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TECHNICAL REPORT

73-32-GP

MEASUREMENTS ON THE RESISTANCE OF FLEXIBLE PACKAGING
MATERIALS TO PUNCTURE, ABRASION, AND FLEXURE AND THE
RELATIONSHIP OF THESE MEASUREMENTS TO THE PERFORMANCE OF
PACKAGES SUBJECTED TO CONDITIONS CAUSING PINHOLE FORMATION

by

Kwoh H. Hu

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January 1973

UNITED STATES ARMY
NATICK LABORATORIES
Natick, Massachusetts 01760



General Equipment & Packaging Laboratory

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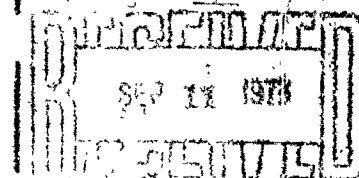
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Tests	10		8		8	
Laboratory	0		0		0	
Measurement			8			
Identifying			8			
Polymeric films			9			
Lamirates			9			
Aluminum foil			9			
Pinholes			4			
Pinhole formation			4		8	
Mechanical properties			8			
Assessment					8	
Piercing					8	
Flexural strength					8	
Abrasive tests					8	
Exposure					10	

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Empirical equations have been established between the laboratory determination of puncture resistance and the probability of package failure in the field to facilitate the prediction of package performance in the field from laboratory measurements.

Although only freeze-dried foods in vacuum-packed flexible packages were used in this field study, the technique of using instrumental measurements to rank materials in the laboratory for the prediction of relative package susceptibility to failure in the field by puncture, abrasion, or flexure could be applied to other types of products, flexible packaging systems, and package use situations for which damage by pinhole formation is important.

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CAUSING PINHOLE FORMATION

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Project Reference:
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ABSTRACT

The objective of this study is to develop the capability to predict the relative performance of flexible packages in the field from measurements of the mechanical properties of the packaging materials in the laboratory.

Methods and appropriate instrumentation were developed to closely simulate and measure three damage modes recognized as occurring in flexible packages under some use situations -- puncture, flexure, and abrasion. Pinhole formation resulting from exposure to these damaging actions was used as the criterion for assessing material resistance to such damage. Selected films and laminates were ranked by these test methods with respect to their resistance to pinhole formation by puncture, flexure, and abrasion. A simulated combat use test was conducted at Fort Lee, Virginia, to ascertain the relevancy between the laboratory instrumental determination of these mechanical properties of laminates and the actual field performance of freeze-dried foods using these laminates. It was found that laboratory instrumental determinations of puncture and abrasion resistances showed excellent rank correlation with the package failures recorded in the field, while the determination of flexing action resistance in the laboratory showed only fair to no correlations with field performance of the food packages tested. It is therefore concluded, supported also by scanning electron microscope examinations, that abrasion and puncture damage which cause pinhole formation in the packages are the main factors causing package failure in this field use situation.

Empirical equations have been established between the laboratory determination of puncture resistance and the probability of package failure in the field to facilitate the prediction of package performance in the field from laboratory measurements.

Although only freeze-dried foods in vacuum-packed flexible packages were used in this field study, the technique of using instrumental measurements to rank materials in the laboratory for the prediction of relative package susceptibility to failure in the field by puncture, abrasion, or flexure could be applied to other types of products, flexible packaging systems, and package use situations for which damage by pinhole formation is important.

Measurements on the Resistance of Flexible Packaging Materials to Puncture, Abrasion, and Flexure and the Relationship of these Measurements to the Performance of Packages Subjected to Conditions Causing Pinhole Formation.

INTRODUCTION

The common practice in the design and selection of packaging is to construct packages from each material under consideration and to fill them with the actual product to be packaged. The filled packages are then subjected to rough handling in a simulated use test to establish the relative practicality of the alternative materials under consideration. This practice is usually time-consuming and costly. A capability to predict relative package performance with respect to a given damage mode from laboratory tests on a variety of packaging materials would be highly desirable, such a capability could markedly reduce the number of alternatives which must be tested.

For packages utilizing foil-laminated flexible packaging materials, the final selection of material must be a compromise recognizing tradeoffs among many factors. Economic and availability factors are highly critical. Furthermore, packages must not fail as a result of poor sealability or as the result of delamination due to poor bonds between the laminae as well as deleterious environmental effects upon the laminae or bonding agents. In addition, the packaging material must be able to withstand physical abuse to which it might be subjected and which can produce breaks or pinholes in the laminate.

Pinhole formation is recognized as an important cause of package failure associated with the generation of breaks in the laminate due to physical abuse and damage. This form of damage is especially evident when products with sharp edges, such as dehydrated or freeze-dried food products, are inclosed in flexible packages and are subjected to application of a vacuum in the course of packaging. However, there are two types of pinhole damage that can be recognized in a foil-laminated package. One involves the breaking of the aluminum foil only, the other layers of material remaining intact. We call this the "foil-break pinhole". The other involves the complete puncture of all layers in the laminate. We call it the "complete-puncture pinhole". It is the complete-puncture pinhole which has been proven to be the most important factor causing the vacuum-pack package to lose vacuum and allowing water vapor and oxygen to enter the package.¹ In this paper we are dealing solely with complete puncture pinholes. They are called simply "pinholes" for brevity in this paper.

It has been recognized in previous studies that pinhole formation can be attributed to three modes of physical damage. These are puncture, flexure, and abrasion.² For example, pinholes due to puncture can occur when products with sharp corners such as dehydrated carrots or apple slices are inclosed in a flexible package. Pinholes due to flexing action can occur when the fold or seal area of a package is flexed during transportation and handling. Pinholes due to abrasion can occur when high points or curved areas of a package rub against another package or against the exterior container.

In military packaging, because of the space saving problem and a greater ease of carrying ration items in a soldier's pockets, vacuum packaging of foods in flexible packages is practiced to a large extent. For this reason much attention will be devoted in this paper to the measurement of package performance for vacuum-packed items.

The first objective of this study is to define the necessary instrumental techniques and to measure those mechanical properties of polymeric films and laminate packaging materials which have bearing on flexible package failure in the field through the formation of pinholes. The second objective is to establish a relationship between the mechanical property data obtained in the laboratory and the package performance observed in a highly destructive field situation. And the third and ultimate objective is to develop a capability to predict relative package performance in the field with respect to pinhole formation from measurements of the mechanical properties of flexible films and laminates made in the laboratory so that it will not be necessary to construct and test an actual package for each alternative packaging material.

METHODS AND MATERIALS

I. Instrumental Techniques for the Measurement of Mechanical Properties of Flexible Packaging Materials

Instrumental methods employed are based on the consideration that pinhole formation in a flexible package represents damage resulting from either puncture, flexure, or abrasion. Most existing test methods for polymeric materials do not simulate the type of pinhole observed in our flexible packaging materials, therefore they were not employed in our testing.³ Two existing test methods were modified and one new technique was developed to provide test procedures which corresponded to pinhole formation due to abrasion, flexing, or puncture.

A. Resistance to Puncture

Methods for measuring puncture resistance of polymeric materials have been described by Furno et al and Lynch.^{4,5} Furno used a 6.4-mm steel rod as a plunger, while Lynch used a rounded cylindrical penetrator, 4.8 mm in diameter. Based on our observations, the pinholes that developed in flexible packages were much smaller in size than the two penetrators employed. Pinholes observed are on the order of 0.25 mm in diameter and sometimes less.¹ An ASTM penetration needle of 0.14- to 0.16-mm diameter at the top of the needle was used for puncture experiments.⁶ The overall testing machine included an Instron Testing Machine with compression-load cell attached. An integrator was connected with the Instron, as shown in Figure 1. Not only maximum force required, but also total energy absorbed in puncturing were measured. The penetration needle was held vertically in the compression cell. The flexible material was placed in a holder which consists of a modified Thwing-Albert Vapometer cup provided with two circular rigid metal disks having a 1.27-cm diameter hole cut in the center of each. A rubber O-ring was placed between the disks along with the sample of flexible material, and the assembly was then clamped into the cup. The completely assembled holder was attached to the movable bar of the Instron machine directly above the compression cell (Figure 2).

The Instron machine was set up as follows:

- a. CB compression cell, range 100 to 2,000 gm.



Figure 1. An Instron Machine with an Integrator Connected for Measuring Forces and Energies Causing Puncture

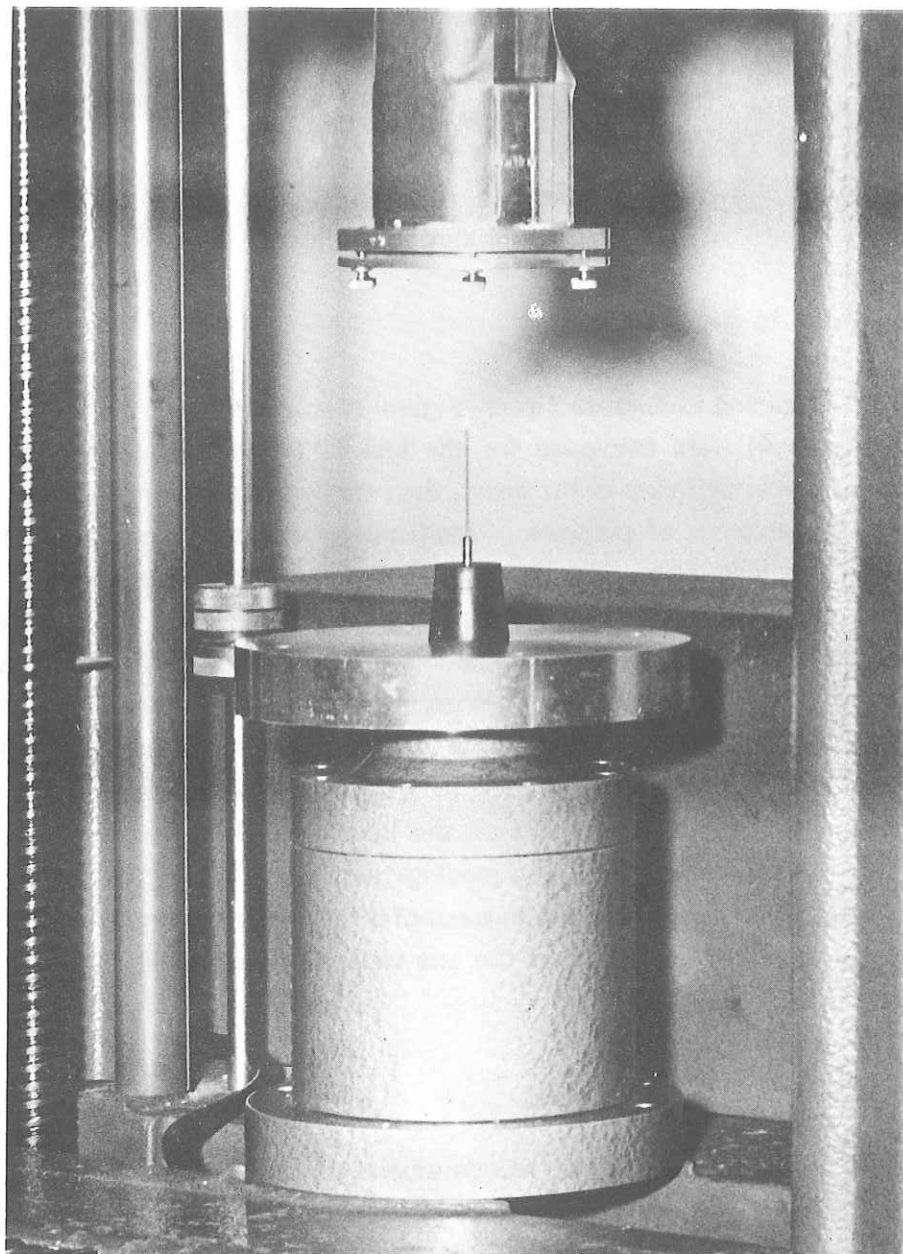


Figure 2. The Assembled Sample Holder and Penetration
Needle Used in Puncturing

b. Jaw speed 0.2 inches per minute.

c. Chart speed 5.0 inches per minute.

Ten readings for each material were taken and an average of these readings was made.

B. Resistance to Flexure

An MIT Folding Endurance Tester (Figure 3) together with a specially designed vacuum box (Figure 4) were employed for the test.⁷ The test sample was first given a predetermined number of flexes in the tester, then removed and mounted on the vacuum box to test for the presence of pinholes. Specifically, the test specimen was cut 14 by 3 cm, folded in half lengthwise, with the heat-sealable surface inside the fold. It was placed in the tester jaws with the folded edge facing the body of the machine. The tension of the jaws was set at 500 g. The speed was set at 180 cycles per minute. After the predetermined flexing cycle was completed, the sample was unfolded and taped to the top of the vacuum box with a strip of filter paper underneath. A drop of dye solution was placed at the flexing point of the test sample. A 59.7-cm vacuum was drawn at the vacuum box. The appearance of dye on the filter paper indicated that flexing had caused a pinhole in the test sample. Ten readings were taken at each number of cycles selected for testing. When nine or ten readings out of a total of ten showed dye penetration, this number of cycles was then taken as the one which causes pinhole formation in the material under test.

C. Resistance to Abrasion

A new corner abrasion tester was developed and used in our testing of flexible package materials.⁸ A 7.6-cm by 7.6-cm section was cut from the test sample. It was folded diagonally once, forming a triangle. It was folded again in the middle of the first fold, forming a second triangle which was half the size of the first triangle. The twice-folded sample was then inserted into a holder in such a manner that its tip was placed against an abrasive material, as shown in Figure 5. The abrasive material, emery polishing paper grit No. 4/0, was mounted on a turntable. Upon the turning of the turntable, the tip of the test sample was rubbed against the surface of the abrasive paper. The speed of the turntable was set at 50 rpm. The test sample was removed from the holder after a predetermined number of revolutions was completed. A drop of dye solution was placed inside the sample fold, and a piece of filter paper was used to touch the

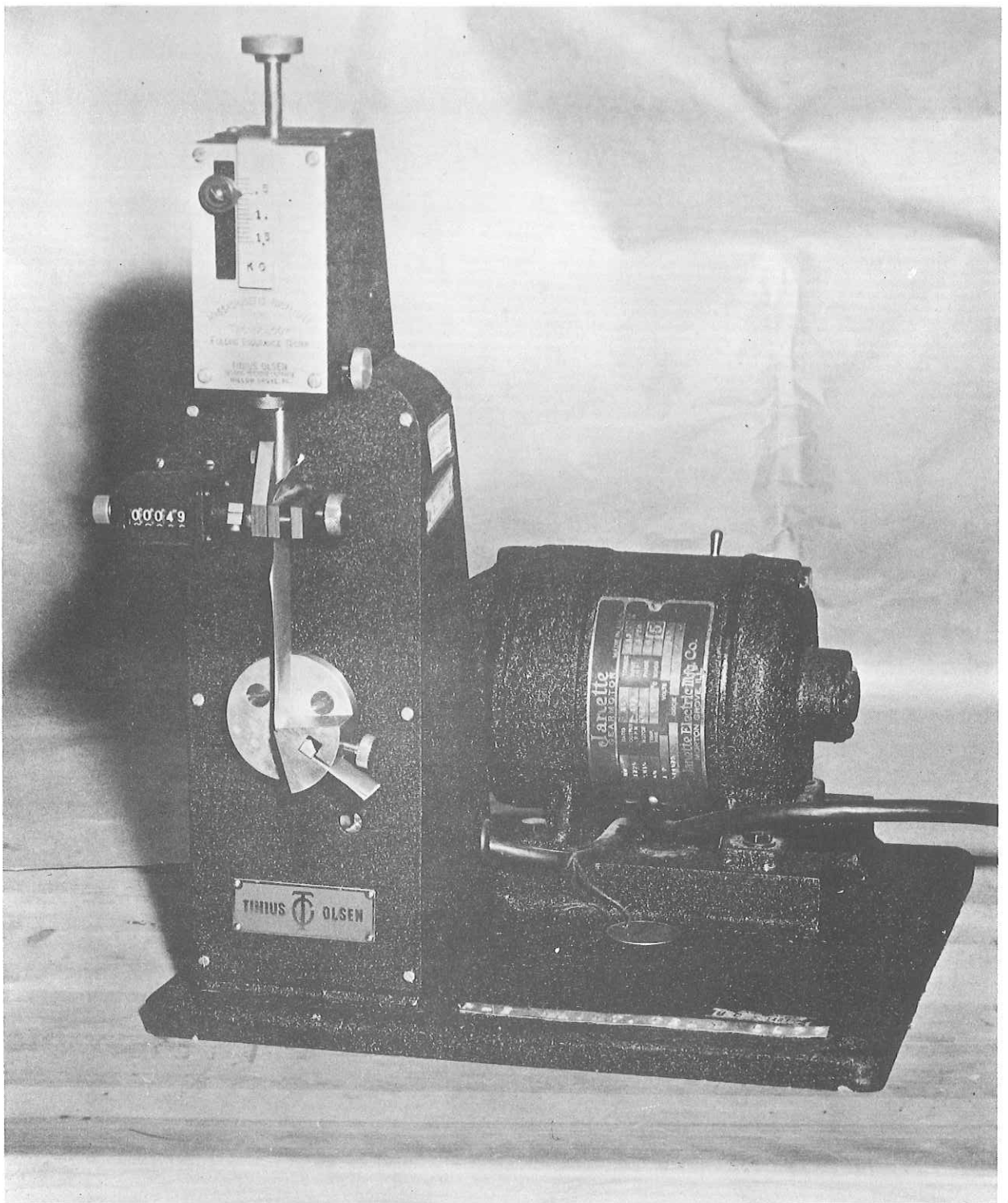


Figure 3. An MIT Folding Endurance Tester used in Testing Flexing Action

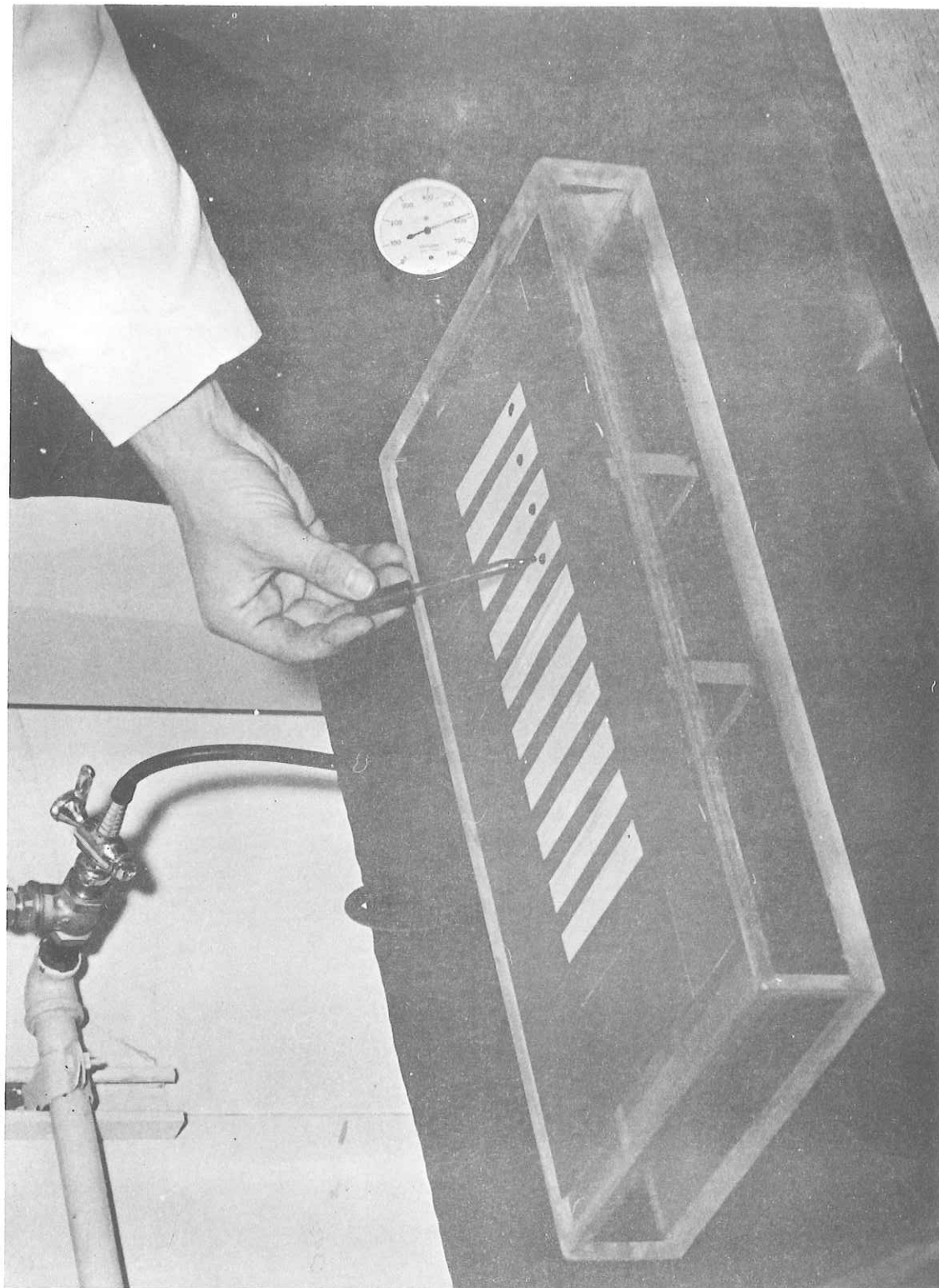


Figure 4. A Vacuum Box used in Detecting Pinholes

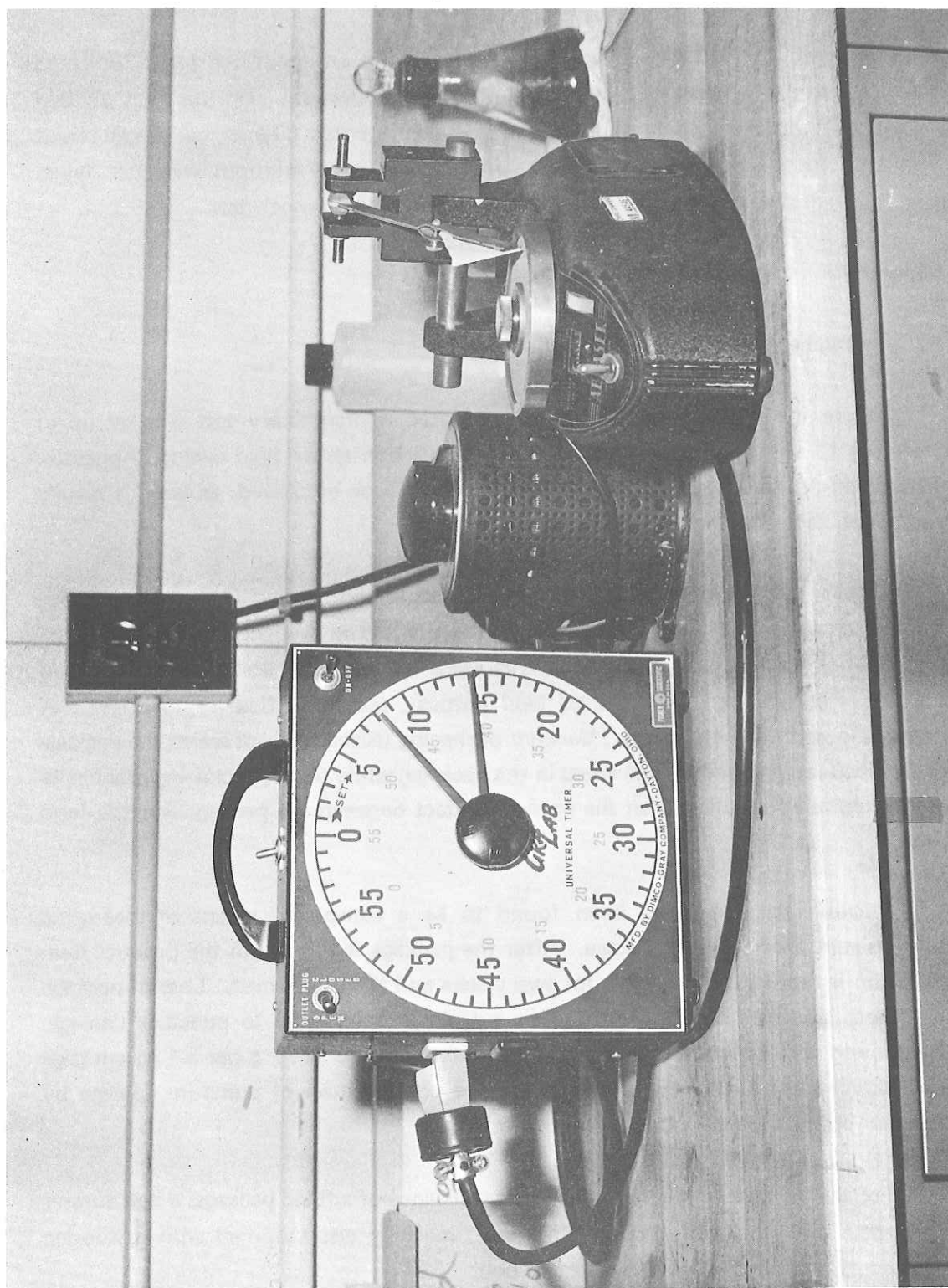


Figure 5. A Corner Abrasion Tester in Operation

tip of the fold on the outside. The appearance of dye on the filter paper indicated that a pinhole had developed in the test sample due to abrasion. Ten readings at each number of revolutions selected for testing were taken. When nine or ten readings out of a total of ten showed dye penetration, this number of revolutions was then taken as the one which caused pinhole formation in the material under test.

II. Laboratory Testing of Food Packages

A. Packaging Materials and Methods Used

Before the actual field testing was initiated a preliminary test was set up in the laboratory to assess the validity of our selection of materials for field testing. Appendix VIII lists package materials, foods, and packaging methods employed, as well as results obtained from this preliminary testing.

The six package materials selected for this study represented a range of resistance to pinhole formation and were selected from those listed in Appendices I to IV. The three food products selected represented three levels of hardness: very hard (dehydrated apple slices), medium hard (freeze-dried beef patties), and soft (flour). Two levels of stress were imposed on the package. Vacuum packaging (evacuated and sealed the package at 73.7-cm vacuum) imposed a high stress in the package, while the air packaging practically imposed no extra stress other than the normal contact between the package and the food contained.

Vacuum packaging has been found to be a convenient means of measuring puncture resistance of a filled package. After the package is filled with the product item and a vacuum is drawn, it is set aside for two weeks and then examined. Loss of package vacuum (loose package) can be regarded as a package failure due to puncture damage. This is followed by a compressed-air under-water test (with a 1816 g per 5.1 sq cm gage air pressure introduced into the package) for the confirmation of puncture damage by watching air bubbles coming out from the puncture.

For the purpose of measuring abrasion resistance of a filled package, a belt abraser was developed. A Rockwell abrasive finishing machine was modified with a wooden frame attached to the top of the abrasive belt.

There were two compartments within the wooden frame as shown in Figure 8. Packages were placed in these compartments so that when the machine was turned on the packages were rubbed against the abrasive belt. Jewelox cloth 320 x 305⁷¹ was selected as the belt material. In each test the belt abraser was run for one minute at 29 rpm.

Efforts were also made to develop a laboratory machine for measuring resistance to flexure for a food-filled package. None of the machines tried showed a sufficient degree of reproducibility. Therefore, the effort was abandoned.

B. Food Products Employed

The hardness and texture of the food products employed were determined by the Food Chemistry Division, Food Laboratory, NLABS. The purpose was to find out the relationship between the product texture and package failure in a vacuum-packed flexible package. Penetration tests were carried out using a 10-mm diameter punch at a speed of 2 cm/min on an Instron Universal Testing Apparatus. In measuring dehydrated apple slices, the only meaningful parameter was the maximum force (F_M) obtained during compression. Dehydrated apple slices were twisted and curled and good contact between the punch and sample, and between sample and load cell, could not be obtained. In measuring freeze-dried beef patties, the parameter of maximum force was not very meaningful because the product lacked a definite rupture point. The force would increase with increasing deformation while the material was being compacted. With flour the penetration force was extremely low. The only meaningful indication was the maximum force. Other important measurements obtained for freeze-dried beef patties, freeze-dried pork with scalloped potatoes, and freeze-dried spaghetti with meat sauce were 1) the force at rupture (F_R) which was related to the ultimate strength of the product, 2) the strain at rupture (E_R) was ultimate strain which was related to the percent of deformation the product would tolerate before rupturing, and 3) the work at rupture (W_R) which was the area under the force-deformation curve.

III. Field Testing of Food Packages

A simulated combat use test of flexible packages filled with freeze-dried foods was conducted at Fort Lee, Virginia, on 3 through 18 June 1971. Five types of flexible

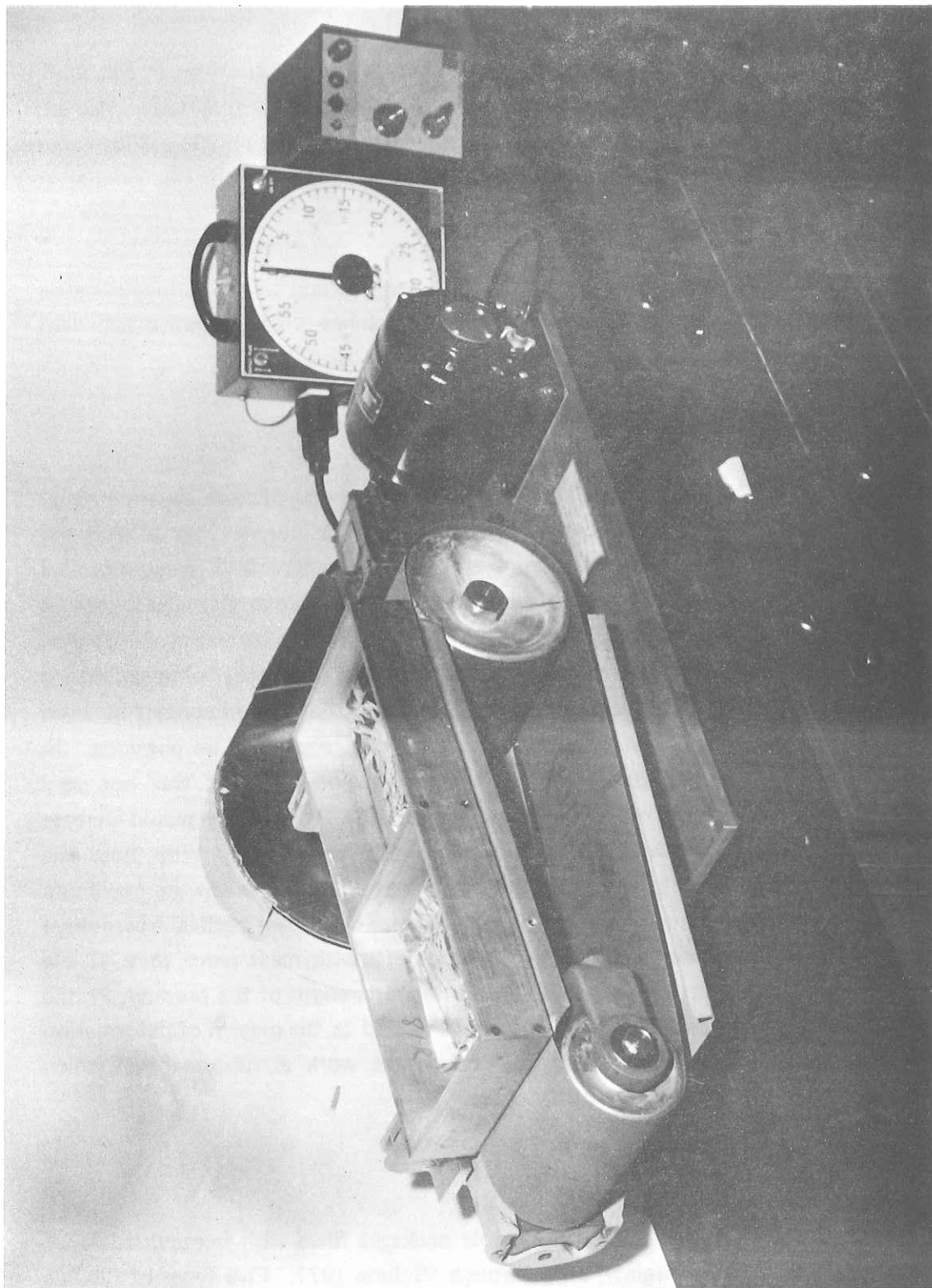


Figure 6. A Belt Abraser in Operation

packages filled with two freeze-dried foods were tested. The package size was 11.4 cm x 17.8 cm, and each package was filled with approximately 40 grams of freeze-dried product. Packages were heat sealed under a vacuum of 74.9 cm. The seal area of the filled package was folded over to reduce the size of the package and the filled package was placed in a 14 cm x 16.5 cm scrim/A1. foil/polyethylene pouch (Military Specification MIL-B-131D) sealed on three sides. Scrim pouch was sealed on three sides to allow the filled packages to be examined as many times as necessary during the course of field testing. Detailed field testing procedures were described in the Final Letter Report⁹ issued by the U. S. Army General Equipment Test Activity at Fort Lee, Virginia. Briefly, ten enlisted men carried flexible packages in the pockets of standard field clothing, including field jackets, while traversing the obstacle course. The course consisted of nine obstacles. Two traversals of the course constituted one test run. All packages were subjected to two traversals of the obstacle course unless failure occurred earlier. There were two inspection stations for each traversal of the course. Four inspections were made to identify the point of package failure during each test run. Figures 7 and 8 show enlisted men traversing two obstacles of the course. One thousand packages were field tested. A detailed visual examination of each package was made at each checkpoint. A loose package showing loss of vacuum constituted a failed package. These package failures were further confirmed in the laboratory under-water test with compressed air.

RESULTS AND DISCUSSION

I. Instrumental Measurements on Films and Laminates

A. Resistance to Puncture

Results of measuring single film resistance to puncture are listed in Appendix I, and laminate resistance to puncture in Appendix II. Testing of different film thicknesses of polyamide (Nylon 6), polyethylene terephthalate, polytrifluoromono-chloroethylene, low density and medium density polyethylenes shows clearly (see Appendix I) that an increase in thickness of flexible material increases its puncture resistance. It should also be noted (see Appendix II) that there are notable differences in forces and energies between the puncture initiated from the "outside" or from the "inside" of a laminate. When the puncture is initiated from the heat seal side, i.e., the polyethylene or vinyl chloride



Figure 7. Enlisted Men Carrying Flexible Packages in Pockets of their Clothing while Traversing Obstacle Course



Figure 8. An Enlisted Man Going Down a Slide and at a Checkpoint

layer side in the laminates used, it is referred to as from "inside"; if initiated from the laminate side corresponding to the exterior of a package, i.e., from the polyethylene terephthalate side of a laminate, it is referred to as from "outside". There has been no general rule found as to which direction of puncturing requires greater force and energy. It seems to depend on the thickness and physical characteristics of the components that make up the laminate.

1. Effects of Lamination

When several films are brought together to form a laminate, thickness is increased, and therefore puncture resistance is increased. When the relationship between the puncture resistance of a laminate and that of the components that make up the laminate is examined, the former is usually less than the sum of the latter. In other words, we usually do not get the total amount of puncture resistance expected on the basis of individual components in the laminate. This is indicated by our results as shown in Table I. For example, in Table I for the first laminate, we determined that the maximum force required to puncture the laminate from the polyethylene side was 178 g, which is smaller than the sum of the components determined separately of 205 g. This effect was also observed for the total energy absorbed in puncturing.

Because of the adhesive used in the laminates and the possible synergistic effect of materials, one might expect the force required to puncture a laminate to be greater than the sum of the components determined individually. Our results show the contrary. An explanation probably lies in the analysis of strength of materials used.

2. The Analysis of Strength of Materials

It is well known that in an axially loaded member of two or more materials of the same length, the unit stress in each is directly proportional to its modulus of elasticity.¹⁰ It is also obvious that, other things being equal, failure resistance of a component is directly proportional to the tensile strength of the material involved. Because of these two determining factors -- modulus of elasticity and tensile strength -- there are several possible combinations in a laminate. For example, the component with high modulus of elasticity may have low tensile strength; or the component with low modulus of elasticity may have high tensile strength, in relationship to the other components. One

TABLE I
Puncture Resistance of a Laminate vs. Individual Components which Make Up the Laminate

Laminate Construction	Individual Components Determined Separately		Laminate Determined as a Unit		
			Maximum Force (gram)		Total Energy (cm-gram)
	Maximum Force (gram)	Total Energy (cm-gram)	from "Inside"	from "Outside"	from "Inside" from "Outside"
0.5 mil* Polyethylene Terephthalate	106	3.72			
0.35 mil Al. Foil	23	0.33	178	203	7.14
3 mil H/D Polyethylene	76	5.33			6.93
SUM	205	9.38			
2 mil Polyethylene Terephthalate	331	11.07			
0.35 mil Al. Foil	23	0.33	422	315	17.68
2 mil Vinyl Chloride	90	10.54			11.20
SUM	444	21.94			

*Since films and laminates are ordered in terms of mils in thickness, it is felt to be more understandable to the reader to express them in mils.

typical example is shown in Figure 9, in which F_1 and F_2 are forces required to cause failure of components 1 and 2 used in a laminate but determined individually; P_1 and P_2 are forces supported by components 1 and 2 when they are in a laminate under Load P_t .

In equilibrium, $P_t = P_1 + P_2$

When load P_t is increased and displacement is increased, component 1 (supposed to have high modulus of elasticity but low tensile strength), as shown in Figure 9, carries more of the load,

until $P_1 = F_1$ (component 1 fails)

then $P_2 = P_t$ (component 2 must now support the entire load),

P_t is increased further

until $P_t = F_2$ (laminate failure takes place)

\therefore at the laminate failure, $P_t = F_2 < F_2 + F_1$

The path of the load on a laminate is indicated by the direction of the arrows in Figure 9.

The above derivation implies that the two components in a laminate break at two different points in the loading process, thus contributing to a lower force than the sum of the two components determined individually. In spite of several possible combinations of modulus of elasticity and tensile strength of one component in relationship to other components, the force required to cause failure of a laminate is always less than, or at the most is equal to, the sum of the components determined individually. This behavior of the axially loaded member coincides with our observations for the puncture of laminates, including the case illustrated in Figure 9, where the two components in a laminate can actually fail at two different points as indicated by two peaks in the force displacement chart as shown in Figure 10. These conclusions were obtained according to our interpretation of strength of materials in a laminate. They appear to be consistent with the results obtained experimentally in our measurements of puncture resistance of flexible laminate materials.



Figure 10. Force displacement chart showing two peaks of two laminates

B. Resistance to Flexure

Results of measuring single film and laminate resistance to flexure are listed in Appendix III. Testing of different film thicknesses of polyethylene terephthalate and polytrifluoromonochloroethylene shows clearly that an increase in film thickness decreases their resistance to flexure. This observation also applies to the effect of lamination. In lamination the thickness is increased; as would be expected, the resistance to flexing action of laminates decreases drastically as shown in Table II.

C. Resistances to Abrasion

Results of measuring single film and laminate resistances to abrasion are listed in Appendix IV. Testing of different film thicknesses of polytrifluoromonochloroethylene shows clearly that an increase in film thickness increases its resistance to abrasion. This observation also applies to the effect of lamination. In lamination the thickness is increased; as would be expected, the resistance to abrasion of a laminate increases drastically, as shown in Table III.

D. Laminate Variations in Resistance to Pinhole Formation

When puncture, flexing action, and abrasion resistance are all taken into consideration, many laminates under study show up strong in one or two resistance properties but weak in others. Typical examples are shown in Table IV. With the available laminates which we have determined, few of them can be considered to be strong in resistance to all three damage modes. For practical applications compromises sometimes have to be made, and the mode of damage most important in the packaging application should be ascertained before an intelligent choice of laminate material can be made.

E. Effects of Variation in Testing Speed

Consideration was given to the use of different speeds in test procedures. The objective was to determine if speeds other than the one used in the adopted test procedure would change the relative position of materials with respect to their resistance to pinhole formation.

TABLE II
Effects of Lamination on Resistance to Flexure

Laminate Construction	Cycles of Flexure to Cause Pinhole Formation		
	3 Individual Components Determined Separately	2-Component Laminate (a) and (b)	3-Component Laminate (a), (b), & (c)
(a) .5-mil Polyethylene Terephthalate	10,000		
(b) .35-mil Al. Foil	23	3,000	700
(c) 3-mil H/D Polyethylene	5,000		

TABLE III

Effects of Lamination on Abrasion Resistance

Laminate Construction	Revolutions of Abrasion to Cause Pinhole Formation	
	3 Individual Com- ponents Determined Separately	3-Component Laminate (a), (b), & (c)
(a) 2-mil Polyethylene Terephthalate	50	900
(b) .35-mil Al. Foil	5	
(c) 2-mil Vinyl Chloride	10	

TABLE IV
Laminate Differences in Resistance to Damage Mode

Laminate Construction	Puncture Resistance (force in gm from "inside")	Flexure Resistance (cycles to pinhole formation)	Abrasion Resistance (revolutions to pinhold formation)
1. 2-mil Polyethylene Terephthalate/.35-mil Al. Foil/2-mil Vinyl Chloride	518	3,000	900
2. .5-mil Polyethylene Terephthalate/.35-mil Al. Foil/3-mil HD Polyethylene	178	700	1,000
3. .5-mil Polyethylene Terephthalate/.35-mil Al. Foil/3-mil Polyolefin Blend	148	more than 10,000	800

NOTE: The No. 1 construction, 2-mil polyethylene terephthalate/.35-mil Al. foil/2-mil vinyl chloride, is too stiff to be used in flexible packages. It is used here only for material comparison purposes.

1. Resistance to Puncture

The jaw speed in the Instron machine (controlling the penetration needle travel) used in our routine testing was 0.2 inches per minute. With our experimental setup we found that the maximum speed which can be employed and still get accurate readings was one inch per minute. At a speed of two inches per minute the laminate usually broke too quickly to get accurate readings. In general, our results indicated that the maximum force and energy required for puncture increased when the speed of puncture was increased. However, when speed was increased from 0.2 to 1 inch per minute, the relative position of laminates did not change, as shown in Appendix V.

2. Resistance to Flexure

Our results did not permit us to draw a firm conclusion as to the effects of flexing speed on flexure resistance. Results from two speeds of flexing action are shown in Appendix VI and show no change in the relative position of materials under study.

3. Abrasion Resistance

Our results show that an increase in the turntable speed of our abraser increases the number of samples with pinhole formation. In other words, increasing abrasion speed has a more damaging effect on the samples tested. In general, the relative position of materials with respect to abrasion resistance did not change. Data obtained are presented in Appendix VII.

II. Laboratory Testing of Food Packages

A preliminary test of food packages was conducted in the laboratory prior to the actual field test to determine if the failure of packages would have a correlation with the resistance to physical damage of the packaging materials. Table V summarizes the mechanical properties of package materials used in the test, while Table VI summarizes the texture and hardness of the food products used. Due to ease of obtaining samples for testing, dehydrated apple slices, freeze-dried beef patties, and cake flour were used for this preliminary testing, while freeze-dried pork with scalloped potatoes and freeze-dried spaghetti with meat sauce were used for actual field testing.

TABLE V
Mechanical Properties of Laminates Selected for Preliminary Testing

Packaging Materials	Resistance to Puncture		Resistance to Flexure (cycles to cause pinhole formation)	Resistance to Abrasion (revolutions to cause pinhole formation)
	Energy (gram-cm)	Maximum Force (gram)		
1. 0.5-mil Polyethylene Terephthalate/0.35-mil Al. Foil/ 3.0-mil Polyolefin	2.14	157	> 10,000	800
2. 0.5-mil Polyethylene Terephthalate/0.35-mil Al. Foil/3.0-mil H.D. Polyethylene	2.63	178	700	1,000
3. 0.5-mil Polyethylene Terephthalate/0.35-mil Al. Foil/2.0-mil L.D. Polyethylene	1.84	138	500	50
4. 1.0-mil Nylon-6/10-ib Polyethylene/0.35-mil Al. Foil/ 2.5-mil L.D. Polyethylene	2.67	110	1,500	600
5. 2.0-mil Nylon-6/0.35-mil Al. Foil/ 3.0-mil Polyolefin	3.51	155	> 10,000	> 1,000
6. 2.0-mil Polyethylene Terephthalate/0.35-mil Al. Foil/ 2.0-mil Vinyl Chloride	10.31	508	3,000	1,000

TABLE VI
Texture and Hardness Determination in Food Products Employed

Food Products	Maximum Force (F_M , Kg)	Force at Rupture (F_R , Kg)	Strain at Rupture (E_R , %)	Work at Rupture (W_R , Kg-H)
1. Dehydrated Apple Slices	13.0	--	--	--
2. Dehydrated Beef Patties	--	3.93	13.4	0.417
3. Cake Flour	0.12	--	--	--
4. Freeze-Dried Pork with Escalloped Potatoes	1.27	0.98	13.5	0.129
5. Freeze-Dried Spaghetti with Meat Sauce	4.67	4.37	17.2	0.738

Results of this preliminary testing, because of their lengthy nature, are summarized in Appendix VIII. In examining the data presented in that Appendix, it is obvious that:

1. The harder the food product was, the greater the package failure for a given laminate material attributed to puncture and abrasion. For example, the dehydrated apple slices were very hard, causing the greatest amount of package failure; freeze-dried beef patties were of medium hardness, causing a medium amount of package failure; and the flour was very soft, causing little package damage in testing (food product hardness is listed in Table VI).

2. The vacuum packaging caused greater package failure, both in puncture (due to stress imposed on the package) and abrasion, than the air packaging.

In the process of evaluating package failure (as a filled package) against the instrumental determination of resistance to mechanical damage of a packaging material, a rank correlation was run.¹¹ The number of package failures observed for the dehydrated apple slices for each testing method were arranged in order of size. Then a rank was assigned to each size of damage. The largest number (size) of package failures was assigned a rank of 1, and the least number of package failures a rank of 6 (because 6 types of package were used in testing). In a similar way, the packaging laminate requiring the largest force to puncture in the instrumental determination was assigned a rank of 6, and that requiring the smallest force a rank of 1. Then a rank correlation coefficient was calculated. This and a comparable rank coefficient for energy to puncture, as well as one for package failure due to abrasion in relation to the testing of packaging material in our abrasion tester are listed in Table VII. A rank correlation coefficient of +1 indicates a perfect agreement. Therefore, the rank correlation coefficients found in our tests, ranging from 0.715 to 0.986 (see Table VII), showed good to excellent agreement between the instrumental determinations on materials alone and the testing of filled packages. These results gave us a strong indication that data obtained from instrumental determinations on films and laminates in the laboratory could be used for predicting package performance in the field.

III. Field Testing of Food-Filled Packages

A. Field Test Data and General Relationships Observed

A simulated combat use test (field test) was conducted at Fort Lee, Virginia, on 8-18 June 1971. Table VIII summarizes the package failures obtained in the field

TABLE VII
Rank Correlation of Package Failure and
Instrumental Determination of Packaging Materials

	Rank of Package Failure due to Vacuum Packaging Test	Rank of Force to Puncture Laminate	Rank of Energy to Puncture Laminate	Rank of Package Failure due to Abrasion Test	Rank of Pinhole Formation in Laminate Abrasion Test
1. 0.5-mil Polyethylene Terephthalate/0.5-mil Al. Foil/3.0-mil Polyolefin	4	4	2	2	2
2. 0.5-mil Polyethylene Terephthalate/0.35-Al. Foil/3.0-mil H.D. Polyethylene	3	5	3	5	4-1/2
3. 0.5-mil Polyethylene Terephthalate/0.35-mil Al. Foil/2.0-mil L.D. Polyethylene	1	2	1	1	1
4. 1.0-mil Nylon-6/10-lb ^g Polyethylene/0.35-mil Al. Foil/2.5-mil L.D. Polyethylene	2	1	4	3	3
5. 2.0-mil Nylon-6/0.35-mil Al. Foil/3.0-mil Polyolefin	5	3	5	6	6
6. 2.0-mil Polyethylene Terephthalate/0.35-mil Al. Foil/2.0-mil Vinyl Chloride	6	6	6	4	4-1/2
Rank Correlation Coefficient		0.715	0.771		0.966

*This is generally referred to as pounds of polyethylene extruded or coated on 3,000 square feet (279 square meters) of substrate material.

TABLE VIII

Package Failures Recorded during the Field Test

Test Code Number	Packages Made of	
1	0.5-mil Polyethylene Terephthalate/0.35-mil AL Foil/3.0-mil Polyolefin Blend	
2	0.5-mil Polyethylene Terephthalate/0.35-mil AL Foil/3.0-mil H.D. Polyethylene	
3	2.0-mil Nylon-6/0.35-mil AL Foil/3.0-mil Polyolefin Blend	
4	1.0-mil Nylon-6/10-lbs L.D. Polyethylene/0.35-mil AL Foil/0.5-mil L.D. Polyethylene	
5	0.5-mil Polyethylene Terephthalate/0.35-mil AL Foil/2.0-mil L.D. Polyethylene	
Food Products		
A	Freeze-Dried Pork with Scalloped Potatoes	
B	Freeze-Dried Spaghetti with Meat Sauce	

Package Code Number	Checkpoints					Checkpoints					Mean
	Package Failures Recorded in the Field, 100 Packages at Beginning of Test					Package Failures Adjusted to % of Those Carried at Each Checkpoint					
	1	2	3	4	Total	1	2	3	4	Total	
A 1	5	2	8	7	22	5	2	9	8	24	6.0
A 2	4	3	1	3	11	4	3	1	3	11	2.7
A 3	0	1	0	0	1	0	1	0	0	1	0.3
A 4	1	2	3	11	17	1	2	3	12	18	4.5
A 5	16	17	10	8	51	16	20	15	19	70	17.4
B 1	5	3	9	5	22	5	3	10	6	24	6.0
B 2	7	9	4	4	24	7	10	5	5	27	6.7
B 3	1	2	4	2	9	1	2	4	2	9	2.2
B 4	8	12	13	6	39	8	13	16	9	46	11.5
B 5	24	26	10	9	69	24	34	20	22	100	25.0

test. The data were taken directly from the final report issued by the U. S. Army General Equipment Test Activity, Fort Lee, Virginia' on their conduct of the field test. It should be noted that 100 of each of the 10 variations in package/food combinations were used in the test. A total of 1,000 packages were field tested. At each checkpoint (4 checkpoints in each test run) any package which showed failure by the evidence of a lost vacuum was taken out and replaced by a dummy package, thus diminishing the total number of packages in the test run to be completed. A calculated adjustment on a 100-package basis for each checkpoint was made and included in Table VIII.

Table IX shows the number of package failures recorded in the field test in relation to the instrumental determination of the packaging material properties conducted in the laboratory. When the data on puncture resistance are examined carefully, it is quite obvious that the combination of energy and maximum force to puncture ($E \times F$) matches the ranking of the probability of package failure much better than either the energy or maximum force related separately. There is a valid reason for this occurrence. The packaging materials which we used are all made of laminates consisting of several layers of material adhered together. Relatively, one layer of material may have a higher force than energy to puncture, while the other layer of material may have higher energy than force to puncture. Therefore, the combined data of energy and force ($E \times F$) show a better correlation to the observed package failures than the energy and force created separately. The factor of " $E \times F$ " is arbitrarily named "puncture resistance factor". It is recognized that a thorough dimensional analysis should be made to gain insight into the correlation between the instrumental determination and field performance. Due to the complex nature of the puncturing of a laminated material, our understanding of the puncture mechanism is not considered adequate for us at this time to proceed with the dimensional analysis on a logical basis.

Also, in Table IX rank correlation coefficients were calculated between the instrumental determinations and food products A and B packaged in five package types used in field testing. A rank correlation of ± 1 indicates a perfect agreement. It is clearly evident that both the puncture resistance factor and abrasion resistance show excellent rank correlations with the probability of package failure, while resistance to flexure shows only fair to no correlation. These correlation indicators imply that abrasion and puncture damage are the main factors in causing package failure in the field. The flexing damage mode apparently played a minor role in this field usage test.

*Refer to the code number listed in Table VIII
for packages and food products.

TABLE IX
The Relationship between Package Failures Recorded during Field Testing and the
Instrumental Determination of the Packaging Material Properties

Package Code Number*	Package Failure (%)	Laminate Resistance to Puncture		Laminate Resistance to Flexure Cycles to Cause Pinhole Formation	Laminate Resis- tance to Abrasion Revolutions to Cause Pinhole Formation
		Energy (gm-cm) (E)	Puncture Resistance Factor (Kenergy X Maximum Force (gm ² -cm)		
A 1	6.0	5.44	853	> 10,000	800
B 1	6.0				
A 2	2.7	6.68	1,194	700	1,000
B 2	6.7				
A 3	0.3	8.92	1,383	> 10,000	1,000
B 3	2.2				
A 4	4.5	6.78	719	1,500	600
B 4	11.5				
A 5	17.5	4.67	645	500	50
B 5	25.0				
Rank Correlation Coefficient:					
With Food A		-0.900		-0.425	
With Food B		-0.900		-0.825	

The instrument used for measuring abrasion resistance was a prototype developed in our own laboratory. It is not yet available outside of the U. S. Army Natick Laboratories. On the other hand, the Instron testing machine used for measuring puncture resistance is readily available and is a well developed instrument equipped with many control features. Since the puncture resistance data are easily and widely obtainable, it was decided to utilize these data for the prediction of package failure in the field. Figure 11 shows plots of probability of package failure against the puncture resistance factor. As can be readily seen, 1) the freeze-dried spaghetti with meat sauce which has a harder texture (see Table VI for texture and hardness determination in food products) creates a much higher package failure level than the freeze-dried pork with scalloped potatoes which has a relatively softer texture; 2) except for the packaging material number 5 (refer to Table VIII for the code number) which has a very high package failure level with low resistance to puncture for the laminate, the rest of the materials seem to have a linear relationship between the puncture resistance factor and the package failure level.

B. Empirical Relation between the Puncture Resistance Property of Laminates and the Probability of Package Failure

The empirical relation between the puncture resistance factor, t , and the probability of package failure under field test is found by assuming a normal distribution for the failure probability. Then a Least Squares polynomial fit is carried out between the reduced normal variable, y , and t^{-1} . The relation between y and t may be expressed as follows:

$$y = \sum_{j=0}^N C_j (t^{-1})^j \quad N = 1 \text{ or } 2$$

C_j are constants. The predicted probability of failure, P , associated with a material having puncture resistance factor, t , then is $P=F(y)$, where $F(y)$ is the distribution function for a standardized normal variable. Table X shows a listing of puncture resistance factor, t , vs. standardized normal variable, y . Since the relative puncture resistance factor of package No. 5 (.5-mil polyester/.35-mil Al. foil/2.0-mil L/D polyethylene) is very low, accompanied by an extraordinarily high package failure rate relative to the other materials tested, this type of material would ordinarily not be considered for use with freeze-dried food products in a vacuum pack under conditions for which the formation of pinholes by puncture was a critical damage mode. Therefore, data on this package were excluded

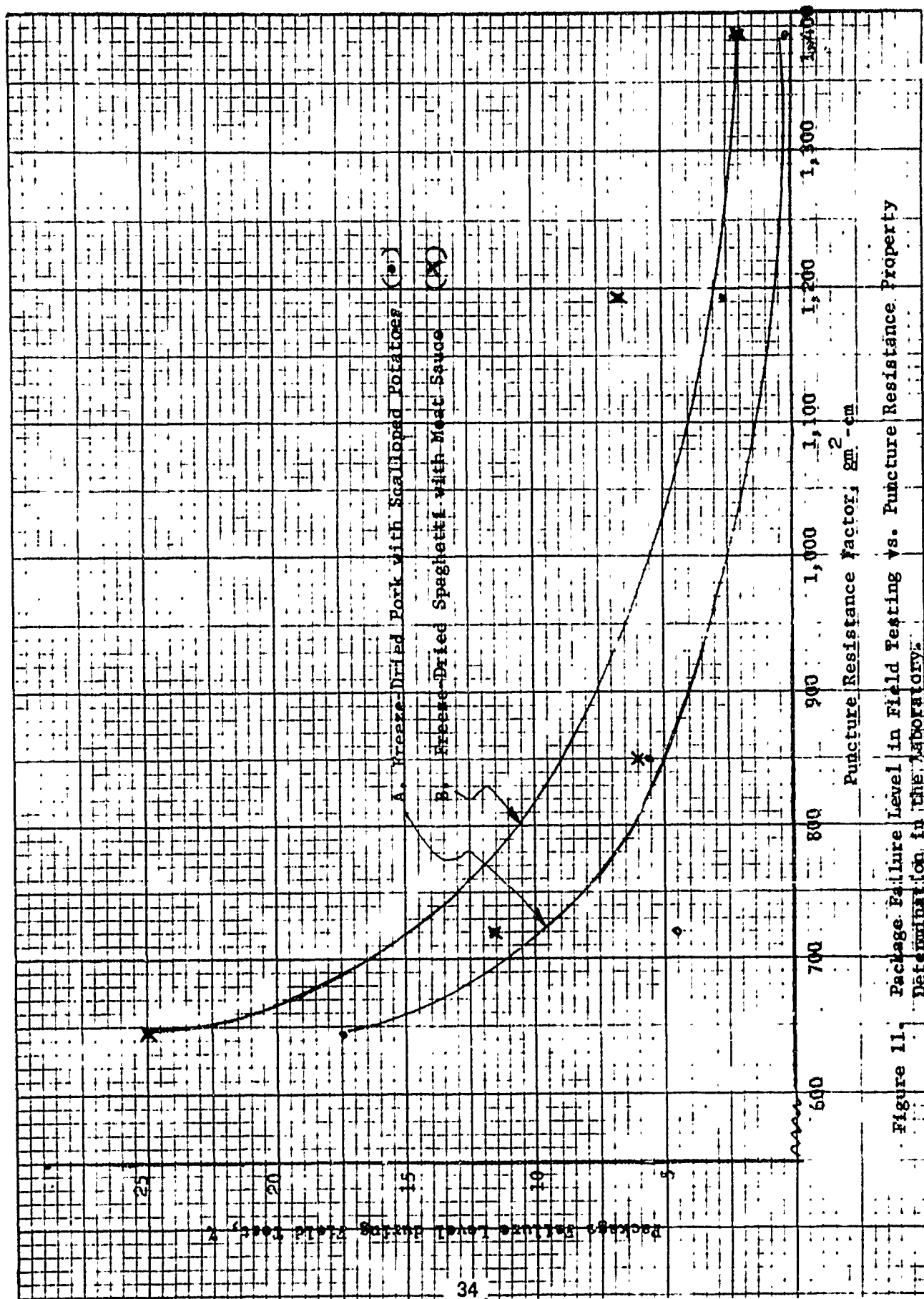


Figure 11. Package Failure Level in Field Testing vs. Puncture Resistance Property Determination in the Laboratory.

TABLE X

A Listing of Puncture Resistance Factor, t ,
and Standardized Normal Variable, Y

Package Code Number	Puncture Resistance Factor, t , $\text{gm}^2\text{-cm}$	$t^{-1} \times 10^2$	Food Code No. A		Food Code No. B	
			Package Failures in Field Testing $\% \times 10^{-2}$	Standardized Normal Variable, Y	Package Failures in Field Testing $\% \times 10^{-2}$	Standardized Normal Variable, Y
3	1,383	.072	.003	-2.75	.022	-2.01
2	1,194	.084	.027	-1.93	.067	-1.50
1	853	.117	.060	-1.56	.060	-1.56
4	719	.139	.045	-1.70	.115	-1.20
5	645	.155	.175	-0.54	.250	-0.68

in developing empirical equations. Table XI shows the linear as well as quadratic equations relating the variables. The linear relations were found to be almost as good as quadratic ones and were adopted for use.

IV. Scanning Electron Microscope for the Examination of Field Testing Samples

Samples from packages damaged during the simulated use test conducted at Fort Lee, Virginia, were examined under a scanning electron microscope. The purpose of the examination was to show evidence, if possible, of the mode of damage which occurred in the field usage. A high-resolution scanning electron microscope manufactured by Advanced Metal Research Corp., AMR Model 900, was used in this examination.

First, the hole size of the damage area was measured under a binocular microscope. The hole shape was quite irregular, ranging from nearly round to stringlike, and to concave in shape. Therefore, the size measurement was only an approximation which ranged from 9.6×10^{-2} to 1.4 sq mm.

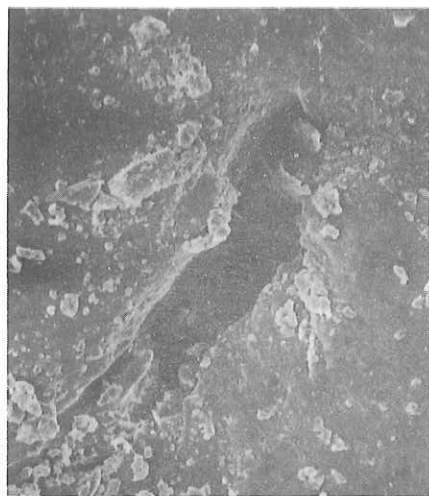
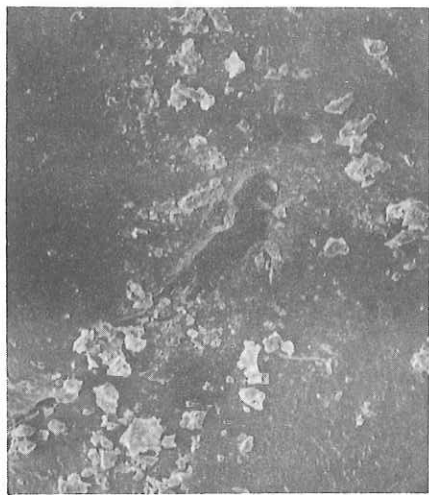
Pictures were taken under the scanning electron microscope. For comparison purposes, the first set of pictures was taken of the inside surface of the sample of a failed package and then of the outside surface of the sample package. Magnifications were 50X, 100X, and 200X. Figures 12 to 16 show the damage area of the five types of package filled with freeze-dried spaghetti with meat sauce. The top row of pictures shows the interior view of the damage area, while the bottom row shows the exterior view. They all seemed to have well defined holes or openings from the interior view, but smaller and sometimes partial openings from the exterior view. Moreover, in some cases, plastic laminates forming the packaging materials seemed to have been pushed out from the interior, causing them to form broken layers opening outward. These effects are especially apparent in Figure 13 and Figure 14.

Examinations made and pictures taken by using the scanning electron microscope indicated that the abrasion damage (friction between the hard edge of the freeze-dried product and the package) was the main factor in causing the package failure. This is consistent with the finding that the probability of package failure in the field has an excellent rank correlation coefficient with abrasion resistance and puncture resistance factors of materials determined in the laboratory.

TABLE XI
Empirical Relations between t and Y

Food Product A Freeze-Dried Pork with Scalloped Potatoes: $Y = -3.408 + 13.817t^{-1}$ $Y = -9.605 + 139.602t^{-1} - 597.465(t^{-1})^2$	Standard Error	Correlation Coefficient
		0.7949 0.9697
Food Product B Freeze-Dried Spaghetti with Meat Sauce: $Y = -2.511 + 9.158t^{-1}$ $Y = -3.074 + 20.591t^{-1} - 54.308(t^{-1})^2$	0.2230 0.3120	0.8387 0.8426

Interior View



Exterior View



50X

100X

200X

Figure 12. Scanning Electron Microscope Examination of Damaged Sample
2.0-mil Nylon-6/0.35-mil Al. Foil/3.0-mil Polyolefin

Interior View



Exterior View



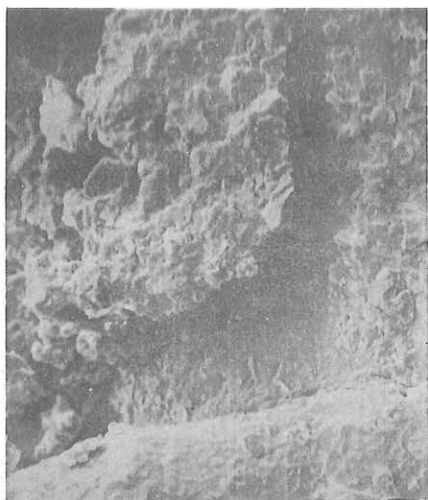
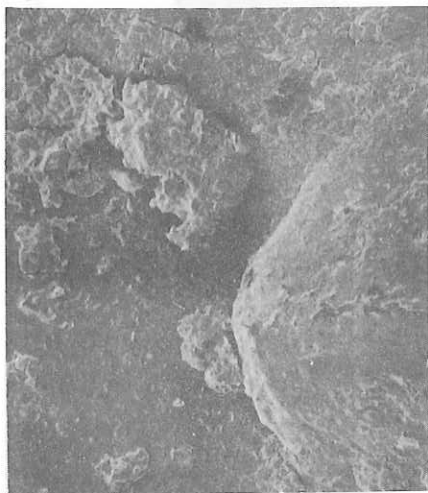
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100X

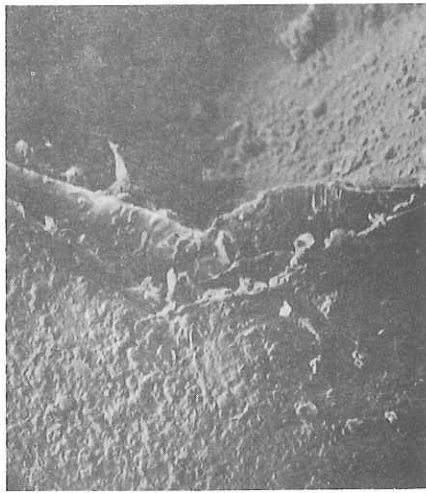
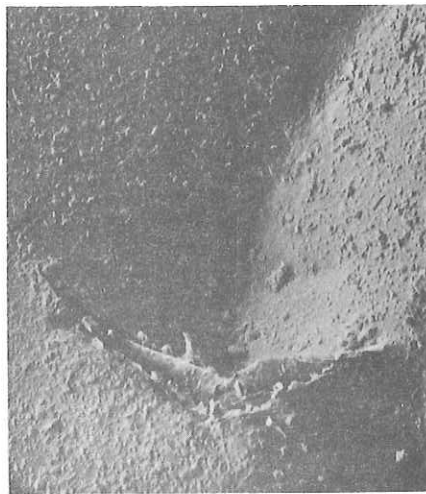
200X

Figure 13. Scanning Electron Microscope Examination of Damaged Sample of 0.5-mil Polyethylene Terephthalate/0.35-mil Al. Foil/3.0-mil Polyolefin

Interior View



Exterior View



50X

100X

200X

Figure 14. Scanning Electron Microscope Examination of Damaged Sample of 0.5-mil Polyethylene Terephthalate/0.35-mil Al. Foil/3.0-mil H.D. Polyethylene

Interior View



Exterior View



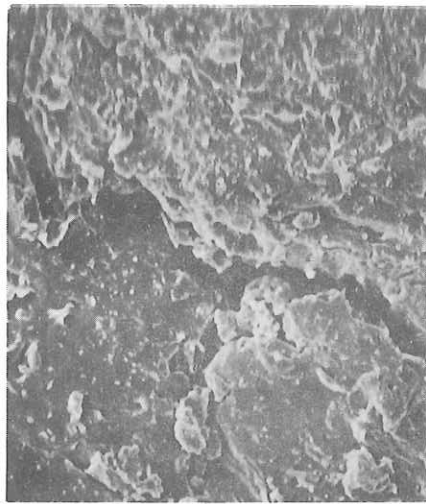
50X

100X

200x

Figure 15. Scanning Electron Microscope Examination of Damaged Sample of 1.0-mil Nylon-6/10-lb L.D. Polyethylene/0.35-mil Al Foil/0.5-mil Polyethylene Terephthalate

Interior View



Exterior View



50X

100X

200X

Figure 16. Scanning Electron Microscope Examination of Damaged Sample of 0.5-mil Polyethylene Terephthalate/0.35-Al. Foil/2.0-mil L.D. Polyethylene

SUMMARY

1. Methods and appropriate instrumentation have been either developed or adapted to closely simulate and measure three damage modes known to occur in flexible packages -- puncture, abrasion, and flexure. Pinhole formation resulting from these damaging actions was used as the criterion for measuring material resistance to such damage.

2. Important findings from laboratory instrumental determinations on films and laminates are:

a. An increase in thickness of flexible material increases its puncture and abrasion resistance but decreases its flexure resistance.

b. When several components (or films) are brought together as a laminate, the puncture resistance of the laminate is greater than the lowest resistance of the individual components, but it is usually less than the sum of the individual components.

c. When several components are brought together as a laminate, the flexure resistance of the laminate is reduced drastically. This is consistent with the results obtained in testing individual components with respect to material thickness in which the increase of material thickness decreases resistance to flexure damage.

d. When several components are brought together as a laminate, the abrasion resistance of the laminate increases dramatically.

e. Changes of speed within the limits allowed in the laboratory testing procedures do not appear to change the relative position of materials ranked with respect to resistance to pinhole formation.

3. Results of a preliminary testing of food packages in the laboratory clearly show that:

a. The harder the food product used, the greater the degree of package failure attributed to puncture and abrasion damage.

b. Vacuum packaging causes a much greater degree of package failure due to puncture and/or abrasion than air packaging (or N_2 packaging).

4. A simulated combat use test was conducted at Fort Lee, Virginia, to ascertain the relevancy of the package performance predictions from relative material rankings based on laboratory instrumental determination of film and laminate properties to the package behavior in actual field usage. It was found that:

a. For puncture damage correlation purposes, the ranking of laminate materials by the combination of energy and maximum force to puncture a laminate as determined in the laboratory (called puncture-resistance factor) matches the ranking of package failure in the field much better than is achieved by ranking materials by either the energy or force separately.

b. Both puncture-resistance factor and abrasion resistance as determined from laboratory measurements show excellent rank correlation with the package failures recorded in the field, while the laboratory determinations of flexure resistance show only fair to no correlations. It is therefore postulated that abrasion damage and puncture damage due to food contact with the packaging material which eventually causes pinhole formation in the packaging material is the main factor causing package failure in this field test situation, while the flexure damage mode only plays a minor role in this field usage test. The above conclusions are applicable only to this field test situation using selected freeze-dried foods in vacuum-packed flexible packages; the results do not imply that flexure cannot be an important damage mode under other field situations.

c. The above conclusion with respect to the important damage mode is further supported by a scanning electron microscope examination of damaged samples.

5. Empirical equations have been established between the puncture-resistance factor which can be determined by laboratory measurements and the probability of package failure in the field.

CONCLUSION

Methods and appropriate instrumentation have been developed to measure mechanical properties of film materials associated with damage by pinhole formation which is recognized as occurring in foil-laminated flexible packages when subjected to conditions permitting puncture, flexure, or abrasion of the laminate. It has been shown that these laboratory instrumental determinations can be used to rank materials in the laboratory with respect to the probability of failure in the field by these damage modes of packages made from these materials. Time and cost savings will be possible in evaluating newer materials and laminates for potential field usage where these damage modes are important through application of these laboratory techniques. Although only freeze-dried foods in vacuum-packaged flexible packages were utilized in this field study, the technique of using instrumental measurements to rank materials in the laboratory for the prediction of relative package susceptibility to failure in the field by puncture, abrasion, or flexure could be applied to other types of products, packaging systems, and package use situations for which damage by pinhole formation is important.

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Appendices

- I. Puncture Resistance of Single Films
- II. Puncture Resistance of Laminated Films
- III. Resistance to Flexure
- IV. Resistance to Abrasion
- V. Puncture Resistance at Different Speeds
- VI. Flexure Resistance at Different Speeds
- VII. Abrasion Resistance at Different Speeds
- VIII. Results of Laboratory Testing of Food Packages

APPENDIX I

Puncture Resistance of Single Films

<u>Films</u>	<u>Thickness (mil)</u>	<u>Energy to Puncture (gram-cm)</u>	<u>Maximum Force to Puncture (grams)</u>
Nylon 6	0.5	3.99	45.3
	0.75	4.55	58.1
	1.0	5.66	78.0
	3.3	9.93	195.0
	5.4	16.89	314.0
Polyethylene Terephthalate	0.5	3.73	106.0
	1.0	6.45	184.0
	1.5	9.37	271.0
	2.0	11.07	331.0
	4.0	18.44	252.0
Polytrifluoromonochloro- ethylene	0.6	1.22	40.5
	1.0	2.54	74.6
	2.0	3.53	107.0
	5.3	9.91	240.0
Low Density Polyethylene	1.25	1.65	21.6
	1.5	1.85	23.9
	2.0	2.31	31.2
	3.0	2.87	39.5
	4.0	3.73	50.5

APPENDIX I - Page 2

<u>Films</u>	<u>Thickness (mil)</u>	<u>Energy to Puncture (gram-cm)</u>	<u>Maximum Force to Puncture (grams)</u>
Medium Density Polyethylene	1.0	1.72	23.8
	2.0	2.46	38.6
	3.0	2.59	43.3
	3.5	2.69	44.4
High Density Polyethylene	3.0	5.33	76.6
Vinyl Chloride	2.0	10.54	90.0
Polyurethane	2.5	23.55	175.0
Modified Polyolefin	3.0	2.49	42.6
Polymer Coated Cellophane	1.5	1.72	89.0
Cellulose Acetate	1.0	2.79	108.0
25-Pound Pouch Paper	1.5	2.64	129.0
Aluminum Foil	0.35	0.33	22.7
	0.5	0.38	26.2
	1.0	1.24	63.0

APPENDIX II

Puncture Resistance of Laminated Films

Laminates	Thickness (mil)	Energy to Puncture (gm-cm)	Maximum Force to Puncture (gm)	
			from Inside	from Outside
Polyethylene Terephthalate/ Foil/Vinyl Chloride (from Company A)	2.0 polyethylene terephthalate			
	0.35 Al. foil	25.07	578	350
	2.0 vinyl chloride			
Polyethylene Terephthalate/ Foil/Vinyl Chloride (from Company B)	2.0 polyethylene terephthalate			
	0.35 Al. foil	17.68	422	315
	2.0 vinyl chloride			
Nylon-6/Foil/Vinyl Chloride	2.0 Nylon-6			
	0.35 Al. foil	12.83	196	208
	2.0 vinyl chloride			
Nylon-6/Foil/Vinyl Chloride (from Company B)	2.0 Nylon-6			
	0.35 Al. foil	11.96	191	209
	2.0 vinyl chloride			

APPENDIX II - Page 2

Laminates	Thickness (mil)	Energy to Puncture (gm-cm)		Maximum Force to Puncture (gm)	
		from Inside	from Outside	from Inside	from Outside
Polyethylene Terephthalate/ Foil/H.D. Polyethylene	0.5 polyethylene terephthalate 0.35 Al. foil 3.0 H.D. polyethylene	7.14	6.93	178	203
Polyethylene Terephthalate/ Foil/Polyolefin Blend	0.5 polyethylene terephthalate 0.35 Al. foil 3.0 polyolefin blend	5.46	5.26	142	152
Polyethylene Terephthalate/ Foil/Polypropylene (from Company C)	0.5 polyethylene terephthalate 0.35 Al. foil 3.0 polypropylene	9.55	9.04	1st peak = 170 2nd peak = 115	1st peak = 178 2nd peak = 100
Polyethylene Terephthalate/ Foil/Nylon-6	0.5 polyethylene terephthalate 0.35 Al. foil 2.0 Nylon-6	10.72	12.07	1st peak = 237 2nd peak = 166	1st peak = 212 2nd peak = 188
Polyethylene Terephthalate/ Foil/Vinyl Chloride	0.5 polyethylene terephthalate 0.35 Al. foil 3.0 vinyl chloride	10.69	15.42	1st peak = 202 2nd peak = 136	1st peak = 178 2nd peak = 177

APPENDIX II - Page 3

Laminates	Thickness (mil)	Energy to Puncture (gm-cm)		Maximum Force to Puncture (gm)	
		Inside	from Outside	from Inside	from Outside
Polyethylene Terephthalate/ P.E./Foil/Vinyl Chloride (from Company C)	1.0 polyethylene terephthalate			1st peak	1st peak
	10 lbs polyethylene			= 261	= 228
	0.35 Al. foil	13.49	12.98	2nd peak	2nd peak
	3.0 vinyl chloride			= 145	= 152
Polyethylene Terephthalate/ P.E./Foil/Vinyl Chloride (from Company D)	1.0 polyethylene terephthalate			1st peak	1st peak
	10 lbs polyethylene			= 223	= 223
	0.35 Al. foil	12.83	9.65	2nd peak	2nd peak
	3.0 vinyl chloride			= 124	= 124
Polypropylene/Cello./Foil/ Polyethylene	0.5 polypropylene			1st peak	1st peak
	1.0 cellophane			= 176	= 203
	0.35 Al. foil	6.55	5.66	2nd peak	2nd peak
	2.5 polyethylene			= 154	= 154
Polyethylene Terephthalate/ Foil/Polytrifluoromono- chloroethylene	0.5 polyethylene terephthalate			1st peak	1st peak
	0.35 Al. foil	8.23	7.29	= 201	= 201
	2.0 polytrifluoromono- chloroethylene			2nd peak	2nd peak
				= 133	= 133

APPENDIX II - Page 4

Laminates	Thickness (mil)	Energy to Puncture (gm-cm)		Maximum Force to Puncture (gm)	
		from Inside	from Outside	from Inside	from Outside
Polyethylene Terephthalate/ P.E./Foil/Polyethylene	1.0 polyethylene terephthalate				
	10 lbs polyethylene	11.68	6.15	295	214
	0.35 Al. foil				
	2.5 polyethylene				
Nylon-6/P.E./Foil/Poly- ethylene	1.0 Nylon-6				
	10 lbs polyethylene	6.40	6.35	121	126
	0.35 Al. foil				
	2.5 polyethylene				
Polyethylene Terephthalate/ P.E./Foil/Polyethylene	0.5 polyethylene terephthalate				
	0.75 P.E.	5.92	5.08	152	142
	0.35 Al. foil				
	2.0 polyethylene				
Polyethylene Terephthalate/ Foil/Polyethylene	0.5 polyethylene terephthalate				
	0.35 Al. foil	4.78	4.24	133	133
	2.0 polyethylene				

APPENDIX II - Page 5

<u>Laminates</u>	<u>Thickness (mil)</u>	<u>Energy to Punc- ture (gm-cm)</u>		<u>Maximum Force to Puncture (gm)</u>	
		<u>from Inside</u>	<u>from Outside</u>	<u>from Inside</u>	<u>from Outside</u>
Polyethylene Terephthalate/ Foil/Polyethylene	0.5 polyethylene terephthalate				
	0.5 Al. foil	5.21	4.90	140	143
	2.0 polyethylene				

APPENDIX III

Resistance to Flexure

<u>Films or Laminates</u>	<u>Thickness (mil)</u>	<u>Cycles of Flexure to Cause Pinhole Formation</u>
Polyethylene Terephthalate	0.5	> 10,000
	2.0	5,000
	4.0	700
Polytrifluoromono-chloro- ethylene	0.5	5,000
	0.75	3,000
	2.0	700
	5.0	300
Polyamide (Nylon-6)	0.5	> 10,000
	2.0	> 10,000
	5.0	> 10,000
H/D Polyethylene	3.0	5,000
Aluminum Foil	0.35	25
	0.5	5
Polyethylene Terephthalate/ Foil/Polyolefin blend	0.5 polyethylene terephthalate	
	0.35 Al. foil	> 10,000
	3.0 polyolefin blend	

APPENDIX III - Page 2

<u>Films or Laminates</u>	<u>Thickness (mil)</u>	<u>Cycles of Flexure to Cause Pinhole Formation</u>
Polyethylene Terephthalate/ Foil/H.D. Polyethylene	0.5 polyethylene terephthalate	
	.35 Al. foil	700
	3.0 H/D polyethylene	
Polyethylene Terephthalate/ Foil/L.D. Polyethylene	0.5 polyethylene terephthalate	
	0.35 Al. foil	500
	2.0 L/D polyethylene	
Nylon-6/P.E./Foil/L.D. Polyethylene	1.0 Nylon-6	
	#10 L/D polyethylene	
	0.35 Al. foil	1,500
	2.5 L/D polyethylene	
Nylon-6/Foil/Polyolefin	2.0 Nylon-6	
	0.35 Al. foil	> 10,000
	3.0 polyolefin blend	
Polyethylene Terephthalate/ Foil/Vinyl Chloride	2.0 polyethylene terephthalate	
	0.35 Al. foil	3,000
	3.0 vinyl chloride	

APPENDIX IV

Resistance to Abrasion

<u>Films or Laminates</u>	<u>Thickness (mil)</u>	<u>Revolution of Abrasion to Cause Pinhole Formation</u>
Polytrifluoromonochloro- ethylene*	0.5	20
	0.75	40
	1.0	140
Vinyl Chloride	2.0	10
Polyethylene Tereph- thalate	2.0	30
Polyethylene Tereph- thalate/Foil/Polyolefin Blend	0.5 polyethylene tereph- thalate	
	0.35 Al. foil	800
	3.0 polyolefin blend	
Polyethylene Tereph- thalate/Foil/H.D. Poly- ethylene	0.5 polyethylene tereph- thalate	
	0.35 Al. foil	1,000
	3.5 H/D polyethylene	
Polyethylene Tereph- thalate/Foil/L.D. Poly- ethylene	0.5 polyethylene tereph- thalate	
	0.35 Al. foil	50
	2.0 L/D polyethylene	

APPENDIX IV - Page 2

<u>Films or Laminates</u>	<u>Thickness (mil)</u>	<u>Revolution of Abrasion to Cause Pinhole Formation</u>
Nylon-6/PE/Foil/L.D. Polyethylene	1.0 Nylon-6	
	10 lbs L/D polyethylene	
	0.35 Al. foil	500
	2.5 L/D polyethylene	
Nylon-6/Foil/Polyolefin Blend	2.0 Nylon-6	
	0.35 Al. foil	> 1,500
	3.0 polyolefin blend	
Polyethylene Tereph- thalate/Foil/Vinyl Chloride	2.0 polyethylene tereph- thalate	
	0.35 Al. foil	900
	2.0 vinyl chloride	
Aluminum Foil	0.35	2

*NOTE: Strathmore drawing board, 100% cotton fibre #235-62 was used as the abrasive material for polytrifluoromono-chloroethylene films, while Emery polishing paper grit No. 410 was used as the abrasive material for the rest of the films or laminates tested.

APPENDIX V

Puncture Resistance at Different Speeds

Laminate Construction	Force, gm.		Energy, gm-cm			
	.2 in/min		1 in/min		.2 in/min	
	From "In-side"	From "Out-side"	From "In-side"	From "Out-side"	From "In-side"	From "Out-side"
1. 2 mil Polyethylene Terephthalate/.35 mil Al. Foil/2 mil Vinyl	508	333	544	401	26.64	12.60
2. .5 mil Polyethylene Terephthalate/.35 mil Al. Foil/3 mil H.D. Polyethylene	178	203	181	208	6.68	7.04
3. 2 mil Nylon-6/.35 mil Al. Foil/3 mil Polyolefin Blend	155	191	171	198	8.92	12.19
4. 5-mil Polyethylene Terephthalate/.35 mil Al. Foil/3 mil Polyolefin	157	169	161	183	5.44	5.51
5. .5 mil Polyethylene Terephthalate/.35 mil Al. Foil/2 mil L.D. Polyethylene	138	145	145	149	4.67	4.27
6. 1 mil Nylon-6/10 mil Polyethylene/.35 mil Al. Foil/2.5 mil L.D. Polyethylene	110	116	119	117	6.78	5.84
					7.59	7.32

APPENDIX VI

Flexure Resistance at Different Speeds

Laminate Construction	Number of Cycles	Number of Samples Showing Pinhole Formation (out of 10 samples)	
		132 Cycles/Min	180 Cycles/Min.
1. 2 mil Polyethylene Terephthalate/.35 mil Al. Foil/2 mil Vinyl	300	0	0
	500	0	1
	3,000	8	10
2. .5 mil Polyethylene Terephthalate/.35 mil Al. Foil/3 mil H.D. Polyethylene	300	1	0
	500	8	4
	700	9	9
3. .5 mil Polyethylene Terephthalate/.35 mil Al. Foil/2 mil L.D. Polyethylene	200	2	1
	300	9	7
	500	8	10

APPENDIX VII

Abrasion Resistance at Different Speeds

Laminate Construction	Number of Revolutions	Number of Samples Showing Pinhole Formation (out of 10 samples)		
		40 rpm	80 rpm	
1. 2-mil Nylon-6/.35-mil Al. Foil/3-mil Polyolefin	500 900 1,500	0 0 0	0 0 4	
2. .5-mil Polyethylene Terephthalate/.35-mil Al. Foil/3-mil H/D Polyethylene	200 500 1,000	2 6 9	3 8 10	
3. 2-mil Nylon-6/.35-mil Al. Foil/2-mil Vinyl Chloride	200 500 900	2 5 9	3 6 10	

APPENDIX VIII

Results of Laboratory Testing of Food-Filled Packages

The complete listing of packaging materials, packaging methods, and food products employed for the preliminary testing are as follows:

Packaging Materials (pouch size: 11.4 × 17.8 cm, heat sealed on four sides)

1. 0.5-mil polyethylene terephthalate/0.35-mil Al. foil/3.0-mil polyolefin.
2. 0.5-mil polyethylene terephthalate/0.35-mil Al. foil/3.0-mil H/D polyethylene.
3. 0.5-mil polyethylene terephthalate/0.35-mil Al. foil/2.0-mil L/D polyethylene.
4. 1.0-mil Nylon-6/ 10-lbs polyethylene/0.35-mil Al. foil/2.5-mil L/D polyethylene
5. 2.0-mil Nylon-6/0.35-mil Al. foil/3.0-mil polyolefin blend.
6. 2.0-mil polyethylene terephthalate/0.35-mil Al. foil/2.0-mil vinyl.

Packaging Methods

- A. Vacuum packaging (evacuated and sealed at 73.7 cm vacuum Hg).
- B. Air packaging (no air pressure differential inside and outside of the package).

Food Products Employed

1. Dehydrated apple slices (32 g. per package).
2. Freeze-dried beef patties (42 g. per package).
3. Cake flour (70 g. per package).

APPENDIX VIII - Continued

The code numbers used in the following table are:

The first number indicates the packaging material. The capital letter following the first number indicates the packaging method. The number following the capital letter indicates the food product employed. For example, 1-A-1 indicates packaging material #1 which is 5 mil polyethylene terephthalate/35 mil Al. foil/3 mil polyolefin blend, with vacuum packaging and filled with food product of dehydrated apple slices.

Code Number for the Combinations of Packaging Materials, Methods, and Food Products Employed	Number of Packages Failed due to Package Stressing by Vacuum Packaging (out of 84 packages)	Number of Packages Failed due to Abrasion Testing on the Belt Abraser (out of 84 packages)
1 - A - 1	5	20
1 - A - 2	1	18
1 - A - 3	0	16
1 - B - 1	0	12
1 - B - 2	0	12
1 - B - 3	0	0
2 - A - 1	4	10
2 - A - 2	1	8
2 - A - 3	0	0
2 - B - 1	0	2
2 - B - 2	0	2
2 - B - 3	0	0
3 - A - 1	40	24
3 - A - 2	21	24
3 - A - 3	0	24
3 - B - 1	0	24
3 - B - 2	0	24
3 - B - 3	0	24

APPENDIX VIII - Continued

Code Number for the Combination of Packaging Materials, Methods, and Food Products Employed	Number of Packages Failed due to Package Stressing by Vacuum Packaging (out of 84 packages)	Number of Packages Failed due to Abrasion Testing on the Belt Abraser (out of 84 packages)
4 - A - 1	12	14
4 - A - 2	2	17
4 - A - 3	0	3
4 - B - 1	0	8
4 - B - 2	0	8
4 - B - 3	0	8
5 - A - 1	2	6
5 - A - 2	1	6
5 - A - 3	0	0
5 - B - 1	0	2
5 - B - 2	0	2
5 - B - 3	0	0
6 - A - 1	0	17
6 - A - 2	0	16
6 - A - 3	0	0
6 - B - 1	0	6
6 - B - 2	0	6
6 - B - 3	0	4