JOURNAL OF APPLIED POLYMER SCIENCE VOL. VI, ISSUE NO. 20, PAGES 217-220 (1962)

Process Control Considerations for Adhesive Bonding in Production

WILLIAM BANDARUK

Materials Department, Plastics Section, Aeronutronic Division of Ford Motor Company, Newport Beach, California

Introduction

The attractive advantages of structural adhesives were of paramount importance in the development of aerodynamically smooth surfaces for missiles and aircraft. The inherent desirable characteristics of structural adhesives were recognized; thus, intense effort was put forth both by Government agencies and industry to improve the reliability of the various adhesives which function as integrated load-bearing units. As a result of this intensive effort, an outstanding epoxy-base structural adhesive was developed and utilized on the supersonic bomber, the B-58.[1,2] Other structural adhesive systems have since found application in other vehicles such as the F102 and F106 airplanes, as well as the Atlas, Blue Scout,[3] Ranger Lunar Probe, and Shillelagh missiles. Reliable structural adhesive systems have been responsible also for the development of sandwich composites utilized as primary load-bearing members.[4,5]

The same adhesive material technology employed in advancing aerospace and aircraft concepts so effectively, can be profitably utilized in the production of ordnance items[6] such as warheads (Hawk, Hercules) and mines (antipersonnel and other types).

Discussion

Because of the rapid advances in adhesive technology, it is necessary to maintain a flexible position in selecting and utilizing the optimum adhesive and process for bonding structures. Thus, it is the objective of this paper to present a system, including process controls considerations, that will achieve adapting the best adhesive and process for bonding ordnance articles representing modern design concepts.

The system by which adhesive bonding is best applied to an ordnance item would probably follow this sequence:

The designer would see the opportunity to utilize the following inherent advantages of an adhesive: (*1*) production of smooth surfaces, (*2*) realization of uniform stress distribution, (*3*) utilization of thinner sheets of metal, (*4*) ability to fabricate complex designs, (*5*) increased resistance to fatigue, (*6*) increased resistance to vibration, and (*7*) reduction of corrosion.

The designer, in cooperation with the adhesive engineer, would determine the type of joint and adhesive which would best fulfill the purpose. The sequence of events will then be in the following order:

1. The engineering laboratory would perform test evaluation of the candidate adhesives under conditions pertinent to the design and functional requirements.

2. After the engineering laboratory has performed its function, the prototype shop would further evaluate the joint and adhesive in a subscale or prototype part.

3. The manufacturing division would assume the final responsibility of producing the end item in production quantities.

An adhesive process sequence which the manufacturing division might utilize is shown in Figure 1 of the paper by Bair, this symposium.[7] As can be seen, in addition to just the bonding aspects of the parts, there are other critical steps which are important; namely, parts preparation, surface preparation, transfer and storage of parts, assembly and inspection.

At this time a number of rigid process controls must be set up to ascertain that the bonded item will meet the engineering and functional requirements to which the ordnance item must be subjected. These process controls can be categorized as (*a*) incoming material control, (*b*) surface preparation control, (*c*) in-process control, and (*d*) final inspection.

Incoming Material Control

Incoming material control includes two types of tests; namely, physical properties such as per cent flow, gel time, and per cent volatiles that are of interest to the process engineer in assuring the quality of the bond. For example, the test on per cent flow is of value in maintaining the bonding process so the adhesive flow will not be too great, which would cause an adhesive starved bond; but too little flow would cause a thick or inadequately filled bond.

TABLE I
Some Specifications and Standard Test Methods for Metal-to-Metal Adhesives

1. MIL-A-5090D	Adhesives, heat resistant, airframe structural, metal to metal.
2. MIL-A-5433A	Adhesive; application of room temperature and intermediate temperature setting resin (phenol, resorcinol, and melamine base).
3. MIL-A-397B	Adhesive, room temperature and intermediate temperature setting (phenol, resorcinol, and melamine base).
4. MIL-A-5534A	Adhesive, high temperature setting resin (phenol, melamine, and resorcinol base).
5. MIL-A-5535A	Adhesive, application high temperature setting resin (phenol, melamine and resorcinol base).
6. MIL-A-8623A	Adhesives, epoxy resin, metal-to-metal structural bonding.
7. MIL-A-9067A	Adhesive bonded metal; process and inspection requirements.
8. ASTM D-903-49	Peel or stripping strength.
9. ASTM D-1337-56	Storage life of adhesives by consistency and bond strength.
10. ASTM D-1002-53T	Strength properties of adhesives in shear by tension loading (metal-to-metal).
11. ASTM D-1062-51	Cleavage test for metal-to-metal adhesive.
12. Federal test method standard No. 175	Adhesives; method of testing.

The mechanical properties of the incoming material are of interest since they are indicative of the structural results to be obtained in the final item.

The various tests and requirements for physical and mechanical properties for structural adhesives are adequately described in various specifications and standard methods such as shown in Table I.

Surface Preparation Control

The second step for assurance of reliable adhesive-bonded parts is surface preparation (see Table II). The fundamental principle of bonding requires that optimum surface conditions are present for the adhesive reactant groups. The preparation of the surface can greatly influence the strengths attained regardless of how carefully the adhesive has been selected and the joint designed. Presence of adsorbed gases, water, or oil on the surface can cause inconsistent results. For example, bond strengths can be reduced by as much as 50% by trace amounts of a waxy substance[8] or the wrong surface treatment.[9]

TABLE II
Tensile Shear Strength of an Epoxy Adhesive on High Strength Steel Utilizing Various Surface Treatments

Surface treatment	Tensile shear strength, psi
Trichloroethylene vapor degrease	3400
Vapor degrease, sandblast, vapor degrease	3400
Scuff sand, vapor degrease	3400
Alkaline degrease	1600
Vapor degrease, alkaline degrease	1800
Solvent wipe with ketone	3200
Oxalic-sulfuric acid	3100
Dilute hydrofluoric acid	2200
Chromic-sulfuric acid	3000
Dilute sulfuric acid	3000
Dilute hydrochloric acid	3400

A typical surface preparation and its process controls are described for metal-to-metal adhesive bonding. The general steps are as follows.

1. Wipe all surfaces with cheesecloth moistened with methyl ethyl ketone to remove lettering and other foreign matter. The cheesecloth must be free from starch and oil or other greasy contamination. The solvent used must not contain greasy contamination or deleterious residue.

2. Degrease the surface in a vapor of trichlorethylene. The trichlorethylene bath must be tested regularly to assure that the solids content does not rise above a certain level. Too high a solids content can cause redeposition of contaminents on the surface of the metal. Solids content can be determined by checking the specific gravity or by evaporation of a sample. The pH of the trichlorethylene should be maintained above a certain value to assure that the halogenated solvent has not decomposed forming free acid which would corrode the metal being degreased.

3. Treat the surface with appropriate metal surface cleaning solution. Here it is most important that the active constituents performing the cleaning be maintained within specified concentration levels for proper control of the process. For example, in surface preparation of aluminum with sulfuric acid-dichromate, both the acid and dichromate must be controlled within close limits to assure quality surface for bonding. Effects of metal surface preparation on adhesive bond strengths are described in considerable detail in various references.[10,11]

4. Remove the surface residue by thoroughly water washing the surface, following with a distilled water spray.

After spraying, the metal surface is examined for breaks in the film of water adhering to the metal surface. A break indicates that the surface has not been properly prepared and the part must be re-immersed in the cleaning solution for recleaning.

In-Process Control of Bonding

The adhesive systems used for structural bonding are of the thermosetting type because they produce high strength bonds under a wide range of conditions and structural requirements. Metal bonding adhesives are available in a number of forms such as (*1*) solvent systems, (*2*) powders, (*3*) solids, (*4*) unsupported films, and (*5*) supported films.

Depending upon the adhesive system and form employed, there are many process control tests which can be performed to assure that the various batches received on the production line are of the same quality as the original samples. Furthermore, their quality should not be reduced because of storage or aging. Some of these significant tests are: (*1*) volatile contents of solvent systems, (*2*) per cent flow of the film adhesive, (*3*) gel time of the solid systems, (*4*) viscosity of solvent and solids systems, (*5*) shrinkage of tape adhesive, (*6*) thickness of dried primer, and (*7*) controls on mixing of two-part systems.

However, the most important process control is the fabrication of a standard test specimen in the same cycle as the part being bonded. This standard test specimen should be a test that is indicative of the prime structural requirement. For example, if the critical item is tensile shear, the standard lap shear specimen should be considered for bonding concurrently as the ordnance part. This is an important point; namely, the process control test specimens that are not integral with the production part must be processed at the same time, under the same conditions, and with the same materials as the production part they represent.

Another approach of fabricating a standard specimen during the same cycle is the incorporation of test tab into the parts being bonded. After bonding, the test tab can be removed by an appropriate method of separation. The test tab can then be tested in accordance to the requirements most significant to the end item.

In addition, process control should maintain checks on ovens, autoclaves, and all bonding tools and fixtures for accuracy in order to assure that the equipment is operating within the specified ranges.

Final Inspection

Visual inspection with the aid of strong light can detect gross flaws and defects. These can be bubbles, lack of bonding agents, voids in the bonds, and misalignment of joints.

Tapping is a simple and effective means of detection for voids or lack of bonding. The simple tools required are a coin or a light hammer. A satisfactory bond will be indicated by a clear tone, whereas a void or poor bond will produce a dull or hollow sound.

Proof stressing is another important method to be considered. The bonded item can be proof tested (*1*) in a manner pertinent to its end usage or (*2*) stressed at levels required by design engineers.

Ultrasonic inspection methods can be useful by employing such ultrasonic units as the Stubmeter,[12] Coinda-Scope, and Fokker bond tester. The Fokker bond tester is a portable electronic instrument consisting of a small oscilloscope, a meter, plus a connecting probe. The probe is held firmly against the bonded area, and the "quality" of the bond is read directly from the instrument.

Proper use of these ultrasonic test instruments calibrated against standard bonded specimens can contribute significantly in assuring the ordnance item has been bonded satisfactorily.

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Synopsis

A system by which adhesive bonding would be best applied to production on ordnance items has been presented. In this system, process controls must be established. Significant and meaningful process controls must be selected and utilized toward producing the quality and reliability expected of the ordnance article.

Résumé

Un système dans lequel la technique d'application des adhésifs serait le mieux employé dans la production de matériel d'ordonnance est présenté. Dans ce système, des contrôles de fabrication doivent être établis. Des contrôles de fabrication vraiment efficients et significantifs doivent être choisis et utilisés afin de produire la qualité et la reproductibilité des propriétés attendues de l'article d'ordonnance.

Zusammenfassung

Ein System zur bestmöglichen Anwendung von Kleheverbindungen bei der Erzeugung von Artillerieartikeln wurde angegeben. Bei diesem System müssen Verfahrenskontrollen eingerichtet werden. Wesentliche und sinnvolle Verfahrenskontrollen müssen ausgewählt und zur Erreichung der für einen Artillerieartikel erwarteten Qualität und Verlässlichkeit benützt werden.