are mixed by the manufacturer and stored in containers until needed. These containers may be large, containing 500 kg or so of adhesive, or they may be small cartridges for manual application. Depending on how the adhesives are mixed and stored, they may contain air and other gases. In laboratory conditions, one-part adhesives may be stirred in vacuum and this can remove most of the entrapped air. However, the process is not easy or cheap.

Two-part adhesives also contain trapped air, and the separate components can also be stirred in vacuum to release all or most of this air. But two-part adhesives

need to be mixed thoroughly just before use. This mixing process can be done in vacuum, but usually it is not. It therefore needs to be done with great care to avoid introducing air, and hence voids, in the cured adhesive.

It is possible to degas a mixed two-part adhesive, but again there are problems. It is to be remembered that such adhesives begin to cure as soon as they are mixed and the viscosity increases with time. Experience shows that degassing to remove air introduced by mixing causes bubbles to form on the top surface of the adhesive. These bubbles may remain trapped in the adhesive, depending on the viscosity and surface tension of the adhesive.

1.3.4
Pouring

Pouring the liquid adhesive into a mold must be done with care to avoid trapping air in the process. Personal experience of pouring a one-part adhesive in vacuum showed that while air was not trapped, voids could be created as the poured column is unstable. The application of pressure after pouring usually solves this problem, but if a pressing sequence is not used, voids can reappear.

1.3.5
Effect of Size

It has been mentioned earlier that there are problems of exothermic reaction with thick specimens. The thicker the specimen, the more likely is overheating due to exothermic reaction likely. The magnitude of the problem depends on the thickness dimension and cure chemistry. Making specimens of the order of 10–15 mm thick is difficult. Dr. Hua Yu working in my laboratory in Bristol cured specimens of epoxy in a metal tube in an oil bath at 60 °C. The specimens were 12.7 mm in diameter and 50 mm long. Thermocouples were placed in the adhesive and oil bath. The oil bath and outer radius of the adhesive showed temperatures of 60 °C as expected. However, a thermocouple at the center of the specimen reached a temperature of 160 °C and the cured material was distinctly brown in the middle. However, the same adhesive with a nominal (oil bath) temperature of 25 °C only reached 26.14 °C inside the specimen. This example is given to show that very high temperatures can be generated if there is an unacceptable combination of geometric dimensions, cure chemistry, and initial (nominal) cure temperature.

1.3.6
Specimen Production

Having mixed and then poured the adhesive into a suitable mold, it is necessary to try and eliminate any voids. One method is to centrifuge the system so that any trapped air can be caused to migrate to one end of the mold. A suitable system for doing this is described in Section 1.2. A rotational speed of 3500 rev/min (which is achievable in a workshop lathe) for 10 min is quite sufficient, and the use of

a high-speed centrifuge is not necessary. The centrifuge technique can also be applied to the mixed adhesive before pouring into a mold. The best results can be achieved by centrifuging the adhesive and then pouring it into a closed mold, which can be subject to hydrostatic pressure. Such a mold is that described in the French standard NFT 76-142 referred to by Jeandrau [7] and described in Section 1.1.

It then remains to select a suitable cure cycle to produce a ''bulk'' specimen.

1.4 Preparing Lap Joints with Flat Adherends

Lucas F.M. da Silva

1.4.1 Introduction

Tests with thin sheet adherends, and in particular the single lap joint (SLJ) test, are very common in the industry. This is because the SLJ reproduces joints encountered in aeronautical structures, which were the pioneers of adhesive bonding technology. Several standards for preparing joints with flat adherends such as the SLJ **(ISO 4587** or **ASTM D 1002)** recommend machining the specimens from two plates bonded together (Figure 1.11). However, this technique has disadvantages such as the effect of cutting, which might introduce cracks in the adhesive, and that cutting fluids might influence the bond. It might be preferable to machine the substrates to the correct dimensions before bonding. There are several geometric parameters that are very important to control rigorously because the adhesive properties to be measured depend on them. The main geometrical aspects to control are substrate alignment, adhesive thickness, and adhesive spew fillets. Generally, molds are used for that effect.

1.4.2 Mold

There are various devices to fix the substrates in place such as springs, clamps, weights, presses, vacuum bags, autoclaves, molds, and so on. Teflon molds are ideal because they do not require a release agent. However, when high-temperature cures are required, it is better to use molds made of the same material as the substrates to reduce the residual thermal stresses. The mold presented in Figure 1.12 can keep the substrates in place and guarantees the substrates' alignment, the overlap length, and the adhesive thickness. It can produce any type of lap joint with flat adherends (SLJ, double lap joint, laminated joints, etc.). The material used to build the mold was carbon steel (e.g., 0.45% C) in the annealed condition. It is cheap, easy to machine, and guarantees good heat dissipation. This mold can be used for any substrate provided the cure is done at room temperature. When bonding aluminum specimens at high temperatures, it is recommended to use an aluminum alloy for

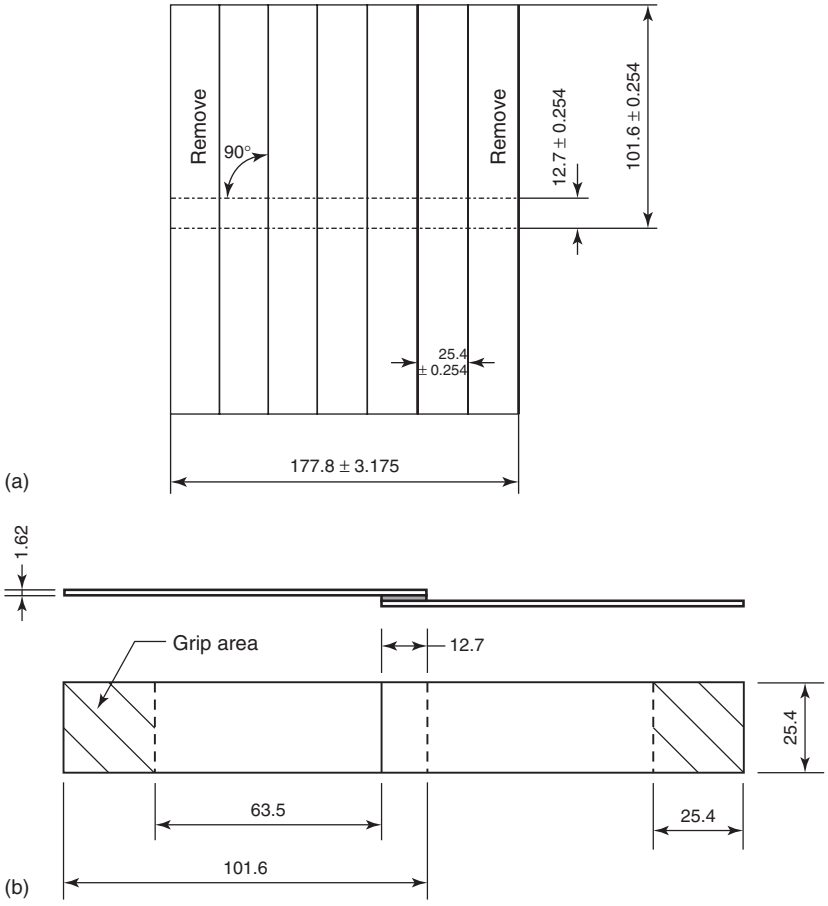


Figure 1.11 Two plates bonded together (a) for the manufacture of single lap joints and (b) according to standard **ASTM D 1002** (dimensions in mm).

the mold. Up to six specimens with a width of 25 mm can be manufactured with this mold. The overlap length can be varied through blocks located laterally.

1.4.3
Substrate Preparation and Mounting

The substrates are generally prepared to remove dust, dirt, oil, oxides, or release agents in order to improve the interfacial bonding. To decrease joint misalignment, tab ends may be used, although the effect of spacers is relatively small [8]. They can be adhesively bonded to the substrates during the adherend bonding or used only during the test by means of a pin (Figure 1.13). If high-strength materials are used, such as heat-treated steel, these cannot be gripped and therefore the adherends must be loaded through a pin. Materials that are easy to grip

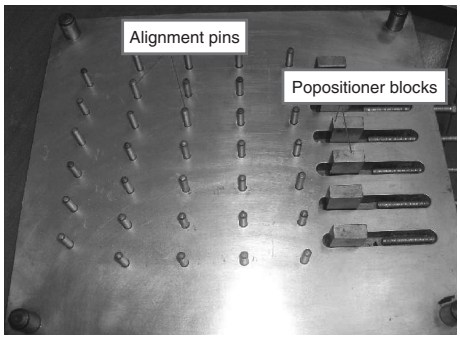


Figure 1.12 Mold for lap joints with flat adherends.

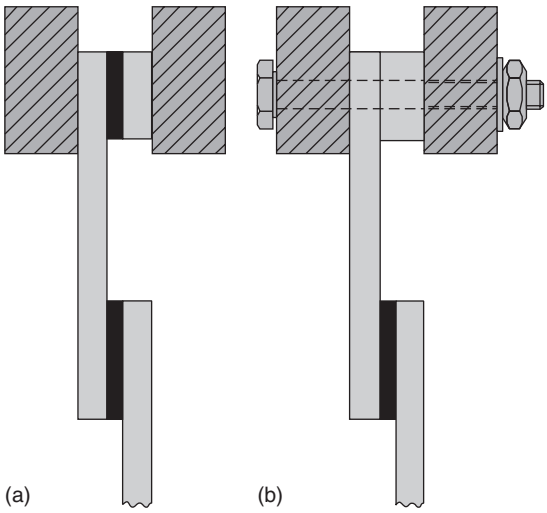


Figure 1.13 Tabs ends bonded (a) and fixed by a pin (b).

(low hardness) such as aluminum, mild steel, or composites are better tested through gripping because the pin force might be sufficient to break the adherend before the joint fails (Section 2.10).

The lower parts of the joints (substrates, tab ends, and shims) are positioned in the mold (Figure 1.14a–c). Before the adhesive application, release agent must be applied to the metallic mold and the shims. The shims have three purposes: to control the overlap, to control the adhesive thickness, and to control the adhesive fillet (Figure 1.15). There are various practical methods to control the adhesive thickness such as use of glass spheres (Figure 1.16), calibrated wires (Figure 1.17), shims, fabric, and so on. Many adhesives come with microspheres in their formulation for an easy means of bondline thickness control. The same principle is used with supported film adhesives. However, the presence of glass spheres, wires, or the fabric may interfere with the adhesive behavior and should be

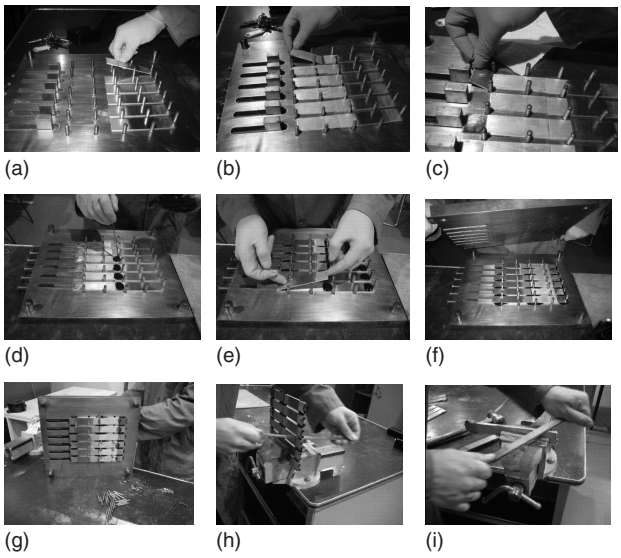


Figure 1.14 Manufacture of lap joints with flat adherends using a mold. The lower parts of the joints, substrates (a), shims (b), and tab ends (c) are positioned in the mold. Adhesive application on the area to bond (d). Substrates assembly (e). Application of the lid to the mold (f). After cure, the specimens are carefully removed from the mold (g). The separation is best done with a saw and not by breaking the adhesive (h). Adhesive excess on the sides of the joints is removed with a file (i).

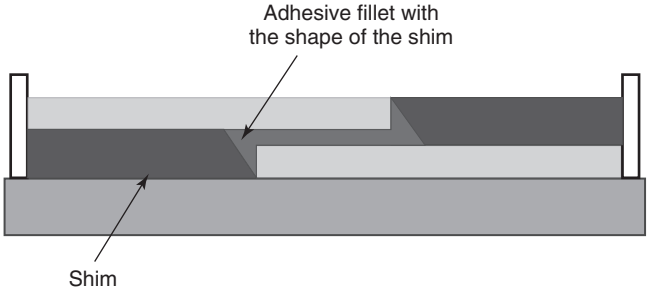


Figure 1.15 Control of adhesive thickness by the use of shims.

taken into account. The wires should be removed before joint testing and therefore may not be very practical.

1.4.4 Adhesive Application and Assembly

The adhesive application depends on the adhesive form. For liquid adhesives, no special care is required, as the adhesive easily flows through the whole area to bond. In the case of film adhesives too the adhesive application is straightforward, even

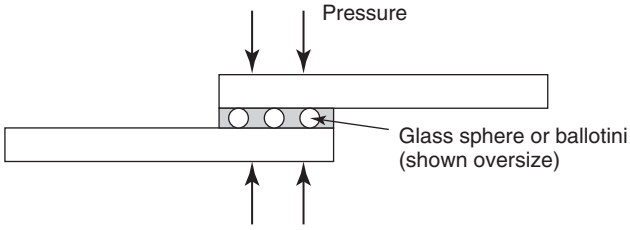


Figure 1.16 Control of adhesive thickness by the use of glass spheres.

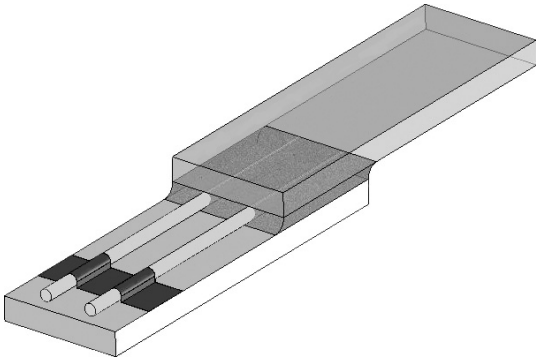


Figure 1.17 Control of adhesive thickness by the use calibrated wires.

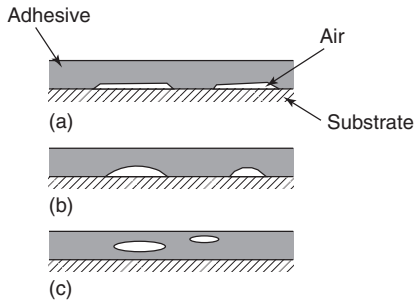


Figure 1.18 Void formation in a film adhesive during various stages. (a) Initial air entrapment, (b) partial displacement, and (c) complete displacement.

though the gaps between the film and the substrate can lead to voids in the adhesive, as shown in Figure 1.18. In that case, applying vacuum and releasing it when the adhesive is liquid can minimize voids [1, 9] (Figure 1.19). The joint is placed in an oven under vacuum without the upper adherend (point 1 in Figure 1.19). The temperature is increased to a point at which the adhesive is most fluid, so that when the vacuum is released the voids collapse to a negligible volume (point 2 in Figure 1.19). The vacuum release technique is applied without the upper adherend to facilitate the evacuation of air. After the vacuum is released, the adhesive is

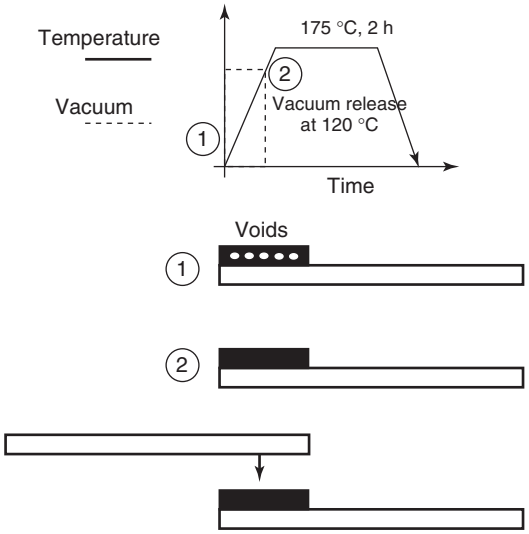


Figure 1.19 Fabrication of adhesives in the bulk (plate) form and in a joint.

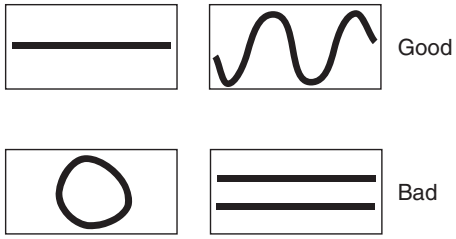


Figure 1.20 Application of paste adhesives.

largely free of voids and in its most fluid state so that it can wet the upper adherend adequately. The upper adherend is then placed on top and the joint allowed to cure. It is to be noted that the details of this method are likely to be system dependent, as elevated temperature will accelerate cure. The method may not be suitable for some chemistries.

For paste adhesives applied with a gun, the scheme described in Figure 1.20 should be followed to avoid air entrapment. In case of paste adhesives applied with a spatula, a generous amount of adhesive should be applied on the area to bond (Figure 1.14d). For two-part adhesives that are mixed manually, the adhesive flow when the top adherend is placed eliminates part of the voids. However, for best results, the mixing should be done so that there are no voids in the adhesive. Recent sophisticated machines in which the mixing is done at high speed can ensure that the adhesive is relatively void free.

The substrates should be assembled in a way to reduce the appearance of voids. There are basically two ways to put in contact two flat substrates, as shown in Figures 1.14e and 1.21. It is preferable to join the two substrates progressively by

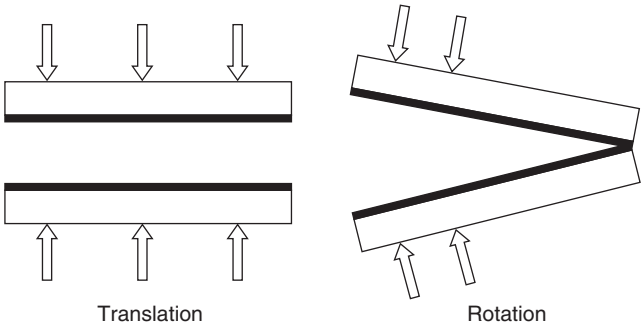


Figure 1.21 Substrates assembly by translation and by rotation. (Source: Adapted from [10].)

rotation as it leads to fewer voids. After complete assembly, the lid is applied to the mold (Figure 1.14f). In the case of thick bondlines with liquid adhesives, special care must be taken to avoid the adhesive to spread out of the overlap. In that case, the overlap area must be sealed so that the adhesive stays in place.

1.4.5 Cure

To guarantee that there is a good contact between all parts of the mold, it is better to apply, in addition to the lid, a weight on the mold or a pressure with a press. In case the adhesive cures with temperature, the hot press is the most practical equipment to apply temperature in a short time. However, it is advised to use a thermocouple close to the adhesive and to count the cure time from the moment the adhesive reaches the cure temperature.

After the adhesive cure, the cooling rate should be slow to guarantee a uniform temperature in the mold and reduce residual stresses. Also, the specimens should not be removed before the mold has reached room temperature; otherwise, the residual stresses may reduce the joint strength.

1.4.6 Specimen Cleaning

After cure, the specimens are carefully removed from the mold (Figure 1.14g). Owing to an excess of adhesive, the joints might be joined to one another in places. The separation is best done with a saw and not by breaking the adhesive, which might introduce cracks (Figure 1.14h). The packing shims should be carefully removed to avoid breaking the joint. The adhesive excess on the sides of the joints is removed with a file (Figure 1.14i). The specimens should be conditioned under controlled temperature and humidity until testing because these factors may influence the mechanical properties of the adhesive.

1.5
Simple Methods for the Preparation of Single Lap Joints Specimens

Edoardo Nicoli

1.5.1
Introduction

The single lap joint (SLJ) specimens have traditionally been used for characterizing adhesively bonded joint systems. This kind of specimen is still among the most widely used for gathering important mechanical information of adhesively bonded systems, namely, the shear strength. Methods for evaluating these measures are described, for example, in the **ASTM D 1002** [11] for SLJs (for calculating the apparent shear strength). SLJ specimens are commonly used because they are rather simple to construct and resemble the geometry of many practical applications. Nevertheless, the construction and the testing of this geometry can present some problems. The correct alignment of the specimens and the control of the bondline thickness can be issues for producing quality specimens. These technical aspects, if not properly considered, can lead engineers and researchers to strive with specimens' preparation and test setup for tests that are standardized and, therefore, are expected to be straightforward.

In this section, a fixture for overcoming the problems of specimen alignment and bondline control in SLJs is presented. This simple solution was implemented by the author during a specific project that required the production of a large number of quality SLJs [12]. The fixture is now used as a laboratory tool for the quick and accurate fabrication of specimens. Other techniques to produce SLJs are presented in Section 1.4.

1.5.2
Single Lap Joint (SLJ) Specimens

Although different lap specimen configurations can be used for evaluating the mechanical properties of bonded joints, the most common geometry is the SLJ configuration recommended in **ASTM D1002**. This standard describes how the apparent shear stress can be measured in bonded metal specimens. The specimens are two metal strips of dimensions 101.6 × 25.4 × 1.6 mm (4 × 1 × 1/16 in.) that are bonded together along an overlap of 12.7 mm (1/2 in.) as shown in Figure 1.22. The specimens are then tested in a traditional load frame and the value of the ultimate applied load at failure, divided by the extension of the bonded area, gives the apparent shear strength of the joint.

Important factors for preparation of specimens, such as bondline thickness, surface preparation of the adherends, and adhesive curing procedure are not described in the **ASTM D 1002** standard, but usually indicated in the technical documentation of the adhesive product. Nevertheless, adhesive layer thickness, surface preparation, and curing procedure influence the shear strength of the specimens and should be controlled when preparing specimens.

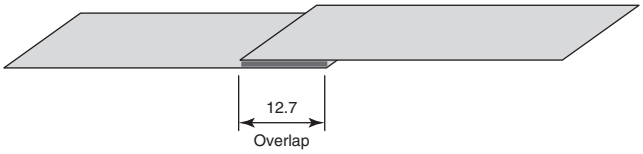


Figure 1.22 Schematic view of single lap joint (SLJ) specimen.

1.5.3
Traditional Methods for SLJ Bonding

The SLJ specimens are often prepared by clamping together the two metal parts after the adhesive application and keeping this configuration during the curing. The clamping is traditionally done in a number of different ways such as using binder clips and C-clamps. There are four major problems that can arise when using these clamps.

1) Guaranteeing the initial alignment of the metal parts is not trivial; the use of some reference blocks held against the adherends can be beneficial, although attention has to be given to not bonding these blocks to the specimen.
2) Maintaining the alignment of the metal part during the cure is not easy, especially if the specimens have to be moved to an oven for exposing them to the temperature required for the proper curing.
3) Controlling the thickness of the bondline is almost impossible, unless shim strips or filler beads are placed inside the bonded area.
4) Preparing a large number of specimens paying attention to correct alignment can be quite time consuming, thus the amount of specimens that can be prepared can be limited by the handling time that the adhesive allows.

1.5.4
The Idea for a New Fixture for SLJ Bonding

The need for developing a new fixture for constructing SLJ arose with a project that compared seven different adhesives in SLJs, where an electrical insulator layer also had to be embedded within the adhesive layer [12]. This activity required testing of a large number of SLJ specimens. The crucial aspect was being able to construct specimens with consistent geometrical characteristics, limiting the specimen to specimen variability, in an efficient and non-time-consuming way. Because of the importance of preparing specimens with good alignment and a consistent bondline thickness, the bonding fixture was developed as the initial step of the project. The goals for this fixture are as follows:

1) guaranteeing proper alignment of the SLJ specimens;
2) controlling the bondline thickness of the SLJ specimens;
3) giving access to the bond area allowing one to remove the excesses of adhesive spew;
4) facilitating the preparation of considerable amounts of specimens.

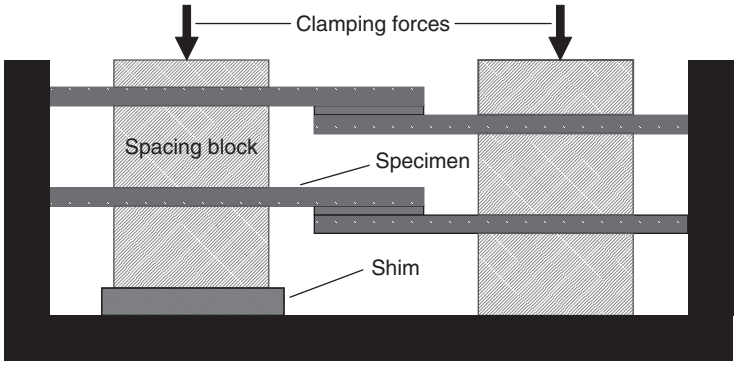


Figure 1.23 Schematic view of the jig and specimens.

The idea for fixture is schematically shown in Figure 1.23. The idea was to place all the specimens in a jig, separated by spacers, and to control the bondline thickness with a changeable shim that imposes an offset to the parts to be bonded. The shim thickness was equal to the sum of thicknesses of one adherend and the desired bondline. The bondline thickness of all the specimens was nominally controlled by this single shim.

1.5.5 The Fixture

The fixture that was developed is an aluminum-made jig, and in the configuration opened before the specimen's placement, is shown in Figure 1.24.

The fixture consists of 50×19 mm ($2 \times 3/4$ in.) aluminum bars connected together. Four of these bars form the frame of the jig, and the other four are lateral constraints that control the proper alignment of the SLJs. Two of the lateral bars are removed during the specimen's positioning and then placed once the specimens are placed inside the jig. Once the adhesive is distributed on the metal parts to be bonded in the SLJ in correspondence to the area of the overlap, the parts are placed in the jig and aligned as illustrated in Figure 1.25, with aluminum blocks used as spacers between the different specimens. The dimensions of the jig and the spacers allow for bonding 10 specimens at a time, but a similar design can be implemented for a different number of specimens.

Once all the specimens are positioned inside the jig, the other two lateral bars of the jig are put in place and the specimens are compressed with the two screws at the top of the jig. As shown in Figure 1.26, the specimens are positioned inside the jig without any residual degree of freedom. The two screws on the top of the fixture are tightened with an Allen wrench after the placement of the specimens and the lateral bars. All the parts of the fixture that are in contact with the specimens and that can possibly get in contact with adhesive are covered with tape for facilitating cleaning of the fixture after use and avoiding unwanted bonding of specimens with the fixture. Little changes in the aluminum bars of the jig would allow the

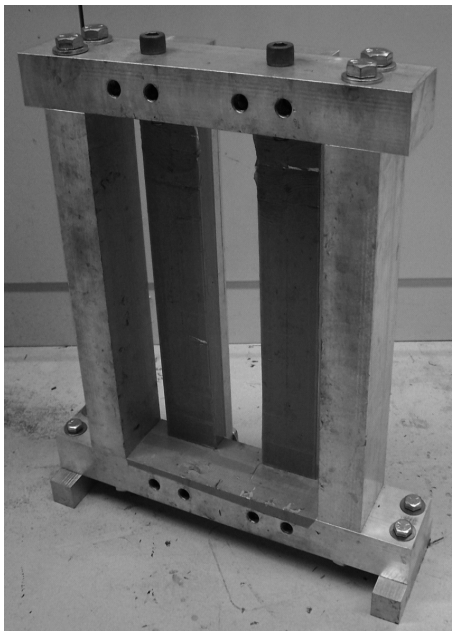


Figure 1.24 SLJ bonding fixture with two of the later bars removed.

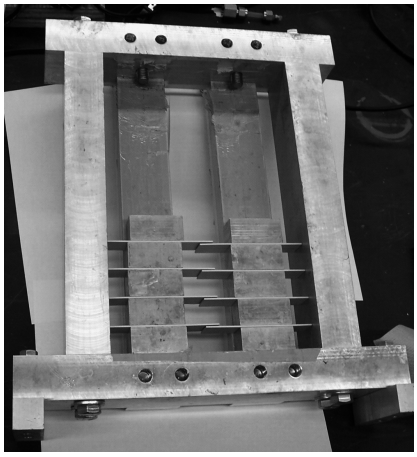


Figure 1.25 SLJ bonding fixture during specimens' loading.

construction of SLJ with geometries different from that designated by **ASTM D 1002**, if needed. A positive aspect of this configuration is that once the jig is closed, it can be moved with all the specimens without issues related to their alignment. The dimensions of the jig allow it to fit inside most of the industrial ovens used for curing.

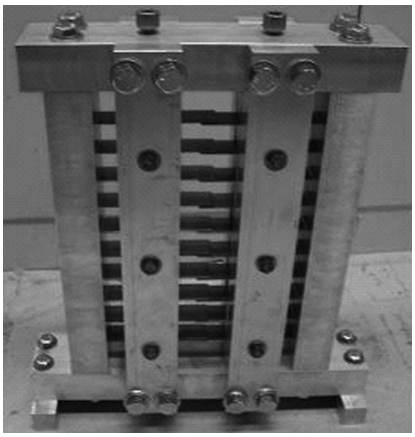


Figure 1.26 SLJ bonding fixture after specimens' loading.

1.6
Preparing Thick Adherend Shear Test Specimens

Lucas F.M. da Silva

1.6.1
Introduction

The thick adherend shear test (TAST) is one of the most popular types of failure strength test because it is easy to make and test the specimens. The conventional single lap shear (SLS) joint, which is mostly used for comparing and quality control of adhesives, puts the adhesive in a complicated state of stress (Section 2.10). Therefore, it is not suitable for the determination of the true adhesive properties. When using stiff and thick metallic adherends, the adhesive is in a state of essentially uniform shear over most of the overlap and peel stresses are reduced. Two forms of the TAST are used, as developed by Krieger [13] **(ASTM D3983)**, in the United States, and Althof and Neumann [14] **(ISO 11003-2)**, in Europe. The main difference between the two tests is the size of the specimen: the Althof specimen is half the size of Krieger's. **ISO 11003-2** recommends machining the specimens from two plates bonded together, as shown in Figure 1.27. However, this technique has several disadvantages. The machining (cut into bars and then into transverse slots) needs to be done without coolant, as it may react with the adhesive or damage the interface and the increase in temperature may affect the adhesive properties. Cutting in the highly stressed region at the end of the adhesive layer may introduce cracks in the adhesive. Cutting the slot is difficult to control and may affect the load transfer. The bonded plates may have an irregular shape or surface, leading to irregular adhesive thicknesses. Last, the specimens produced by this method cannot be reused, which may be an important factor in terms of cost. Adams and coworkers [15] have proposed that a better solution is to machine the adherends to the correct dimensions before bonding with the geometry shown in Figure 1.28.

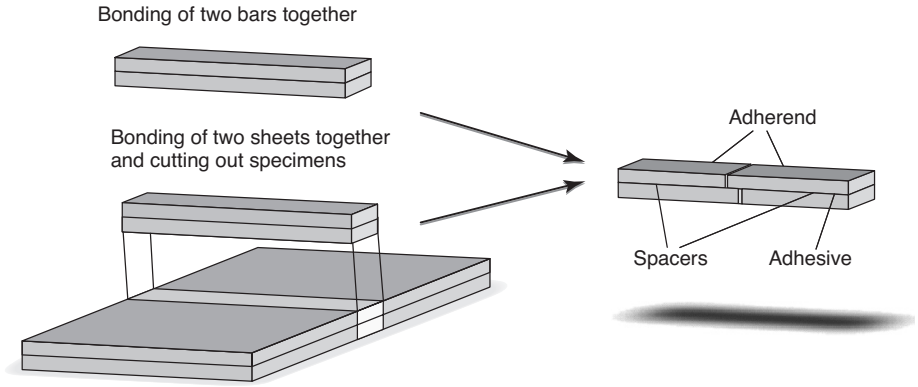


Figure 1.27 **ISO 11003-2**-recommended specimen manufacture.

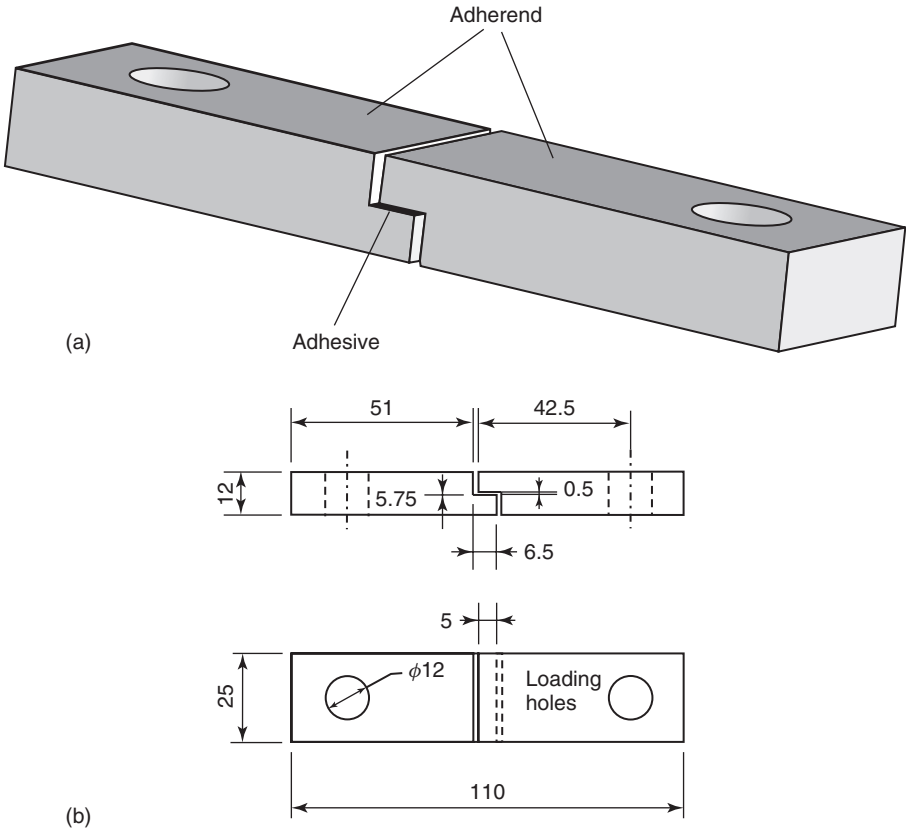


Figure 1.28 Thick adherend shear test (TAST) geometry (a) and dimensions in millimetres (b) (**ISO 11003-2**).

The bending stiffness is higher than when the joint is composed of two bonded bars and therefore reduces the peel stresses. This technique is described in the following discussion.

1.6.2 Mold

A specially built jig was designed for aligning and holding the specimens (Figure 1.29). To control the overlap and fillet, steel shims are inserted into

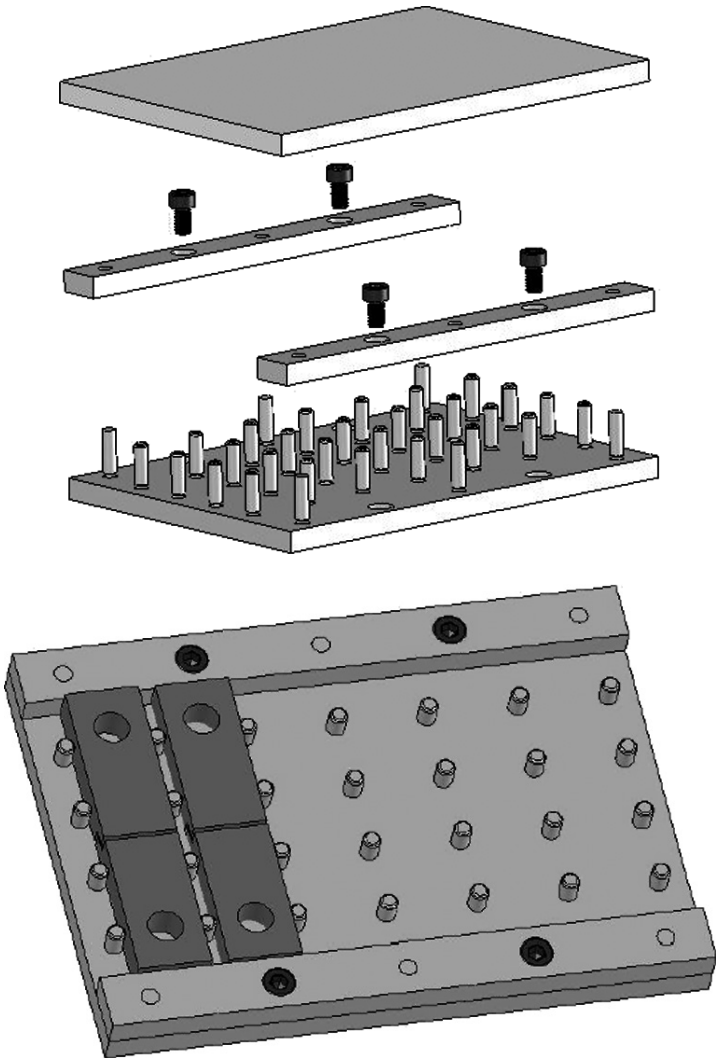


Figure 1.29 Mold for producing TAST specimens.

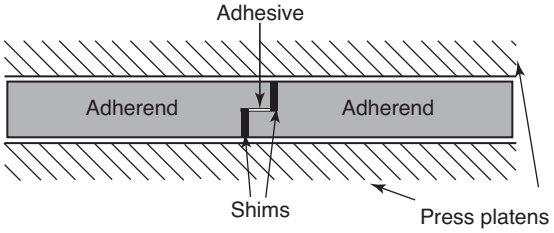


Figure 1.30 Shims used to control the overlap and the adhesive fillet.

the gaps once the adherends have been brought together (Figure 1.30). The shim can have a 45° chamfer, which is a good way to reduce the stress concentration in the adhesive and to avoid premature failures (Section 2.8). The material used to build the mold was carbon steel (e.g., 0.45% C) in the annealed condition. It is cheap, easy to machine, and guarantees good heat conduction. Up to six specimens with a width of 25 mm can be manufactured with this mold.

1.6.3 Substrate Preparation

The substrates are always prepared to remove dust, dirt, oil, oxides, or release agents in order to improve the interfacial bonding. For most adhesives, the surface preparation consists of degreasing with acetone or another solvent, shot blasting, and degreasing again. If the failure is not cohesive in the adhesive, a chemical treatment should be used. It is preferable to use steel instead of aluminum because the higher stiffness of the steel minimizes the adherend deformation and rotation, which creates a more uniform shear stress state in the adhesive. Also, since the mold described above is made of steel, it does not introduce residual thermal stresses in the adhesive because of differences in thermal expansion between the mold and the specimens when high cure temperatures are required. Care should be taken in drilling the holes for specimen loading (Section 2.8) because misalignments lead to unwanted rotation and bending. The lower parts of the joints (substrates and shims) are positioned in the mold (Figure 1.31a). Before adhesive application, release agent must be applied to the metallic mold and the shims.

1.6.4 Adhesive Application and Assembly

Adhesive application (Figure 1.31b) is similar to that used for joints with flat adherends (Section 1.4). In the case of low-viscosity adhesives, the adhesive may flow out of the bonded area. In that case, the joint sides should be sealed so that the adhesive stays in place. After adhesive application and placement of the top substrate, a lid is applied to ensure that the adherends are parallel (and therefore control the bondline) and to maintain the joint in contact (Figure 1.31c).

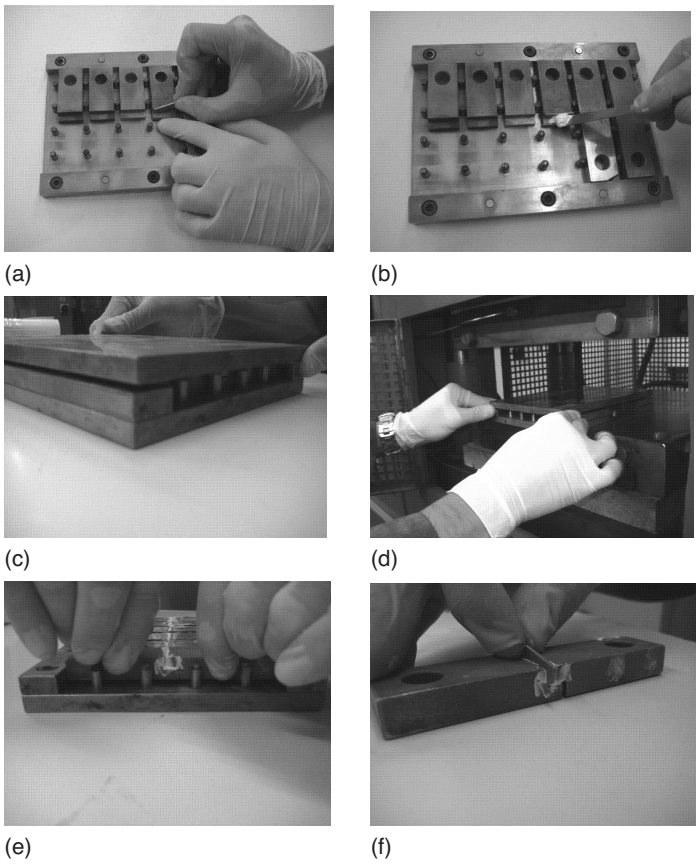


Figure 1.31 (a–f) Manufacture of TAST specimens using a mold.

1.6.5 Cure

As in the case of joints with flat adherends (Section 1.4), cure is best done in a heated press, which ensures that there is a good contact between all parts of the mold and that the temperature distribution is uniform (Figure 1.31d).

1.6.6 Specimen Cleaning

After cure, the specimens should be carefully removed from the mold (Figure 1.31e). Because an excess of adhesive is used, the joints might be bonded. Separation is best done with a saw and not by breaking the adhesive, which might introduce cracks. The packing shims should be carefully removed to avoid breaking the joint (Figure 1.31f). The adhesive excess on the sides of the joints

is removed with a file. The joint geometry should be carefully checked before testing. This is best done with a ruler coupled to a microscope. Alternatively, the adhesive thickness can be measured by subtracting the total thickness of the joint from the adherend thickness as measured before bonding. The specimens should be conditioned under controlled temperature and humidity because these factors influence the mechanical properties of the adhesive.

1.7 Modified Thick Adherend Shear Test

Jean-Yves Cognard and Romain Créac'hcadec

The TAST [16] is a logical extension of the SLS test method, which is widely used to evaluate adhesive systems. It was developed in Germany and the United States in the 1970s. The test is detailed in the standards **ISO 11003-2** and **ASTM D3983**, and in the manufacture of quality specimens – TAST. Using thick adherends, typically 10 mm thick (or 6 mm thick for the Althof test [16]), and a short overlap enables the peel stresses, which complicate the SLS, to be significantly reduced. A suitable extensometer allows the full shear stress–shear strain curve to be obtained. This setup is often used to analyze the mechanical behavior of adhesives in bonded assemblies under various conditions [17–19]. A precise analysis of the experimental results can lead to observation of crack propagations close to the substrate–adhesive interfaces [19, 20] and can lead to incorrect analysis of the behavior of the adhesive. The experimental and numerical analyses of the mechanical behavior of such bonded joints are made particularly difficult by stress concentrations because of edge effects [20–23]. Therefore, understanding how stress is distributed in the adhesive can lead to improvements in adhesively bonded assemblies. Particularly, since it can be difficult to take the effects of stress concentrations into account when analyzing experimental results, it is useful to design experimental fixtures that strongly limit edge effects in order to obtain reliable data [24, 25].

This section presents a modified TAST fixture [26], which gives a more homogeneous adhesive stress state in the adhesive joint and strongly limits edge effects in order to obtain reliable experimental results.

1.7.1 Specimen Geometry

The modified TAST fixture and a TAST specimen are presented in Figure 1.32. Figure 1.33 presents the geometry and the fixing system of the bonded specimen. The first idea was to use small bonded samples ((1) in Figure 1.33), which represent the useful part of the TAST specimen (a parallelepiped of height ∼20 mm with an adhesive area $S_c = 9.53\ \text{mm} \times 25.4\ \text{mm}$). The second point was to use a rigid support (2) in order to limit the bending of the device, which increases the peel stress in the joint. A fastening device (3) is used to fix the bonded sample. A special system was used (noted (4) in Figure 1.33) to obtain precise positioning of

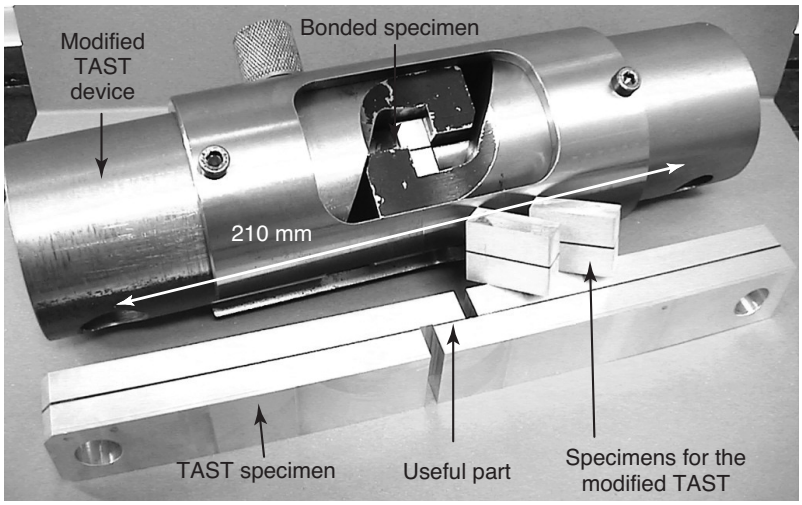


Figure 1.32 Comparison of the TAST and the modified TAST.

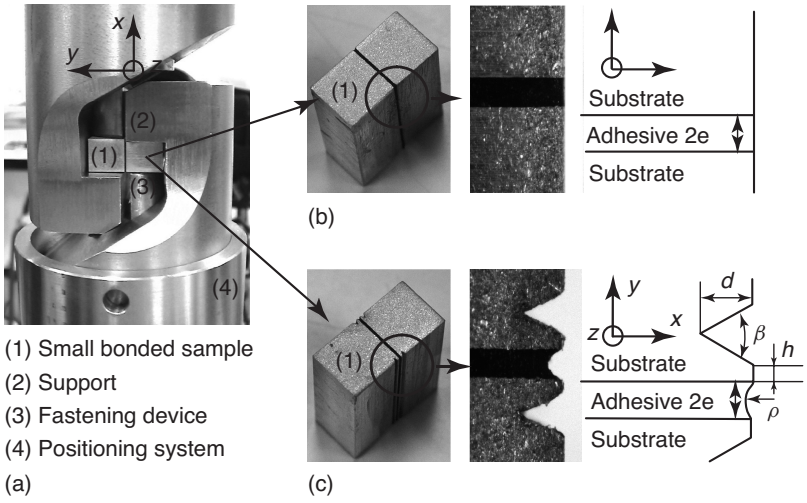


Figure 1.33 Geometry and fixing system of the bonded specimen. (a) Fixing system, (b) specimen without beak, and (c) use of beak and cleaned edges.

the specimen in the support. Moreover, the part is used to limit the preloading of the specimens during the mounting of the specimen in the tensile testing machine (Figure 1.34a). Before performing a mechanical test of the joint, part 4 is descended to prevent friction effect (Figure 1.34b). In order to prevent parasitic loadings, connections to the tensile testing machine, allowing rotations, were used (Figure 1.34): a universal joint ensures radial positioning of the device and the axial load is transmitted by a pin in order to control the geometrical constraints.

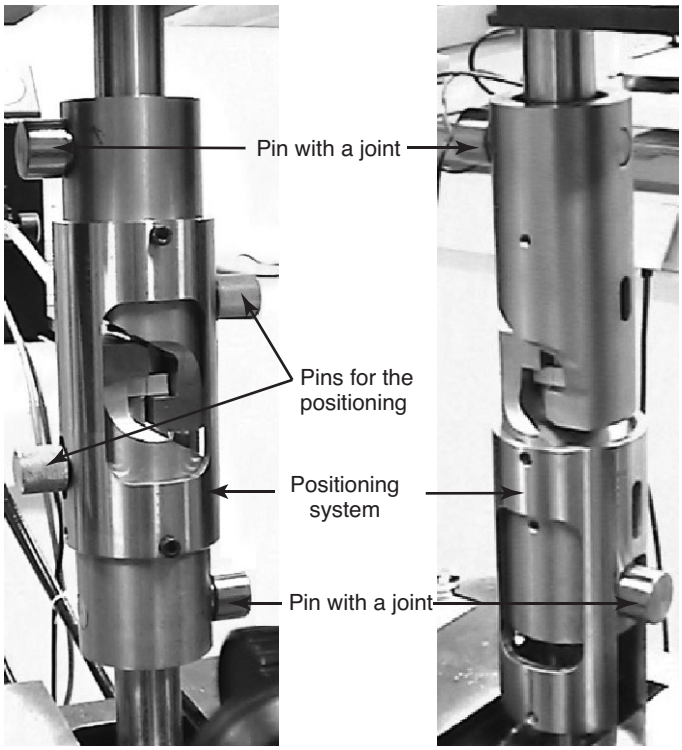


Figure 1.34 The modified TAST. (a) Assembly configuration and (b) test configuration.

1.7.2 Bonded Specimen Geometry

The design of this test is based on experience acquired while improving the Arcan assembly design [25]. Precise finite element computations were also used to analyze stress distributions in the adhesive joint, taking into account the influence of the fixing system in order to optimize the design of this test [26]. This section describes the use of 2D models to analyze the stress distribution throughout the thickness of the adhesive and to analyze the edge effects in the modified TAST. Various simulations have shown that good numerical results are obtained using meshes with 20 linear rectangular elements for a 0.1 mm thickness of adhesive [26]. For these computations, the average shear stress in the joint was normalized to 1 MPa in order to make analyses easier.

The modified TAST specimen can be modeled under 2D assumptions in an initial study. In order to reduce the computing cost, only half of the specimen was modeled by applying adequate boundary conditions [26]. For these models, the loading results are presented for aluminum substrates (Young's modulus:

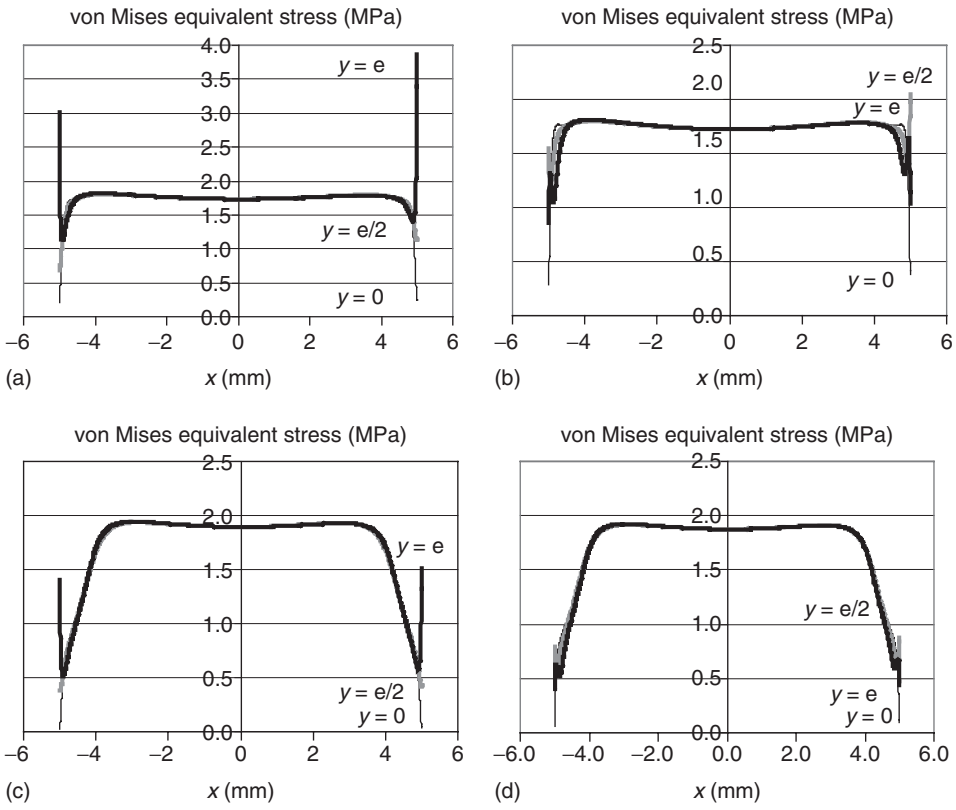


Figure 1.35 von Mises equivalent stress with respect to the overlap length for 2D computations for an average shear stress of 1 MPa. (a) Straight substrates and straight joint. (b) Straight substrates and cleaned joint. (c) Substrates with beaks and straight joint. (d) Substrates with beaks and cleaned joint.

$E_a = 75$ GPa, Poisson's ratio: $\nu_a = 0.3$). The material parameters for the studied adhesive were $E_j = 2.2$ GPa, $\nu_j = 0.3$.

Figure 1.35 presents the evolution of the von Mises equivalent stress in the adhesive for a joint thickness of $2e = 0.4$ mm with respect to the overlap length (the center of the joint is associated with $x = 0$). Four geometries were used: straight substrates (Figure 1.33b), substrates with thin beaks ($h = 0.1$ mm, $d = 0.5$ mm, and $\beta = 60^\circ$, Figure 1.33c), straight free edges of the adhesive, and cleaned edges of the adhesive-free edges ($\rho = \infty$ and $\rho = 1.5$e, Figure 1.33c). The von Mises equivalent stress, which takes into account the different stress components, is used to qualify the edge effects. The different curves are associated with a position y in the adhesive; $y = 0$ represents the average plane of the adhesive and $y = e$ is close to the adhesive–substrate interface. It can be noted that for straight substrates and straight ends of the joint (Figure 1.35a) quite large stress concentrations can be observed closed the free edges of the joint. Such results also exist for the TAST,

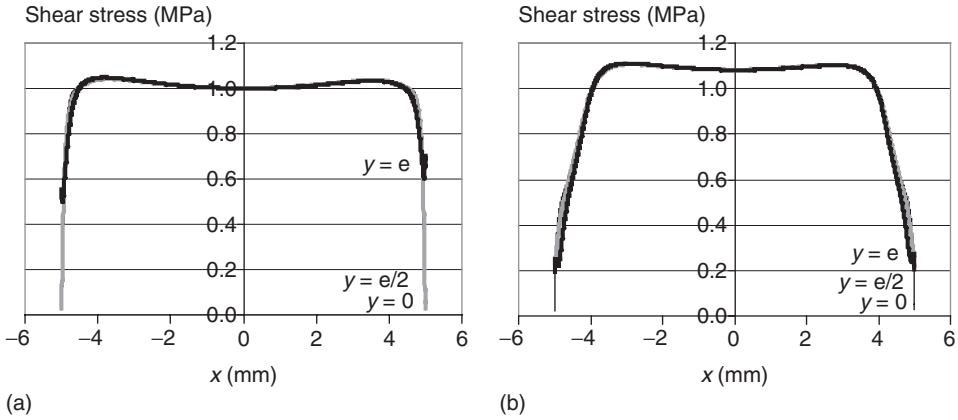


Figure 1.36 Shear stress with respect to the overlap length for 2D computations for an average shear stress of 1 MPa. (a) Straight substrates and straight joint. (b) Substrates with beaks and cleaned joint.

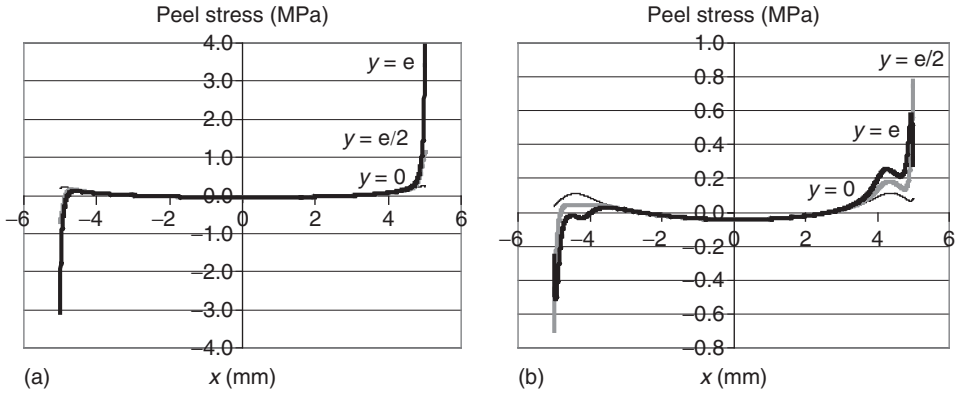


Figure 1.37 Peel stress with respect to the overlap length for 2D computations for an average shear stress of 1 MPa. (a) Straight substrates and straight joint. (b) Substrates with beaks and cleaned joint.

with a little larger value as the rigidities of the supports are different [20, 26]. The use of substrates with thin beaks and cleaning of the free edges of the joint can strongly limit the stress state close to the free edges of the joint (Figure 1.35d). Moreover, it can be noted that for such geometries, the maximum value of the stress is obtained within the joint and not at the substrate–adhesive interface. Figure 1.35b,c underlines the effect of the geometry of the adhesive-free edges on the influence of edge effects.

Figures 1.36 and 1.37 present the evolution of the shear and peel stresses for straight joints and for substrates with beaks and cleaned joint. It can be noted that the geometry of the bonded joint, close to the adhesive-free edges, has only a little

influence on the shear stress distribution in the adhesive and that the edge effects are mainly associated with the peel stress.

Assuming linear behavior, the maximum value of the shear stress is obtained starting from finite element computations, and thanks to the average stress based on the load measured during the experimental test (F_T) and the section of the adhesive plane (S_c):

$$\tau_{xy\,\text{maxi}} = 1.11\tau_{xy\,\text{average}} \text{ with } \tau_{xy\,\text{average}} = F_T/S_c$$

The large stress gradients, observed for straight joints, can be difficult to model when the nonlinear behavior of the adhesive is taken into account in a finite element computation, as crack initiation and crack propagation models have to be used. Thus, in order to analyze the nonlinear behavior of an adhesive in an assembly, it seems interesting to use tests that strongly limit the influence of edge effects.

1.7.3 Machining of the Samples with Beaks

It has been shown numerically that small bonded samples with beaks on only two sides can be used [26], but it is difficult to machine these geometries starting from bonded plates. However, the shapes can be obtained quite easily starting from bonded bars in which beaks have been previously machined (Figure 1.38a). Precise positioning of the two bars during bonding is not difficult (Figure 1.38b). Moreover, cleaning of the adhesive, on the free edges, can be quite easily done before the curing process. Finally, the small samples can be cut out of bonded bars using a water jet cutting system in order to limit the influence of machining on the adhesive behavior (Figure 1.38c).

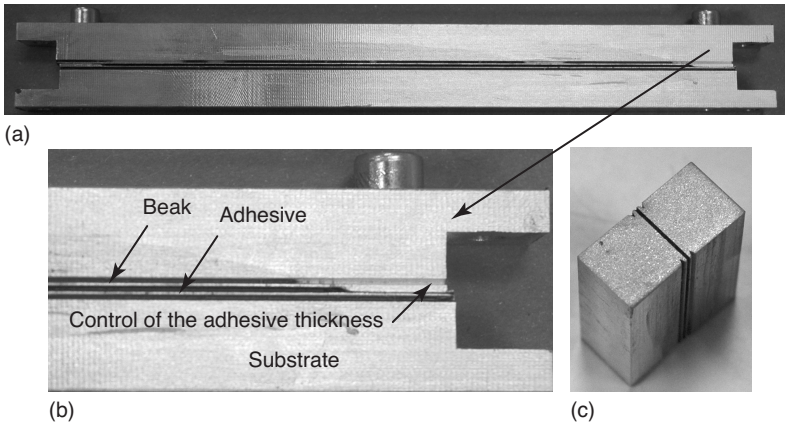


Figure 1.38 Manufacturing of small bonded samples with beaks on only two sides. (a) Bonding of bars with beaks. (b) Bonding of bars (close-up view). (c) Bonded specimen.

1.8
Preparing Butt Joints

Lucas F.M. da Silva, Stefanos Giannis, and Robert D. Adams

1.8.1
Introduction

The butt joint with solid substrates can be used to measure the tensile properties (''poker chip'' test) or the shear properties of the adhesive through a torsion test. There are two ASTM standards for the tensile test. The first, **ASTM D 897**, uses short circular specimens made of metal or wood, and the second **(ASTM D 2095)** is more general and includes round and square geometries. Other standards include **ISO 6922**, **BS EN 26922**, and **BS 5350 – Part C3**. When testing under torsion, the specimen is ideally free of stress concentrations, which enables larger strains to failure than other types of tests where stress concentrations exist. Also, the adhesive displacement generated is higher, which gives a higher accuracy for the strain than other joint methods. A mold is presented in **ASTM D 2095** for controlling the adherend alignment and adhesive thickness. A procedure to manufacture joints based on that mold is presented here. An additional manufacturing technique developed by Adams and coworkers [15] is presented in the Section 1.8.7.

1.8.2
Mold

The mold presented in Figure 1.39 can keep the substrates in place, controlling both substrate alignment and adhesive layer thickness. It is designed to produce specimens of the geometry shown in Figure 1.40. The material used to build

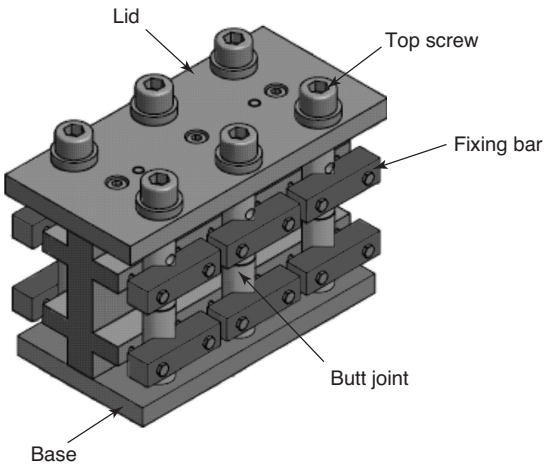


Figure 1.39 Mold for manufacturing butt joints (**ASTM D 2095**).

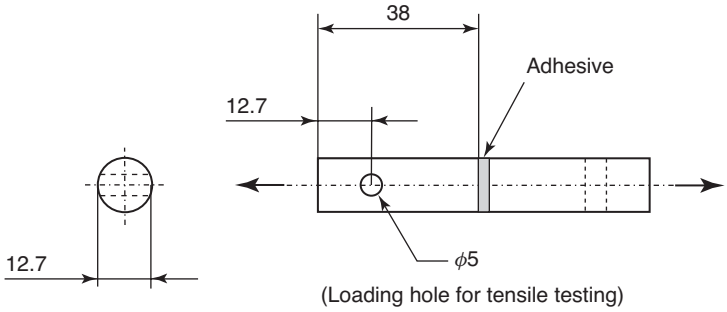


Figure 1.40 Butt joint geometry (dimensions in millimetres) (**ASTM D 2095**). For torsion testing, the loading hole is not necessary.

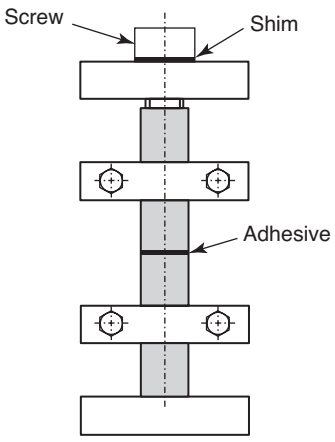


Figure 1.41 Control of adhesive thickness by the use of shims.

the mold is carbon steel (e.g., 0.45% C) in the annealed condition. It is cheap, easy to machine, and offers good heat dissipation. This mold can be used for any substrate, provided the curing is done at room temperature. When bonding substrates different from steel, residual thermal stresses may arise when high cure temperatures are used. Up to six specimens can be manufactured with this mold. The adhesive thickness can be adjusted with shims located at the top (Figure 1.41).

1.8.3 Substrate Preparation

The substrates are always prepared to remove dust, dirt, oil, oxides, or release agents in order to improve the interfacial bonding. For most adhesives, surface preparation consists of degreasing with acetone or another solvent, shot blasting, and degreasing again with a solvent. If the failure is not cohesive in the adhesive, chemical treatment may be used. It is preferable to use steel instead of aluminum because the higher stiffness of steel minimizes adherend deformation. Also, since the mold described above is made of steel, when high cure temperatures are

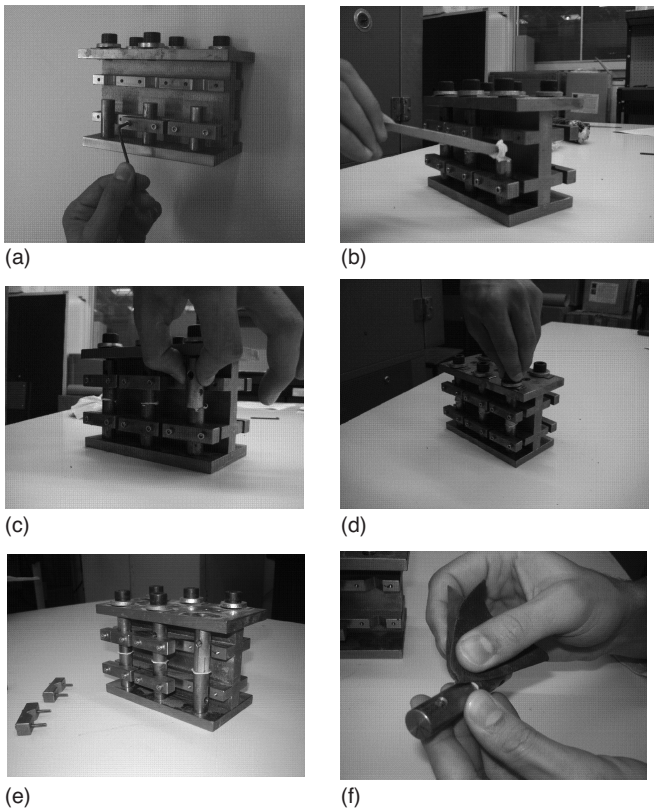


Figure 1.42 (a–f) Manufacture of butt joints (ASTM D 2095).

required residual thermal stresses is not introduced in the adhesive because of differences in thermal expansion between the mold and the specimen. In case the butt joint is used for tensile testing, care should be taken in the drilling of holes for specimen loading because misalignments lead to unwanted rotation and bending. The lower substrates are positioned in the mold (Figure 1.42a) fixed by the support bar. The screws of the top bar should be tightened so that the adherend does not move, and the adherend can be displaced when the top screw is tightened down. Before adhesive application, a release agent should be applied to the metallic mold and the support bars to facilitate removal after cure.

1.8.4
Adhesive Application and Assembly

Application of the adhesive depends on the adhesive form. For liquid adhesives, thin bondlines should be used to avoid spreading out of the adhesive. In the case of film adhesives too the adhesive application is straightforward, even though gaps between the film and substrates can lead to voids in the adhesive (Section 1.4). For

two-part adhesives that are mixed manually, adhesive flow when the two adherends are pressed against each other eliminates part of the voids. However, for best results, mixing should be done so that there are no voids in the adhesive. Recent sophisticated machines in which the mixing is done at high speed can ensure that the adhesive is relatively void free.

The adhesive is applied in the bottom substrates (Figure 1.42b). The upper substrates are then positioned and fixed lightly by the support bar (Figure 1.42c). The top screw is then tightened down until it reaches the shim for bondline thickness control (Figure 1.42d).

1.8.5 Cure

In case the adhesive cures above room temperature, the mold should be placed in an oven with good air circulation. It is advised to use a thermocouple close to the adhesive and to count the cure time from the moment the adhesive reaches the cure temperature.

After the adhesive cures, the cooling rate should be slow to minimize mold temperature nonuniformity and reduce residual stresses. Also, the specimens should not be removed before the mold has reached room temperature; otherwise, the residual stresses can reduce the joint strength considerably.

1.8.6 Specimen Cleaning

After curing, the specimens are carefully removed from the mold (Figure 1.42e). The adhesive excess on the sides of the joints can be removed with a file or other technique (Figure 1.42f). The specimens should be conditioned under controlled temperature and humidity because these factors influence the mechanical properties of some adhesive bonds.

1.8.7 Alternative Manufacturing Method

Another type of butt joint specimen is commonly used for testing adhesives or sealants in torsion. To enable gripping the specimen on a test machine that is capable of applying an angular displacement, aluminum or steel adherends with square ends might need to be used. The geometry of such adherends is shown in Figure 1.43. Preparation of the adherends before bonding is the same as with those used for butt joints intended for tensile loading.

Generally, the manufacturing of this type of butt joints involves application of the adhesive/sealant on both surfaces of the adherends and the use of a horizontal V-block-type jig, which holds the adherends together and determines the bondline thickness. This jig can be used as an alternative to the jig described earlier. It is necessary to produce good-quality joints in order to measure as

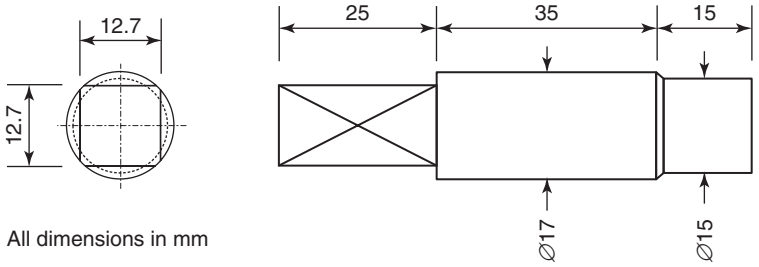


Figure 1.43 Geometry of the adherends used for butt joints (dimensions in mm).

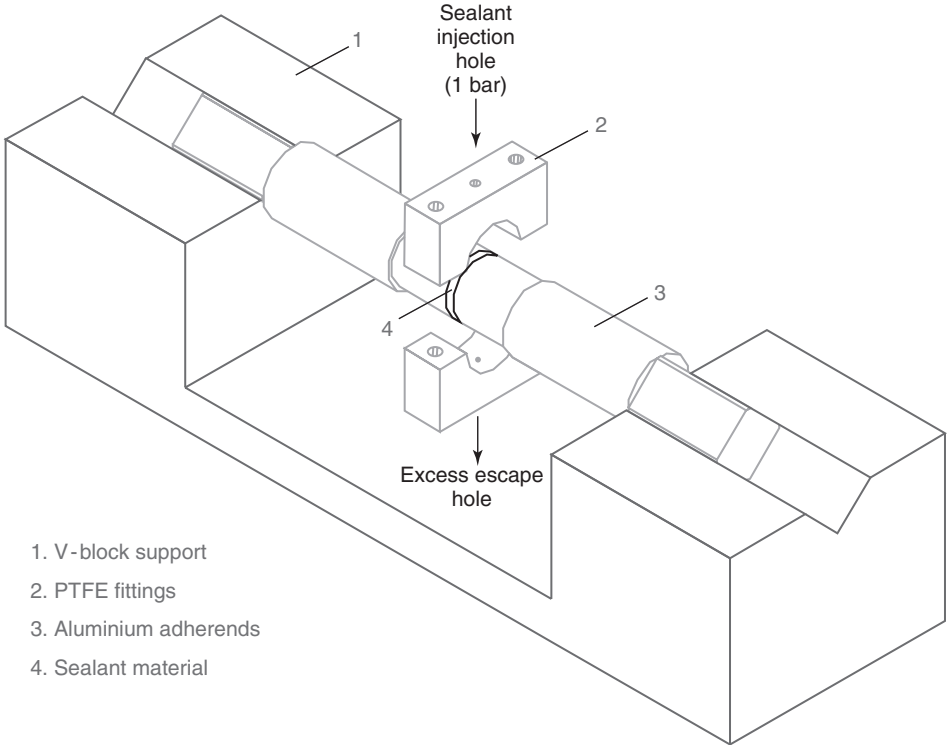


Figure 1.44 Schematic representation of the jig and the method used to manufacture the butt joints.

accurately as possible the shear properties of the examined material. For one-part and two-part adhesives, degassing the material before applying on the adherends ensures minimal air voids in the joint. For these types of materials, the procedure described in Section 1.8.4 might be more adequate. However, as sealants are very viscous, another approach should be followed for manufacturing the butt joint specimens. For example, a typical range for the viscosity of sealants is from 1500 to 1800 Pa·s, while a conventional two-part epoxy structural adhesive has viscosity in

the range 25–35 Pa·s. Thus, it is very difficult to remove the entrapped air, which is introduced during mixing, by degassing the mixed sealant, in less time than the working life of the material. For reducing the amount of voids in the butt joints, the sealant is injected into the joint under pressure. To achieve that, the following procedure is adopted. The two adherends are supported in a V-block jig, as shown in Figure 1.44, which keeps them level. Two pieces of polytetrafluoroethylene (PTFE), machined to the appropriate dimensions, are fitted around the two adherends. The PTFE fittings ensure that the two adherends are a certain distance apart, which corresponds to the desired bondline thickness. The sealant is then injected from the upper hole under 2 bar pressure, using an air pressure gun. As the sealant enters the joint, it displaces the air. In addition, due to the pressure applied, a number of voids existing in the mixed sealant collapse at the entrance of the injection hole. When the gap between the aluminum adherends is filled, the sealant material starts to flow out through the lower release hole and the injection is stopped. To avoid any misalignment of the aluminum adherends, when using this technique in manufacturing, the butt joints should be handled with special care after injection of the material.

1.9
Preparing Napkin Ring Specimens

Robert D. Adams

1.9.1
Introduction

Butt joints are commonly used for testing adhesives in the thin film form. Various standards exist for testing, examples being **ASTM E 229** and **ISO 11003-1**. The manufacture of suitable joints is less easy than it might appear. Solid butt joints are described elsewhere in this book (Section 1.8); such joints can be tested in tension or torsion, but there are problems in that the stress distributions are complex [27, 28]. For this reason, joints made between two rings are often used. In torsion, the radial shear stress variation is small, but in tension, the stress is almost as complex as in a solid butt joint.

Because the adhesive consists of a thin film between two metallic substrates, the full cure cycle can be used with no fear of the endothermic effect found with bulk specimens (Section 1.3). By taking appropriate care, adhesives supplied as a film, paste, or liquid can be used.

1.9.2
Adherends

A typical specimen is shown in Figure 1.45. The dimensions can be varied as is convenient. The specimen consists of two metallic pieces, which could be steel, aluminum, or titanium; the choice of substrate is not critical, as it is the adhesive

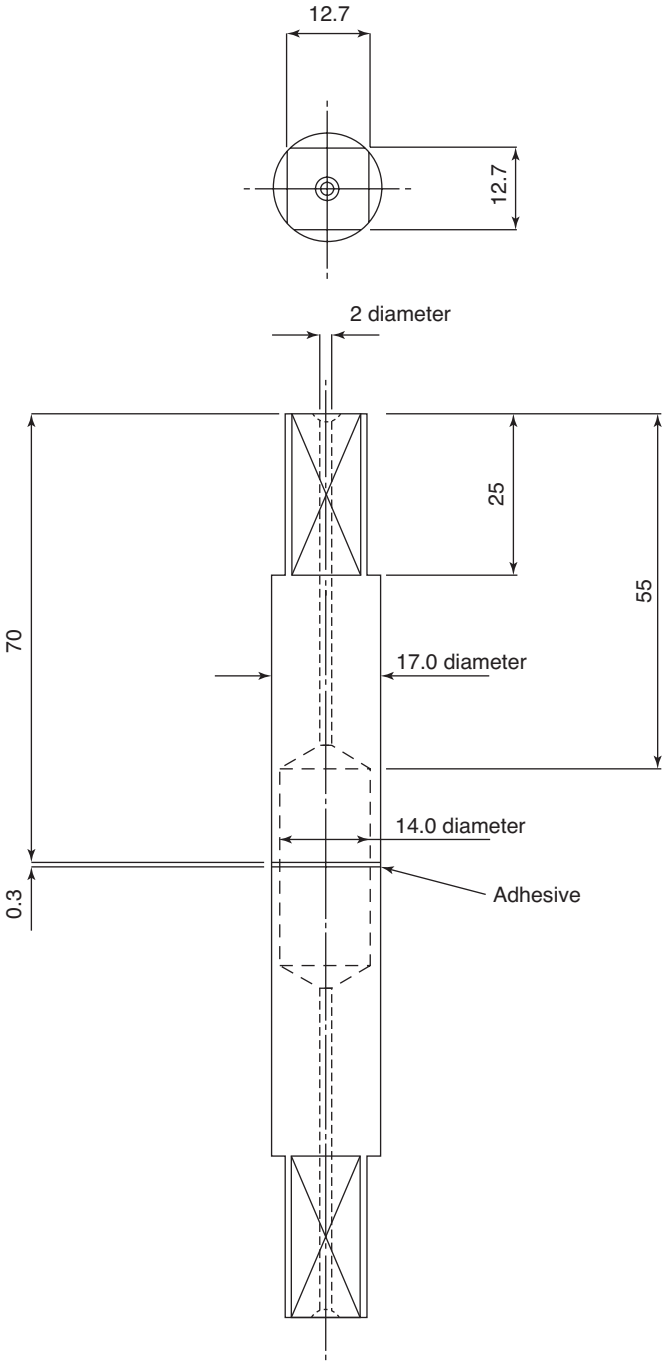


Figure 1.45 Typical dimensions (millimetres) used for a napkin ring specimen.

that is expected to fail in the test. The only necessity is that the bonded surface should be easy to prepare and that the dimensions be such as to be easy to manufacture and align accurately in the bonding process. It helps, therefore, to use a substrate material that is inexpensive, readily available, and easy to machine. Since in a laboratory specimens may be dropped, it is better not to use mild steel or a soft grade of aluminum alloy, as these can be damaged. Experience and material availability will usually lead to a sensible solution.

Specimens can be machined from a bar with a nominal diameter of 20 mm. First, centers are determined at each end of the bar. Next, using these centers as locations, the square cross sections are milled at each end. The outside diameter (17 mm) of the annulus is then created in a lathe. The bar is then cut in two, and the 17 mm circular section is used as the location datum. From this datum, the 14 mm internal radius can be created by drilling or boring. Finally, a 2 mm diameter hole is drilled along the axis of the specimen. Apart from the final 2 mm hole, all dimensions have to be accurately controlled and the alignment has to be true. The depth of the 14 mm internal hole can be chosen to suit. In practice, it is common to reuse the specimens after each test, cutting back to the metal in order to remove all traces of the original adhesive, so an initial depth of 15 mm is recommended. It should be noted that there must not be a chamfer on the inner or outer edges of the adherends so that the adhesive in the joint is a true cylindrical section.

1.9.3
Joint Manufacture–Alignment Jig

The two adherends must be accurately aligned, and this alignment must be maintained throughout the creation of the joint. In order to do this, an accurately machined jig is necessary (Section 1.8). Also, this jig should be checked at least annually and, if necessary, reground. The jig should be made from the same material as the specimen. In other words, aluminum specimens are best made in an aluminum jig; this minimizes problems related to differential thermal expansion if a hot cure is used. For steel specimens, a tool steel grade is recommended. The jig contains V-shaped grooves, one for each specimen. Typically, specimens would be produced in a batch of six, and whether a single jig with multiple grooves or several jigs with one groove is made depends on the machine shop and curing oven available.

The purpose of the groove is to provide alignment of the two adherends. The adherends are placed and lightly clamped in position. The clamps will also allow the jig to be carried to the cure oven without disturbing the adhesive. One of the adherends is then adjusted until a predetermined space is created between the adherends, which will define the bondline thickness. Usually, a bondline of about 0.5 mm is used. If the bondline is too thin, the rotational deflection across it will be too small to measure with accuracy. If it is too large, geometric imperfections can occur, which will mask the measurement accuracy.

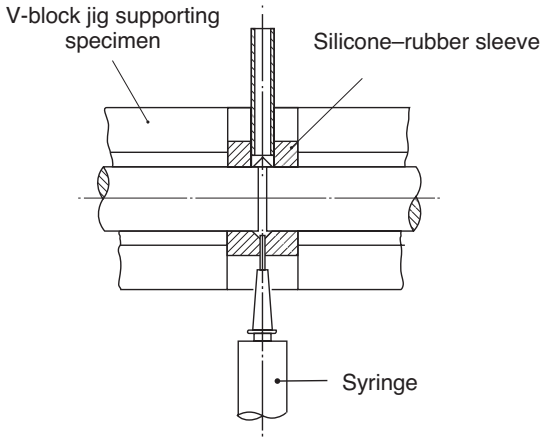


Figure 1.46 Injection of adhesive into napkin ring butt joint.

1.9.4 Introduction of the Adhesive

Various possibilities exist for inserting the adhesive. The objective is to have a truly cylindrical layer of adhesive between the two adherends. For liquid and paste adhesives, it has been found best to use some form of mold around the outside of the specimens, as shown in Figure 1.46. The mold can be of PTFE or of silicone rubber. The objective is to create a fillet on the outside of the adhesive layer when it has cured. This fillet will then be removed by machining. Experience has shown that this is preferable to trying to form a perfect outer radius using only the mold. In addition, a cylindrical plug is placed inside the tube such that there is negligible clearance between the outer diameter of the plug and the inner diameter of the tube. This plug forms the internal part of the mold. If the diameter of the plug is too large, it will be compressed radially, except at the bondline where it can expand into the bondline cavity, thus distorting the cured shape. If the plug diameter is too small, the adhesive will leak away from the joint.

After placing the plug and outer mold, the adhesive is injected into the lower part of the outer mold through a hole. Air inside is displaced through a vent at the top of the mold, and the joint is assumed filled when the adhesive appears at the top of this vent. Experience shows that a silicone rubber mold is adequate for liquid adhesives, but PTFE is necessary for the higher pressures used for injecting viscous or paste adhesives.

The jig is now placed in the curing oven (if a hot cure is required) and the usual cure cycle applied. Because of the higher thermal mass of the jig and the adherends compared with the thin adherends of a lap joint, a longer time is usually needed. Test runs with a few thermocouples in and around the bondline will show the temperature–time profile so that real joints can be cured correctly.

1.9.5
Final Specimen Preparation

After cure, the molds are removed and the internal plug is pushed (via the 2 mm axial hole) so as to clear the inner radius of the adhesive. The specimen is put in a lathe, and the fillet formed on the outside of the joint is carefully removed. There is no need to machine the inner bore of the joint as the plug has provided a proper smooth internal radius to the joint. Indeed, it is physically impossible to machine internally unless a much larger initial bar diameter is used. Note that it is essential to have no fillet with torsion joints as these fillets actually introduce a stress concentration [28].

The napkin ring butt joint for the torsion testing of adhesives can be made with accuracy provided the above-mentioned procedure is followed. It is not essential to use the exact same dimensions as given above, and other experimenters will vary the procedure with the confidence of experience. Carefully made joints take time and effort, but these will give true results. Carelessly made joints will give a lot of scatter in results, and the mean will always be lower.

1.10
Preparing T Joint Specimens

Lucas F.M. da Silva and Robert D. Adams

1.10.1
Introduction

Peel tests have been used for many years to compare the relative strengths of different adhesives, different surface preparation techniques, and so on. However, they can also be used to determine the adhesive toughness by suitable data treatment. One of the advantages of peel testing is that various combinations of mode I and mode II loadings can be applied by varying the peel angle, although it has been shown that when total input energy is corrected for plastic deformation in the peel arm, the adhesive toughness is independent of the peel angle (at least, for flexible peel arms) [29]. There are various peel test methods: fixed arm, T-peel, wedge peel, floating roller, climbing drum, and mandrel peel. These peel methods differ in the way that the load is applied to the specimen, but they are very similar in principle.

The T joint is not as common as lap joints loaded in shear. However, in some cases such as the automotive industry, it provides a geometry that is well adapted for spotwelds and is commonly used. This joint is composed of two adherends bent at 90° to each other and bonded together as shown in Figure 1.47. The amount of adhesive spew fillet is an important variable in this type of joint, and it affects the joint in a way similar to the lap shear specimens described in Sections 1.4 and 2.10. The joint geometry, including the adhesive fillet, is difficult to control manually.

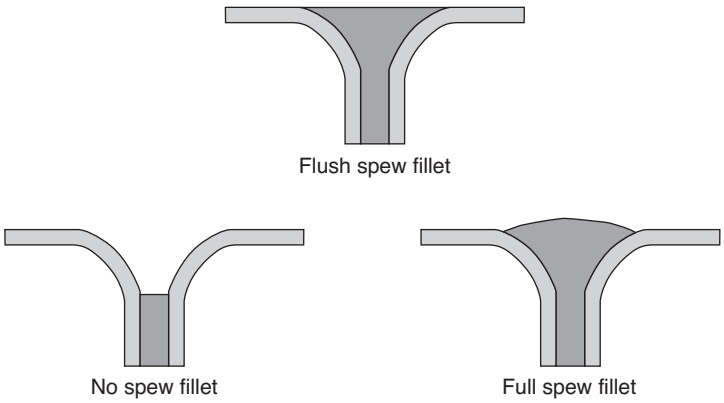


Figure 1.47 T-joints with different types of spew fillets.

Standard **ASTM D1876** recommends bonding two plates, cutting specimens, and bending at 90° the free length for griping. However, this procedure may introduce microcracks in the adhesive during manufacture, and the bending operation is hard to control. A preparation using a mold that rigorously controls all the geometric parameters is described here. The substrates are machined and bent to the correct dimensions before bonding. However, the procedure is only suitable for short overlaps (less than 25 mm). For longer overlaps, the adherends should be bent after bonding to facilitate the adhesive application.

1.10.2 Mold

The T-joints are produced using a metal jig to hold the joint in place and cured by placing the jig in an oven. Figure 1.48 shows the design of the jig. This jig was initially developed by Grant *et al.* [30] and has been recently improved by da Silva *et al.* [31]. The material used to manufacture the mold was carbon steel with 0.45% carbon in the annealed condition. It is easy to machine, and guarantees good heat dissipation. In order that the fillet is indeed flush with the outside of the adherend, the following procedure is followed. One set of the adherends is placed on one side of the jig (Figure 1.49a), and the top block is placed on them and tightened down (Figure 1.49b). The other set is placed on the movable part of the mold using the positioning pins and shims to hold the adherends apart at the correct distance for controlling the adhesive thickness (Figure 1.49c). A bar beneath the adherends is then tightened down with screws so that the adherends are fixed to the movable block (Figure 1.49d). The movable block is removed (Figure 1.49e). The jig is then turned through 90°, so the flange leg of the adherend is horizontal. The adhesive is then placed on the flange leg (Figure 1.49f). The jig is then laid flat again, while the movable block with the second set of adherends is applied against the first set. Up to six specimens with a width of 25 mm can be manufactured with this mold. The overlap length can be adjusted during the adherend manufacture.

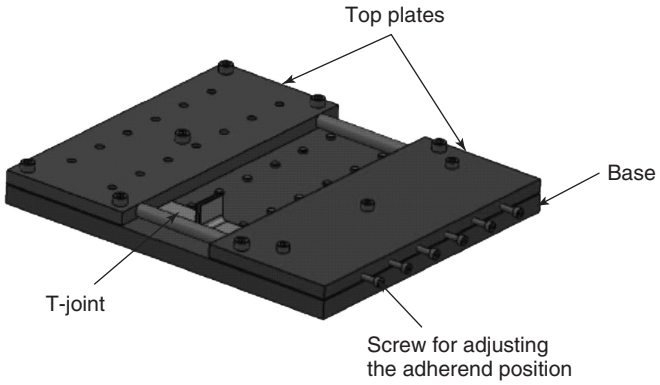


Figure 1.48 Diagram of the jig to produce T-joints [30].

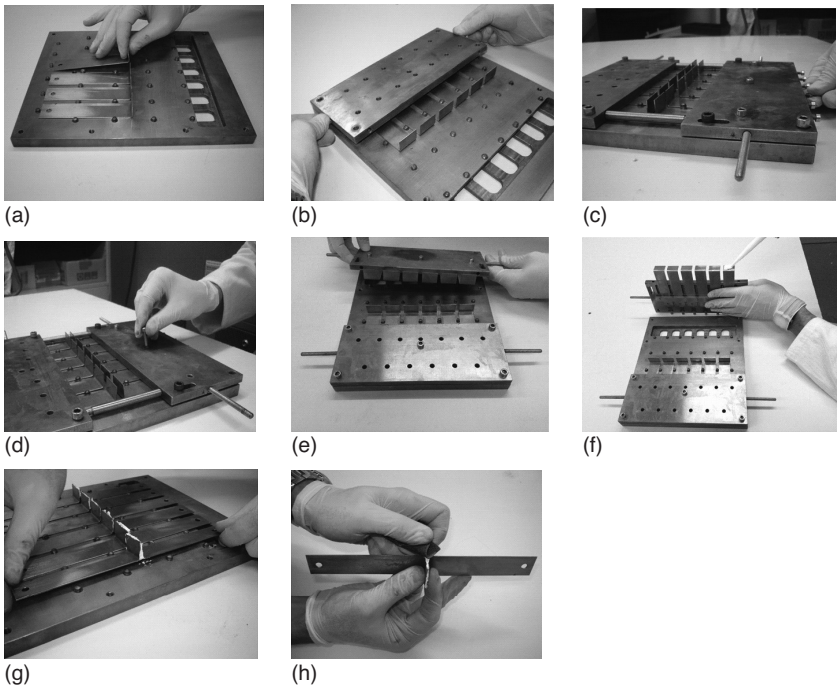


Figure 1.49 (a–h) Manufacture of T-joints.

1.10.3
Substrates Preparation

The geometry usually used with the mold presented above is shown in Figure 1.50. However, the bending radius, the adhesive thickness, and the overlap can be altered easily. The adherend radii is formed in a manual bending machine and

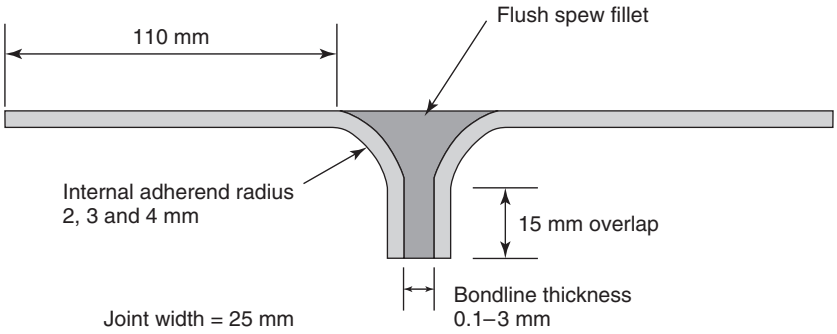


Figure 1.50 T-joint geometry [29].

controlled with radius gauges. A flush fillet is obtained with this procedure. The substrates are always prepared to remove dust, dirt, oil, oxides, or release agents in order to improve the interfacial bonding. For most adhesives, the surface preparation consists of degreasing with acetone or another solvent, shot blasting, and degreasing again with a solvent. If the failure is not cohesive in the adhesive, a chemical treatment should be used. It is preferable to use steel instead of aluminum because the mold described above being made of steel, when high cure temperatures are required residual thermal stresses is not introduced in the adhesive because of differences in thermal expansion between the mold and the specimens.

1.10.4
Adhesive Application and Assembly

The adhesive application depends on the adhesive form. For liquid adhesives, thin bondlines should be used to avoid spreading out of the adhesive. In the case of film adhesives too the adhesive application is straightforward, even though the gaps between the film and the substrate can lead to voids in the adhesive (Section 1.4). For two-part adhesives that are mixed manually, the adhesive flow, when the top adherend is placed, eliminates part of the voids. However, for the best results, mixing should be done so that there are no voids in the adhesive. Recent sophisticated machines in which mixing is done at high speed can ensure that the adhesive is void free. The adhesive should be applied following the steps described Section 1.10.2.

1.10.5
Cure

In case the adhesive cures above room temperature, the mold should be placed in an oven with good air circulation. It is advised to use a thermocouple close to the

adhesive and to count the cure time from the moment the adhesive reaches the cure temperature.

After the adhesive cure, the cooling rate should be slow to guarantee uniform temperature in the mold and avoid residual stresses. Also, the specimens should not be removed before the mold has reached room temperature; otherwise, the residual stresses can reduce the joint strength considerably.

1.10.6 Specimens Cleaning

After cure, the specimens are carefully removed from the mold (Figure 1.49g). The adhesive excess on the sides of the joints is removed with a file or emery paper (Figure 1.49h). The specimens should be conditioned under controlled temperature and humidity because these factors influence the mechanical properties of the adhesive.

1.10.7 Results

Peel testing is usually associated with a large scatter, especially for structural adhesives that are very sensitive to defects such as voids and microcracks. The procedure described here for producing T-joints enables a uniform bondline layer to be obtained and the control of all the geometrical parameters. The results obtained with joints prepared with the mold described above show very little scatter, as shown in Figure 1.51. The adherends were degreased with acetone to remove surface contamination, especially oil, and were then grit blasted to give a surface finish of 2.5 μm. The grit blasted surface was again degreased using acetone. The geometry of the tests presented in Figure 1.51 is that shown in Figure 1.50 (with a 2 mm radius). The adhesive was a paste epoxy.

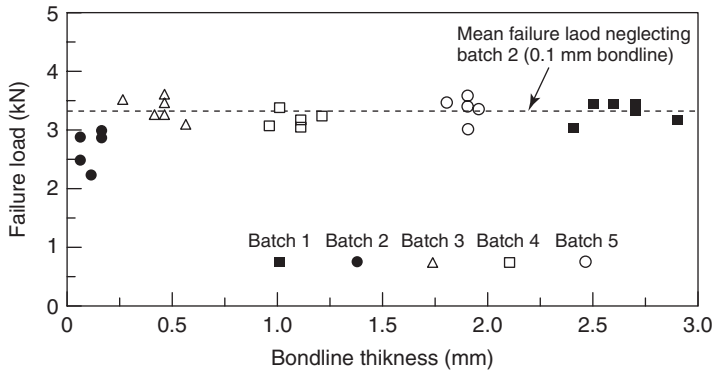


Figure 1.51 Failure load versus bondline thickness for T-joint specimens (2 mm radius) [30].

1.11
Preparing Flexible-to-Rigid Peel Specimens

Stefanos Giannis

1.11.1
Introduction

The peel specimen is widely used for quality control and to assess the peel performance of adhesives and sealants. Various forms of the peel test are used, as shown schematically in Figure 1.52. There are a number of ASTM and ISO international standards describing the test procedures of the various types of peel tests. **ASTM C 903** and **ISO 8510-2** describe the 180° peel test for a flexible-to-rigid assembly, while **ISO 8510-1** refers to the same type of assembly but specifies testing at a 90° peel angle. There are also specific standards for the T-peel test **(ASTM D 1876)**, the climbing drum peel test **(ASTM D 1781)**, and the floating roller peel test method **(ISO 4578)**. Also, there is a specific procedure for testing sealant materials at a 180° peel angle, described in **ASTM C 794**. The emphasis in this section is on the manufacturing of specimens used for 180° peel testing of adhesives and sealants on flexible-to-rigid assemblies.

1.11.2
Mold

The international standard test procedures do not specify a particular mold for preparing peel specimens with paste or film structural adhesives. However, it is recommended that, wherever possible, peel specimens are cut from bonded panels rather than bonding individual specimens. In this way, more reproducible specimen geometries are achieved, with minimal edge effects on each sample. A universal bonding fixture is composed of a metallic plate with locating pins to accurately position the rigid adherend. This can be aluminum, steel, or FRP (fiber-reinforced plastic) composite. Another metallic plate is used to sandwich the

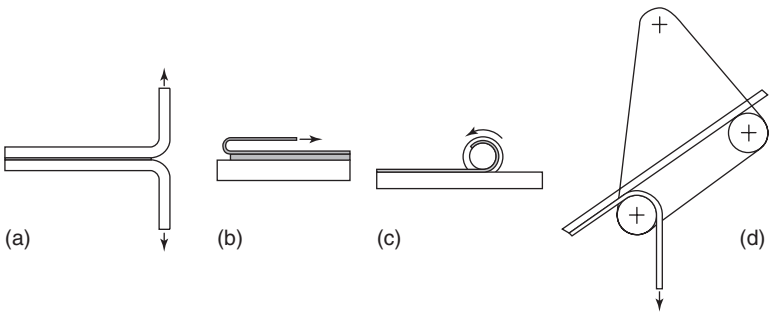


Figure 1.52 Various forms of the peel test: (a) T-peel test for flexible-to-flexible adherends, (b) 180° peel test for flexible-to-rigid adherends, (c) climbing drum peel test, and (d) floating roller peel test.

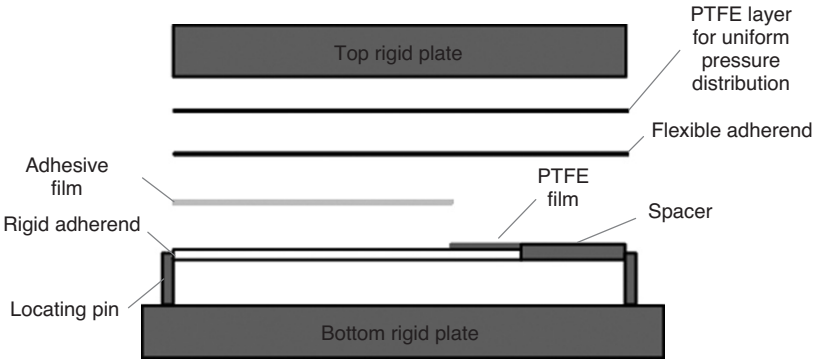


Figure 1.53 Molding arrangements for manufacturing peel specimens with paste or film adhesives.

assembly of rigid adherend, adhesive, and flexible adherend and ensure application of light pressure (i.e., enough pressure to keep all parts of the mold together firmly) during curing. The flexible adherend can either be a thin aluminum or a steel sheet (Figure 1.53). When the flexible adherend is a tape material, already containing a thin layer of adhesive (i.e., self-adhesive tape), it is directly placed on top of the rigid adherend and cured, if needed, according to the manufacturer's instructions. Pressure should be applied through the top metallic plate of the mold. The above described molding procedure works very well for the case of manufacturing peel specimens with paste and film adhesives or self-adhesive tapes.

When testing sealants, or rubbery materials, cutting the specimens from a bonded panel is not generally recommended. Moreover, the flexible adherend used to apply the peel load is usually either a stainless steel mesh or a fiberglass cloth **(ASTM C794)**. For manufacturing these types of peel specimens a special mold is needed in which individual specimens, rather than panels, are molded. A PTFE mold, as shown in Figure 1.54, can be used. This mold provides an easy and flexible way of manufacturing five peel specimens at a time [5]. By simply changing only one part of the mold, both the sealant thickness and the thickness of the top layer sealant, used to ensure that the flexible adherend is well impregnated and would not peel away from the material under test, could be varied accordingly.

1.11.3 Adherend Preparation

The adherends must be prepared to remove dust, dirt, oil, oxides, or release agents in order to enhance the interfacial bonding. For most structural adhesives, the surface preparation consists of degreasing with a solvent (i.e., acetone, methyl-ethyl-ketone), grit blasting, and degreasing again with a solvent. In some cases, chemical treatment of the surface might be needed to improve bonding and result in cohesivetype failure of the tested adhesive. When structural adhesives or sealants are tested against aerospace-grade aluminum adherends, primer might be applied to the

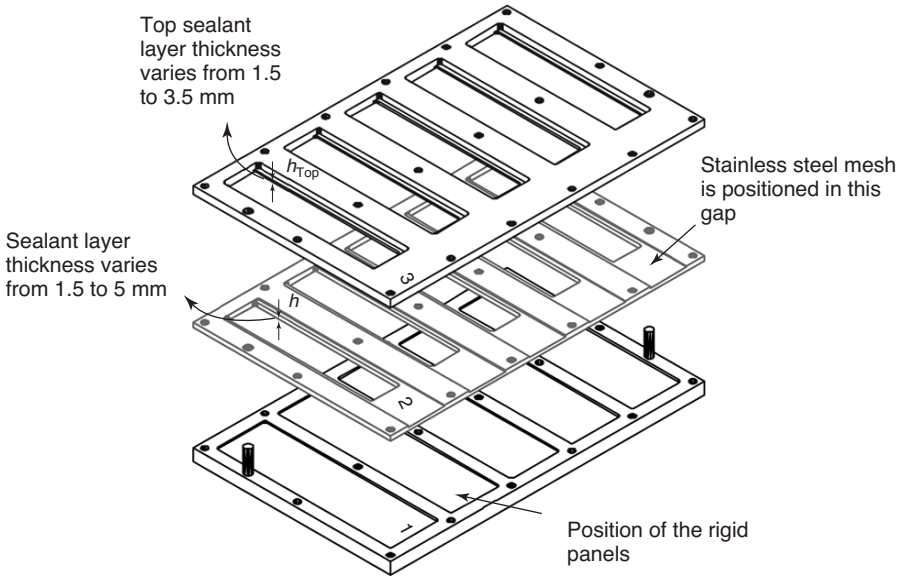


Figure 1.54 Mold for manufacturing sealant peel specimens.

surface of the aluminum before bonding. It is preferable to use thicker steel rigid adherends rather than aluminum to ensure that deformation of the adherend is minimal and influences the peel forces. However, when an adhesive material has to be tested against a particular adherend (e.g., thin aluminum) special care should be taken during the testing phase to ensure minimal deformation via an appropriate gripping arrangement.

1.11.4
Adhesive Application and Assembly

Adhesive application depends on the adhesive form. For paste structural adhesives, thin bondlines should be used to avoid large spew fillet. In the case of film adhesives, adhesive application is simple but care should be taken to minimize the gaps between the film and the adherend, which can lead to voids in the adhesive. For two-part adhesives that are mixed manually, the adhesive flow when the two adherends are pressed against each other eliminates part of the voids. Before the manufacture of bonded panels, release agent must be applied to the metallic mold and the locating pins. For the case of sealants and when the PTFE mold in Figure 1.54 is used, release agent is not required. The upper surface of the rigid adherend, where the flexible adherend will be bonded, should be treated according to the adhesive manufacturer's specification (i.e., grit blasted, chemically etched, etc.) and accessible so as to apply the paste or film adhesive. To control the bondline thickness, glass beads of specific diameter can be dispersed in the adhesive. Care should be taken to dry the glass beads to remove any moisture from their surface

before dispersing in the adhesive. The concentration of the glass beads should not exceed 5% per volume of the adhesive layer, in order not to affect the properties and influence the test results. Finally, a rigid plate is also needed to apply uniform pressure on top of the bonded assembly. A PTFE film should be placed as shown Figure 1.53 to provide an initial unbonded area between the rigid and the flexible adherends, as required for this type of test. The length of the unbonded area is not critical for this type of test, and it should be long enough to allow gripping the specimen on the machine for testing.

For making sealant peel specimens, using the mold shown in Figure 1.54, the following method should be used. Rigid adherends of the appropriate size are placed in the cavities at the first part of the mold. The second part of the mold, which determines the sealant layer geometry and positions the flexible adherend, is fitted on top. The sealant is then mixed according to the manufacturer's instructions and applied on the panels. In order to achieve a good-quality specimen, each cavity is filled with a large amount of sealant. The sealant is then drawn down using a suitable scraper, creating a continuous layer. A strip of the stainless steel mesh or fiberglass cloth is impregnated with the sealant and laid on top of the sealant layer, and then the scraper is carefully drawn across its surface to remove any entrapped air. Finally, the third part of the mold is placed on top and a thin layer of sealant is put over the steel mesh.

1.11.5 Cure

For adhesives that cure at room temperature, the mold should be placed in an air circulated oven at ambient conditions. Some control of the humidity level within the oven is desirable, as the cure of a number of adhesives can be affected by moisture in the environment. In the case that curing takes place at temperatures higher than the ambient room temperature, this should be monitored with a thermocouple placed as close possible to the adhesive layer. The actual curing time should be determined from the moment that the adhesive reaches the curing temperature. Cooling should be slow and the specimens should be allowed to reach ambient conditions before being removed from the mold. Most sealants cure at room temperature so it is essential to control the humidity levels of the environment in which curing takes place. Most laboratories have constant temperature and humidity levels, but using an oven at ambient temperature and humidity is recommended.

1.11.6 Final Specimen Preparation

After curing, the bonded panels are carefully removed from the mold. Cutting specimens from the bonded panels is done using a diamond-coated saw at very slow speeds to minimize the introduction of defects at the edges of the joints. Typically, 20–25 mm wide specimens are cut (Figure 1.55). During cutting, and in order to avoid heat built up, a coolant should be used. Because of possible

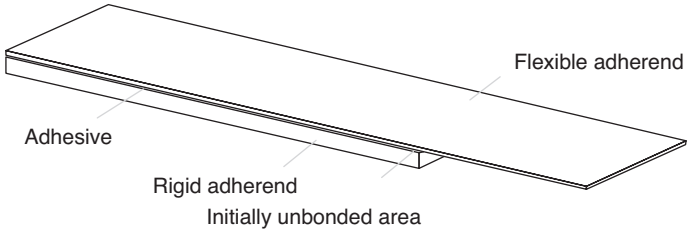


Figure 1.55 Typical peel specimens with flexible-to-rigid adherends.

potential water ingress in the adhesive layer, it is recommended to lightly warm up the specimens in an oven after cutting in order to remove any surface moisture. Temperature and time strongly depend on the particular system. It is recommended that the temperature be below the cure temperature of the system and the time be in the order of 10–15 min. For the case of sealant peel specimens, cutting is not required since these are prepared as individual specimens rather than bonded panels. Specimens should then be placed in an incubator chamber set at ambient temperature and humidity levels.

1.12
Preparing Specimens for Fracture Properties: Double Cantilever Beam and Tapered Double Cantilever Beam

Bamber R. K. Blackman

1.12.1
Introduction

The double cantilever beam (DCB) and tapered double cantilever beam (TDCB) adhesive joint test specimens have been widely employed for fracture-mechanics-based testing since the early work of Ripling, Mostovoy, and coworkers in the 1960s [32, 33]. Their work led to the publication of an ASTM standard [34]. This method was developed for metallic substrates, namely, brass, copper, aluminum, steel, and titanium. More recently, partly due to the increased popularity of fiber-reinforced polymer matrix composites, the test has been revisited [35, 36] and a new international standard has been published [37]. The test specimens are shown in Figure 1.56. The test method is described in Section 3.2.

The method requires that DCB and/or TDCB test specimens be manufactured for the determination of the critical strain energy release rate (SERR) in mode I (the tensile opening mode), that is, G_{Ic}. DCB specimens are simple to manufacture and are frequently the specimens of first choice. TDCB specimens are more complex and expensive to make but have the advantage that the height taper provides a linear change in compliance with crack length, such that they are useful when it is not possible, or desirable, to measure crack lengths during the test.

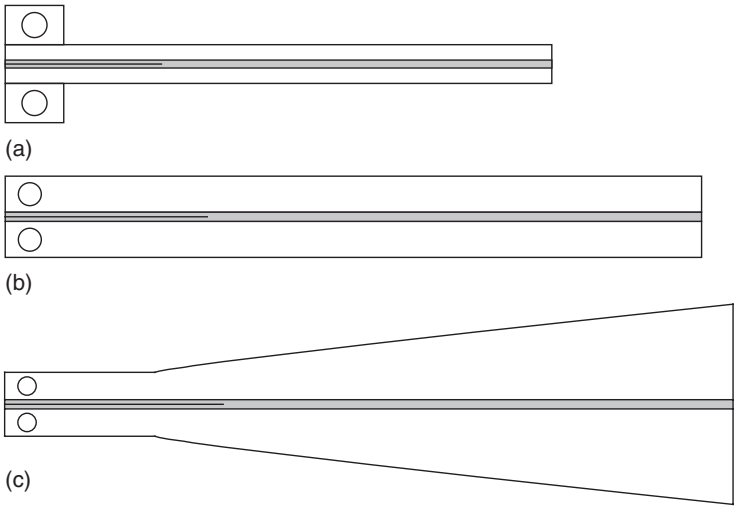


Figure 1.56 (a) DCB with load blocks, (b) DCB with drill holes, and (c) TDCB adhesive joint specimens.

DCB specimens can be manufactured in two ways. Plates can be adhesively bonded, and specimens can then be subsequently cut from the bonded plates. Alternatively, substrates can be machined to final size and can be subsequently bonded in a support frame or jig. Each method has some advantages over the other, and the user should consider various factors before choosing which preparation technique to follow. These factors include (i) whether it will be possible to cut the adhesively bonded plates into specimens without damage to the adhesive occurring and (ii) whether the bondline thickness can be controlled across the plate or in the individual specimens (Section 1.12.5.2). TDCB specimens are manufactured using the second method.

1.12.2 Bonding Jigs

If DCB specimens are to be manufactured from bonded plates, the procedures of **ASTM D3433** [34] can be followed and the recommended plate sizes used. The plates should be flat and smooth with a maximum surface roughness of 4.1 μm and with edges free from burrs. Once the plates are prepared, surface treatment takes place before adhesive bonding. Figure 1.57a,b shows the bonding of DCB plates. If individual substrate pairs are to be bonded to form the specimens, a bonding jig as shown in Figure 1.57c,d may be used to maintain the orientation of the substrates and also to provide means to apply a fixed pressure during adhesive cure. The pressure may be applied either by attaching bolts to the threads and tightening or by placing weights on top of the upper plate. The jigs can be made

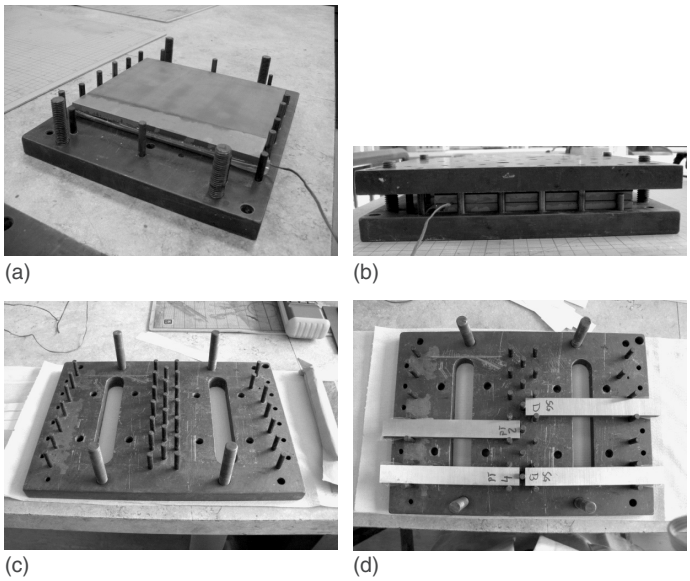


Figure 1.57 DCB bonding jig: (a) with two bonding plates in position; (b) with top plate in position; (c) lower plate for individual substrate pairs; and (d) with four joints in position.

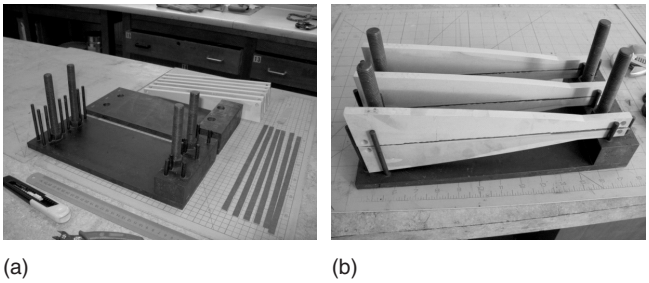


Figure 1.58 (a) TDCB bonding jig with upper and lower plates, adhesive film strips and substrates and (b) loaded with three joints awaiting top plate.

from carbon steel. A thermocouple wire is shown leading from the bondline of the joint in Figures 1.57a,b, as described in Section 1.12.6.

If TDCB specimens are to be manufactured, then pairs of substrates are bonded together in a jig as shown in Figure 1.58. Figure 1.58a shows a typical style of bonding jig with an upper and a lower plate, together with specimen alignment pins. Figure 1.58b shows the jig loaded with TDCB specimens awaiting attachment of the top plate. Again, the jig maintains the orientation of the specimens and provides the means to apply a fixed pressure during cure. The jigs should be clean and coated with an appropriate mold release agent before use, to facilitate the removal of the specimens after curing the adhesive.

1.12.3 Specimen Dimensions

1.12.3.1 DCB Specimens

ASTM D3433 specifies the following dimensions for the DCB substrates: length = 356 mm, width = 25.4 mm, and height = 12.7 mm, (14 × 1 × 0.5 in.). Then there are a number of further factors to consider before dimensions are finalized. First, the minimum height of the substrate may be determined via the need to avoid plastic deformation of the substrates during the test. This is determined in **ASTM D3433** via Eq. (1.1)

$$h_{\min} = \sqrt{\frac{6Ta}{B\sigma_y}} \tag{1.1}$$

where $h_{\min}$ is the minimum permissible substrate thickness to ensure that only elastic deformations of the substrates occur, T is 150% of the maximum load to start the crack in the adhesive layer, B is the joint width, and σ_y is the tensile yield strength (or proportional limit) of the substrate. As it is necessary to perform a test to obtain T, this technique can assist the user to refine the h value after initial testing. Similarly, if the G_{Ic} value is known, the minimum permissible substrate thickness can be determined from Eq. (1.2)

$$h_{\min} = \frac{3EG_{Ic}}{\sigma_y^2} \cdot n \tag{1.2}$$

where E is the Young's modulus of the substrate in the direction along the specimen length and n is a safety factor ($n = 1.5$ would be equivalent to the 150% factor of safety used in Eq. (1.1)). Second, to minimize the contribution of shear to the total deformation of the joint, the ratio of h/a should be kept to a minimum. Thus, substrates with large h values will require longer crack lengths and hence longer total lengths. **ISO 25217** does not prescribe the dimensions of the substrates to be used for the DCB tests, but experience has shown that joint widths in the range of 20–25 mm and lengths in the range of 150–250 mm are usually satisfactory.

1.12.3.2 TDCB Specimens

ASTM D3433 recommends substrates of length 241.3 mm (9.5 in.) and of width 25.4 mm (1 in.). Substrates of length 310 mm and of width 10 mm have been shown to give satisfactory results [35, 36]. The height profile of the TDCB specimens is required such that the rate of change of compliance, C, with crack length, a, that is, dC/da, is constant over a suitable range of crack lengths in the specimen.

The shear-corrected beam analysis defines the profile in terms of a geometry factor, m, such that

$$m = \frac{3a^2}{h^3} + \frac{1}{h} \tag{1.3}$$

where a is the crack length, measured from the applied load line in the test, and h is the substrate height, a function of a, This nonlinear profile ensures that dC/da remains constant; however, at $a = 0$ and for small values of a, the profile requires

values of h below h_{min}, that is, below that required to avoid plastic deformation of the substrates. There is also the need for a certain minimum thickness at $a = 0$ to allow for the application of the load via pins inserted though holes drilled through the substrates. Therefore, an initial length of the substrate has constant height. D3433 specifies this initial height to be 12.7 mm (0.5 in.), extending 48.62 mm from the far end of the joint. The dimensions of the TDCB employed in Refs. [35, 36] are shown in Figure 1.59.

Different values of the geometry factor can be used; a smaller value of m will lead to a steeper taper and a stiffer substrate, while a larger m will give a shallower taper and more compliant substrate. The ratio of bending to shear deformation of the substrates will alter, depending on the value of m chosen. ASTM recommends $m = 90$ in.$^{-1}$ (3.5 mm^{-1}). Much of the supporting work in the development of **ISO 25217** used $m = 2$ mm^{-1}, with the profile section as tabulated in Figure 1.59.

1.12.4 Substrate Conditioning and Preparation

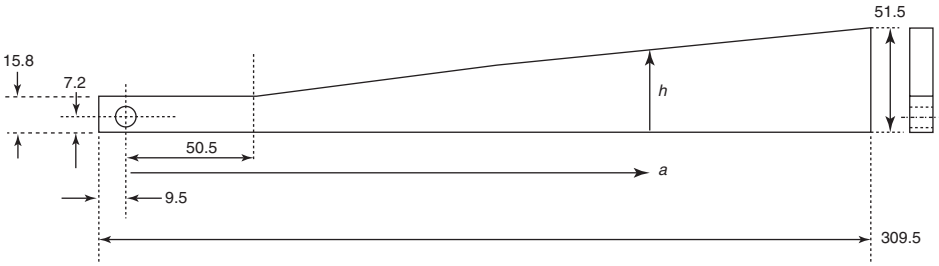
1.12.4.1 Storage and Substrate Conditioning

After the production of plates or individual substrates for bonding, it is necessary to ensure that metallic substrates do not corrode or otherwise deteriorate unduly before surface pretreatment. It may be necessary to maintain metal substrates in a dry environment during this time. Polymeric substrates that absorb water will need to be dried before surface pretreatment and subsequent adhesive bonding are undertaken. Users should consult the substrate material supplier for recommendations. When bonding with certain adhesives, absorbed water has been found to reduce joint toughness significantly when carbon fiber reinforced polymer (CFRP) substrates are employed [38]. Procedures for drying CFRP can be found in [39].

1.12.4.2 Surface Pretreatment

Whether plates or individual substrate pairs are to be bonded, the surfaces should be carefully pretreated before adhesive bonding. Such a step is usually an essential part of the joint manufacture. Some adhesives have been designed to work in the presence of certain surface contaminants. Users are referred to specific product information, and the many bonding guidelines, provided by adhesive manufacturers, for example, by 3M and Henkel. The most recent internationally agreed guidelines for surface preparation of metals and plastics before adhesive bonding can be found in [40].

For metallic substrates, it is usually necessary to remove any oil and grease and to roughen the surface before final cleaning. Some metals will require subsequent treatment to remove weak oxide layers and replace these with oxides grown under closely controlled conditions, for example, via anodizing. Frequently, a primer is then applied to enhance adhesion and provide surface protection. For fiber-reinforced composite substrates, it will be necessary to remove any residual mold release agents from the surfaces of the substrates via careful grit blasting.



a (mm)	h (mm)
50	15.80
55	16.72
60	17.71
65	18.67
70	19.61
75	20.53
80	21.42
85	22.30
90	23.16
95	24.00
100	24.83
105	25.65
110	26.45
115	27.24
120	28.02
125	28.79
130	29.54
135	30.29
140	31.03
145	31.76
150	32.48
155	33.20
160	33.90
165	34.60
170	35.30
175	35.98
180	36.66
185	37.33
190	38.00
195	38.66
200	39.32
205	39.97
210	40.61
215	41.25
220	41.88
225	42.51
230	43.14
235	43.76
240	44.38
245	44.99
250	45.60
255	46.20
260	46.80
265	47.39
270	47.99
275	48.58
280	49.16
285	49.74
290	50.32
295	50.89
300	51.47

Figure 1.59 TDCB substrate showing dimensions in millimetres and profiled values of a and h for $m = 2$ mm^{-1}. These substrates may be used to form a TDCB specimen for testing using **ISO 25217**.

In this respect, wet grit blasting is preferred to dry grit blasting as the process is less aggressive and is less likely to drive contaminants into the surface. Surface cleaning will be required, and if thermoplastics are being used, it may be necessary to modify the surface with an electrical technique (e.g., plasma or corona) to enhance the adhesion of the surface.

1.12.5 Adhesive Application and Forming the Joint

1.12.5.1 Adhesive Handling and Application

Adhesives are typically supplied in either film or paste formats, and the pastes can be single or two part, room or high-temperature curing. Film adhesives are usually supplied in designated thicknesses and partially cured, and hence are stored in a freezer before use. It is important to ensure that the adhesive has reached room temperature before use and that the adhesive film is fully insulated from atmospheric moisture until used. It is therefore essential that the film is maintained in a sealed bag until it has reached ambient temperature. Unused adhesive should be returned to the bag and be resealed before return to the freezer. Paste adhesives may be supplied in drums or cartridges, and these are also frequently stored in subambient temperatures. These should be allowed to reach ambient temperature before use. Owing to high viscosity, it is recommended that certain paste adhesives be heated above ambient temperature before application. The manufacturers' literature should be consulted.

It is typical to apply one layer of film adhesive to the bonding surface of one substrate only. One (or more) layers may be applied to the bonding surface of each substrate, but if more than one layer is to be used, it should be noted whether the adhesive has a designated tacky side, and the adhesive manufacturers' advice should be sought. If two-part paste adhesives are used, it is important to ensure proper mixing of the constituents and to observe the handling time quoted by the manufacturer. The paste adhesive is applied to the substrate in bead form, and this is spread evenly across the bonding area when forming the joint.

1.12.5.2 Control of Bondline Thickness

Except for the most brittle adhesives, the results of the DCB and TDCB test are strong functions of the bondline thickness. It is therefore important to carefully control this dimension. While film adhesives have a nominal layer thickness, it is unlikely that this will be achieved closely in the cured joint without some additional control. Some paste adhesives contain fillers of known maximum particle size (or particle size range), and this will add some control of layer thickness, but it may be insufficient or may not be the desired thickness.

Strategies to control bondline thickness usually involve the placement of shims at each end of the joint. Lengths of stainless steel wire (of known diameter) can be placed across the width of one bonding surface after the application of the adhesive. Provided the substrates have high flexural rigidity, these will control the bondline. For very long joints, or when the substrates are more flexible, it may be necessary to add additional shims or wires to the midjoint region. Preferably, they should be outside the length of intended crack propagation in the fracture specimen, that is, outside of the range $a_0 < x < a_f$ where a_0 is the initial crack length (see next section) and a_f is the final intended crack propagation length. If this is not achievable, it might be possible to place short wires, staggered with longitudinal alignment, within the propagation length.

Another way to control bondline thickness is to add glass spheres (ballotini) to the adhesive. These can be purchased in known sizes or size ranges. Used in very small quantities, and dispersed uniformly throughout the adhesive, they will not alter the adhesive properties. They should, however, be dried before use, as they will tend to adsorb atmospheric moisture onto the surfaces, which may be damaging to the adhesive.

Both test standards [34, 37] assume that the adhesive layer makes negligible contribution to the overall compliance of the joint. In the development of the standard in Ref. [37], round-robin and supporting studies demonstrated that acceptable results could be obtained for epoxy adhesives with bondlines in the range 0.1 mm $< h_a <$ 1.0 mm. Values of $h_a >$ 1 mm may be used, but the test validity has not been demonstrated with these thicker layers, or with low-modulus adhesives. More details on specifying the adhesive thickness are available in [37].

1.12.5.3 Introduction of the Initial Crack

The DCB and TDCB specimens both require an initial disbond, from which a mode I crack initiates and subsequently propagates during the test. In **ASTM D3433**, this is a ''nonadhesive shim'' of length 51 mm placed at one end of the bonding surface before closing the joint. **ISO 25217** recommends the use of a ''nonstick film,'' preferably PTFE, of thickness <13 μm. Experience has shown that very thin films (<8 μm) may be too delicate and may rupture in use. Fluoropolymer films supplied for the vacuum bagging of composites with a thickness of ~12.5 μm have been used very successfully. The upper service temperature of the film should exceed the cure temperature of the adhesive to be bonded. Sections of film are cut and carefully placed on the adhesive layer, as applied to one substrate, before closing the joint.

1.12.6 Cure

For individual DCBs or TDCBs, the top plate of the jigging fixture should be closed and the pressure should be applied. If a heat cure adhesive is being used, the jig should be placed in an air circulating oven for the required time, as recommended by the manufacturer of the adhesive. It is advisable to monitor the temperature of the adhesive in the bondline of one joint via the insertion of a thermocouple to allow for the effects of thermal inertia. It is important to ensure that the adhesive layer reaches the intended temperature and is maintained at this temperature for the required cure duration. If a room temperature curing adhesive is being used, the joints should remain in the jigging, under pressure, for the recommended time. The comments also apply to the bonding of plates.

1.12.7 Final Specimen Preparation

If plates have been bonded, **ASTM D3433** should be consulted for advice on cutting out the specimens. If individual specimens have been bonded, any adhesive fillet

will need to be removed to leave a joint with smooth sides. If it is not possible or desirable to drill loading holes through the substrates, loading blocks should be used. These can be aluminum or another suitable metal, with a loading hole drilled centrally through each block. The blocks should be adhesively bonded to the substrates, and a jig may be required to ensure proper block alignment. Local surface pretreatment should be applied to the substrates and the bonding surface of the blocks. It may be necessary to consult [40]. A two-part, room temperature curing epoxy adhesive has been shown to be suitable for block attachment. As an alternative to loading blocks, piano hinges can be used to introduce the load to the specimen, provided the loads are not sufficiently high to cause permanent deformation of the hinges. However, unless it is known that the adhesive is of low strength, the use of loading blocks in preference to piano hinges is strongly recommended.

The specimens should be moisture and temperature conditioned before testing. Users should consult [41] for standard atmospheres for conditioning and testing. Finally, it is common practice to coat one side of the specimen with a thin layer of white typewriter correction fluid to facilitate the measurement of crack growth. As some solvents can damage the adhesive, an aqueous solvent is recommended. Lines can then be drawn along the side of the specimen, perpendicular to the plane of the adhesive, to aid the crack length measurement.

1.13
Preparing Bonded Wood Double Cantilever Beam (DCB) Specimens

Hitendra K. Singh, Edoardo Nicoli, and Charles E. Frazier

1.13.1
Introduction

Few materials have been used throughout history as much as wood. This is due to the relative abundance and the properties of this material. Wood has unique characteristics among natural materials in terms of strong anisotropy, providing good strength-to-weight and stiffness-to-weight ratios in the grain direction. In one direction wood is strong and stiff, whereas in another direction it is weak, soft, and easily machined. The combination of these characteristics has facilitated, for example, wood's use in construction: it is relatively easy to split or saw wood parts from a large block or a tree trunk. Considering also the general low density and the renewable nature of wood, the crucial role of this material in human civilization becomes evident. Even now, wood is prized for various applications, ranging from the paper industry to building construction [42]. Most North American homes are built from wood and wood-based composites; no other material matches the performance-to-cost ratio; and this performance includes reduced energy consumption and carbon footprint in comparison to competitive materials such as steel, concrete, and aluminum (*http://www.corrim.org/*).

Adhesively bonded wood constructions are frequently used as assembly techniques in many structural and nonstructural applications [43, 44]. The technique of adhesive bonding plays a critical role in the wood products industry. Adhesives are used for manufacturing building materials, such as plywood particleboard and flakeboard, and also in structural applications such as framing, I-joints, and laminated timbers. Because of widespread application of bonded wood structures, understanding the durability and reliability of bonded joints, subjected to various environmental conditions, and a variety of loading modes, is very important for the proper use of this material. For understanding fracture properties and failure mechanisms of bonded wood joints, tests are often performed using double cantilever beam (DCB) type specimens. One of the important prerequisites for understanding various aspects of the bonded geometry and obtaining reliable information from fracture testing is the preparation of good-quality specimens. As one points out "the quality of the data is as good as the quality of the specimens used to obtain that data"; the aim of this section is to provide information on the methods and procedures used to fabricate good-quality DCB specimens so that meaningful and repeatable results could be obtained from fracture testing of adhesively bonded wood systems.

1.13.2 Aspects of Wood Bonding

Structurally, all wood is composed of basic units called *cells*, which are long and narrow elements that, aligned with the trunk, form the characteristic wood grain. These cells resemble drinking straws, but with closed ends as if the straws were pinched at the ends. Therefore, wood is considered orthotropic at the microscopic scale. In addition, in most temperate climates, common trees present annual growth rings, where layers of earlywood and latewood are alternated as growing conditions cycle from year to year. Earlywood is characterized by cells with relatively large internal void space, usually a lighter color and a relatively low density. Latewood has thicker cell walls and greater density. The different densities of these two layers result in different stiffness and strength and, given the disposition of these layers, result in a material that is orthotropic also at the macroscopic scale. The described characteristic aspects of wood, such as its porosity, orthotropic nature, and the presence of natural variations influence the nature of the bond formation and bond durability in the material. In light of this, several aspects of wood bonding are rather unique within the spectrum of bonding of materials.

Proper wetting of the surfaces is important in the construction of bonds, but this aspect is even more critical in wood bonding, where the adhesive has to flow not only between the surfaces to be bonded but also into the cellular structure to promote interlocking and reduce stress concentrations near the bondline, increasing the bond strength, especially with the mechanisms of diffusion and mechanical interlocking. The diffusion of the adhesive inside the adherends is greatly influenced by the chemistry of the adhesive resin and its viscosity, but aspects such as the porosity of the wood and grain orientation and consolidation pressure play an equally important role. The penetration of adhesive in a direction

perpendicular to the grain is less effective, and it rarely occurs; most adhesive penetration occurs along the grain because the wood cells tend to intersect the bonding surface such that cell lumens are almost always exposed and available for flow parallel to the grain. Nevertheless, the grain orientation choice during the preparation of specimens has to also account for the orthotropic nature of the adherends. In fact, the mechanical characteristics of wood are highly dependent on the fiber orientation and, in particular, the resistance to fracture occurring between layers of earlywood and latewood is usually relatively limited [45].

1.13.3 Sample Preparation

Preparation of bonded wooden DCBs has to address the following particular needs of the material systems: (i) proper penetration of adhesive into the adherends and (ii) the orientation of the fibers that lowers the risk of crack formation/propagation inside the adherends. Other aspects, such as the creation of a precracked area and of features that permit the DCB to be tested in a traditional load frame, are also important.

1.13.3.1 Wood Preparation

In the experience of the authors, specimen preparation usually starts with sawn lumbers of thickness ~50–55 mm. These parts are then band sawed to 12 mm thick laminates, which are then planed to the final thickness, which can be 8–10 mm. The other dimensions of the boards are mainly driven by the size of the available press. Large boards are not recommended, since they may easily lead to uneven distribution of adhesive. An important aspect is that the 12 mm thick laminates are carefully cut so that the orientation of the wood grain is between 3 and 5° with respect to the intended bond plane, as described by Gagliano and Frazier [46]. There are at least two important reasons for the choice of maintaining the grain orientation fairly constant. First, this configuration is beneficial in preventing the crack from moving from the bonded layer into the adherend substrate in common wood such as yellow poplar and yellow pine. Second, a fairly constant grain orientation provides more consistent stiffness characteristics, limiting the sources of variability in the experiments. For the same reason, boards containing knots or macroscopic defects should not be used for specimen construction, since knots and defects have different elastic properties and are likely to be subjected to dissimilar adhesive penetration, when compared to plain wood. Wood properties also depend on the water content, so it is preferable to have the boards conditioned at $20 \pm 1\,^{\circ}C$ and $65 \pm 1\%$ relative humidity (RH) in an environmentally regulated chamber for at least two weeks before proceeding with further processing. This step aims to obtain 12% equilibrium moisture content (EMC) in the wood, which is a reference value for wood properties, and minimizes any differential swelling in the specimens for testing purposes, fully recognizing that bonded wooden joints exposed to service conditions can experience additional debonding energies because of the presence of residual stresses [47]. When the boards are ready to be

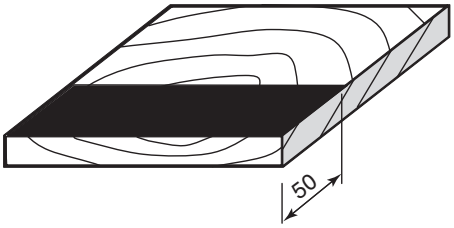


Figure 1.60 Wooden board for specimen construction. The shaded area is used to obtain a precrack (the dimension shown is in millimetres).

bonded, an easy way for obtaining a precrack is to color a 50 mm (≈2 in.) wide region of the board near one of the edges with a wax crayon to limit adhesion in the final bonded specimens. One such board is illustrated in Figure 1.60. The painted area has to be in the side of the board at which the fiber orientation converges at an angle 3–5° in the direction of crack growth (please refer to Section 1.13.3.3 for additional details).

1.13.3.2 **Adhesive Types**

A large range of products is available in the market for several different applications that require bonding of wood. Polyurethanes, phenol-, resorcinol-, and urea-formaldehydes, epoxies, and cyanoacrylates are among the most common synthetic resins that are typically used to bond wood laminates. The curing mechanisms of the resins include activation by the moisture naturally present in the wood, reaction of the two components, solvent loss, and thermal activation. The choice of the adhesive should follow manufacturer's recommendations for a given application, and technical sheets should always be consulted before preparing the samples.

1.13.3.3 **DCB Specimen Preparation**

The procedure for fabricating the DCB specimens starts by bonding the environmentally conditioned boards using the adhesive. To bond these boards, one of them is placed on a precision balance and, after zeroing the load, an amount of adhesive within the limits indicated on the technical sheet is poured on the board. Then, removing the board from the balance, the adhesive is spread to obtain a uniform distribution and the second board is placed on top of the adhesive layer, as shown in Figure 1.61a. The fiber orientation of the two boards is converging at an angle 3–5° in the direction of crack growth from both of the boards, as illustrated for the final specimen in Figure 1.61b.

Finally, the assembly is placed in a cold or hot press, depending on the adhesive system, and, during this pressing phase, correct alignment of the boards to be bonded is facilitated by means of lateral constraints (lateral pressing of the two boards for keeping a precise overlapping of the two). The bonded boards are often wrapped in aluminum foil to prevent any expelled liquefied adhesive from leaking onto the press. Adhesive quantity, applied pressure, and curing time imposed are those indicated in the technical sheets of the adhesives. Sometimes, multiple

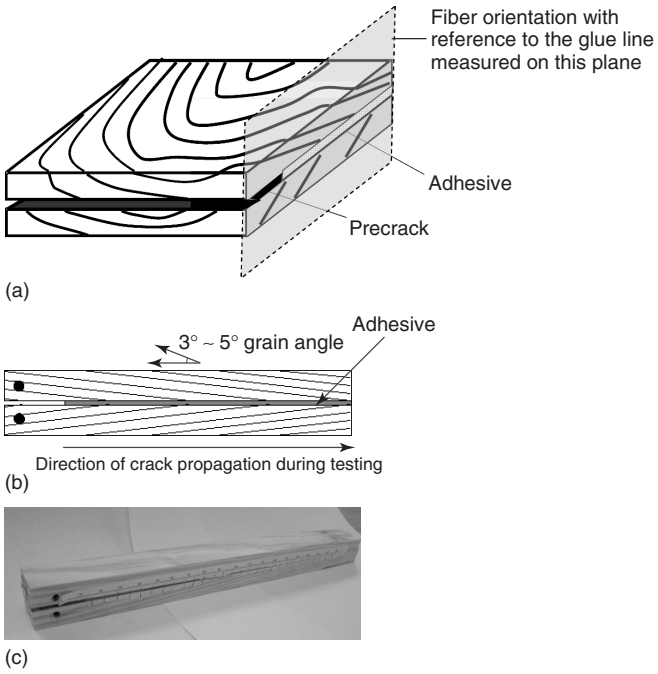


Figure 1.61 DCB sample preparation method: (a) two wooden laminates bonded together with adhesive; (b) illustration of a typical DCB specimen showing the grain angle orientation with respect to the direction of crack propagation; and (c) DCB sample painted with white correction fluid and marked with parallel lines along with the attached paper scale.

stacks of bonded boards are placed in the press and compressed at the same time. Particular care is taken to complete the bonding procedure without exceeding the prescribed handling time, which can often be of the order of a few minutes and thus limits the amount of bonded boards that one can produce at a time.

When moisture escapes during curing of moisture cure adhesives, wood has a tendency to curve in the opposite direction of the growth ring curvature. This characteristic becomes more pronounced as the moisture content in the wood decreases, the rate and level of which is dependent on temperature and atmospheric humidity. For this reason, in order to reduce residual stresses and ensure more consistent bondline thickness, the boards were laid so that their growth ring curvatures were in the same direction. An example of this is shown in Figure 1.61a. This ensured that distortions that would have taken place, due to wood drying in the oven during the long curing process, would be in the same direction, the important result being that residual stresses are minimized so that they do not contribute to crack growth, thereby lowering the measured mode I fracture energy.

After curing, the bonded pieces are cut to the appropriate width using a table saw, with the laminate's outer 10 mm edges being discarded to avoid edge effects.

The cut DCB specimens, that have a width in the order of 20 mm, are then returned to the same environmental chamber for further conditioning at $20 \pm 1\,^{\circ}C$ and $65 \pm 1\%$ RH for at least two weeks. Additional operations are typically performed on the specimens after this conditioning time, to prepare them for fracture testing. Two holes are drilled through the width direction, as shown in Figure 1.61c on the debonded end of each adherend. These holes provide a way to connect the specimens to loading clevises in the testing machine by using pins. One side of the bonded specimens can be painted with typewriter correction fluid to enhance the ability to detect the crack tip and, finally, a paper ruler is bonded on the same side of the specimen to facilitate crack length measurements. As already mentioned, attention regarding the orientation of the wood grain is given throughout the assembly procedure.

1.13.3.4 Judging the Sample Quality by Adhesive Penetration

The contributions of the different adhesion mechanisms in bonded wood samples are generally different from the contributions one can estimate in bonded metals or composites. During the production of bonded wood specimens, pressure applied to the bonding pieces rather than the bondline thickness is generally controlled. Adhesive penetration in the wooden beams usually results in a thin bondline [42]. Bondline thickness is a factor that influences the extension of the plastic zone and the value of critical strain energy release rate [48, 49]. The variation in bondline thickness may lead to significant scatter in the measured values. It is therefore important to examine the bonded DCB specimens to make sure that consistent bondline thickness is obtained during sample preparation.

An estimate of the bondline thickness can be obtained using a stereomicroscope along the finely machined edges of the DCB specimen. A generally more detailed procedure for obtaining the same measurements can also be based on the ideas and techniques applied by Frazier and Zheng [50] and Johnson and Kamke [51], and the methods described in [52]. The analysis is destructive and generally requires a microtome for preparing microscope samples and a fluorescence microscope working with transmitted light. A DCB has to be taken and cut in parts of section 5 by 5 mm, where the bondline is roughly in the middle. Then, these parts are further cut, with the microtome, into 40 μm thick slices. For increasing the contrast between the adhesive and the wood, several staining techniques can be applied, coloring these slices with solutions such as 0.5% safranine O or 0.5% toluidine blue O in water. Application of the color has to be done for few seconds, after which the slices are rinsed in distilled water. The slices that are obtained can be analyzed under the microscope where, again, with use of fluorescence and different light filters, the contrast between adhesive and wood is enhanced in order to be able to assess the adhesive layer thickness. This analysis procedure requires initial tests and can be improved for the different material systems. This kind of microscopic analyses can show interesting results: in some material systems, for example, the adhesive layer thickness may vary within a single specimen, as a result of roughness of the wood surface and the spacing of earlywood and latewood in the two bonded

boards. It is also common to see that the adhesive penetrates into the adherend to a depth that can be an order of magnitude larger than the adhesive layer thickness.

1.14
Modified Arcan Test

Jean-Yves Cognard, Laurent Sohier, Bernard Gineste, and Romain Créac'hcadec

The development of reliable tools is necessary for the optimization of adhesively bonded assemblies. Thus, a large database of experimental results under various tensile–shear loadings is required in order to develop representative numerical models [53]. Such results can be obtained using cylindrical bonded specimens loaded under combined loadings in tension and torsion [53], but these tests require a tension–torsion testing machine. In order to be widespread in laboratories, the use of a usual tensile testing machine is an important advantage. Scarf joints can be used but for each tensile–shear ratio a specific specimen is required and stress concentration can generate cracks at the substrate–adhesive interface [54, 55]. An Arcan-type device [56] allows a same bonded specimen to be loaded under various proportional tensile–shear loadings. Moreover, the geometry of the bonded specimen can be optimized in order to prevent crack initiation, that is, to limit the influence of edge effects.

1.14.1
The Arcan-Type Device

The principle of the Arcan device [56], which has been used for various experimental studies [57, 58], is presented in Figure 1.62a; this device allows tensile–shear loadings of the specimen. A variant of this fixture has been adopted in order to be able

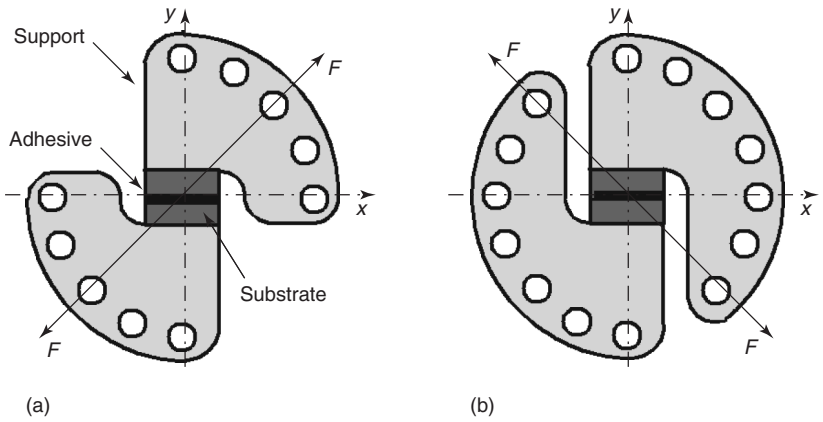
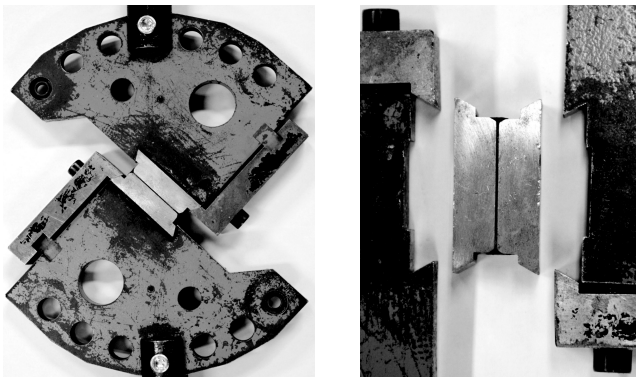


Figure 1.62 Principles of Arcan-type fixture. (a) Classical Arcan-type fixture. (b) Modified Arcan-type fixture.



(a) (b)

Figure 1.63 Classical Arcan fixture. (a) Classical Arcan-type fixture. (b) Bonded specimens and fixing systems.

to analyze the behavior of bonded assemblies under tensile/compression–shear loadings [58], as this is of interest for various applications.

Figure 1.63 presents the geometry of an Arcan-type device and bonded specimens often used. On one hand, the geometry of the substrates generates stress concentrations associated with edge effects for bimaterial components [25], and on the other hand, the fixing system of the bonded specimen on the Arcan support can generate a preloading of the bonded joint. Those two points can have an influence on the experimental results of these tests.

1.14.2 A Modified Arcan Test

In order to be able to accurately study the behavior of the adhesive as a function of the normal stress component and to use a traditional tensile testing machine, a modified Arcan fixture [25, 59] has been developed, which enables compression or tension to be combined with shear loads. Figure 1.64 presents the modified Arcan device considering four directions of loadings. The orientation between the loading direction and the mean plane of the adhesive is defined by the angle γ. For tension tests, γ is equal to 0° (Figure 1.64a); for traction–shear tests with the same load in tension and shear, $\gamma = 45^\circ$ (Figure 1.64b); for shear tests, $\gamma = 90^\circ$ (Figure 1.64c); and for compression–shear tests, $\gamma = 135^\circ$. Other directions can be used. Numerical simulations under linear elasticity assumption for bimaterial structures show that the use of a special geometry for the substrate (a beak close to the adhesive joint) strongly reduces the contribution of the singularities due to edge effects [25, 59], Section 1.7 (Figure 1.65).

Two important points were studied when designing this assembly. First, is the generation of a stress field in the adhesive joint, which is as uniform as possible with a maximum stress at the center of the joint. This is important in order to

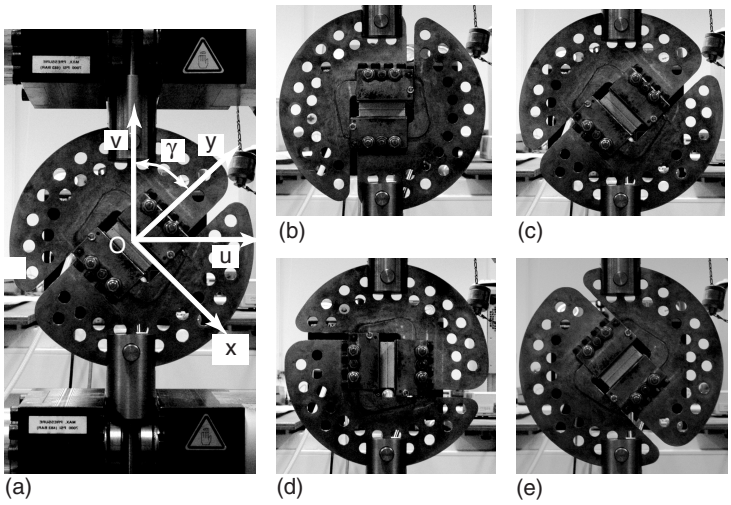


Figure 1.64 Modified Arcan fixture. (a) Modified Arcan fixture; (b) tensile, $\gamma = 0^{\circ}$; (c) tensile–shear, $\gamma = 45^{\circ}$; (d) shear, $\gamma = 90^{\circ}$; and (e) compression–shear, $\gamma = 135^{\circ}$.

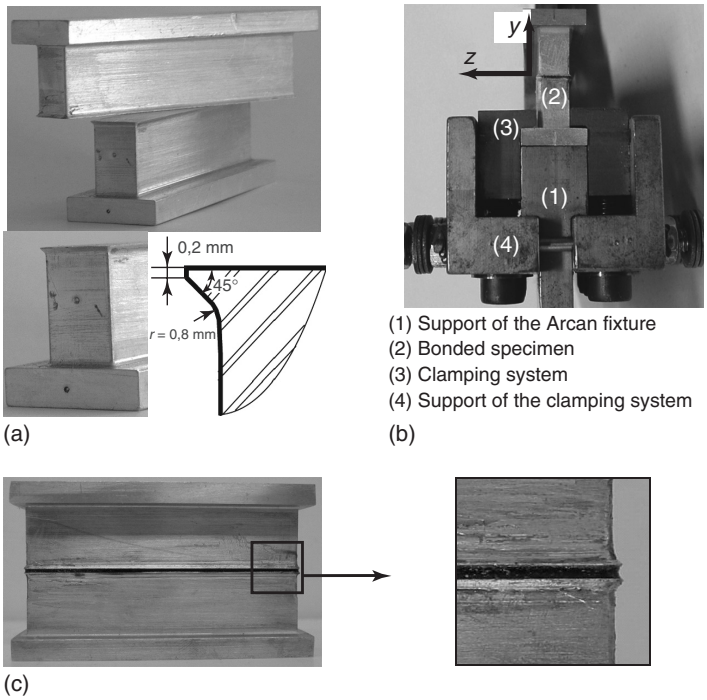


Figure 1.65 Geometry of the bonded specimens and mounting of the specimen on the modified Arcan fixture. (a) Geometry of the substrates. (b) Mounting of the specimen. (c) Bonded specimen.

limit the influence of defects. Second, the system fixing the substrates on the supporting fixture has to be designed in order to prevent preloading of the adhesive joint (Figure 1.65b). The optimization of the complete fixture was made using three-dimensional nonlinear simulations taking into account the fixing system, with refined meshes in order to be able to analyze the possible edge effects [25]. Moreover, in order to be as realistic as possible, the finite element models used take into account the contacts with friction and the assembly procedures.

A specimen with rectangular section (section of $70 \times 10\,mm^2$) was proposed by taking into account the constraints involved in machining (Figure 1.65). The beak is machined with an angle of 45° and a blending radius of 0.8 mm (other values of the parameters can be used). The definition of a beak with a sharp angle leads to manufacturing and handling problems. Simulations showed that an extra thickness of about 0.2 mm on the beak did not change the stress field in the adhesive. Figure 1.65c presents the geometry of the free edges of the adhesive, which is obtained by cleaning before curing; such geometry reduces the influence of edge effects [59].

Other geometries that strongly limit the influence of edge effects can be used for the substrate design [25, 59, 60].

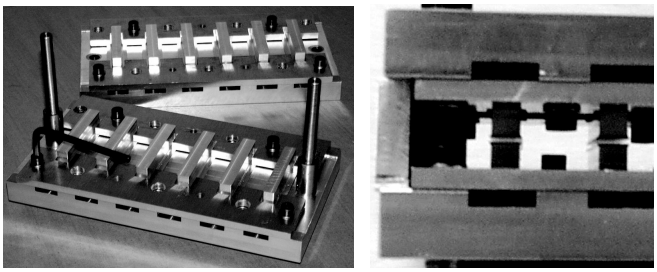
1.14.3 Interfaces Assembly Machine

As the dimensions of the adhesive joint are quite small, it is necessary to avoid the generation of parasite loadings, associated in particular with bending, in order to limit modification of the stress distributions in the adhesive joint. The important points to control are

- the geometrical quality of the various parts;
- the relative positioning of the two substrates during bonding;
- the positioning of the specimen in the Arcan fixture;
- the connection between the Arcan fixture and the tensile testing machine.

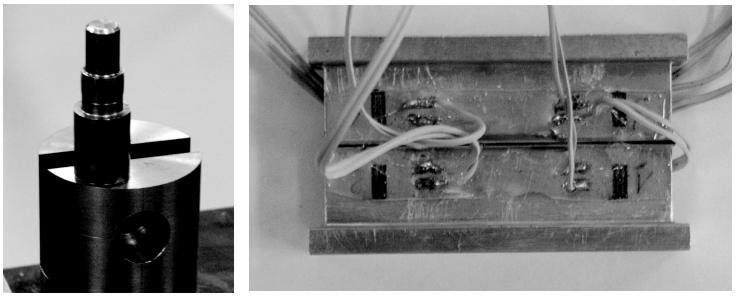
The assembly as well as the substrates have to be machined with the greatest care. Precise positioning of the substrates is obtained by the use of a special fixture during bonding (Figure 1.66). Moreover, this system allows a precise control of the joint thickness, which is an important parameter for the experimental tests. Subsequent control of these substrates carried out with a three-dimensional measuring machine confirmed the geometrical quality of the different parts.

In order to prevent parasite loadings, the link between the Arcan fixture and the testing machine is made using a specific pin to apply the loading in the middle plane of the fixture (Figure 1.67a). Instrumented assemblies, with strain gages in both sides of the specimen, are used to verify the stress distribution in the bonded joint (Figure 1.67b).



(a) (b)

Figure 1.66 Bonding fixture for six assemblies. (a) Substrate positioning and (b) adhesive thickness control.



(a) (b)

Figure 1.67 Partial link between fixture and test machine, and an instrumented assembly. (a) Loading fixture. (b) Instrumental assembly.

1.14.4
Stress Distribution in the Joint

The numerical analysis of stress distribution in the adhesive is important to analyze the experimental results. Three-dimensional fine element simulations have been performed under elastic assumption taking into account the real geometry of the bonded assembly. For these models, the loading results are presented for aluminum substrates (Young's modulus: $E_a = 75$ GPa, Poisson's ratio: $\nu_a = 0.3$). The material parameters for the studied adhesive were such that $E_j = 2.2$ GPa, $\nu_j = 0.3$. The symmetry with respect to the loading plane (O,x,y), O being the center of the specimen (Figure 1.64a), and the antisymmetric loading make it possible to reduce this three-dimensional simulation to one-fourth of the specimen by applying adequate boundary conditions. After the simulation of the fastening of the specimen, a tension–shear loading is imposed on the upper face of the support [25]. Precise numerical results require large models. A tensile–shear loading with the same load components in the x- and y-direction is prescribed. Figure 1.68a presents the stress distribution in the mean plane of the adhesive in the loading plane (O, x, y). The evolution of the stress components xx, yy (peel), and xy (shear) with respect to the overlap length (x-direction) are drawn (the maximum

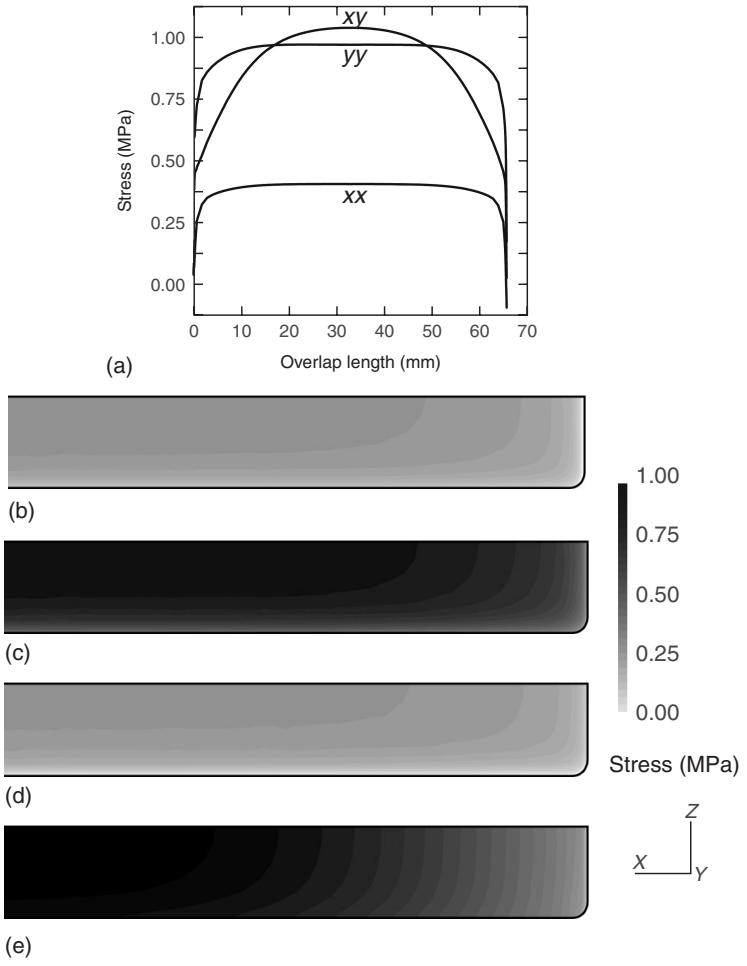


Figure 1.68 Stress in the mean plane of the adhesive for a 3D model. Only a quarter of the adhesive overlap is represented. (a) Stress components with respect line (O,*X*); (b) stress component *xx* (>0.00; <0.40); (c) stress component *yy* (>0.00; <1.00); (d) stress component *zz* (>0.00; <0.40); (e) stress component *xy* (>0.00; <1.00).

stress component is chosen equal to 1.). Figures 1.68b–e presents for a quarter of the mean plane of the joint the stress distribution for the components *xx*, *yy*, *zz*, and *xy*. The other two shear components are nearly equal to 0. For each component, the extreme values are noted under the drawings. The stress distribution in the adhesive is almost constant. It is important to note that the maximum value is located at the center of the joint and that the stress level is lower all around the free edges of the adhesive.

Under the assumptions of calculation, the maximum values of the components of the stress are obtained from the finite element results and from the average

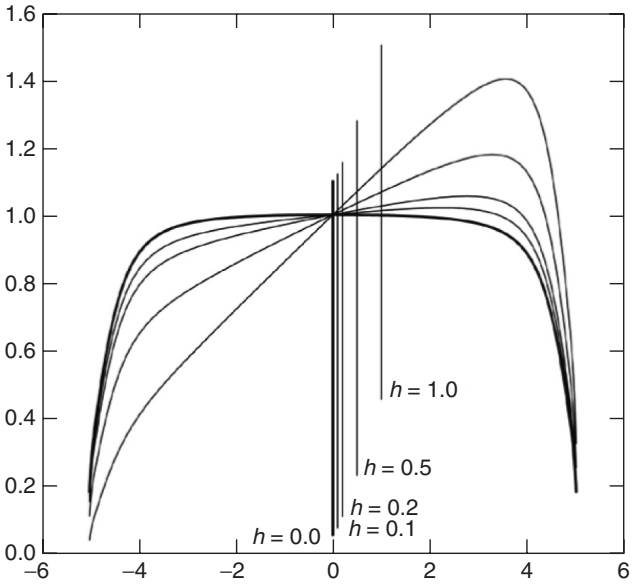


Figure 1.69 Influence of a position defect h on the stress distribution.

stress based on the loads measured during the experimental test and the section of the adhesive plane (S_c)

$$\sigma_{\gamma\gamma\,\text{maxi}} = 1.12\sigma_{\gamma\gamma\,\text{average}} \rightarrow \text{with } \sigma_{\gamma\gamma\,\text{average}} = F_N/S_c$$

$$\sigma_{x\gamma\,\text{maxi}} = 1.29\sigma_{x\gamma\,\text{average}} \rightarrow \text{with } \sigma_{x\gamma\,\text{average}} = F_T/S_c$$

where F_N and F_T are the components of the applied load, respectively, in the normal and tangential directions in the mean plane of the adhesive joint (i.e., in the γ- and x-direction, Figure 1.64).

These simulations underline that the stress state in the adhesive is quite complex; thus a correct analysis of the experimental results requires the use of inverse identification. Under shear loadings (load in the x-direction, $\gamma = 90^\circ$, Figure 1.64), the joint is nearly loaded under shear ($x\gamma$ component), but under tensile loadings (load in the γ-direction, $\gamma = 0^\circ$, Figure 1.64), the joint is mainly loaded with three stress components (xx, $\gamma\gamma$, and zz).

In order to respect the loading conditions associated with the Arcan device, a specific loading fixture is used (Figure 1.67a). To evaluate the maximum acceptable defects, in order to obtain good experimental results, various simulations were carried out starting from 2D modeling in the (O, γ, z) plane. Figure 1.69 presents the influence of a loading offset h, with respect to the center of the specimen, on the normal stress distribution in the adhesive. It is important to note the sensitivity of this parameter: according to these calculations, to limit to 5% the modification of the maximum normal stress one needs to keep the loading offset below $h = 0.2$ mm. These calculations confirm the need for checking the various points quoted previously in order to guarantee the accuracy of the tests.

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