

Adhesive Testing: A Necessary Cost

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Introduction

Testing is an extremely critical function in the adhesive and sealant industry for several reasons. One cannot reliably predict the characteristics of the joint based solely on parameters of the adhesive, substrate, and joint design. Therefore, testing is necessary to confirm expectations. The performance of the adhesive bond is often variable and will depend on the consistency of the materials and processes utilized. Therefore, test methods are also important to ensure reliability and uniformity in the bonding process. Importantly, testing and test results are also a means of communication between the adhesive formulator and the end-user.

This article reviews the importance of testing in the adhesive and sealant industry and why it is a necessary cost that must be dealt with by both the adhesive formulator and end-user. The importance of standardized test methods is emphasized as well as the process of designing an adequate test program. Fundamental material property tests, tests to determine the working characteristics of the adhesive, and performance tests are identified but not expounded in detail. Most of these test methods, their results, and unique characteristics have been discussed in previous SpecialChem articles.

Necessity of Adhesive Testing

To determine and ultimately control the strength of the joint, one must resort to testing. Through testing the many factors that can affect the strength of the joint can be separated and measured. Sometimes it is difficult to disconnect these variables, but at least through testing, a vehicle exists to provide a quantitative approximation of their consequences.

Standard test methods are very useful for comparing and determining the consistency of materials and processes. It is less valuable in accurately predicting the actual strength of specific production joints. Usually, prototype tests must be designed for this purpose.

Measuring the effectiveness or strength of adhesives has been a necessary challenge since the dawn of the industry. Originally, these test methods were crude and qualitative. Now one has modern instrumentation to quantify the characteristics of these systems and standard test methods to ensure their accuracy and reproducibility.

Adhesives tests are conducted for a variety of reasons. They are used to:

1. Choose among materials or processes, such as adhesive, adherend, joint design, or curing conditions
2. Benchmark one's formulation against a competitive formulation
3. Monitor the quality of incoming materials to make certain that they have not changed since the last time they were verified for use in the bonding process
4. Confirm the effectiveness of bonding processes, such as surface cleaning or curing
5. Investigate parameters or process variables that may lead to differences in performance (i.e., solve adhesive quality issues)
6. Communicate requirements or specifications necessary for a particular application.

There are two categories of general tests for adhesives and sealants: fundamental property tests and end-use tests. End-use tests, such as peel and shear, are those that try to simulate the type of loading and service conditions to which a joint will be subjected. These tests are relatively straightforward, but experience is required to establish the correct sample type and testing procedures, to judge the reliability of the resulting data, and to interpret the results and apply them to a practical application.

The determination of a fundamental or bulk property, such as viscosity, hardness, or setting rate, is usually simpler and more reproducible than end-use tests. However, a correlation between fundamental properties and the results of end-use testing is often difficult. Fundamental property tests are usually employed to assess the consistency of the incoming adhesive or substrate once the bonding process is verified as suitable for a specific application. Unfortunately, fundamental property testing is often done only after there is a failure or unexplained occurrence. In this way testing is used to determine if a change in the incoming material may have been the possible culprit.

Importance of Standardized Testing

It is critically important that in any testing program the procedures be standardized and broadly applicable to different types of suppliers and end-users. Tests that are performed at different locations and with different personnel should be identical. Fortunately, there are a number of organizations that have developed standard test methods for adhesives and sealants. There are approximately 900 international standards (ASTM, ISO, FINAT, etc.) used in all sectors of the adhesives and sealants industry.

In the United States, ASTM (American Society of Testing Materials) standards are the most commonly referenced test methods. However, other standard organizations are popular in certain parts of the world, and government or military organizations will often contribute test procedures. Table 1 identifies several of these international standard organizations and provides an example of their format. The International Standards Organization (ISO) tries to merge the individual regional standards into standards that can be used globally.

Several end-user organizations are also active in developing standards for adhesive and sealant testing. These are generally defined by the market in which they are operating and its unique requirements. These organizations are summarized in Table 2.

Region	Publisher	Example
United States	American Institute of Testing Materials	ASTM D1002
Australia	Australian Standards	AS 2990
UK	British Standards Institute	BS 2990
Canada	Canadian Standards Board	CGSB 25.14
Germany	Deutsche Institut fur Normung	EN 60454
Japan	Japanese Industrial Standards	JIS Z9900
Switzerland	International Standard Organization	ISO 9653
Netherlands	Netherlands Normalisatie Istutuut	NEN 10244-7
France	Norme Francaise (AS-NOR)	NF T76.103
Norway	Norges Standardiseringsforboud	NS 4828
Sweden	Standariserigns-Kommissionen I Sverige	SEN 01 03 45

Table 1: International Standard Organizations Involved in Adhesive and Sealant Testing

Industry	Organization	Example
General	American Society of Mechanical Engineers	ASME B31.3
Aerospace	Aerospace Materials Specifications, Society of Automotive Engineers	AMS 1374
Automotive	Society of Automotive Engineers	ARP 1524
Pulp and Paper	Technical Association for the Pulp and Paper Industries	TAPPI 207
Tapes and Labels	Pressure Sensitive Tape Council	PSTC
Steel	American Institute of Steel Construction	AISC M011
Concrete	American Concrete Associations	ACI 211.1
Building and Construction	American Society of Civil Engineers, American Architectural Manufacturers	ASCE 10-90 AAMA 1407.1
Plastics	Society of Plastics Institute	SPI B 151.2
Aluminium	Aluminium Association	AA53

Table 2: Professional Organizations Active in Testing of Adhesives and Sealants

Fundamental Material Property Tests

Fundamental tests can be used for monitoring the consistency of incoming products that are used in the adhesive bonding or sealing operation. These tests may be used to characterize either the adhesive, adherend, or other materials, such as primers or solvents, used in the joint or in its construction. These tests may also be referred to as bulk property tests because they define the properties of the material in the bulk form and not within the adhesive or sealant joint.

Once a combination of materials is verified to provide acceptable joints, the materials are tested and a "fingerprint" is generally made of the resulting properties. Samples taken from a subsequent lot or suspicious materials can then be subjected to testing and the results compared to the original fingerprint. A significant difference in a measured fundamental property could be evidence supporting further investigation.

Material property tests are usually employed to determine the consistency of incoming adhesive or sealant products. Table 3 lists a number of selected ASTM test standards that are used for this purpose. The most commonly used fundamental property tests measure the viscosity, softening point, solids content, filler content, specific gravity, and color.

ASTM Designation	Test Method	Comments	Use
ASTM D898	Applied weight per unit area of dried adhesive solids	Measures the quantity of adhesive solids applied in a spreading or coating operation.	W
ASTM D899	Applied weight per unit of liquid adhesives	Measures the quantity of liquid adhesive applied in a spreading of coating operation. Intended only for wood bonding.	W
ASTM D1084	Viscosity of adhesives	Measurement of viscosity of free flowing adhesives by various methods (cup and viscometer).	C
ASTM D1146	Blocking point of potential adhesive layers	Measures the blocking point of thermoplastic or hygroscopic layer of coating. Generally applicable to heat or solvent/water reactivatable adhesives.	W
ASTM D1337	Storage life of adhesive by consistency and bond strength	Measures practical shelf life by viscosity or bond strength.	W
ASTM D1338	Working life or liquid or paste adhesive by consistency and bond strength	Similar to ASTM D1337 but aimed at working life instead of storage life.	W

ASTM D1489	Nonvolatile content of aqueous adhesives	Determination of nonvolatile content on aqueous adhesives. Principally dextrin, starch, casein and animal based adhesives.	C
ASTM D1490	Nonvolatile content of urea-formaldehyde resin solutions	Determines the apparent nonvolatile content of urea-formaldehyde solutions used as wood adhesives.	C
ASTM D1875	Density of adhesive in fluid form	Cup method of determining density of liquid adhesives.	C
ASTM D1917	Penetration of adhesives	Tests for penetration of liquid adhesives into porous substrates. Generally applied to paper.	W
ASTM D2183	Flow properties of adhesives	Measures flow of adhesives that are generally used as a film or semi-solid. Information useful in determining pressure requirements for optimum bond line thickness.	W
ASTM D2556	Apparent viscosity of adhesive having shear rate dependent flow properties	Measurement of viscosity by spindle or other viscometer capable of change of rate of shear. Measurements are made for each rate of shear.	C
ASTM D3236	Viscosity of hot melt adhesive and coating materials	Determines melt viscosity using a Brookfield type viscometer. Applicable for materials showing a viscosity of 200,000 cps at 175°C.	C
ASTM D1002	Lap shear strength of an adhesive joint	Measures the ultimate strength when the adhesive joint is tested in a tensile-shear configuration.	P
ASTM D1876	T-peel strength of an adhesive joint	Measures the peel strength of an adhesive as the substrates are separated to for a "T" geometry.	P
ASTM D1062	Fatigue test	Measures the resistance of a joint to a given load that is repeatedly applied.	P
ASTM D950	Impact test	Used to determine the impact resistance of an adhesive joint. One adherend is struck with a pendulum hammer and the energy of impact at failure is reported.	P

Table 3: Examples of Standard ASTM Test Methods Used for Assuring Consistency of Incoming Adhesives and Sealants

There are several standard tests that are used to measure the working characteristics of adhesives and sealants. These tests are also often used to determine consistency of a material from lot to lot and as a means of specifying an adhesive for a specific application.

Table 3 also identifies several working characteristic standards that have been developed by ASTM. The most commonly used are shelf life, working life, and cure rate. These tests are direct measurements of the reactivity of the adhesive or sealant at various stages during the bonding process. They provide information important to the end-user regarding the time limits required for storage, application, and cure of the adhesive or sealant.

Standardized Test Method to Determine Performance of an Adhesive in a Joint

Many standardized tests have been developed to measure the performance properties of an adhesive joint. These tests include tensile-shear strength, peel strength, impact resistance, tack, heat and moisture resistance and virtually any other set of environmental conditions or types of stress that can act on an adhesive joint while it is in service. These test methods are also well documented by the various testing organizations mentioned above. Reference to the appropriate standards will adequately equip one with the background necessary to conduct the test or a version of it.

Tests on adhesive joints are only useful if they can be reproduced. It is important that the same results can be measured by both the adhesive developer and the end-user. It is also important that the results are

reproducible with time and with different testing personnel. The accuracy and reproducibility of test results depend on the conditions under which the bonding process is performed.

Unless otherwise specified, the standard conditions surrounding the specimens at least 24 hours prior to and during the test are controlled to 23 +/- 1°C and 50 +/- 4% relative humidity. The individual standard usually describes the number of samples required along with other details regarding the test procedure and how the results are to be reported.

The following variables will also influence the results of the test and, therefore, they must be strictly controlled:

1. The physical characteristics of the joint design being tested (length of overlap, bond line thickness, substrate material, substrate thickness, etc.)
2. Procedures for cleaning, etching, and drying the surfaces of the substrates prior to application of adhesive.
3. The time between surface preparation and application of adhesive and the environmental conditions present during this period. This includes the temperature and percent relative humidity. Usually standard atmospheric conditions are specified (23°C and 50% relative humidity).
4. Complete procedures for mixing the adhesive components, if more than one.
5. Conditions and methods for application of the adhesives to the substrate surface.
6. Curing conditions, including the pressure, temperature, and time of the curing cycle. It should be specified whether or not the temperature is measured within the glue line, or on some other point on the substrate, or at some location within the curing oven. The temperatures could vary significantly at these different locations depending on the weight and size of the assembly. When an adhesive producer specifies a temperature and time for cure, it refers to the conditions of the actual adhesive within the bond-line.
7. Conditioning procedures for specimens after curing and prior to testing.
8. The rate at which the sample is loaded during test. Peel and impact tests especially are very dependent on the speed at which the sample is tested.

A standard test report usually documents the resulting measurements such as tensile shear strength, peel strength, etc. It should also indicate all of the pertinent conditions that are required to assure reproducibility in subsequent testing. It is often also very useful to describe the failure mode of the tested specimens. An analysis of the type (or mode) of failure is an extremely valuable tool to determine the cause of adhesive failure. The failed joint should be visually examined to determine where and to what extent failure occurred. The percent of the failure that is in the adhesion mode and in the cohesion mode should be provided.

A description of the failure mode itself (location, percent coverage, uniformity, etc.) is often useful. The purpose of this exercise is to establish the weak link in the joint to better understand the mechanism of failure.

Designing a Test Program and “Non-Standard” Test Methods

Designing a test program to evaluate adhesives, adhesive processing methods, or joint designs can be a formidable task. Above all else, one must keep in mind that the final product will never be a T-peel specimen or a lap shear specimen. ASTM and other standard test methods are excellent tests to offer relative comparisons of adhesives and / or bonding conditions. They also offer a valuable starting point for adhesive evaluation. However, there may be better ways to get information for predicting performance of the actual joint in service. The standard test methods are well defined and, therefore, can be well controlled; but their

relationship, if any, to performance in the proposed product must be established by a well-designed test program.

The nature of the test and evaluation program requires careful thought. The critical features are that the tests must have a known relationship to the final product. Often this requires either developing creative, non-standard tests that stress the part in a mode that is more indicative of its service load, or producing actual prototype specimens with the adhesives and bonding conditions that are intended to be used in production. These prototype specimens then would be subjected to simulated service environments. The environmental exposure can be accelerated to reduce testing time. Caution needs to be exerted so that the acceleration does not cause reactions or mechanisms within the materials or bond-line that will not actually be present in the intended real environment. These "non-standard" test methods should be controlled so that the tests are repeatable and the variability is low. Among the obvious variables that need to be controlled are surface cleaning and other preparation processes, joint geometries, method and extent of material mixing, method of application, fixtures utilized, and cure conditions.

To develop a joint with an adequate service life and with a realistic design margin, the use of a "Mathes" ladder is suggested to establish a testing hierarchy. In this process, shown in Figure 1, testing proceeds from simple, standard tests of basic materials where well-defined test specifications are available to increasingly complex tests. Depending on the application and the types of information from the lower rungs of the ladder, the need for more complex testing may be reduced or even eliminated. However, the need to completely understand the simpler tests is mandatory. The unexpected failure in service is often associated with a lack of understanding of the effects of the service environment on the basic materials or on a lack of understanding of the test variables (e.g., rate of loading).

References

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