DESIGN AND DEVELOPMENT OF A TEST METHOD FOR LAMINATE TUBES SEALABILITY

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ABSTRACT

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FOR LAMINATE TUBES SEALABILITY

by Gregory J. Reed

This study involves the investigation of differences in the sealability of laminate tubes and a report of a reproducible test method that classifies laminate tubes by their seal strength.

The testing of heat seals is one of the most difficult tasks in packaging. For example, although standard laboratory procedures and equipment evaluate seal strength, such values may have little bearing on environmental performance of the tube later on.

One of the major development problems in Packaging is the sealing of laminate tubes. The method that is outlined in the study explains how laminate tube seals can be classified to determine their sealability before they are used in production. A test method like this is quite

valuable because it reduces stock wastage, expenditures on material, and satisfies the consumers' needs during the use of the product.

DESIGN AND DEVELOPMENT OF A TEST METHOD FOR LAMINATE TUBES SEALABILITY

by

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I wish to express my sincere thanks to Lyle Ritze of Proctor & Gamble who gave me advice during my preparation of the project.

I also wish to give thanks to Bill and Maureen, my guardians, who have helped me a great deal during my undergraduate studies.

And to my Mother, who has provided for me during the years.

Thanks Vee

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INTRODUCTION

SUMMARY

The objective of this report is to develop a relatively simple and reproducible test method that can be used as a quality control test to classify tubes by their sealability. The investigation was not directed toward formulating a test method for all laminate tubes, but only those with a certain thickness.

In order for such a test to be developed, an investigation of sealing differences had to be established. This was done by sealing tubes and evaluating their bursting strength with a pressure apparatus (Fig. 1). There were three groups of tubes that were studied for differences; see Table 1.

It was found that there were differences in bursting strength of each group, although they were made the same. Therefore, there was a need to develop a method to discriminate between good and bad tubes.

TABLE 1

LAMINATE TUBE VARIABLES

GROUP	AGE	QUALITY OF SEALS
I	6 Mo.	Good
II	1 Yr.	Bad
III	2 Mo.	Good

BACKGROUND

A relatively new type of tube that has been developed, has a body comprised of a laminate structure of polyethelene foil and paper. This construction should provide a whole new dimension in collapsible tube packaging. Products not previously packagable in plastic tubes can now be handled (1).

The tube is composed of laminated sheets and a layer of metal foil. In the manufacturing method, a base layer or substrate is coated in succession with an adhesion layer, a metal foil layer, and another coating of adhesion layer.

The method provides a laminated structure which resists delamination.[a]

One of the major development problems of the laminate tube, is sealing the tube effectively after it has been filled with a product. Three different types of tubes have been studied, and it has been found that laminate tubes do cause a sealing problem, if there are differences in their lamination.

GROUP I	GROUP II	GROUP III
6 Months Old	1 Year	2 Months Old

The three tube groups above were studied with an ultrasonic sealer and an induction sealer. Ultrasonic,

> relies on the hammering of two films (or foil) together at high frequency, causing them to weld without generating a temperature that would de-orient the material that is being sealed. (1)

Induction sealing depends greatly on the heat that is momentarily applied to the area that is being sealed.

It was discovered that different laminate tubes that are set up to be sealed, with any present sealing method,

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[a]Refer to Appendix for complete structure of the laminate tube.

will vary in seal strength. For example, if the ultrasonic sealer is set to give good seals on Group I or Group III, Group II would give very poor seals. Aging was thought to be the reason for differences in seal strength, until a fourth group was analyzed that was two years old. The fourth group's seal strength was equal to that of Group I or Group III, both of which had good seals.

A good seal was defined as a tube capable of holding 25 pounds of pressure per square inch, or more, for 15 seconds.

Hypothetically, the construction of the tube was believed to be the cause for sealing differences. Differences may occur if the laminated layers have not adhered to each other properly. This could result if temperature settings were changed in the laminating cycle. There were not any satisfactory explanations for this phenomenon. Since this condition existed, there was a need to develop a test method that could identify laminate tubes and determine if they had good sealing properties before they were put into production.

Reasons for the Test

Tube variations can occur in laminate tubes.

1. A test was developed to insure that the consumer has a reliable tube during the life of the product.

2. A quality control test increases the probability of protecting the product.

3. A sealing test identifies bad tubes from the sample before the tubes are used in production. A sealing test will decrease the amount of product being wasted.

4. The test greatly reduces the cost on material.

TEST CRITERIA

The test criteria were defined as:

1. The test must be reproducible, so that the manufacturer can install the test to detect bad tubes for distribution.

2. The test has to be simple to set up and operate.

Test Equipment

SENTINEL SEALER

The test equipment that was used to determine the sealability of the laminate specimens was the Sentinel sealer and Instron, Figs. 2 & 3. The Sentinel sealer was used because it has the ability to duplicate seals that were made by the ultrasonic and induction methods.

Three settings are required to operate the Sentinel sealer effectively; they are:

Right Heat

Temperature settings are made by turning pointer of thermostat to temperature readings on dial of etched plate (200 to 500 degrees) of thermostat panel. These are approximate temperatures. (If a more accurate temperature reading is desired, a hole is drilled in one end of heater jaw into which a laboratory Weston thermometer may be inserted; turn thermostat pointer to increase or decrease heat to correspond with desired thermometer reading.)

Right Pressure

Pressure setting is made on control cabinet panel, by turning regulator knob clockwise to increase and counter-clockwise to decrease pressure. Pressure gauge markings are especially calibrated to show pounds per square inch pressure of the sealing jaw.

Pressure is important to a good heat seal. Unless otherwise specified, a setting of a minimum of 60 pounds per square inch is recommended when using heavy duty barrier materials.

Right Time

Dwell-time of the sealing jaw is controlled by sweep hand of timer (right center of control cabinet panel). Timer dial is divided into quarter seconds, up to 15 seconds. Lever pointer is set to the desired time and can be readily changed at any time.

INSTRON

To determine whether the seal was a good or bad one, the Instron was used to evaluate the seals made by the Sentinel sealer. The Instron tested the tensile strength of the sealed area by separating the seal.

The Instron is a very precise evaluator of tensile strength. The machine is electrically power driven. It is equipped with a set of jaws to hold the sealed specimen. It also has a recorder to record the results.

The testing procedures for operating the Instron are not difficult.

1. The first step of running the Instron is to zero and balance the control, for an accurate recording.

2. The machine has to be calibrated with calibrating weights, extended from the load cell.

3. The selection of the crosshead speed must be determined.

For example, assume that a sample with a 2 inch gage length is to be tested at a rate of 25% per minute, and that the expected ultimate extension will be 50%, on 1.0 inch total. On this basis, the proper crosshead speed is 1/2 inch per minute, and approximately 1 minute will be required for rupture.

TEST METHOD

SCOPE

This method covers a procedure for determining the sealability of laminate tubes. Two specimens shall be sealed on a Sentinel sealer. An Instron is used to measure the force that it takes to separate the seal. The settings for the sealer are for certain laminate structures, depending on thickness of the material. The results are reported in units of force (lbs./inch).

APPARATUS

The Sentinel sealer with the following characteristics was used (Fig. 2).

1. jaws, upper and lower that apply pressure and heat for sealing specimens (Each jaw is to have a 1 inch wide strip of Teflon tape to prevent specimen from sticking.),

2. two temperature gauges which control the heat for the upper and lower jaw,

3. pressure regulator, that controls the jaw pressure applied for sealing, and a

4. timer, that regulates the jaw dwell time on the material.

TEST SPECIMENS

Test specimen can be any tube size made from laminate stock. (See Appendix A)

1. The specimen can be cut from a roll stock by 2-3/4" x 3-3/4".

2. The specimens can be taken from a tube and be prepared to 2-3/4" x 3-3/4".

METHOD OF TEST

A. Sealing Procedures

   1. Adjust both temperature gauges to 325°F.

2. Set main timer for 3 seconds.

3. Turn pressure regulator to 28 lbs.

4. After machine is warmed, the specimen's inner surfaces are placed evenly between the jaws of the sealing machine.

5. The specimen should be sealed 1/2" to 3/4" from its 2-3/4" edge.

6. Press pedal, to begin sealing operation.

B. Testing Seals

1. The sealed specimens shall be cut 1" wide with a sample cutter.

2. The Instron machine must be balanced and calibrated for testing. Turn on recorder for calibrations.

3. The unsealed strips of sealed specimen are separated and placed in the grips of the Instron as shown in Figure 3.

4. The crosshead speed of the Instron can be from .2 in./min. to 2 in./min.

5. The sealed specimen is ready to be evaluated for strength.

REPORT

1. Results will be measured by the height of peak, which is the force required to separate the sealed specimens.

2. The units are recorded in pounds per inch.

3. Four sealed samples shall be sufficient to determine the sealability of the laminate stock.

4. The averages of the four samples are to be computed and measured with the standards below (refer to discussion of test method for explanation of standards):

STANDARDS

Unacceptable	Acceptable
0-11(lbs./in.)	$\geq$ 12(lbs./in.)

DISCUSSION OF TEST METHOD

A series of tests were conducted with the Sentinel sealer and an air pressure apparatus (Fig. 1) before the Instron was substituted for a precise test method. When using the pressure apparatus after sealing the tubes with the Sentinel, it was found that it took sixteen tubes for an evaluation to determine the seal strength of each group studied.

After sealing as many as 100 samples and varying the temperatures, the results had shown that Group I and Group III averaged 25 pounds of pressure or more before the tubes burst. Group II did not average more than 25 psi., as shown in Table II. After testing 100 tubes and compiling the results, a good tube was defined as one that was capable of holding 25 psi. This was arbitrarily selected from the test results.

TABLE II

AVERAGE PRESSURE STRENGTH SEALED AT 28 PSI AND 3 SECONDS ON SENTINEL SEALER

Sealing Condition of	Gr. I	Gr. II	Gr. III
275	8 psi	3 psi	28 psi
300	28 psi	14 psi	46 psi
325	45 psi	22 psi	60 psi

In order to determine the seal strength with the air pressure device, it was found that the test method was not accurate, because large amounts of samples needed to be tested. A better test was sought to do the job of the pressure apparatus. The Instron was substituted for the pressure evaluator.

The Instron tested 50 samples of each group studied to check if it was better than the pressure apparatus. This was done by correlating the tensile strength of the samples that were evaluated by the Instron, to the pressure strength of the samples tested by the pressure apparatus. For instance, Group III tube average 24 lbs./in. of force. These tubes would hold 60 lbs. of pressure. All samples were sealed at the same condition. The results are indicated in Table III which showed that the Instron was a consistent instrument for such a test method in conjunction with the Sentinel Heat Sealer. Most important, when testing the samples for seal strength, only four samples were needed to determine which groups had the best seal strength. Another significance of the test method was that it determined which tubes had good sealing properties. The importance of the sealing properties, was that they gave an idea of which tube was capable of giving a good seal before production was set up.

After the Instron proved to be a reliable instrument for testing seal strength, the Sentinel sealer was checked for accuracy in producing good seals if any of the controls were varied during testing. Each of the settings on the Sentinel sealer were varied ± 10% from its original setting while the other two remained constant during sealing. The results are tabulated in Tables V-VII.

Many settings were checked for effects on sealing. The sealing device proved that it was capable of producing good seals if the controls did vary ± 10% from any of its settings. The greatest differences of seal strength were recorded at 325°F, 28 lbs. pressure, and 3 seconds. This setting was chosen as a setting to seal and evaluate all samples for future evaluation. However, different tube sizes required different settings. It was found that by cutting the tubes into strips of 2-3/4" x 3-3/4" only one setting was required for the sealer. The results were the same regardless of the tube size. After compiling data on 50 samples, it was established that Group II seal strengths ranged from 0-8 (lbs./inch), Group I ranged from 12-15 (lbs./inch), and Group III ranged 15-25 (lbs./inch). A cut-off point was selected to discriminate good tubes from bad ones. Refer to Table III for the cut-off point. Figs. 4 & 5 also indicate these cut-off points.

TABLE III

STANDARDS FOR TUBES SEAL STRENTH

Unacceptable (lbs./in.)	Acceptable (lbs./in.)
0-11	≥ 12

TABLE IV

CORRELATING SEAL STRENGTH AND PRESSURE STRENGTH

Sealing Condition °F	Seal Strength (lbs./in.)			Pressure Strength (Psi)		
	Gr. 1	Gr. II	Gr. III	Gr. I	Gr. II	Gr. III
275	2	1	6	8	3	28
300	6	4	13	28	14	46
325	4	7	24	45	22	60

*Press. 28 psi; Time 3 secs.

TABLE V

TEMPERATURE VARIANCES RESULTS
Effects of deviating sealing settings from its test setting.

Temp. F	Gr. I	Gr. II	Gr. III
315	13	8	20
325	14	8	24
335	16	9	24

*Force (lbs./in.); Time 3 secs.; Press. 28 psi.

TABLE VI

TIME VARIANCE RESULTS
Effects of deviating sealing setting from its test setting.

Time (Secs.)	Gr. I	Gr. II	Gr. III
2.5	7	4	18
3.0	14	8	24
3.5	22	8	25

*Force (lbs./in.); Temp. 325°F, Press. 28 psi.

TABLE VII

PRESSURE VARIANCE RESULTS
Effects of deviating sealing setting from its test setting.

Press. (psi)	Gr. I	Gr. II	Gr. III
24	11	6	20
28	14	8	24
32	15	8	26

*Force (lbs./in.); Time 3 secs.; Temp. 325°F.

CONCLUSIONS

1. The use of the Sentinel Sealer and Instron proved to be a very effective method of determining the sealability of laminate stock.

2. The two test apparatus performances were consistent and reliable when using them as evaluators of laminate tubes.

   The controls of the sealer did not change during testing.

   If fluctuation did occur, it would not change the results unless the control varied more than 10%.

3. The test method is simple and reproducible, which was the objective of the assignment.

PRESSURE APPARATUS

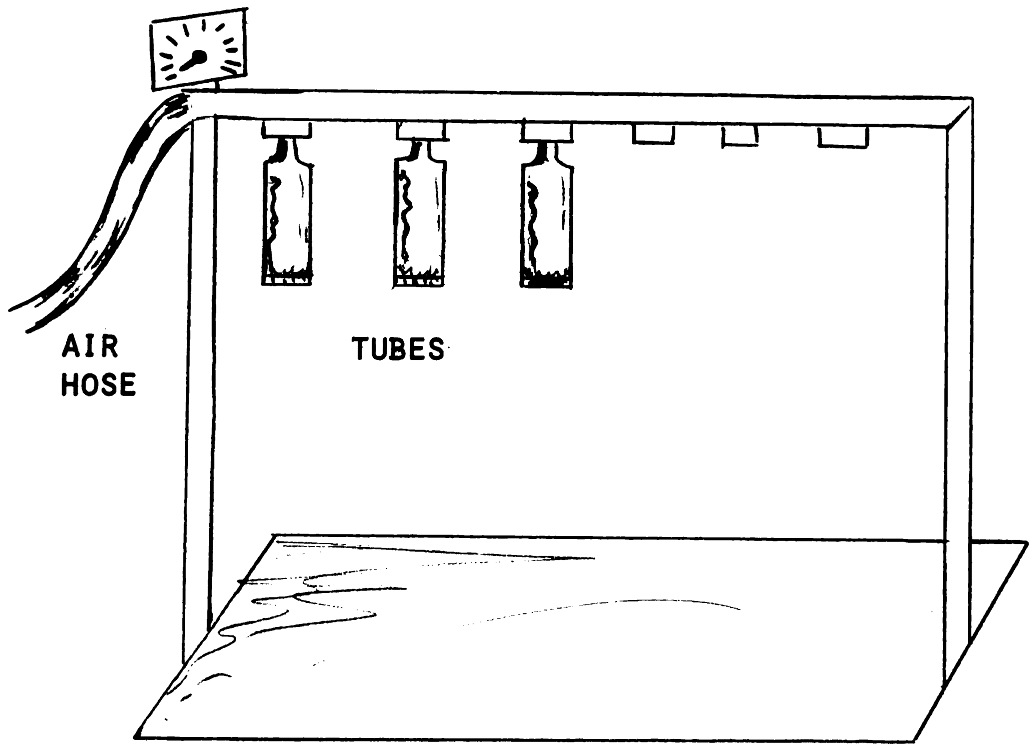


FIG. 1

SENTINEL SEALER

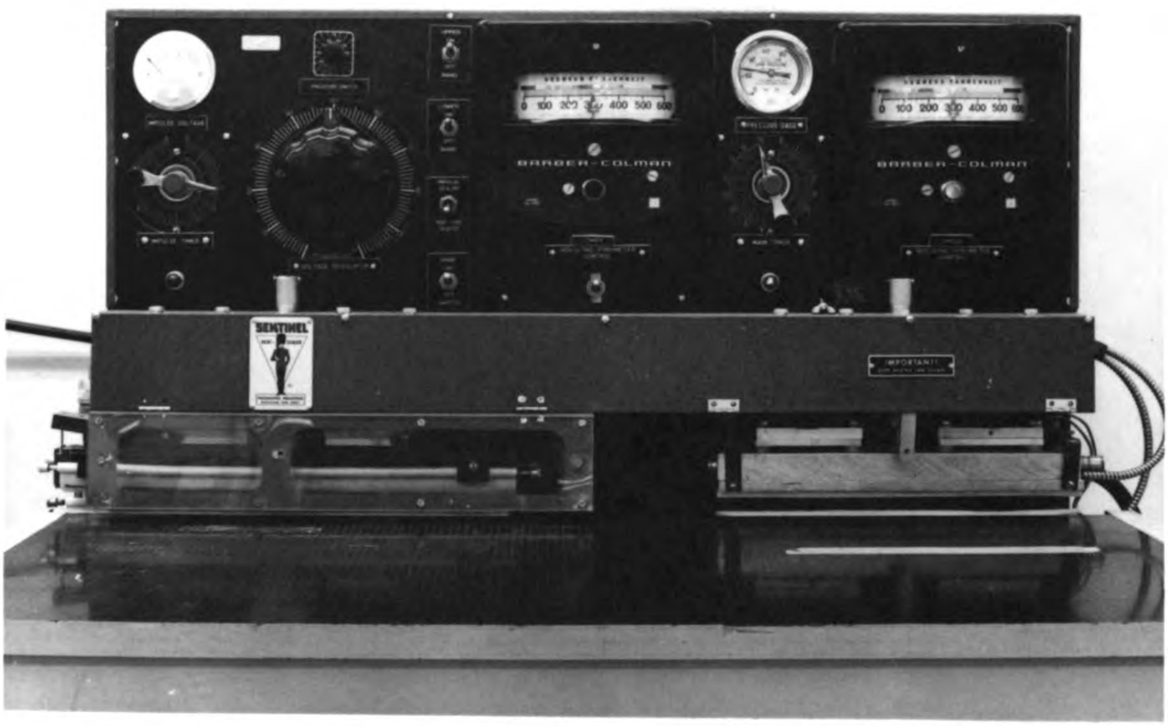


FIG. 2

INSTRON

FIG. 3

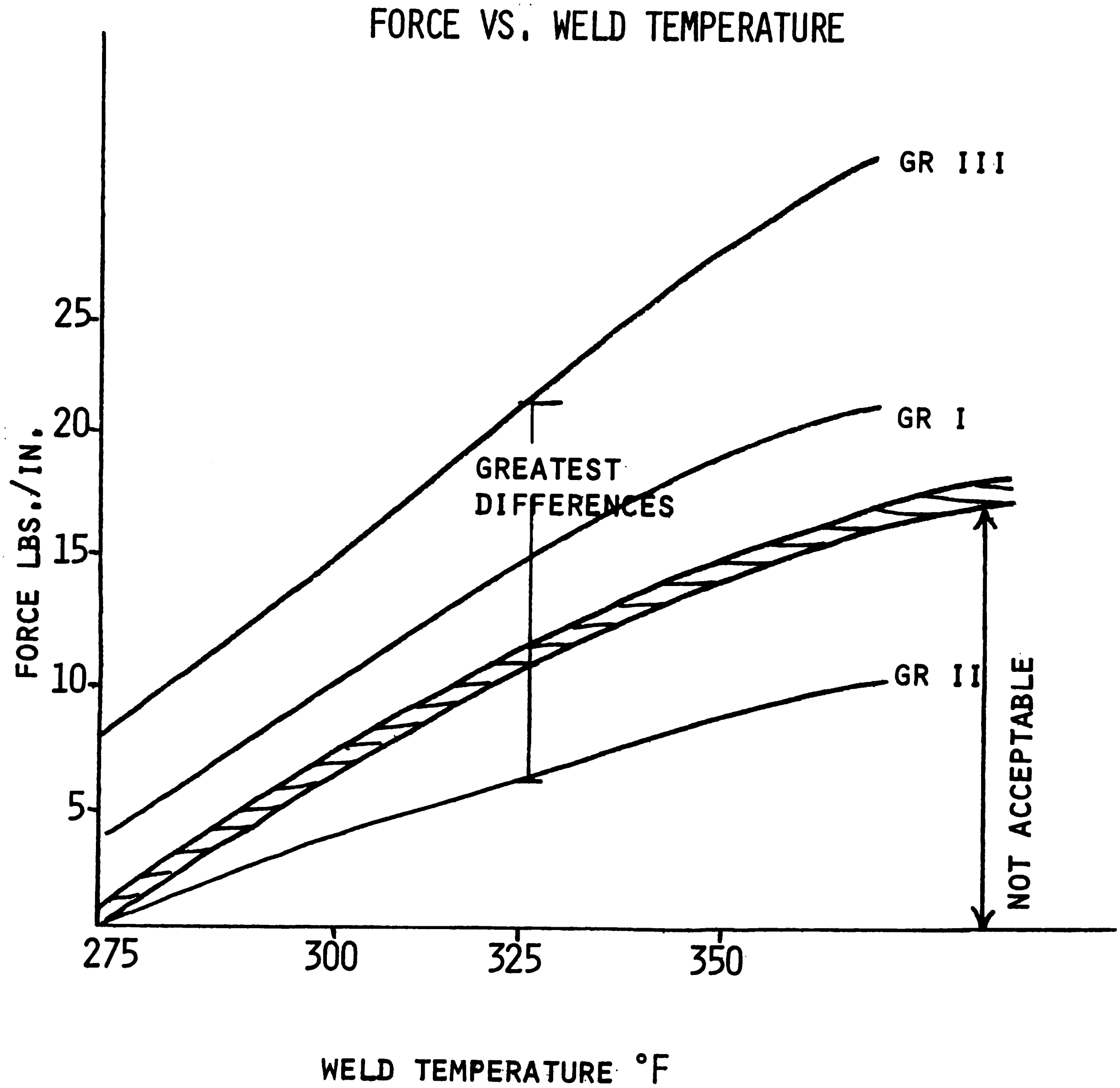

FORCE VS. WELD TEMPERATURE
GR III
GR I
GR II
GREATEST DIFFERENCES
NOT ACCEPTABLE
FORCE LBS./IN.
25
20
15
10
5
275
300
325
350
WELD TEMPERATURE °F

FIG. 4

VARIATION IN LAMINATE TUBE SEAL STRENGTH

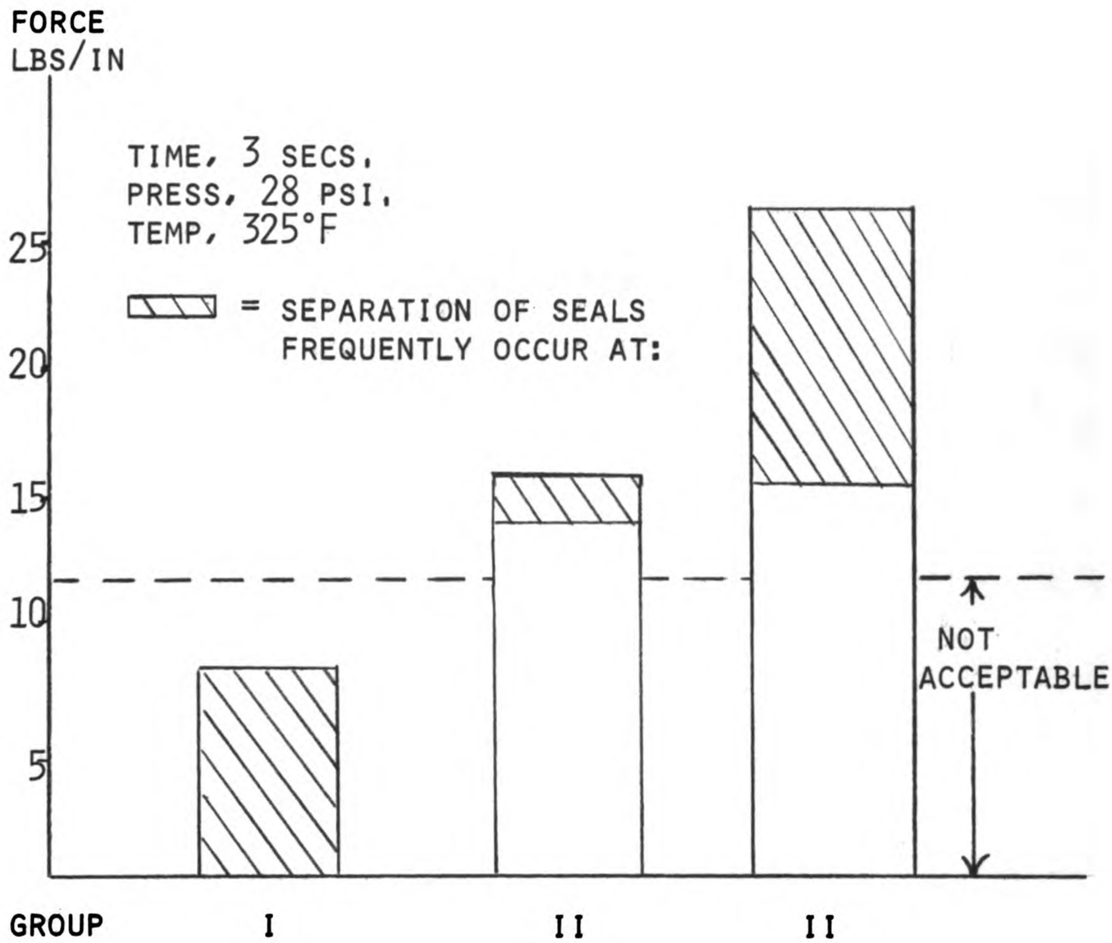


FIG. 5

APPENDIX

APPENDIX A

METHOD OF LAMINATING SHEET MATERIAL BY COATING METAL FOIL WITH A COPODYMER OF ETHYLENE AND AN ETHYLENICALLY YNSATURATED CARBOXYLIC ACID

ABSTRACT OF THE DISCLOSURE

A method for manufacturing laminated sheets having a layer of metal foil. In the method a base layer or substrate is coated in succession with an adhesion layer, a metal foil layer and another coating of the adhesion layer. The method provides a laminated structure including a metal foil layer which resists delamination.

The present invention relates to laminated sheet material and more particularly to a method for manufacturing laminated sheets which are used for making the walls of collapsible tubes.

Plastic collapsible tubes have been widely known and used for packaging cosmetics and personal preparations such as shampoo. Plastics like polyethylene are relatively inert and can resist chemical attack from many products which react chemically with metal tubes. Heretofore plastic tubes have been limited in their application because they provide a poor barrier to moisture, oxygen, and volatile ingredients like essential oils or perfumes.

In order to extend the use of collapsible plastic tubes to products like mustard having volatile ingredients, plastic tubes may include a barrier layer of metal foil laminated to the plastic. The combination of plastic and metal foil layers yields an improved tube prevents loss of essential ingredients and the plastic in the wall of the improved tube and protects the metal foil barrier layer against chemical attack.

An improved tube comprising layers of plastic and metal foil is disclosed in the Brandt and Kaercher U.S. Patent No. 3,260,410 granted July 12, 1966, owned by the assignee of the present invention. In the Brandt et al. application the preferred plastic laminate is a co-polymer of an olefin such as polyethylene and an ethylenically unsaturated carboxylic acid such as acrylic acid. This copolymer adheres well to metal foil and provides a tube wall which resists delamination.

While the preferred co-polymer in said application exhibits good adhesion to metal foil, difficulty has been encountered in obtaining good adhesion between the co-polymer and the metal foil using known methods to produce commercial quantities of the sheet material.

The present invention provides a method for manufacturing laminated tube walls whereby an effective and durable bond is obtained between metal and plastic layers of the laminated wall of a collapsible tube.

Another object of the present invention is to provide a method for continuous coating of a metal foil barrier layer in a composite sheet material.

These and other objects of the present invention will become apparent upon an understanding of the preferred embodiment, selected to describe the present invention. The preferred embodiment of the invention has been chosen to illustrate the principles of the present invention and may be varied without departing from the spirit and scope of the present invention.

FIGURE 1 is a side elevation view of a collapsible tube having a laminated wall which is prepared according to the present invention.

FIGURE 2 is an enlarged section view taken along line 2--2 of FIGURE 1 to illustrate the individual layers which may comprise the tube wall according to the present invention.

FIGURE 3 is a schematic view illustrating the apparatus for laminating sheet material for tube balls according to the present invention.

FIGURE 4 is a section view of a laminated sheet corresponding to the various stages of lamination performed upon the sheet in FIGURE 3.

The preferred embodiment of the present invention is described with particular reference to collapsible tubes. It will be understood that the sheet material according to the present invention may be used for other purposes than collapsible tubes.

Referring to FIGURE 1 of the drawings, a collapsible tube 1 including a tube wall 2 which is heat sealed at its lower end 3. A headpice 4 and a cap 5 enclose the upper end 6 of the tube. The tube wall 2 comprises a laminated sheet 7 which is rolled into tube form and joined in a side seam (not shown) at the edges of the sheet.

As best shown in FIGURE 2, the tube wall includes a plurality of layers 8-15. Outer layers 8, 9, 10, 11, 12, 13, 14, and 15 may be referred to as barrier layers.

The tube wall 2 may be manufactured in any suitable manner to achieve the decorative layers 8-11 are laminated first and then are joined to barrier layers 12-14. After joinder of the decorative and barrier layers, a plastic coating 15 is applied to the inner surface of the tube wall.

In preparing the decorative layers 8-11, a flat sheet of glassine paper 11 is coated with a layer of polyethylene 10 which layer is pigmented for opacity and to give a solid color background. A layer 9 of printed data is applied against the background layer 10. Next an outer layer 8 of clear polyethylene protects the layer 9 of printed data and adds to the attractiveness of the tube wall 2.

The glassine paper 11 lends dimensional stability to the decorative layers 8-11 which include polyethylene. Thus the decorative layers may be rerolled for subsequent lamination with the barrier layers 12-15. The barrier layers 12-15 prevent migration of the essential oils, oxygen, moisture and the like through the tube wall.

As shown in FIGURE 3, the decorative layers 8 through 11 are mounted on an unwind roll 20 for processing in the form of a sheet 21 through a laminating apparatus, indicated generally at 22. During the unwinding operation, the layer of glassine paper 11 appears on the upper surface of the sheet 21 for joinder with the barrier layers 12-14. The clear polyethylene layer 8 is on the underside of the sheet 21 and is not coated. A suitable roller 23 may be used for supporting the sheet 21.

As shown in FIGURE 3, the sheet 21 moves toward a priming station 24 as it enters the laminating apparatus 22. At the priming station 24 the upper surface, i.e. glassine paper surface 11, of the sheet is primed, that is, prepared to be laminated to another layer 12. Preferably, a preheater 25 estends across the sheet 21 and heats sheet surface 11. Alternatively, polyethylene imine may be used to prime the sheet surface.

After the priming operation the sheet 21 moves into the nip of a pressure roll 26 and a chill roll 27 which define a first coating station shown generally at 28. At this station, a layer 13 of metal foil is joined to the sheet 21 by means of a suitable plastic layer 12. Preferably the layer 13 is aluminum foil and the layer 12 is a co-polymer of polyethylene and acrylic acid which co-polymer exhibits good adhesion to aluminum foil. The co-polymer of polyethylene and acrylic acid may be applied in any suitable manner as for example by extrusion under suitable heat and pressure through an extrusion die 29. The co-polymer is extruded in a thin layer (about 1.3 mils thin) which extends across the width of the metal foil layer 13 and the sheet 21.

Applicants have determined that both surfaces of the metal foil layer 13 should be coated on both surfaces with a suitable plastic without removing the sheet 21 from the laminating apparatus 22. Applicants believe that contamination of the uncoated metal foil surface 30 occurs if the sheet 21 is put in roll form between coating operations. If rolled, the uncoated metal foil surface 30 contacts a coating on the other side of the foil which is believed to be the source of contamination. The coating causing contamination may be layer 8, layer 9, or layer 10 depending on sequence of adding plastic layers. Applicants believe that such contamination interferes with proper bonding between the co-polymer and the metal foil. By avoiding this contamination, a durable bond between co-polymer and metal foil is obtained.

Accordingly, the sheet 21 is passed over a suitable support roller 31 to a second priming station 32 where the sheet 21, metal foil surface 30 uppermost, is pre-heated. Wrinkling of the metal foil 13 is prevented by adjusting the preheater temperature or the speed of the sheet 21.

After preheating, the sheet 21 moves to a second coating station 34 defined by a pressure roller 35 and a chill roller 36. Here a layer 14 of suitable plastic, i.e., the co-polymer of ethylene and acrylic acid is adhered to the upper surface 30 of metal foil layer 13. Again the coating operating is accomplished by extruding the co-polymer through an extrusion die 37 as at the first coating station 28.

After passing through the second coating station 34, the sheet 21 is removed by a rewind roll 38.

FIGURE 4 illustrates in section the layers of the sheet as they progress through the extrusion apparatus of FIGURE 3.

The innermost layer 15 (FIGURE 2) is a coating of polyethylene which gives better adhesion to the headpiece 4 than does ethylene-acrylic acid co-polymer. The innermost layer 15 may be added in a subsequent coating operation, as by extrusion.

While the present invention has been described in particular reference to a co-polymer of an olefin and an ethylenically unsaturated carboxylic acid, it is to be understood that any suitable plastic may be employed in carrying out the present invention.

It will be seen that applicants have provided a method for the continuous coating of a laminated sheet which gives collapsible tubes an extended shelf life without delamination of the individual layers of the individual layers of the tube wall. A collapsible plastic tube which does not delaminate has the feel of a single wall tube similar to that experienced with collapsible metal tubes.

Having thus described our invention, we claim:

1) A method of making a laminated web including a base layer the surface of which has been primed by heat treatment or by using polyethylene imine comprising the continuous steps of moving the primed base layer to a first coating station, coating the base layer with an intermediate layer of a copolymer of an ethylene and an ethylenically unsaturated carboxylic acid, coating the intermediate layer with a sheet of metal foil, moving the web to a priming station, priming the surface of the metal foil, moving the web to second coating station, and coating the metal foil with a layer of a copolymer of an ethylene and an ethylenically unsaturated carboxylic acid.

2) The method of claim 1 wherein metal foil layer is aluminum.

April 7, 1970 D. J. HAAS ET AL 3,505,143

METHOD OF LAMINATING SHEET MATERIAL BY COATING METAL FOIL WITH A COPOLYMER OF ETHYLENE AND A ETHYLENICALLY UNSATURATED CARBOXYLIC ACID

Filed March 10, 1968

Fig. 1

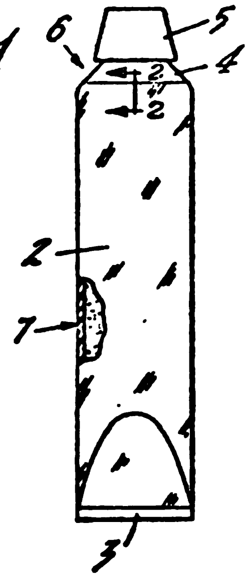


Fig. 2

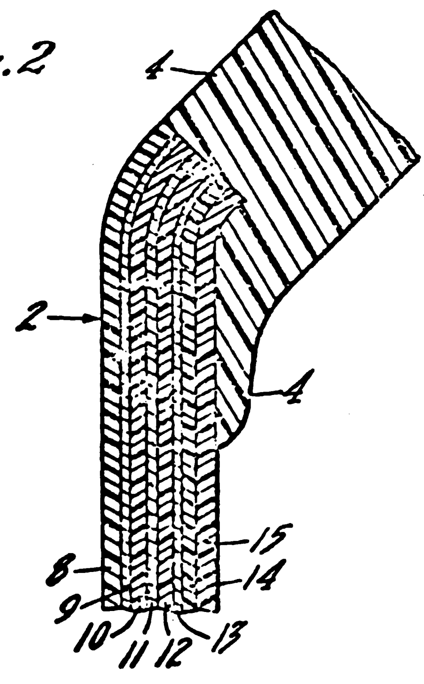
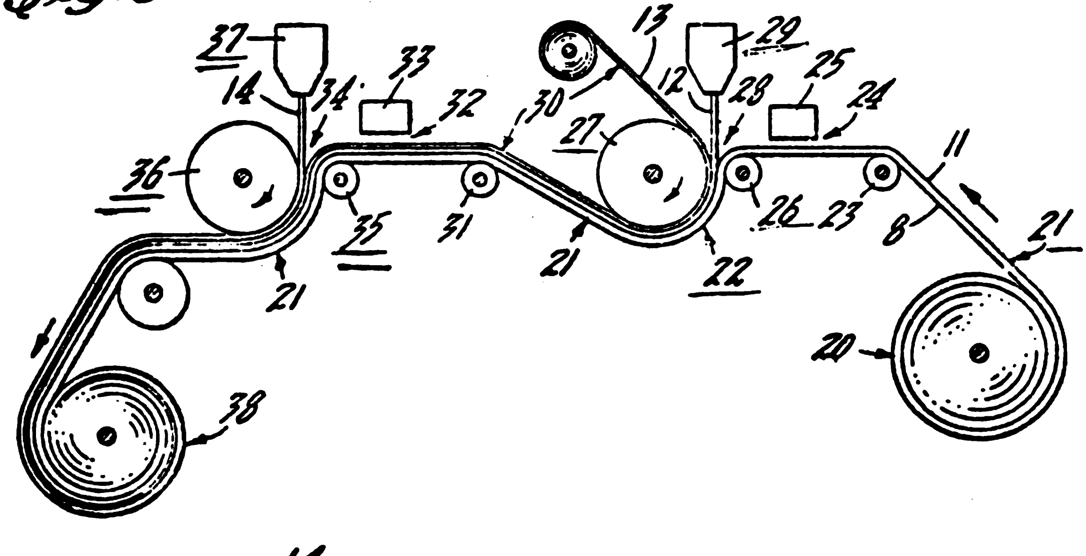


Fig. 3



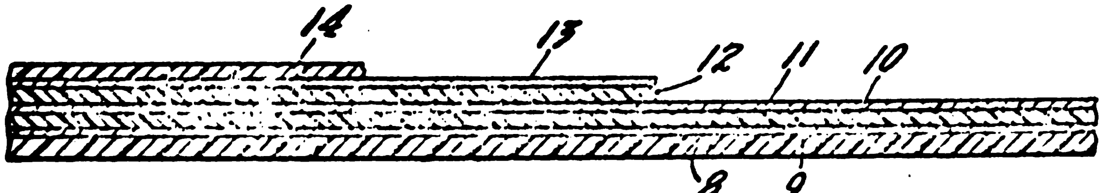


Fig. 4

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KURT FRITZ ROESCH
BY Patrick J. Welch
ATTORNEY

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