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DELAMINATION IN SCREW CAP CLOSURES

Thesis for the Degree of M. S.  
MICHIGAN STATE UNIVERSITY  
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1957

DELAMINATION IN SCREW CAP CLOSURES

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A THESIS

Submitted to the School of Graduate Studies of Michigan  
State University of Agriculture and Applied Science  
in partial fulfilment of the requirements  
for the degree of  
MASTER OF SCIENCE

Department of Forest Products

1957



## THESIS ABSTRACT

Delamination in bottle cap closures refers specifically to a part, or all, of the liner in the cap separating, splitting or falling out of the cap. The problem was to discover the factors that bring about the failure of the liner and of the adhesive individually or as a unit in the complete closure.

It was therefore decided to tackle the problem in these three parts: adhesives, liners and complete closures, and to determine the factors that in each case will bring about delamination.

In the case of adhesives, this necessitated a study of the physical and chemical factors that influence adhesion, for an inadequate bond will aid delamination. In addition, a brief review of existing commercial adhesives and their properties was carried out.

The study of liners involved not only the complete liners, but the various types of facings and backings that are commercially available. Furthermore, tests were carried out on liner board to determine the effects of various liquids on the laminate strength of the board.

Rigid plastic and metal screw cap closures were examined and the factors that bring about delamination of their liners carefully analyzed. These include the pH factor, hydrodynamic action, excessive or insufficient torque and liquid penetration. A series of tests were carried out to examine the effects of liquid penetration on the laminate strength of the closure assembly and further tests made to determine the effects on delamination of wax on liner facings.



It was concluded that an incorrect choice of the adhesive to be used in the closure would result in an inadequate bond which could result in delamination. The adhesive film must be as strong as the other materials used in the closure and capable of resisting any chemical or physical forces to which it might be subjected.

Similarly, an incorrect choice of liner materials will result in delamination, for in addition to the liner being capable of resisting chemical and physical forces applied to it, it must be able to resist liquid penetration which will otherwise seriously lower its laminate strength.

Finally, the combination of adhesive and liner in the complete screw cap closure must be capable of resisting the torque forces applied to it, the hydrodynamic forces to which it might be subjected, pumping and sweating, liquid penetration and chemical attack.

It is recommended that further research be carried out on the wider application of direct plastic laminations as liners, for these will eliminate many of the aforementioned factors which can cause delamination.

6/24/57  
215-570

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## ACKNOWLEDGEMENTS

The author wishes to express his sincere thanks to Mr. J. W. Goff and to Dr. H. J. Raphael of the Department of Forest Products for their guidance and helpful suggestions, particularly during the laboratory tests and to Dr. J. Toulouse of the Owens-Illinois Company for his very considerable contribution of technical information. Thanks are also due to Mr. R. K. Lancaster, Mr. J. E. Shottafer and Mr. R. F. Frisosky for their help at various stages of the project and finally to Miss Marilyn Wilt who typed this paper.



## INTRODUCTION

Delamination refers specifically to the separating or splitting action usually caused by lack of adequate or sufficient adhesion in laminated or plied goods. In the case of bottle cap closures, the delamination may be caused by failure of the adhesive adhering the liner to the inside of the bottle cap or by the separation of the liner material itself.

This delamination is most undesirable from both the manufacturer's and the consumer's standpoint. After delamination occurs, the closure no longer acts as an efficient seal and the enclosed product may be contaminated as a result, the product may leak out, or in the case of the packaging of highly volatile products, excessive evaporation will occur. This defeats the whole purpose of an efficient reusable closure and is a source of considerable annoyance to both consumer and manufacturer, for the latter may lose a considerable amount of sales as a result of the former's adverse reactions.

The delamination may occur on the initial opening of the closure or after several openings and closings. If the former occurs, it is probably due to bad container and closure design and engineering or incompatibility of the adhesive and liner with the contents of the container.

The second type of delamination is due to a variety of causes, the most commonly encountered one being the splitting of the pulp backing material after repeated uses of the product. This condition results from the product being used from the container and some of it being left on the glass threads. This then gets on the inside of the cap and the edges of the liner material, eventually soaking into the backing and aiding delamination.

It is the purpose of this paper to analyze the causes of the second type of delamination; to devise means of testing liners and adhesives for delamination; to draw conclusions on these tests; and finally, to suggest means of overcoming these problems on the basis of observations made during the tests.

## CHAPTER I

### ADHESIVES

#### THEORY OF ADHESION

##### Introduction

Many factors influence adhesive action and these may be divided according to their physical and chemical properties. In addition, the adhesive action will be closely related to the surface chemistry and physical properties of the adherends. Today, tackiness in adhesives is considered of little importance as it has no correlation with the strength of the final assembly. In fact, adhesives that retain their tackiness may show an increase in the rate of creep under stress compared with those having little or no tackiness.

In the general overall picture, the selection and adaptation of an adhesive depends upon the chemical and physical properties of the adhesive itself, the adherends and the product being packaged. Although there are some very versatile adhesives capable of application to most surfaces, no one at present knows of an adhesive with outstanding properties for all bonding problems.

##### Physical factors influencing adhesion

1. Surface tension - The suitability of a liquid adhesive for a given surface is related in part to the ability

of that liquid to wet the surface. The wettability in turn depends on the viscosity of the liquid adhesive and on its surface tension. The tendency of a liquid to spread or wet the surface is measured by the wetting angle, which is the angle of contact formed between the liquid and the surface. As the adhesion of the liquid for the surface increases, the angle of contact diminishes until a point is reached where the work of adhesion of the liquid to solid equals the cohesion of the liquid and the angle of contact vanishes. The surface tension in a liquid adhesive can be lessened by the use of heat in curing the adhesive and causing it to flow more freely before setting. The work of adhesion (work required to separate the two) between the solid and liquid has been shown to depend upon the surface tension of the liquid and the lowering of the free surface energy of the solid through equilibrium with the saturated vapour of the liquid.<sup>1</sup>

Solid surfaces are often contaminated with foreign matter, usually in the form of a thin film of grease, an adsorbed film of gas or an oxide film. Grease may be removed by washing with suitable chemicals but the adsorbed gas is most difficult to remove. In the practical application of adhesives, the presence of the adsorbed gas film can be assumed, but if it can be removed the adhesion between the

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<sup>1</sup>Harkins, W.D., and Livingston, H.K., Journal Chem. Phys. 1942.

solid and the liquid would be improved. Generally speaking, therefore, the cleaner the surface of the solid, the better the adhesion.

2. Porosity of surface and relative smoothness - The performance of adhesives is greatly influenced by the relative porosity of the surface to which they are applied. Wood, paper and leather are porous in that numerous capillaries are present in them and these will conduct away the more highly mobile portions of the adhesive, disturbing the balance of the solute and solvents or the molecular weight distribution of the polymers. This is often advantageous in that it develops quick drying properties. However, too rapid a disappearance of the mobile portions of an adhesive may leave a starved glue joint as sometimes occurs in plywood manufacture.

3. Physical properties of adhesive film - The characteristics of a cemented assembly are very largely influenced by the physical properties of the adhesive film and in determining these physical properties it is advisable that tests should be run using a thickness of adhesive film that will, in fact, be employed in practical use. In addition, drying of the test film should be performed under conditions similar to those of the intended application. Furthermore, the mechanical properties of the adhesives depend upon humidity and the rate of loading in certain cases, and these must be carefully controlled during the test. In this respect, the

synthetic resin adhesives behave much more satisfactorily at moisture extremes than do adhesives from animal or vegetable sources.

One of the most important physical properties of adhesive films is the modulus of elasticity, which reflects the ability of the glue to absorb and distribute the loads from one surface to another. It may be pointed out that from a structural viewpoint, a modulus at the glue line comparable to that of the materials being bonded is most desirable.<sup>1</sup> However, this is very difficult to achieve, particularly when metals and organic plastics are being bonded because of the wide differences in the moduli of the two materials.

In structural applications where adhesives may be under sustained loadings, creep under stress may seriously affect the efficiency of the glue joint. More highly plasticized materials are apt to creep under stress more readily than the stiffer, more rigid thermosetting materials. During the process of stressing, shear or tensile loads rupture the adhesive bonds originally established after application. A true permanent adhesive bond possesses negligible creep under stress.

The retention of volatiles at the glue line also contributes to the weakening of the adhesive film and the loss of its cohesive strength. The removal of these volatiles is dependent upon the manner of application of the adhesive and

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<sup>1</sup>Delmonte, J. The Technology of Adhesives, p. 330.

the surface conditions of the adherends. The non-porous types will retain the volatiles more readily and will result in lower strength films at the glue line.

4. Thickness of the adhesive film and viscosity of adhesive solution - The physical properties of the adhesive film are related to the thickness of the film and the percentage of voids due to the evaporation of solvents. The viscosity of the adhesive solution is related to the film thickness in that more viscous solutions tend to form the thickest films with the most voids. Hence, the greater the probability of loss in cohesive strength of the adhesive film when employing highly viscous adhesives. When the pressure of assembly exercises sufficient control of glue line thickness, this factor is less important. Generally, the thinnest films prepared from solvent type adhesives exhibit the greatest bond strength. It has been shown experimentally that there is a direct relationship between bond thickness in inches and bond strength in pounds per square inch of tension, i.e. the thinner the bond, the greater the bond strength.<sup>1</sup>

Finally, three stages of failure are evident in the film:

a. Failure in the glue film due to insufficient cohesive strength.

b. Failure at the interface - partly in the adhesive and partly as evidenced by pockmarks or separated fibers on the faces of the adherends.

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<sup>1</sup>de Bruyne, N. A., and Houwink, R. Adhesion and Adhesives.

c. Complete failure in the adherend - an ideal condition from the adhesive point of view.

5. Methods of application of adhesives - This influences the strength of the bond in several ways depending on three variables: pressure, temperature and time. The last two will determine when a film has been completely dried and the first factor will determine the thickness of the film. On uneven or porous surfaces, pressure is particularly desirable until the adhesive has developed sufficient cohesive strength to keep the assembly together.

6. Relation between strength of bond and area of adhesive film - Tests carried out by the College of Forestry at New York State University show that as the area of adhesion increases so does the strength of the bond. In fact, the strength of the bond was reduced by 17.1% when the bond area was decreased by one half.<sup>1</sup> However, Mr. Yavorsky argues that owing to the non-uniform stress distributions which occur in conventional test specimens, the load at failure, rather than the stress (load divided by area under test) should be employed as the criterion of strength of the glue joint.<sup>2</sup>

The area of the bond does not appear to be as important however, as the length of the periphery of the bond. It has

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<sup>1</sup>Yavorsky, J., Cunningham, J. H. and Hundley, N. G. "Survey of Factors Affecting Strength Tests of Glue Joints," Forest Products Journal, October 1955.

<sup>2</sup>Ibid.



been found that the plot of bond strength against length of periphery of the failure area resulted in a straight line. Presumably, once the periphery fails, the specimen fails.<sup>1</sup>

### Chemical factors influencing adhesion

1. Polar characteristics - That the polar characteristics of an adhesive influence the strength of adhesion is generally accepted, but the importance of this has been the subject of considerable controversy. Generally, substances with feeble polar groups possess small adhesion since polar groups are strongly adsorbed. Strong joints can never be made to polar surfaces with non-polar adhesives and vice-versa. Outstanding adhesives generally have strong polar groups in them. For example, the OH group in the phenolic resins, and the carboxyl group of the polyester resins are strongly polar.<sup>2</sup> Chlorinated rubber derivatives have shown good bonding to metal surfaces for the C-Cl polar bond is strongly adsorbed at clean metal surfaces.

2. Polymerization and molecular weight - Somewhere between the more highly polymerized and the unpolymerized fractions lies a range of molecular weight or degree of polymerization best suited for adhesive purposes. Sticky and tacky vinyl polymers for example, are formed within the range of fifty to three hundred degrees of polymerization.

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<sup>1</sup>Marra, Alan A. Ph.D. Thesis, University of Michigan, 1954.

<sup>2</sup>de Bruyne, N. A. and Houwink, R. Adhesion and Adhesives.

As the highest degree of polymerization yields the toughest films with the best cohesive strength and that the lowest degree of polymerization yields compounds which are usually mobile liquids, the optimum range of adhesive properties for thermoplastic polymers will be found in that range where good specific adhesion to the surface can be established and where the film possesses sufficient cohesive strength to establish permanency in the assembly.

3. Side groupings on polymer chains - The nature of the side grouping on the polymer chain is of the utmost significance in determining the merits of the polymer as an adhesive. Two factors appear to be directly related to the influence of side chains on adhesive action, firstly, polar characteristics, and secondly, chemical compatibility. For example, a polymer may have good polar characteristics but may be extremely difficult to dissolve due to its incompatibility with most solvents.

4. Evaporation and diffusion of volatiles from adhesive films - This is very important in the development of good adhesive strength. Except for the temporary benefits of permitting molecular rotation and polar adjustments, volatiles do not contribute much to the final adhesive properties. However, excessively fast evaporation should be avoided to prevent skinning-over of the adhesive film before the parts are pressed together. If the presence of volatiles is detrimental to the final bond, the sooner the volatiles are evaporated the better.

The wetting power of the volatile solvent and its penetrating characteristics may considerably alter the strength of the bond. With better penetration, there is a tendency towards starvation of the glue line and consequently lower strength. Another bad effect is that a surface, such as wood or paper, is more likely to become brittle if it is easily penetrated by a resin. To prevent this excessive penetration, thermoplastic polymers in small percentages may be added to thermosetting formulations to obtain higher strength.

5. Acidity or alkalinity of the glue line - The influence of the pH of the glue line upon the adhesive strength has been the subject of much investigation. It is generally recognized that strong acids and strong alkalies are detrimental to the adhesive bond, particularly if they exert a pronounced effect upon the materials being bonded. Cellulosic bodies such as wood and paper are affected more than non-porous organic plastic solids or metallic bodies.

### Conclusion

It must be stressed that the preceding comments are merely an introduction to the theory of adhesion and many detailed studies are available on the subject. However, one may conclude by quoting N. A. de Bruyne's two basic rules of adhesion which are that an adhesive should be used that wets the surface, i.e. that has a similar polarity, and that on solidification the adhesive must not develop residual stresses

sufficient to disrupt the bond.<sup>1</sup> In addition, adhesives should be used which are compatible with the adherends and the product and which are strong enough to resist any normal range of forces to which the bond may be subjected.

Testing procedures on adhesives are fairly numerous and can be found in the Handbook of the American Society for Testing Materials, D 897-46T.

## CHEMICAL CLASSIFICATION AND PROPERTIES OF ADHESIVES

### Thermosetting synthetic resin adhesives

These include the following adhesives:

Adhesive	End Use
Phenol-formaldehyde	Woods, cloth, paper, misc.
Phenol-furfuraldehyde	Woods, cloth, paper, misc.
Resorcinol-formaldehyde	Woods, plastics, rubber
Urea-formaldehyde	Woods, textiles
Melamine formaldehyde	Woods, textiles
Furanes	Plastics, rubber, misc.
Polyester resins	Cloth, glass, fabrics
Aniline formaldehyde	Woods, plastics
Polyurethanes	Woods, misc.

Through the action of heat, catalysts, or both, these resins may be converted into an infusible and insoluble state.

Generally, the phenolics are insoluble in water and are resistant to acids and mild alkalies. They are however, soluble in a solution of alcohol and monochloronaphthalene. Both urea and melamine are insoluble in water when set, the latter resin having a particularly low water absorption rate.

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<sup>1</sup>de Bruyne, N. A. "Some Basic Ideas," Structural Adhesives. Lange, Maxwell & Springer, p. 3.

They are both soluble to a certain extent in alcohol but are highly resistant to aromatics (benzene compounds). Ureas tend to set in an acid medium whereas melamine resins may set in neutral or faintly alkaline conditions. Aniline formaldehyde is insoluble in water when set as are the furanes. All thermosetting resins are soluble to some extent in alcohol and acetone.

### Thermoplastic synthetic resin adhesives

These include the following adhesives:

Adhesive	End Use
Polyisobutylene	Tapes, foils, misc.
Polyvinyl acetate	Metals, paper, misc.
Polyvinyl chloride	Misc.
Polyamides	Metals, woods, misc.
Silicones	Insulating materials
Polymethacrylic & polyacrylic esters	Safety glass, misc.
Polyvinyl butyral	Safety glass, misc.
Polystyrene	Shoe cements
Polyvinyl ethers	Paper, wood, misc.
Maleic anhydride adducts-glycerol-fatty acid resins	Plasticizers, misc.

Polyvinyl acetate adhesives are very popular and have excellent compatibility characteristics with other synthetic resin adhesives and various plasticizers and solvents. In this respect, they are far superior to the chlorinated polyvinyl esters which have poor compatibility. As general purpose adhesives for porous and some non-porous materials, polyvinyl acetate and chloride are widely used. Polyvinyl acetate is soluble in water to a slight extent and very soluble in methyl acetate, toluene, alcohol and ether. However,

its solvent retention is very pronounced and forced drying is often necessary to remove all the solvent. Polyvinyl ethers are not widely used as adhesives nor are polystyrene and the acrylic resins. Polyvinyl chloride is usually halogenated to give a polymer which is much more readily soluble in cheap lacquer solvents such as butyl acetate, benzene, acetone and ethylene dichloride. Polyvinyl acetates are generally in the form of white or light colored emulsions which give strong, dried films. Thermoplastic films are generally fast setting, light colored, humidity and grease resistant.

### Rubber adhesives

These include the following adhesives:

Adhesive	End use
Rubber latex	Cloth, paper, misc.
Natural rubber	Rubber, metal, misc.
Chlorinated rubber	Rubber, metal, misc.
Cyclized rubber	Rubber, metal, paper
Polychloroprene	Leather, misc.
Butadiene-acrylo-nitrile copolymer	Paper, metals, misc.
Synthetic resin-rubber combinations	Metals, plastics, misc.
Chlorinated synthetic rubbers	Misc.

Rubber adhesives are generally characterized by good flexibility, high initial tack and in certain cases, high specific adhesion to metal surfaces. In addition, they have other specific advantages over the more rigid synthetic resin adhesives. Not only are they capable of developing good specific adhesion for various surfaces but their high elongation permits the adhesive film to absorb a large amount of strain

compared with a more brittle resin adhesive in which greater stress concentrations will exist when loaded.

Latex adhesives tend to be slow drying due to the presence of slowly evaporating water and have not a high initial tack, but do have the advantage of containing no toxic solvents. They are soluble in naphtha, carbon tetrachloride and benzene. Natural rubber dissolved in naphtha, benzene, carbon disulfide, carbon tetrachloride or trichloroethylene forms highly viscous solutions which are capable of depositing tacky adhesive films. Cyclization of rubber is accomplished by treating rubber with concentrated sulfuric acid and then thoroughly washing it to remove traces of the acid. Rubber conditioned in this manner can form a much more efficient bond than untreated rubber. Synthetic resin-rubber combinations have produced some very strong adhesives, for example, phenol-modified rubbers possess an adhesion to smooth surfaces far superior to other rubber derivatives. Numerous adhesives made from polychloroprene are widely used to bond rubber to metal. Dried rubber films are usually very water resistant.

#### Cellulosic and starch derivatives

These include the following adhesives:

Adhesive	End Use
Cellulose nitrate	Leather, paper, misc.
Cellulose acetate	Leather, paper, misc.
Ethylcellulose	Cloth, paper, misc.
Methylcellulose	Paper, modifier, thickener
Sodium carboxymethyl cellulose	Paper, thickener
Starch, dextrin	Paper, wood, textiles

Cellulose derivatives have taken a prominent position in the adhesives industry and generally are capable of forming tacky films, well suited to the bonding of paper, leather and similar products. The softening point and chemical resistance of this type of adhesive depends greatly on the degree of substitution as well as on the chain length of the substituent. Thermoplastic cements prepared from cellulose nitrate or acetate in combination with a polybasic acid-polyhydric alcohol synthetic resin, yield strong, durable adhesives which can be heat-welded, are waterproof, flexible and resistant to the action of oils and greases.

An analysis of solvents for the cellulose esters is important to the formulation of adhesives from these materials. The cementing of two chemically incompatible bodies will not result in a strong assembly, even if the solvent cementing agent is satisfactory for each material individually, therefore chemical compatibility is most important to obtain a good bond. Cellulose derivatives are soluble in methyl acetate, acetone, ethyl acetate, methyl ethyl ketone, ethyl alcohol, butyl and amyl acetate. Cellulose acetate, however, is insoluble in ethyl alcohol. Ethylcellulose is soluble in the coal tar hydrocarbons and ketone. Methylcellulose is unique among the commercial cellulose esters in that it is completely water soluble. Sodium carboxymethyl cellulose is only slightly soluble in water.



Chemically, starch has the same constitution as cellulose  $(C_6H_{10}O_5)_x$ . Starch is slightly soluble in water and dextrins are even more soluble. Most dextrins are fluid, filmy, comparatively fast-setting materials.

### Protein adhesives

These include the following adhesives:

Adhesive	End Use
Casein	Wood, paper, misc.
Soyabean protein	Plywood
Zein	Paper, misc.

Adhesives from protein substances are widely used in the manufacture of plywood and for general purpose wood bonding. Since the development of synthetic resin adhesives, protein adhesives have suffered as a result of their poor performance in resisting moisture, although they are still widely used in the manufacture of coated papers. Casein adhesives have moderate to high water resistance and can be diluted with ammonia or alcohol. The dried films are strong, continuous and fairly light in color and are usually soluble in alkaline solutions. They have better adhesion to plastics, inked, or varnished surfaces than vegetable or animal glues and usually have alkaline pH.

### Adhesives of animal origin

These include the following adhesives:

Adhesive	End Use
Glue (hide, bone extract, green bone)	Wood, tapes, misc.
Fish offal	Wood, misc.
Blood albumen	Modifier

Hide glue possesses great strength whereas green bone glue has very weak strength characteristics. They are all soluble in water, the amount of solubility depending on the physical form of the glue, the more readily soluble types being more finely divided. Animal glues are also slightly soluble in glycerine and glucose. The adhesiveness of animal glues and gelatin has been improved by adding alcohols of higher molecular weight or sulfonation products thereof. The dried films exhibit good strength and have a slightly acid pH.

#### Natural resins and oils as adhesives

These include the following adhesives:

Adhesive	End Use
Rosin and rosin esters	Plasticizers and modifiers
Shellac	Insulation, misc.
Ashphalts	Minerals, misc.
Gum tragacanth	Thickeners for protein and starch
Ester gum	Plasticizer and modifier
Linseed oil (oxidised)	Linoleum, misc.

Natural resins are often sticky in themselves and are definitely thermoplastic in nature. Various water soluble gums are derived from the exudations of the various kinds of Acacias, particularly gum arabic, which is soluble in two or three times its weight of water. Gum tragacanth is not as soluble in water as gum arabic and manila gum is only soluble in denatured alcohol. Shellac is very soluble in ethyl or methyl alcohol. Ashphalts are a composition of many compounds and are abundant, low in cost, resistant to water and susceptible to temperature changes.

### Inorganic adhesives

These include the following adhesives:

Adhesive	End Use
Sodium silicate	Corrugated paper, paper products
Plaster of Paris	Ceramics, misc.
Portland cement	Mineral aggregates

Sodium silicate has wide applications in the paper industry and is manufactured by the fusion of sand and sodium oxide to yield a soluble silicate, usually sold in a concentrated water solution. Because of the abundance of raw materials, sodium silicate adhesives are extremely cheap. They are not as strong as animal or protein adhesives but give better strength than some starch and dextrin types. They lack high tackiness but demonstrates a great change of viscosity with changes in the moisture content above a certain range. Sodium silicate adhesives have a strong alkaline reaction which prevents them from being used in certain special cases. They set rapidly, depending for their setting properties upon diffusion of water into a porous medium such as paper and wood.

### CONCLUSION

There are at present, about twenty thousand adhesives commercially available and one has therefore considerable choice in selecting an adhesive for a specific purpose. If the cap liners are going to be adhered to the inside of the cap, then in the light of the information contained in the earlier parts of this chapter on the theory of adhesion and

the chemical and physical properties of adhesives, the following points should be borne in mind when choosing the adhesive:

1. The nature of the adherends or in the common vernacular, "what do you want to stick to what?". Certain adhesives will not adhere to metals unless the metals are coated and this is very important when using metal caps. In addition, the nature of various paperboards will affect the adhesive bond as a result of their sizing, coating and density.

2. Is color a factor? Certain adhesives are colored or can be colored whilst others are transparent.

3. Is odour a factor? Food packaging calls for adhesives with a minimum of odour.

4. Is excessive dry heat a factor in the production process? For example, are the bottles sealed while the product contained therein is hot? The adhesive when set, must have a higher melting point than any temperature to which it is subjected.

5. Is dry cold a factor? This will occur often in storage or transportation in cold climates. The adhesive when set, must retain its bond strength at low temperatures and the adherends must also remain stable.

6. Is moisture resistance, excessive humidity or sweating a factor? This is most important in toilet articles which are opened in steamy bathrooms, for example. Reference to the chemical properties of the adhesive will result in the choice of a water resistant type.

7. Is immersion in liquid a factor? This is particularly important as the product may creep around the liner and into the backing and adhesive. An adhesive must be chosen which is not soluble in any of the chemical solvents contained in the product being packaged.

8. Is toxicity a factor? If food products are to be packaged then a non-toxic adhesive must be used.

9. Is pH a factor? The alkalinity or acidity of the adhesive and adherends must be determined and an adhesive chosen which is chemically compatible.

10. Should the adhesive contain an additive to give further properties to it such as increased water resistance? For example, fillers can be added to most resins to increase their resistance to water or other chemicals without affecting their strength.

11. How is the adhesive to be applied? The speed at which the adhesive is to be applied as well as the method of application is important, for different setting times are required for different adhesives. Adhesive manufacturer's specifications will always include the setting time required as well as information as to whether pressure should be applied to the bond while it is setting.

12. What consistency of adhesive is required? This will affect the spreading of the adhesive and the wetting angle which will in turn influence the strength of the bond.

13. Is there air drying time before combining?

Excessive air drying time may result in a skin forming on the adhesive and thus weakening the eventual bond.

14. Can heat be used to reduce viscosity but retain high solids? Heat must sometimes be applied to make the adhesive flow more freely in order to obtain a better bond but it must not cause a deterioration in the adhesive or adherends.

15. Is strength a factor? If excessive loads or torques are to be applied to the bond, the adhesive must be capable of resisting these forces and must also be capable of resisting any warping that may occur in the adherend.

16. A final check should always be made if government or consumer specifications are stated to insure that these will be met in the choice of the particular adhesive.

If all these questions are considered in selecting an adhesive to glue a liner to a screw cap, a satisfactory bond will result and the adhesive will not be the cause of any delamination that occurs.

## CHAPTER II

### LINERS

#### LINER BACKINGS

##### Introduction

A liner backing is generally a material of varying degrees of resilience and compressibility which will conform to the contour of the sealing surface of the bottle. They provide the necessary cushion behind suitably chemically inert facing materials to which they are bonded, and they produce a gasket effect or seal between the container and the cap closure.

##### Pulpboard and newsboard

These are the most widely used materials for backings and are generally manufactured on a cylinder machine with bleached or semi-bleached pulp. Pulpboard contains no size and therefore has extremely low water resistance. With a suitable facing, these backings perform satisfactorily and are of very low cost. However, their resilience is lowered after excessive application of torque and as a result their performance as an effective seal diminishes. This latter consideration is most important when volatile products are being packaged.

##### Composition cork

This is often used as a backing because, being composed

of scrap cork flakes and granules combined with a binder such as casein or vegetable glue, it is of lower cost than pure natural cork which is extremely expensive and is dependent on foreign suppliers. It shows much greater resilience than pulpboard at normal cap-sealing pressures. However, composition cork has generally to be backed with paper in order to reduce its tendency to curl, which sometimes causes the liner to fall out of the cap. Cork backing with the proper facing material is widely used for sealing certain liquors, perfumes, volatile solvents and essential oils.

#### Gray felt and white felt

These backing materials are used in fairly large quantities in closures for those products where pulpboard and newsboard have been found to be too hard and where the excellent sealing efficiency of composition cork is not required. Liners backed with white or gray felt have been found effective in molded closures having a relatively thin construction and subject to breakage when torque is applied to them. By using a felt backing, greater compression into the liner is made by the glass finish at low sealing pressures. Felt backings have been found adequate for many volatile products.

#### Plastic-lined caps

Theoretically, in this type of closure, the facing, backing and adhesive are all one and the same material. Vinyl-



resin plastisol composition is molded into the caps with a thin center section and a series of concentric rings in the sealing area, to provide for multiple sealing points and easier flow of liner material around irregularities in the glass finish. Plastic liners of this type are usually lighter in weight than composition cork, are not subject to the excessive drying and loss of sealing properties of cork liners, have a more uniform performance than composition cork and can be modified to become chemically resistant to certain products. Furthermore, they are odourless and supplies are not dependent on foreign sources. Under test, their product retention capabilities have proved to be as good as those of composition cork and have generally shown excellent resealing efficiency. Additional important advantages of vinyl-resin liners over composition cork are in the absence of liner discolouration, the comparative freedom from particles of the liner sticking to the glass finish and their generally cleaner appearance.<sup>1</sup>

## LINER FACINGS

### Introduction

Recent developments in synthetic resins have made available a large number of facing materials for use in bottle cap liners. The following is a brief description of the more common facings used at present in the industry and some of their characteristics.

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<sup>1</sup>Brockett, H. E., Modern Packaging, September 1956, pp. 135-40.

### Tin foil

This material is the ideal facing for packaging medicinals, pharmaceuticals and biologicals containing highly volatile organic chemicals, or preparations containing an equivalent alcohol content in excess of fifty per cent. However, it is relatively expensive, depends on overseas sources for raw materials, and synthetic resins not dependent on overseas sources of supply are being developed to take its place.

### Aluminium foil

This material is fairly extensively used as a facing, particularly in crown closures in the brewing industry. Laminated to a suitable backing, it is used in continuous thread caps for sealing non-alkaline products of a not too volatile nature but containing ingredients that readily affect varnish, synthetic resin or wax coatings. The foil is usually laminated to a composition cork backing, because this foil has a hard surface and a resilient cork backing permits better conformity to the glass finish. It is, however, important to remember that very thin aluminium foils have a considerable moisture vapour transmission rate due to the presence of minute pinholes in thin foils. This can result in the product passing through the facing and into the backing material and this will contribute to the delamination of the liner facing from the backing.

### Lead foil

This has been used for sealing concentrated sulfuric acid, petroleum hydrocarbons and aromatics but it cannot be used on food products or cosmetics because of the possibility of lead poisoning resulting from the toxicity of lead.

### Pliofilm

This film of rubber hydrochloride provides a very versatile facing and is used in large quantities for sealing alcoholic beverages, chlorinated washing fluids, drugs and pharmaceuticals. As a rubber derivative, it should not be used for sealing oils, oily products or for hot-packed products. It has a very low water vapour transmission rate and excellent conformity to glass finishes.

### Laminated cellophane

This is manufactured for liners by combining moisture-proof and non-moistureproof transparent cellophane or regenerated cellulose to paper. It is recommended for use with mineral, vegetable and fish oils and on certain essential oils and organic solvents.

### Laminated saran

This is made by combining seventy-five or one hundred gauge poly-vinylidene-chloride film to paper with a thermoplastic adhesive to produce a very resistant liner. It has

outstanding properties of high gloss and attractive appearance, toughness, flexibility, minimum odour and taste, chemical resistance and strong bonds can be formed between the film and the backing. It has a very low vapour transmission rate and is very satisfactory for sealing perfumes, face creams, lotions, deodorants and many drugs and chemicals. A direct saran lamination to white-lined pulpboard produces an attractive, efficient, fairly low cost liner.

#### Varnish and resin coated paper

These liner materials are produced in very large quantities by applying resistant oleoresinous baking varnishes to bleached kraft paper. These liners have high gloss, good flexibility, and adequate resistance to water and to vegetable oils. They are widely used in the food packing industry for they are very inexpensive.

#### Synthetic-coated paper facings

The principal facing used in the closure industry is composed of urea-formaldehyde-melamine resin applied to pure or bleached kraft paper. Its principal properties are solvent and oil resistance and it is used as a substitute for tin or aluminum foil on products where a white liner is required and liner costs are very important. This facing has good water resistance but does not conform well to glass finishes and as a result, relatively high evaporation loss will occur on aque-

ous or volatile products. These losses are particularly evident when the bottles are of small volume and have a relatively large diameter opening.

#### Dark brown acid resisting paper

This is manufactured by coating kraft paper with an oleoresinous varnish containing gilsonite or an asphalt base. It provides a liner with unusually good resistance to acid products.

#### Dark brown acid resisting paper (casein coated)

Basically the facing is similar to dark brown acid resisting paper but a casein coating has been added for resistance to organic solvents and oils.

#### Casein coated tin foil

This is used where greater sealing efficiency is required than can be obtained with plain tin foil, e.g. in sealing mineral oils, vegetable oils and various organic solvents. This greater sealing efficiency is a result of the casein coating providing a better conformity to the glass finish than the plain tin foil.

#### Waxed composition cork

This is used in packaging products where the high sealing efficiency of cork is required and where the product is

of a mild nature and will not attack the wax which is readily soluble in such organic compounds as carbon tetrachloride, toluene, heptane and decane. Waxes, being esters of higher monohydroxy alcohols, can be used in packaging products with a high alcohol content. In addition, wax coatings are applied to many other liners to improve the sealing efficiency of the closure and to act as a lubricant when torque forces are applied to the screw cap and prevent the liner sticking to the glass finish.

#### Calendered coatings

Paper, calendered and coated with vinyl chloride acetate, has been used for many years without outstanding success in the drug and pharmaceutical field. This type of liner has excellent chemical resistance to weak acids and alkalies and many other chemical solutions. In addition, it has a very attractive appearance, is low in cost but has a rather high water vapour transmission rate. Calendered polyethylene is made by a similar process to the vinyl chloride acetate. It has a high chemical resistance and a very low water vapour transmission rate.

#### Wax coatings

These liners have a base coating of refined paraffin wax, microcrystalline wax, ceresine wax or a formulation of all three. They are not suitable for products containing mineral, vegetable, fish or essential oils nor for products containing solvents which might affect the wax.

## DELAMINATION TESTS ON LINER BACKING MATERIAL

### Objective

The object of these tests was to show the effects of various liquids on the delamination of backing material and to see whether specific liquids, or liquids generally, will increase the possibility of delamination.

### Test equipment

1. Baldwin-Emery S. R. 4 testing machine, Model FGT
2. 20 steel blocks with milled faces, 2 x 1/4 x 1/4 inches
3. 10 glass beakers (500 cc.)
4. Bleached cylinder-board, thickness .0025 inches
5. Adhesive: Weldwood contact cement
6. Standard reagents (see test results)

### Procedure

At present there are no accurate scientific tests that have been standardized to measure the delamination of paper-board. Indeed, the American Society for Testing Materials' test is literally a rule of thumb judgement.<sup>1</sup>

Ten specimens of cylinder-board were cut to a size, 1/4 x 1/4 inch, giving a surface area of .625 square inches which is equal to the surface area of the ends of the steel blocks. Each specimen was bonded between two steel blocks using Weldwood contact cement (which has excellent adhesive

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<sup>1</sup>ASTM Standards on Paper and Paper Products, p. 251. Sept. 1955.

properties to metal and paper and is insoluble in water), the assembly then being allowed to dry for three hours under standard conditions of 72° F. and 52% relative humidity. After each part of the test, the assemblies were again prepared in the above manner. The ten assemblies were then subjected to a direct tensile test on the Baldwin-Emery testing machine at a platen speed of .050 inches per minute until failure of the specimen occurred. The pounds of force required to bring about total failure of the assembly were then recorded.

### Results

Part I - Steel blocks and liner assemblies were stored at standard conditions for six hours prior to the test.

Sample number	Lbs. of force at which complete delamination occurs
1	0.92
2	3.24
3	1.53
4	1.38
5	2.18
6	2.67
7	2.83
8	3.23
9	2.02
10	5.22
	Mean 2.53

Part II - Steel blocks and liner assemblies were immersed for thirty minutes in distilled water, and all assemblies failed before removal from the water.

Part III - Steel blocks and liner assemblies were immersed for thirty minutes in a 10% sodium hydroxide solu-



tion, and all assemblies failed before removal from the solution.

Part IV - Steel blocks and liner assemblies were immersed for thirty minutes in a 1% sodium hydroxide solution, and all assemblies failed before removal from the solution.

Part V - Steel blocks and liner assemblies were immersed for thirty minutes in a 10% sodium chloride solution, and all assemblies failed before removal from the solution.

Part VI - Steel blocks and liner assemblies were immersed for thirty minutes in 95% ethyl alcohol, and all assemblies failed before removal from the alcohol.

Part VII - Steel blocks and liner assemblies were immersed for thirty minutes in 50% ethyl alcohol, and all assemblies failed before removal from the alcohol.

Part VIII - Steel blocks and liner assemblies were immersed for thirty minutes in acetone, and all assemblies failed before removal.

Part IX - Steel blocks and liner assemblies were immersed for thirty minutes in ethyl acetate, and all assemblies failed before removal.

Part X - Steel blocks and liner assemblies were immersed for thirty minutes in N-heptane, and all assemblies failed before removal.

Part XI - Steel blocks and liner assemblies were immersed for thirty minutes in a 3% solution of sulfuric acid, and all assemblies failed before removal.

Part XII - Steel blocks and liner assemblies were immersed for thirty minutes in a 30% solution of sulfuric acid, and all assemblies failed before removal.

### Conclusions

A measure of the pounds of force necessary to bring about complete delamination of cylinder-board can be found using a tensile test on the Baldwin-Emery testing machine.

The laminated strength of the cylinder-board is greatly reduced by contact with liquids for any length of time.

### THEORY OF FAILURE OF PAPER AND BOARD UNDER TENSILE STRESS

In Part I of the above test, a progressive failure in the board was evidenced by the erratic movements of the recording needle on the testing machine. This progressive failure may be accounted for by the mechanical properties of the paper or board and their actions when subjected to strain. The strain on the paper consists of four components: 1) the extension of individual fibers which is a recoverable elastic strain; 2) uncoiling of the fibers which is an unrecoverable creep; 3) slipping of unbounded fibers over one another which is also an unrecoverable creep; 4) relative movement of bound fibers which is primary creep associated in the paper or board with the bonding between the individual fibers.<sup>1</sup>

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<sup>1</sup>Steenberg, B. "Paper as a visco-elastic body," Svensk Papp. Tidn., Part 1.

Finally, there are two other important factors that influence the strength of paper and board and which, as a result, will influence delamination. Firstly, the degree of beating of the pulp is important in that continued beating will progressively increase the area of adhesive cellulose which will result in a progressively stronger bond structure up to a point where the effect of the break-up of the fibers results in a lower overall strength structure.<sup>1</sup>

Secondly, it is a well-recognized fact that cylinder-board, if made on more than one cylinder is considerably weaker than a similar thickness of fourdrinier board, for in the former, the individual layers of pulp tend to separate very easily compared with the homogeneous structure of fourdrinier board.

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<sup>1</sup>Meredith, R. Mechanical Properties of Wood and Paper, p. 212.

## CHAPTER III

### SCREW CAP CLOSURES

#### INTRODUCTION

The discussion that follows is limited to metal caps and rigid plastic caps for semi-rigid closures such as polyethylene caps do not generally contain liners and the delamination problem does not arise. The screw type closure is widely used in the glass packaging industry as it offers a mechanically simple means of applying sufficient force to provide an efficient seal, not only on initial application but on subsequent resealings.

#### TYPES OF SCREW CAP CLOSURES

##### Metal caps

In using metal caps the liners are generally not adhered to the inside of the cap, but depend on a tight press fit, the liner being held in place by the protruding metal threads on the inside of the cap. This enables a small air pocket to form between the inside of the metal cap and the liner backing and since the metal conducts heat rapidly, a decrease in the outside temperature causes a different rate of cooling to take place between the cap and the liner backing. As a result, condensation will occur in this air space and the moisture may warp the liner or lower its laminate strength so that it is no longer a tight fit and it will easily delaminate or fall out of the cap.

This increase in moisture between the backing and the inside of the cap is known as sweating action and is much more noticeable in metal caps than in plastic caps.<sup>1</sup>

Very little tensile force is required to pull liners out of metal caps as they are not adhered to the inside of the cap and the slightest sticking of the liner facing to the glass finish will result in delamination of the liner from the metal cap. This often occurs if the product is of an adhesive nature and some of it remains on the glass finish after pouring, adhering the liner to the glass finish on resealing.

Metal screw caps are more resistant to shock than rigid plastic caps but are easily oxidized and corroded by many chemicals unless a coating is applied to them which, however, will raise their cost.

Metal caps, as has already been stated, do not generally have their liners adhered to them, and as a result shear forces set up when torque is applied are lessened slightly as the liner can rotate in the cap.

Changes in temperature in addition to causing the sweating effect can also cause a pumping effect to take place by changing the pressure in the air pocket between the liner backing and the inside of the metal cap. The pumping and sweating effect combined, can loosen the pulp backing from

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<sup>1</sup>Borg, Henry A. Unsolved Delamination Problems in Screw Cap Closures Used on Certain Bristol Myers Products (term paper, Forest Products Dept., M. S. U., 1956), p. 35.

the liner facing causing delamination. This can be avoided by applying a small amount of soft wax between the pulp backing and the metal cap in conjunction with an insulating type of coating inside the metal cap.

### Rigid plastic caps

In this type of cap the liner is generally adhered to the inside of the cap to keep it in place. However, this results in shear forces occurring when torque is applied to the cap. These forces can be overcome only by using an adhesive and liner, the combination of which is sufficiently strong to overcome these forces in all normal torque ranges.

These shear forces can be reduced by using wax on the facing of the cap liners. This wax acts as a lubricant and also prevents the liner facing from sticking to the glass finish. Tests were made with and without wax on the facing at various torque ranges and the results (see test below) confirm this property of the wax.

### Torque test on waxed and unwaxed liners

**Objective** - The object of this test was to show that wax on liner facings acts as a lubricant and prevents the liner from sticking to the glass finish.

#### **Equipment -**

1. Owens-Illinois torque tester 0-25 inch lbs.
2. Eighty test bottles, 28 MM, ½ oz. amber flasks
3. Eighty plastic caps, 28 MM, 400 LUS
4. Eighty vinyl faced pulpboard liners
5. N-heptane

Procedure - All of the test was carried out in the conditioning room under standard conditions (72°F., 52% relative humidity). The liners were already adhered in the caps and on forty of these the wax was removed from the facing by lightly rubbing it off with a cloth soaked in N-heptane. Care must be taken while doing this as N-heptane dissolves the vinyl as well as the wax. However, wax is much more soluble in the heptane than the vinyl and light rubbing followed by rubbing with a cloth soaked in distilled water will remove the wax without affecting the facing to any extent. The caps were then screwed onto the bottles at various torque levels from ten to twenty-five inch pounds of torque, ten waxed and ten unwaxed liners being used at each torque level. The caps were then removed from the bottles and the amount of torque required for removal measured and recorded.

Results - In inch pounds of removal torque at selected applied torque levels:

Applied torque:	10" lbs.		15" lbs.		20" lbs.		25" lbs.	
	W*	U*	W	U	W	U	W	U
	7.0	8.5	9.5	10.5	15.0	16.5	20.0	20.5
	6.5	9.0	9.5	11.0	13.5	19.0	19.5	19.0
	8.0	10.0	11.5	12.5	14.0	18.0	18.0	21.0
	7.0	10.0	11.0	12.5	15.0	15.5	19.0	23.5
	6.5	8.5	10.5	11.0	15.0	15.0	11.5	24.5
	8.0	9.0	10.0	13.0	17.0	18.0	18.0	23.0
	7.5	10.0	11.5	13.0	15.5	17.0	18.0	25.0
	7.0	9.0	10.5	14.0	16.5	18.0	18.0	23.5
	7.5	10.0	11.0	12.5	13.5	15.5	17.5	24.0
	7.0	10.0	9.0	12.0	16.5	16.0	19.5	24.5
Mean:	<u>7.2</u>	<u>9.4</u>	<u>10.4</u>	<u>12.2</u>	<u>15.15</u>	<u>16.85</u>	<u>18.5</u>	<u>22.85</u>

\*W - Waxed liners, U - Unwaxed liners

Statistical analysis of torque test results - To see if the results on waxed and unwaxed liners are statistically different at each applied torque level.

Applied torque	Waxed $\Sigma d^2$	Unwaxed $\Sigma d^2$	Degrees of freedom
10 inch lbs.	2.6	3.9	18
15 " "	7.08	10.6	18
20 " "	15.225	16.525	18
25 " "	7.5	36.025	18

Using the formula:

$$"T" = \frac{M_1 - M_2}{\sqrt{\frac{\Sigma d_1^2 + \Sigma d_2^2}{(n_1 + n_2) - 2} \left( \frac{1}{n_1} + \frac{1}{n_2} \right)}}$$

Where:

$M_1$  = Mean of waxed liners

$M_2$  = Mean of unwaxed liners

$n_1$  = Number of samples of waxed liners (10)

$n_2$  = Number of samples of unwaxed liners (10)

$\Sigma d_1^2$  = Sum of the squares of the deviations from  $M_1$

$\Sigma d_2^2$  = Sum of the squares of the deviations from  $M_2$

Results:

at 10" lbs. of applied torque the "T" value= 8.18.

Therefore the level of significance ( $18^0$  of freedom) = .01

and therefore the difference is statistically significant at the 1% level.

at 15" lbs. of applied torque the "T" value= 4.091.

Therefore the level of significance ( $18^0$  of freedom) = .01

and therefore the difference is statistically significant at the 1% level.



at 20" lbs. of applied torque the "T" value= 2.872. Therefore the level of significance ( $18^{\circ}$  of freedom) = .02 and therefore the difference is statistically significant at the 2% level.

at 25" lbs. of applied torque the "T" value= 6.547. Therefore the level of significance ( $18^{\circ}$  of freedom) = .01 and therefore the difference is statistically significant at the 1% level.

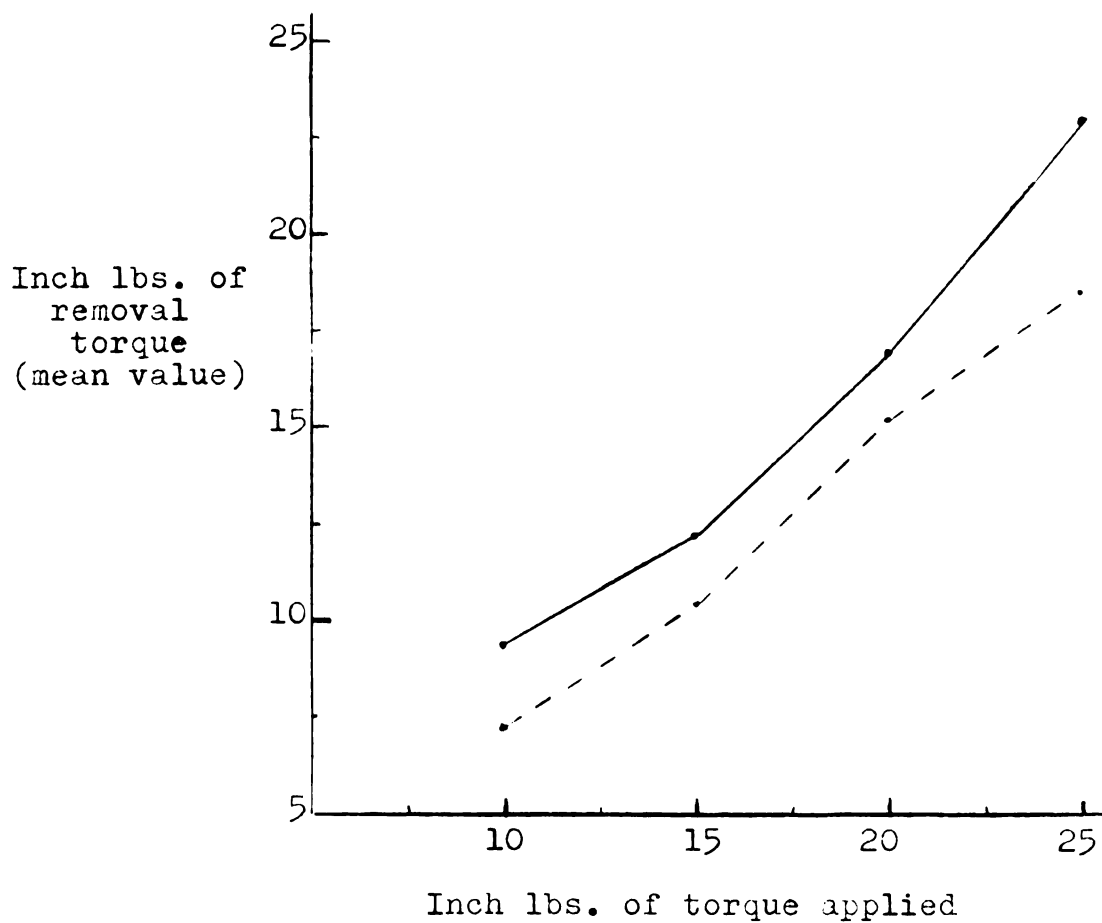
Conclusions on statistical analysis: There is significant difference between the removal torque required for waxed and for unwaxed liners at every selected torque level. The values for the waxed liners are lower at every torque level indicating that the wax acts as a lubricant and prevents the liner from sticking to the glass finish. As a result, less torque is required to remove the cap, and the liner and adhesive are therefore less subject to excessive shear forces which can cause delamination. For a graphical representation of the test results see page 42.

#### GENERAL FACTORS CAUSING DELAMINATION

##### pH factor

Materials that are strongly acidic or alkaline can undergo changes that bring their pH closer to natural resulting in a change in their physical and chemical properties. If a liner and adhesive are selected that are incompatible with the product or with each other, decomposition of the adhesive, the liner or the product may occur. It is therefore

A GRAPHICAL REPRESENTATION OF THE RELATIONSHIP  
BETWEEN APPLIED AND REMOVAL TORQUES ON WAXED  
AND UNWAXED LINER FACINGS IN SCREW CAPS



- - - - - = waxed liners

————— = unwaxed liners

most important, as has already been stated, to insure that the product, the facing, the backing and the adhesive are all chemically compatible.<sup>1</sup>

### Marking the facing

Certain manufacturers stamp an identification number on the facing inside inside the cap e.g. on Bristol Myers' Vitalis. Sometimes this stamping passes completely through the facing and as a result destroys its utility for the product can then penetrate the backing and cause delamination.

### Hydrodynamic effects on delamination

Hydrodynamic action in a filled glass container is caused by a sudden acceleration of the glass container with respect to its contents, followed by a sudden deceleration of the container. Besides causing possible breakage of the container, hydrodynamic action causes a "sucking" effect to take place within the container. This "sucking" action has a tendency to weaken the adhesive bond between the facing and backing of the closure, contributing to the delamination problem.

### Torque ranges

Determining the proper torque range for each liner combination will give longer shelf life to the product and keep

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<sup>1</sup>For liner compatibility tests see Packaging Institute Test Procedures, PI Closures 2t-51.

delamination at a minimum. As yet there is no scientific method of determining the correct amount of torque necessary for each cap size and liner combination. An empirical chart designed by the Owens-Illinois Company of Toledo, Ohio, lists a general guide for use in determining approximate torque values on closure sizes from 15 to 70 millimeters. The figures listed below are entirely empirical and constitute a general guide rather than definite recommendations to fit specific circumstances.<sup>1</sup>

CHART OF SUGGESTED TORQUE RANGES

Closure size in millimeters	Suggested tightness of applica- tion in units of inch pounds of torque as applied by hand
15	6 - 9
20	8 - 12
24	9 - 15
28	10 - 18
33	12 - 21
38	15 - 25
43	17 - 27
48	19 - 30
53	21 - 36
58	23 - 40
63	25 - 43
70	28 - 50

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<sup>1</sup>Packaging Research Division, Owens-Illinois Glass Company, Toledo, Ohio.

Torque settings can cause liner delamination to take place in two ways:

1. Over-torque can cause a crushing of the pulp, cracking of the facing or cause the facing to stick to the glass finish, so that in subsequent removals of the closure the contents of the container may penetrate into the pulp weakening the liner bond and causing delamination.

2. Under-torque can cause a seepage of the contents under the facing and into the pulp, gradually dissolving the adhesive bond between the pulp, cap and facing, causing delamination to take place.

Finally, the consumer will often spill the contents of the container onto the glass finish and often onto the liner facing. The closure is then replaced and tightened onto the glass container. This tightening of the closure forces some of the product up into the edges of the liner as well as sticking the liner to the glass finish. This process may be repeated many times and will contribute greatly to delamination.

#### Deterioration of liners caused by liquid penetration

We have already seen in Chapter II the deleterious effects on cylinder-board of liquid penetration and a similar effect takes place in complete closures, and is a very pertinent factor in bringing about delamination caused by failure of the backing, the bond between the facing and backing or the bond between the backing and the inside of the cap.

### Test for delamination in screw cap closures

Objective - To determine the effect on delamination of various liquids coming into contact with the complete cap assembly, i.e. liner adhered to the inside of the cap.

#### Equipment -

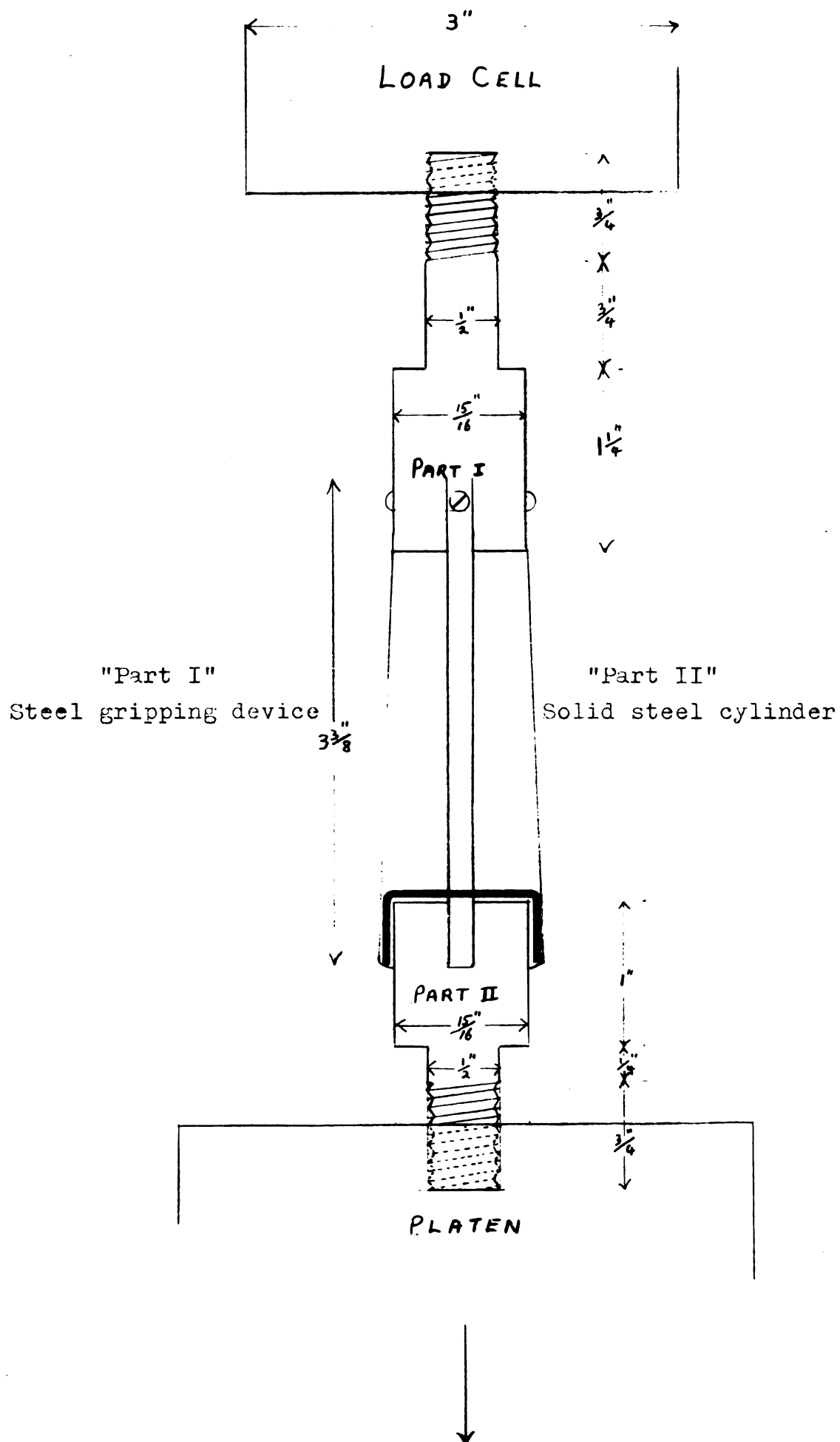
1. Baldwin-Emery S.R. 4 testing machine, Model FGT
2. Baldwin S.R. 4 load cell
3. Steel gripping device (see "Part I" page 47)
4. Solid steel cylinders with milled faces and opposite ends threaded (see "Part II" page 47)
5. Plastic caps (28 MM, 400 LUS) and vinyl faced pulpboard liners adhered to caps
6. 500 cc. glass beakers
7. Distilled water
8. 95% ethyl alcohol
9. Mineral oil
10. 10% sodium hydroxide solution
11. 3% sulfuric acid
12. N-heptane and toluene for cleaning purposes

Procedure - The facings of the liners in the caps were cleaned with N-heptane to remove the wax and to enable the milled face of each solid steel cylinder to be adhered to each liner with Weldwood contact cement. This adhesive was chosen because it is insoluble in water and oils, has a neutral pH and forms excellent bonds between plastics and metals. It requires neither heat nor pressure while drying and the manufacturers recommend a drying time of three hours.<sup>1</sup>

After drying under standard conditions for three hours, the steel cylinders with the caps adhered to them were removed from the conditioning room for the test. A steel cylinder and cap assembly was screwed into the platen of the Baldwin-Emery testing machine and the claws of the clamping device

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<sup>1</sup>United States Plywood Corp. Technical Data Sheet 200-1.



were hooked over the edge of the cap (see page 47). A tensile load was then applied at a platen speed of .050 inches per minute until final failure of the specimen occurs and the load in pounds at failure recorded (see test results). As in the case of the tensile tests on cylinder-board in Chapter II of this paper, a progressive failure in the structure occurs up to final delamination.

#### Results -

Part I - After drying, the ten assemblies were kept under standard conditions for twelve hours and the load at final failure recorded for each one.

Sample No.	Failure load	Remarks
1	5.32 lbs.	Complete delamination of pulpbd.
2	5.86 lbs.	Complete delamination of pulpbd.
3	5.81 lbs.	Complete delamination of pulpbd.
4	4.46 lbs.	Delamination of facing from backing
5	7.66 lbs.	Complete delamination of pulpbd.
6	5.18 lbs.	Complete delamination of pulpbd.
7	5.60 lbs.	Complete delamination of pulpbd.
8	5.18 lbs.	Complete delamination of pulpbd.
9	4.42 lbs.	Complete delamination of pulpbd.
10	5.87 lbs.	Complete delamination of pulpbd.

Part II - After drying, the ten assemblies were placed in distilled water for thirty minutes prior to test.

Sample No.	Failure load	Remarks
1	0.94 lbs.	Complete delamination of pulpbd.
2	1.33 lbs.	Complete delamination of pulpbd.
3	1.11 lbs.	Complete delamination of pulpbd.
4	2.38 lbs.	Complete delamination of pulpbd.
5	1.65 lbs.	Complete delamination of pulpbd.
6	0.98 lbs.	Complete delamination of pulpbd.
7	2.86 lbs.	Complete delamination of pulpbd.
8	2.16 lbs.	Complete delamination of pulpbd.
9	1.27 lbs.	Complete delamination of pulpbd.
10	1.02 lbs.	Complete delamination of pulpbd.



Part III - After drying, the ten assemblies were placed in 95% ethyl alcohol for thirty minutes prior to test.

Sample No.	Failure load	Remarks
1	1.91 lbs.	Complete delamination of pulpbd.
2	1.90 lbs.	Complete delamination of pulpbd.
3	0.87 lbs.	Complete delamination of pulpbd.
4	0.88 lbs.	Delamination of facing from backing
5	1.97 lbs.	Complete delamination of pulpbd.
6	1.39 lbs.	Complete delamination of pulpbd.
7	1.94 lbs.	Complete delamination of pulpbd.
8	1.32 lbs.	Complete delamination of pulpbd.
9	1.27 lbs.	Complete delamination of pulpbd.
10	1.68 lbs.	Complete delamination of pulpbd.

Part IV - After drying, the ten assemblies were placed in mineral oil for thirty minutes prior to test.

Sample No.	Failure load	Remarks
1	3.36 lbs.	Complete delamination of pulpbd.
2	6.58 lbs.	Complete delamination of pulpbd.
3	9.16 lbs.	Complete delamination of pulpbd.
4	7.13 lbs.	Complete delamination of pulpbd.
5	8.96 lbs.	Complete delamination of pulpbd.
6	3.92 lbs.	Complete delamination of pulpbd.
7	1.43 lbs.	Delamination of facing from backing
8	3.90 lbs.	Complete delamination of pulpbd.
9	2.84 lbs.	Complete delamination of pulpbd.
10	1.73 lbs.	Delamination of facing from backing

Part V - After drying, the ten assemblies were placed in a 10% sodium hydroxide solution for thirty minutes prior to test.

Sample No.	Failure load	Remarks
1	0.67 lbs.	Complete delamination of pulpbd.
2	0.38 lbs.	Complete delamination of pulpbd.
3	0.71 lbs.	Complete delamination of pulpbd.
4	0.51 lbs.	Complete delamination of pulpbd.
5	0.57 lbs.	Complete delamination of pulpbd.
6	0.42 lbs.	Complete delamination of pulpbd.
7	0.50 lbs.	Complete delamination of pulpbd.
8	0.61 lbs.	Complete delamination of pulpbd.
9	0.54 lbs.	Complete delamination of pulpbd.
10	0.66 lbs.	Complete delamination of pulpbd.

Part VI - After drying, the ten assemblies were placed in 3% sulfuric acid for thirty minutes prior to test.

Sample no.	Failure load	Remarks
1	1.41 lbs.	Delamination of facing from backing
2	0.79 lbs.	Delamination of facing from backing
3	1.06 lbs.	Delamination of facing from backing
4	0.99 lbs.	Delamination of facing from backing
5	1.28 lbs.	Complete delamination of pulpbd.
6	1.12 lbs.	Delamination of facing from backing
7	1.03 lbs.	Delamination of facing from backing
8	0.91 lbs.	Complete delamination of pulpbd.
9	1.16 lbs.	Complete delamination of pulpbd.
10	0.87 lbs.	Delamination of facing from backing

Statistical analysis of tensile test results - To see if there are statistically significant differences between the failure loads of each of the other parts of the test.

Test part	Mean failure load	$\Sigma d^2$	Degrees of freedom
I	5.54 lbs.	7.3846	18
II	1.57 lbs.	4.0814	18
III	1.51 lbs.	1.6521	18
IV	4.90 lbs.	73.0919	18
V	0.62 lbs.	0.2173	18
VI	1.06 lbs.	0.3698	18

Using the formula:

$$"T" = \frac{M_1 - M_2}{\sqrt{\frac{\Sigma d_1^2 + \Sigma d_2^2}{(n_1 + n_2) - 2} \left( \frac{1}{n_1} + \frac{1}{n_2} \right)}}$$

Where:

$M_1$  = Mean of Part I of the test

$M_2$  = mean of the other part of the test being compared with Part I

$\Sigma d_1^2$  = Sum of the squares of the deviations from  $M_1$

$\Sigma d_2^2$  = Sum of the squares of the deviations from  $M_2$

$n_1$  = Number of samples in Part I of the test

$n_2$  = Number of samples in the other part of the test being compared with Part I

#### Results:

Part I compared with Part II; the "T" value = 11.15, therefore the level of significance ( $18^\circ$  of freedom) = .01 and therefore the difference is statistically significant at the 1% level.

Part I compared with Part III; the "T" value = 12+, therefore the level of significance ( $18^\circ$  of freedom) = .01 and therefore the difference is statistically significant at the 1% level.

Part I compared with Part IV; the "T" value = 0.0705, therefore the level of significance ( $18^\circ$  of freedom) = .5+ and therefore the difference is not statistically significant, for the level is greater than 50%.

Part I compared with Part V; the "T" value = 16+, therefore the level of significance ( $18^\circ$  of freedom) = .01 and therefore the difference is statistically significant at the 1% level.

Part I compared with Part VI; the "T" value = 15+, therefore the level of significance ( $18^\circ$  of freedom) = .01 and therefore the difference is statistically significant at the 1% level.

Conclusions - With the exception of the mineral oil test (Part IV) there is a significant difference between the load at final failure of the dry liners (Part I) and the load



at failure of the liners when soaked in liquid. In these cases the liquid had crept around the edge of the liner and soaked completely through the backing causing the pulpboard to lose its strength and delaminate more readily.

The lack of significance between Part I and Part IV is statistically, a result of the wide deviation between the sample readings. The writer is at a loss to explain the reason for this, and more particularly, the reason why some readings are as high or higher than those recorded in Part I of the test. A partial explanation may lie in the fact that the surface tension of mineral oil is much higher than that of any of the other liquids used, and as a result, less mineral oil penetrates the porous material (pulpboard) of which the backing is composed. This will result in failure loads nearer to those of Part I. In addition, the surface tension of the mineral oil is a force which has to be overcome by the tensile force as well as the force required to delaminate the pulpboard. In that fibers have to be pulled through the surface of the mineral oil to bring about delamination, a force is required to do this, and this additional force has to be added to the original force that is required to delaminate the pulpboard. It is recommended that further investigation on the effects of mineral oil on the tensile strength of paper and board be carried out.



## CHAPTER IV

### CONCLUSIONS

#### INTRODUCTION

When delamination occurs in a screw cap closure, the sealing efficiency of the closure is destroyed. This is a state of affairs the packager wishes to avoid as it is a source of considerable irritation to both retailers and consumers.

The delamination may be caused by one factor or a combination of factors, and when designing the closure, each factor must be considered.

#### ADHESIVE DELAMINATION

An incorrect choice of the adhesive to be used to adhere the backing to the cap or to adhere the facing to the backing will result in a weak bond which will aid delamination when torque forces are applied. The adhesive must be as strong as the other materials used in the cap and liner assembly and must be capable of resisting the forces applied to it when torque is applied to the complete closure. The adhesive must be compatible with the adherends and with the product being packaged. If the product comes in contact with the adhesive it must have no deleterious effects on the strength of the bond, and in addition, the surface chemistry of the adherends must be considered. In this latter respect, strong joints





cannot be made to polar surfaces with non-polar adhesives and vice versa. Outstanding adhesives generally have strong polar groups. Strong acids and alkalies are generally detrimental to an adhesive bond which must also be unaffected by any changes in temperature to which it might be subjected. The adhesive film must be as thin as possible, for de Bruyne has shown that thickness of the adhesive film greatly affects the strength of the bond; the thinner the film, the stronger the bond. Finally, the area of coverage should be specified in that the periphery of the glue line is related to the strength of the bond; the longer the periphery, the stronger the bond, other things being equal.

#### LINER DELAMINATION

An incorrect choice of liner materials will result in delamination, for if the backing and facing are not sufficiently strong to resist the forces applied to them when torque is applied to the complete closure delamination will occur. If the liner facing is incompatible with the product, the latter will either dissolve or seep through the facing and into the backing causing it to delaminate or warp. In connection with product seepage through the facing, manufacturers should avoid stamping identification numbers on the facing, for this will aid product penetration. Facings with a high moisture vapour transmission rate should also be avoided for the same reasons.

Unwaxed liner facings show a greater tendency to stick to the glass finish than waxed facings and this results in excessively high removal torque having to be applied to the closure. This sets up unnecessarily high shear forces in the liner and adhesive which will aid delamination. This can be prevented by using wax on the facings to act as a lubricant.

The backing must be of a material, the laminate strength of which is sufficiently great to resist the shear or tensile forces which may be applied to it. In this respect, it should be pointed out that the most common backing material in screw type closures is pulpboard manufactured on a cylinder machine. Cylinder-board, of several plies, delaminates much more readily than a board of similar thickness manufactured on a Fourdrinier machine. The cost between the two processes would not differ greatly for a board of similar composition throughout, and the Fourdrinier board would resist delamination much more than the cylinder-board. Furthermore, pulpboard is made from unsized mechanical pulp and the addition of size would aid the resistance of the backing to liquids and help prevent delamination.

#### COMPLETE CLOSURE DELAMINATION

##### Metal caps

In these caps, the liner is not generally adhered, and as a result, it may warp under changing temperature conditions or when the humidity increases in the air pocket or when

liquid penetration occurs. In addition, if the liner sticks to the glass finish very little force is required to pull it out of the cap. Better performance would be obtained by adhering it to the cap, although this will increase the cost of the closure.

### Rigid plastic caps

In this type of cap, the liner is adhered and the combination of facing, backing and adhesive must be sufficiently strong to resist the shear forces to which they may be subjected. Furthermore, the plastic from which the cap is made must not be affected by the product, liner or adhesive.

### General delamination factors

Liquid penetration - Liquid penetration is generally brought about by the liquid creeping or being forced between the edge of the liner and the inside of the cap. This will happen if insufficient torque is applied to the closure or if the product remains on the glass finish and the application of torque forces this product up into the gap between the edge of the liner and the inside of the cap. The product then seeps into the backing and considerably weakens its laminate strength.

pH factor - The pH of the product, facing, backing, adhesive and cap must be carefully considered. Strong acidity or alkalinity in one of these parts may cause a deterior-

ation in one of the other parts which may lower its strength and aid delamination.

Hydrodynamic factor - The bond between the facing and backing and between the backing and the cap must be strong enough to resist the continual "sucking" effects that occur as a result of hydrodynamic action, otherwise delamination may occur.

Torque ranges - Excessive torque causes crushing of the backing, cracking of the facing, or causes the facing to stick to the glass finish and these three effects all weaken the liner assembly and aid delamination. In addition, excessive torque will force any liquid on the glass finish up into the gap between the edge of the liner and the inside of the cap and into the backing. Insufficient torque enables the product to seep between the liner facing and the glass finish and then up into the gap between the edge of the liner and the cap and into the backing.

## RECOMMENDATIONS

### Mineral Oil

In view of the wide divergence of the results in Part IV of the tensile test on complete liner and cap assemblies in Chapter III, it is recommended that further research be done on the effects of mineral oil on the properties of paper and board.

### Plastic lined caps

Further investigations should be carried out on the efficiency of vinyl-resin plastisol laminated directly to the inside of metal caps. Other direct laminations of a white rubber-based plastic are also being used in small quantities in the industry and these appear to be particularly suitable on wide-mouthed jars. They are applied to the inside of the cap as a ring around the edge where the glass finish comes into contact with the cap. Provided they are compatible with the product, seepage of the product will not affect them for it cannot occur as the liner ring is a homogeneous mass. Hydrodynamic action will not affect it, nor will pumping or sweating caused by changes in temperature for there is not an air pocket in existence. It appears to be sufficiently resilient to conform well to the glass finish and adsorb a certain amount of torque forces. Its adhesive characteristics are such that a strong bond is formed between it and the metal screw cap.

Both the vinyl-resin plastisol and the rubber-based plastic used as direct laminations have a further advantage in that they are produced from raw materials obtainable in the United States and no reliance is therefore required on high cost foreign sources of supply, as for example, occurs when composition cork is employed. This type of liner is gradually finding wider use in bottle closures and many wider applications are envisaged for the future.

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