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ABSTRACT

THE EFFECT OF ADHESION VARIABLES IN THE BONDING OF CORRUGATED FIBERBOARD

by James Edward Shottafer

This investigation was conducted to examine the effects of selected factors of adhesion on the bonding of corrugated fiberboard, and the interaction of these factors with one another. The variables selected for study were: adhesive formulation, adhesive spread, bonding temperature, bonding pressure, bonding press time, and paperboard moisture content. The factor of press time could not be sufficiently reduced to simulate the very short dwell times characteristic of the conversion of corrugated fiberboard, but was investigated as a source of related information. The other variables, constituting the coincident study of five factors, were incorporated in the investigation at levels such that significant response was reasonably certain. It was thought that the detection of significant interaction effects could be best assured by the inclusion of main factors at potentially significant levels.

A series of preliminary investigations were conducted to establish a method of preparing and evaluating a single adhesive bond similar to that found in typical A-flute corrugated board. Exploratory studies were also undertaken as a qualitative examination of the nature of the typical bond structure. The test method developed provided for the loading of the experimental bond to failure in nominal shear, in the plane of the bond and parallel to the flute.

Employing a single combination of typical liner, medium, and

starch adhesive, specimens were prepared at the designated levels of the variables specified, and tested by the method developed for the conduct of the study. The test data were evaluated by conventional analysis of variance statistical techniques, complemented by qualitative and quantitative examination of the results.

The evaluation of all main factors denoted significant effects, as did their response in interaction with one another, with the following exceptions: the interaction between adhesive spread and formulation, and the interaction between adhesive spread, formulation, bonding pressure, and moisture content of the paperboard materials. The adhesive bond produced by experimental methods appeared similar in structure and general characteristics to the typical bond in converted corrugated fiberboard.

Based on the results of this research, it was concluded that the shear strength of the adhesive bond typical of corrugated fiberboard will be significantly affected by variation in the principal factors studied as inherent to it. The interaction of these variables is significant, and to maintain satisfactory strength in the bond, adjustment of the level of one factor must always be made with regard to the respective levels of all others. Under the conditions prevalent in the conversion process, the bond tends to form without a distinct interface, with the fibers of the adherends tending to mingle and form a contexture at the bond, with limited interpenetration of the adherends. Adhesion results principally from fiber-starch-fiber bonds with some direct fiber-to-fiber bonding, but the relative contribution of each to total bond strength is unknown. The raw starch component of the adhesive is probably the principal

source of total bond strength, but the cooked starch portion, which functions as a carrier, appears to contribute to the bond. The level of moisture present at the interface is critical, as are the rate and extent of its removal under pressure. The starch mixture definitely appears to function as an adhesive system, rather than a single bonding agent.

Areas recommended for further research are the possible evaluation of corrugated fiberboard adhesive bonds by shear loading on the interface, and the application of systems analysis and feedback control to the conversion process.

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IN THE BONDING OF
CORRUGATED FIBERBOARD

By

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TABLE OF CONTENTS

	Page
ACKNOWLEDGMENTS	ii
LIST OF TABLES	v
LIST OF ILLUSTRATIONS.	vi
INTRODUCTION	1
The manufacture of corrugated fiberboard.	3
Review of the literature.	9
Purpose and scope of the study.	15
DEVELOPMENT OF A TESTING TECHNIQUE	18
Review of testing methods and related literature.	18
Preliminary considerations in evaluating the adhesive bond	21
Observed failure in corrugated containers	25
Development and evaluation of a test technique.	26
Preliminary tests	33
Qualitative examination of the adhesive bond.	41
EXPERIMENTAL PROCEDURE	45
Selection of materials.	45
Selection of experimental variables	46
Material selection and designation	51
Sample preparation.	53
Testing procedure	57
DISCUSSION AND ANALYSIS OF RESULTS	61
Effect of adhesive bond formation factors	61
Effect of adhesive bond formation factor interaction. . .	77
General observations during sample evaluation	87

Table of Contents. - Continued.

	Page
Analysis of the adhesive bonding mechanism in corrugated fiberboard	89
Recommendations for further research.	93
CONCLUSIONS	98
LITERATURE CITED	101
APPENDIX A	105
APPENDIX B	111
APPENDIX C	130
APPENDIX D	136
APPENDIX E	140

LIST OF TABLES

Table		Page
1	Average breaking load and percent paper failure in shear specimens bonded in the study of five adhesive bond formation factors	62
2	Summary analysis of variance of adhesive bond breaking loads in the study of five bond formation factors	63
3	Results of tests of shear specimens to determine liner tab width	114
4	Results of tests of shear specimens to determine adhesive bond length.	116
5	Results of sample misalignment on the breaking strength of shear specimens	120
6	Comparison of the tensile strength of liner material as determined by two test methods	122
7	Results of tests of adhesive bond strength by shear and tensile test methods	125
8	Strength of discontinuous adhesive bonds in specimens tested in shear and tension	128
9	Breaking load and percent paper failure for samples bonded at six pounds per inch of glue line with standard spread .	141
10	Breaking load and percent paper failure for samples bonded at twelve pounds per inch of glue line with standard spread	142
11	Breaking load and percent paper failure for samples bonded at six pounds per inch of glue line with heavy spread . .	143
12	Breaking load and percent paper failure for samples bonded at twelve pounds per inch of glue line with heavy spread .	144
13	Analysis of variance of adhesive bond breaking loads in the study of five bond formation factors	145
14	Analysis of variance of adhesive bond breaking loads in the study of five bond formation factors (Revised) . . .	146
15	Breaking load in pounds for specimens bonded with single face formulation at various press times	147
16	Breaking load in pounds for specimens bonded with double back formulation at various press times	148

LIST OF ILLUSTRATIONS

Figure		Page
1	Illustration of glue line shear device (front view). . . .	28
2	Illustration of glue line shear device (section side view)	29
3	Illustration of details of glue line shear device with typical specimen	30
4	Partially disassembled glue line shear device for testing of simulated corrugated fiberboard adhesive bond	31
5	Glue line shear test device.	32
6	Fixtures for tensile test of simulated corrugated fiberboard adhesive bond	34
7	Adhesive bond tensile strength specimen mounted in fixtures for testing	35
8	Partially disassembled press device used in preparing simulated corrugated fiberboard adhesive bonds for testing	37
9	Press device assembled and ready for use	38
10	Typical simulated corrugated adhesive bond test specimens	40
11	Photomicrograph of double back adhesive bond in corrugated fiberboard (40x)	42
12	Photomicrograph of single face adhesive bond in corrugated fiberboard (40x)	43
13	Typical modes of failure in adhesive bond shear specimens	60
14	Response of shear specimens bonded with single face and double back adhesive formulations to variation in press time	64
15	Hypothetical adhesive strength response surface for three bond formation variables in single facer adhesion . . .	96

INTRODUCTION

This investigation was undertaken to evaluate the influence of some of the factors of adhesion present in the conversion of corrugated paperboard, and the interaction, or mutual response of these variables. While the basic process involved is the adhesive bonding of paper, the nature of both the formation and physical character of the corrugated glue bond are in some respects unique, as compared to other adhesion phenomena.

The manufacture of paper is a combined science and art which has been notable for its very long history of economic and technological prominence. No aspect of the area of forest utilization has received the degree of attention, from the standpoint of research, that has been devoted to subjects related to the pulp and paper industry. The development of paper for purely structural uses as a primary material element in a fabricated construction, however, has occurred predominantly in the last few decades. A special emphasis on research in this area occurred during the World War II period when aircraft requirements for strong, light weight structures encouraged the development of cellular paper core materials, and when the necessity of high quality shipping containers was critical. The demands that such uses imposed on the physical and environmental integrity of structural papers stimulated investigation of both materials and composite products. Sharing this increased technological attention was corrugated fiberboard, which had been widely used as a material for shipping containers for some time. The use of corrugated paper structures has continued to increase and become more diverse to the present. The container industry continues to enlarge,

and corrugated paper laminates have appeared as core materials on a large scale in the aircraft and furniture industries, and are currently finding application on an increasing scale in the building industry.

The principal investigative study in regard to corrugated fiberboard for packaging has been in three general areas, where the immediate need for technological or economic product development has existed. The paper elements have received considerable scientific attention, especially in regard to research into fiber character, and in the development of new coating and impregnating materials to enhance the physical strength, and environmental and biological characteristics of paperboard. In the area of the fabrication of paper laminates, many process improvements were made possible by the relatively rapid development of improved synthetic resin and starch-based adhesives, and by general advances in the field of adhesives technology. Significant attention has been devoted to the problem of testing structures made of corrugated fiberboard, especially shipping containers and the internal supporting elements used in them. The degree of success with which the various methods of evaluation may be related to the fabrication process has varied considerably. The results of some physical tests show excellent correlation with variation in materials or methods, while others appear to exhibit little or no relationship. The technology of manufacture in the fabrication of corrugated fiberboard has not received the investigative attention devoted to raw materials and finished products until very recently. What research has been undertaken by the industry itself has been of a highly applied nature, often limited to specific machines or materials.

The manufacture of corrugated fiberboard. -- In order to provide a basis for the discussion of the purpose and scope of this study, some consideration of the corrugated fiberboard bonding process, and the bonding of paper in general, is in order. The conversion of corrugated board involves the bonding of at least two or more types of adherends, using an adhesive material as a bonding agent. The conditions of bonding become critical when the limitations of one or more of the constituents related to the specific adhesion phenomenon are involved. Typical corrugated board is basically a three-element composite structure, with the center element, or medium, usually a corrugated shaped paper of 0.009 to 0.012-in. straw or semichemical stock. This is bonded on both sides to a 0.010 to 0.030-in. Kraft paperboard, which acts as facing material. Multiples of two and three composites may be combined to form "double wall" and "triple wall" board. A great many variations in the physical character of corrugated board are possible, depending on geometrical properties, such as flute size and shape, and such material characteristics as paper finish, basic weight, and fiber constituents.

In the basic corrugated board conversion process, the medium and one liner board are threaded from horizontal roll stands through a series of steaming devices and preheaters and into the single facer machine. Here the medium is formed by fluted corrugating rolls into its characteristic shape, and adhesive is applied by **transfer** rolls to the tips of the flutes in the medium. Almost simultaneously, the liner board which has been threaded into the single facer is bonded to the medium in the nip of a heated pressure roll, thus forming single face corrugated board. A considerable variation in critical conditions may be encountered in this fundamental forming and bonding process.

The steaming and preheater rolls are employed to produce a plasticizing effect on the medium so it will deform more readily in the corrugating rolls, and bring both medium and liner board up to a satisfactory temperature for bonding. Commonly the entire heating system, including preheaters, pressure and corrugating rolls, and double backer drying plates are heated by steam pressure to temperatures in excess of 300°F. The recommended operating temperature for the equipment surfaces, from the standpoint of the bonding process is about 340°F., but this may vary considerably with lineal machine speed. Little is known of the moisture content of the board when it enters the machine, but the opinion of most operators seems to be that it is probably in the neighborhood of 6-10 percent, based on oven-dry weight. The bonding pressure between the nip of the corrugating and pressure rolls, is usually 75-100 lbs. per lineal inch, but this is also quite variable, depending upon machine speed, type of adhesive, and other formation factors. Sodium silicate adhesives were widely used in the past, but in recent years starch adhesives have come into almost universal use. In order to react the two-phase starch mixture which characterizes the adhesive, the heating system must produce a temperature of 140-150°F. at the glue line, despite a dwell time that may theoretically be as short as 0.002 sec. (based on C-flute board run at 500 ft. per minute). The adhesive is applied from a heated storage pan with an applicator roll and gels almost instantly with the application of sufficient heat.

Subsequent to the formation of the single face material, it is conveyed by an overhead belt and roller system to the double backer machine, where the second liner board is applied. The basic bonding operation here is similar to that performed on the single facer, with a few notable exceptions. The bonding pressure is quite low, (commonly about

10 lbs. per lineal inch), since pressures such as those used on the single facer would crush the flutes. The formulation of the starch adhesive is slightly different for the double backer operation, being more viscous, with a higher content of cooked starch. The use of steam is limited, since the single face element will delaminate with excessive moisture, but the liner board may still be heated.

After the second bonding operation the finished board, held together by the initial "tack" of the adhesive, is carried between heated plates mounted on cotton belts, which apply heat and a slight pressure to the material and complete the cure of the adhesive. The board is then processed on cutting devices, such as a printer-slotter machine, into corrugated containers or cut flat sheets of finished board. Again, this description is of a general nature, and a more complete analysis of the bonding process, per se, is presented subsequently.

While it is not intended to completely discuss the physico-chemical character of the adhesive bond in corrugated fiberboard here, since a study of significant magnitude might be devoted to this subject alone, some analysis of it and the related literature is appropriate. In the light of microscopic examination, and consideration of the nature of the fabrication process, at least two distinct types of bonding are probably involved, more distinguishable by their physical rather than chemical character.

Without question, a three-element bond exists between the two adherends and the adhesive, the starch molecule acting as a bridge between the cellulose molecules of adjacent fibers. This is what might be considered the conventional situation in regard to an adhesion phenomenon.

The starch molecule is particularly well suited to the bonding of paper, since it not only has the necessary available chemical side groupings for satisfactory cross linking, but is in fact almost identical to the basic cellulose unit structure. Chemically, starch is similar to cellulose, the difference in the substances lying in the geometrical configuration of the molecule. Starch is characterized by α glucosidic linkages joining the unit structures, and cellulose by stereospecific β glucosidic linkages, which tend to form long, relatively stiff polymeric chains (2) (18) (24).

Unlike the typical adhesive bond, however, a laminate of paper may be formed by direct inter-fiber bonding of the adherends themselves, under proper conditions of contact. Accessible hydroxyl and hydrogen groups will readily form cross-linkages, bonding adjacent molecules, and consequently individual fibers that are in close proximity with one another. The extent to which this fiber bonding process takes place in the formation of the original paper mat is dependent on a host of physico-chemical factors, and these phenomena and the extent of their occurrence determine in large part the physical strength of the finished paper. It is fairly well established that the conditions and extent of heating, fiber strength and geometry, the affinity of the pulp for water, hemicellulose content of the pulp, and many other factors are significant in paper formation (11) (25) (37) (44) (13). In terms of the finished paper, however, Swanson (48) describes the strength of paper as depending in general, on the length and strength of the particular fiber, and the strength, number, and distribution of fiber-to-fiber bonds, regardless of the way the correct level of these characteristics is achieved.

The extent to which this direct fiber-to-fiber bonding may occur in the adhesive bonding of corrugated fiberboard is unknown on a quantitative basis. It is probable that some fiber bonding occurs between the medium and liner boards, but there is a notable lack of published comment in regard to this phenomenon, which is undoubtedly involved to some extent in single face bonding and may be present in the adhesion of the double back liner.

At the moment that liner and medium are joined on the single facer both have been heated and moistened, probably to a moisture content of 8-10 percent, a condition described in the trade as plasticizing the material (36) (49) (50) (51). This added moisture will certainly tend to dissociate the hydrogen bonds between adjacent fibers, especially where a bond between two secondary hydroxyl groups is involved, thus rendering molecular surface areas available for re-bonding under the proper conditions. A mechanical factor is introduced when the medium, as the adhesive is applied to the tips of the flutes, passes under the fingers which guide the material against the corrugating roll. If these fingers have been relieved to allow the material to move slightly away from the roll at the base of the finger, the surface fibers, on the convex surface of the flute, will tend to lift away from the surface. These minute fiber ends will then be more likely to mingle physically with the fibers of the liner material at contact, so that bonding can occur where the fibers are intimately in contact. The same effect may occur on the convex surface where the liner turns on guide rolls, or on the pressure roll before contact in the nip. While the contribution of this factor to the degree of inter-fiber bonding is probably very small in the quantitative sense, it does tend to encourage the phenomenon. As the

medium and liner enter the nip between the pressure and corrugating rolls, where the adhesive bonding of the materials occurs, they are subjected to 75 to 150 lbs. per lineal inch of pressure, or about 2400 to 5000 psi (32) (42) (50) (51). This pressure literally inbeds the medium material in the surface of the single face liner board, as shown by Eoller et. al. (5), and causes an indistinct interface to be formed.

The moisture content of paper during the basic forming processes of its manufacture varies considerably, but most sources agree that fiber bonding occurs below 60-75 percent, as the paper is dried to a final moisture content of 6-7 percent. Fiber bonding has been found to coincide with sheet shrinkage, in that the sheet strength develops as the shrinkage of the mat progresses. If the paper is re-wet, a definite hysteresis effect may be noted, as in all wood-based substances, since new hydrogen bonds are created during the original drying process that are difficult to break (12) (39). The material will thus never achieve the original swelling properties, and degree of inter-fiber bonding present in formation of the basic paper mat. A very similar effect may be noted in the bonding of non-homogenous layers of multi-ply board made on a cylinder type paper machine. The inter-ply bond is never as strong as the bonding in a homogenous sheet made on a Fourdrinier machine (11) (23) (48). The various types of fiber bonding common to paper and evidence of the forces that form and support the fiber network of the paper mat have been illustrated by Simmonds and Chidester (44).

Since the more important conditions required to permit fiber bonding appear to be present in the adhesive bonding process at the single facer, it is reasonable to conclude that some degree of bonding directly

between adjacent fibers must occur. Water is provided to the material by the steam showers and from the adhesive, the fibers are forced into intimate contact, and then drying occurs as moisture migrates away from the glue line and into the sheet and as the single face board is dried in subsequent operations.

In the double backer gluing operation, moisture is introduced, but to a lesser extent, and pressures are very low, probably about 10 lbs. per lineal inch. While some fiber bonding may occur, it is probably not significant (22) (42).

The adhesive bonding of corrugated fiberboard thus involves at least two physico-chemical phenomena: the conventional bond, where a starch adhesive serves as a bridge between the adherends and bonds in turn to each, and a direct adherend-to-adherend bond, where the adhesive serves only to enhance the molecular contact between the specific surfaces of the adherends (48). The quantitative role played by the two phenomena is difficult to predict, since the degree of fiber bonding is unknown, but the indistinct interface caused by inter-penetration of the adherends appears typical of the structure. No doubt the adhesive bond, rather than the fiber bond is the chief source of inter-laminar strength between adherends, especially in the double back bond where physical contact is limited.

Review of the literature. -- Literature of a definitive nature is very limited in relation to the bonding of corrugated fiberboard - much of it is highly qualitative in nature, and orientated to a very specific process or system of machines. A considerable amount of the available published material is also connected with the journals of

specific firms, or is based on research of a proprietary nature.

A large part of the published comment on the conversion of corrugated boxboard is based on the presumed use of silicate type adhesives, which have been largely replaced by starch formulations, in recent years. De Bruyne and Houwink (15) discuss the process by which corrugated board is made (based on silicate adhesives) and describe the various engineering characteristics of good boxboard. A number of studies regarding the effect of certain factors involved in the bonding process are reviewed and commented on. De Bruyne notes the use of pressures of about 150 psi at the single facer and about 15 psi at the double backer. (Note: presuming an 80-in. width of corrugating roll, this would be equivalent to about 15 lbs. per lineal inch). A rather complete review of work related to the silicate adhesive per se is also presented.

McCready and Katz (31) investigated the engineering characteristics of boxboard at considerable length, but included only a limited number of actual formation variables in their study. They considered differences between adhesives, adhesive spread, and the effect of extenders on silicate glue. It was noted that using a pin adhesion test as a criterion, the material fabricated with starch adhesives at a normal spread evidenced greater bonding strength than comparable silicate-made-board. At heavy spread levels, however, the silicate bonds were stronger than those made with starch adhesives, possibly due to the greater tendency to penetration of the medium noted in the case of the silicate. Other critical variables were controlled at a single level considered normal for actual fabrication process. This work was most notable as a complete base-line study of the engineering properties of corrugated paperboard made under a restricted set of conditions, rather than a comprehensive

analysis of the effect of adhesion variables.

More recent literature provides discussion based on starch adhesive formulations, but most deals with the question of bonding variables only in a qualitative manner. Delmonte (16), Werner (50), Yezek (53), and Sherman (43) all present general discussions of the bonding phenomena, without analyzing the effects of specific variables or the levels at which they may be present.

Goettsch (19) considers the influence of machine and applicator roll speeds as an interaction, related specifically to their effect on glue line quality by the intensity and character of the glue spread. It was noted that at the single facer the amount of adhesive transfer did not seem to vary significantly with relative changes in machine and applicator roll speeds. At the double backer, however, the ratio of applicator roll speed to machine speed appeared critical to adhesive transfer. This may indicate a time-temperature-pressure interaction at the single facer machine, or the need for heavier spreads to achieve strength in the bond at slower speeds because of an interaction between moisture content redistribution and glue line temperature. It should be noted, however, that the effect of speed was assumed critical only in that the proper amount of adhesive be delivered to the flute tips. The actual strength of adhesive bonds was not considered.

A discussion of adhesion theory of the double backer was undertaken by Harrison (22), again on a primarily qualitative basis. As in Goettsch (19) and various Stein Hall Company technical publications (3) (6), the use of two formulations of starch glue for single face and double backer applications was recommended in this publication. It was noted that excessive spread creates very wide "shoulders" between flute

tip and liner, and differential drying of the board due to these areas of adhesive accumulation will cause a characteristic defect known as "washboarding". In the Harrison study moisture content and roll weight (pressure) were considered critical in their relation to adhesive spread, but the discussion is entirely qualitative with no specific values for the variable levels included.

A rather comprehensive study was made by Koenig (28) of common processing defects at the single facer machine. A number of bonding factors are cited in relation to board and container defects, including moisture content, finger settings, equipment condition, web tension and various basic properties of the adhesive. Unfortunately, this discussion is based primarily on specific machine characteristics, i.e.: stripper adjustment, roll settings, etc., rather than actual variable control. The variables are also treated only in the sense of attributes such as "too much" and "too little", rather than specifically identified factor levels. This investigation could be of considerable aid to the operator or engineering manager of a corrugated board conversion process, but is of little use in a critical analysis.

In a series of tests of the effect of nip pressure on the flat crush quality of corrugated fiberboard, Max (36) also used a single face formulation of starch adhesive. The board studied was A-flute material run at a machine speed of 100 ft. per minute, with nip pressures varying from 7.8 to 158 lbs. per lineal inch. Little change was noted in the caliper of board beyond 30 lbs. per lineal inch, and pressures above 60 lbs. per lineal inch apparently had little effect on flat crush tests. It should be noted that the material was apparently bonding satisfactorily at all nip pressure levels. A second but very important point in the

comments on this study, was the consensus of the panel discussion subsequent to the presentation of the paper. Apparently those persons present considered 9 percent moisture content as not only the most common for the paperboard at bonding, but also viewed it as an optimum level on the basis of experience.

A report of a TAPPI (Technical Association of Pulp and Paper Industries) committee industry survey indicates average adhesive spreads of about 1.3 lbs. per thousand square feet of material, with a range of 0.98 to 1.75 lbs. per thousand square feet of finished board, as an average for 61 firms responding (42).

Sherman (43), in a study of the effect of machine speed, noted no significant change in pin adhesion test values for materials run at speeds from 100 to 600 ft. per minute, so long as the proper ratio of adhesive roll speed to machine speed was maintained. This implies a lack of interaction between spread and other adhesion variables. A starch adhesive was used, with a spread of 2.0 to 2.5 lbs. per thousand square feet. A study by Wilson (51) also refers to an average nip pressure of 75 to 150 lbs. per inch of glue line, where flute contour was the primary subject of the paper.

A very significant series of experiments was conducted by McKee (32) at the Institute of Paper Chemistry at Appleton, Wisconsin. The variables studied were primarily operational rather than material, although four types of medium material were used with a 42 lb. Kraft linerboard. The materials were fabricated into A-flute single face board, using a starch adhesive in the 140-144°F. gel point range. Samples of the board, fabricated at different levels of machine speed, web tension, nip pressure, and steam pressure, were subjected to various conventional

tests. The following conclusions were drawn from the results, based upon an evaluation of bond strength by the pin adhesion test (21):

1. Speed, varying from 150 to 1000 ft. per minute, had no significant effect on bond strength below 600 ft. per minute. Variation beyond this speed was considered to be the result of the time-temperature interaction effect on the gel point of the adhesive.
2. There was no evidence of a significant effect of nip pressure on bond strength, from 220 to 865 lbs. per lineal inch pressure.
3. The effect of steam pressure did not appear significant in relation to bond strength.
4. There was no significant effect evidenced by varying the amount of web tension.

Variation in the operating levels of these factors, in addition, produced no significant response in the caliper of finished board. The moisture content of the material at the time of fabrication varied from 6.9 - 8.4 percent. The study utilized a laboratory size single facer machine, resembling its conventional counterpart except in the width of the machine, which was 12-in. across the rolls. McKee's investigation is of considerable interest, especially from the standpoint of the apparent response of the primary variables involved, and the levels at which these factors were included. Together with other published information, it aided in establishing criteria for the selection of the factors and factor levels incorporated in the study at hand. It should be noted that on the basis of McKee's graphical analysis, some of the various physical characteristics of the experimental board were significantly affected

by variations in the bonding factors studied, despite the fact that the bond strength, per se, did not appear to be.

In summary, it may be noted that in the available literature, the emphasis of experimental investigation of the adhesion of corrugated fiberboard has been confined, at best, to the evaluation of main factor effects. Limited quantitative information is provided regarding the influence of these main factors, and none regarding their interaction.

Purpose and scope of the study: -- The objective of this investigation was to determine the primary and interaction effects of certain selected factors involved in the adhesive bonding of corrugated fiberboard. The study was made, primarily, from the standpoint of those variables related to the formation of the bond, rather than the nature of the adherends or the adhesive.

Defining the scope of the study required the selection of factors for investigation based on time restrictions and the existence of laboratory facilities, and a series of preliminary investigations necessary to the conduct of the principal research.

Aside from information that might be gained from the preliminary work associated with the development of a test technique, it was decided to limit the study to the consideration of six main variables, treated in two combinations. These were: the weight of applied adhesive, the type of adhesive formulation, bonding temperature, bonding pressure, press time, and the moisture content of the paper adherends. The criteria for the selection of the factors to be included in the investigation, and the levels of those factors, are explained in detail under the description

of the experimental procedure. These decisions were based on the review of the pertinent literature, and the results of the preliminary tests conducted prior to the principal phase of the study. The levels at which the variables were incorporated in the investigation were selected to detect the effect of factor extremes, or to indicate the influence of those conditions thought most representative of the corrugated conversion process. The variables selected also reflect the previously stated emphasis in this study on operational rather than material adhesion factors.

In order to implement the conduct of this investigation of the factors inherent in the adhesion of corrugated fiberboard, it was necessary to select methods of preparing and evaluating representative adhesive bonds to serve as criteria. After careful consideration of existing laboratory techniques of producing corrugated adhesive bonds, and testing the quality of these bonds, it was concluded that other methods of specimen fabrication and evaluation were desirable. It was also decided that a limited investigation of the nature of the typical bond found in corrugated board would aid in the interpretation of experimental results, and might provide additional pertinent information. Preliminary studies to establish an evaluation method and provide qualitative information regarding the adhesive bond were therefore conducted in support of the investigation of adhesion variables.

The conduct of this investigation may thus be summarized as resolving itself into the following facets:

1. The principal study of the effects of factors related to the adhesion of corrugated fiberboard.
2. Preliminary investigations conducted prior to the principal study to (a) establish acceptable methods of specimen

preparation and evaluation, and (b) provide qualitative information as to the character of the adhesive bond, through microscopic examination, and limited evaluation of the role of direct fiber-to-fiber bonding in the subject adhesion phenomenon.

The development of the evaluation method, and the qualitative studies of the adhesive bond were, of necessity, considered first.

DEVELOPMENT OF A TESTING TECHNIQUE

A satisfactory method of evaluating bond quality is required in the critical examination of the factors involved in the adhesive bonding of corrugated fiberboard. The common approach to this type of problem is to devise a specimen that is subjected to the characteristic loading imposed on the material in service, and evaluate the bond in terms of pounds for a geometrically defined specimen, or psi at fracture. Alternative methods involve loading the bond in some convenient manner, without regard to mode of failure in service; or purely qualitative tests based on observation. The latter are less desirable than a derived test, since they often evaluate properties not related to the failure of the bond in service, or being dependent on qualitative properties, have no definitive basis as a criterion.

Review of testing methods and related literature. -- If those techniques based on observation are discounted as insufficiently reliable for use in a critical study, several possible methods of evaluation may be considered that have served in previous investigations as criteria of bond strength, and appear in the literature specifically related to the testing of corrugated paperboard. The pin adhesion test has been in use for some time and has served as an evaluation basis for several studies (21) (31) (32) (43). It is the standard for corrugated bond strength evaluation currently proposed to the American Society for Testing Materials, (ASTM) (1). The basis of this test is to insert steel rods through the flutes of the material, and then pull alternate rods in opposing directions, with the plane of the specimen normal to the imposed tensile load. The

widespread use of this test has grown despite certain distinct inconsistencies in its behavior, which are subsequently described.

Another approach to the question of evaluation has been the use of lap joint shear specimens. These were used by Carlson (8), and in several other investigations examining the general engineering characteristics of corrugated fiberboard (5) (9) (41). Specimens of this general type are very widely used for evaluation of adhesive bonds where wood, metals or plastics serve as the adherends.

Peeling tests have been used in measuring the quality of glue bonds in paper products (1) (15), as well as in evaluations of the adhesive properties of thin films, paints and tape. An application of a peel test was made by Broughton, Chu, and Kaswell (7), by pulling the liner and medium in cleavage, the load being applied to a series of flutes in succession. The quality of the bond was judged by the amount of energy required to effect separation, and by quantitative examination of the load-deformation curves showing the progressive rupture of the bond at successive flutes. This paper (7) not only advocates the use of an energy-based peel test, but discusses the advantages of a testing device based on a constant rate of deformation rather than a pendulum counterweight. (The pendulum-type tensile testing machine is very widely used in the paper industry, but incorporates neither a constant rate of loading nor a constant rate of elongation.)

A number of comments are pertinent regarding these test techniques from a theoretical standpoint (15) (52). On the basis of existing studies devoted to the engineering properties of corrugated fiberboard, (8) (27) (31), we may be assured that the quality of adhesive bonding is a significant factor in establishing the properties of the composite material.

It is interesting to note, however, that there is little evidence of direct correlation between these tests of the adhesive bond and the strength characteristics of the finished board or container (5) (7) (15) (31). This indicates that the bond tends to form in only two modes of existence, i.e., satisfactory and unsatisfactory, or that the tests are index estimates which do not reflect the forces encountered in the finished material. The first alternative is not technologically sound from the standpoint of the general theory of adhesion, but the second is, however, quite possible. It should be noted that all of the evaluation methods described above, together with their more common modifications, test several bonds, or actually measure an effective "average". The peel test proposed by Broughton et. al., (7) reveals the strength of individual bonds, but still requires their composite evaluation, and even refers to the successive levels of failure as one criterion of uniform bonding. The lap joint specimen is designed to test resistance to shearing forces, but there exists ample evidence that the stresses that are critical in establishing the level of failure in the specimen are not shear, but bending or tension at the lap edge ("tearing" stresses) (15) (52). The various peel tests and the pin adhesion test evaluate the tensile strength of the bond, and are very similar to cleavage tests in terms of the manner of loading. Such cleavage forces have been demonstrated to produce high level tensile stresses at the point of separation between the adherends, and these tensile stresses are dependent, in part, on the flexural strength of the adherends (4) (15) (52). All of these test specimens exhibit considerable inherent variability, despite the attempt to "average out" local defects by testing the bonds in multiples. The specimens involved in the pin adhesion and peel test present no

particular problems in regard to preparation, but the shear or lap test specimens, which require backing by another material such as wood, demand lengthy fabrication and experimental procedures. All the types of specimens considered to date have indicated an apparent insensitivity to many of the operational factors which are apparently critical to satisfactory bond formation.

Due to the evident inadequacies in the principal existing evaluation techniques, it was decided that a more responsive and precise method of evaluating the strength of the adhesive bond in corrugated fiberboard was desirable, and effort was directed to establish such a satisfactory criterion.

Preliminary considerations in evaluating the adhesive bond. -- The contribution of an adequate adhesive bond to the structural strength of corrugated fiberboard has been convincingly confirmed by a number of investigators who have concerned themselves with this relationship. McCready and Katz (31) studied the effect of adhesive bond variables on the structural character of corrugated board, and in turn used the elastic properties of the material to predict the compressive strength of containers. Carlson (8) and Kellicutt and Landt (27) all relate the rigidity of corrugated fiberboard to the elastic character of the component materials, and identify the strength quality of board with adequate bond formation. Carlson demonstrated the tendency of square corrugated tubes to form a recurring wave along the flutes when loaded in compression in the same direction, with the length of the wave between 12 and 16-in., depending on the component materials. All of these studies established a definite relationship between the flexural properties of corrugated board and

fabricated containers, and noted the effect of score lines, poor flute formation, and such adhesive defects as insufficient spread and finger marks, on container compressive strength.

McNair (33) used electrical resistance strain gages to investigate progressive failure of containers under dynamic loads. The same compound curves, or reverse "dishing" effect was noted in compressive impact loading that others (8) (9) (10) (27) reported in regard to static compression of containers. This study suggests that flexural failure, which actually occurs long before evidence of gross failure is visible, is directly dependent upon the relative inability of corrugated fiberboard to assume a compound surface, i.e., a surface curved in two planes perpendicular to one another. This implies the existence of considerable shear stress in the plane of the sheet, and coincides with the importance of shear deformation in the flexure of corrugated fiberboard as noted in the results of the study by Carlson (9). Schupp and Boller (41), and Boller, Lander and Morehouse (5) show a more direct relationship between adequate bond quality and structural compressive strength. These studies call attention to the supporting character of the small truss-like structures formed between either side of the flute and the inner surface of the liner board. McCready (31) also noted the strengthening effect of the adhesive deposited on the surface of the liner. All the above investigations observed that compression failures in containers invariably started at points of weakness in the adhesive bond. Schupp and Boller (41) observed that paper and adhesive variations had little effect on bond structure, as such. Only where the "shoulders" failed to form at the bond, was the structure significantly deficient in strength. These "shoulders" may be noted in the microphotographs of

bonds shown in Figure 11 and Figure 12 in the section devoted to qualitative examination of the adhesive bond.

The presence of shear forces at the bond interface is not unexpected, considering the tendency of the material to buckle in flexure when the container is compressed, the normal eccentricities of the structure, and the anisotropic character of the board due to its internal geometry. In considering the action of a container side panel under column loading from a theoretical standpoint, it becomes evident that shear forces must be present at the interface between liner and medium.

If transverse and anticlastic bending are disregarded for purposes of simplicity, the panel of corrugated board under end load in the flute direction may be assumed to be an aggregate of narrow adjacent strips, each behaving as a column. This assumption is a common device resorted to in the analysis of plywood and other laminated structures (37). In such a section of corrugated fiberboard the geometry will produce an effect very similar to that evident in a composite I-beam used as a column. Before gross bowing, while the column is in a stable state, it may be assumed deformation or strain is the same in both medium and liner, with the adhesive acting as a rigid bond between them. There will almost invariably be some bending action, however, due to minor imperfections and eccentricities in the nominally flat surface.

If there were no glue lines, or if there were a significant area without bonding, the liners would probably tend to bow normally to the medium, provided the loading was absolutely symmetrical. This would indicate a tensile stress on the glue lines, which normally restrain

the liner from bowing in such a fashion. There is, however, shear at the nominal interface due to invariable flexure effects, as noted above.

The more usual conditions of fixity caused by container score lines tend to make the side panel of a box behave in a manner more similar to a pin-ended column, with a limited amount of restraint. Such a condition will commonly induce flexure, and produce the wave effect described previously. If the analogy to a beam in flexure, especially an I-beam, is again made, it is assured that significant shearing stresses exist in the plane of the panel, indicating why often beam structures are used to test adhesive joints in shear (15). The close relationship noted between the EI value of corrugated board, and the compressive strength of containers noted in prior investigations (8) (9) (31) tends to support the conclusion that bending stresses are both present and critical, with the inherent shear stress present at the assumed rigid transition between liner and medium.

It is thus reasonable to conclude that the primary forces on the adhesive bond are shear forces, with some tension effects where finger marks, or other discontinuities of the bond are present. The board in end-wise compression will fail by buckling in flexure. In some instances this buckling failure may be accompanied by "rippling" of the liner, indicating some normal tensile force must have been present, but the flexural failure invariably implies shearing stress at the bond. On the basis of the forces encountered in service, a test method that imposes shearing loads at the glue line would therefore appear most logical.

Containers often fail from loads other than compression, especially puncture and tearing failures at the score lines or closures. These

failures would seem, however, to be a function of the nature of the materials, rather than the structure. At the score line the structure does not exist, and in a puncture failure, deformation of the composite structure remains a bending phenomenon.

Observed failure in corrugated containers. -- In order to further ascertain the nature of structural failure in corrugated containers, a limited field study was made in several locations.

The subjects of the study were limited to conventional regular slotted (RSC) boxes, with contents, and in commercial use at the time of failure. Observations were made at the following places:

1. New York Central Freight Terminal - Utica, New York
2. Federal Post Office - Freeport, L.I., New York
3. Greyhound Bus Station - Lansing, Michigan
4. Wrigley Market - Lansing, Michigan

An attempt was made to include various modes of transportation and contents, although close examination of the containers and their contents was not attempted in such a limited preliminary test. In order to avoid the size effects discussed by Carlson (8) only containers 12-in. or larger in all dimensions were considered, and those boxes showing environmental damage (moisture, etc.) were disregarded.

In summary, 163 shipping containers were considered. Of these, 155 indicated failure in compressive flexure; 150 by buckling and 5 by lifting or rippling of the exterior liner. The remaining 8 cartons, all filled with canned goods exhibited failure by end crushing in compression. Only eight of the cartons examined (all of which had failed by buckling), showed any evidences of tensile forces at the glue line.

Material was taken from these eight containers, and large areas of separation with inadequate adhesive bonds were noted, in addition to fiber distortion indicating the presence of tensile forces. Only one incidence of puncture failure was noted.

This limited examination of container failure in service suggests that failure in flexure, with the acknowledged shear forces at the glue line, imposes the most critical stresses on the adhesive bond. Without doubt the quite common failures due to puncture, flat crushing, and end crushing as a column occur in significant numbers, but these would appear to be primarily dependent on the integrity of the adherends, rather than the adhesive bond.

Development and evaluation of a test technique. -- On the basis of previous studies (5) (7) (15) (26) (31), the limited field study of container damage, and the probable nature of the loads imposed on the nominal bond interface in service, a test technique was designed to incorporate the various features thought necessary to a representative evaluation. It was evident from both logical conjecture and observed failures in corrugated boxes, that the bond is predominantly subjected to shear loading along the flutes, with some possibility of tensile forces normal to the plane of the board.

Two test specimens have been discussed (7) (31), both of which involve loading which is essentially shear. Both lap specimens and reinforced shear specimens have inherent disadvantages which seemed desirable to avoid. It was considered essential that only a single glue line be involved, to avoid the variability that might be encountered with multiple bond samples. The versatility of the specimen, ease of preparation and

testing, and validity in comparison to service conditions of stress imposition were also necessary considerations.

A shear testing technique, based on the combined features of a lap-joint test and the maple-block shear test used to evaluate adhesive joints in wood, was developed after extensive trials of various test device designs. The details of this device are illustrated in Figures 1, 2, and 3, and in the photographs presented as Figure 4 and Figure 5.

As may be noted from these illustrations, the specimen consists of a section of liner material bonded to medium material. The latter is stretched over a triangular steel support, and firmly held with restraint plates and wing screws against the sides of the support. The apex of the support describes a 0.090-in. radius, which duplicates quite closely the radius at the point of contact between the flute tip and liner in A-flute corrugated board. This dimension was arrived at by measurements made on thin sections of production-run A-flute board, and from measurements made by Goff (20) and McCready (31).

The specimen is mounted on the support, and the entire system inserted into precision ways at the back of the test device. The tab of the liner extending below the medium is inserted into the sash section of the device and restrained. As load is applied by a testing machine to the rounded top of the sash, the liner is pulled down away from the medium, producing a force that is essentially shear on adhesive bond which joins the paper elements. Some tensile forces certainly exist in the system, especially at the top of the bond between liner and medium. There must also be some tendency for the medium to distort in the area along the top of the restraint plates, between the plates and the comparatively ridged bond area. The forces imposed on the nominal interface

Notes:

1. Device made by Metals Machining Co., Lansing, Michigan.
2. Critical dimensions: (as measured to 0.0001-in.).
 - a. Line of action of sash must be parallel with no meas. tol. with center line of medium support flute.
 - b. Lateral movement of medium support on stop blocks must not exceed 0.001-in.
 - c. Lower edge of medium support triangular element and grip section of sash must meet with no meas. tol.
 - d. Ways in device for insertion of medium support must permit removal and return with 0.001-in. lateral tol.
3. Not to scale. Terms sample and specimen used synonymously.

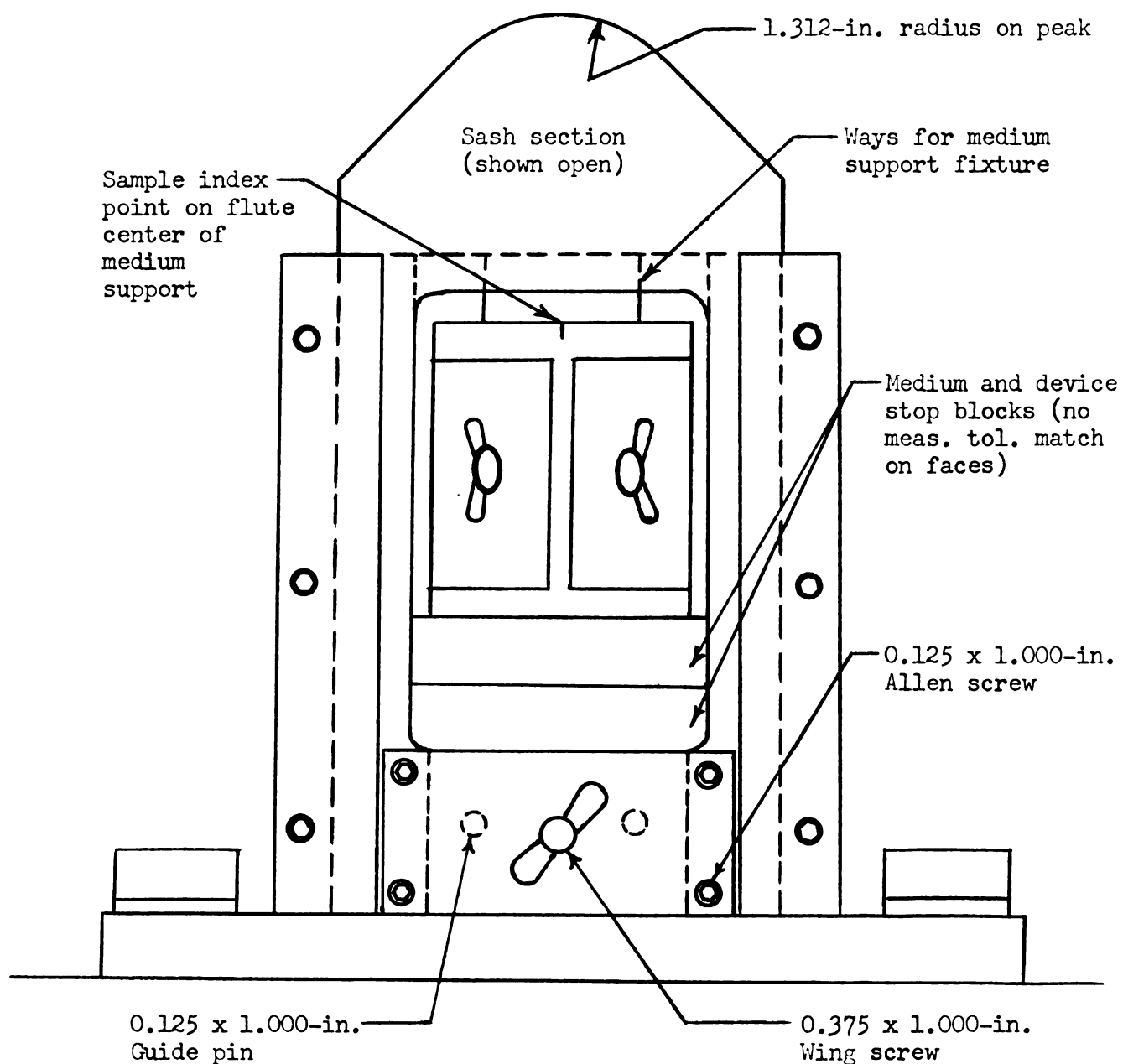


Figure 1. Illustration of glue line shear device (front view).

Notes:

1. Illustration shows section taken through center line of device front view (pg. 28).
2. Critical dimensions: (as measured to 0.00.-in.).
 - a. Line of action of sash grip plate must be parallel to flute surface of medium support fixture with no meas. tol.
 - b. Stop block sections of medium support and device must match with no meas. tol.
 - c. Device weighs 25.1 lbs. Sash in situ weighs 1.2 lbs.
3. Not to scale.

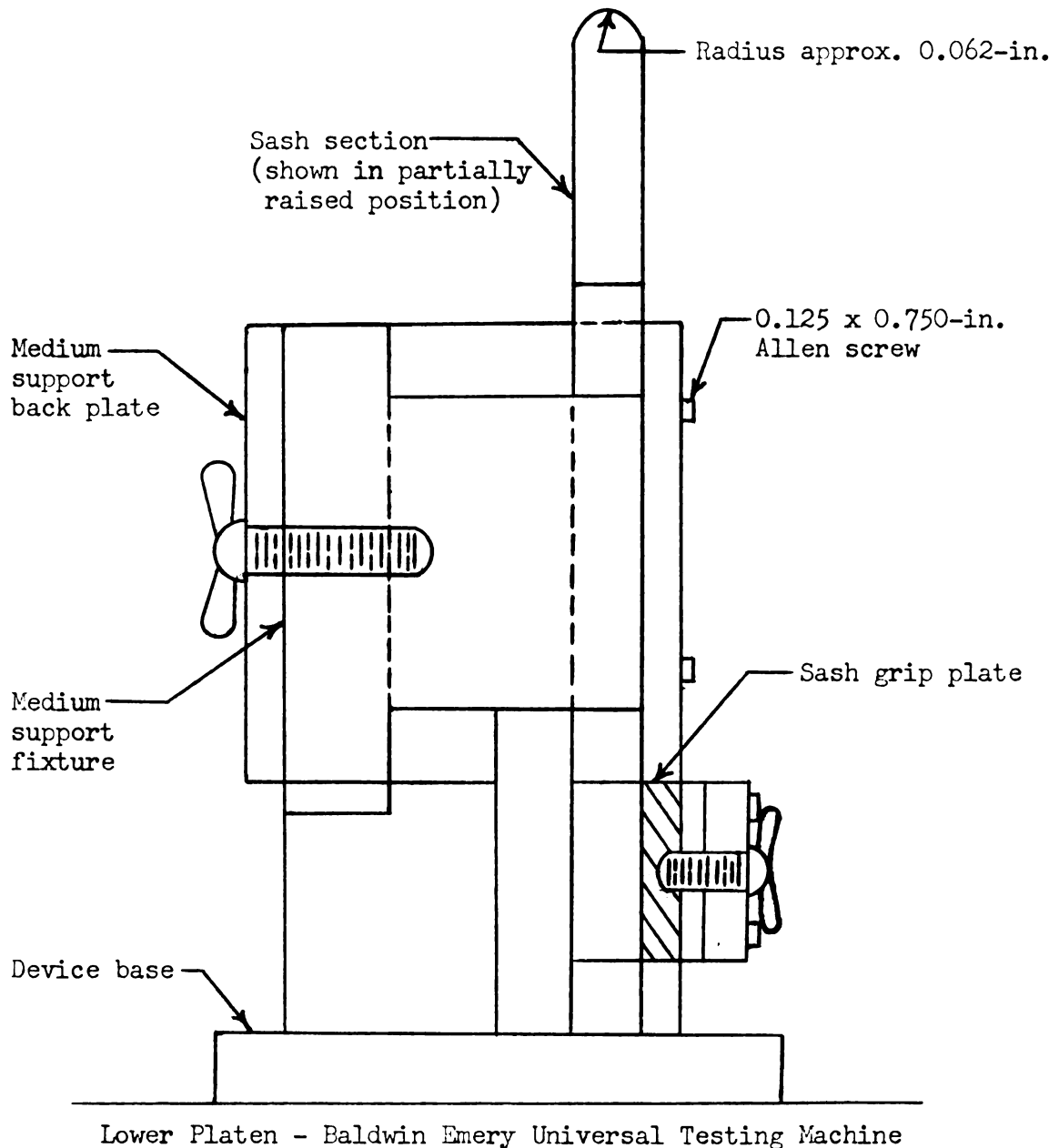
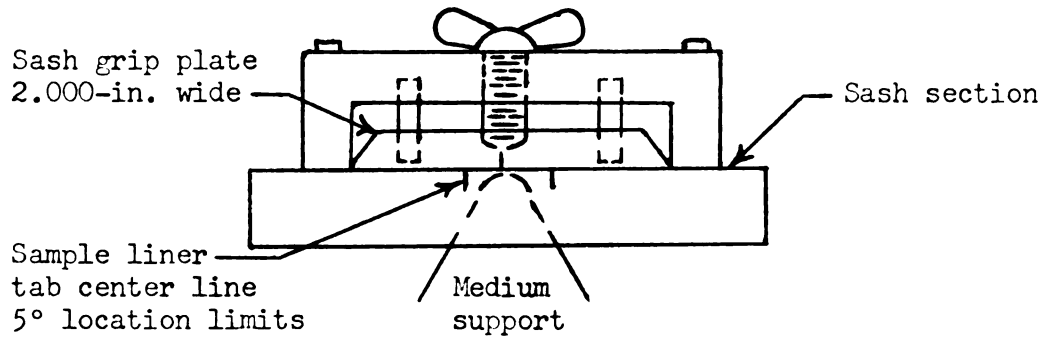


Figure 2. Illustration of glue line shear device (section side view).

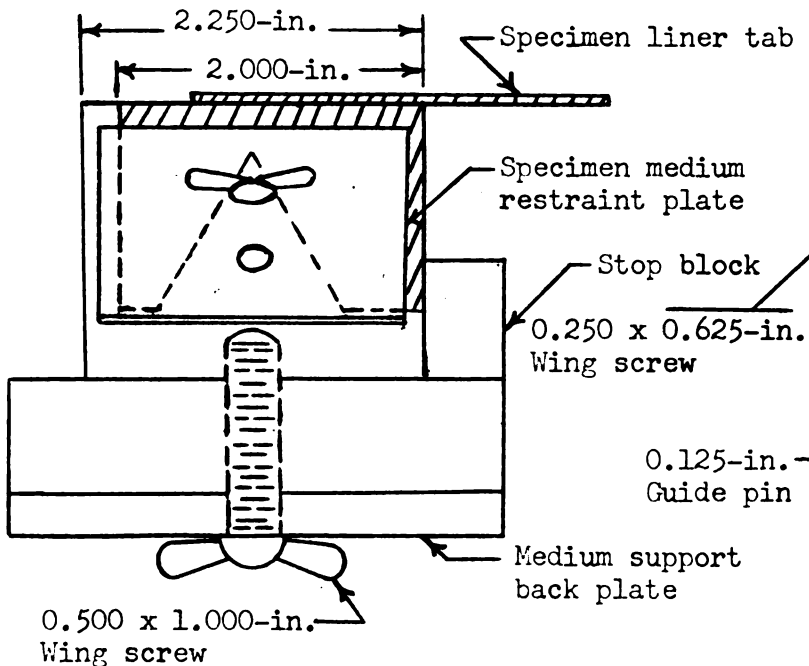
- a. Top view of liner grip plate portion of shear device sash section.



Notes:

1. Tol. of device dimensions ± 0.001 -in.
2. Tension test fixture identical to triangular element of medium support with 0.300 hole drilled through end to end, 0.250 from base on vertical center line.
3. Not to scale. Terms sample and specimen used synonymously.

- b. Side view of medium support fixture of shear device with specimen.



- c. Top view of medium support fixture of shear device with specimen.

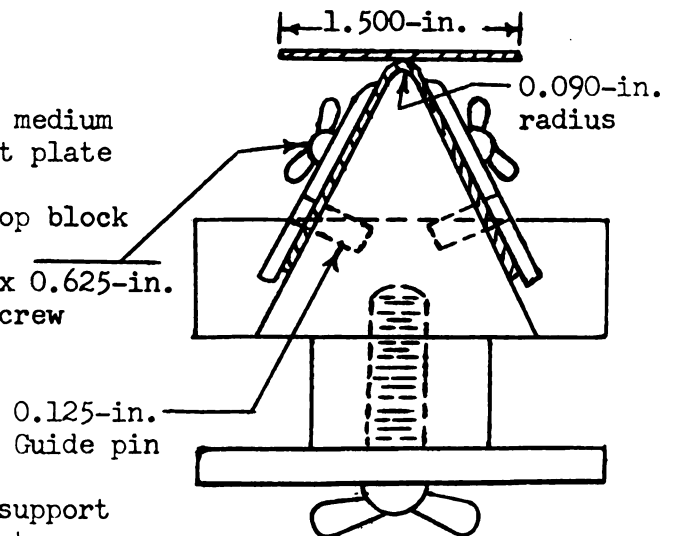


Figure 3. Illustration of details of glue line shear device with typical specimen.

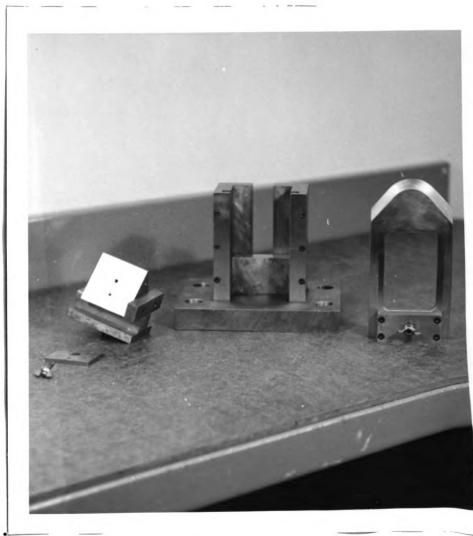


Figure 4. Partially disassembled glue line shear device for testing of simulated corrugated fiberboard adhesive bond. Left to right: medium support restraint plate with wing screw; medium support fixture; device frame and base; sash assembly with liner tab grip plate section (front).

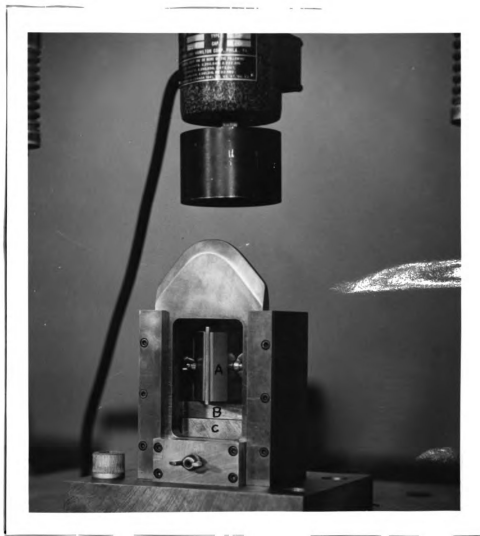


Figure 5. Glue line shear test device. Device is shown ready for use, with sash section in the down position. Note: medium support fixture with material restraint plates (A), medium support fixture stop (B), device frame stop (C), and 500 lb. capacity Baldwin SR-4 load cell for applying and measuring test load.

are primarily shearing in nature, however, as evidenced in the section describing the preliminary tests. In the course of developing the experimental technique a series of preliminary tests was used to confirm the validity of the method, and establish its limitations and peculiarities. These tests, together with comments pertinent to the evaluation method, may be found in Appendix B in detail.

As shown in Figure 6, the removable support for the medium component may be mounted in a set of universal joints, and the liner element bonded to a block of hardwood or similar material. With the wood support block also attached to a universal joint system, the adhesive bond may be subjected to tensile loading in a universal testing machine, as shown in Figure 7.

Preliminary tests. -- The primary objective of the preliminary experiments was to evaluate the two potential methods of testing the adhesive bond, in order to select one as the criterion for examination of selected bonding variables. It was necessary to establish the specimen dimensional characteristics, and to compare the reliability and response, or sensitivity of the alternative test methods. Theoretical analyses and field observations had given predominant support to a shear type test, but the certainty of some tensile stress at the bond, as either a primary source of failure, or in association with shear stresses, indicated the consideration of some mode of evaluation based on a test of bond tensile strength.

Prior to the construction of the glue line shear device, a wooden prototype was assembled in order to assure the practicability of the proposed test methods. This model, made of hard maple (Acer spp.) and paper

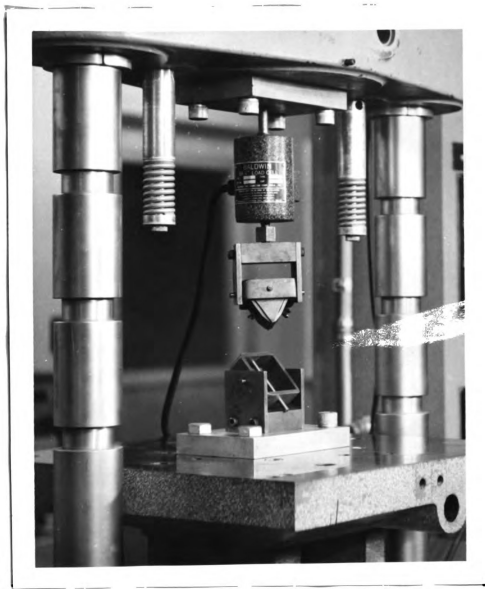


Figure 6. Fixtures for tensile test of simulated corrugated fiberboard adhesive bond. Note universal joint effect at both the Baldwin SR-4 load cell and the lower test machine platen.

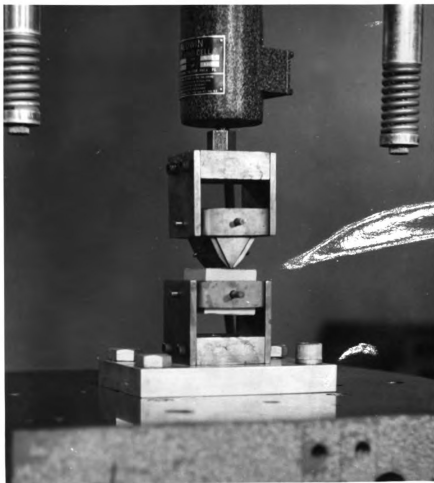


Figure 7. Adhesive bond tensile strength specimen mounted in fixtures for testing. Medium element is held by restraint plates against triangular support fixture (load cell universal joint). Liner is bonded to maple block (pinned in lower platen universal joint).

based phenolic laminate material, was generally similar to the finished device illustrated in Figure 1, except for various non-critical dimensions. By trial and error methods, together with small group comparisons of five or ten specimens, the optimum positioning of the device in a universal testing machine, rate of loading, effect of relative humidity, and the general facets of the test technique were established.

The feasibility of using the triangular medium support from the test device as a lower platen in fabricating the test specimens was also determined. As illustrated in Figure 8, the upper platen of the press device was the electrically heated, rheostat-controlled platen from a small Carver laboratory press. This was attached to a plywood mounting plate with handles, for ease of removal from between the vertical plywood side supports of the press device. A point contact was mounted on the left side support (as seen from the front of the device) to assure a consistently level position. Using a hydraulic compressometer, the press device was repeatedly checked for uniformity of loading, and less than 0.10 lb. variability was noted under a 10 lb. load. The press is shown in the bonding position in Figure 9, with the platen in place.

The technique of testing the single bond specimen in tension was also examined to establish procedural methods. The triangular medium support was drilled, to be pinned into a universal joint mounted in the test machine. The liner segment of the specimen was bonded with a neoprene latex contact cement to a block of hard maple (Acer spp.), which was, in turn, pinned into an lower universal joint. As may be noted in Figure 7, the resulting assembly permits loading of the medium-to-liner bond in nominal tension, with the required freedom of lateral movement

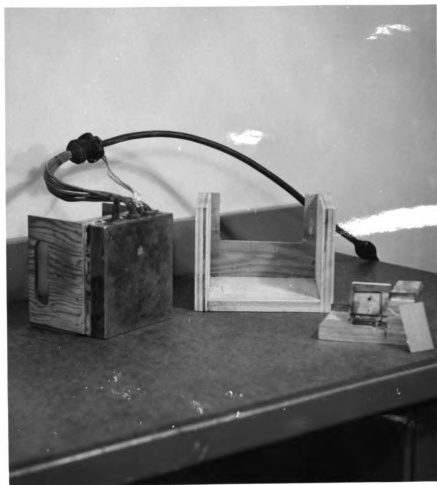


Figure 8. Partially disassembled press device used in preparing simulated corrugated fiberboard adhesive bonds for testing. Left to right: heated Carver press platen with plywood handle frame attached, press device frame, and medium support fixture mounted on plywood and showing spring clips for retraining medium material during the bonding procedure.



Figure 9. Press device assembled and ready for use. Medium support fixture is hidden by front of plywood slide. Pressure applied by dead load added to top of Carver press platen.

in the test system. Both tensile and shear specimens are illustrated in Figure 10.

The complete details of the sequence of preliminary tests may be found in Appendix B. The techniques of specimen preparation and testing established for use in the adhesion factor study are described in the experimental procedure. The results of the preliminary tests are summarized below:

1. The optimum width of the specimen tab formed by the liner element was established to be 1-1/2-in.
2. The optimum length of bonded interface was determined to be 1-1/2-in.
3. In a test of the effect of misalignment in the bonding of the liner tab to the medium portion of the specimen, it was found that misalignment of less than 5 degrees between the center lines of medium and liner elements did not effect a significant difference in test results. A misalignment of 5 degrees or more from the mutual centerline is visibly detectable.
4. It was considered desirable to establish some definite relationship between the proposed shear test and an accepted standard testing method. If the liner portion of the specimen is of the appropriate width, failure in tension will occur in the tab, rather than at the medium-liner interface. Based on a paired group experimental design, no significant difference in tensile strength values was determined, using the glue line shear test device, and a standard ASTM test method for the tensile strength of paperboard.

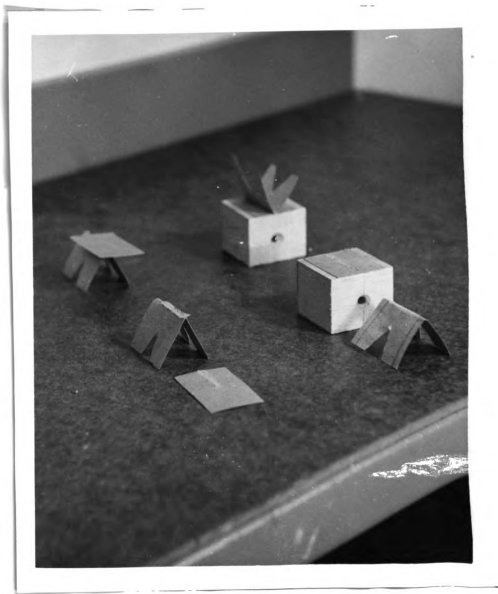


Figure 10. Typical simulated corrugated adhesive bond test specimens. At left: shear strength specimen ready for testing, and a failed specimen showing 100% paper failure in the liner. At right: tensile strength specimen ready for testing, and a failed specimen showing partial paper failure in the liner. Note liner bonded to maple (Acer. spp.) block for testing with load normal to the glue line.

5. In comparing the sensitivity or response of single liner-to-medium adhesive bonds to shear or tensile loading, the test employing shear exhibited the greater and more consistent relative sensitivity.
6. In evaluating the comparative reliability of shear and tension testing methods, it was found that the coefficients of variation of the test techniques were 16 percent and 23 percent respectively, based on several test replications. Comparison of these estimates of relative variation indicates greater reliability inherent in the shear test. For proposed experimental purposes, a minimum sample size of five shear specimens was estimated by conventional statistical techniques.

The results of the preliminary test sequence served to establish the experimental technique and the characteristics of the specimen to be used in the evaluation of bonding variable effects. From the combined results of theoretical analysis, preliminary testing, and the limited field study of container failure, the use of the shear test was indicated, with the details of methodology delineated by the results and experience gained from the preliminary evaluation sequence.

Qualitative examination of the adhesive bond. -- As a preliminary study related to this investigation, photomicrographs of both single face and double back bonds were made. These illustrations, shown in Figure 11 and Figure 12 were made of actual production-run glue bonds, sectioned and mounted in paraffin. The difference due to the differing degree of pressure in the two processes is quite distinct.

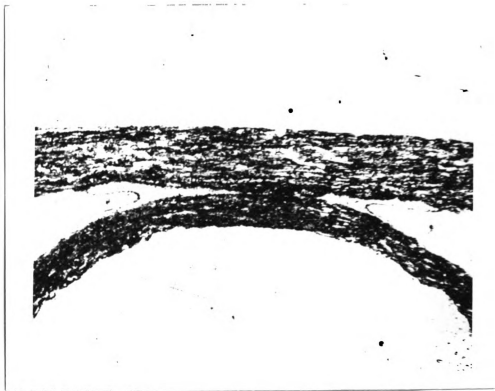


Figure 11. Photomicrograph of double back adhesive bond in corrugated fiberboard (40x). Note minimal contact between liner and medium elements, and shoulder effect of the adhesive either side of the contact interface.

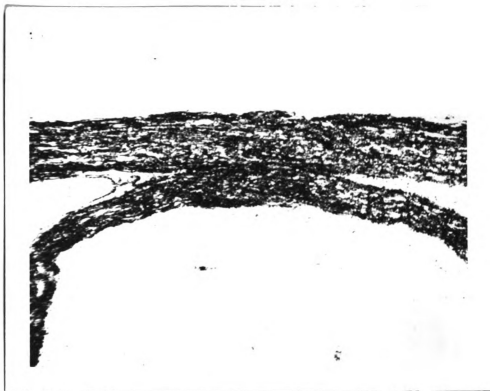


Figure 12. Photomicrograph of single face adhesive bond in corrugated fiberboard (40x). Note embedding of curved medium material into liner at interface and shoulder effect of adhesive either side of the contact interface.

An attempt was also made to bond specimens of the same type utilized in studying the adhesive bond but without adhesive, to examine the fiber bonding effect. These specimens, resembling a single flute of medium bonded to a section of liner board, were fabricated only after considerable trial-and-error procedure, and then with only limited success. The bonds achieved were very fragile, and far too weak to test with the method associated with the evaluation aspect of the study. Some bonding was noted, however, and with careful handling the specimens remained intact for several hours. The results implied some direct fiber-to-fiber adhesion in the corrugated paperboard adhesive bond, but the contribution to total bond strength did not appear significant at the levels of bonding variables used.

These above preliminary procedures are discussed in detail in Appendices A and C, respectively.

EXPERIMENTAL PROCEDURE

In considering the details of the experimental procedure to be employed in the investigation of adhesion variables, it became immediately apparent that some severe limitations were required of the number of factors and factor levels to be included in the study. The general order of the investigative procedure was as follows:

1. Selection of test specimen materials; primarily the adherend paperboard and adhesive formulations employed.
2. Designation of the main variables to be included in the study, and the respective levels of these variables.
3. Selection of specimen elements from the designated materials, and sample preparation by the methods and criteria determined.
4. Evaluation of the specimens by the shear test technique previously established.

The procedure is described subsequently in detail.

Selection of materials. -- Based on the difficulties encountered in the preliminary tests, the existence of published information, and the desire for the maximum effective glue line area available, it was decided to limit the geometry of the sample to 1-1/2-in. of glue line and a medium curvature equivalent to representative A-flute board. (average 36 corrugations per foot) The decision was made to limit the study to a typical liner, medium, and adhesive combination, based on the availability of material and information, and the previously stated scope of the proposed study. The liner material was 16 pt. Kraft paperboard with a nominal basis weight of 69 lbs. The corrugating medium selected

was a 9 pt. semichemical type, with a 26 lb. nominal basis weight. The adhesive chosen for the principal test sequence was a two-stage starch mixture, typical of the raw starch - cooked starch mixture used in the industrial manufacture of corrugated boxboard. The specific adhesive formulations used appear in section D. of the Appendix.

To verify the identity and character of the materials selected, a series of control tests were conducted, including evaluation of the caliper, basis weight, tensile and bursting strength of the paper elements. All tests were performed according to ASTM standards (1). The paper materials used were supplied by the Ohio Boxboard Company of Ohio, and the adhesive by the Stein Hall Company of New York.

Selection of experimental variables. -- The choice of main factors to be incorporated in the investigation was based on the results of existing published research, discussion with persons active in the industry, and the limitations of the existing laboratory facilities.

It was immediately apparent that press times approximating those in production circumstances were impractical, considering the other limitations regarded as critical. In gluing operations the primary function of heat, however, is to facilitate solvent or carrier removal, and motivate any necessary chemical reactions. In most adhesive processes the time-temperature interaction can be varied, within practical limits, by simply adjusting one factor to complement the other, so long as the necessary physical response is produced at the glue line.

It was therefore decided to incorporate the effects of temperature, pressure, moisture content, and spread (unit weight of adhesive applied), as experimental factors, since these are conventionally accepted and

logically critical elements in the bonding of most cellulose-based adhesives. Since the suppliers of the adhesive recommended the use of slightly different adhesive formulations for the two distinct bonding processes involved in the conversion of corrugated board, it was felt that the presumed effect of press time on the adhesive should be confirmed for both formulations. While the gel temperatures of the respective formulations assured cure of the adhesive to some extent, it was evident that some further knowledge of the rate and degree of cure of both variants would be of value in the analysis of experimental results. The study investigation thus constituted itself into two primary experimental parts, with temperature, moisture content, pressure, spread, and formulation incorporated in the five factor study or first phase, and press time and adhesive mix in the second phase. The basis for the choice of factor levels is described in detail below.

1. Formulation: -- The Stein Hall Company, in correspondence, recommended two formulations as laboratory approximations of those suggested by the company for use in the single face and double back bonding operations. These appear in Appendix D. As may be noted, the chief differences in the properties of the formulations are their respective pH, gel point, solids content, and viscosity values. The pH, solids content, and gel point of both formulations were verified for the several mix replications used in the study. Distilled water and reagent grade chemicals were used for the other adhesive ingredients. No adhesive mix was kept for more than six hours pot life, though the supplier stated that uniform behavior could be expected of either mix over a period of several

days of mixed storage life. The approximate solids content and the gel point of each individual mix of adhesive used was also verified. Except for temperature of gelation (gel point), all verification tests were conducted as prescribed by ASTM procedures where possible. Gel point was measured directly with a thermometer as the adhesive was heated and gently stirred in a small beaker.

2. Weight of adhesive spread: -- Several of the literature sources noted the weights of adhesive spread in the respective subject studies. The most current references (42) (51) indicated a spread of 2 - 4 lbs. per thousand square feet of finished board, based on the weight of dry starch without noting the actual adhesive spread. It was therefore decided to base the spread used in the study on the general Stein Hall recommendation of about 1-1/2 - 2 gallons, or 15 lbs. of adhesive per thousand square feet of finished board. If the conventional 36 flutes per inch for A-flute board is assumed, this reduces to approximately 0.017 gr. of cured adhesive per specimen. It was decided to include two levels of spread in the study, 0.02 gr. per specimen (light or standard), and 0.04 gr. per specimen (heavy), based on an approximate 20 percent solids content. A more exacting control of the weight of applied adhesive was not practicable, especially in view of the number of test samples potentially involved. The lighter spread was within the approximate range of values noted in the literature, and the heavier was adjudged to be a sufficient increase to detect any factor

differences that might exist. Attempts to utilize a lighter adhesive spread than 0.02 gr. per specimen produced very erratic results in trial tests.

3. Bonding temperature: -- As noted in section D of the Appendix, the approximate temperatures of gelation of the single face and double back formulations are 150°F. and 140°F., respectively. In a series of tests, employing a potentiometer with the conventional copper-constan thermo-couples, the temperature at the glue line with different platen temperatures was determined for a 10 second press time. On this basis, the following platen temperatures and corresponding interface temperatures were selected for use as factor levels:

<u>Platen Temp (°F.)</u>	<u>Glue Line Temp (°F.)</u>	<u>No. Trials</u>
160	140-143	10
180	146-149	10
200	152-156	15

A glue line temperature of 145°F. was noted with a platen temperature of 200°F. in about 4 seconds. It was felt that this choice of variable levels provided a sufficient range of values to detect any significant temperature effects, since the temperature sensitivity of the adhesive in regard to gel point indicated that a wider range of levels could serve no useful purpose, and a more precise measure of temperature was not reproducible. Since the temperature at the glue line during bonding is a result of the usual press-time - platen temperature interaction, it is commonly more practical to vary press time to obtain precise cure conditions at the interface, both in experimental work and in general usage.

4. Moisture content of paper: — As previously noted, the few available published reports of paperboard moisture content during conversion indicate a variety of levels, from a low of 6 percent to a maximum of 12 to 13 percent. Most of these values were established at the single facer, or the point of determination is not cited. The opinion of operators in the field tended to favor a value of 9-11 percent, at both single face and double back bonding operations (32) (36) (42) (51).

On the basis of the available information, and to provide a range of values that would detect significant interaction effects, moisture content levels of approximately 6, 12 and 20-21 percent, based on oven dry weight, were selected for testing. The 12 percent value was chosen as representative of service conditions, and 6 and 20 percent as logical extremes of the factor.

5. Bonding pressure: — The selection of test levels for pressure at bonding was based primarily on results of the investigations by Max (36) and McKee (32), and upon the premise that some measurable effect of the factor was desirable. The literature implied that beyond possibly intensifying the rate of heat transfer, the effect of pressure was of minor importance. It was thought that a very low pressure, of about 6 lbs. per lineal inch, and about double this value, or 12 lbs. per lineal inch, should detect evidence of the effect of pressure on the bond. A brief exploratory test, made on typical specimens

confirmed the premise that 12 lbs. per inch would produce an adequate bond based on the presumed bonding procedure.

For the examination of the effect of press time on bond quality, series of test samples were bonded at press times varying from 1 to 35 seconds, using both Stein Hall starch formulations. The samples, conditioned for a period of four days to a 12 percent moisture content, were bonded using a light or standard spread cured at a 200°F. platen temperature under a pressure of 6 lbs. per lineal inch of glue line.

In the selection of some of the variable levels noted above, a main factor effect was virtually assured, the interaction effects being the prime object of interest. In general, it was thought that detection of significant interaction effects could be best assured by the inclusion of potentially significant main factor levels.

Material selection and designation. -- The paper stock for liner and corrugated medium materials was contributed by the Ohio Boxboard Company in roll and sheet form, respectively. Since no logical selection sequence could be applied to the medium sheet stock, it was arbitrarily decided to take twenty sample items from each 16 x 16-in. sheet, the thirty-six required sheets selected at random from a lot of about one hundred. In the selection of liner material elements, in order to avoid any systematic discrepancy due to variation along or across the roll due to the effects of paper formation, specimen elements were taken from the full width of the stock at the beginning, middle, and end of the roll. The medium and liner specimen parts were cut to the size indicated as optimum by the preliminary tests. The medium portion of the sample was 3-1/2-in. wide by 2-in. along the flute, and the liner tab

1-1/2-in. wide and 3-in. in length. Maximum tolerances of ± 0.003 -in. were permitted on those dimensions considered critical; the 2-in. medium dimension, the liner tab width, and the bond lap length. The orientation of both materials was such that the machine direction was effectively normal to the flute direction and the adhesive bond. In the case of both materials, the elements were completely mixed, and specimens selected at random.

Specimen designation was composed by simply abbreviating the selected test variable levels, as follows:

A-B-C-D-E-F

where:

A = moisture content level (6, 12, 22)

B = adhesive spread intensity (L, H)

C = cure temperature (160, 180, 200)

D = adhesive formulation (S, D)

E = cure pressure level (6, 12)

F = the no. of the specimen in the group (1-10)

for example:

12 L 180 D 6 - 5

is the fifth specimen in a lot conditioned to 12 percent moisture content, and bonded with a light double backer type adhesive formulation, at 180°F. under 6 lb. per inch of glue line pressure. In other designations the number elements are self explanatory, i.e., H and S would indicate heavy spread and single facer formulation, respectively. This specimen identification system encompassed all the experimental conditions employed in the five-factor study, the press time phase requiring no additional designations.

The various samples were selected by pooling the entire aggregation of specimens after cutting, mixing it thoroughly, and drawing the appropriate lots of ten for each experimental treatment combination at random. It was concluded that by a random choice of samples and representative material selection from that available, any trends in inherent material properties would be randomized across the entire investigation. Both liner and medium specimen elements were segregated by this procedure. In the manner described, a total of 720 specimens were selected to provide ten replications of each of three main factors at two levels, and two factors at three levels.

A second array of samples was chosen at random for the test involving investigation of press time and adhesive formulation, requiring again ten items for each test design cell, or a total of 620 specimens.

All material was stored under conditions of $50 \pm 2\%$ r.h. and $72 \pm 3^\circ\text{F}$. prior to the bonding procedures.

Sample preparation. -- The liner and medium materials for the five-factor study, cut to size, were divided into three lots for conditioning at each of the selected moisture content levels. It was found that the conditions maintained as standard in the test and storage area of the laboratory of the School of Packaging ($50 \pm 2\%$ r.h. and $72 \pm 3^\circ\text{F}$.) provided an average moisture content of 6.6 percent in both materials. This was considered suitable for the sample to be conditioned to 6 percent moisture content prior to bonding. After a number of trials, it was found that moisture content levels of 12 to 13 percent and 22 percent could be reliably obtained utilizing a Blue M glass-topped humidity chamber. Extra material was included with each lot of specimens for

verification of moisture content level. All specimens were bonded in the area adjacent to the humidity chamber at ambient conditions (60-67% r.h. and 71-76°F.). It had been predetermined that a minimum of 3-1/2 minutes was required to effect a 1 percent change in the moisture content of a specimen. Since the bonding procedure took less than one minute, it was apparent that the effective moisture content at bonding could be presumed to be that of the conditioning environment. Specimens were removed from the humidity cabinet in samples of 10, and kept in a polyethylene bag prior to use. The specimens conditioned to 6 percent were divided into several groups, and each protected by a polyethylene bag during the gluing procedure and during transportation from the conditioning environment. All specimens were conditioned for a minimum period of four days prior to bonding. It was considered most convenient to bond all samples at a given temperature within a moisture content level at the same time, since these two variables were the most difficult to reproduce exactly. The Carver press platen was adjusted to the desired temperature, as indicated by a potentiometer, and periodic checks were made during the bonding procedure to assure that the desired temperature level was maintained. The platen, ready for use, is shown in Figure 8.

The required adhesive mixtures were prepared exactly as detailed in Appendix Section D, with fresh adhesive prepared for each sample sequence. It was found most satisfactory if the dry starch adhesive components were added very slowly to both the cooked and raw starch portions of the formulations. Otherwise, both mixes tend to become excessively lumpy, with poor general dispersion of the starch. The mixes required very frequent stirring to maintain good consistency, and it was found that the temperature required for preparation of the cooked

starch component was extremely critical, since a stringy condition at the surface was induced with excessive heat. Without doubt, use of an electric heat jacket or water bath, rather than a hot plate, would have facilitated the preparation of the adhesives.

With the assembled, heated press ready and the adhesives prepared, the sample medium was taken from the protective bag and immediately applied to the medium support fixture. It was found helpful if the material was drawn sharply across a rounded corner, such as a table edge, to assure a proper fit on the fixture. It had also been noted in previous experience that the proposed bond surface must not be touched with the hands, or a good bond cannot be assured. With the medium firmly held in place by the spring clips shown in Figure 8, the fixture was then placed in the press in an indexed position, and the designated adhesive mix applied.

Perhaps no part of the preliminary work necessary before the preparation of the samples for the principal portion of the study gave as much difficulty as the determination of a reproducible, uniform method of applying the proper amount of adhesive. After extensive trial and error determinations based on weighed measures of adhesive deposit on specimens, the following method was established as the most practical, reproducible, and analagous to the method of adhesive application in the actual corrugated bonding process. It was found that a 0.04 gr. spread of adhesive could be applied to the specimen by dipping a stainless steel chemist's spatula into the adhesive, permitting the excess to run off, and then pressing the spatula firmly against the glue line area of the specimen flute tip. The standard, or lighter spread was achieved by brushing a layer of adhesive on the spatula blade with a stiff camel's

hair brush, waiting a few seconds for the adhesive film to become uniform, and then pressing the blade on the specimen for adhesive transfer. The variation in spread was less than 0.005 gr. for the 0.02 gr. per specimen spread, and not greater than 0.008 gr. with the 0.04 gr. application, as determined by preliminary tests using an automatic balance accurate to four decimal places. Because of the very small weights of adhesive involved, close control of the variable was quite difficult. It was thought, however, that the above technique, together with the size of the samples involved, would produce representative values. It should be noted that it was found necessary to clean the spatula with water between each specimen, and the brush between each sample. Clean, dry equipment was absolutely critical to this phase of the experimental procedure.

With the medium support fixture in place in the press and the adhesive applied to the medium flute tip, the liner adherend was firmly pressed in place to form a 1-1/2-in. overlap, using a center line drawn on the tab surface and marks on the fixture to assure proper alignment. The Carver press platen, with the necessary tare weight to produce the desired glue line pressure, was then set in place. At the end of the 10 second press time, as measured by a stop watch, the platen was lifted, and the fixture removed from the press. The specimen was carefully removed from the fixture and set aside for storage at the end of the preparation sequence. At the completion of the bonding of the moisture content lot, the entire sample array was stored at $50 \pm 2\%$ r.h. and $72 \pm 3^{\circ}\text{F}$. for a minimum of five days prior to testing.

The preparation of specimens for the second phase of tests, involving various press times, followed the same general procedure. All

specimens conditioned to the 12 percent moisture content level were bonded in a single sequence under the conditions previously specified (page 53). The samples prepared with the single face formulation were bonded in one lot, and those with the double back mix in a second.

While the above explanation of the gluing procedure is, of necessity, somewhat lengthy, it should be emphasized that the actual procedure required rapid implementation, especially during the period extending from when the material was removed from environmental protection until the press platen was applied to the specimen. Interruption of the bonding cycle can result in excessive adhesive migration into the medium, partial drying of the adhesive on the application spatula, migration of the adhesive into the liner before heat and pressure are applied, and a host of other difficulties that can result in a non-representative, or even nonexistent bond. In addition, the bonded specimens were extremely fragile in some respects, and required very careful handling during removal from the support fixture and later during the testing procedure.

Testing procedure. -- During the five day pre-test conditioning period, at $50 \pm 2\%$ r.h. and $72 \pm 3^\circ\text{F}$. in the Packaging School Laboratory, the samples from the three preparation sequences of the five-factor study were completely mixed, so that specimens could be selected in random groups of ten specimens for testing. This procedure was employed in order to randomize any sequential effect that might be present or develop in the test equipment system. The specimens constituting the second part of the investigation were similarly mixed and selected at random, but were not intermingled with those of the five-factor phase.

The more general aspects of the conduct of the tests are discussed in the section of this study dealing with the development of the glue line shear test device and the technique related to its use. The specific aspects of procedure used for this investigation are described below.

The samples were tested in the same temperature - humidity controlled area of the Packaging Laboratory in which they were stored prior to evaluation, i.e., at $50 \pm 2\%$ r.h. and $72 \pm 3^{\circ}\text{F}$. The samples from the five-factor phase of the investigation were tested in two sequences, and the press time study specimens in a third and fourth.

The specimen to be tested was carefully fitted to the medium support fixture, and the restraint plates tightened with the attached wing screws, as shown in Figure 3. It was necessary to exercise particular care that the medium element was tight against the support the entire length of the specimen, and to align the index marks on it with those on the fixture, to assure the proper response to the test load. As the support and specimen are then inserted in the ways at the back of the test device, the stop of the support section must meet flush with the stop of the device, as shown in Figures 2, 3, and 4. Otherwise, misalignment of the liner tab will result, with subsequent improper loading of the adhesive bond. The sash section was then raised into position against the medium support, and the liner secured by the sash grip plate. Index marks on the upper surface of the grip plate indicated the proper position of the tab within 5 degrees of true alignment. With the application of load to the upper surface of the sash, the specimen was failed along the apex of the support fixture.

The load at failure was noted and recorded for each specimen, as

well as the percent of paper failure, to the nearest 25 percent. Experience gained in the design trials of the fixture and the preliminary tests indicated that more precise estimates of the proportion of paper to bond failure were neither feasible nor meaningful. The details of the test equipment are illustrated in Figure 5. A specimen is not shown in the mounted position so that the details of the fixture might be more evident, and the sash section of the device appears in the down position. As may be noted in Figure 5, the position of the device on the lower platen of the Baldwin universal testing machine was maintained by an Allen screw in the front corner of the base, and an index mark at the rear. The 500 lb. capacity type SR-4 Baldwin load cell employed to load the sash appears above the device. A 0.50-in. per minute rate of deformation was used in the testing of all specimens.

Typical specimens after failure appear in Figure 13, illustrating the characteristic modes of failure in the adhesive film, in the paper element, and across the liner tab. The latter type of failure did not occur except in the preliminary tests, of course, since the tab width had been specifically selected to avoid it. The specimen shown in Figure 13 was deliberately failed in this manner to illustrate the mode of failure. As explained in the section dealing with the discussion of the experimental results (page 73), the entire sample array for the study of the effect of press time on the bond quality of the single face and double back formulations was not tested. As this phase of the sequence progressed, it became evident that no useful information would be contributed by some of the groups bonded at the more extended press times.

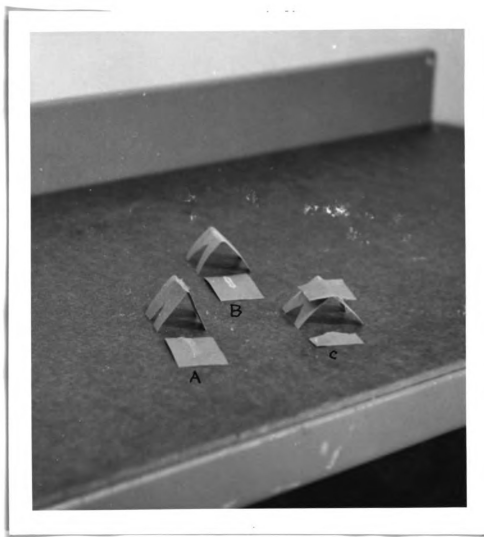


Figure 13. Typical modes of failure in adhesive bond shear specimens. A. paper failure in liner adherend, B. failure in adhesive, and C. tensile failure in line tab element.

DISCUSSION AND ANALYSIS OF RESULTS

The results of the experiments to determine the effect of the various factors of adhesion incorporated in the investigation are summarized in Tables 1 and 2 and in Figure 14. Table 1 presents the response in strength and type of bond failure exhibited by the shear specimens prepared in the coincident study of five adhesion variables. The effect of press time on the strength of specimens bonded with two variations of the starch adhesive formulation is illustrated graphically in Figure 14. The results of the five-factor study were evaluated by conventional statistical methods, and a summary of the analysis of variance of these data is presented in Table 2. The press time-formulation test series was analyzed qualitatively from the graphical presentation of the data in Figure 14. The complete data and analyses related to the various aspects of the investigation may be found in Appendix E. The techniques of statistical analysis employed were conducted as recommended by Davies (14) and Snedechor (47).

Effect of adhesive bond formation factors. -- The effects of the principal factors studied are reflected by the average breaking loads of the respective samples as shown in Table 1 and Figure 14. The levels at which the main factors chosen for evaluation had been imposed in the preparation of the samples were selected in the hope of producing significant effects, but some of the differences caused by the effects of these factor variations are nonetheless striking.

The summary analysis of variance presented in Table 2 indicates a 99 percent level of significance in the differences that may be ascribed

Table 1. AVERAGE BREAKING LOAD AND PERCENT PAPER FAILURE¹ IN SHEAR SPECIMENS BONDED IN THE STUDY OF FIVE ADHESIVE BOND FORMATION FACTORS^{2,3}

Adhesive formulation type		Single face bond						Double back bond						
Applied bonding pressure	Adhesive spread	Heavy			Standard			Heavy			Standard			
	Platen temperature at bonding (°F.)	160	180	200	160	180	200	160	180	200	160	180	200	
	6 Pounds per lineal inch	6	(O)	(O)	(*)	(I)	(I)	(*)	(I)	(*)	(II)	(II)	(III)	(*)
		12	10.3	15.7	31.1	28.4	28.9	31.4	25.3	16.8	27.6	26.1	27.1	29.2
		22	(O)	(O)	(I)	(O)	(I)	(II)	(I)	(*)	(II)	(O)	(II)	(*)
12 Pounds per lineal inch	Average paperboard moisture content at bonding (percent)	(O)	(O)	(O)	(O)	(O)	(II)	(O)	(O)	(O)	(O)	(O)	(*)	
		14.1	17.4	20.4	15.5	24.3	24.4	22.8	17.8	28.8	16.1	27.1	30.9	
		0	4.2	15.5	0	2.5	28.7	0	9.2	17.9	0	16.0	30.7	
	6	(I)	(I)	(*)	(II)	(*)	(*)	(II)	(III)	(*)	(III)	(*)	(*)	
	21.6	25.1	30.8	29.3	29.8	31.0	26.0	29.5	31.0	29.1	30.9	32.5		
22	Average paperboard moisture content at bonding (percent)	(O)	(I)	(I)	(I)	(III)	(*)	(O)	(II)	(*)	(O)	(III)	(*)	
		14.2	24.9	26.8	20.7	26.9	30.6	13.3	22.9	30.2	25.1	28.1	31.5	
		(O)	(O)	(I)	(O)	(I)	(II)	(O)	(I)	(I)	(O)	(I)	(I)	
0	23.4	29.7	0	22.9	26.2	0	21.5	26.7	12.9	23.5	23.3			

¹ Percent paper failure designated by: (O) 0-25%, (I) 25-50%, (II) 50-75%, (III) 75-100%, (*) 100%.

² Description of bond formation factors appears in the text section EXPERIMENTAL PROCEDURE, pg. 45.

³ The complete test data appear in Tables 9, 10, 11 and 12, APPENDIX E.

Table 2. SUMMARY¹ ANALYSIS OF VARIANCE OF ADHESIVE BOND BREAKING LOADS IN THE STUDY OF FIVE BOND FORMATION FACTORS

Source of variance	Degrees of freedom	Sum of squares	Mean square	F ratio	Level of significance (%)
Spread	1	3,122.50	3,122.50	223.4	99
Formulation	1	890.22	390.22	27.9	99
Temperature	2	20,958.30	10,479.15	749.5	99
Pressure	1	4,045.12	4,045.12	289.4	99
Moisture (content)	2	21,266.07	10,633.04	762.7	99
SxF	1	3.01	3.01	0.2	N.S. ³
SxT	2	151.43	75.71	5.4	95
SxP	1	172.67	172.67	12.4	99
SxM	2	137.08	68.54	4.9	95
FxT	2	182.66	91.33	6.5	95
FxP	1	156.98	156.98	11.2	99
FxM	2	66.32	33.16	2.4	N.S. ³
TxP	2	1,299.38	649.69	46.5	99
TxM	4	7,009.87	1,729.98	123.7	99
PxM	2	550.17	725.09	19.7	99
Residual ²	693	9,688.24	13.98		
Total	719	69,700.02			

¹ Complete analysis appears in Table 13, Appendix E.

² Within; second, third and fourth order interaction sources of variance pooled as residual term.

³ Non-significant at the 95 percent level of significance.

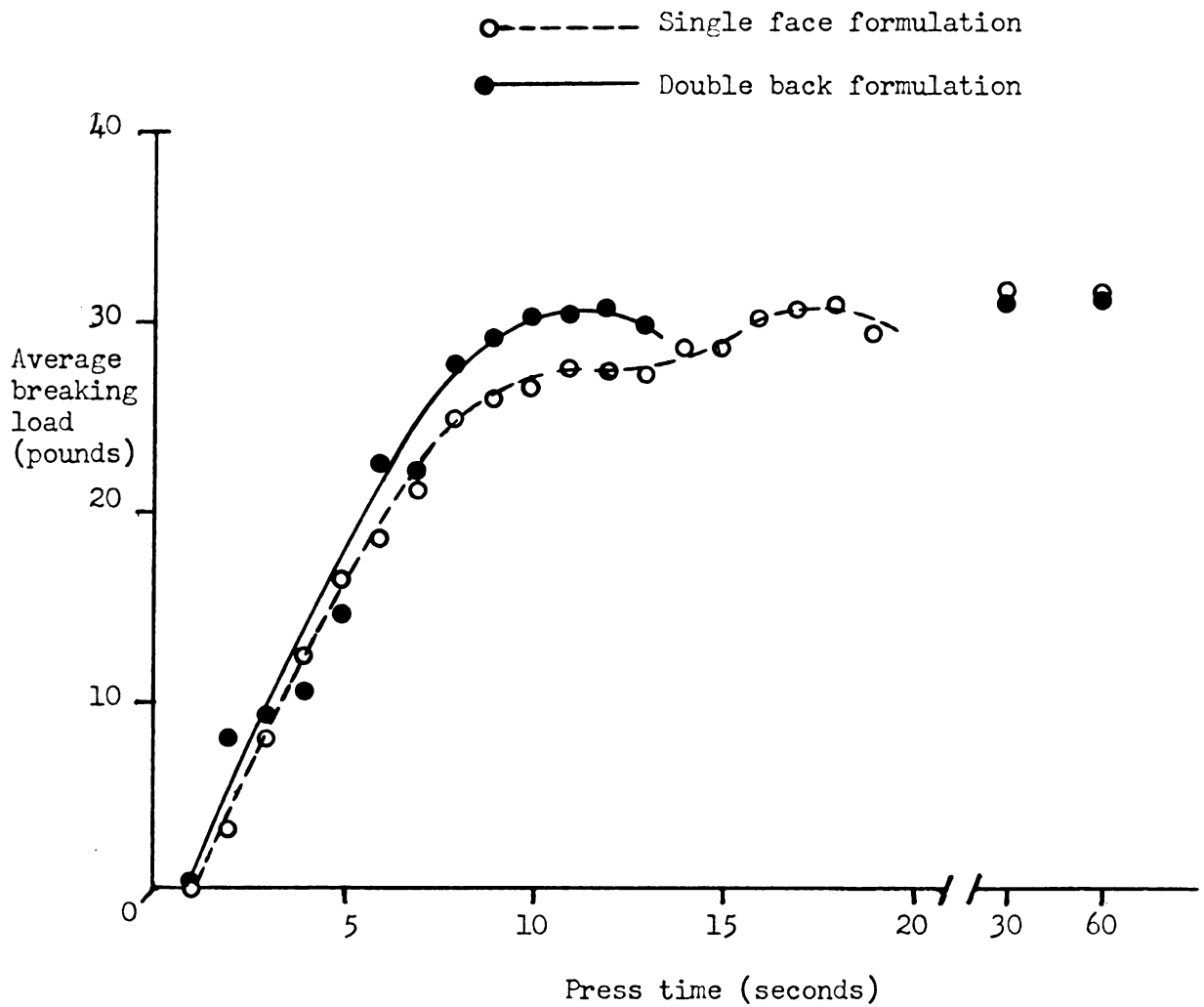


Figure 14. Response of shear specimens bonded with single face and double back adhesive formulations to variation in press time. Each point represents a sample of ten specimens.

to the variations in bonding temperature, pressure, adhesive formulation, paperboard moisture content, and weight of spread incorporated in the investigation. A complete analysis of the data may be found in Table 13, in Appendix E. While the interaction effects present in this phase of the study are subsequently discussed in detail, it must be noted here that almost all exhibited a high level of significance. Under these circumstances, the pooling of interaction terms in analysis and the comparing the averages for levels of one factor by the summation of data over other factors are questionable procedure, in terms of analytical precision. It is common practice, however, (14) (47), particularly in experiments based on industrial processes, to disregard higher level interaction effects and simply make statistical comparisons based on a residual term containing these interaction effects and whatever within variation is present due to specimen replication. This may be justified, since any induced error tends to conservatism, or the suppression of small differences. The physical interpretation of high level interactions is difficult with any degree of reality, and the relative magnitude of mean square values derived in the analysis of variance may permit pooling of the data for purposes of comparison. Consulting the complete analysis presented in Table 13, it is evident that with a single exception, the mean square values for the main effects are much larger in magnitude than those ascribed to their various interactions. The mean square value for formulation is fairly small, but the F test value assigned is still far in excess of the level required for significance. The large interaction value for temperature - moisture content interaction suggests caution in comparing the mean effects of these variables, placing reliance principally on the complete description

of factor combination averages shown in Table 1 when comparing the various mean breaking loads. Accepting the reality of the interaction effects as detailed in the complete analysis, the third, and fourth order interaction effects were successively pooled with the within variation for purposes of discussion. By this means, the relative significance of the main factor and first order interaction effects was demonstrable. As may be noted in Table 2, all main factor effects remained significant when tested with a residual mean square incorporating within variation with second, third and fourth order interactions. The evident effects of these main factors and press time on adhesive bond shear strength in the specimens, are now considered in detail.

1. Adhesive spread: -- The use of excessive adhesive is undesirable from an economic standpoint in the manufacture of corrugating board, but in the context of this study, such practice appears to reduce bond strength. The overall average strength of samples bonded with a standard weight of adhesive was 23.1 lbs., but effectively doubling the amount of adhesive decreased the mean breaking load to 19.4 lbs. Examining the results in Table 1 it is evident that the incidence of little or no paper failure in the samples (0) associated with the heavier weight of spread used, is double that exhibited by the samples bonded with less adhesive. Without doubt the effect of excessive spread is highly dependent on the level of paper moisture content and press platen temperature involved, as may be noted by the significant interaction effects. Some investigators (43) (51) have reported that in some instances, especially where

varying rates of machine speed are involved, heavy adhesive application is desirable. It should be noted, however, that the "heavy" spread used in this study was an excessive application, not merely heavy within the normal range of spread used in the industry. Since the raw starch component of the adhesive cures by temperature, and no difference in the comparative degree of intimate contact of the adherends is involved, the causal agent immediately suspect is the water component of the adhesive. Excessive moisture will inhibit the proper starch-to-fiber formation and the drying of the cooked starch adhesive component, and redistribution of this moisture through the sample after bonding may tend to deteriorate what bonds do exist between paper fiber and adhesive. The strength loss commonly ascribed to the use of heavy adhesive spreads in bonding less porous adherends does not appear to be directly involved, since here the paper adherends tend to form a contexture, without a distinct interface. Casey (11) reports a decrease in the rate of paper strength increase as additional amounts of starch are used at the beater in paper making, but not a reversal effect. The use of excessive adhesive must therefore be regarded as tending to produce adhesive bonds with comparatively low shear strength.

2. Adhesive formulation: -- It had been assumed that notable differences in bond strength would be caused by the two variations of starch adhesive used in the study. Such a response is evident in the average breaking loads presented in Table 1.

The average breaking load in shear was 22.4 lbs. for samples bonded with the double back type adhesive mix, and 20.1 lbs. for those prepared with the formulation recommended for single facer operation. This is expected, since the double back formulation gelatinizes at a lower temperature level, as illustrated in Appendix D. Considering the temperature levels delivered to the glue line by the heated press platen (page 49), it is evident that the raw starch component, which is responsible for much of the bond strength, is gelatinizing at two of the three temperature levels incorporated in the investigation in the case of the double back mix. The single face variety, in comparison, will gel only at the 200°F. platen level, or one of the three experimental temperatures. In actual practice, however, the single face formulation is used under pressure, while the other is not, and the single face bond is commonly stronger. An analogy to the single face operation in Table 1 may be taken as the average breaking load for samples bonded at 12 percent paper moisture content, under 12 lbs. per inch of glue line pressure with the standard weight of spread. The minimum platen temperature to assure gelatinization of the raw starch component is 200°F. In the case of the double backer, similar conditions are selected, except that the minimum platen temperature required is 180°F., and the lower platen pressure is involved. The average breaking load for the single face sample is now 30.6 lbs., and 27.1 for the double back formulation. Thus, under what might be termed conditions of use, the single face bond is as strong, or in

most instances stronger than that formed by the double back formulation. This contention is supported if the levels of paper failure in the bonds at test are compared.

The effects of the different formulations would seem to be dependent on their physical properties, specifically temperature of gelatinization and viscosity. Since the control of these is defined by the gluing operation, it must be presumed that adequate bonds may be achieved with either formulation, so long as proper cure conditions are provided. At either pressure level, there is probably a greater tendency of the single face formulation to migrate away from the glue line into the paper, due to its' lower viscosity, though there was no visible evidence of it in the interface areas of the test specimens.

3. Press platen temperature: -- The effect of the various press platen temperatures was assured, since those selected produced levels of temperature at the glue line well above and below the gelatinization points of the adhesive formulations included in the investigation. The average breaking loads for samples bonded at 160°F., 180°F., and 200°F. platen temperatures were 14.6, 21.5, and 27.8 lbs., respectively. Upon examination of Table 1, the effects of increasing temperature are quite evident, especially at the 12 percent moisture content level. An unexplained paradox in the response of bond strength to temperature, however, is also evident in the behavior of the samples bonded with a heavy spread of the double back formulation under 6 pounds per inch of glue line pressure.

Rather than a rise in bond strength with increasing platen temperature, a reversal effect appears at the 180°F. temperature level. This phenomenon was noted at the time of test, but examination of the specimens, and verification of the formation variable conditions did not suggest an explanation. The degree of paper failure and intimate contact of the adherends does not suggest a strength loss due simply to excessively thick glue lines with the heavy mix. The most probably cause of these low values in bond strength is suggested by the high level of paper failure noted in the samples. The strength of the liner material in these specimens apparently suffered a definite reduction at both 6 percent and 12 percent moisture content levels. The randomization of specimen materials would seem to preclude a basic paper defect, and no factor of sample preparation, storage or test technique could be detected as a potential cause of paper strength loss. Since it is the cross-machine strength of the paper in question, some defect related to moisture is suspect, as it is in this direction that the paper is most responsive to moisture effects. It is possible that moisture, in migration from the heavy adhesive spread or from some unknown source in the specimen bonding or storage environment, caused the loss of inter-fiber bond strength in the liner. In any event, the causal agent and its source remains unknown, since the same materials, adhesive mix, and specimen preparation sequence produced specimens whose strength response did not conflict with logical expectations.

4. Applied platen pressure: -- The influence of pressure at the time of adhesive bond formation is readily evident in Table 1. The effect of doubling the applied pressure per inch of glue line is to increase the degree of intimate contact between the adherends, and as noted in Appendix C, pressure seems critical to the formation of direct fiber-to-fiber bonds between the adherends. The improvement of bond strength by increasing pressure is especially notable at the higher moisture content levels, as seen in Table 1. The degree of paper failure is also generally increased, verifying an improvement in adhesion. The average breaking load for all specimens prepared at 6 lbs. per glue line inch of pressure was 18.9 lbs., and 23.7 lbs. for those bonded under 12 lbs. per glue line inch of pressure. As will be noted subsequently, the interaction effects of pressure and temperature are of particular interest, as well as the participation of pressure in interaction effects with other variables. It should be noted that the actual nip or bonding pressures used in the manufacture of corrugated fiberboard are much greater in the single face bonding operation than those employed in this investigation. Related investigations, such as those by McKee (32) and Max (36) indicate little improvement in adhesive strength over the range of these nip pressures, however.
5. Press time: -- The response of samples tested to evaluate the effect of time under heat and pressure during specimen preparation on adhesive bond shear strength is illustrated in Figure 14.

The press times in this study are, of course, much longer than the actual dwell times present in the modern corrugated conversion process. As in the case of the pressure variable, however, simulation of the actual bonding process in the laboratory was not considered practicable.

As press time was increased, the average bond strength increased in an apparently slightly curvilinear relationship, with both adhesive formulations. At about 12 seconds press time an effective maximum is evident in breaking loads of the samples prepared with the double back formulation. This strength level of about 30 lbs. is apparently constant up to a full minute of press time. The average bond strength of the samples glued with the single face formulation exhibits some tendency to become constant at the 11 second press time, then rises to a maximum of about 30 lbs. at 18 seconds. Some increase in breaking load is evident at 30 seconds and 1 minute, but the increase is so slight that it may be reasonably ascribed to random variation. The increasing difference between the average breaking loads of the formulations in the 6 to 14 second press time range may be ascribed to the advanced cure condition probable in the double back formulation. The difference in bond strength at the initial press times may be attributed, to a large extent, to the lower gelatinization temperature of the double back formulation. As noted on page 49, this formulation gels, or "pastes up" in 3 to 4 seconds at the prescribed 200°F.

platen temperature. As the press time increases the greater viscosity of the double back mix, no doubt, becomes a factor, again advancing the cure level beyond that of the single face mix at a given press time. The fact that by 6 seconds both formulations have certainly reached, and probably passed, the gel point of the raw starch component suggests that the continued increase of bond strength up to press times in the 10-12 second range is due to removal of moisture from the carrier, and possibly the amylopectin component of the raw starch, which does not gelatinize but tends to remain stable in viscosity (45) (46).

At those pressure periods where the average breaking load values for the respective formulations tended to become constant, it was suspected that no subsequent change in average strength would be effected by increasing press times. Consequently, samples were prepared and tested incorporating press times of 30 and 60 seconds. As illustrated in Figure 14, no notable increase in average strength was apparent. Several samples prepared with both formulations were therefore deleted from the test sequence, as shown in Tables 15 and 16 in Appendix E, since such additional data would contribute little information of real value.

Despite the unrealistic length of the press times incorporated in the investigation as compared to actual dwell times in the production process, the press time study does present several pertinent inferences. The behavior of

the two formulations at the shorter press intervals suggests that the time factor is of importance only in regard to the transmission of sufficient temperature to the adhesive bond, to facilitate gelatinization of the raw starch component. If the time is not sufficient to permit conduction of the minimal required heat, differences in formulation behavior tend to disappear, and bonds of both type become very weak. So long as the press time permits delivery of the required temperature level at the glue line, the relationship between formulations will probably remain as shown in Figure 14, even though the press temperature is quite high and the time interval very short, as in the corrugated bonding process. This explanation will apply, however, only in the case of delivered temperature levels near the adhesive gelatinization point. It is questionable if the relationship shown in Figure 14 between the formulations in the 5-10 second press time range exists in a similar state at very short dwell times. The rate of moisture movement out of the adhesive and into the paper or surrounding air after the gelatinization of the raw starch glue component is suspected of affecting bond strength to some extent, as will be discussed subsequently. In the actual corrugated board conversion process the very short dwell times, application of steam and heat to the paper materials to facilitate processing, and differences in the level of heat used at the single facer and double backer probably alter the relationships shown in Figure 14 in the 5-10 second

press time range. In the manufacturing procedure, time will affect not only the rate of temperature transmission, but the rate of water vapor movement out of the adhesive and through the paper as well.

In relation to this study, the results illustrated by Figure 14 assure the fact that complete cure was achieved at some levels of the five-factor study, in which both a 10 second press time and 200°F. platen temperature were employed.

6. Moisture content of the paperboard: -- As had been anticipated in the statistical design of the five-factor study shown in Table 1, the general effect of increasing the moisture content of the paperboard components was to decrease the strength of the adhesive bond and the incidence of paper failure at the bond interface. The summarized average breaking loads for samples bonded at 6, 12 and 22 percent moisture content levels were 26.9, 23.1, and 13.9 lbs., respectively. As noted in the previous discussion of other main factors in the study, the removal of moisture from the adhesive appears to have a definite role in the development of the full strength of the adhesive bond. Whatever strength loss was induced in the paper materials by the higher levels of moisture content should have been regained in the post-bonding conditioning period of four days at $50 \pm 2\%$ r.h. and $72 \pm 3^\circ\text{F}$. Since the percent of paper failure generally decreased with increasing moisture content levels, as is evident in Table 1, the

immediate inference is a significant weakening of the adhesive bond at these higher levels. This strength loss is evidently present not only in the starch-to-fiber bonds, but in the cohesive strength of the adhesive bond film as well. This may be noted in those sample averages in Table 1 where, other factors being constant, a strength loss with moisture content is evident with no change in the low level of paper failure. If the breaking loads of samples bonded at various moisture content levels with no paper failure are considered (Tables 9, 10, 11, and 12, Appendix E), the decrease in starch film strength with increasing material moisture becomes certain. If any intermingling of adherend fibers occurred under pressure the contexture was destroyed when the pressure was removed.

The main effects of the factors incorporated in the investigation are, in the general, in agreement with qualitatively expected results. As previously indicated, averages based on data summarized over more than one factor are of comparative value only, because of the significant interaction effects present in the experiment. Interest in the absolute average strength values of the adhesive bonds of the various samples must be directed toward the specific variable level combinations, as presented in Table 1. The effects of variation in adhesive formulation are almost certainly related to the respective differences in gelatinization temperature and viscosity. The use of excessive adhesive spread appears to produce negative effects, possibly related to problems of moisture removal. The results of increases in bonding pressure and press time appear to produce the conventional reactions of increased strength common to many

adhesion phenomena. If its relationship to moisture is not considered, the influence of temperature on bond strength appears to relate directly to the gel point of the adhesive formulation.

Complete analysis of response to moisture, both as related to the adherend materials and the adhesive, must be reserved until the interaction effects with other variables are examined. The general effect of increasing the moisture content of the paperboard was to detract from adhesive bond strength, but the specific effect on the adhesion mechanism, both in this study and in the manufacture process, must be considered further.

Effect of adhesive bond formation factor interaction. -- As indicated by the results of the analyses of variance of adhesive bond breaking loads, highly significant interaction effects are present in the test data, and are of considerable interest from a practical standpoint. None of the bond formation variables are present in the corrugated fiberboard conversion process, except in intimate association with one another. It was the role and significance of these interaction effects that was of prime interest in this investigation, for unlike the main factor effects, such interactions cannot be readily anticipated with real certainty. The statistical evaluation of these interactions appears in Tables 13 and 14 in Appendix E, and in summary in Table 2. Interpretation of these effects is based on Table 2 unless otherwise noted, and is subject to the limitations previously indicated (page 65) related to the pooling of higher level interaction values. The basis of discussion is the breaking load values of the simulated corrugated paperboard adhesive bond shear specimens.

1. Formulation x weight of adhesive spread: -- This interaction was not significant, indicating that both formulations responded similarly to increases in the weight of applied adhesive.
2. Formulation x moisture content: -- On the basis of the summary analysis, this interaction was not significant, indicating that both formulation types tended to respond similarly to increases in the moisture content of the adherends. The significance denoted in Table 13, in Appendix E, may be ascribed, in general, to the fact that a more uniform trend in strength loss with increasing moisture content was exhibited by the double back formulation (see Table 1).
3. Formulation x press time: -- This interaction is illustrated qualitatively in Figure 14. It is probably significant, due to the more pronounced rate of strength increase with press time evident in the double back adhesive. The difference between increase rates is ascribed to the lower gelatinization temperature and greater viscosity of the double back formulation.
4. Formulation x platen temperature: -- On the basis of Table 2, this interaction is significant at the 95 percent level. The effect is real, but not so pronounced as some of the coincident interactions, such as formulation x bonding pressure level. The single face and double back mixes at 160°F., 180°F. and 200°F. platen temperatures resulted in summarized breaking loads of 12.8, 20.5, and 27.3 lbs.;

and 16.4, 22.6, and 28.3 lbs., respectively. The differential response of the two formulations tends to decrease as the platen temperature level increases. In terms of the response of the respective formulations to temperature increase, a fairly uniform increase in bond strength is evident between temperature levels, except between 180°F. and 200°F. in the case of the double back mix. The decrease in the strength differential with increasing heat here may be attributed to the fact that the formulation is effectively gelatinized at the 180°F. level. The increase in strength with the increase to a 200°F. platen temperature here must be ascribed to a cause other than the gelatinization of the adhesive raw starch component, such as moisture migration. The significance of the interaction effect is directly related to the gel point and viscosity characteristics of the adhesive formulations, which tend to become less critical as platen temperature increases.

5. Formulation x platen pressure: -- Platen pressures of 6 and 12 lbs. per inch of bond yielded summarized average bond shear strengths of 17.4 and 23.1 lbs. with the single face formulation, and 20.5 and 24.3 lbs. with the double back variety. The response of the single face mix is somewhat greater to changes in platen pressure than the double back, and the strength differential between formulations at the 6 lb. level is twice that exhibited at 12 lbs. per inch of contact pressure. Either aspect of the interaction

effect is probably related to the fact that the influence of the higher gel point and greater fluidity of the single face formulation tends to become less significant at greater platen pressures.

6. Weight of adhesive spread x platen temperature: -- The relative significance of this interaction, in the frame of reference provided by Table 2, is somewhat less than those found significant at the 99 percent level. Average bond strengths at 160°F., 180°F. and 200°F. platen temperatures were 16.9, 24.0, and 29.3 lbs. for the standard spread, and 12.2, 14.0, and 26.4 lbs. for the heavy spread. The difference in the response of the two spread weights to changes in platen temperature levels are quite evident. The strength difference between temperature levels tends to decrease in the case of the standard spread, and increase abruptly with the heavier weight of applied adhesive. Considering the effects of adhesive gel point became dominant at 180°F. particularly with the double back mix which "pastes up" at this platen temperature level, some factor apparently inhibits a corresponding increase in strength with the heavier spread until a platen temperature of 200°F. is introduced. Again, the effect of the added moisture introduced by the heavier weight of adhesive during sample preparation is suspect. Where gelatinization does not occur under pressure the heavy spread may also tend to produce a more distinct interface with a thicker, weaker starch film.
7. Weight of adhesive spread x platen pressure: -- This

interaction is significant at the 99 percent level in Table 2, again denoting a high level of significance for the effect. Pressure levels of 6 and 12 lbs. per contact inch produced average bond strengths of 21.5 and 25.3 lbs., and 16.4 and 22.1 lbs. with the standard and heavy spread weights, respectively. The effect indicated suggests that the heavier adhesive spread responds more to changes in pressure level than the standard application, and that the strength differential between spread weights is greater at the lower bonding pressure level. The inference from either standpoint is that the higher pressure serves to force the heavy adhesive application out of the interface, improving bond quality. The degree of fiber association between adherends, and thinner, stronger film forming of the adhesive, may be more encouraged by an increase in pressure with the heavy spread. As noted in the discussion of other main factor and interaction effects, the additional moisture inherent to the heavy glue spread is suggested as a negative influence on the formation of strong adhesive bonds.

8. Weight of adhesive spread x moisture content: -- The interaction of these factors, which considers the concurrent behavior of the two prime determinents of moisture level at the bond interface, is significant at the 95 percent level. The range in paperboard moisture content levels; 6, 12 and 22 percent, resulted in average adhesive bond strengths of 29.6, 25.1 and 15.6 lbs. with the standard adhesive spread, and 24.2, 21.1 and 12.4 lbs. with the

heavy application. Considering these values, it is evident that the standard spread responds more to moisture content than the heavy, with the differences in bond strength between moisture content levels increasing with moisture content in the case of both weights of adhesive application. It may also be noted that the differential in bond strength between the respective spreads decreases as the material moisture content increases. The principal difference in the spreads, other factors remaining constant, is the amount of moisture present in the interface at the time of bond formation. The solids content of the adhesive should not tend to deteriorate the bond, presuming sufficient pressure is provided for intimate contact of the adherends. The comparatively lower sensitivity of the heavy spread to increasing moisture content levels consequently suggests that the moisture present in the adhesive is sufficient to mask the effects of water content in the adherends. In short, as the adherend moisture content decreases, the moisture in the heavier spread becomes increasingly the dominant factor in determining the differential in bond strength between spreads. The increasing capacity of the paper to absorb water at lower moisture contents is evidently not sufficient to accelerate the improvement in heavy spread bond strengths so as to render them comparable to those obtainable with the more standard adhesive application. Here the heavier spread may also detract from bond strength by producing thicker adhesive films between fiber surfaces.

9. Platen pressure and temperature: -- The interaction of these factors appears significant at the 99 percent level in Table 2. A bond formation pressure of 6 lbs. per contact inch produced average adhesive bond breaking loads of 13.2, 17.3 and 26.4 lbs. at platen temperatures of 160°F., 180°F. and 200°F., respectively. At the 12 lb. pressure level, the average bond strengths were 16.0, 25.8, and 29.3 lbs. for the 160°F., 180°F. and 200°F. platen temperature levels. Examining these values, it is evident that the difference between temperature levels tends to increase in the case of the 6 lb. bonding pressure, and decrease at the 12 lb. formation pressure. This response may be attributed to more efficient temperature conduction to the glue line at the higher pressure, producing more complete gelatinization in the adhesive, especially in the case of the double back formulation. Examining the breaking load averages, it is probable that the influence of adhesive gelatinization becomes dominant at the 180°F. level under 12 lbs. per contact inch pressure, and at a 200°F. platen temperature when a 6 lb. pressure level is employed. Relating the effect to corrugated board manufacture, increasing nip pressure will aid temperature transmission, resulting in improved gelatinization of the adhesive. After the raw starch adhesive component is gelled, the strength difference effected decreases, and probably tends to become constant. This response is in agreement with the results reported by Max (36) and McKee (32) in

regard to the effect of increasing nip pressure.

10. Platen pressure x moisture content: -- Significant at the 99 percent level, this interaction denotes a definite effect of platen pressure on specimen strength response to changes in adherend moisture content. At a bonding pressure of 6 lbs. per glue line inch, the summarized average breaking loads for samples bonded at 6, 12 and 22 percent moisture content were 24.8, 21.6 and 10.4 lbs. Comparable values for samples bonded under 12 lbs. pressure were 28.9, 26.4 and 17.5 lbs., respectively. The degree of bond strength loss is not pronounced at either pressure level as the material moisture content increases from 6 to 12 percent. With an increase from 12 to 22 percent, however, the strength loss is much greater in the case of the lower bending pressure. Without pressure to promote the transfer of heat to the glue line and facilitate cure, the effect of moisture in the adherends on the inhibition of bond formation is apparently increased appreciably. In analogy to the corrugated fiberboard bond, the effect of high paperboard moisture content levels on bond quality is probably more pronounced at low formation pressures.
11. Platen temperature x moisture content: -- The average bond shear strength values at 6, 12 and 22 percent moisture content levels in the adherends are summarized, in order, as follows:
 - a. 160°F. platen temperature - 24.5, 17.7, and 1.6 lbs.
 - b. 180°F. platen temperature - 25.5, 23.7, and 15.4 lbs.

c. 200°F. platen temperature - 30.7, 27.9, and 24.8 lbs.

As may be determined by examination of the average breaking load values, the improvement of bond strength with increased temperature is more pronounced at the higher moisture content levels. Conversely, as temperature level increases, the loss of bond strength with high moisture content tends to be minimized. The total effect is to indicate that moisture induces a negative response in bond strength, and heat at the time of bond formation will tend to reduce this influence. If the corrugated bonding process is considered, higher moisture contents in the adherend materials will tend to weaken the adhesive bonds, unless increased roll, nip, and preheater temperatures are introduced to offset the effect.

12. Second order interactions: -- The second order interactions are not discussed in detail, since in most cases the relationships involved can be better compared by directly referring to the sample averages shown for the various factor level combinations in Table 1. For the discussion of these second order interactions, the reader is referred to the revised analysis of variance presented as Table 14 in Appendix E.

In evaluating the second order interaction it is immediately noted that the response of bond shear strength to variations in platen temperature and pressure are similar with both formulations. This is evident since the subject interaction (F_xT_xP) is not significant. All other second

order interactions in the five-factor study display a high order of significance at the 99 percent confidence level. The immediate point of interest is why one interaction shows no significant effect, while all others definitely do. In this respect, the consideration of some common elements seems pertinent.

It is immediately evident that every other subject interaction involving the concurrent effect of three formulation factors contains at least one variable wherein the variation in factor level involves a change in moisture level at the adhesive bond during sample preparation. Both paperboard moisture content and weight of applied adhesive affect the amount of water present at the bond when it is cured, and one or both are considered in all interactions other than the only one evidencing a lack of significance. In short, where factors contributing moisture variation to the bond are segregated in analysis, significance is evident. Where the moisture effect is not considered, no significance is exhibited. It is now certain that moisture has some influence on bond strength beyond the inhibition of delivered temperature level at the glue bond, for the adhesive gel point serves to define the effect of formulation, and significance is evident in interactions that do not include formulation effects.

Further interpretation of interaction effects was not considered practicable, since the procedure becomes primarily one of comparing the average breaking load values for the adhesive bond assigned to the specific

factor level combinations presented in Table 1. This conclusion is emphasized by the significance of the fourth order interaction shown in Table 13. It should be noted that the practice of extending inferences drawn on high level interaction effects can often lead to erroneous and physically unrealistic conclusions. Such complex relationships are best examined by series of sequential investigations (14) (47).

General observations during sample evaluation. -- A number of qualitative results were observed during the conduct of the testing procedure, and deserve comment.

Some curiosity regarding the physical behavior of the starch film, as removed from its role as a bonding agent, was aroused. Specimens bonded at high moisture content levels or with the excessive adhesive spread exhibited a tendency, in some instances, to curl along the axis of the flute. Since this was the plane of greatest moisture response in the paper, concern was felt for possible "frozen" stresses in the paper (37). Measurement of a few of the liner elements, however, did not indicate any notable increase in the length dimension of the tabs; certainly not of sufficient magnitude to curl the specimen. It was therefore conjectured that the starch film was shrinking at the interface. This suggests a possible condition of residual stresses in the adhesive bond of corrugated board caused by the shrinkage of the starch film. The formation of a distinct interface is also implied, with direct contact between adherend fibers. This apparent shrinkage raised the question of the inherent strength of the starch film. An estimate was obtained by scanning the data from the five-factor study, and selecting values from factor groups where five or more specimens exhibited no paper failure.

In each group so selected, those values related to 0 paper failure were averaged, and an estimate of the shear strength of the starch adhesive film thus obtained. The strongest example of film, with 22.5 lb. breaking load, occurred in specimens bonded at 12 percent moisture content and 160°F. platen temperature, using a standard spread of double back adhesive, and glued at 12 lbs. per contact inch platen pressure. It was felt that this could possibly serve as a criterion of bond strength for some industrial applications. It could also serve as a basis of judgment in relating percent paper failure to shear failure in corrugated fiberboard adhesive bonds. Such application would answer the question, "Strong adhesive or weak paper?", where paper failure was high, but bond strength seemed marginal or low.

Some specimens bonded with heavy and light spreads were examined at random, to determine if any notable difference in bond width resulted from adhesive squeeze-out in the press. No distinct differences were evident with an optical pocket comparator. The same question arose regarding the lower viscosity of the single face adhesive mix. Specimens at the same spread level were examined in similar fashion, with no evident difference between single face and double back adhesive types.

The general appearance of the better quality bonds was quite similar to the typical adhesive bond in A-flute corrugated paperboard. The shoulder effect was in evidence with both formulations, exhibiting the typical appearance shown in Figure 11. In regard to general bond appearance, the experimental single-line bond seemed an adequate analogy to the adhesive bond found in typical converted material.

It was observed that all paper failure occurred in the liner element. Failure appeared to be what is sometimes referred to as "rolling

shear", or a twisting effect of the fibers out of the paper mat. In so far as could be determined with a hand lens, fibers were both broken off and pulled out of the paper matrix, suggesting that failure in the paper adherend is related to the cohesive strength of the material in tension, as well as shear. The forces inducing failure, however, tend to act in the plane of the adherend, rather than normal to it.

Analysis of the adhesive bonding mechanism in corrugated fiberboard. -- in attempting to relate the experimental results of this investigation to the bonding phenomenon as it exists in the manufacture of corrugated fiberboard, a critical examination of the bonding process is first in order.

In the corrugated bonding process, the adhesive is not actually a single entity, but an adhesive system. The nature of this system may be determined by examination of the formulations in Appendix D, but generally three principal elements are involved. Raw starch, or more accurately, ungelatinized starch is the critical component which permits the almost instant adhesion of the medium and liner at the point of contact. The cooked starch portion acts as a carrier for the raw starch element, and water imparts the proper fluidity to the paste. The adhesive is thus a colloidal sol, with raw starch suspended in a cooked starch dispersion in water, with sodium hydroxide added to adjust the gel point of the raw starch, and borax to maintain the proper fluidity of the mixture. The mixed system is constantly stirred and maintained at a minimum temperature, until it is fed onto the flutes in the corrugated medium by an applicator roll.

It is at this point that the description of the bonding

phenomenon, as found in the literature, seems to become inadequate. The following is presented as a description of the possible sequence of physical events as the bond is formed.

As the liner feeds under a heated roll and contacts the adhesive coated flute types of the medium, heat and some degree of pressure are applied to the adhesive and adherends as they form an interface. The amylose element in the raw starch (about 25 percent) gelatinizes, absorbing water rapidly. The fibers of the paper adherends come into intimate contact, and, especially at the single facer where pressures are high, begin to form fiber-to-fiber bonds, either as a result of inter-molecular forces or by the hydrogen bonds typical in paper. Which of these bonds is the more important is a matter of conjecture (2) (11) (30) (34) (40). (The more recent theories (40) suggest that hydrogen bonds are of minor importance in the inter-molecular bonding of cellulose). The cure of the bond is now completed, as the amylopectin element of the raw starch, the cooked starch carrier, and possibly the fiber entanglement between paper elements, lose water and bond by inter-molecular forces. It is in the latter stages of the bonding phenomenon that some areas of the literature seem at variance with the description above and the results of this study.

Werner (50) has stated that most bond strength is contributed by the raw starch component of the adhesive system, and that the carrier of cooked starch does not participate in the bond. There seems no logical reason to support this contention, since a cooked starch paste will gelatinize with moisture loss, though not "in situ" as the raw starch does. Cooked starch films are weaker than similar films formed from raw starch (29) (45), but it would seem reasonable that both the carrier and the

amylopectin component of the raw starch add some strength to the corrugated bond. The results illustrated in Figure 14, and the strength increases that may be noted in Table 1, for temperature levels beyond the gel point of the adhesive tend to support this conjecture.

Max (36) has stated that the water released by the carrier in bonding is absorbed by the raw starch as it swells and gelatinizes. This may be true of a portion of the adhesive water component, but the same source indicates that the "green" board does not exhibit the strength of cured board. In this statement, in fact, he terms the bond superficial at a point just off the corrugator. The results of this investigation, especially the evident interaction effects, have suggested repeatedly that the ultimate strength of the bond is closely related to moisture level at the bond at the time of cure, and to factors that effect the removal of water from the bond. In summary, the bond must benefit, and perhaps appreciably, by the contribution to bond strength made by the carrier, provided the means for water removal (low paper moisture content, high cure temperature, etc.) are provided.

The use of excessive adhesive application is definitely undesirable. It is not only uneconomical, but the adhesive bond is weakened when excessive spreads are employed. This may not be as critical on the double backer, where higher temperatures are often employed, but is certainly critical to the single face bond, where water must be removed very quickly to facilitate a high degree of cure.

A delicate balance between paper moisture content and applied heat apparently exists in corrugated board manufacture. A certain level of moisture content must be maintained to permit processing of the liner and medium material, for such defects as cracked board or poor flute

formation can result with low moisture content, but if it is too high, or the delivered heat is insufficient, a tendency toward loss in bond strength will result. This interaction will be more critical at the double backer operation, because of the probability of higher moisture content in the adherends, and the lower bonding pressure involved. In the formation of both single face and double back adhesive bonds, the interaction between moisture content of the adherends, applied pressure, and applied temperature is probably the single most important relationship. As determined in the analysis of the results of this study, these factors are intimately associated in their relationship to bond quality, and the level of one cannot be altered without consideration of the response of the others to such an alteration.

The most important characteristic of the adhesive is, no doubt, its gel point, or temperature of gelatinization. The importance of viscosity, stressed in the literature (3) (6) seems more related to the application of the adhesive than to its actual participation in the bond. It should be noted, however, that viscosity as it applies to the adhesive mix is related to gel point, presuming the proper proportion of adhesive components. Using a funnel viscometer as recommended (3), viscosity will be easier and more rapid to employ as a control technique than gel point in the production process. The degree of interaction with other variables detected in the five-factor study certainly recommends the use of distinct formulations for the single face and double back bonding procedures. (Note significant FxT and FxP interactions).

If the general trends in breaking load and degree of paper failure illustrated in Table 1 are accepted as criteria of bond strength, it appears that low paper moisture content, high temperatures, maximum pressures and

minimal adhesive spreads tend to encourage high quality adhesive bonds. If a single factor is to be maximized to offset other effects, it would appear that increasing temperature will yield the most satisfactory results. The most probable causes of poor adhesion are inadequate temperature and residual moisture, either from too high a paperboard moisture content or use of excessive amounts of adhesive. In addition, certain mechanical limitations of processing, such as paper runability, may limit the extent to which adhesion factors may be optimized.

The results of this study and the pertinent literature (11) (45) (48) indicate the most important element in the adhesion of corrugated fiberboard must be assumed to be the amylose-paper fiber (cellulose) bond, probably formed by inter-molecular forces and, to some extent, by hydrogen bonding. The other starch components of the adhesive system participate in the bond, but are dependent on water removal for complete cure. Some fiber-to-fiber bonding must occur, but the extent of its contribution to total interface strength is unknown.

Recommendations for further research. -- The results and analysis of this investigation suggest a number of potential areas of further research, both in the laboratory and on an industrial basis.

The development and successful use of a test specimen based on shearing forces on the adhesive bond in corrugated fiberboard suggests the possible development of a related test for industrial use. The test employed in this study has no application to multi-bond samples from formed board, but the principal may be employed. A sample of the same size and type as the common plywood shear specimen (1) could be cut from formed corrugated board, with the flutes parallel to the length of the sample.

By cutting the liner on alternate sides of the sample at points a predetermined distance apart, shear planes parallel to the plane of the adhesive bond can be formed. The medium can be removed from the ends of the specimen and spacer blocks inserted between the liner elements, so that the specimen can be mounted in the grips of a conventional tensile testing machine, and loaded to failure. The loads on the bond will not be entirely shearing in nature, as demonstrated by De Bruyne (15) and Yavorsky (52), but with the proper dimensions determined, a more realistic test than those in current use is possible. As discussed in the development of the glue line shear device, the peel and pin adhesion tests commonly employed at present impose tensile forces on the bond normal to the interface, while service loads appear to impart shear stresses to the interface, with the tensile forces in the adherends predominantly in the plane of the paper.

The contribution of fiber bonding, and the role of the raw and cooked starch adhesive components, in the adhesion of corrugated fiberboard should be studied quantitatively. The results of this investigation indicate that all have some part in bond formation, but their relative contribution should be studied. Some aspects of the fiber bonding phenomenon bear an interesting resemblance to "tack" bonding in certain types of synthetic and urethane rubber materials (4).

The relationship of adhesion variables to other corrugated paperboard defects, such as "wash boarding" should be evaluated. The movement of moisture and adhesive carrier element into the board is of particular interest, since any accessible cellulose polymer chains may be locally stiffened by these materials (35) (38). Such an effect could produce a considerable difference in the strength and hygroscopicity of

the liner in the bonded area.

In conclusion, an interesting area of industrial process research suggests itself through the prominence of interaction effects present in the adhesion of corrugated board. Such a process, where many significant factors are present and interacting, suggests the application of modern methods of systems analysis and automatic control (17). If a hypothetical bonding process is conceived, where only three formation variables are present and interacting, an example may be presented of such analysis and control.

Assume a single facer machine is operating, and only speed, paper moisture content, and temperature tend to vary. Other factors known to affect bond formation are presumed to be closely controlled. If some minimum can be set as a criterion of acceptable bond quality, a limited number of experiments with the machine will produce a relationship such as that illustrated in Figure 15. This type of figure, constructed by conventional analytical techniques such as those recommended by Davies (14) is a response surface. Any point on this response surface denotes a particular combination of machine speed, roll temperature, and paper moisture content that will produce, on the basis of past evaluation, the minimum acceptable bond strength. The surface itself describes the manner in which minimum acceptable bond strength, or any other specified level of bond strength, varies with the three formation factors as they interact on it. If the level of one or more of the factors changes, the surface denotes the extent to which the remaining factors must be adjusted to maintain minimum adhesive bond quality.

The application of modern instrumentation and feed-back systems will provide for the automatic sensing of the levels of the respective

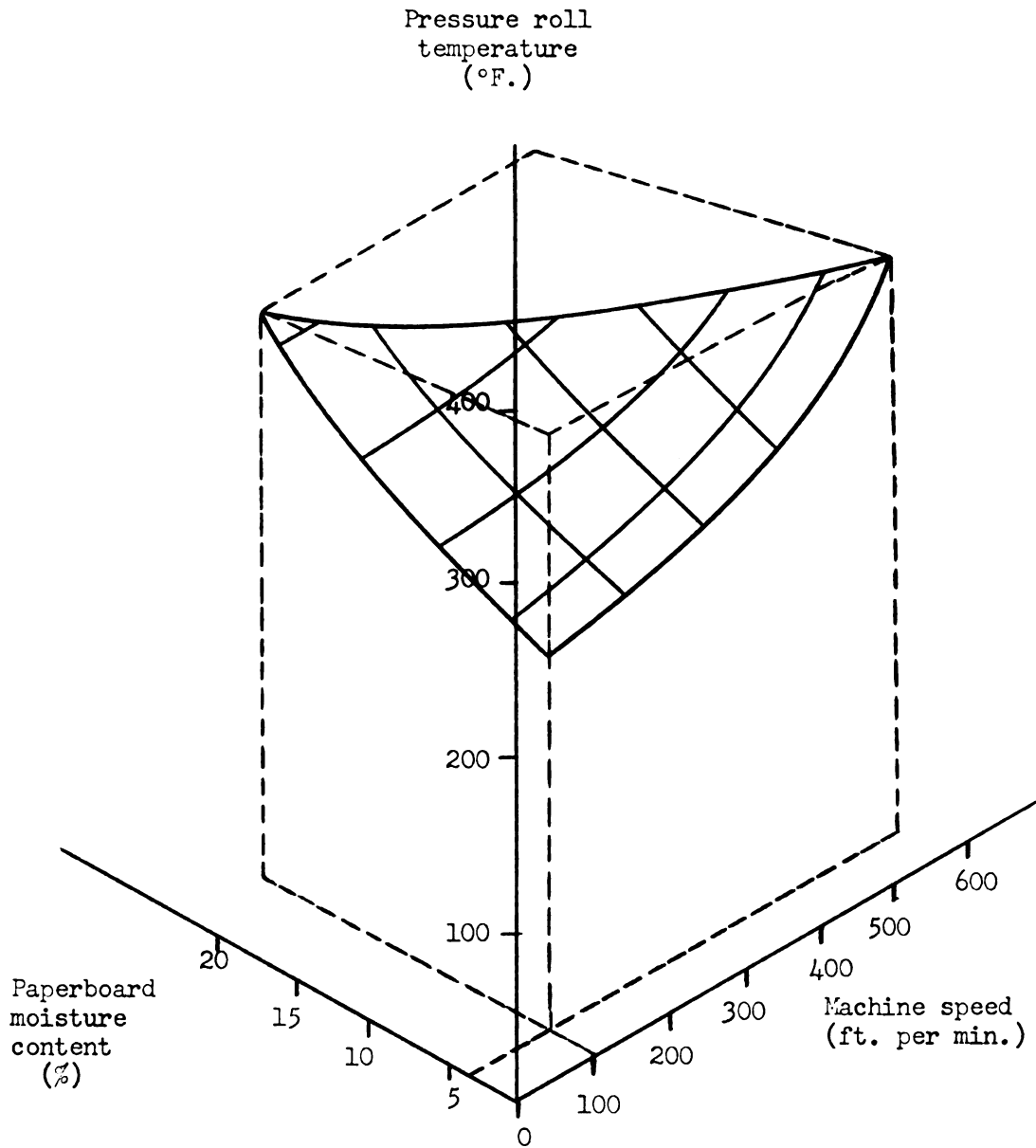


Figure 15. Hypothetical adhesive strength response surface for three bond formation variables in single facer adhesion.

factors, and their continuous adjustment to maintain the quality of the bond at the level described by the response surface. A departure from the conventional systems employed at present, where other factors are adjusted to conform to a set machine speed, would be to "sense" the level of the other variables, and adjust machine speed automatically to maintain adhesive bond quality. Such instrumentation could evaluate variable levels before the bonding event, and adjust speed to whatever level is required. The only way in which the operator could increase the machine speed would be through the manipulation of one or more of the other variables, such as increasing the temperature. Automatic systems would be capable, of course, of controlling a much more complicated relationship than the simple one illustrated in Figure 15.

CONCLUSIONS

Consistent with the limitations of this study, the following conclusions may be drawn:

1. Variation in all of the factors related to the adhesive bonding of corrugated fiberboard included in this investigation; bonding temperature, bonding pressure, moisture content of the adherends, duration of press time (or its industrial reciprocal, machine speed), weight of adhesive application, and adhesive formulation (as relates specifically to temperature of gelatinization and viscosity), will produce significant differences in the shear strength of the adhesive bond.
2. All of the adhesive bond formation factors enumerated in 1, above, react significantly with one another, with the following exceptions: The interaction of adhesive formulation with weight of applied adhesive; and the interaction of weight of applied adhesive, moisture content of the adherends, adhesive formulation, and bonding pressure. The relative magnitude of these interactions is not uniform, but tends to follow the magnitude of the main factors effects involved.
3. In evaluating the influence of the main factors enumerated in 1, above, or their interactions, on the nominal shear strength of the corrugated fiberboard adhesive bond, comparisons of factor levels must be made on the basis of specific factor level combinations.

4. Consistent with the limitations of the conversion process, the optimum conditions for the adhesive bonding of corrugated fiberboard tend to be: low moisture content of the adherends, high bonding pressures, high bonding temperatures, low machine speeds, and minimum weights of adhesive application. The specific level of any of these variables must be determined with regard to the specific levels of all others, due to significant interaction effects. The physical properties of the adhesive mix define the basis of the potential variation of other factors.
5. The starch adhesive is a colloidal sol, not a single element glue. The conversion of this sol to a solid film resembles the physico-chemical process of coagulation, rather than drying or polymerization alone. The nature of the adhesive system is extremely complex, and predictions regarding its behavior must be made with caution.
6. At least three distinct adhesion phenomena are present in corrugated fiberboard: fiber-to-fiber bonding of the paper adherends; raw starch-to-fiber bonds, produced by gelatinization; and cooked starch-to-fiber bonds, produced by water loss.
7. The quantitative contribution of the above bond types to total adhesive bond strength is undetermined. The raw starch-to-fiber bonds are thought to be the most important, and the fiber-to-fiber bonds the least, in regard to total bond strength.
8. The cooked starch adhesive component should be considered

a participant in the adhesive bond, in addition to its primary function as a carrier for the raw starch adhesive ingredient.

9. The removal of water from the adhesive bond is apparantly necessary to achieve optimum shear strength at the interface. The degree of water removal required is a function of the amount of water present at the bond during formation, in excess of that which can be absorbed by the raw starch adhesive component in gelatinization.
10. The corrugated single face and double back bonding processes are specifically different in nature, requiring different levels of critical adhesion factors, and forming distinctly different types of adhesive bond.
11. The evaluation of a simulated corrugated fiberboard adhesive bond was satisfactorily performed, employing a device designed to apply shear loads to the nominal bond interface. The testing of conventional bonds in shear as well as tension should be considered, based on theoretic analysis, the results of comparative evaluation studies, and the apparant nature of the forces imposed on the bond under service conditions.

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APPENDIX A

SECTIONING AND PHOTOMICROGRAPHY OF ADHESIVE BONDS IN CORRUGATED FIBERBOARD

PREPARATION OF PHOTOMICROGRAPHS OF CORRUGATED FIBERBOARD

Introduction. -- An initial aspect of examining adhesion in corrugated fiberboard was the study of **typical** bond lines under magnification, to determine the characteristics of their physical appearance. It was assumed that this examination would reveal evidence of the effect of some of the more critical formation variables such as pressure, and the general physical state of the adherends and adhesive at the bond interface.

Investigative procedure. -- The procedure by which thin sections of corrugated paperboard were prepared for microscopic examination is described in summary on page 109. This procedure was based on notes made available to the writer during a course of instruction under Dr. R. J. Raphael of the School of Packaging at Michigan State University, and the cited references.

Discussion of results and observations. -- Two principal points of difficulty were encountered in the preparation of material for examination: the writer was unable to secure satisfactory sections less than 20 μ thick, all of the paraffin embedding material could not be successfully removed without destruction of the section. These facts, together with the available time that could be reasonably devoted to this preliminary phase of the overall study, prevented a fiber-by-fiber examination of the bond. The gross microscopic features of the bond were readily discernable, however, and microphotographs of these are presented in Figure 11 and Figure 12 on pages 42 and 43. The effect of the greater bonding pressure used in single face bonding is quite evident, with the medium element

distinctly embedded into the liner material. The double back bond, where very low pressure is employed, does not exhibit this characteristic. There did not appear to be any residual deformation of medium or liner as a result of the pressure used in the single face operation, but subsequent cyclic moisture content conditions could have concealed such evidence. The lack of a distinct interface, with the adherends tending to form a contexture is evident, due to the degree of intimate contact between the paper elements.

The starch film can be readily discerned and seems quite continuous in both bonds, with the characteristic concavity present where the film has shrunk in the "shoulder" structure joining the liner and the curved medium element. The double back bond appears to be more dependent than the single face on the film for a "bridging" effect, though there definitely is some fiber-to-fiber contact in the former. The degree of intimate contact exhibited by the two bonds supports the contention that, presuming some fiber-to-fiber adhesion does occur, it is greater at the single face interface because of the embedding effect. While both bonds may gain strength from the shoulder effect caused by adhesive flow away from the contact line, it appears more important to the double back bond because it constitutes a larger part of this structure than its counterpart found in the single face variety.

While not evident in the illustrations, individual starch particles were observed under direct examination, probably consisting of a conglomerate of actual starch granules. These particles were apparent well into the structure of the medium and liner materials in both bond types, especially in the region of the flute tip. In contrast to the embedding effect observed in the single face bond, the liner in the

double back bond appeared to actually permit some deformation of surface fibers across the interface to maintain contact with the medium. This may indicate that high shrinkage in the starch film at the double back interface actually tends to destroy the fiber-to-fiber relationship if the flexibility and length of the liner fibers are not sufficient to maintain contact.

SECTIONING OF CORRUGATED FIBERBOARD

A small piece of A-flute corrugated fiberboard, 1/2-in. wide and 1/4-in. along the flute, was cut from converted material manufactured by the Ohio Boxboard Company. After conditioning for a period of about one month at $50 \pm 2\%$ r.h. and $72 \pm 3^\circ\text{F}$. The following procedural sequence was then followed in preparing the material for microscopic examination. The time periods indicated are approximate.

1. Dehydration series.

- a. 70 percent ethyl alcohol - 10 minutes.
- b. 95 percent ethyl alcohol - 10 minutes.
- c. 50 percent absolute ethyl alcohol, 50 percent xylene - 3 changes, 10 minutes each.
- d. Xylene - 10 minutes.

2. Impregnation and embedding.

- a. Thirty minutes in saturated solution of paraffin ($50-52^\circ\text{C}$. m.p.) in 50 percent absolute ethyl alcohol, 50 percent xylene.
- b. Thirty minutes in melted paraffin ($50-52^\circ\text{C}$. m.p.).
- c. Thirty minutes in melted paraffin ($53-55^\circ\text{C}$. m.p.).
- d. Thirty minutes in melted paraffin ($56-58^\circ\text{C}$. m.p.).
- e. Embed in paraffin ($56-58^\circ\text{C}$. m.p.).

3. Sectioning.

Sections for microscopic examination were cut on a heavy duty type sliding microtome to an approximate thickness of $20-25\mu$. A film of 2 percent collodion was applied to the surface of the material and allowed to dry, to facilitate

sectioning.

4. Slide preparation and examination.

Sections placed on microscope slides were washed in alcohol and ether (50-50 sol.), to remove the collodion, and then in xylene to remove the paraffin. Sections for immediate examination were mounted in Karo or Permount. Sections for photomicrography were flooded with alcohol and covered with cover-glasses. They were then transported directly to the Photomicrography laborabory of the Michigan State University Botany Dept., where the embedding material was removed and the photographs taken.

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APPENDIX B

PRELIMINARY ADHESIVE BOND

STRENGTH TESTS

INTRODUCTION TO PRELIMINARY TESTS

Purpose of the tests. -- The general purpose of this evaluation sequence was to provide procedural information for the conduct of the principal portion of the investigation. It was necessary to establish certain aspects of technique related to the glue line shear test that had been developed (see page 26) and verify the use of a shear type test rather than one based on tensile loading.

General procedure. -- After a series of trial and error experiments using the device, various adhesives and specimen types, certain aspects of procedure were standardized and became common to all the preliminary tests.

The same paper materials employed in the main factor studies were used in these exploratory tests; 16 pt., nominal 69 lb. basis weight Kraft liner, and 9 pt., nominal 26 lb. basis weight semichemical medium. The same size medium element, 3-1/2-in. wide by 2-in. in the flute direction, with the machine direction normal to the flute and glue line, was used throughout the tests. The adhesive selected was a commercially prepared polyvinyl acetate resin emulsion, rather than one of the starch systems incorporated in the principal portion of the investigation. This adhesive was used, since at the time of the preliminary studies the behavioral characteristics of the starch formulations in the test situation were unknown. The emulsion cures under heat with water loss, in a manner similar to the reaction of a starch glue, and it displayed the practical advantages of very rapid tack properties and uniform flow characteristics, and required no preparation other than stirring. A uniform spread of

approximately 0.1 gr. per inch of glue line was maintained by application of the adhesive with a medicine dropper, the tip of which had been trimmed to permit the desired flow.

The specimens were bonded under conditions of $50 \pm 2\%$ r.h. and $72 \pm 3^{\circ}\text{F.}$, and stored for a period of two weeks before and 48 hours after fabrication in the same environment. The environmental conditions at the time of test were noted for each of the respective portions of the investigation. Employing the press device shown in Figure 8 and Figure 9 on pages 37 and 38, all preliminary test specimens were bonded with a platen temperature of $230 \pm 2^{\circ}\text{F.}$ under 10 lbs. total pressure, for a press time of 30 seconds.

The preliminary tests were conducted using the test fixtures shown in Figure 5 and Figure 6 on pages 32 and 34. The shear test specimens were loaded in a National Forge compression testing machine, with the exception of the study which compared the shear and tensile testing techniques.

The preliminary tests are described in sequence, rather than as a group, for purposes of clarity and to emphasize the chronological nature of the studies as a source of supporting information.

DETERMINATION OF LINER TAB DIMENSIONS

Purpose. -- The purpose of the test was to establish the optimum dimensions for the size of the shear specimen liner tab, so that failure at the bond interface, rather than in the liner material, might be assured.

Materials and methods. -- The samples were prepared as described in the general procedure, incorporating tab widths of 1, 1-1/2, and 2-in. widths and a 2-in. bond length. The samples were tested at a 0.2-in. per minute rate of deformation under conditions of 52% r.h. and 76°F.

Results. -- The results of the investigation are summarized below.

Table 3. RESULTS OF TESTS OF SHEAR SPECIMENS TO DETERMINE LINER TAB WIDTH

Liner tab width (inches)	Breaking load in pounds		
	1	1-1/2	2
Test data	20.2, 19.3, 18.3, 18.3, 20.1, 18.0, 20.7, 18.8, 18.0, 22.3	25.0, 24.1, 24.1, 26.7, 28.0, 26.2, 25.5, 27.2, 27.1, 24.8	27.9, 26.3, 24.1, 26.1, 27.4, 24.2, 24.1, 24.6, 24.5, 25.4
Mean	20.1	27.1	26.7
Range	4.3	3.9	3.8
No. of bond failures	0	9	10

Discussion of results and conclusions. -- All samples responded in a generally satisfactory manner. The tab failure noted in the 1-1/2-in. width sample was ascribed to an discernable defect in the paper. It was concluded that a liner tab width of 1-1/2-in. would be satisfactory for further testing.

DETERMINATION OF TEST BOND LENGTH

Purpose. -- The purpose of the test was to establish the optimum dimension for the length of glue bond in the shear specimen, so that, presuming a 1-1/2-in. liner tab width, failure at the interface would be assured.

Materials and methods. -- All samples were prepared in accordance with the general procedure previously outlined. Samples with glue bond lengths of 1/4, 1/2, 3/4, 1, 1-1/2 and 2-in. were evaluated for breaking load at failure, and observed for general behavior during loading. All specimens were loaded in a National Forge compression testing machine at 0.2-in. per minute rate of deformation, in an environment of 76°F. and 44%r.h.

Results. -- The results of the tests are summarized in Table 4 on page 116.

Discussion of results and conclusions. -- The results indicate that the 2-in. length is unsatisfactory, since failure at the bond is not certain. Considering the values of range breaking loads, it is obvious that the relative variability will be least for the 1-1/2-in. bond length. A few specimens of each bond length were tested qualitatively, with a 0.001 Ames gage positioned to indicate deformation of the medium normal to the flute at the index mark shown in Figure 1 on page 28. The specimens were given some freedom of movement by inserting small pieces of 0.010 shim stock under the fixture restraint plates at the lower edge of the specimen. Deformation in the medium, i.e., a tendency to pull away from the fixture at the index mark, was noted only with the 2-in. bond length. This

Table 4. RESULTS OF TESTS OF SHEAR SPECIMENS TO DETERMINE ADHESIVE BOND LENGTH

Adhesive bond length (inches)	Breaking load in pounds					
	1/4	1/2	3/4	1	1-1/2	2
Test data	3.4	9.5	16.1	16.9	21.5	27.4
	3.1	9.7	16.4	18.2	21.4	28.6
	2.8	8.5	16.0	19.4	20.8	29.5
	2.5	10.6	15.9	17.6	21.4	27.4
	2.5	9.8	15.1	17.1	20.0	29.2
	3.4	10.6	14.9	19.0	24.0	28.1
	4.1	11.0	17.3	18.4	20.8	28.3
	1.0	10.4	15.3	18.1	22.6	28.3
	2.1	9.3	16.2	17.3	20.6	27.8
	3.7	8.9	16.2	19.1	27.0	25.9
Mean	4.1	10.0	17.1	19.3	22.7	29.3
Range	3.1	2.5	2.4	2.5	2.6	3.6
No. of bond failures	10	10	10	10	10	6

suggested that the tendency for the mechanical moment caused by the liner-medium couple to be effective was present only with the 2-in. glue line. Some wrinkling of the medium was noted in all specimens.

It was concluded that a 1-1/2-in. glue line, used in conjunction with a liner tab width of 1-1/2-in., would be the most satisfactory for use in the main factor investigation.

DETERMINATION OF THE EFFECT OF LINER TAB ALIGNMENT

Purpose. -- In planning the main factor experiments, it soon became evident that the number of specimens involved would require a rapid but reliable procedure for bonding the specimen, removing it from the medium support, and accurately re-mounting it for testing. By the use of center lines at the edge of the medium and along the length of the liner element, and alignment index marks on the medium support fixture and sash section of the test device, it was evident that repeated alignment of the sample within 5 degrees of true center was possible. The subject test was therefore conducted to evaluate the effect on breaking load of misalignment of the bond line 5 degrees to left and right of the true centerline.

Materials and methods. -- The specimens were prepared in accordance with the general procedure previously described. As indicated by prior test results, a liner tab width and bond length of 1-1/2-in. were used. The specimen elements, both medium and liner, were selected from the paper stock in groups of three adjacent pieces, taken in the cross-machine direction of the paper. Particular care was taken that in bonding, the centerline of the liner tab was aligned with the flute direction, as closely as could be measured with a straight edge. Subsequently, when the samples were tested, one sample from each group was remounted on the fixture as it was bonded, i.e., true with the line of action down the apex of the medium support and the indexed center of the sash, as shown in Figure 3 on page 30. One sample of the same group was mounted with the tab deliberately misaligned 5 degrees to the left, and one with the tab 5 degrees to the right,

as previously established by index marks on the sash. Replicating the procedure fifteen times, the specimens were failed in the shear device as previously described. Load application was with a National Forge compression testing machine operated at 0.20-in. per minute. Environmental conditions at test were 69% r.h. and 79°F. The results of the tests were compared by conventional statistical techniques (47) to evaluate the effect of the misalignment.

Results. -- The results of the investigation are summarized in Table 5 on page 120.

Discussion of results and conclusions. -- No tab failure was noted in any samples, and general behavior of the specimens and device was satisfactory. The analysis of the results indicated that up to 5 degrees misalignment of the sample at test could be tolerated. Since index marks on the fixture permitted rapid use of the device with actual alignment well within these limits, the use of the technique was adjudged satisfactory for the main factor tests. The satisfactory general behavior of the samples and the absence of any evident alignment problems suggested the inletting of the medium for the wing screws of the restraint plates. Six extra samples from the test array were failed, with no apparant movement of the medium under load. Since these inlets, shown in the samples in Figure 13, greatly facilitated the mounting of the test specimens, the procedure was incorporated in all subsequent preliminary tests, and in the main factor investigations. In previous tests, and in the subject test, small holes had been made in the medium to permit access for the wing screws. The use of inlets was more rapid, and reduced the amount of handling the bonded specimen was subjected to in attachment to the test device.

Table 5. RESULTS OF SAMPLE MISALIGNMENT ON THE BREAKING STRENGTH OF SHEAR SPECIMENS.

Specimen misalignment (degrees)	Breaking load in pounds		
	5 Left	0	5 Right
Test data	21.0	18.0	16.7
(paired specimens	20.0	17.5	17.6
in order)	16.8	17.5	17.9
	17.8	20.3	19.4
	19.4	18.9	20.1
	20.2	19.5	17.1
	19.9	19.4	19.9
	19.8	18.6	17.7
	17.6	20.3	20.9
	18.4	20.8	19.1
	17.1	19.0	16.3
	20.8	20.0	20.3
	17.2	18.0	18.8
	17.8	20.1	17.5
	18.7	19.1	18.0
Mean	18.8	19.1	18.5
Standard deviation	1.5	1.1	1.4
Statistical comparison by "T" test	No significant difference for 5 left vrs. 0, 5 right vrs. 0 at the 90% level of confidence (47).		

COMPARATIVE DETERMINATION OF LINER TAB TENSILE STRENGTH

Purpose. -- The purpose of the study was to determine the relationship between the delivered load to the liner tab, as imposed by the glue line shear device, and as measured by a standard and accepted evaluation method. If the load system that exists in the shear device functions as presupposed, the measured load delivered to the top of the shear device should be transmitted directly to the cross section of the liner tab, producing a tensile stress in the tab as it is restrained on one end by the adhesive bond and on the other by the restraint plate of the sash section of the shear device. If the load sustained by the liner, when failed in the shear device, proved radically different than when the material is tested for tensile strength in the more conventional manner, the immediate inference would be that the measure of the load by the test machine recording device is erroneous in terms of the load sustained by the glue line. A force component imposed on the glue line outside the plane of the line of action of the adhesive bond and the sash of the device would be a serious disadvantage in the use of the technique.

Materials and methods. -- The shear specimens were prepared in accordance with the previously described general procedure. A paired group experimental design was employed, taking the liner tab elements by adjacent pairs, and assigning one of each pair to the shear test and one to the conventional tensile test. The liner tab segment of the shear specimens was 1-in. in width, to correspond with the cross section of standard tensile specimen, and a 2-in. adhesive bond was used, to insure failure in the tab material. In all liner elements, the machine direction

of the paperboard was normal to the direction of load application. The tensile tests were conducted in accordance with ASTM Standard Procedure D328-48 in a Schopper pendulum-type test machine operated at 4-in. per minute, with a jaw spacing of 3-3/4-in. The shear specimens were loaded in the shear test device, employing a National Forge compression testing machine at 1.0-in. per minute rate of deformation. Both tests were conducted under conditions of 54% r.h. and 74°F. The average loads at failure by the two methods were subsequently compared by standard statistical techniques.

Results. -- The results of the comparison tests are summarized in Table 6.

Table 6. COMPARISON OF THE TENSILE STRENGTH OF LINER MATERIAL AS DETERMINED BY TWO TEST METHODS

Test device used	Breaking load in pounds	
	Schopper machine	Glue line shear device
Test data	20.0, 19.3, 19.2, 18.3, 19.0, 20.0, 19.4, 19.4, 18.6, 19.0, 18.0, 19.2, 20.0, 18.8, 19.0, 20.5, 19.5, 20.0, 19.8, 19.0	20.0, 20.0, 20.0, 19.0, 20.0, 18.0, 18.5, 20.5, 20.0, 20.5, 20.0, 17.5, 21.0, 20.5, 20.0, 21.0, 20.0, 17.5, 20.0, 20.5
Mean	19.3	19.7
Standard deviation	.7	1.0
Statistical comparison by "T" test	No significant difference between methods at the 90% level of confidence.	

Discussion of results and conclusions. -- The results of the investigation clearly indicated that the two test methods measured load at failure in the liner material with no apparant difference. The immediate conclusion was that the recorded load delivered to the top of the sash of the shear device was transmitted to the liner element, and therefore the adhesive bond, without significant alteration. Provided care in specimen mounting and alignment was observed, direct loading parallel to the bond and 0.008-in. from it (one-half the liner thickness) could be assumed, tending to verify the concept of the test technique. The failure level of the liner by both techniques seemed low for the basis weight and caliper of the paperboard involved, but later check tests appeared to verify the values. Since direct comparison of paired specimens tested by the respective methods was involved, however, the inferences regarding differences were presumed justified.

BASELINE STUDY OF SHEAR AND TENSILE BOND TEST METHODS

Purpose. -- The object of this investigation was to establish the comparative quality of the proposed shear test and a tensile test technique as criteria in the evaluation of single adhesive bonds similar to those characteristic of A-flute corrugated fiberboard. The two techniques are qualitatively described and illustrated in the Experimental Procedure section of the main factor study.

Materials and methods. -- The material for use with both test methods was selected by cutting specimen elements as adjacent pairs, assigning one element to the shear test sample, and one to the tensile test group. The shear specimens were prepared in accordance with the previously described general procedure, using a 1-1/2-in. liner tab width and a 1-1/2-in. glue line. It had been observed in the prior preliminary tests and trial-and-error investigations with the device, that various aspects of the bonding technique might be altered to improve bond strength. These supposed improvements included more care in handling the specimen elements, "breaking" the medium by pulling it over a rounded edge a few times to improve fit on the support fixture, deletion of the pencil line on the medium bond interface to mark the center line, care that the rough side of both medium and liner were at the interface, and permitting the adhesive "squeeze out" to remain on the specimen rather than wiping it away after removal from the press. The removal of excess adhesive had been practiced in the previous preliminary tests for the sake of uniformity, but it was felt that it should remain on the specimens in the main factor study, to simulate the "shoulder" buildup of adhesive

characteristics in production-run corrugated board. The tensile specimens were bonded in a manner identical to the shear specimens, the difference being in the geometry of the former. In the tensile specimen, the 1-1/2-in. bond was centered in the 2-in. long medium element and a 2 x 2-in. piece of liner material. After 24 hours the liner was, in turn bonded with a neoprene contact cement to a drilled wood block, yielding the finished glue joint specimen illustrated in Figure 10 on page 40. In all specimens the machine direction of the paper materials was normal to the adhesive bond. The mode of testing of the tensile specimen is illustrated in Figure 7 on page 35. Both samples were tested in a Baldwin Emery universal testing machine fitted with a Baldwin SR-4, 500 lb. capacity load cell. All tests were conducted at 0.50-in. per minute rate of deformation under conditions of 54% r.h. and 72°F.

Results. -- The results of the tests are summarized in Table 7, below.

Table 7. RESULTS OF TESTS OF ADHESIVE BOND STRENGTH BY SHEAR AND TENSILE TEST METHODS

Test method	Breaking load in pounds	
	Tensile system	Glue line shear device
Test data	11.8, 12.0, 13.0, 13.0, 12.8, 13.2, 12.6, 13.4, 12.6, 13.8, 11.8, 12.0, 11.4, 11.8, 13.2, 12.4, 12.8, 12.0, 13.0, 11.8	31.4, 30.4, 29.8, 29.0, 32.0, 30.0, 31.0, 32.8, 29.8, 32.4, 31.0, 30.6, 30.8, 31.8, 29.6, 28.8, 32.0, 32.2, 31.4, 30.4
Mean	12.5	30.9
Standard deviation	0.7	1.2
Coefficient of variation	5.3%	3.8%

Discussion of results and conclusions. -- Examination of the statistical information available from the tests reveals that both methods evidence satisfactory behavior. The coefficient of variation, a measure of relative variability, and therefore reliability, is somewhat smaller for the shear test, but is quite acceptable for both methods. The tensile test technique appears quite satisfactory as a method of evaluating single bonds in a paper structure analogous to corrugated fiberboard. Factors favoring the use of the shear test method are, in addition to the theoretical justifications previously advanced and the results of the box failure field study, the relatively greater magnitude of the shear test results, suggesting greater potential range for variation, and its comparative simplicity and the ease of specimen preparation and manipulation. The evident increase in the magnitude of failure loads in shear may be ascribed to the development of the adhesive "shoulder" at the bond interface, and the general improvements in bonding technique employed. All failure was in the paper rather than at the bond interface, and entirely in the liner material in the case of the shear test. The bond width developed in the shear test specimens was measured, and found to be 0.08-0.10-in.

DETERMINATION OF THE RESPONSE LEVEL OF SHEAR AND TENSILE ADHESIVE BOND TEST TECHNIQUES

Purpose. -- The purpose of the study was to determine the relative response level, or sensitivity to strength variations in the adhesive bond, of the two subject evaluation methods. A high level of response or sensitivity, is an advantage in utilizing such test methods as a quantitative criteria of adhesive bond quality.

Materials and methods. -- The specimens for the investigation were prepared and tested as described in the general procedure and in the preliminary tests to evaluate the shear and tensile bond test techniques. In order to determine response to a defect in adhesive bond, a weak or discontinuous bond was simulated by controlled gaps in the glue line. These discontinuities, centered in the interface, were 1/4, 1/2, 3/4 and 1-in. in length. Ten specimens of each gap type were evaluated, employing the shear and tensile methods of testing with the Baldwin Emery testing machine, at 0.50-in. per minute rate of deformation as previously described (page 125).

Results. -- The results of the investigation are summarized in Table 8 on page 128.

Discussion of results and conclusions. -- In comparing the response of the two subject methods to discontinuities in the adhesive bond by the coefficient of variation values, it is immediately evident that the shear test exhibits a more uniform response to changes in the glue line. Both techniques show response to gaps in the adhesive bond, but

Table 8. STRENGTH OF DISCONTINUOUS ADHESIVE BONDS IN SPECIMENS
TESTED IN SHEAR AND TENSION

Gap length (in.)	Tested in tension		
	Average breaking load (lbs.)	Standard deviation	Coefficient of variation
1/4	10.5	0.8	7.6%
1/2	7.0	0.5	7.1%
3/4	5.8	0.2	4.1%
1	4.9	0.2	4.5%

Gap length (in.)	Tested in shear		
	Average breaking load (lbs.)	Standard deviation	Coefficient of variation
1/4	30.8	1.3	4.4%
1/2	26.3	0.6	2.3%
3/4	24.5	0.8	3.3%
1	21.1	0.9	4.3%

the rate of change in shear strength appears more uniform and greater in magnitude, with greater reliability in this response, than does the comparable tensile test. The tensile test also appears to exhibit some tendency to become insensitive to changes in bond quality with larger defects, as compared to the shear method.

APPENDIX C

STUDY OF FIBER BONDING IN
CORRUGATED FIBERBOARD

STUDY OF FIBER BONDING EFFECTS IN CORRUGATED FIBERBOARD

Purpose. -- The object of this investigation was to verify the existence of direct fiber-to-fiber adhesion between liner and corrugated medium materials brought into intimate contact under conditions analogous to the conversion process. If such bonding could be achieved, it was intended that some measurement be made of the strength of such bonds. The problem was, in essence, to achieve a bond directly between liner and medium materials without an adhesive.

Materials and methods. -- The same materials incorporated in the main factor studies; 16 pt., nominal 69 lb. basis weight Kraft liner and 9 pt., nominal 26 lb. basis weight semichemical medium, were used in the fiber bonding study. The specimen medium and liner components were taken at random from paperboard stock that had been conditioned for a minimum of several weeks at $50 \pm 2\%$ r.h. and $72 \pm 3^{\circ}\text{F}$. The specimen medium element was 3-1/2-in. wide by 2-in. along the flute, and the liner tab 2-in. wide and 4-in. in length.

The samples were bonded in the press device illustrated on page 37, and used for the main factor study. A press time of 30 seconds with various platen temperatures and pressures, was used. The lap, or proposed bond length, was 2-in., the full width of the medium element. The general procedure followed was similar to that described on page 53 and that employed in the preliminary bond strength tests, with the notable exception that no adhesive bonding agent was employed. The medium was stretched over the medium support fixture of the shear test device and held in place by spring clips. The liner was placed along the flute

surface, and with the fixture placed in the press device, the upper press platen applied heat and pressure to the materials. All tests were conducted under conditions of $62 \pm 4\%$ r.h. and $75 \pm 3^\circ\text{F}$. Samples conditioned to high or low moisture contents were protected by a polyethylene bag during the test procedures.

Experimental procedure and results. -- The various bonding trials were conducted as a series of sequential tests, and are presented below in chronological order.

1. A series of 10 specimens at approximately 6 percent moisture content were pressed at ambient temperature under pressure of 10 lbs. per inch of contact. No bond resulted.
2. A series of 5 specimens at approximately 21 percent moisture content were pressed at ambient temperature under a pressure of 5 lbs. per inch of contact. No bond resulted.
3. A series of 5 specimens with a moisture content of approximately 21 percent were pressed under 10 lbs. per inch of contact pressure with a press platen temperature of 200°F . All specimens showed some evidence of adhesion, and four formed definite bonds (sufficient to permit removal from the fixture). These bonds were quite fragile, however, and failed without loading after 24 hrs. conditioning at $50 \pm 2\%$ r.h. and $72 \pm 3^\circ\text{F}$.
4. A series of 5 specimens with a moisture content of approximately 21 percent were pressed with a platen temperature

of 200°F. and a contact pressure of 5 lbs. per inch. No bond resulted.

5. A series of 5 specimens with a moisture content of 13 percent were pressed under 10 lbs. per contact inch pressure with a platen temperature 200°F. No bond resulted.
6. A series of 5 specimens at a moisture content of 13 percent were pressed under 5 lbs. per contact inch pressure with a platen temperature of 200°F. No bond resulted.
7. A series of 5 specimens at 21 percent moisture content were pressed with a platen temperature of 180°F. and a contact pressure of 5 lbs. per inch. No bond resulted.
8. A series of 10 specimens at 13 percent moisture content were pressed under a pressure of 10 lbs. per inch of contact pressure with a platen temperature of 180°F. All specimens bonded, and eight permitted removal from the support fixture. The bonds remained intact, but were too weak for shear or tension test measurements. The test was replicated once, with all specimens again exhibiting bonds that permitted handling.

In the test sequence described above, the moisture content of the paperboard was determined by ASTM methods applied to extra specimens, and the platen temperature measured directly with a potentiometer.

Discussion of test sequence and conclusions. -- The tests are described in sequence, rather than summarized as a table, to emphasize the fact that, despite common controlled conditions of treatment, the

the effect of unknown and uncontrolled variables is highly suspect. While the results of the sequence are hardly definitive, the conditions under which bonding did occur permit some reasonable inferences regarding the fiber bonding phenomenon.

As is evident from the results of the investigation, a physical situation similar to the fabrication of corrugated paperboard does permit some fiber-to-fiber adhesion, presuming the proper relationship between applied temperature, pressure, and the moisture content of the paper. There is an apparant necessity for pressure to provide sufficient contact between the fibers of the adjacent material elements, and the adhesive must not be present at the point of contact. In this study, the materials involved evidently require a minimum pressure of 10 lbs. per contact inch to permit fiber-to-fiber bond formation. A minimum level of moisture in the paper is apparantly required, probably to permit sufficient deformation of the materials into intimate contact, and to provide the necessary hydrogen and hydroxyl groups for the water cross-linkages common in paper. There is an apparant balance necessary between moisture content and applied temperature level, however. An adequate moisture level is necessary, but sufficient heat is evidently required to facilitate the removal or migration of this moisture during the particular dwell time involved.

It appears certain that, considering the moisture content levels of the paperboard during the conventional corrugated conversion process and the temperature levels required at the bond for gelatinization of the adhesive, some direct fiber-to-fiber adhesion does occur in the adhesion of corrugated board. The extent to which this phenomenon contributes to the total strength of the bond is indeterminate from the

results of this study, but the inference is that it is of secondary importance. The much higher pressure levels and shorter dwell time encountered in the conversion process render this only a logical conjecture, however. A very definite time-temperature-pressure-moisture content interaction is assuredly present, and conclusions concerning the degree of fiber bonding in standard corrugated fiberboard must be viewed with caution.

APPENDIX D

ADHESIVE FORMULATION

FORMULATION AND PREPARATION OF STARCH ADHESIVE

The following formulations and the starch adhesive component therein were supplied by the Stein Hall Company of New York. The formulations are designed to duplicate, in laboratory scale quantities, the same formulations recommended for use in the production of corrugated fiberboard.

General purpose formulation

1. Water - 500 gr.
2. Add corn starch - 50 gr.
3. Add 10 percent NaOH solution - 80 gr.
4. Heat at 71°C. for 10 minutes
5. Add water - 800 gr.
6. Mix for 5 minutes
7. Add borax, 10 mol. Hyd. - 9 gr.
8. Add corn starch - 300 gr.
9. Mix thoroughly 15 minutes before use
10. Physical properties
 - total solids content 21 percent
 - pH (approximately) - 11.7
 - gel point - ---
 - viscosity - ---

Single facer formulation

1. Water - 260.0 gr.
2. Add corn starch - 25.5 gr.
3. Add 10 percent NaOH solution - 42.0 gr.
4. Heat at 71°C. for 10 minutes

5. Add water - 405.0 gr.
6. Mix for 5 minutes
7. Add borax, 10 mol. Hyd. - 4.2 gr.
8. Add corn starch - 153.0 gr.
9. Physical properties
 - total solids content - 21 percent
 - pH (approximately) - 11.0
 - gel point - 150-155°F.
 - viscosity - 28-30 seconds

Double backer formulation

1. Water - 260.0 gr.
2. Add corn starch - 30.0 gr.
3. Add 10 percent NaOH solution - 48.0 gr.
4. Heat at 71°C. for 10 minutes
5. Add water - 405.0 gr.
6. Mix for 5 minutes
7. Add borax, 10 mol. Hyd. - 4.8 gr.
8. Add corn starch - 149.0 gr.
9. Physical properties
 - total solids content - 20 percent
 - pH (approximately) - 10.9
 - gel point - 140-145°F.
 - viscosity - 40-45 seconds

The general purpose formulation was used in the trial-and-error preliminary work to establish adhesive spread control, develop the specimen pressing and test techniques, and other exploratory tests. The more specific single face and double back mixes were those used in the main

factor studies of bonding variable effects. The formulation details described above are exactly as received from the Stein Hall Company, except for point number 4, where it was found that stirring gently while the heat was applied was necessary to produce a uniform mixture. Formulation components are indicated in grams rather than proportions of the total mixture, since the adhesive quantity indicated is the minimum recommended for representative properties.

APPENDIX E

TEST DATA AND STATISTICAL ANALYSIS

Table 9. BREAKING LOAD¹ AND PERCENT PAPER FAILURE² FOR SAMPLES BONDED AT SIX POUNDS PER INCH OF GLUE LINE WITH STANDARD SPREAD

Formulation ³	Platen Temperature					
	160°F.		180°F.		200°F.	
	S	D	S	D	S	D
6 Percent moisture content	29.0 ⁺	28.6 [\]	28.0 [/]	27.8 [*]	31.0 [*]	27.4 [*]
	28.6 [\]	26.2 ⁺	28.0 [/]	25.4 [*]	32.2 [*]	29.2 [*]
	27.2 [/]	24.0 [/]	29.4 ⁺	29.6 [*]	31.0 [*]	30.6 [*]
	29.2 [/]	26.6 ⁺	27.8 [/]	26.2 [\]	30.8 [*]	29.8 [*]
	28.0 [/]	25.4 ⁺	30.2 [/]	25.8 [\]	32.6 [*]	27.8 [*]
	29.2 [\]	24.4 [\]	29.6 [.]	28.0 [*]	32.0 [*]	30.0 [*]
	28.0 ⁺	28.8 [\]	28.4 [/]	28.2 [*]	30.6 [*]	30.2 [*]
	28.8 ⁺	24.2 ⁺	30.0 ⁺	26.8 [\]	31.8 [*]	29.6 [*]
	27.8 ⁺	27.0 [*]	28.8 ⁺	26.0 [*]	31.4 [*]	30.0 [*]
	28.2 [/]	26.0 [/]	29.0 ⁺	27.4 [\]	30.8 [*]	27.8 [*]
12 Percent moisture content	15.2 ⁺	17.6 ⁺	23.4 [/]	29.4 [*]	24.0 [\]	30.8 [*]
	16.0 ⁺	13.6 ⁺	23.8 [/]	27.6 [\]	24.0 [\]	32.8 [*]
	15.4 [.]	16.8 ⁺	25.0 [/]	25.4 [/]	26.0 [*]	32.0 [*]
	15.0 [.]	15.6 ⁺	24.4 ⁺	24.4 [/]	23.4 [\]	29.4 [*]
	15.8 ⁺	16.4 ⁺	24.0 ⁺	26.8 [\]	24.8 [*]	28.2 [*]
	15.4 ⁺	17.2 [.]	23.0 ⁺	29.0 [*]	22.0 [/]	32.6 [*]
	15.0 [.]	16.2 ⁺	24.0 ⁺	27.4 [\]	22.8 [/]	32.0 [*]
	15.2 ⁺	15.8 ⁺	26.0 ⁺	28.0 [\]	25.2 [*]	31.4 [*]
	16.2 ⁺	14.2 [.]	24.2 ⁺	28.4 [/]	25.2 [*]	31.2 [*]
	16.0 ⁺	17.2 ⁺	25.6 ⁺	24.8 ⁺	26.2 [*]	29.0 [*]
22 Percent moisture content	0 [.]	0 [.]	2.0 [.]	12.6 [.]	22.8 [.]	28.8 [*]
	0 [.]	0 [.]	4.4 ⁺	20.2 ⁺	33.0 [*]	31.8 [*]
	0 [.]	0 [.]	0 [.]	16.6 ⁺	28.0 [/]	32.2 [*]
	0 [.]	0 [.]	3.8 [.]	16.0 ⁺	31.6 [*]	30.0 [*]
	0 [.]	0 [.]	5.0 ⁺	15.6 [.]	25.2 ⁺	31.6 [*]
	0 [.]	0 [.]	0 [.]	14.8 ⁺	30.8 [\]	28.0 [*]
	0 [.]	0 [.]	3.2 [.]	15.8 [.]	31.4 [/]	32.2 [*]
	0 [.]	0 [.]	4.0 [.]	13.4 ⁺	29.2 ⁺	28.8 [*]
	0 [.]	0 [.]	0 [.]	19.6 ⁺	25.8 [/]	30.6 [*]
	0 [.]	0 [.]	2.8 [.]	15.6 ⁺	28.8 [/]	33.2 [*]

¹ Adhesive bond breaking load in pounds as measured with the glue line shear device.

² Designations for the percent of paper failure noted at the bond interface; ^o - 0%, + - 25%, / - 50%, \ - 75%, * - 100%.

³ Denotes single face (S) and double back (D) formulations.

Table 10. BREAKING LOAD¹ AND PERCENT PAPER FAILURE² FOR SAMPLES BONDED AT TWELVE POUNDS PER INCH OF GLUE LINE WITH STANDARD SPREAD

Formulation ³	Platen Temperature					
	160°F.		180°F.		200°F.	
	S	D	S	D	S	D
6 Percent moisture content	30.6 ⁻	31.2 [*]	30.6 [*]	30.0 [*]	34.2 [*]	31.4 [*]
	27.2 ⁻	27.2 [*]	31.4 [*]	32.6 [*]	29.8 [*]	34.0 [*]
	27.8 ⁻	29.2 [*]	28.6 [*]	29.4 [*]	32.0 [*]	32.6 [*]
	30.6 [*]	28.6 [*]	29.2 [*]	30.4 [*]	31.6 [*]	33.0 [*]
	27.4 ⁻	28.8 [*]	28.0 [*]	29.8 [*]	30.0 [*]	31.8 [*]
	30.0 ⁻	30.0 [*]	30.8 [*]	30.6 [*]	33.2 [*]	32.8 [*]
	30.8 [*]	29.0 ⁺	31.0 [*]	32.2 [*]	33.6 [*]	31.8 [*]
	28.8 [*]	29.4 [*]	30.0 [*]	30.4 [*]	30.8 [*]	32.4 [*]
	29.4 ⁻	30.8 ⁻	29.6 [*]	31.6 [*]	31.4 [*]	32.0 [*]
	30.6 ⁻	27.0 ⁻	29.0 ⁻	32.0 [*]	32.8 [*]	32.8 [*]
12 Percent moisture content	19.8 ⁺	26.0 ^o	21.2 ⁻	25.6 ⁻	32.0 [*]	31.8 [*]
	20.0 ⁺	23.8 ^o	22.4 ⁻	25.6 ⁻	30.4 [*]	30.0 [*]
	22.4 ⁻	24.6 ^o	31.0 ⁻	31.0 [*]	31.6 [*]	30.4 [*]
	20.2 ⁺	26.0 ⁺	26.6 ⁻	26.6 [*]	30.8 [*]	33.0 [*]
	22.2 ⁻	25.2 ^o	29.0 [*]	28.8 [*]	30.0 [*]	31.8 [*]
	20.4 ⁺	25.4 ^o	29.8 [*]	28.0 [*]	28.6 [*]	32.4 [*]
	21.2 ⁻	24.8 ^o	28.0 [*]	30.4 [*]	31.0 [*]	29.6 [*]
	19.6 ⁺	24.6 ^o	30.4 [*]	27.8 [*]	31.2 [*]	30.6 [*]
	20.6 ⁻	25.2 ^o	24.6 ⁻	26.0 ⁻	29.8 [*]	31.8 [*]
	20.8 ⁻	25.0 ^o	26.4 ⁻	31.0 [*]	30.4 [*]	33.2 [*]
22 Percent moisture content	0 ^o	11.8 [*]	19.6 ⁺	20.0 ⁺	30.0 [*]	21.0 ⁺
	0 ^o	15.8 ⁺	27.8 ⁺	27.6 ⁺	22.8 ⁻	26.0 ⁻
	0 ^o	9.4 ^o	23.2 ⁺	21.0 ⁺	28.6 [*]	23.4 ⁻
	0 ^o	14.0 ^o	19.0 ^o	20.6 ⁻	26.6 ⁻	23.0 ⁺
	0 ^o	12.8 ^o	27.0 ⁺	22.0 ⁻	25.2 ⁻	24.4 ⁻
	0 ^o	14.6 ⁺	20.8 ⁺	25.4 ⁻	29.2 [*]	26.2 ⁻
	0 ^o	13.8 ^o	20.4 ⁺	26.0 ⁻	27.6 ⁻	21.8 ⁻
	0 ^o	14.8 ⁺	20.6 ⁺	26.6 ⁻	24.4 ⁺	22.0 ⁻
	0 ^o	12.0 ^o	24.2 ⁺	21.8 ⁻	24.0 ⁺	23.4 ⁺
	0 ^o	10.4 ^o	26.0 ⁺	24.4 ⁻	23.4 ⁺	21.6 ⁻

¹ Adhesive bond breaking load in pounds as measured with the glue line shear device.

² Designations for the percent of paper failure noted at the bond interface: ^o - 0%, ⁺ - 25%, ⁻ - 50%, [\] - 75%, ^{*} - 100%.

³ Denotes single face (S) and double back (D) formulations.

Table 11. BREAKING LOAD¹ AND PERCENT PAPER FAILURE² FOR SAMPLES BONDED AT SIX POUNDS PER INCH OF GLUE LINE WITH HEAVY SPREAD

Formulation ³	Platen Temperature					
	160°F.		180°F.		200°F.	
	S	D	S	D	S	D
6 Percent moisture content	8.4 [°]	27.8 [°]	15.8 ⁺	15.6 [*]	31.8 [*]	30.0 [*]
	12.4 [°]	23.2 ⁺	15.0 ⁺	17.6 [*]	30.4 [*]	27.2 [°]
	10.0 [°]	25.0 ⁺	16.4 ⁺	16.6 [*]	31.2 [*]	30.2 [°]
	11.4 [°]	23.4 ⁺	16.0 [°]	17.8 [*]	32.2 [*]	26.6 ⁺
	10.6 [°]	26.6 ⁺	14.6 [°]	15.8 [*]	30.8 [*]	28.2 [*]
	9.8 [°]	26.0 [°]	16.8 ⁺	16.6 [*]	32.0 [*]	26.0 [°]
	9.0 [°]	26.2 [°]	15.6 [°]	16.4 [*]	30.0 [*]	27.4 [*]
	10.6 [°]	26.0 ⁺	16.2 ⁺	15.8 [*]	31.4 [*]	27.0 [°]
	11.2 [°]	23.8 [°]	15.2 [°]	18.0 [*]	30.0 [*]	26.8 ⁺
	10.0 [°]	25.0 ⁺	15.0 [°]	18.2 [*]	31.0 [*]	27.2 [°]
12 Percent moisture content	12.4 [°]	20.8 ⁺	15.4 [°]	16.0 [*]	22.2 ⁺	31.2 [*]
	16.0 [°]	25.4 [°]	16.0 [°]	19.2 [*]	18.6 [°]	26.0 ⁺
	14.6 [°]	23.4 ⁺	20.2 ⁺	18.2 [*]	18.4 [°]	30.6 [*]
	15.4 ⁺	22.2 ⁺	17.0 [°]	17.4 [*]	20.4 [°]	28.4 [*]
	14.2 [°]	24.2 [°]	15.0 [°]	16.8 [*]	19.2 [°]	31.0 [*]
	12.0 [°]	19.4 [°]	16.6 [°]	18.0 [*]	21.6 ⁺	27.2 [°]
	15.0 [°]	20.0 ⁺	18.8 ⁺	18.8 [*]	20.8 ⁺	27.0 [*]
	13.8 [°]	20.6 [°]	21.4 [°]	17.6 [*]	19.2 [°]	29.4 [*]
	12.8 [°]	26.8 ⁺	17.6 [°]	19.0 [*]	21.8 ⁺	30.2 [*]
	14.6 [°]	24.8 ⁺	16.0 [°]	16.6 [°]	21.6 ⁺	26.8 [°]
22 Percent moisture content	0 [°]	0 [°]	5.0 [°]	10.4 [°]	12.0 [°]	18.8 ⁺
	0 [°]	0 [°]	3.0 [°]	8.4 [°]	14.6 [°]	16.0 [°]
	0 [°]	0 [°]	4.6 [°]	6.8 [°]	17.2 ⁺	20.0 [°]
	0 [°]	0 [°]	6.2 [°]	11.2 [°]	15.2 ⁺	16.2 [°]
	0 [°]	0 [°]	5.2 [°]	7.2 [°]	16.4 [°]	19.0 [°]
	0 [°]	0 [°]	0 [°]	8.0 [°]	13.8 [°]	18.4 [°]
	0 [°]	0 [°]	3.4 [°]	10.8 [°]	14.0 [°]	19.2 ⁺
	0 [°]	0 [°]	4.0 [°]	8.0 [°]	18.0 [°]	18.0 [°]
	0 [°]	0 [°]	5.6 [°]	11.0 [°]	16.2 ⁺	17.0 [°]
	0 [°]	0 [°]	4.8 [°]	10.6 [°]	17.6 ⁺	16.6 [°]

¹ Adhesive bond breaking load in pounds as measured with the glue line shear device.

² Designations for the percent of paper failure noted at the bond interface: ° - 0%, + - 25%, / - 50%, \ - 75%, * - 100%.

³ Denotes single face (S) and double back (D) formulations.

Table 12. BREAKING LOAD¹ AND PERCENT PAPER FAILURE² FOR SAMPLES BONDED AT TWELVE POUNDS PER INCH OF GLUE LINE WITH HEAVY SPREAD

Formulation ³	Platen Temperature					
	160°F.		180°F.		200°F.	
	S	D	S	D	S	D
6 Percent moisture content	19.8 /	25.6 /	28.4 \	27.6 *	30.2 *	30.0 *
	24.0 +	27.6 \	23.0 +	31.8 *	30.6 *	32.0 *
	20.0 +	24.4 /	26.8 /	29.0 *	30.0 *	31.4 *
	21.4 /	26.4 *	22.2 +	28.8 *	29.4 *	29.8 *
	22.6 +	26.4 /	23.8 +	31.0 *	32.6 *	30.2 *
	22.0 +	26.8 /	24.8 /	29.6 *	30.8 *	30.8 *
	23.0 /	25.0 +	28.0 /	29.8 \	31.4 *	31.6 *
	20.6 +	25.8 /	26.0 /	30.4 *	31.6 *	31.4 *
	21.8 +	27.2 +	25.4 +	28.2 \	30.0 *	32.2 *
	21.2 +	24.4 +	22.4 °	29.2 \	31.6	31.0
12 Percent moisture content	18.6 +	11.6 °	24.8 +	20.0 +	28.0 /	30.0 *
	11.6 °	16.4 +	22.4 +	22.0 *	28.8 \	31.0 *
	15.4 +	12.8 °	26.0 /	25.0 \	25.0 +	28.0 *
	13.0 °	13.4 +	27.8 /	24.6 *	29.6 \	33.0 *
	11.8 °	12.2 °	22.8 +	25.2 *	26.2 +	30.2 *
	14.4 °	13.2 °	24.0 +	20.8 +	24.8 +	28.6 *
	12.6 +	10.8 +	26.6 /	22.2 /	25.6 +	29.0 *
	15.0 +	13.6 °	23.8 +	24.4 *	25.0 +	30.6 *
	14.4 °	14.6 +	24.4 +	23.6 /	28.2 /	30.8 *
	14.8	14.4 +	26.2 /	21.6 /	27.0 /	31.2
22 Percent moisture content	0 °	0 °	24.0 +	18.0 °	28.0 /	29.2 \
	0 °	0 °	21.8 °	26.6 \	23.6 °	26.0 +
	0 °	0 °	25.0 +	20.0 +	28.0 /	25.2 +
	0 °	0 °	21.4 +	19.6 °	30.2 /	24.6 +
	0 °	0 °	22.8 +	21.6 +	31.6	24.8 +
	0 °	0 °	22.0	22.4 +	32.4 *	29.0 \
	0 °	0 °	24.0 +	20.4 +	30.8	28.4 +
	0 °	0 °	25.8 +	21.8 /	32.4 *	25.0 +
	0 °	0 °	22.8 +	24.8 +	30.0 /	27.2 /
	0	0	24.6 °	20.2	29.8 /	27.8 /

¹ Adhesive bond breaking load in pounds as measured with the glue line shear device.

² Designations for the percent of paper failure noted at the bond interface: ° - 0%, + - 25%, / - 50%, \ - 75%, * - 100%.

³ Denotes single face (S) and double back (D) formulations.

Table 13. ANALYSIS OF VARIANCE OF ADHESIVE BOND BREAKING LOADS
IN THE STUDY OF FIVE BOND FORMATION FACTORS

Source of variance	Degrees of freedom	Sum of squares	Mean square	F ratio	Level of significance(%)
Spread	1	3,122.50	3,122.50	846.2	99
Formulation	1	890.22	390.22	241.3	99
Temperature	2	20,958.30	10,479.15	2,839.0	99
Pressure	1	4,045.12	4,045.12	1,096.2	99
Moisture (content)	2	21,266.07	10,633.04	2,881.5	99 ¹
SxF	1	3.01	3.01	0.8	N.S. ¹
SxT	2	151.43	75.71	20.5	99
SxP	1	172.67	172.67	46.8	99
SxM	2	137.08	68.54	18.6	99
FxT	2	182.66	91.33	24.8	99
FxP	1	156.98	156.98	42.5	99
FxM	2	66.32	33.16	9.0	99
TxP	2	1,299.38	649.69	176.1	99
TxM	4	7,009.87	1,729.98	468.8	99
PxM	2	550.17	275.09	74.6	99
SxFxT	2	101.97	50.98	13.2	99
SxFxP	1	144.20	144.20	39.1	99
SxFxM	2	526.31	263.15	71.3	99
SxTxP	2	1,013.38	506.69	137.3	99
SxTxM	4	436.14	109.03	29.6	99
SxPxM	2	393.11	196.55	53.3	99
FxTxP	2	62.41	31.20	8.5	99
FxTxM	4	379.89	94.93	25.7	99
FxPxM	2	145.83	72.91	19.8	99
TxPxM	4	692.36	173.09	46.9	99
SxFxTxP	2	503.48	251.74	68.2	99
SxFxTxM	4	601.04	150.26	40.7	99
SxFxPxM	2	7.23	3.61	1.0	N.S. ¹
SxTxPxM	4	1,181.32	295.28	80.8	99
FxTxPxM	4	1,013.48	328.18	88.9	99
SxFxTxPxM	4	105.30	26.32	7.1	99
Within	648	2,380.79	3.69		
Total	719	69,700.02			

¹ Non-significant at the 95 percent level of significance.

Table 14. ANALYSIS OF VARIANCE OF ADHESIVE BOND BREAKING LOADS
IN THE STUDY OF FIVE BOND FORMATION FACTORS (REVISED)^{1,2}

Source of variance	Degrees of freedom	Sum of squares	Mean square	F ratio	Level of significance(%)
Spread	1	3,122.50	3,122.50	360.1	99
Formulation	1	890.22	390.22	102.7	99
Temperature	2	20,958.30	10,479.15	1,208.6	99
Pressure	1	4,045.12	4,045.12	466.6	99
Moisture (content)	2	21,266.07	10,633.04	1,226.4	99
SxF	1	3.01	3.01	0.3	N.S. ³
SxT	2	151.43	75.71	8.7	99
SxP	1	172.67	172.67	19.9	99
SxM	2	137.08	68.54	7.9	99
FxT	2	182.66	91.33	10.5	99
FxP	1	156.98	156.98	18.11	99
FxM	2	66.32	33.16	3.8	95
TxP	2	1,299.38	649.69	74.9	99
TxM	4	7,009.87	1,729.98	199.5	99
PxM	2	550.17	275.09	31.7	99
SxFxT	2	101.97	50.98	5.9	99
SxFxP	1	144.20	144.20	16.6	99
SxFxM	2	526.31	263.15	30.4	99
SxTxP	2	1,013.38	506.69	58.4	99
SxTxM	4	436.14	109.03	12.6	99
SxPxM	2	393.11	196.55	22.7	99
FxTxP	2	62.41	31.20	3.6	N.S. ³
FxTxM	4	379.89	94.93	10.9	99
FxPxM	2	145.83	72.91	8.4	99
TxPxM	4	692.36	173.09	19.9	99
Residual	668	5,792.64	8.67		
Total	719	69,700.02			

¹ Complete analysis appears in Table 13.

² Within, third and fourth order interaction sources of variance pooled as residual term.

³ Non significant at the 95 percent level of significance.

Table 15. BREAKING LOAD IN POUNDS FOR SPECIMENS BONDED WITH SINGLE
FACE FORMULATION AT VARIOUS PRESS TIMES

Press time (seconds)	Breaking load	Average breaking load
1	no measurable bond	0
2	8.6,0,4.8,4.2,04.2,3.2,0,2.0,6.0	3.3
3	7.4,8.0,6.2,10.6,4.0,11.8,4.8,9.6,10.2,8.8	8.1
4	10.8,15.4,9.2,12.2,14.6,10.4,12.8,10.0,14.0,13.6	12.3
5	15.0,15.8,16.0,17.8,16.2,17.0,19.2,16.4,18.0,17.0	16.6
6	16.2,20.4,18.0,19.2,21.0,18.8,20.2,17.6,16.8,20.8	18.9
7	19.4,20.0,23.6,22.8,18.2,18.8,22.2,20.6,21.4,24.6	21.2
8	23.4,26.4,28.0,26.6,26.0,26.8,24.6,22.8,23.6,22.0	25.0
9	28.0,28.0,25.8,25.0,27.2,27.0,22.8,26.6,25.2,24.8	26.0
10	30.4,27.0,28.6,28.2,24.8,26.0,25.8,26.4,24.6,27.6	26.9
11	28.6,29.8,28.2,27.4,28.2,26.2,29.8,26.8,26.0,27.4	27.8
12	30.2,29.4,29.0,25.6,26.6,28.2,27.4,26.8,28.0,24.4	27.6
13	29.8,29.4,26.8,28.4,27.6,26.0,24.8,28.2,26.4,27.6	27.5
14	30.2,29.0,28.8,29.2,31.4,30.6,26.4,27.8,26.8,29.6	28.9
15	26.2,28.0,30.0,29.4,26.8,31.2,27.8,28.0,31.2,30.0	28.9
16	31.8,29.4,28.0,29.4,30.8,30.2,29.6,31.4,32.8,30.4	30.4
17	31.8,29.0,30.2,30.0,32.2,29.4,31.2,31.6,33.0,31.2	30.9
18	30.4,30.2,33.2,30.4,32.6,32.0,31.0,30.6,28.4,31.6	31.0
20	27.1,29.2,27.0,32.0,28.4,31.0,28.0,29.2,30.4,32.0	29.5
22	test omitted	--
24	" "	--
26	" "	--
28	" "	--
30	29.2,29.0,30.0,32.8, 30.6,30.4,32.0,30.8,31.6,31.6	30.8
60	30.8,29.2,31.2,33.8,32.0,30.0,31.0,33.6,30.4,32.4	31.6

Table 16. BREAKING LOAD IN POUNDS FOR SPECIMENS BONDED WITH DOUBLE BACK FORMULATION AT VARIOUS PRESS TIMES

Press time (seconds)	Breaking load	Average breaking load
1	2.0,0,0,0,0,0,3.2,0,0,0,0	0.5
2	6.2,8.4,8.6,10.4,6.0,8.2,7.0,7.4,11.0,6.6	7.9
3	10.6,10.4,12.2,11.0,8.2,7.4,7.4,11.2,6.8,8.2	9.3
4	10.6,14.4,9.2,10.4,10.2,7.4,13.2,9.6,8.4,12.6	10.6
5	14.2,18.4,16.2,18.4,14.0,16.0,12.6,11.0,12.6,14.4	14.8
6	24.0,26.6,20.4,25.2,22.4,25.0,24.0,20.2,19.2,19.4	22.6
7	21.4,20.4,21.6,23.0,18.8,20.6,26.2,22.8,23.2,22.8	22.1
8	28.0,29.4,28.2,29.2,28.2,27.6,29.0,26.0,24.6,29.0	27.9
9	30.8,32.4,31.0,30.0,28.4,32.6,27.4,27.2,26.2,25.4	29.1
10	30.6,31.8,28.6,29.4,32.0,32.2,28.6,30.6,31.2,29.0	30.4
11	31.8,32.6,28.0,28.4,30.8,30.0,31.6,29.2,32.2,29.6	30.4
12	31.8,32.4,30.8,33.6,28.2,29.6,29.0,30.8,31.8,32.0	30.9
13	32.0,31.4,30.2,28.0,32.2,30.6,31.8,29.8,29.8,24.4	30.0
14	test omitted	--
15	" "	--
16	" "	--
17	" "	--
18	" "	--
20	" "	--
22	" "	--
24	" "	--
26	" "	--
28	" "	--
30	34.2,32.6,33.2,29.0,31.2,30.2,28.6,32.2,29.4,30.6	31.1
60	32.8,31.6,31.8,30.4,29.6,32.2,31.6,29.0,30.2,32.2	31.2

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