AdTech
European Harmonized Training for Adhesive Bonding Technology

EUROPEAN ADHESIVE ENGINEER

MODULE 4.9

DURABILITY- WEATHERING AND AGEING EFFECTS ON ADHESIVE JOINTS

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ERASMUS + REFERENCE 2015 -I- PT 01- KA202 - 012915

Co-funded by the Erasmus+ Programme of the European Union

This project has been co-funded with support from the European Commission.
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4.9 Weathering and Ageing Effects on Adhesive Joints

Scope:

- ✓ Definition of an environment
- ✓ Short term testing
- ✓ Long term testing
- ✓ Comparison of short and long term testing
- ✓ Environmental effects (moisture, water, chemicals)
- ✓ Combined effects

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Definition of an environment [1]

Types of environments

- Natural weathering (outdoor) in different types of environments (vacuum, UV-radiation, seacost, marine, industrial)

- Simulated weathering (indoor) in laboratory conditions (acceletated testings)

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Definition of an environment [2]

Adhesives are generally not significantly affected by moderate outdoor weathering. However, there are certain circumstances that could affect the permanence of joints exposed to outdoor service. It is important that these be considered early in the design of the adhesives joint and selection of materials.

By far the most detrimental factors influencing adhesives aged in a non-sea coast environment are heat and humidity. Near the sea coast, however, salt water and salt spray must also be considered when designing an adhesive joint. Thermal cycling, oxygen, ultraviolet radiation, and cold are relatively minor factors with most structural adhesives.

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Definition of an environment [2]

Non-sea coast environment

When exposed to weather, structural adhesives may rapidly lose strength during the first 6 months to 1 year. After 2–3 years the rate of decline usually levels off at strength that is 25–50 % of the initial joint strength depending on the climate zone, adherend, adhesive and stress level.

Adhesive systems that are formulated specifically for outdoor applications show little strength degradation over time in a moderate environment.

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Definition of an environment [2]

The following generalizations are of importance in designing a joint for outdoor service:

- The most severe locations are those with high humidity and warm temperatures.
- Stressed panels deteriorate more rapidly than unstressed panels.
- Stainless-steel panels are more resistant than aluminum panels because of corrosion.
- Heat-cured adhesive systems are generally more resistant than room temperature cured adhesive systems.
- For the better adhesives, unstressed bonds are relatively resistant to severe outdoor weathering, although all joints will eventually exhibit some strength loss.

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Definition of an environment [2]

Sea coast environment

For most bonded metal joints that are exposed to outdoor service, corrosive environments are a more serious problem than the influence of moisture. The degradation mechanism is corrosion of the metal interface resulting in a weak boundary layer.

Surface preparation methods and primers that make the adherend less corrosive are commonly employed to retard the degradation of adhesive joints in these environments.

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Definition of an environment [2]

The quickest method to measure corrosion dominated degradation in bonded metal joints is to store the bonded parts in a salt spray chamber with a continuous 5 % NaCl salt solution spray in 95 % RH at +35°C.

Usually only several hundred hours of exposure are needed to show significant differences in corrosion resistance of various adhesive joint systems. Resistance of the adhesive joint to salt climates depends not only on the type of adhesive, but on the method of surface preparation and on the type of primer used.

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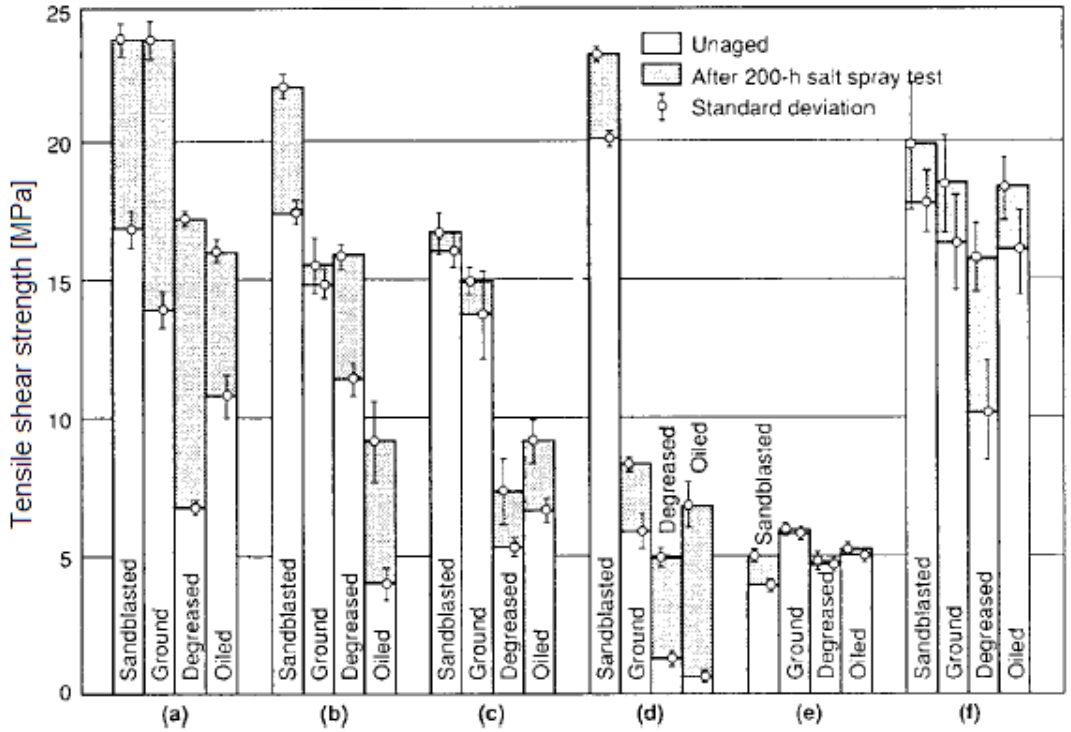
Definition of an environment [2]



a.) Two-part cured epoxy
b.) Two-part acrylic
c.) Anaerobic acrylic
d.) Cyanoacrylate
e.) PVC plastisol
f.) One-part heat cured epoxy

Shear strength of mild steel adhesive bonded joints, exposed to 200 h salt spray test at +35°C

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Short term testing [1]

Short-term testing is carried out at duration of maximim 1 year (52 weeks) at the laboratory conditions.

Accelerated ageing is required to assist the process of determining long-term performance from short-term testing. A number of techniques have been employed to accelerate testing of adhesives joints.

Short-term tests at high stress levels and high temperatures are generally set-up first to check the procedure and to identify technical issues that may not have been apparent prior to testing.

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Short term testing [4]

Selection of standard laboratory ageing conditions for testing bonded joints – ISO 9142

This standard describes laboratory ageing conditions under which adhesive joints may be exposed to various environmental influences (climatic or chemical) for the purpose of assessing the effects of such influences on certain properties. The ageing conditions are applicable to bonded assemblies and may be used to constitute a set of tests to the evaluation of an adhesive.

The results obtained using the procedures described in this standard are not necessarily applicable to the determination of the service life of a bonded assembly because there is no direct relation between the test results and the behaviour of a bonded assembly over a period of time under service conditions. However, for certain specific applications, experience with the procedures may enable a correlation to be established.

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Short term testing [4]

Apparatus for performing short term-testing of adhesive bonded joints:

- **Conditioning chamber**, meeting the requirements of ISO 483, capable of being maintained at a temperature of +(23 ± 2) °C and a relative humidity (RH) of (50 ± 5) %.
- **Dry-heat chamber**, ventilated and adjustable to a temperature between +20 °C and +200 °C.
- **Humid chamber**, meeting the requirements of lSO 483, adjustable to a relative humidity between 25 % and 100 %.
- **Cold chamber**, adjustable to temperatures of (− 20 ± 3) °C and (− 40 ± 3) °C.
- **Controlled-pressure chamber**, capable of operating at a pressure of 600 kPa (6 bar) and at an underpressure of 92 kPa (0,92 bar).

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Short term testing [4]

Procedures:

A.) Tests before ageing: Specimens shall be conditioned in the conditioning chamber for a minimum duration of 24 h before being subjected to ageing. The conditioning shall be carried out after the adhesive has been cured in accordance with the materials specification or the methods specified by the manufacturer of the adhesive.

B.) Ageing: Expose conditioned specimens to the environmental conditions as defined in Annex A to Annex F, as required.

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Short term testing [4]

C.) Tests after ageing: Determine the properties after ageing in accordance with the procedures of the appropriate standards. Unless there are indications to the contrary in the Annexes, condition test specimens in the conditioning chamber before determining the properties:

- for (3 ± 1) h in the case of specimens exposed at a relative humidity higher than 50 %,
- for at least 24 h for all other specimens.

Expression of results: For properties that can be measured, the results obtained are expressed as either absolute values or relative variation with respect to the initial value (e.g. tensile shear strength). For properties that cannot be measured, the nature of the effects observed shall be noted, e.g. rupture between the substrate and adhesive, failure of cohesion of the adhesive joint due to partial or total corrosion of the adherend beneath the adhesive, etc.

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Short term testing [4]

General conditions of ageing:

Preferred temperatures:

-40, -20, +20, +23, +30, +40, +50, +55, +60, +70, +90, +100, +105, +125, +175°C

Preferred relative humidities (RH):

15 %; (25 ± 5) %; (50 ± 5) %; (65 ± 5) %; 90 %-100 %.

Preferred exposure periods (cycles):

Hours: 1 — 2 — 3 — 4 — 6 — 16 — 24 — 48 — 72 — 96

Weeks: 1 — 2 — 3 — 4 — 6 — 8 — 12 — 16 — 26 — 52

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Short term testing [4]

Atmospheric conditions for single-variable ageing

In this type of ageing, the effect on the test specimen of only one environmental variable (temperature) is considered, the other variables being fixed. Specimens are exposed to dry heat in dry-heat chamber or to cold in cold chamber. The temperature and period of exposure being selected from the lists of preferred values from the slide 15.

Atmospheric conditions for multi-variable ageing

In this type of ageing, the simultaneous effect of two or more environmental variables on the specimen is considered. In some cases, transfers from one chamber to another are necessary; unless otherwise specified, such transfers shall be carried out in less than 3 minutes.

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Short term testing [4]

The specimen is exposed in the humid chamber to the simultaneous action of heat and humidity. The temperature, exposure period and humidity are selected from the lists of preferred values from the Slide 15. A further-accelerated ageing test using humid heat may be carried out at +(70 ± 2) °C and 90 % - 100 % relative humidity for 1000 h.

Atmospheric conditions for cyclic ageing

In this type of ageing, the specimen is exposed for several successive periods, under single-variable and/or multi-variable conditions, on a cyclic basis. In some cases, transfers from one chamber to another are necessary; unless otherwise specified, such transfers shall be carried out in less than 3 minutes.

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Short term testing [4]

The following cycles may be used:

Cycle D1 — Heat and humidity cycle
Cycle D2 — Heat, cold (thermal shock) and humidity cycle
Cycle D3 — Heat, cold and humidity cycle
Cycle D4 — Humid-heat and cold cycle
Cycle D5 — Dry-heat, humid-heat and cold cycle
Cycle D6 — Immersion in water and drying
Cycle D7 — Alternating immersion in water and drying
Cycle D8 — Immersion in boiling water and drying
Cycle D9 — Immersion in boiling water followed by drying, and then dry heat and re-immersion in boiling water
Cycle D10 — Immersion in boiling water followed by drying, and then re-immersion in boiling water
Cycle D11 — Immersion in water, exposure to cold and exposure to dry heat
Cycle D12 — Immersion in salt solution and drying

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Short term testing [4]

Example: Cycle D2 — Heat, cold (thermal shock) and humidity cycle

This cycle requires three chambers, as follows:

- a humid chamber A , kept at a temperature θ_A of either +(40 ± 2) °C or +(70 ± 2) °C and at a relative humidity not lower than 90 %;

- a cold chamber B, kept at a temperature θ_B of either (– 20 ± 3) °C or (– 40 ± 3) °C;

- a humid chamber C, kept at a temperature θ_C of either +(70 ± 2) °C or +(100 ± 2) °C and at a relative humidity of (50 ± 5) %.

The cycle consists of the following exposure periods:

a) (16 ± 1) h in chamber A;

b) (3 ± 1) h in chamber B;

c) (5 ± 1) h in chamber C.

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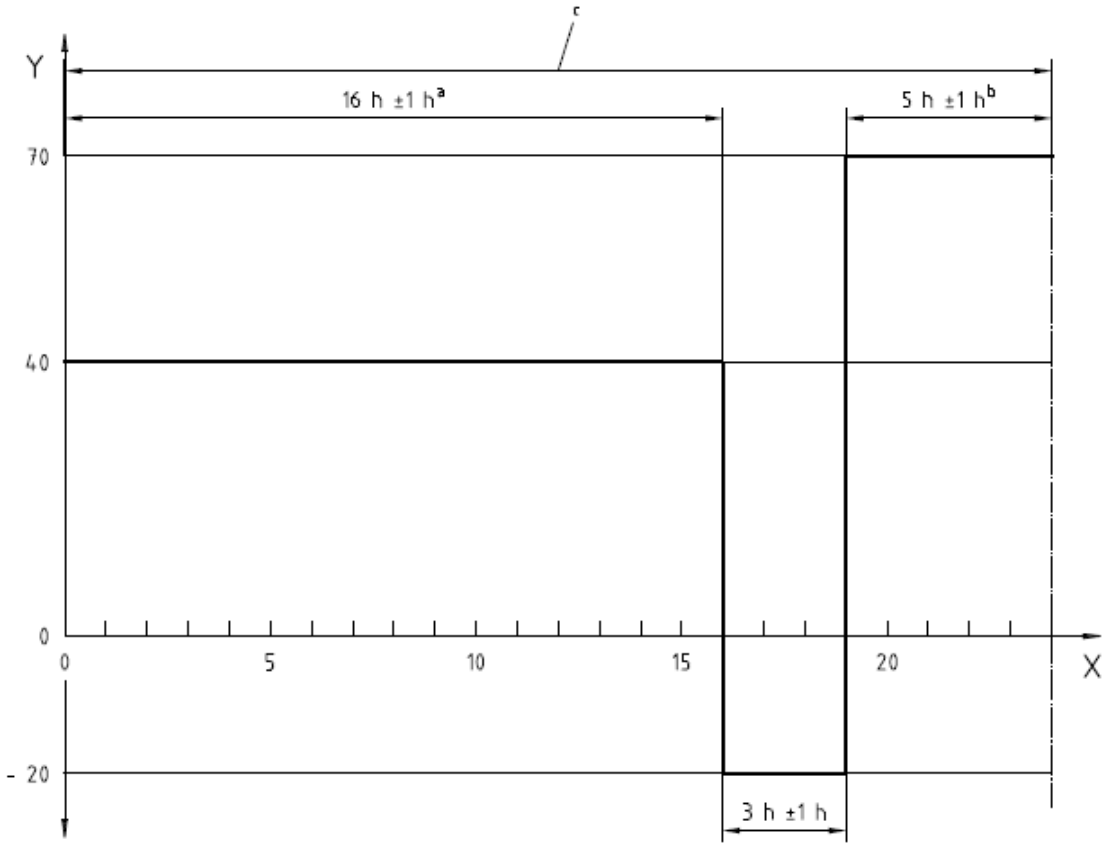
Short term testing [4]



X exposure period (h)
Y temperature (°C)
[a] Not less than 90 % R.H.
[b] (50 ± 5) % R.H.
[c] Cycle repeated *n* times

Typical conditions for cycle D2, in which θ_A, θ_B and θ_C are taken as +40° C, −20° C and +70° C, respectively

Short term testing [4]

Special procedures of short-term testing

1.) Exposure to neutral salt spray: Expose the specimens to salt spray under the conditions specified for the NSS (neutral salt spray) test in ISO 9227. Condition the exposed specimens at +(23 ± 2) °C and (50 ± 5) % R.H. for 24 h before measuring the property of interest.

2.) Exposure to laboratory light sources: Expose the specimens to artificial light in accordance with the relevant part of ISO 4892.

3.) Exposure to neutral salt spray and elevated temperature and humidity: Expose the specimens to salt spray under the conditions specified for the NSS (neutral salt spray) test in ISO 9227. After this, transfer the specimens to a humid chamber, kept at +(40 ± 2) °C and a relative humidity between 90 % - 100 % for a further exposure period. Condition the exposed specimens at +(23 ± 2) °C and (50 ± 5) % R.H. for 24 h before measuring the property of interest.

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Short term testing [5]

ASTM D 1183: Standard Practice for Resistance of Adhesives to Cyclic Laboratory Ageing Conditions

These practice cover the determination of the resistance of adhesives to cyclic accelerated service conditions by exposing bonded specimens to conditions of high and low temperature and high and low relative humidities. The extent of degradation is determined from changes in mechanical strength properties as a result of exposure to the test conditions.

It is recognized that no accelerated procedure for degrading materials correlates perfectly with actual service conditions, and that no single or small group of laboratory test conditions will simulate all actual service conditions. These practices/conditions are intended for use with specimens described in the approved mechanical strength test methods for adhesives.

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Short term testing [5]

Apparatus for performing cycling laboratory ageing testing of adhesive bonded joints:

- **Circulating Air Ovens,** capable of being controlled at the required temperatures.
- **Rooms, Cabinets or Desiccators,** with means for controlling the relative humidity (RH) of the air in them at the required values.
- **Vessels with Water,** for immersion tests.
- **Substitute Ocean Water** according to ASTM D 1141.
- **Other Apparatus** for the particular strength property used to evaluate the extent of degradation.

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Short term testing [5]

Procedures:

1.) Condition all specimens for 7 days at a relative humidity of (50 ± 2) % at +(23 ± 1)°C.

2.) Subject specimens or panels to the number of cycles of one of the test conditions listed in Slide 25, as specified in the material specifications.

3.) After completion of the exposure of the test specimens or panels to one of the test conditions in Slide 25, condition the test specimens or panels for 7 days at a relative humidity of 50 % at +(23 ± 1)°C and then test them immediately for the specified strength properties, unless otherwise stated in the material specifications.

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Short term testing [5]

Test Condition Designation	Name	Period, h	Temperature °C	Relative Humidity, %
A	Interior	24	23 ± 1	85 to 90
		24	48.5± 3	<25
		72	23 ± 1	85 to 90
		48	48.5 ± 3	<25
B	Interior	48	60 ± 3	<15
		48	38.5 ± 2	85 to 90
		8	− 18 ± 2	about 100
		64	38.5 ± 2	85 to 90
C	Exterior, land and air	48	71 ± 3	<10
		48	23± 1	immersed in water
		8	−57 ± 3	about 100
		64	38.5 ± 2	about 100
D	Exterior, marine	48	71 ± 3	<10
		48	23 ± 1	immersed in substitute ocean water
		8	−57 ± 3	about 100
		64	23 ± 1	immersed in substitute ocean water

Test conditions for cyclic laboratoray ageing of adhesive bonded joints according to ASTM D 1183

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Long term testing [6]

Long-term testing is carried out at duration of minimum 1 year at the outdoor conditions.

ASTM D 1828: Standard Practice for Atmospheric Exposure of Adhesive-Bonded Joints and Structures

The atmospheric exposure tests described in this practice will evaluate the stability of the adhesive bond only in terms of a particular natural atmosphere. Since the atmospheric conditions vary greatly from year to year, these results will not be as reproducible as those derived from laboratory aging procedures.

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Long term testing [6]

Considerable research has shown that laboratory artificial weathering tests will not give consistently good correlation with outdoor test exposures.

Suitable test methods for evaluation of the effects of exposure include nondestructive qualitative or quantitative observations on the same sample at prescribed intervals, or destructive tests on separate sets of specimens (tensile shear strength).

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Long term testing [6]

Procedure:

1.) Prior to exposure, condition all samples at the standard conditions of +(23 ± 1)°C and (50 ± 2) % relative humidity for not less than 40 h. Then one part of samples expose to long term testing, and second part keep in the above conditions until the conclusion of the exposure period.

2.) Measure the dimensions and note the appearance of the test specimens in accordance with the exposure test specifications and record these values.

3.) Test for a minimum of one year, unless a shorter time is necessary because of the lack of durability of the adherends. Where tests of less than one year duration are to be used, start exposure in the spring and continue for the first few weeks of cool weather. In tropical areas where climatic conditions are more uniform throughout the year, the time of year when short-term exposure is carried out may be less critical. In all localities the longer period of exposure produces more reliable results.

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Long term testing [6]

4.) After exposure, again note and record the appearance of the specimens. When performing other than visual appearance tests, condition the specimens for not less than 40 h at +(23 ± 1)°C and (50 ± 2) % relative humidity. Measure the dimensions, including warpage if any, of the specimens after the conditioning period just prior to testing.

5.) Measure the dimensions, including warpage if any, of the stored control specimens, and test these specimens at the conclusion of the exposure period along with the exposed specimens.

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Comparison of short and long term testing [3]

It is often possible to establish the timescale of the longer term tests on the basis of these short term results. This is important where measurements need to be completed with in a set period of time, as specified in a number of standards.

Testing was carried out at 3-24 months at water-soak exposures and 3-96 months in industrial atmospheres. Specimens were made from AW-6061-T6 adherends jointed with on one-part and two-part epoxies. Overall deterioration of all differently treated joints after 2 years in water soaking comparing to 8 years in the industrial atmospheres do not reflect the wide variance in different degrees of life predictability.

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Comparison of short and long term testing [3]

It can be possible to calculate an „acceleration ratios“ for each different combination of of adherend, adhesive, surface pretreatment conditions and weathering conditions.

An overall average retention in the 2 year water immersion environment was 76 % as compared with an overall average strength retention of 88 % for the same collective joints after 8 years in the industrial atmosphere.

- average acceleration ratio for one-part, heat cured epoxy: 31
- average acceleration ratio for two-part, room-temperature cured epoxy: 6

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Comparison of short and long term testing [7]

Structural Adhesives - Mechanical behaviour of bonded joints subjected to short and long terms exposure at specified conditions of temperature (EN 14258:2004)

This document specifies how adhesively bonded joints can be charracterised:

- at different temperatures,
- after having been submitted to continuous exposure under specific conditions of time and temperature.

Characterisation can be determined on different standardised shear specimens. Due to diversity of possible applications and interactive influences of different adhesive adn adherend properties, it is not appropriate to define or determine exposure performance limits.

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Comparison of short and long term testing [7]

Apparatus:

- Machines for performing shear tensile tests at room temperatures,
- Tensile test machine for short-term testing: Equipped with an environmental chamber suitable for conditioning and testing the specimens at desired temperature,
- Environmental chamber for long-term testing

Procedure for short-term testing:

1.) Before testing all specimens shall be conditioned for 1 day at +(23 ± 2)°C and relative humidity of (50 ± 5)%.

2.) The temperature should be chosen according to the specific application and according to ISO 9142 (see Slide 15). The specimen shall be held at the relevant temperature until temperature equilibrium is reached.

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Comparison of short and long term testing [7]

Procedure for long term thermal exposure testing:

1.) Unaged reference specimens shall be prepared and evaluated at +(23 ± 2)°C

2.) An exposure time shall be chosen according to the specific applications. The specimens shall be conditioned at the desired temperatures in a suitable environmental chamber, accurately monitored and controlled for the desired periods of time.

3.) After thermal ageing all specimens shall be conditioned at +(23 ± 2)°C and a relaitev humidity of (50 ± 5)% for 24 h prior to testing. The shear specimens shall be evaluated according to the standard test methods for tensile shear strength testing at +(23 ± 2)°C .

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[3]	J. D. Minford: Handbook of Aluminum Bonding Technology, Marcel Dekker, 1993
[4]	ISO 9142:2003 Adhesives - Guide to selection of standard laboratory ageing conditions for testing bonded joitns
[5]	ASTM D 1183-03: Standard Practices for Resistance of Adhesives to Cyclic Laboratory Aging Conditions
[6]	ASTM D 1828-01: Standard practice for Atmospheric Exposure of Adhesive Bonded Joints and Structures
[7]	EN 14258:2004 Structural adhesives - Mechanical behaviour of bonded joints subjected to short and long terms exposure at specified conditions of temperature

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