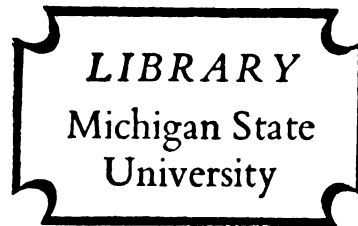


ANALYSIS OF METHODS USED TO
EVALUATE HEAT SEAL BONDS

Thesis for the Degree of M. S.
MICHIGAN STATE UNIVERSITY
Edward H. Graft
1962



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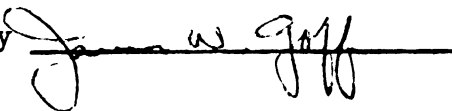
ABSTRACT
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By
Edward H. Graft

A THESIS
Submitted to
Michigan State University
in partial fulfillment of the requirements
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MASTER OF SCIENCE
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ABSTRACT

ANALYSIS OF METHODS USED TO EVALUATE HEAT SEAL BONDS

by Edward H. Graft

This study was undertaken to determine the validity of methods which have been used to evaluate a heat seal bond.

The initial work includes the results of a literature search conducted to determine existing methods for evaluating the bond formed by adhesives, coatings, tapes, and seals formed by heat.

It was found that little had been done to correlate the results obtained by the various testing methods. This pointed out the need for the comparative evaluation which forms the second part of this thesis.

Four different test methods were used on identical seals under similar conditions. The tests can be described as: burst, compression, quick-leak, and peel tests.

As a result of this study, it appears that seal

Edward H. Graft

evaluation should be based on the results of the test method which simulates the treatment the package will be subjected to in distribution and use.

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PART I

INTRODUCTION

The use of heat for sealing the contacting surfaces of packaging material has increased in recent years. In the early days of packaging, glues were usually employed for sealing. Paper and paperboard were the only common flexible and semirigid packaging materials then in use; their absorptive properties rendered the glue tacky almost immediately, so that a modest application of pressure for a short time would produce primary sealing. Primary sealing is defined as that degree of adhesion which is adequate to resist the springback tendency of the packaging materials (84). Further absorption and evaporation of the glue solvent, usually water, ultimately "sets" these primary seals into functional seals, which are defined as those of adequate strength to meet their intended function (84).

The use of a glue requires its even application in the area to be sealed and time for it to become tacky enough for primary sealing. This time may be taken either before the glued surfaces are brought into contact or after; if after, pressure must be maintained to hold the glued surfaces together until primary sealing occurs. Heat is often used in automatic gluing equipment to reduce the time factor to

a minimum by promoting rapid evaporation of the glue solvent. Pregluing is also employed to reduce sealing time and is probably responsible for the earliest use of heat sealing in packaging. Glue is first applied to packaging material in the areas to be sealed later, and allowed to dry. The pre-glued material is then machine processed into packages; the application of heat to the material in the regions to be sealed softens the glue, conditioning it for a primary seal without requiring any drying time. Pre-gluing is essential if appreciable machine speed is to be achieved when sealing areas do not include absorptive packaging material.

The advent of waxed papers and boards provided a natural field for heat sealing since wax and wax-based coatings have excellent sealing qualities. Although wax-type seals are quite weak, this weakness is useful for many packages as, for example, in the case of bread wrappers which are rather easily opened without tearing the wrapper. Since the adhesive is everywhere on a waxed wrapper, wrapping machines using waxed paper require no glue application or drying devices and, therefore, often operate at greater speeds than glue-type equipment.

One of the first flexible packaging materials

not involving paper was cellophane. Since cellophane is relatively non-absorptive, it is offered with a heat-sealable coating making it useable in high-speed wrapping machinery. An added advantage of a heat-seal coating over glue is that the sealed area is almost indistinguishable from the rest of the material. This is an important appearance feature with transparent wrappers.

As the packaging art progressed and more convenience items, particularly food products, were developed, hermetically sealable flexible packages were required. Similarly, ways to evaluate the formed seal were needed to determine whether the seal was adequate to perform the job it had to do.

The first part of this paper contains the results of a literature search that was conducted by the writer to uncover the various methods that had been used to evaluate the bond formed by the different methods of fastening. These include: adhesives, coatings, tapes, and seals formed by heat.

In the sections that follow, methods of evaluating the bonds formed by the various methods are expanded upon. At the end, a list of eighty-four selected references pertaining to bond evaluation is included.

WAX SEALS

The rapid progress in wax formulations and in the introduction of new packaging materials to which waxes are applied has made imperative the development of standard wax testing procedures and performance specifications.

Measurement of the sealing strength of paraffin and microcrystalline waxes is a most difficult task. Numerous factors, including types of surfaces sealed together, conditions of coating and cooling of sealed surfaces, the specific angle at which the sheets are separated, the thickness, and flexibility of the material coated all effect the results obtained and require standardization.

In order to be certain that a sufficiently strong heat-sealed closure will be provided, it is necessary to be able to measure the sealing ability of the wax used, to compare waxes available from different sources, and to select a wax that can be counted on to meet the particular requirements.

In the past, many have depended merely on a qualitative test of one type or another. These tests are characterized by sealing with a hot iron and then peeling apart by hand to obtain an estimation of sealing qualities. Others have developed quantita-

tive methods based on a wide variety of techniques.

In one of the earlier methods, the Elmendorf Tear Tester was modified to determine sealing strength by indicating the energy absorbed in separating two sealed strips of specified area (59). Paralleling this were methods based on peeling a sealed specimen at a constant rate and measuring the required force by using a simple Jolly Spring arrangement or gravity weighing system such as the basis-weight scales employed by the paper industry. Others used similar apparatus but, instead of the Jolly Spring, obtained an indication of the force by measuring the vacuum or pressure developed in a piston and cylinder arrangement (23).

Kinsel and Schindler (47) have described a method for determining the adhesion properties of microcrystalline wax. But, due to the method of sealing test specimens and high load range of the seal tester (Suter Tensile Tester), it is not satisfactory for paraffin waxes.

MacLaren (50) describes a modification of the method developed by Des Autels of the Kalamazoo Vegetable Parchment Company, for determining sealing strength of both paraffin and microcrystalline waxes.

This instrument utilizes a vacuum weighing system. Test data correlates well with data obtained by laboratory tests for sealing strength and blocking.

Another method for determining the sealing strength of paraffin wax, reported by Funk, et al. (33), uses a spring weighing system and records the force graphically on polar coordinates. The sealed specimens are separated at the rate of 21 inches per minute.

Separation is dependent on buoyancy in the method developed by Padgett, et al. (63). In this method one separated end is attached to a float in a mercury-filled column, and the other is drawn downward by a constant speed motor. The machine is calibrated by finding the relationship between the weight applied and the depression of the float. The distance the float is pulled down before the strips separate gives a measure of the seal strength.

Whereas most of these methods were based on peeling the sealed specimen at 180 degrees, the Socony-Vacuum Oil Company has developed a technique of determining sealing strength by peeling at small angles (62). This method gives satisfactory results as long as the angle of separation is maintained.

constant when making comparisons.

Since wax is used primarily as coating on some other material, some work has been done on measuring the bond between wax and the coated material. The method developed by Salvesen and Eosefow (67) was primarily designed to measure the adhesion of paraffin wax to milk carton stock. A specified area of dairy carton paper stock is waxed on one side under controlled conditions. Using specially constructed attachments adapted for the incline plane this wax coating is stripped off. The force required to break the bond between the wax and the paper gives a measure of the wax adhesion.

TAPE TESTING

Tape testing was included in the survey because of the similarity between the area of seal formed by a tape to its adherent and adhesive seals in general. The resistance to separation or peel of a tape (particularly pressure sensitive tape) may be called: tack, grab, stick, and so on. A literature survey (15) of tack-testing methods led to their classification into two groups - those designed to duplicate end-use performance and those intended to correlate one property of adhesiveness, such as viscosity, with tack. End-use tests require many determinations and lack sufficient reproducibility, whereas property tests are usually sensitive and reproducible but may not correlate well with end-use performance.

The point common to most end-use tests is that the tests are run on the packaged surface (72) rather than a standard such as a flat steel panel. From this point on, the methods used by individual companies are those which most nearly answer their individual needs.

There are five methods which represent the main categories of testing for tack not involving the packaged surface.

These are:

1. Chang method, peeling tape from a flat steel panel;
2. Rotating surface method, peeling tape from a rotating steel drum;
3. Inclined plane method;
4. Douglas curved track method;
5. Hercules probe method.

CHANG METHOD (25)

In this method the tape is to be put on a stainless steel panel, with no other pressure than the weight of the tape itself. The contact between the adhesive and the testing panel is kept to the lowest possible minimum. The peeling is done with the free end of the tape at an angle of 90 degrees to the adherent and the testing panel is made to move at a speed equal to the peeling rate, 12 inches per minute.

In the actual application of pressure-sensitive tape, the user may or may not apply pressure on the backing. The practical advantage of this method is that it would be of interest to the manufacturer or designer of tape in both conditions, applied with and without pressure.

ROTATING SURFACE METHOD (26)

A roller with a diameter of $1\frac{1}{4}$ inch is used as a rotating surface. One end of the tape to be tested is clamped in the upper jaw of the tensile tester and the other is let down slowly to make contact with the roller. The rate of peel is 12 inches per minute.

INCLINED PLANE METHOD (26)

In this method, a steel ball rolls down over an adhesive surface with an inclination of 30 degrees. The rolling distance is taken as a measure of tack. In this method the size of ball used is such that it is small enough to avoid difficulty in determining the rolling distance on one hand and large enough to make this distance larger than one inch so as to assure 10% precision in the resulting reading.

HERCULES PROBE METHOD (82)

Wetzel suggested the use of a carefully machined 1/16th inch brass probe to measure the tack of pressure sensitive adhesive film deposited on glass. The film approaches and contacts the probe at 20 inches per minute, and moves away from the probe at the same speed after a contact time of one second. The pressure applied is equivalent to 7 psi. The pulling is done

with an Instron Tensile Testing machine and a Sanborn high speed recorder is used for recording the load on the probe. The average of ten readings of the stress required to break the interfacial bond between adherent and adhesive is taken as tack.

In addition to testing for tack, Dahlquist (28) has attempted to define tack and goes into the theory of tack.

METHODS OF SEPARATING (PEELING) SEALS TO CAUSE FAILURE

Any adhesive joint comprising a flexible member as one of its adherents presents the possibility of peeling as a mode of failure. Peel, as a particular form of tearing, is essentially a boundary process with the physical work of peel and progressive bond destruction localized at the moving edge of the bond.

Several attempts have been made to give a thorough explanation of peel (27, 42, 43, 44, 45). By necessity they involve a mathematical presentation.

Standard tests have been developed to measure seal strength by pulling (peeling) the seal apart. Two such tests are ASTM D 903-49 and TAPPI RC-272. Another is the dead weight test which accomplishes the peel by having a static load on the seal for a specified amount of time.

Various machines have been developed to peel the specimen apart at a constant rate such as:

1. Instron Tensile Tester
2. Schopper Tensile Tester
3. Scott Tensile Tester
4. Suter Tensile Tester

to mention a few.

ADHESION TESTING

The problem of accurately measuring the degree of adhesion of a plastic to the substrate has existed since the extrusion coating process was first used commercially.

In the field of paper coating it has been conventional practice to measure adhesion by hand peeling the plastic from the paper. This test is qualitative in that it only determines whether the adhesion is good or bad. The need for a quantitative test existed in order to determine the intermediate levels of adhesion.

The test adopted in many paper-coating laboratories has been the use of the conventional tensile tester to measure the force required to strip the coating from the paper. This test has been moderately successful, but has certain limitations. In the case of good adhesion the paper often tears before the bond fails.

An instrument capable of measuring adhesion values between no adhesion and perfect adhesion is the Perkins-Southwick Bond Tester (38, 39, 73). This tester includes a chamber with an orifice of one square inch area over which a test specimen of coated material is clamped with the coated side up. Most test materials are more

or less porous, and air controlled pressure supplied to the chamber is passed through the orifice and against the specimen so that it seeps or leaks through the base against the coating. The air pressure tends to lift the coating from the base, and its measure determines the adhesive power.

A similar method developed by Donahue and Verseput (30) for use in determining the ply-bond strength of paperboard employs the Jumbo Mullen Tester as the means of applying and measuring the force required to rupture the sample.

Another direct, quantitative method for measuring adhesion of organic coatings utilizes an electrodynamic system for producing longitudinal ultrasonic vibrations in a metal cylinder (56, 57). An organic film attached to the free end of the cylinder separates from the substrate when the force due to acceleration exceeds the force of adhesion at the interface. The acceleration is determined by the frequency and amplitude of vibration.

All of the above methods are destructive testing methods. A sensitive heat detector can be used to record differences in heat transfer through a laminated web passing over a hot roller (17). This nondestructive

tive testing is based on the principle that heat transfers more readily through a good bond than through a poor one.

Several less complicated tests for adhesion have also been developed, such as:

1. Wax Streak Test (12)
2. Bell Laboratory Mar Adhesion Test (65)

ADHESIVE BOND

Although the bond formed by an adhesive may be thought of in terms of adhesion testing, the term bonding strength is also used. Several methods or approaches were uncovered in the literature search.

The initial work always seem to start with an evaluation by tearing apart simple pasteups (first by hand, noting the type of tear and later obtaining quantitative measurement with a tensile tester). (71)

TAPPI RC-267 (68) attempts to measure the bonding quality of an adhesive by measuring the force (in grams or pounds) required to lift a block from its anchorage. An advantage of the method is that materials to be bonded with a test adhesive do not have to be attached to rigid fixtures with some stronger adhesive. With only minor variations, the procedure can be used to study the bonding of paper to paper, the attaching of label stock to glass, or for measuring the adhesive strength of gummed tape.

Bartlett (18) stresses the need to carry out bonding strength tests by using a combination of tests. He points out that almost directly opposite

results can be obtained for the same bond depending on the test used.

Several pieces of equipment have been designed specifically for adhesive testing. One of these is an impact testing machine (2). The specimen, specially shaped for the purpose, is fastened to the end of a long rod vertically mounted. Annular weights designed to slide freely down the rod provide impact. The energy level of the blow is governed both by the weight of the ring and height of the fall. Among the test results are the number of blows required to effect a failure at a given energy level.

Another method uses the Concora Torsion Tear Tester to evaluate the nature and strength of an adhesive bond (49). Laboratory evaluation may be used to establish the best adhesive, film thickness, and pressure time to duplicate production conditions.

The problem of determining bond strength has been approached in several ways. Nichol (60) viewed the problem from the mechanical aspect, of which the most important part is board absorption. The two absorption tests described are the oil test (in which the distance a given amount of oil runs is measured -

the shorter the distance the greater the absorption) and the drying speed test (in which the time needed to effect a fiber tear bond is recorded).

Immersion testing has been conducted to determine bond strength when the adhesive is subjected to a selected reagent (54).

Kane (46) developed a test to evaluate the water resistance of a corrugator adhesive bond. The force required to break the wetted bond is measured (the force being applied to and in the planes of the liner material).

Another possible evaluation of bond strength may come through use of nine test methods (14), some of which have been used primarily for wood (plywood).

1. Tensile Strength Test
2. Shear Tests
3. Block Shear
4. Plywood Shear
5. Single lap joint shear
6. Double lap joint shear
7. Scarf joint shear
8. Cylindrical Single Shear
9. Johnson double shear

HEAT SEAL BOND

A heat seal bond should be treated in the same manner as a liquid adhesive bond. As with all adhesive applications, tests must be conducted to determine whether or not the final adhesive bond is satisfactory.

Belletire (20) describes a test which has shown itself to be indicative of final adhesion. After the bond has been made by use of heat, the adhered area is allowed to cool to room temperature. The samples are then placed in a refrigerated temperature of 25 degrees F. If the bond is not positive it usually will break in less than 30 minutes.

The Packaging Institute (5) has developed a method for testing the tear strength of seals of thermoplastic materials using a tensile tester.

Several different types of apparatus have been designed to test the strengths of films and seals using air pressure to burst the film or pouch. Among these are the work done by Hu and Nelson (41), Olsson and Pihl (61), Mannheim, et al. (51), Davis, et al. (29). The pressure reading at burst and area of failure is recorded.

A precise check on the continuity and quality of heat seals in plastic materials has been found possi-

ble by visual examination with polarized light using a Heat-Seal-O-Scope (55). However, alone it can not be considered a quantitative instrument because it must be used with representative samples of good and poor seals.

Several non-destructive leak detection methods that essentially test seals are spelled out in specification MIL-P-116 (13).

One of the tests specified is the quick-leak test. There are two versions of this test. For small packages, the test package is placed under water in a vacuum chamber and the inspector looks for bubbles rising through the water from the package.

The other version of the quick-leak test, which would be used on packages too large for the vacuum chamber, uses heat rather than vacuum to force escape of air from the package being tested. The package is immersed in a tank of hot water and systematically rotated so that each side in turn is parallel to and approximately one inch below the water surface. Again, a continuous stream of bubbles indicates a leak.

Also mentioned are pressure and vacuum retention tests. These tests are intended to be used for large packages that cannot easily be handled in the manner

necessary to run the quick-leak test. In these tests each package must be fitted with either temporary or built-in connections to permit either pressurizing or evacuating the interior of the package. By means of connected manometers or gauges, the tests determine the ability of the sealed package to hold a specified pressure or vacuum, as the case may be, within certain tolerances over a specified period of time.

Powell (66) has also done some work on leak testing methods. His methods involve the use of detectable, inert, gaseous contaminants.

In one method a radioactive gas is forced to enter a leaking package and this package is later detected as a leaker when it is passed under a scintillation counter. This method is used for detecting extremely low order leaks in electron tubes and hermetically sealed units.

The other method is to insert a small amount of Freon or other halogenated gas or vapor inside the package just prior to final sealing. Forced leakage is detected with a General Electric Halogen Detector, a portable instrument that is extremely sensitive to halogenated gases and vapors.

Another method examined by Powell takes advantage

of the pillowing effect on flexible packages when subjected to vacuum conditions in a closed test chamber. Using a sensitive expansion detection device, it is possible to reliably differentiate between good seals and those with leaks.

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PART II

INTRODUCTION

One point which stood out as the literature search progressed was the fact that very little comparative data existed for test results on a given set of seals. It seems that each writer has his method of test, and little is done to correlate the results obtained by the various testing methods. This pointed out the need for the comparative evaluation, which forms the second part of this paper.

Four different tests were run on identical seals under similar conditions. Three tests were designed which tested the bond in the same general manner. These tests can be described as: burst, test, compression test, and quick-leak test. A complete description and procedure for performing each follows below. The fourth test was a peel test performed on a tensile tester. This test was selected because of its frequent use within the industry. It was performed in accordance with P. I. Test Method -3, for heat sealing strengths and characteristics.

Testing was done on seals made from four materials:

1. Polyethylene (Dupont 2 mil.)
2. Polypropylene (Avisun Olefane AT-2)
3. Polyvinylchloride (1 mil.)
4. Saran (Dow Type 12 gauge 200SW)

A comparison of test methods can not be achieved if the seals being tested are not fabricated under the same conditions of temperature, pressure and dwell time. The laboratory heat sealer used must be one which lets the operator control the three variables of heat sealing. The Olin Mathieson Chemical Corporation (3) has developed such a sealer and because of its unique abilities, it was used to fabricate the seals for three of the test materials: Polyethylene, Polypropylene, and Polyvinylchloride.

The instrument consists of two parts: the sealer and the control cabinet. The sealer is built in the form of a small table containing an opening through which the sealing bar can move freely. In its rest position, the bar is one-half inch below the top of the table and it is moved up into its sealing position and down again by means of a small air piston.

The sealing element is constructed in two parts. The lower portion, consisting of a steel bar six inches long with square cross section of one inch, containing a one-half inch diameter hole at its center extending almost through its entire length. A 150 watt heater is inserted in this hole. The second part forms the surface which comes into contact with the heat-

sealable packaging material. This bar is made of aluminum and is Teflon coated. The entire assembly, consisting of the steel bar with its heating element and the sealing bar, is in turn fastened to the air piston through a heat insulator block.

The top metal plate contains two screws and three studs which project from it. The screws at either end act as stops for controlling the height to which the sealing-bar assembly may move. These are set to allow a rise of approximately 1/16 inch above the surface of the table. The three remaining studs form locators for the sealing pressure weight (during the sealing, these three studs were removed because they interfered with the sheet of film needed to make the test pouch - the pressure weight was located by placing it over the holes the studs formerly occupied).

The sealing pressure is controlled by dead weight (the pressure weight weighed three pounds eleven ozs., and was covered with paperboard). It is possible to make heat seals for testing purposes at a variety of sealing pressures ranging from a minimum equal to the weight of the

upper piece of film to a maximum of several pounds per square inch.

Temperature and dwell time are regulated respectively, by means of a temperature controller and an electric timer located in the control cabinet. For an accurate estimate of the temperature of the surface of the sealing bar, a thermocouple is located in the center of the sealing bar and as close to the sealing surface as possible. The electric timer is started by means of a micro-switch adjusted so that the sealing cycle is timed from the instant the sealing bar first touches the test specimen. The timer, in turn, opens and closes a solenoid valve to the small air piston which moves the sealing bar into position and back down at the end of the sealing cycle.

Each of the three sealing variables: pressure, sealing temperature, and dwell time is controlled independently of the others and it is possible, for all practical purposes, to make heat seals at an infinite number of sealing conditions.

The seals for the fourth material (saran) were fabricated on an electronic heat sealer. The sealer used was a Callanan 1 KW Generator with standard press.

This electronic sealing equipment is made up of three basic units: (1)

1. A generator or frequency converter to change the commercial low frequency power to high frequency electrical energy;
2. A pneumatically operated press to raise and lower the die and to supply the necessary pressure during the sealing cycle;
3. A die (or electrode) of the required outline and dimensions to seal the desired shape and area of the film or sheet.

High frequency sealing is accomplished by an electric power usage known as dielectric hysteresis. The plastic material is subjected to a high frequency electrostatic field. As the polarity changes, a corresponding stress reversal is experienced by each molecule in the material being sealed. The distortion and reorientation of the molecular elements results in more or less friction, depending upon the material's composition. The frictional heat generated causes melting and consequent uniform bonding together of those areas of the film which were subjected to the electrostatic field.

The great advantage of this method in heat sealing is that the heat is always uniform; it is exactly the same at the sealing surfaces as on the exterior surfaces.

In ordinary heat-sealing, the heat must penetrate through the material and the exact temperature at the critical sealing surface is difficult to determine or control.

The seal can be controlled by varying the power, pressure and time. The sealing cycle is accomplished in the following manner. The film to be sealed is placed on the plate of the flat bed press. The die is lowered and the necessary air pressure applied. The high frequency energy is applied at a fixed rate for the required time cycle. The die is then raised and the finished piece removed from the press bed.

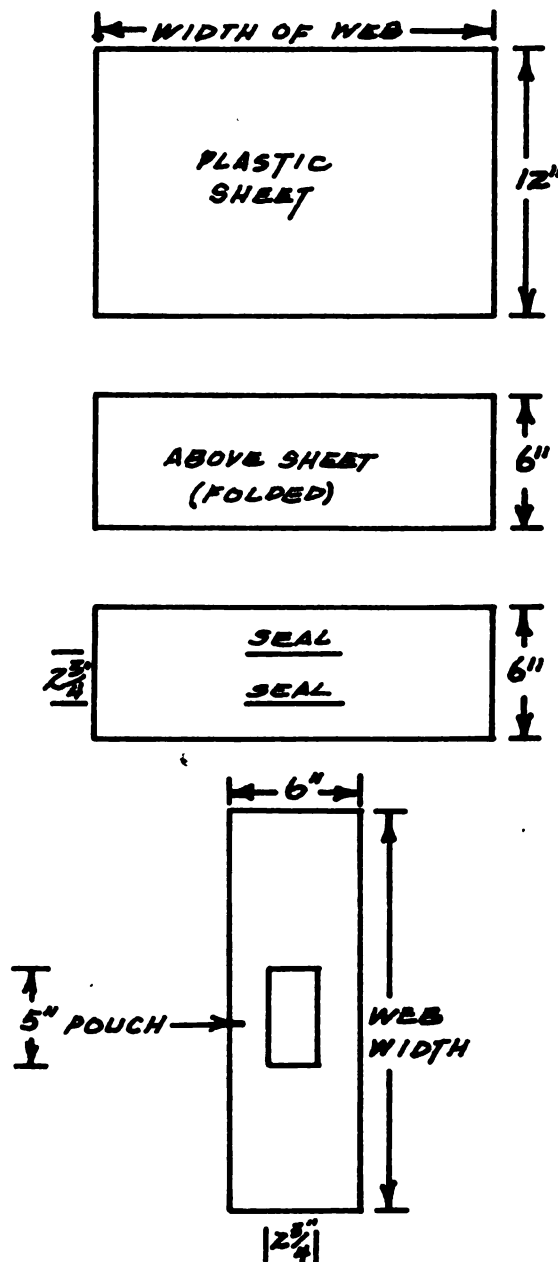
When the material to be sealed is very thin, .004 inches or less, it is usually necessary to use a buffer between the material and the bed of the press. A buffer tends to lessen the heat lost by thermal conductivity to the bed plate and die. It also prevents arcing. The usual material for the bed plate buffer is phenolic impregnated paper.

Because the saran film used was .002 inch, a 3/64 inch thick phenolic buffer was used for all seals on the electronic sealer.

EXPERIMENTAL PROCEDURE

The burst, compression, and quick-leak tests were run on a pouch whose inside dimensions were 5 inches by 2 $\frac{3}{4}$ inches.

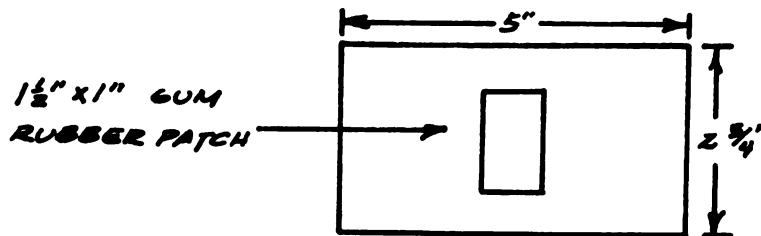
Each pouch was fabricated in the following manner to make certain that the center section of the web was used for all pouches. The smallest variation in thickness across the web occurs in the center section.



Because of certain inherent characteristics of the tests and of one of the materials (~~saran~~), certain modifications were made on the pouch, none of which changed its basic dimensions.

Seal failure in the burst and compression tests is brought about by the force exerted on the seals by air and water respectively.

Putting air or water into the pouch for each test necessitated the attaching (with rubber cement) of a $1\frac{1}{2}$ inch by 1 inch piece of gum rubber centered on the side of the test pouch.



Air or water is injected into the pouch by puncturing the center of the gum rubber patch with a hypodermic needle. The gum rubber seals up the hole upon removal of the needle (this property makes the compression test possible).

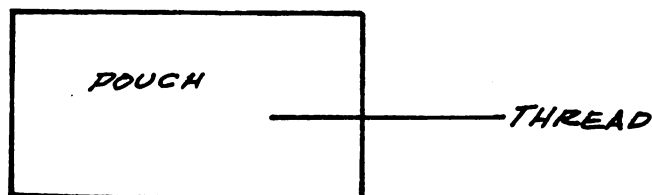
The pouches used for the quick-leak test contained an insert* (one polyethylene semi-rigid tube $2\frac{1}{2}$ inches

* Polyethylene Tulox container supplied by Extruded Plastics Incorporated., New Canaan Ave., Norwalk, Connecticut.

long and 1 inch in diameter, open on one end). The tube was placed in the pouch (closed end first) so that the length of the tube was parallel to the 5 inch side of the pouch, by having the insert in the pouch, enough air was trapped in the pouch to cause failure when the vacuum was drawn during the test.

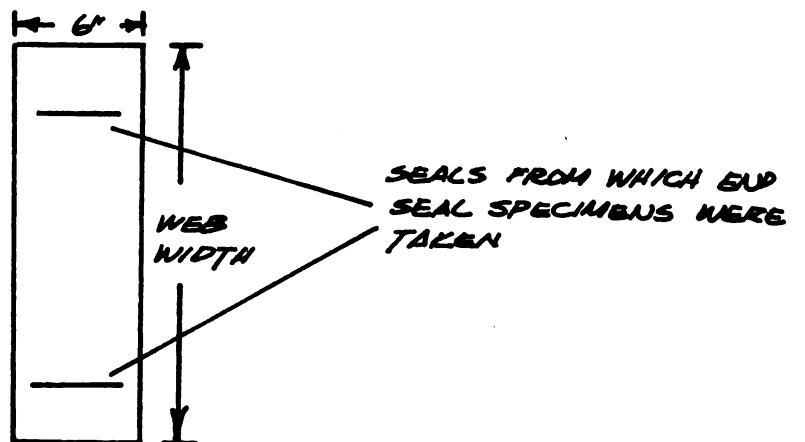
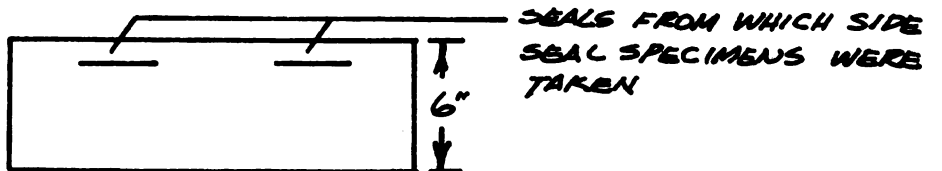
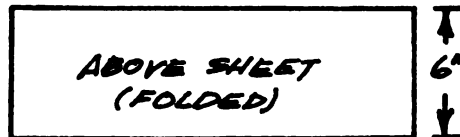
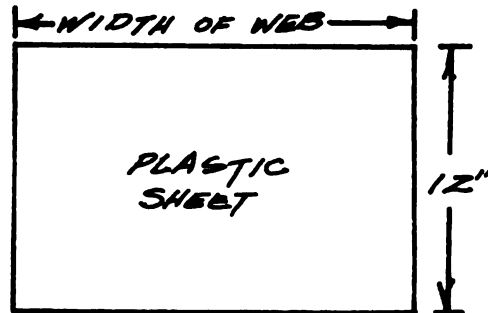
Before the actual testing of the pouches began, it was found that when the needle was injected into the saran pouches, there was a tendency for the material to split out from the point of injection. To eliminate this problem, a very thin (.001 inch) coating ($1\frac{1}{2}$ inch by 1 inch in area) of cellulose acetate butyrate was applied to one inside face of the pouch (actually to the sheet material before the pouch was fabricated). The position of the coating coincided with the position of the gum rubber patch.

An additional modification was made on all control pouches. A piece of ordinary sewing thread was sealed in one end.



The thread was pulled out after the seal was made. The control pouches were included to obtain an indication of the sensitivity of each test method (to leaks).

The test specimens for the peel test were made in the following manner:



The control seals were made by sealing a piece of thread (same as used for pouch control) within the end and side seals. The thread was pulled out after the seals were made. Before each control peel specimen was tested, the area where the thread had been was inspected to see that the thread went entirely through the seal.

All specimens for the peel test were cut from the representative side and end seals using a Thwing-Albert Model JDC 25 specimen cutter.

In order to realize the control over seal fabrication which the Olin Heated-Bar and Callanan Electronic sealers are capable of, the following procedures for their operation were drawn up.

A. Procedure for operating electronic sealer.

1. Open the valve from the air line one full turn counterclockwise.
2. Make sure the sealer jaw is in the raised position.
3. Check to see that the brass electrode is clean, level, and securely in position.
4. Position the dielectric sheet on press bed.

5. Turn on generator (allow five minutes for warm up).
6. Set sealer- timer, Powerstat, and pressure.
7. Position material to be sealed.
8. Seal.
9. After each sealing cycle, recheck settings before next seal is made.

B. Procedure for operating heated-bar sealer.

1. Turn the air regulator handle counter-clockwise until it is free of tension.
2. Open the valve from the air line one full turn counterclockwise.
3. Slowly turn air regulator handle clockwise to the desired setting.
4. Adjust the Powerstat to a value of 85.
5. Turn on the machine.
6. Set the temperature control and timer to desired setting.
7. When machine reaches correct temperature, place material across top plate of the sealing unit in correct position.
8. Place the weight on top of the material.
9. Quickly press and release foot control.
10. Remove weight and specimen.
11. Check settings after each seal.

After the pouches were fabricated, the excess material was trimmed off. The gum rubber patches were placed on the appropriate pouches, and then all test pouches and seals were placed in the conditioning room* for 24 hours before testing.

To insure consistency in each test, the following procedures were set up:

A. Burst Test

Equipment

1. Tank of nitrogen fitted with flow control mechanism.
2. Desiccator jar.
3. Stop watch.
4. Specially designed injecting valve (see figure 1).

Procedure

1. Partially fill desiccator with water (have enough water in dessicator to completely cover the pouch).
2. Set flow meter to 7.5 lbs. per square inch.
3. Inject needle through center of gum rubber patch and into pouch.

* Room conditions controlled at 73 ± 2 degrees F. and 50 ± 2 percent relative humidity.

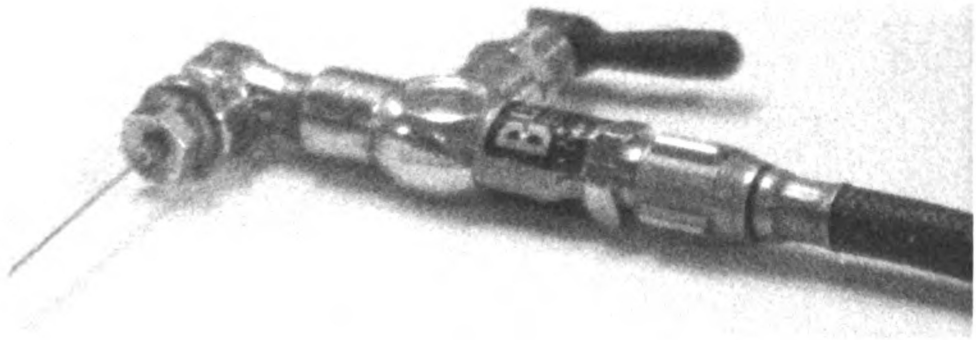


Figure 1

4. Completely submerge pouch in water.
5. Simultaneously start air flow and stop watch.
6. At first sign of failure (bubbles), stop watch and air flow.
7. Record elapsed time, area, and type of failure*.

B. Quick-Leak Test

Equipment

1. Vacuum pump (Cenco Hyvac 2).
2. Manometer (Meriam Instrument Co.) and, or vacuum gauge (U. S. G. gauge).
3. Vacuum desiccator.

Procedure

1. Partially fill the vacuum desiccator with water (have enough water in the desiccator to completely cover the pouch).
2. Attach lead weight to pouch using spring clip.
3. Place test pouch and weight in the desiccator.
4. Place cover on desiccator and connect the desiccator to vacuum pump by means of rubber tubing.

*Adhesive failure - within the adhesive layer.

Cohesive failure - in the material itself.

5. Attach a manometer or vacuum gauge in the line.
6. Start the vacuum (simultaneously start stop watch).
7. Record the elapsed time, vacuum attained, area, and type of failure if bubbles (indicates failure) appear before reaching 10 inches of vacuum.
8. If no failure occurs after reaching 10 inches of vacuum, stop the pump for .1 minute and observe (noting if failure occurs).
9. If no failure occurs after .1 minute, start pump and proceed to 15 inches of vacuum (noting if failure occurs before this).
10. If no failure occurs after attaining 15 inches of vacuum, stop the pump for .1 minute and observe (noting if failure occurs).
11. Continue this process at intervals of 5 inches up to 25 inches.

The appearance of any bubbles, in the water, or on the surface of the water, coming from the pouch indicates that it has failed. A few bubbles on the surface of the pouch, but not released from it, are not indications of failure.

C. Compression Test

Equipment

1. National Forge Compression Tester (Model TM 51008).
2. 50 c.c. hypodermic syringe.
3. Potassium Permanganate Solution (dilute solution used to color the water to make failure more easily recognized).
4. Polyethylene bags.

Procedure

1. Inject needle through center of gum rubber patch and into pouch.
2. Fill syringe.
3. Connect needle to syringe and inject 50 c.c. of weak permanganate solution.
4. Repeat steps 2 and 3.
5. Remove needle from pouch.
6. Place pouch in polyethylene bag (to catch solution when failure occurs).

7. Make sure side with patch is up
and pouch is lying flat.
8. Place test pouch in compression tester.
9. Set compression tester speed to .2
inch per minute.
10. Check zero point on scale.
11. Start machine.
12. When failure occurs, record pounds
(0-100 lbs. scale) at failure, area,
and type of failure.
13. Raise upper platen of machine and re-
move test pouch.

D. Peel Test

Equipment

1. Schopper Tensile Tester.

Procedure

1. Level the machine (Schopper Tensile
Tester) in both directions.
2. Anchor the free ends of the specimen
in the two clamps*, taking care to
see that the specimen is lined up
parallel to the clamps.

* Slipsheets made from paperboard were used on
the clamps.

3. Check to see that the machine is set to run at 12 inches per minute, jaw speed.
4. Loosen upper clamp.
5. Trip the machine to begin the pull on the specimen.
6. Record the highest reading from the proper scale and note the type of failure.
7. Reverse machine and remove fractured specimen.

The burst, quick-leak, and peel tests were run in the 73° F. 50% R. H. controlled condition room. The compression test was run in the physical testing room at the School of Packaging.

ANALYSIS OF TEST DATA

The results that follow are from tests run on seals made from the four materials. Seals were made at two different sealer settings for each material. Five samples were made up for each test, at each setting, for all materials.

In addition, five control samples were made up for each test for every material.

Tables I-IV contain the maximum, minimum, and mean values for the five samples run on each test. Also included is the type and area of failure having the greatest frequency of occurrence.

The mean values for all tests on all materials is presented in table V.

SARAN*

Table I

Sealer Setting		Values	Burst		Compression		
			Time	Type	Area	Lbs.	Type Area
Pressure	15	Max.	.07			15	
Time	1 sec.	Min.	.05	**	**	8	**
Power	20	Mean	.06	AD (3)	Side (4)	12	AD 4: Side (5)
Pressure	15	Max.	.08			17	
Time	$\frac{1}{2}$ sec.	Min.	.06			11	
Power	30	Mean	.07	AD (5)	End (3)	13	AD 5: Side (3)
Pressure	15	Max.	.03			3.5	
Time	$\frac{1}{2}$ sec.	Min.	0			0	
Power	30	Mean	.02	AD (5)	End (5)	2	AD 5: End (5)

POLYETHYLENE

Table II

Sealer Setting		Values	Burst		Compression		
			Time	Type	Area	Lbs.	Type Area
Pressure	14.5	Max.	.09			19	
Time	$\frac{1}{2}$ sec.	Min.	.06	**	**	10	**
Temp.	285 deg.	Mean	.08	COH(5)	End (4)	16	COH (5) End (5)
Pressure	14	Max.	.09			20	
Time	.7 sec.	Min.	.06			15	
Temp.	275 deg.	Mean	.08	COH(5)	End (3)	17	COH (5) End (5)
Pressure	14	Max.	.04			3	
Time	.7 sec.	Min.	.01			1	
Temp.	275 deg.	Mean	.02	AD (5)	End (5)	2	AD (5) End (5)

* All saran seals were 3/32 inch thick because of the die used on the electronic sealer.

** Indicates number of occurrences out of five.

*** Adhesive layer starts to separate and then material tears.

Table I (con't)

QUICK LEAK				End Peel		Side Peel	
Time	Vacuum	Type	Area	Lbs/In.	Type	Lbs/In.	Type
.30	16	:	:	12.45	:	11.50	:
.25	14	:	** :	1.95	:	1.25	:
.27	14	:	COH Side	7.86	:	6.46	:
		:	(5) (5)		:		:
.30	15	:	:	11.80	:	12.10	:
.27	14	:	:	2.00	:	2.55	:
.29	15	:	AD(5) Side	7.61	:	7.22	:
		:	(3)		:		:
0	0	:	:	5.00	:	6.00	:
0	0	:	:	2.20	:	2.55	:
0	0	:	AD(5) End	3.62	:	4.43	:
		:	(5)		:		:

Table II (con't)

QUICK LEAK				End Peel		Side Peel	
Time	Vacuum	Type	Area	Lbs/In.	Type	Lbs/In.	Type
.29	16	:	:	3.65	:	3.75	:
.25	14	:	** :	3.50	:	3.35	:
.27	15	:	COH End	3.55	:	3.54	:
		:	(4) (5)		:		:
.29	16	:	:	3.45	:	3.70	:
.25	14	:	:	3.20	:	3.15	:
.26	15	:	COH End	3.33	:	3.42	:
		:	(4) (5)		:		:
0	0	:	:	2.95	:	2.80	:
0	0	:	:	2.75	:	2.50	:
0	0	:	AD (5) End	2.80	:	2.61	:
		:	(5)		:		:

POLYPROPYLENE

Table III

: Sealer Setting		Values :	Burst			: Compression		
			:Time:	Type	:Area	:Lbs.:	Type	:Area
:Pressure	14	: Max.	:.05 :	:	:	:15 :	:	:
:Time	1.1 sec.	: Min.	:.04 :	** :	** :	5 :	** :	**
:Temp.	275 deg.	: Means	:.04 :	COH(5):End	:11 :	COH(5):End(5)	:	:
:		:	:	:	(5)	:	:	:
:Pressure	13.5	: Max.	:.03 :	:	:	:17 :	:	:
:Time	1.8 sec.	: Min.	:.03 :	:	:	:9.5:	:	:
:Temp.	285 deg.	: Means	:.03 :	COH(5):End	:13 :	COH(5):End(5)	:	:
:		:	:	:	(5)	:	:	:
:Pressure	13.5	: Max.	:.02 :	:	:	:1 :	:	:
:Time	1.8 sec.	: Min.	:.01 :	:	:	:1 :	:	:
:Temp.	285 deg.	: Means	:.01 :	AD(5):End	:1 :	AD (5):End(5)	:	:
:		:	:	:	(5)	:	:	:

POLYVINYLCHLORIDE

Table IV

: Sealer Setting		Values :	Burst			: Compression		
			:Time:	Type	:Area	:Lbs.:	Type	:Area
:Pressure	14	: Max.	:.08 :	:	:	:31 :	:	:
:Time	2 sec.	: Min.	:.06 :	** :	** :	25 :	** :	**
:Temp.	275 deg.	: Mean	:.07 :	AD COH:Side	:28 :	AD(5) :	End(5)	:
:		:	:	(5)	(4)	:	:	:
:Pressure	13.5	: Max.	:.08 :	:	:	:31 :	:	:
:Time	1.8sec.	: Min.	:.06 :	*** :	:	:20 :	*** :	:
:Temp.	285 deg.	: Mean	:.07 :	AD COH:End	:25 :	AD COH: End(4)	:	:
:		:	:	(5)	(4)	:	(5)	:
:Pressure	13.5	: Max.	:.03 :	:	:	:2 :	:	:
:Time	1.8 sec.	: Min.	:.01 :	:	:	:0 :	:	:
:Temp.	285 deg.	: Mean	:.02 :	AD (5):End	:1 :	AD (5): End(5)	:	:
:		:	:	:	(5)	:	:	:

** Indicates number of occurrences out of five.

*** Adhesive layer starts to separate and then material tears.

Table III (con't)

Quick Leak				End Peel		Side Peel	
Time	Vacuum	Type	Area	Lbs/In.	Type	Lbs/In.	Type
.08	8			5.95		6.40	
.06	6	**	**	3.95	**	4.05	**
.07	7	AD(5)	End(5)	5.14	COH (5)	5.65	COH (5)
.10	10			6.20		5.5	
.05	5			5.60		4.35	***
.07	7	AD(5)	End(5)	5.91	COH (4)	4.68	AD COH(5)
0	0			5.40		5.50	
0	0			4.80	***	4.20	
0	0	AD(5)	End(5)	5.02	AD COH (5)	4.75	AD COH(5)

Table IV (con't)

Quick Leak				End Peel		Side Peel	
Time	Vacuum	Type	Area	Lbs/In.	Type	Lbs/In.	Type
.46	18			1.90		1.95	
.43	16	**	**	1.50	**	1.05	**
.45	17	COH (5)	End(4)	1.73	AD (5)	1.62	AD (5)
.47	18			2.00		1.70	
.42	16	***		1.45		1.45	
.45	17	AD COH (5)	End(4)	1.75	AD (5)	1.59	AD (5)
0	0			1.60		1.45	
0	0			.95	***	1.15	***
0	0	AD(5)	End(5)	1.19	AD COH (5)	1.27	AD COH (5)

MEAN VALUES FOR ALL TESTS

Table V

Material	Burst Min.	Compression Lbs.	Quick-Leak Min.	End Peel Lbs./In.	Side Peel Lbs./In.
Saran					
1st. setting	.06	12	.27	7.86	6.46
2nd. setting	.07	13	.29	7.61	7.22
Control	.02	2	0	3.62	4.43
Polyethylene					
1st. setting	.08	16	.27	3.55	3.54
2nd. setting	.08	17	.26	3.33	3.42
Control	.02	2	0	2.80	2.61
Polyvinylchloride					
1st. setting	.07	28	.45	1.73	1.62
2nd. setting	.07	25	.45	1.75	1.59
Control	.02	1	0	1.19	1.27
Polypropylene					
1st. setting	.04	11	.07	5.14	5.95
2nd. setting	.03	13	.07	5.01	4.68
Control	.01	1	0	5.02	4.75

COMPOSITE SEAL STRENGTH RANKING* BY
MATERIAL AS DETERMINED BY EACH TEST

Table VI

Tests	Saran	Polyethylene	Polypropylene	Polyvinylchloride
Burst	3	1	4	2
Comp.	3	2	4	1
Peel	1	3	2	4
Quick-Leak	2	3	4	1

* Number 1 indicates strongest seal for each test.

Table VII

RESULTS OF t-TEST RUN ON RAW DATA TO
TEST THE EFFECT OF SEALER SETTINGS

H_0 : Seal₁ = Seal₂ t distribution with 8 d.f.
 $\alpha = .05$

Material	Rejection Region	t value
<u>Saran</u>		
Burst	$ t \geq 2.306$	1.667
Compression	"	.621
Quick Leak	"	2.000
End Peel	"	.080
Side Peel	"	.272
<u>Polyethylene</u>		
Burst	"	0
Compression	"	.534
Quick Leak	"	1.000
End Peel	"	3.929
Side Peel	"	.300
<u>Polyvinylchloride</u>		
Burst	"	0
Compression	"	1.389
Quick Leak	"	0
End Peel	"	.182
Side Peel	"	.060
<u>Polypropylene</u>		
Burst	"	5.0
Compression	"	.862
Quick Leak	"	0
End Peel	"	1.925
Side Peel	"	.540

Table VIII

RESULTS OF t-TEST RUN ON END AND SIDE
MEAN PEEL VALUES FOR EACH MATERIAL

$$H_0: \mu_{END} = \mu_{SIDE}$$

$$\alpha = .05$$

t distribution with 4 d.f.

Material	Rejection Region	t value
Saran	$ t \geq 2.776$	.198
Polyethylene	"	.108
Polyvinylchloride	"	.324
Polypropylene	"	.452

CONCLUSIONS

1. It appears that the method of test does have an effect on the results obtained as is brought out by the ranking in Table VI. A unanimous selection of the strongest seal is not possible from the results obtained. Because of the effect exhibited by the tests on results, seal evaluation should be based on the results of the test method which simulates the treatment the package will be subjected to in distribution and use.
2. The results of the t-tests performed on the data (Table VII) show that there is consistency within each method. The two sealer settings used for each material were selected by the writer to give (for all practical purposes) equally strong seals. This was done by using the three heat sealing variables in such a combination that, for example, a lower temperature in one setting would be offset by a higher pressure value in that setting (see sealer settings Tables I-IV). The results of the t-tests show that in 18 out of 20 tests, there is no significant differences at the .05 level between the values obtained from the two sealer settings for all materials and tests.

3. The peel test was run on samples of both the side and end seals of the pouches. It wasn't known whether the orientation of the film would effect the seal strength values obtained. Analysis of the data showed there to be no significant difference between the values obtained for the end and side seals for all materials tested (Table VIII).
4. The control specimen results show that the burst, compression, and quick-leak tests are much more sensitive to the type of seal control used than the peel test. In fact, some control values for peel are higher than some of the standard peel test specimens.
5. The type of failure is dependent on the strength of the seal. A seal which is stronger than the material will invariably result in a cohesive failure at the inside edge of the seal. This is the weakest point because of the thinning of the material in this area when the pressure and heat are applied to fabricate the seal. Adhesive failure rarely occurs because a good seal is usually stronger than the sealed material.
6. The area of the majority of the failures was in the end seal. This may have been brought about because of the shape of the pouch.

SUGGESTIONS FOR FURTHER WORK

1. Plot correlation curves for the various tests by running tests at a number of different sealer settings.
2. Expand experiment to include other materials and tests.

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