



EUROPEAN ADHESIVE ENGINEER

MODULE 4.10

DURABILITY- DURABILITY ASSESSMENT AND LIFE PREDICTION FOR ADHESIVE JOINTS



4.10 Durability Assessment and Life Prediction for Adhesive Joints

Scope:

- ✓ Durability test techniques - general
- ✓ Diffusion dominated durability: spring loaded shear specimens
- ✓ Adhesion dominated durability: wedge test, wet peel test
- ✓ Corrosion dominated durability: salt spray test

Durability test techniques – general [1]

Range of adhesively bonded joints durability testing:

- Tests with moisture
- Tests with temperature (creep testing)
- Wedge test, wet peel test, salt spray test
- Different modes of fatigue and fracture tests

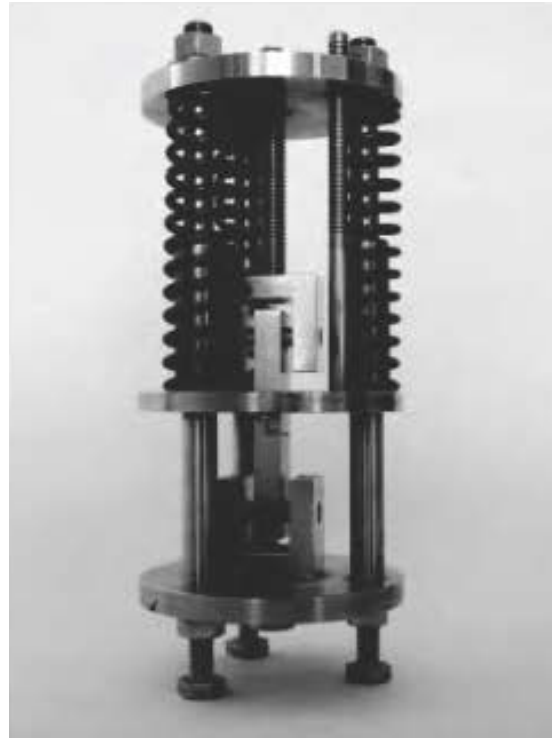
Diffusion dominated durability [1]

Spring loaded shear testing for conducting creep tests

For the purpose of **conducting creep experiments with adhesively bonded joints under natural or accelerated aging conditions**, several spring-loaded creep test instruments have been suggested. Since **constant stress creep tests are desirable**, the usual common practice would be using constant dead weight load tests with gravity causing the predefined level of load.

If the total deformation or extension in the adhesive is small, the change in spring load can be neglected. Spring-loaded units have the advantage of reduced weight compared to creep testers applying the required creep load directly by means of masses.

Diffusion dominated durability [1]



Spring loaded creep unit for thick adherend specimens

Diffusion dominated durability [2]

ASTM D 2293: Standard Test Method for Creep Properties of Adhesives in Shear by Compression Loading (Metal-to-Metal)

This test method covers the determination of the creep properties of adhesives for bonding metals when tested on a standard specimen and subjected to **certain conditions of temperature and compressive stress** in a spring-loaded testing apparatus.

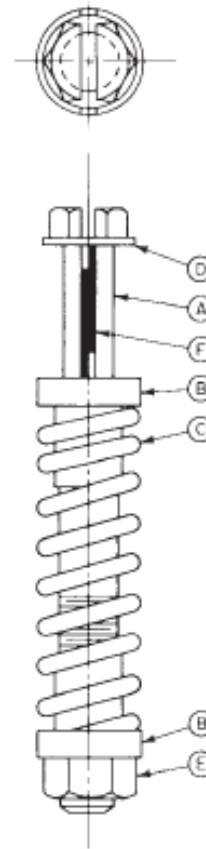
This test method is useful in research and development for comparison of creep properties of adhesives, particularly as those properties are affected by changes in adhesive formulation or expected service conditions, including temperature, moisture level and duration of loading.

Diffusion dominated durability [2]

Procedure:

- 1). To conduct a creep test, center the specimen within the slot between the washer and bushing of the apparatus as shown in Slide 8. Compress the spring between the two bushings to the desired load by tightening the nut. The correct load can be applied by deflecting the spring a given measured amount as determined from a calibration curve.
- 2.) To measure total deflection, observe the average displacement of fine razor scratches across the centers of both sides of the lap joints with a calibrated microscope having 100 × magnification.
- 3.) Record the total observed average deflection, the magnitude and duration of the compressive stress, and the test temperature for each specimen.

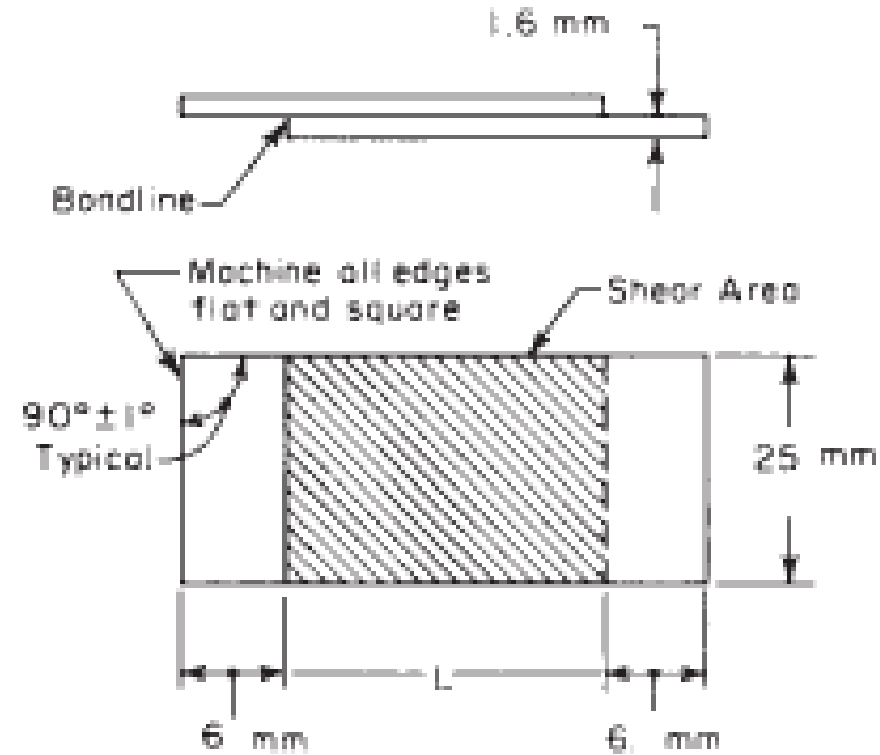
Diffusion dominated durability [2]



- A—Slotted bolt
- B—Bushing
- C—Spring—piano wire cylindrical helical compression spring with six active coils, eight total coils, wound closed, ground square, and cadmium plated
- D—Washer—22.25-mm OD, 12.7-mm ID, 1.6 mm thick
- E—Nut— M12
- F—Test specimen

Assembly of Compression Creep Test Apparatus

Diffusion dominated durability [2]



Form and dimensions of test specimen

Diffusion dominated durability [3]

ASTM D 2294: Standard Test Method for Creep Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal)

This test method covers the determination of the creep properties of adhesives for bonding metals when tested on a standard specimen and **subjected to certain conditions of temperature and tensile stress** in a spring-loaded testing apparatus. This test method is applicable to the temperature range from -55°C to $+260^{\circ}\text{C}$.

This test method is useful in research and development for comparison of creep properties of adhesives, particularly as those properties are affected by changes in adhesive formulation or expected service conditions, including temperature, moisture level and duration of loading.

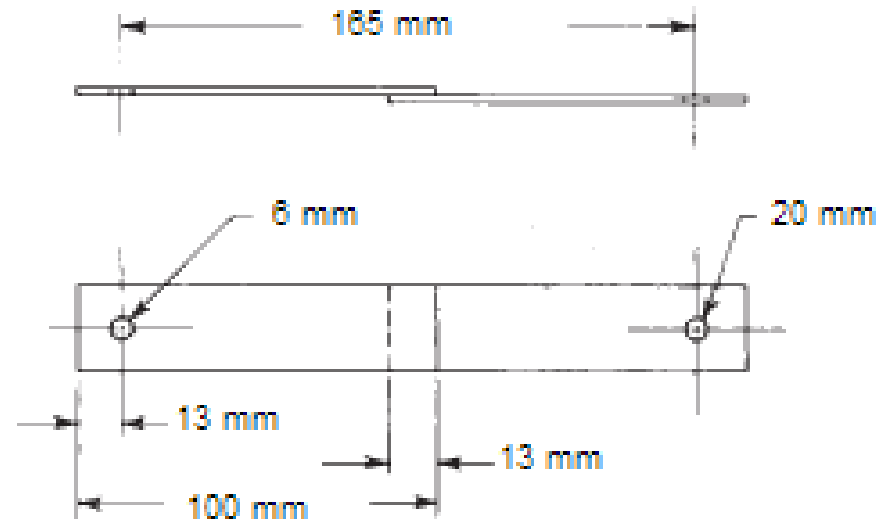
Diffusion dominated durability [3]

Procedure:

- 1.) Attach the test apparatus to a testing machine and condition to a prescribed test temperature. Place a specimen within the load chamber of the test apparatus (Slide 13) and attach to the chamber and load shaft by means of pins.
- 2.) Apply the load by the test machine at a rate of 8-10 MPa/cm²/min. After reaching the desired load, turn up the knurled supporting ring to make contact with the disk (that is, touch plus 1/4 turn) supporting the compressed spring. Unload the testing machine, remove the entire creep test apparatus (except the loading yoke) from the testing machine, and place it in the desired environment.
- 3.) To measure total deflection, observe the average displacement of fine razor scratches across the centers of both sides of the lap joints with a calibrated microscope having 100 × magnification.

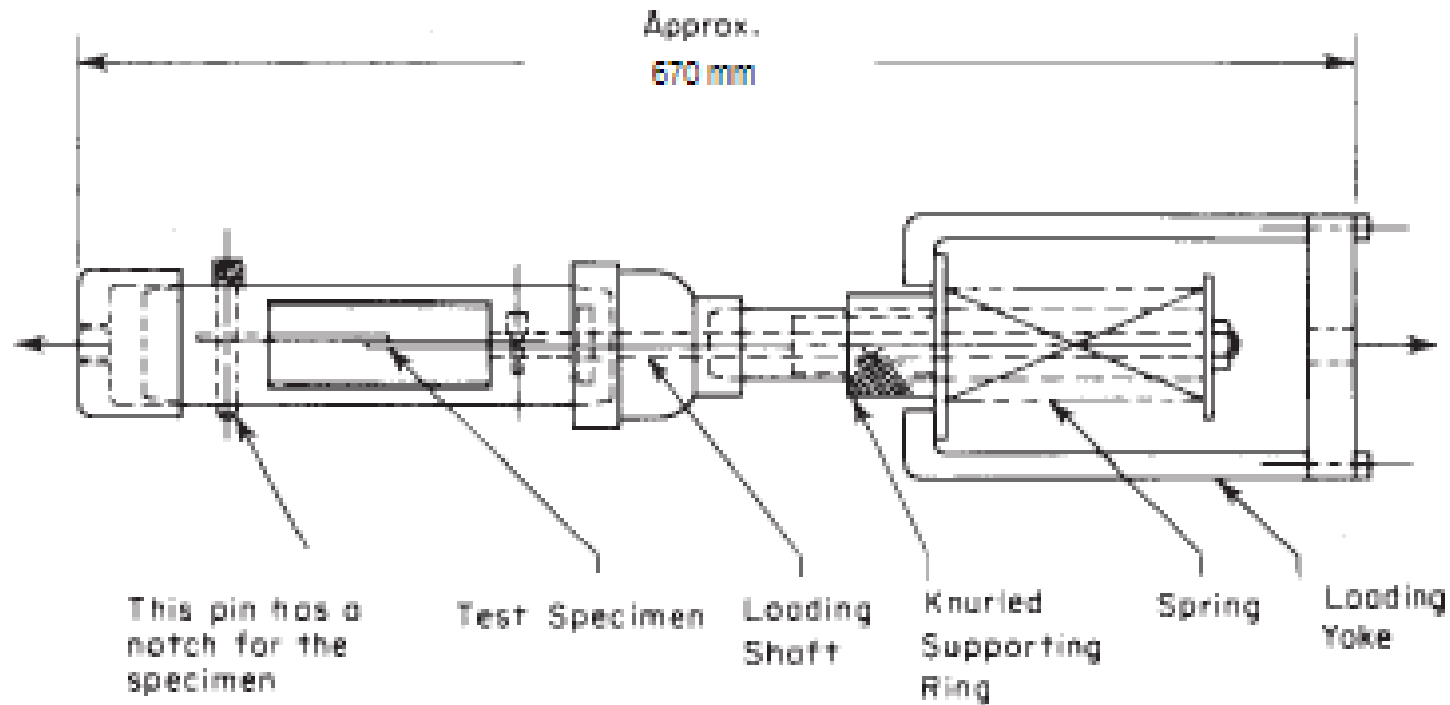
Diffusion dominated durability [3]

4.) Record the deflection at periodic intervals during the test, the total deflection, the magnitude and duration of the tensile stress, and the test temperature for each specimen. Express all loads in [Mpa] and if possible, report to three significant figures.



Form and dimensions of test specimen

Diffusion dominated durability [3]



Tension Creep Test Apparatus

Adhesion dominated durability ^[1]

Wedge test

An effective method of evaluating the resistance of an adhesive bond is by cleavage at constant displacement. **Wedge test is the most effective method of evaluating bond adhesion.** It has been adopted by many specialists and is well adapted to the high strength of modern structural adhesives. **The length of fracture is directly related to the fracture energy. The wedge test gives information not only on the quality of the bond but also on its durability.**

The thickness of the adhesive is not of importance as long as it is thin. It has been shown that in the range of 50-200 μm there was no influence of the adhesive thickness.

Adhesion dominated durability [4]

EN 14444: Structural Adhesives – Qualitative assessment of durability of bonded assemblies – Wedge rupture test (ISO 10354 modified)

This standard simulates in a qualitative manner the forces and effects on an adhesive-bonded joint at metal-adhesive/primer interface. It can also be used as a method of checking the surface preparation of adherends, with a limited detection level. **The test is applicable to the adhesive bonding of aluminium and titanium alloys using thermosetting structural adhesives and primers.** Consideration is given to the thickness and rigidity of the adherends.

The same test is specified in ASTM D 3762: Standard Test Method for Adhesive-Bonded Surface Durability of Aluminum (Wedge Test).

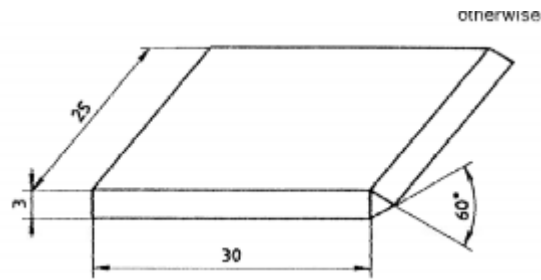
Adhesion dominated durability [4]

Principle: The test consists of the progressive introduction of a wedge between two plate adherends, bonded together with the adhesive under test. **The length of the crack thus produced is measured**, as is any propagation of the crack caused by laboratory ageing tests.

Apparatus:

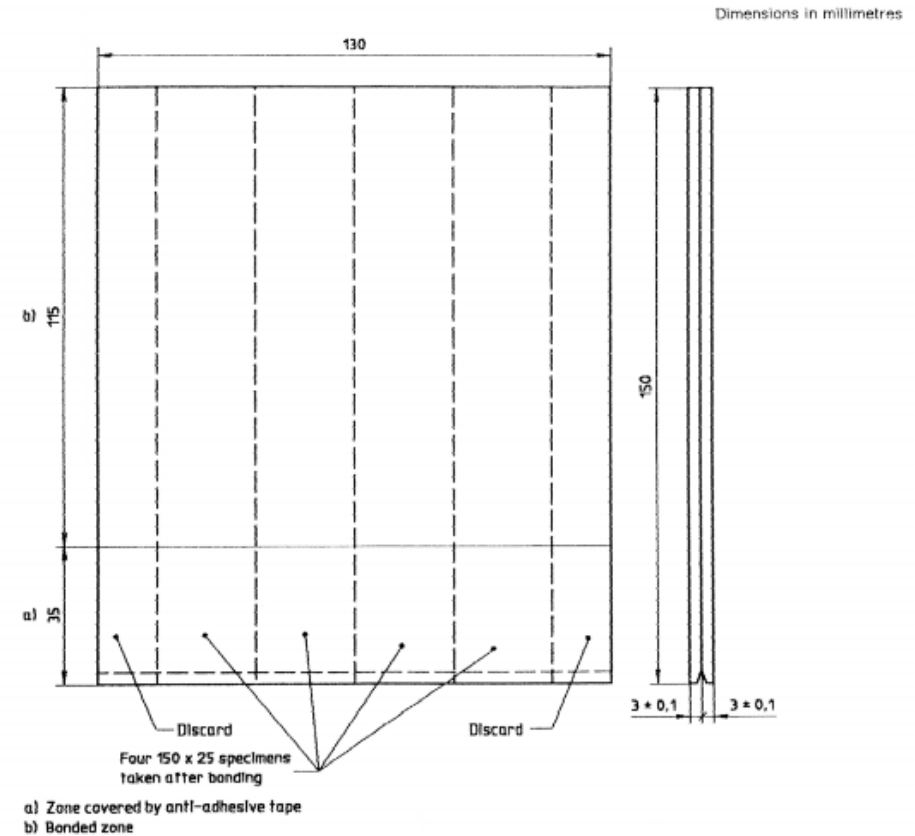
- 1.) Stainless steel wedge
- 2.) Device for driving the wedge, which provides slow and regular penetration (30 ± 5) mm/min, but excludes the possibility of oblique penetration of the wedge
- 3.) Binocular magnifying glass (magnification $10 \times$ to $40 \times$)
- 4.) Stopwatch, accurate to nearest 1 sec.
- 5.) Micrometer, accurate to nearest 0,01 mm

Adhesion dominated durability [4]



Dimensions of wedge [mm]

Dimensions of bonded plate from which specimens are cut

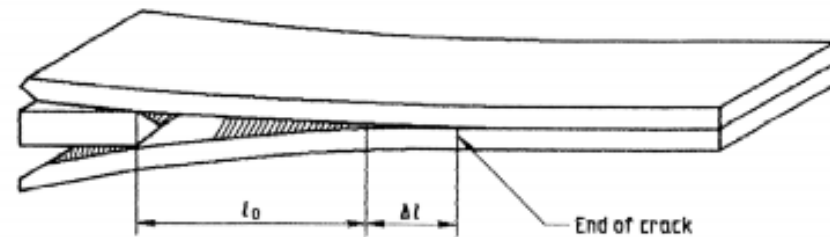
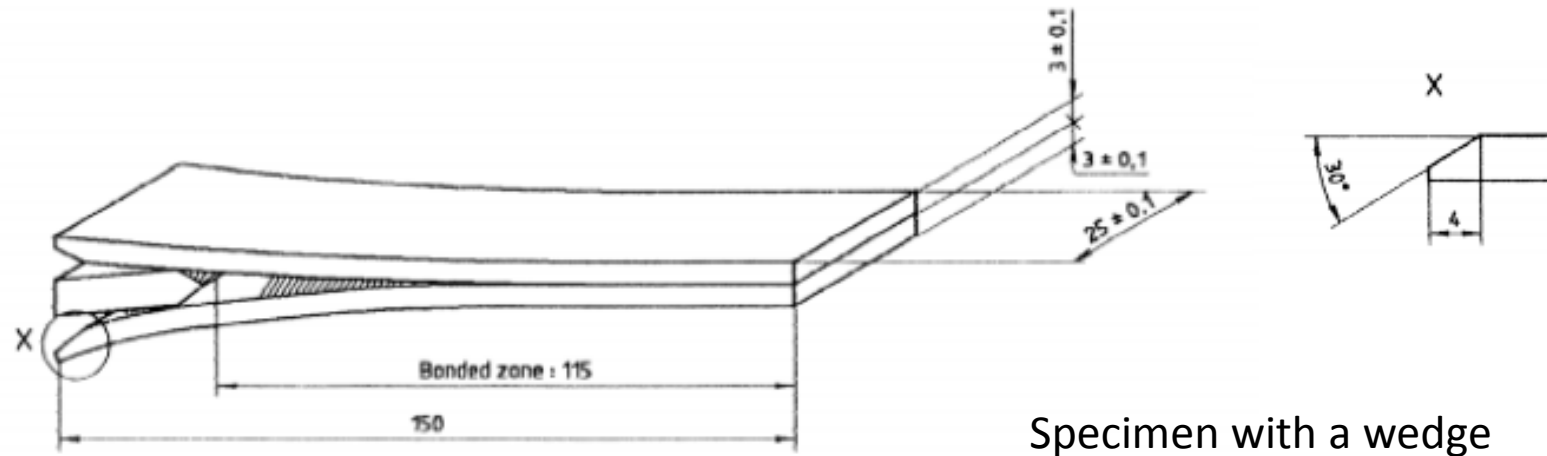


Adhesion dominated durability [4]

Determination: For each specimen drive the wedge fully home with device. Push the wedge into the joint to permit slow and regular penetration. Positioning the wedge so that the end and sides are approximately flush with the sides of the specimen. Determine the position of the crack on each side of the specimen by examination with the binocular magnifying glass and mark it.

Measure the initial crack length [mm], from wedge/adherend contact point to the mark, on each side of the specimen. Note the mean initial crack length L_0 . **Age the specimen under one of the sets of conditions specified in ISO 9142.** After ageing allow specimen to stand for (60 ± 10) min at ambient conditions to stabilize. Measure the final length L_F [mm] of the crack on each edge of the specimen.

Adhesion dominated durability [4]



Evaluation of crack propagation

Adhesion dominated durability [5]

Wet peel test

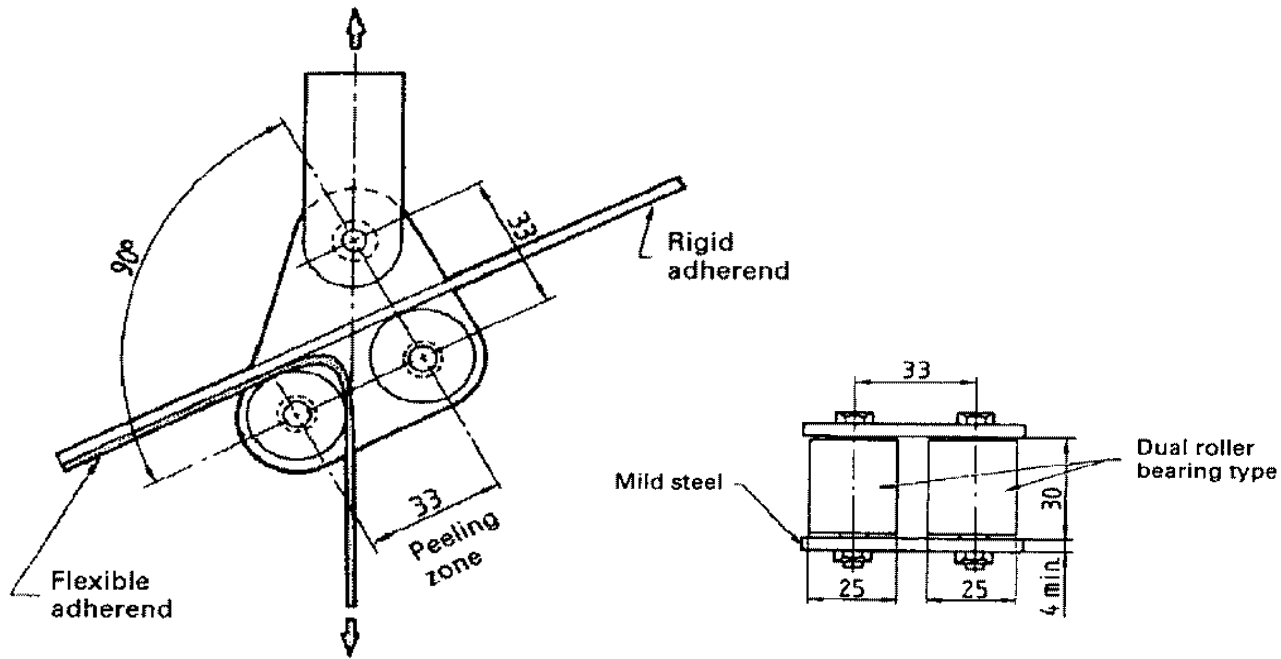
EN 1464 Adhesives – Determination of peel resistance of adhesive bonds – Floating roller method

Floating roller method for the determination of the peel resistance of adhesive bonded joints between one rigid adherend and one flexible adherend when tested under specified conditions produces more constant numerical data than other peel methods. Flexible adherend shall conform to the surface of the roller.

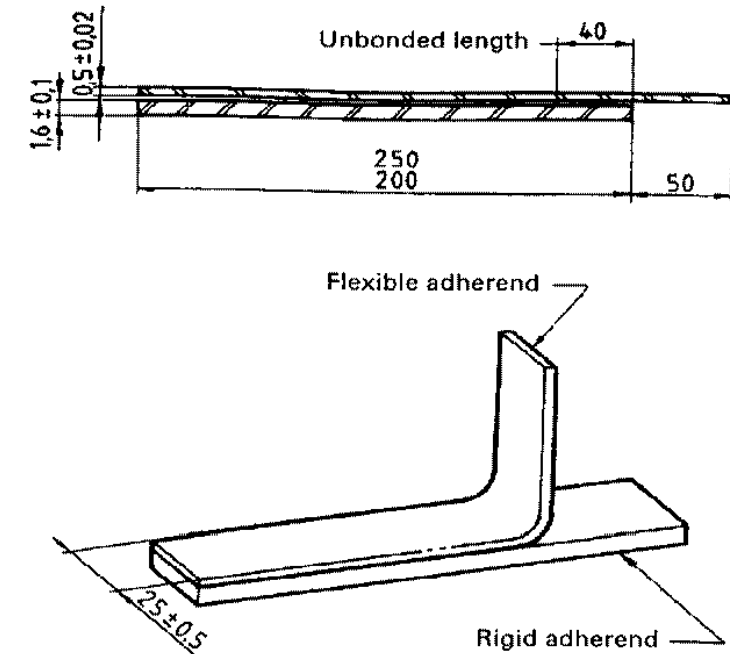
Apparatus:

- 1.) Tensile testing machine with constant crosshead rate of 100 mm/min
- 2.) Peel test fixture (Slide 21)

Adhesion dominated durability [5]



Peel test fixture



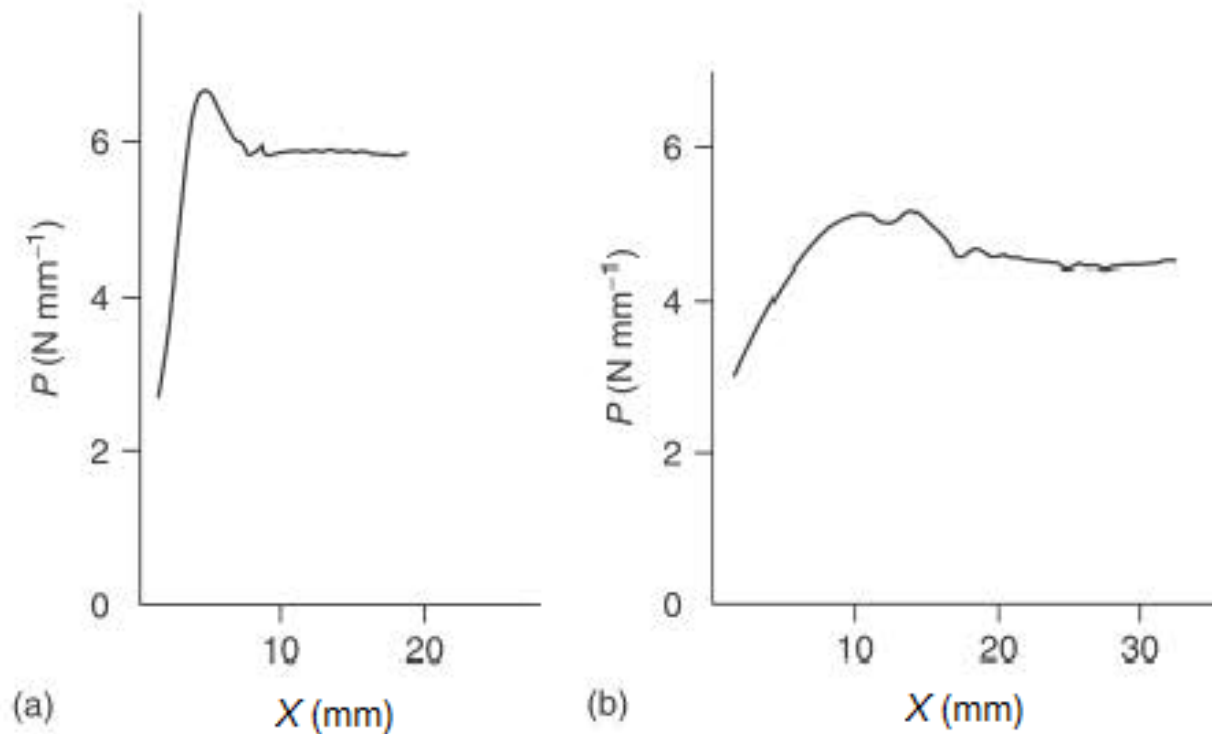
Example of test specimen

Adhesion dominated durability [5]

Test specimens (Slide 21) may be prepared individually or cut from bonded panels. The adherends and the surface pretreatment shall be in accordance with the intended application and process. The thickness of the flexible adherend shall be $(0,5 \pm 0,02)$ mm and that of the rigid adherend shall be $(2,5 \pm 0,1)$ mm.

Procedure: Insert the test specimen into the peel test fixture (Slide 21) with unbonded end of the flexible adherend gripped in the jaw of the tensile testing machine. Peel the specimen at a constant crosshead rate of (100 ± 5) mm/min. Stop the crosshead after peeling about 75 mm of the bonded length. **Then apply several drops of water containing a wetting agent to the crack opening (total volume $(1 \pm 0,5)$ mL). The solution may typically contain 0,5 – 1 % detergent.** After the application of this liquid, the peeling process shall be immediately commenced. The test shall be continued until the complete sample is peeled. **Recording of force versus crosshead distance peeled shall be made. From this record, average peeling resistance P in [N/mm] of the test specimen width (required to separate the adherends) shall be determined.**

Adhesion dominated durability [1]



Peel load P [N/mm] versus peeled distance for floating roller test with adherends: a.) AW-3103-0; b.) AW-2024-T3

Corrosion dominated durability [6]

Salt spray test

Saline solutions are also commonly employed by immersion or spray to mimic sea, coastal, or road conditions. Initially developed to evaluate the corrosion resistance of coatings for metals, the salt spray test can be used to evaluate the durability of adhesive bonded assemblies. The apparatus for testing consists of a closed testing chamber, where a salted solution most commonly, a solution of sodium chloride (NaCl) is sprayed by means of a nozzle. This produces a corroding environment in the chamber and thus, pieces in it are attacked under this severe corroding atmosphere. Testing periods can range from a few hours (e.g., 8 or 24 h) to more than a month before the determination of physical properties for comparison with their initial values.

Corrosion dominated durability [6]

Salt spray testing is popular because it is cheap, quick, well standardized and reasonably repeatable. There is, however, only a weak correlation between the duration in salt spray test and the expected life in service, since the corrosive processes involved are very complicated and can be influenced by many external factors. Nevertheless, salt spray test is widely used in the automotive, construction and aerospace industries.

Important international standards for salt spray testing:

- ISO 9772: Corrosion tests in artificial atmospheres – Salt spray tests
- ASTM B 117: Standard Practice for Operating Salt Spray (Fog) Apparatus

Corrosion dominated durability [6]



Adhesive bond line after salt spray testing (duration 480 h in 5 % NaCl aqueous solution at +25°C)

Bibliography

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| [1] | L.F.M. da Silva, D.A. Dillard, B. Blackman, R.D. Adams, Ed.: Testing Adhesive Joints – Best Practices, Wiley-VCH Verlag, 2012 |
| [2] | ASTM D 2293-96: Standard Test Method for Creep Properties of Adhesives in Shear by Compression Loading (Metal-to-Metal) |
| [3] | ASTM D 2294-96: Standard Test Method for Creep Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) |
| [4] | EN 14444:2005 Structural Adhesives – Qualitative assessment of durability of bonded assemblies – Wedge rupture test (ISO 10354 modified) |
| [5] | EN 1464:2010 Adhesives – Determination of peel resistance of adhesive bonds – Floating roller method |
| [6] | A. Öchsner, R. D. Adams, L. F. M. da Silva, Ed.: Handbook of Adhesion Technology, Springer, 2011 |