NATIONAL BUREAU OF STANDARDS REPORT

10 255

THE WARD CONTAINER DELAMINATION PROBLEM

to

Army Natick Laboratories

Natick, Mass.

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS

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For

Army Natick Laboratories

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TABLE OF CONTENTS

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1. Introduction

1.1 Background

At the request of the General Equipment and Packaging Laboratory (NLABS) the Structures Section of NBS undertook an investigation of the sandwich panel material used in the Ward Containers (Must Hospital Units) being received (March 1970) by the Army. A high percentage of these containers have exhibited panel delaminations shortly after delivery to the Army depot.

Cursory observations of these delaminated panels, while in place on the containers, indicated that the adhesive bond to the skins was poor. The reason for the poor bond could not be determined.

Later, the delaminating, hinged roof panel on a container (Unit 101) was cut so that exterior aluminum could be peeled from the honeycomb core. Visual observation of the partially peeled skin showed that although the adhesive bond was very poor over much of the skin area some areas were well bonded.

This observation implied that, unless the adhesive properties varied significantly, the skins had been improperly processed prior to application of the adhesive or had deteriorated following application of the adhesive.

In an effort to obtain test data which might pinpoint the cause of the delaminations NLABS prepared a test plan (Appendix A) to be used on samples of panels from containers in stock at Atlanta General Depot. Another objective of the study was to determine if the samples met the requirements of the procurement specifications.

The Test Plan prepared by NLABS, dated 24 March 1970, is reproduced as Appendix A to this report. This plan itemizes the samples to be secured, the preparation of specimens and the methods to be used in testing the samples cut from representative containers.

The Test Plan called for samples to be cut from only three containers (Serial No. 90, 139 & 143), but additional samples were cut from two additional containers (Serial No. 134 & 137). These additional samples were secured in order to have test data for panels produced by the three manufacturers supplying panels to the prime contractor.

Four types of tests were specified in the Test Plan; shear, tension, flexure and peel. In addition, three specimen conditioning methods were specified. Most specimens were to be conditioned and tested in lab air at $73 \pm 2^{o}F$ and $50 \pm 4\%$ relative humidity. Two 12 in x 12 in specimens from each container sample were to be preconditioned for 7 days at $120 \pm 5^{o}F$ and 100% relative humidity. Two additional 12 in x 12 in specimens from each container sample were to be preconditioned (aged) for 7 days in an oven at $175 \pm 5^{o}F$.

Three verification samples were received from the contractor. These samples were also conditioned and tested at 73 ± 2°F and 50 ± 4% rh.

In general the test methods were to be as described in MIL-STD-401B with certain exceptions indicated in the Test Plan.

1.3 Paper Honeycomb Sandwich Panel Specifications

The sandwich panels for the Ward Containers were to be made in accordance with a specification denoted as ZSMS-25 (October 10, 1967, revision). This is a general purpose specification for paper honeycomb sandwich panels made with metallic and non-metallic skins. The type and thickness of the skins, type and density of the core, and thickness of the panels were to be as specified in other documents.

ZSMS-25 specified values for certain test data, but with the specified values dependent on type, density and thickness of the core.

NLABS provided the information that the panel material for this investigation was to have been made with .040 in aluminum skins laminated to 2 in. thick honeycomb.core conforming to MIL-H-21040A, Type II, Class 2. (4 pcf density). This specification requires a minimum shear strength of 150 psi and shear modulus of 14,000 psi for 1-in thick core when tested dry in the "L" direction. ZSMS-25 requires a minimum shear strength of 220 psi under the same conditions.

From these requirements it would appear that the minimum shear strength and modulus for the core should be 220 and 14,000 psi respectively. However, the above values are for core specimens 1 in. thick. The cores in the samples for this investigation were approximately 2 in. thick.

Another requirement in ZSMS-25 (Section 3.3.5 and Fig. 6) states that the shear strength of the bond should meet the requirements of a curve relating shear strength with core-thickness to 1 3/4 in. Extrapolating beyond this curve to 2 in. thickness indicates that a minimum shear strength of about 190 psi would be required for the bond. By implication this is a requirement of 190 psi minimum shear strength for the 2 in. thick core.

The only, explicitly stated requirement of ZSMS-25 for the laminated panels was the peel strength. The minimum peel strength (Section 3.34) was to be 4-in-lb of torque per inch of width.

There is a statement in ZSMS-25 (7.5.1) regarding bond strength which requires that there be some indication of core failure rather than bond failure in the peel test. The indication is evidence of "shreds" of core material remaining on the peeled skins.

2. Summary of Conclusions

2.1 Panel Properties

2.1.1 Shear Properties of Core

The plate shear strength of the cores used in samples No. 90, 139 and 143 were significantly higher than those in No. 134 and 137 when tested dry. The shear strengths of the cores in samples 134 and 137 were less than the 190 psi indirectly required by the ZSMS-25 specifications while those for the other samples were greater.

The plate shear moduli for all samples were greater than the 14,000 psi required by MIL-H- 21040A, but those for the cores in samples 90, 139 and 143 were much greater than 134 and 137.

The shear values determined by the flexural test were slightly lower than those determined by the plate shear test and with greater scatter in the data. This scatter could be expected because the position of the splices in some of the specimens affected the results to a greater extent than in the plate shear test.

Of the 30 flexural specimens conditioned at 100% rh and 120^0F only 4 failed in shear. The other 26 specimens failed either in bond or in crushing of the core under the load application point. This means that the maximum shear stress attained for the wet specimens were not indicative of the shear strength of the wet core.

2.1.2 Tensile Bond Strength

The tensile bond data indicate that the blue and pink adhesives (cold bond) were not nearly as effective as the yellow adhesive (hot bond). Furthermore, the results of the flexural tests indicate that the blue and pink adhesive-bonded panels were very susceptible to deterioration from high humidities.

2.1.3 Peel Strength

Although the drum peel test is not considered to be good for determining small differences in adhesive bond, the results show that the yellow adhesive was much more effective than the blue or pink.

ZSMS-25 required a minimum peel strength of 4 in-lb of torque per inch of specimen width. None of the 24 specimens bonded with the blue adhesive attained this value. Only 1 of the 9 specimens bonded with the pink adhesive attained a peel strength of more than 3.8 in-lb/in.

All the specimens bonded with the yellow adhesive had a peel strength considerably greater than 4.0 in-lb/in. In fact 8 of the 12 specimens had a peel strength exceeding twice the required value.

2.1.4 Fire Resistance

The fire resistance of the panel material could not be evaluated using the procedure of ZSMS-25.

2.2 Causes of Delamination

2.2.1 Poor Initial Bond

No test data was developed in this study which would directly relate the delamination problem with the use of the chromate coating on the skins or with contamination of the skins prior to lamination. However, technical representatives of two major aluminum producers stated that the conversion coatings used on the skins of the blue and pink adhesive panels are considered to be unstable compounds. The adhesive bonding characteristics of these coatings deteriorate with time, especially under high humidity conditions. These producers do recommend the use of these coatings as a bonding aid for paints, but only when the paint can be applied shortly after processing of the aluminum.

It was learned that the fabricator of the blue adhesive panels procured, at one time, a large supply of aluminum with the conversion coating. If this is true it would explain the obvious lack of initial bond observed on certain areas of delaminated blue adhesive panels. The skins at these areas were observed to be entirely free of any evidence that honeycomb had ever been bonded to the skins.

2.2.2 Degradation of Bond

On other completely delaminated areas a light foggy deposit was observed on the skins. This deposit, which appeared to be a corrosion product, formed a pattern on the skin outlining the position of the honeycomb core. This observation implies that the conversion coating had changed in some manner following the lamination process.

The skins of specimens subjected to high-humidity conditions were examined following the tests. The skins of the blue-adhesive specimens, and to a lesser degree the pink adhesive, exhibited a similar but heavier pattern to that mentioned above. However, these patterns were obviously formed by a corrosion which attacked the aluminum base metal and/or the conversion coating. No such corrosion was found on the yellow adhesive specimens.

From these observations the conclusion was reached that one of the primary causes of the delamination is the deterioration of the bond induced by the penetration of moisture to the skin. Microscopic observations on the skins from the high-humidity specimens led to the conclusion that this moisture travels down the core paper to the skin causing a corrosion which spreads underneath the adhesive.

These conclusions mean that unless the skin is protected with a coating unaffected by moisture the bond will eventually deteriorate in sandwich systems such as those tested here.

2.3 Critique of Test Procedures

2.3.1 Shear Tests

Two tests were used in this study to determine the shear properties of the panel material. 1) The compressive plate shear test and 2) the center-point loading flexure test.

The plate shear test (MIL-STD-401B Para. 5.1.5) is a direct test suitable for determining the shear properties of a sandwich core. It has two disadvantages. First, the results probably depend to a certain extent of the size and thickness of the specimen. The test procedure should specifically state the size of specimen for each core thickness and to assist the user of test data, correction factors could be introduced to compensate for the various core thickness.

The second disadvantage of the plate-shear test is that it is expensive to perform. Two machined heavy steel plates have to be cemented to each specimen. For a few specimens the expense may not be excessive, but in any large scale investigation it would be.

In general the plate shear test is the best standard test now in use for determining shear properties of sandwich panel cores.

The center-point loading flexure test MIL-STD-401B, Para. 5.2.4 was used for specimens dimensioned so that the core would fail in shear when the adhesive bond strength was adequate.

In general the shear strength values determined by this method are considered to be a fair estimate of the shear strength of the core, but unless the bond strength is very poor the bond strength cannot be evaluated.

The shear modulus values determined in this manner are poor estimates, especially when there are splices in the core. This is because the maximum shear occurs over a very small area of the specimen and if there is a splice close to this area the shear properties of the splice control the test values.

2.3.2 Tension Tests

This test (MIL-STD-401B Para. 4.2.3) is considered to be a good test for determining the strength of the core-to-facing bond except when the bond strength is greater than the tensile strength of the core.

2.3.3 Drum Peel Test

The Drum peel test (MIL-STD-401B Para. 5.2.6) is intended to be used in determining the relative peel resistance of the bond between the facing and the core. The precision of these peel test values is considered to be poor.

The primary reason for this lack of precision is that a large part of the force required to peel the skin from the sandwich is used just to bend the skin. Some value for this bending force must be deducted from the force required to peel the sandwich in order to determine the peel resistance of the bond. The value of this bending force depends on the thickness and stiffness of the adhesive layer as well as the skin itself.

The peel values reported here are considered to be approximate, but there is no doubt that the relative differences reported between the yellow and the blue and pink adhesive panel material are real.

2.3.4 General

The primary purpose of this study was to determine the cause or causes of the delamination. Delamination is due to insufficient bond between the facing and core.

The only test from which reliable bond strength values were obtained was the tension test. The flexure test did show that high humidity degraded the bond. However, it was obvious that the bond strength varied from good to poor within the same panel.

This means that coupon specimens such as were used here may or may not indicate the efficiency of the bonding operation. It is suggested that some work be done on developing a test procedure that will test the bond over most of the area of a panel.

An edgewise, axial compressive test would induce tensile stresses in the bond over most of the specimen area. With such a test the application of a proof load, say 85% of the theoretical ultimate compressive load, would cause face wrinkling at poorly bonded area.

3. Test Specimens

3.1 Description of Samples

Eleven samples were taken from five different Ward Containers in stock at Atlanta General Depot by Army personnel and shipped to NBS. Two samples were taken from the folding roof panel of each of four containers. Three samples were taken from the folding floor panel of the fifth container. Three "verification" samples were received from the container contractor.

All samples were "coin" tested upon receipt.

Table 1 is a listing of all samples received and includes a brief description of each. All samples were approximately 2 in. thick. The paper honeycomb core appeared to be similar and included about 1-in. of foamed plastic in the cells next to the exterior skin. The major differences between the various samples were the color of the adhesive.

The color of the adhesive supposedly can be used to identify the manufacturer of the panels. Thus, the panels with the blue adhesive would be Met-L-Wood, the pink would be Dow Chemical and the yellow would be Aerospace Technology.

The yellow adhesive included a scrim cloth carrier and appeared to be "hot" bonded. The other two adhesives appeared to be "cold" bonded.

The thicknesses of the skins on the samples varied slightly, but in general the skins for the verification samples and for the samples from containers No. 134 and 137 were about 0.040 in. thick. The skins of the samples from containers No. 90, 139 and 143 were about 0.045 in. thick.

3.2 Preparation of Test Specimens

The samples were cut into the various specimens as described in the Test Plan. The verification samples were cut into 3 flexural and 3 tension specimens. All flexural and shear specimens were cut so that the honeycomb paper ribbons ran lengthwise with the specimen (TL direction). A 20-tooth, carbide tipped, table saw blade was used to cut all specimens.

Upon cutting the individual specimens each was marked with an identification symbol in accordance with the Test Plan. The Plan called for only three tension specimens from each sample, but more then three were cut from some samples.

The Test Plan implied that the aging and humidity specimens were to be conditioned in the 12 in. x 12 in. size and after conditioning were to be cut to flexural test size (4 1/4 in. x 12 in.). These specimens were cut to the final size before conditioning because of the danger of inducing delamination in the conditioned specimens.

3.3 Conditioning of Test Specimens

Three different methods were used to condition test specimens prior to test. All test specimens were tested at 73°F.

1. <u>Conditioning Method No. 1</u> (73°F - 50% RH)

   The specimens conditioned by this method were stored prior to test in the laboratory for 3 to 7 days in air controlled at 73 ± 3°F and 50 ± 5% relative humidity.

2. <u>Conditioning Method No. 2</u> (120°F - 100% RH)

   All humidity specimens were conditioned for 7 days prior to test in a chamber controlled at 120 ± 2°F and at nearly 100% relative humidity. The air in the chamber, which was heated with a constant flow of steam, appeared to be fully saturated, but the relative humidity was not measured.

   The humidity specimens were placed in a fog room at 73°F for about one hour before testing.

3. <u>Conditioning Method No. 3</u> (160°F)

   All aging specimens were conditioned for 7 days in a ventilated oven controlled at 160 ± 2°F. The temperature of the oven was to have been 175 ± 5°F according to the Test Plan. However, through an oversight, the aging temperature (160 ± 5°F) specified in ZSMS-25 was used.

   All aging specimens were stored in the lab at 73°F for about one hour before testing.

4. Test Procedures

4.1 Compressive Shear Tests on 4 1/4 in. x 25 1/2 in. Specimens

The test procedure was as described in MIL-STD-401B and as shown in Figure 1.

The test specimens were bonded to 7/8 in. steel plates with an epoxy resin. The compressive load was applied through a spherical loading head to the steel plates so that the line of action of the force passed through the diagonally opposed corners of the sandwich.

The 0.001 in dial gage visible in Figure 1 measured the relative movement between the steel plates. The shear strain and modulus reported are for the composite sandwich. The secant value for shear modulus was computed for a shear stress of 90 psi for all specimens. The secant value is the slope of a straight line drawn on the stress-strain curve between the origin and the 90 psi stress value. For these materials this secant is very close to the initial tangent.

4.2 Flatwise Tensile Tests on 2 in. x 2 in. Specimens

This test procedure was as described in MIL-STD-401B and as shown in Figure 2. Steel blocks (2 in. x 2 in. x 1 in. thick) were bonded to both skins of the 2 in. x 2 in. specimens with epoxy resin. The tensile load was applied through 3/4 in. dia. pull rods attached to the steel blocks which were tapped to receive the rods. The other end of the rods were connected to the testing machine through spherical seats. This allowed the specimens to be aligned axially during the test. No measurements were made other then the maximum load.

This test evaluates the lowest of either the strength in tension of the skin-to-core bond, the tensile strength of the core, or the cohesive strength of the adhesive.

4.3 Flexure Tests on 4 1/4 in. x 12 in. Specimens

This test was conducted as shown in Figure 3 with center point loading on specimens on a 10 in. span. Quarter point loading as stipulated in MIL-STD-401B was not feasible with the short specimen. Two inch wide bearing plates were used at the reactions and load points. This procedure conformed closely to that described in ZSMS 25 Section 4.2. The humidity and aging specimens were tested in flexure after normalizing at 73°F for one hour.

The size of the specimen had been chosen so that the method of failure would be by shearing of the core when the skin-core bond strength was sufficient.

4.4 Climbing Drum Peel Tests on 3 in. x 12 1/2 in. Specimens

The test procedure was that described in MIL-STD-401B and as shown in Figure 4.

The specimens were attached to the drum peel test apparatus and loaded at a rate of one inch of cross-head travel per minute in a 10,000 lb. testing machine. The rate of specimen peel was four inches per minute. Autographic curves were made of each specimen, and these were used to determine the median peeling force for each sandwich specimen. In order to determine the force required to bend the aluminum skin around the drum a skin from each sample was removed from the sandwich and tested by itself. The force required to bend the skin was deducted from the force required to peel the sandwich. The difference was recorded as the peeling resistance of the adhesive bond.

The peeling resistance (P) is that force required to overcome the adhesive bond. MIL-STD 401B requires the calculation of the peeling torque (T). For the equipment used in this study

$$T,\ \text{in-lb/in} = \frac{0.5P}{W}$$

Where W = width of the specimen, in.

P = peeling force, lb.

4.5 Fire Resistance of 4 in. x 12 in. Specimens

The test procedure was that described in ZSMS-25 Section 4.6.

The test specimens were mounted at forty five degrees to the table top and exposed to 1/2 in of a 1 1/2 in. yellow bunsen burner flame for 30 seconds.

The specified test specimen (5 in. x 8 in.) were not available for this test so the flexure specimens were used following the flexure tests. Only three specimens were tested.

Samples of the core materials (paper honeycomb and plastic foam) for each manufacturer were given a simple match test which was not described in 4.6.1. This consisted of igniting the material with a match and noting acceptability as described in ZSMS-25 Section 4.6.1.

4.6 Miscellaneous Tests and Observations

A number of tests were made in an effort to explain the poor adhesive bond of some panels. These efforts can be broken down into three categories:

1. Contamination or poor processing of skins prior to lamination
2. Poor adhesive and lamination control
3. Degradation of bond following lamination

The test methods are explained below in Section 5.6, Miscellaneous Test Results.

5. Test Results

5.1 Compressive Shear Tests

Results of these tests for the individual specimens are given in Table 2. The shear modulus values reported are assumed to be for the core alone as the failure occurred in the core of all specimens. No adjustment was made to eliminate the small effect from the skin on the shear modulus.

Since all specimens failed in shear of the core, the strength and modulus data can be used to compare the cores of the panel material. However, when a splice was presented in the core, initial shear failure occurred at the splice inducing bond failure between the core and the skin. Figure 5 is a typical core shear failure.

All specimens were cut so that the direction of the ribbon was parallel to the length of the specimen.

5.2 Flatwise Tensile Tests

The results from the flatwise tensile tests are presented in Table 3a and 3b. Most of the specimens failed in bond or in the adhesive interface between the core and the skin. The remaining failures were in the core. Reliable values of core tensile strength are obtained only from specimens having core failures.

However when specimens had core splices the values were higher than for those without splices because of the difference in bonding area and the strength of the splice material. Typical failures are shown in Figures 6, 7 and 8. Figure 7 and 8 show specimens with splices.

5.3 Flexure Tests

The results of the flexure tests are presented in Table 4a, 4b & 4c. The dry, room-temperature specimens failed by shear fracture of the core. However, when splices occurred the failures generally occurred near the splice. The shear strengths of the spliced specimens did not differ appreciably from those without splices. Figure 9 shows a typical shear failure.

Local compressive failures (core crushing) occurred in the core of some specimens conditioned at 100% relative humidity and with a noticeable decrease in maximum shear stress. The local failures appeared under the center loading plate. A typical crushing failure is shown in Figure 10. Some humidity specimens delaminated prior to test. Figure 11 is a picture of one of these specimens.

There was no significant difference between data collected from specimens conditioned at room temperature and data taken from specimens conditioned at 160°F.

5.4 Climbing Drum Peel Tests

Results of the peel tests are presented in Tables 5a and 5b. All specimens tested exhibited some cohesive failure within the adhesive. Some specimens had adhesive bond failure in combination with cohesive failure. The specimens with the yellow adhesive failed primarily in the core but with some cohesive failure in the adhesive at the scrim cloth.

The peel values reported can be used to compare the bond of the various panels, and are a rough indication of the torque required to peel the skin from the sandwich.

Splices in the core caused a large peak value in load as the skin was peeled off the spliced area. This peak value was not used when determining the values presented, because it was not considered to be representative. Figures 12, 13 and 14 are examples of each type of peel specimen after test.

5.5 Fire Resistance

Results of these tests showed that the test method stipulated in ZSMS 25 was not valid for determining fire properties of these panels. Of the specimens tested, none of the skins got warm enough to blister the paint; in fact, the only visible damage was a black spot about 1 1/2 inch in diameter at the point of flame application.

The match test on the core material revealed that none of the papers in the cores was self-extinguishing. The fire resistance of the foams used in the core cells was questionable using this match test.

5.6 Miscellaneous Tests and Observations

5.6.1 Contamination or Poor Processing of Skins

a. An attempt was made to determine if the contamination present on the skins of delaminated panels was present when the panels were laminated. No conclusion was reached.

b. The chromate coating (Alodine 1200?) on the skins of delaminated, blue adhesive samples was slight foggy. In some areas this foggy appearance formed a pattern on the skin resembling the outline of the honeycomb core. The fog appeared to be a corrosion product and was heavier in the area around but not right at the area where the core contacted the skin. This means that the fogging must have occurred after lamination. See Section 5.6.3 for more on this.

c. The peeled skin from a fairly well bonded blue adhesive peel specimen was examined under the microscope. There was evidence that the gold colored, chromate coating was pulled from the skin with the adhesive.

d. The peeled skin from a pink adhesive peel specimen was examined. There was no clear evidence that the skin had ever had a conversion coating or that fogging of the skin had occurred although the pattern of the core was visible on the skin.

5.6.2 Adhesive Tests

a. About 1000 specimens of the blue adhesive were peeled from delaminated panels. These specimens were the hexagonal shaped pieces of the adhesive left on the skin after delamination. The thickness of about 250 of these specimens was measured in an effort to determine the consistency of the adhesive control. The average thickness was .011 in with a range of from .001 in. to .017 in. This is not the thickness of the adhesive as applied because some of it is drawn to the paper core forming the fillet. The fillet is the portion of the adhesive that provides the bond strength.

b. Specimens of blue and pink adhesive from the skins of peel test specimens were collected. These adhesive specimens were arbitrarily rated according to the ease at which they were peeled from the skin.

These specimens were used in the Thermal-Mechanical Analysis instrument in an attempt to discover any appreciable difference in the adhesive from good and poor bonded areas. There was some differences from specimen to specimen, but the only clear-cut conclusion was that the softening point of the pink adhesive was higher than the blue.

5.6.3 <u>Degradation of Bond Following Lamination</u>

a. It was observed following the 100% humidity conditioning of the specimens that the bond deteriorated on the blue and pink adhesive specimens. (See Figure 11).

The skins of the blue adhesive specimens appeared to be corroded and very little of the chromate coating was visible (See Figure 15). The skins and cores of these specimens were examined under the microscope. This examination indicated that something spreading out from the edges of the core paper was attacking the coating and/or the aluminum of the skins. Figure 16 is a close-up view (2X) of one of these skins. The chromate coating (pink striated surface) is still present in the areas close to the adhesive (blue) and directly under the edge of the paper where it had been pressed on the skin. (Note the dark lines between the white areas.)

b. Similar investigation of the pink adhesive skin after 100% humidity conditioning gave no evidence of corrosion although the bond had been reduced considerably. The pattern of the honeycomb core was visible on the skin from an amber colored stain. This stain covers an area much like the corrosion on the blue adhesive skins.

c. These microscopic observations led to the development of a simple test program to determine if the corrosion and amber staining are caused by something being carried from the paper or foam by the moisture.

Samples of core materials from pink and blue adhesive specimens, with and without the plastic foam, were immersed in small amounts of distilled water (ph 7.4) in semi-sealed (plastic wrap) beakers. The beakers were placed in a 140°F oven for four days. A strong phenolic odor was noticed almost immediately after the beakers became warm.

After the 4 days exposure the water in the beaker with the blue adhesive core had a Ph of 5.7 while the pink was practically unchanged (Ph 7.3). There was no difference between the samples with and without the plastic foam.

Samples of skin material with well bonded blue adhesive were placed in these beakers. The liquids covered about 1/8 in. of the bottom of 2-in long skin material. After 3 days at 73°F in the unsealed beakers there was no apparent change in the skins.

The beakers were then heated until the liquids boiled for 4 hours. The bond deteriorated significantly, but there was no apparent change in the skins.

The same test was run overnight, but with a dilute solution of phenolic and distilled water as the liquid. All the adhesive dropped off the skin which was slightly fogged.

6. Discussion of Results

6.1 Comparison of Panels

6.1.1 Honeycomb Cores

The requirements for the core are not clearly stated in ZSMS-25, but from the discussion of Section 1.3 (above) it would seem that the core should have a minimum shear strength of 190 psi with a minimum shear modulus of 14,000 psi.

Two tests in this study were used to determine these values; the compressive shear and the flexural tests. The plate-shear values obtained from the compressive shear test are average properties of a fairly large specimen. The flexural-shear values are for the weakest section in a relatively small specimen. This means that the shear plate values should be slightly higher than the flexural shear values for identical cores.

In this study the core material was not identical from specimen to specimen of the same panel because of the presence of core splices in some specimens. ZSMS-25 states (Section 3.2.3) that the core splices "shall maintain structural integrity." This requirement means that the performance of spliced specimens should be as good or better than un-spliced specimens.

The average shear data presented in Tables 6 and 7 for the 73°F - 50% RH specimens indicate that shear strength values for the two tests are comparable but with the plate shear values slightly higher.

The shear modulus values are comparable for the 134 and 137 unspliced specimens but not for the others. A splice, being more rigid than the core, is a variable whose effect depends on the test method and on its position in the specimen.

The plate shear values are considered to be more reliable than the flexural values because of the assumptions made in calculating the flexural shear modulus and because of the effect of the core splices.

The results indicate that the effective shear strength of the core in the blue adhesive samples was less than the 190 psi presumedly required. The shear strengths of the cores in the pink and yellow adhesive specimens exceeded this requirement. The maximum shear stresses listed in the tables can be considered shear strengths only for those specimens which failed in core shear. Only 4 of the 30 flexural specimens conditioned at 100% RH (Table 4b) failed in shear. This means that the shear strengths of the wet cores were not determined.

All samples met the plate shear modulus requirement of 14,000 psi for dry honeycomb. The shear moduli of the wet specimens were considerably less than for the dry specimens.

6.1.2 Adhesive Bond

The tensile and peel test results (Tables 3a, 3b and 7) point out the one large difference between the various panels. Clearly the bond of the yellow adhesive samples was much better than for the pink and blue adhesive samples.

The flexural shear test results for specimens conditioned at 120°F - 100% RH indicate bond failure in 7 of 12 blue adhesive specimens, in 6 of 6 pink adhesive specimens, and none in 12 yellow adhesive specimens. Since none of the dry specimens failed in bond we can safely say that the high humidity must have greatly affected the bond of the blue and pink adhesive panels.

From the discussion in Section 5.6.1 and 5.6.3 (above) it would also be safe to say that lower humidities would also affect the bond of the blue adhesive panels. Because of the appearance of the skins from some samples it cannot by said the primary cause of the delamination in the Ward Containers. was due to this humidity effect after lamination.

A standard text book on adhesives [1/] states that epoxy-nylon adhesives (epoxy-polyamide) "deteriorate very rapidly when exposed to moisture or high-humidity conditions." Furthermore, he states that the cohesive strength of these adhesives decreases rapidly with an increase in bond-line thickness. This factor is complicated by the expansion of the adhesive which can fill a gap of as much as .020 in. with a film of only .010 in. thick.

The blue and pink adhesives used in the panels were epoxy-polyamide formulations. Test are now underway to determine the effect of various environmental factors on the bond and cohesive strength of the blue adhesive.

[1/] Cagle, Charles V., "Adhesive Bonding" McGraw-Hill Book Company, 1968.

The precision of the peel test data reported here is not good. This lack of precision is due to two factors.

1. The major part of the force required to peel the skin from the sandwich is used just to bend the relatively thick skin and the adhesive layer on the skin. The significant data for this test is the difference between the sandwich peeling force and the skin bending force. This difference was as small as 8 percent of the measured peeling force. The difference varies with the thickness and physical properties of the adhesive layer as well as the skin.

   In our tests an attempt was made to prepare the skin for bending so that the adhesive thickness was about the same as on the skin peeled from the sandwich. The thickness and amount of adhesive left on the skin varied considerably from specimen to specimen and sometimes even on the same specimen.

2. The values reported for the peel tests neglect the effect of the splices which was very significant.

7. Acknowledgements

Of the many individuals and agencies who assisted and cooperated in this work special mention should be made of the following:

Mr. John Wheeler, Army Natick Laboratories

Mr. Frank Rankin and his staff of the NBS Structures Laboratory

Mr. K. F. Plitt, NBS Plastics and Textiles Section

Mr. Max Tryon, NBS Materials and Durability Section

Table 1 Sandwich Panel Samples Received

Sample[1] No.	Identification[1]	Date Received	Size of Sample in x in	Adhesive Color	Condition
134A	Bottom hinged	4/21/70	27 x 32	Blue	Good
B	Section; top edge	4/21/70	27 x 32	Blue	Good
C	L to R from level	4/21/70	27 x 32	Blue	Delam-one corner
137A	Top hinged	4/21/70	27 x 32	Blue	Good
B	edge	4/21/70	27 x 32	Blue	Good
90A	Hinged roof panel	4/14/70	27 x 32	Pink	Good
B		4/14/70	27 x 32	Pink	Good
139A	Hinged roof panel	4/14/70	27 x 32	Yellow	Good
B		4/14/70	27 x 32	Yellow	Good
143A	Hinged roof panel	4/14/70	27 x 32	Yellow	Good
B		4/14/70	27 x 32	Yellow	Good
C13 S/N-1	Verification Sample	4/10/70	15 x 15	Blue	Good
C13 S/N-2	Verification Sample	4/10/70	15 x 15	Blue	Good
C17 S/N-1	Verification Sample	4/10/70	15 x 15	Blue	Good

[1] Identification and sample No. marked on samples by supplier.

Table 2 - Individual Test Results - Compressive Shear

(4 1/4" x 25 1/2" Specimens)

Sample No.		Specimen No.	Max Shear Stress psi	Modulus psi	Splice
134	A	S1	168	15,700	No
	A	S2	207	16,700	No
	B	S3	148	14,800	No
	B	S4	202	15,800	No
137	A	S1	123	14,800	No
	A	S2	153	16,800	No
	B	S3	165	17,400	No
	B	S4	212	17,900	No
90	A	S1	237	29,200	Yes
	A	S2	186	25,200	Yes
	B	S3	215	28,700	Yes
	B	S4	240	32,500	Yes
139	A	S1	262	23,100	Yes
	A	S2	259	24,900	Yes
	B	S3	245	19,400	Yes
	B	S4	246	30,500	Yes
143	A	S1	256	23,100	Yes
	A	S2	260	25,000	Yes
	B	S3	259	23,300	Yes
	B	S4	253	21,000	Yes

Notes:

1. Modulus is the secant value at 90 psi shear stress
2. All specimens failed by shear of the core
3. All specimens conditioned in lab air at 73°F - 50% RH

Table 3a - Average Tensile Test Results [1] - Container Samples

(2" x 2" Specimens)

Sample No.	Adhesive Color	Maximum Tensile Stress Average psi	Maximum Tensile Stress Range psi	No. of Specimens	Typical Failure	Spliced Specimens
134	Blue	114	28-225	7	Bond	0
137	Blue	62	12-175	7	Bond	0
90	Pink	135	13-391	17	Bond	6
139	Yellow	435	290-554	8	Core	4
143	Yellow	411	300-560	6	Core	2

1 Specimens conditioned at 73°F - 50% RH.

TABLE 3b Individual Tensile Test Results[1] - Verification Samples

(2" x 2" Specimens)

Specimen[3]	Maximum Tensile Load lb	Maximum Tensile Stress psi	Thickness[2] of Adhesive
C13 S/N-1 A	770	193	Medium
B	650	163	Medium
C	800	200	Medium
Average	740	185	--
C13 S/N-2 A	340	85	Light
B	160	40	Light
C	580	145	Medium
Average	360	90	--
C17 S/N-1 A	370	93	Light
B	1330	333	Heavy
C	220	55	Light
Average	640	160	--
Grand Average	--	145	--

[1] Initial failure for all specimens was by cohesive failure in the adhesive. Specimens conditioned at 73°F - 50% RH.

[2] Thickness of adhesive - visually judged light, medium or heavy by observation of the relative size of the fillet.

[3] The adhesive for all these specimens was blue.

Table 4a - Individual Test Results - Flexural Shear

(4 1/4" x 12" specimens conditioned at 73°F - 50% RH)

Sample No.	Specimen No.	Max Shear Stress psi	Shear Modulus* psi	Splice in Specimen?	Type of Failure
134A	F1	198	16,300	No	Core shear
A	F2	115	13,600	No	Core shear
B	F3	211	17,600	No	Core shear
B	F4	129	14,100	No	Core shear
137A	F1	201	15,600	No	Core shear
A	F2	135	12,100	No	Core shear
B	F3	187	13,200	No	Core shear
B	F4	213	12,400	No	Core shear
90A	F1	256	22,700	Yes	Core shear & Bond at splice
A	F2	216	16,300	Yes	Core shear
B	F3	187	20,900	Yes	Core shear
B	F4	206	19,900	Yes	Core shear
139A	F1	238	19,700	Yes	Core shear & bond at splice
A	F2	143	19,900	Yes	Core shear & at splice
B	F3	278	18,400	No	Core shear
B	F4	286	19,700	No	Core shear
143A	F1	140	17,100	Yess	Core shear
A	F2	107	17,700	Yes	Core shear at splice
B	F3	251	19,700	No	Core shear
B	F4	249	19,700	No	Core shear

*Secant modulus at 90 psi shear stress

Table 4b - Individual Test Results - Flexural Shear

(4 1/4" x 12" specimens conditioned @ 120°F - 100% RH)

Specimen No.	Max Shear Stress psi	Shear Modulus* psi	Splice in Specimen?	Type of Failure
134 A-H1-1	98	8,200	No	Core Shear
H1-2	116	7,100	No	Core Crushing
H1-3	65	11,800	No	Bond
B-H2-1	85	8,200	Yes	Core shear and bond at splice
H2-2	101	7,700	No	Bond
H2-3	66	11,000	No	Bond
137 A-H1-1	38	--	No	Bond
H1-2	45	--	No	Core Crushing
H1-3	59	--	No	Bond
B-H2-1	52	--	Yes	Core Crushing and bond
H2-2	54	--	No	Bond
H2-3	63	--	No	Bond
90 A-H1-1	11	--	No	Bond
H1-2	0	--	No	Separated before test
H1-3	0	--	No	Separated before test
B-H2-1	26	--	No	Bond
H2-2	28	--	No	Bond
H2-3	23	--	No	Bond
139 A-H1-1	143	7,400	No	Crushing of core
H1-2	136	8,580	No	Crushing of core
H1-3	137	7,580	No	Crushing of core
B-H2-1	118	7,590	No	Crushing of core
H2-2	148	7,070	No	Crushing of core
H2-3	141	7,720	No	Crushing of core
143 A-H1-1	89	--	Yes	Core Shear at splice
H1-2	112	7,540	No	Core Shear
H1-3	97	--	Yes	Crushing at splice
B-H2-1	128	7,410	No	Crushing of core
H2-2	122	7,180	No	Crushing of core
H2-3	104	--	No	Crushing of core

*Modulus @ 90 psi shear stress

Table 4c - Individual Test Results - Flexural Shear

(4 1/4" x 12" specimens conditioned @ 160°F - 0% RH)

Specimen No.	Shear Strength psi	Shear Modulus* psi	Splice in Specimen?	Type of Failure
134 A-A1-1	124	14,600	No	Core shear
A1-2	206	18,600	No	Core shear
A1-3	127	19,700	No	Core shear
B-A2-1	122	16,500	No	Core shear
A2-2	181	18,100	No	Core shear
A2-3	105	12,200	No	Core shear
137 A-A1-1	89	14,600	No	Core shear
A1-2	126	13,900	No	Core shear
A1-3	44	--	No	Core shear
B-A2-1	134	12,400	No	Core shear
A2-2	226	16,700	No	Core shear and bond
A2-3	124	15,600	No	Core shear
90 A-A1-1	115	17,500	Yes	Core shear near splice
A1-2	99	19,300	Yes	Bond
A1-3	125	16,400	Yes	Core shear at splice
B-A2-1	148	19,700	No	Core shear
A2-2	164	22,600	Yes	Core shear
A2-3	173	19,200	No	Core shear
139 A-A1-1	255	15,500	Yes	Core shear
A1-2	223	21,600	Yes	Core shear
A1-3	277	19,700	Yes	Core shear at splice
B-A2-1	234	20,400	Yes	Core shear
A2-2	160	18,800	Yes	Core shear across splice
A2-3	156	16,600	No	Core shear
143 A-A1-1	227	20,900	Yes	Core shear next to splice
A1-2	249	21,600	Yes	Core shear
A1-3	216	16,000	No	Core shear
B-A2-1	145	20,500	Yes	Core shear across the splice
A2-2	163	20,600	Yes	Core shear
A2-3	202	16,700	Yes	Core shear at splice

Table 5a Individual Peel Test Results, Container Samples
(3" x 12 1/2" Specimens Conditioned at 73°F - 50% RH)

Specimen	Sandwich Peeling Force		Skin Bending Force		Median Peeling Torque[1]		Type[2] of Failure
	Maximum	Median	Maximum	Median	Sandwich	Adhesive Bond	
	lb.	lb.	lb.	lb.	in.-lb./in.	in.-lb./in.	
134A -1	110	95	89[3]	84[3]	12.6	1.8	2
2	104	101	--	--	13.6	2.8	2
3	107	99	--	--	13.3	2.5	2
B -4	111	98	--	--	13.1	2.3	2
5	109	97	--	--	13.0	2.2	2
6	112	99	--	--	13.3	2.5	2
C -1	120	109	103	98	15.0	1.8	2
2	127	114	109	104	15.8	1.7	2
3	121.	110	100	96	16.3	2.3	2
137A -1	113	98	90[3]	86[3]	13.1	2.0	2
2	106	95	--	--	12.6	1.5	2
3	109	98	--	--	13.1	2.0	2
B -4	109	96	--	--	12.8	1.7	1 & 2
5	108	95	--	--	12.6	1.5	2
6	102	93	--	--	12.2	1.2	2
90A -1	125	106	84[3]	80[3]	14.5	4.3	1 & 2
2	117	103	--	--	14.0	3.8	1 & 2
3	109	97	--	--	13.0	2.8	1 & 2
B -4	106	94	--	--	12.5	2.3	1 & 2
5	105	93	--	--	12.2	2.2	1 & 2
6	104	94	--	--	12.5	2.3	1 & 2
139A -1	163	132	77[3]	72[3]	18.8	10.0	2 & 3
2	149	124	--	--	17.5	8.7	2 & 3
3	154	127	--	--	18.0	9.5	2 & 3
B -4	185	144	--	--	20.8	12.0	2 & 3
5	177	142	--	--	20.5	11.7	2 & 3
6	190	151	--	--	22.0	13.2	2 & 3
143A -1	166	128	89[3]	87[3]	18.2	6.8	2 & 3
2	175	144	--	--	20.8	9.5	2 & 3
3	157	133	--	--	19.0	7.7	2 & 3
B -4	158	137	--	--	19.7	8.4	2 & 3
5	162	129	--	--	18.3	7.0	2 & 3
6	138	119	--	--	16.6	5.3	2 & 3

[1] & [2] See note [1] & [2] Table 5b
[3] Average of two tests

Table 5b Individual Peel Test Results, Verification Samples

(3" x 12 1/2" specimens conditioned @ 73°F - 50% RH)

Specimen	Sandwich Peeling Force		Skin Bending Force		Median Peeling Torque[1]		Type of[2] Failure
	Maximum lb.	Median lb.	Maximum lb.	Median lb.	Sandwich in.-lb./in.	Adhesive Bond in.-lb./in.	
C13 S/N-1 A	102.5	90.7	79.5	76.0	11.9	2.5	1, 2 & 3
B	102.0	90.7	79.5	76.0	12.7	2.5	1 & 2
C	93.2	83.6	79.5	76.0	11.5	1.3	1 & 2
C13 S/N-2 A	103.5	89.2	79.0	75.6	12.5	2.3	1 & 2
B	89.8	82.3	79.0	75.6	11.2	1.1	1, 2 & 3
C	102.1	89.2	79.0	75.6	12.5	2.3	1 & 2
C17 S/N-1 A	96.8	85.7	83.0	80.5	11.9	0.9	1 & 2
B	106.0	94.0	83.0	80.5	13.3	2.3	1, 2 & 3
C	97.0	86.3	83.0	80.5	12.0	1.0	1 & 2

[1] Peeling Torque, in - lb./in. = .167 (F_p - F_o)

Where F_p = Sandwich peeling force, lb.

For the sandwich, F_o = Force required to overcome the drum troque, lb. (19.3 lb. for C13-S/N-1A sandwich and 14.5 lb. for all others)
For the bond, F_o = Skin bending force, lb.

[2] Failures:

1) Bond to facing
2) Cohesive failure in adhesive
3) Core failure

Table 6 - Average Test Results[1] - Compressive Shear

Sample No.	Adhesive Color	Maximum Shear Stress psi	Spliced Specimen	Modulus[2] psi
134	Blue	178	None	15,700
137	Blue	163	None	16,700
90	Pink	220	4	28,900
139	Yellow	253	4	24,500
143	Yellow	245	4	23,200

[1] Average of 4 specimens conditioned @ 73°F - 50% RH

[2] Secant modulus at 90 psi shear stress

Table 7 - Average Test Results - Flexural Shear

Sample	Adhesive Color	Conditioned at 73°F-50% rh			Conditioned at 120°F-100% rh			Conditioned at 160°F-0% rh		
		Specimens Spliced	Max. Shear Stress psi	Modulus* 10^3 psi	Specimens Spliced	Max. Shear Stress psi	Modulus* 10^3 psi	Specimens Spliced	Max. Shear Stress psi	Modulus* 10^3 psi
134	Blue	None	163	15.4	1	105	9.0	0	144	16.6
137	Blue	None	184	12.2	1	52	---	0	124	14.6
90	Pink	4	216	19.9	0	22	---	4	137	19.1
139	Yellow	2	236	19.4	0	137	7.7	5	218	18.8
143	Yellow	2	186	18.6	2	109	7.4	5	200	19.4

*Shear Modulus @ 90 psi shear stress

Table 8 Sandwich Peel, Average Test Results[1]

Sample	Number of Specimens	Median Peeling Torque: Sandwich in-lb/in	Median Peeling Torque: Adhesive Bond in-lb/in
134A, B & C	9	14.0	2.2
137A & B	6	12.7	1.7
90A & B	6	13.1	3.0
139A & B	6	19.6	10.9
143A & B	6	18.8	7.5
C13 2/N-1[2]	3	12.0	2.1
C13 S/N-2[2]	3	12.1	1.9
C17 S/N-1[2]	3	12.4	1.4

[1] See Notes for Table 5b

[2] Verification samples

Table 9 - Average Weight Change of Flexural Specimens

Sample	Adhesive	Conditioned @ 120°F - 100% RH Weight Gain Percent	Conditioned @ 160°F - 0% RH Weight Loss Percent
134	Blue	15	0.7
137	Blue	12	0.7
90	Pink	18	1.0
139	Yellow	14	1.0
143	Yellow	13	1.0

Note: Weight gain and loss is computed from initial weight at 75°F 50% RH.

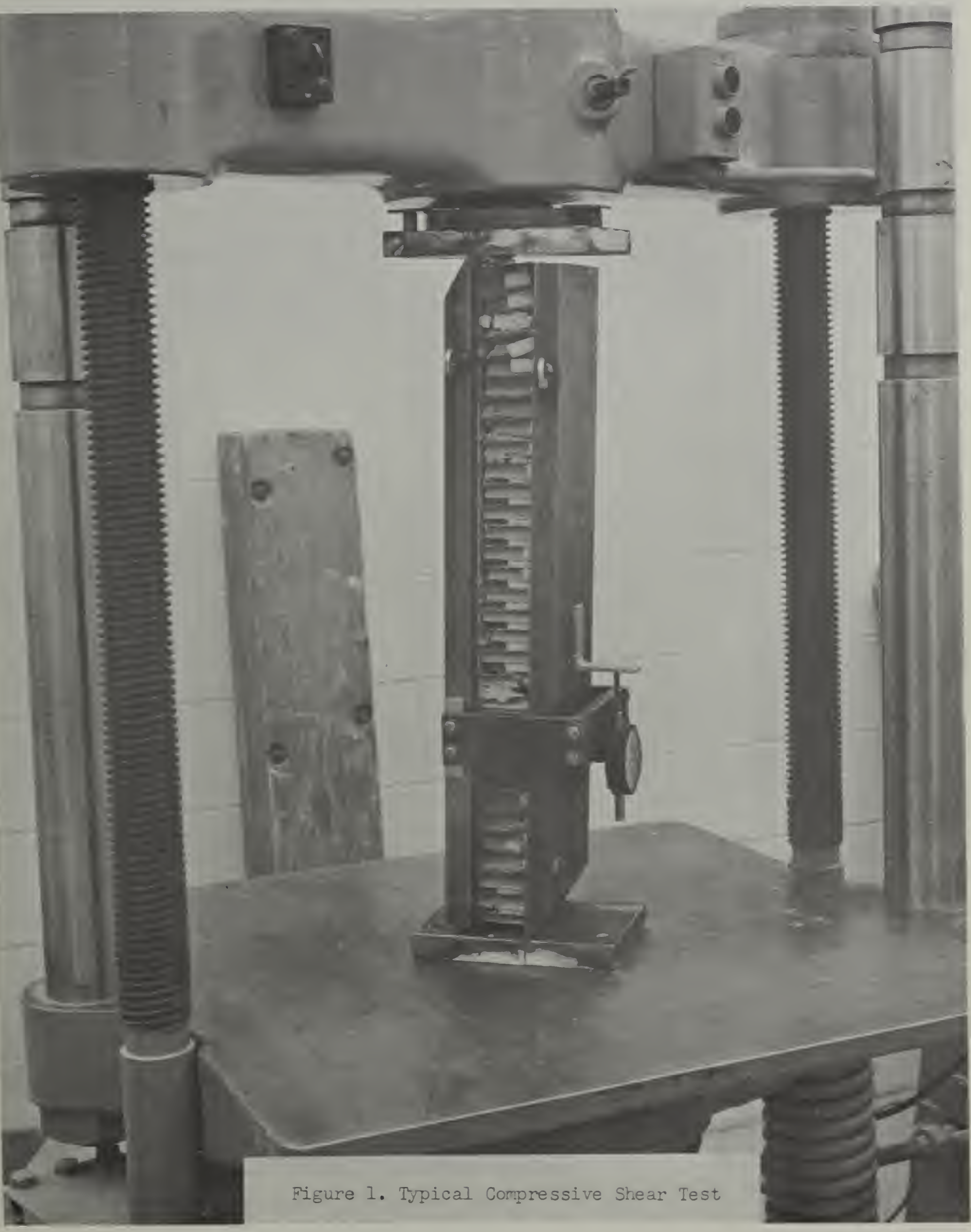


Figure 1. Typical Compressive Shear Test

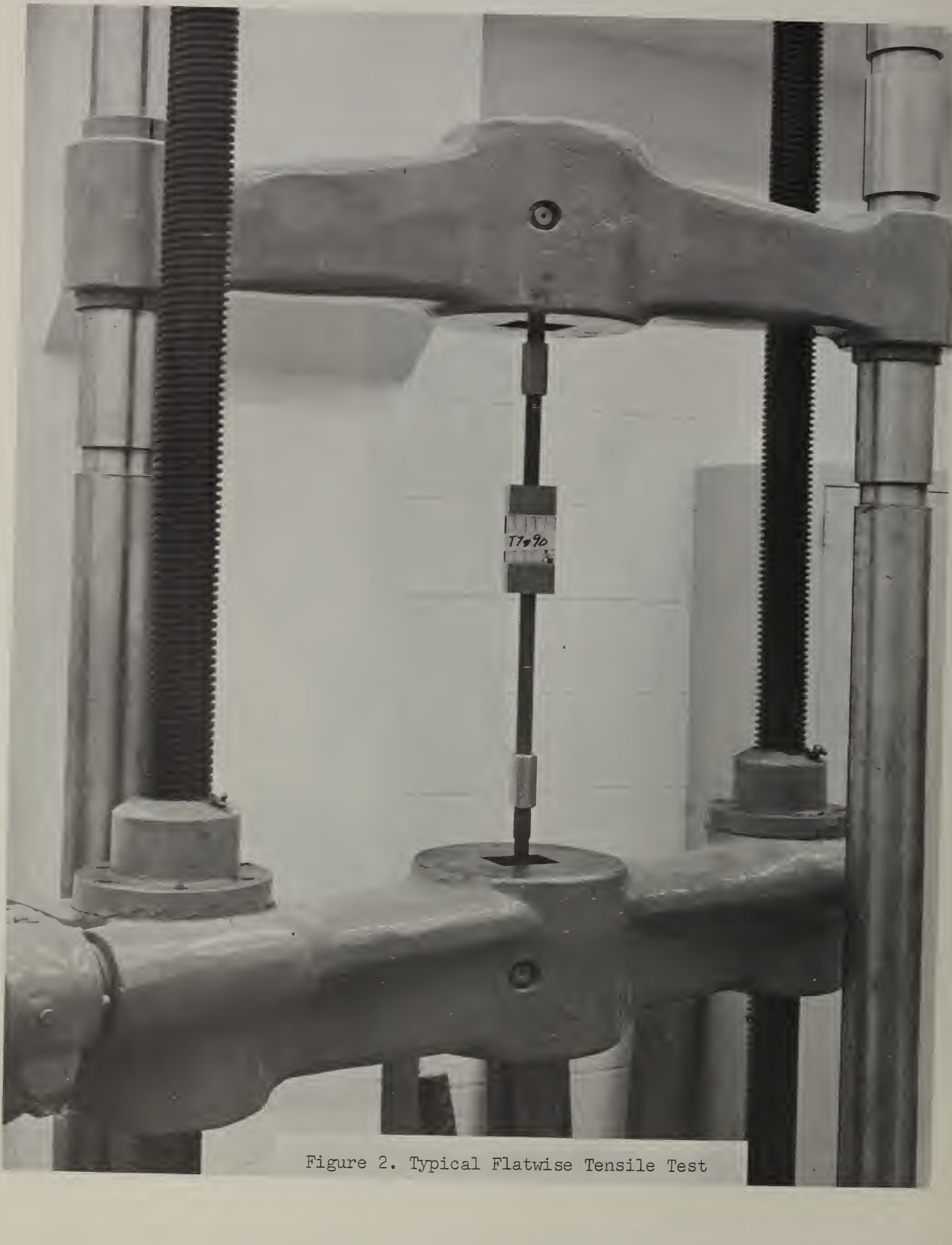


Figure 2. Typical Flatwise Tensile Test

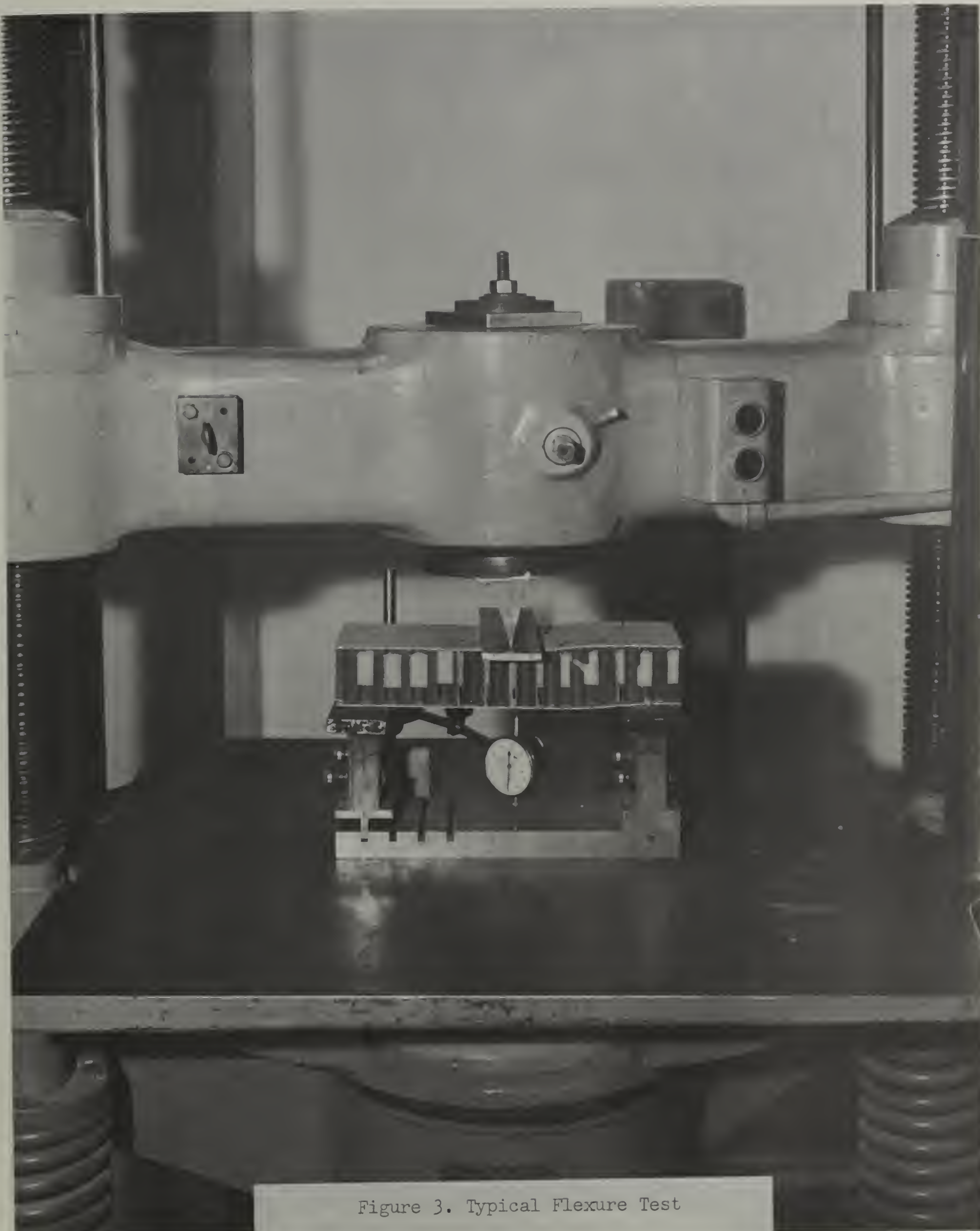


Figure 3. Typical Flexure Test

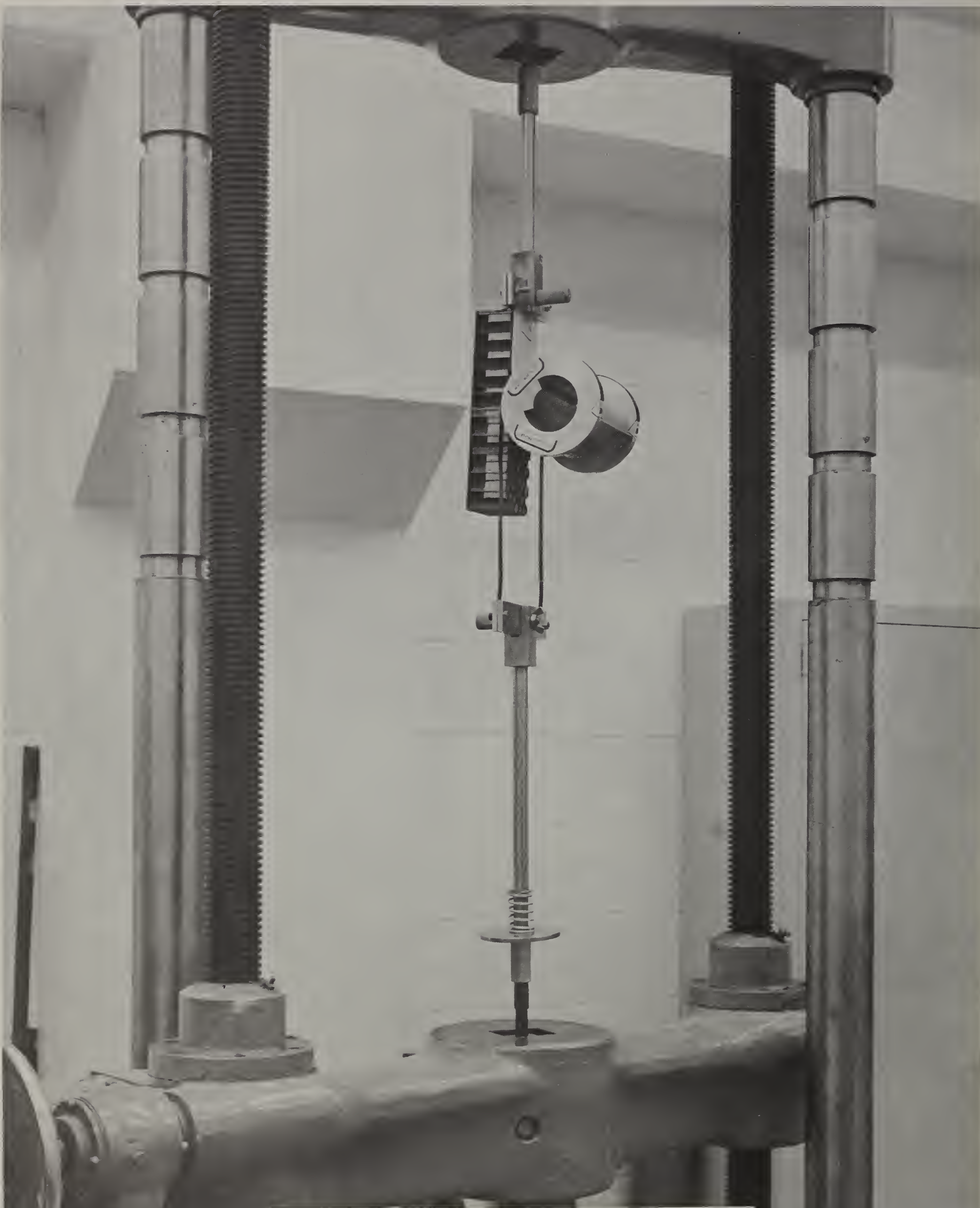


Figure 4. Climbing Drum Peel Test

Figure 5. Compressive Shear Specimen After Test

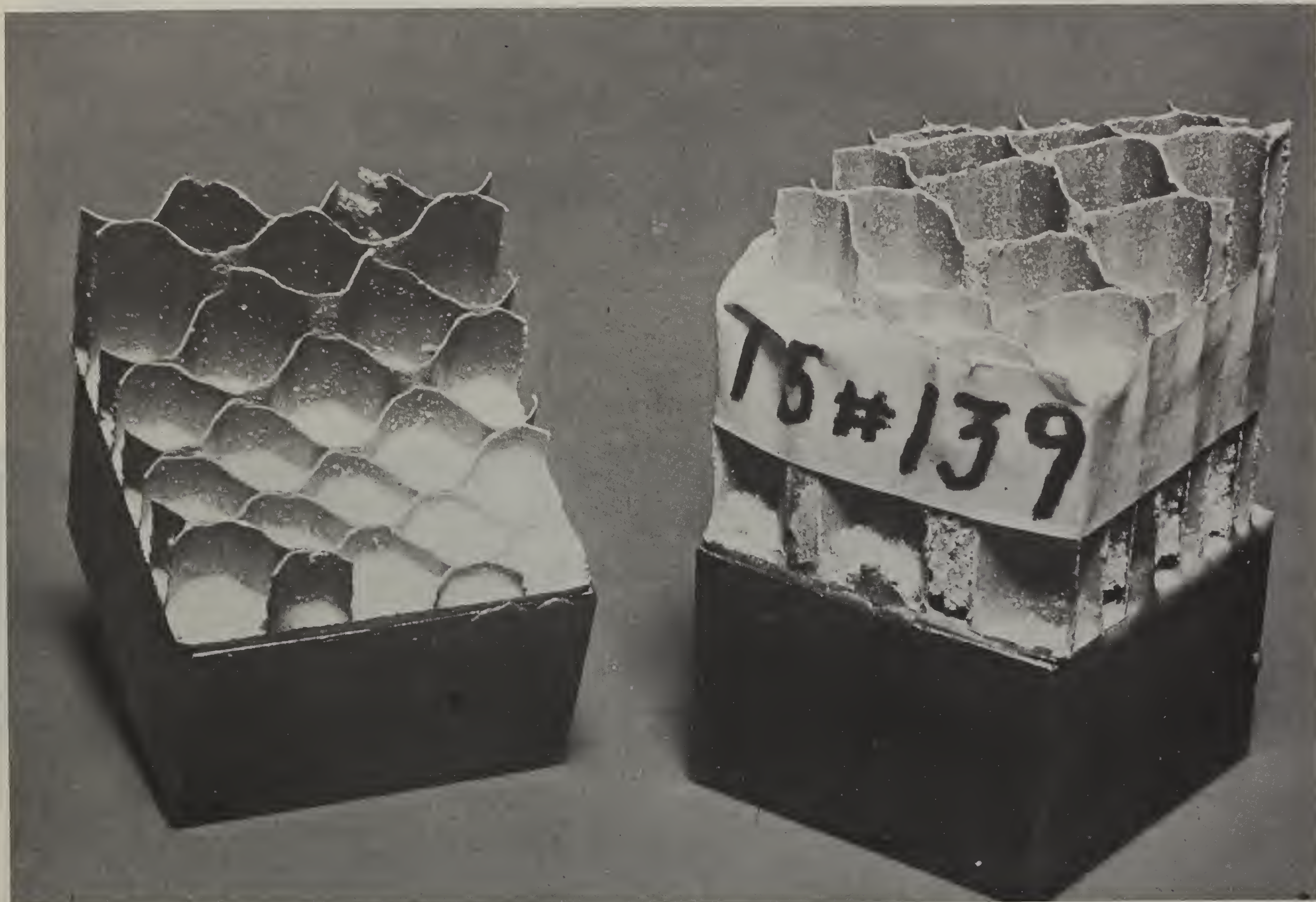


Figure 6. Flatwise Tensile Test - Core Failure

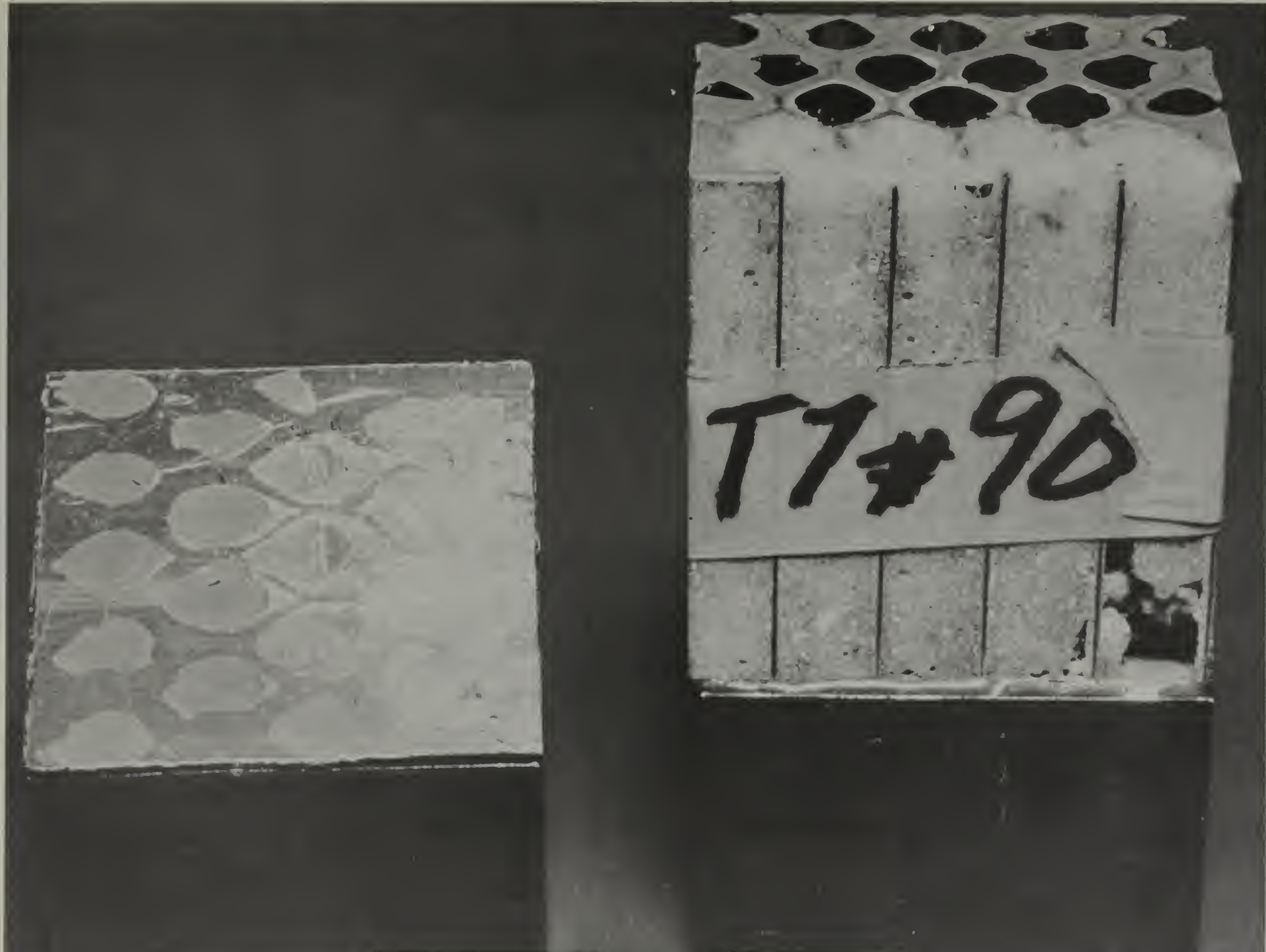


Figure 7. Flatwise Tensile Test - Bond Failure with Spliced Specimen

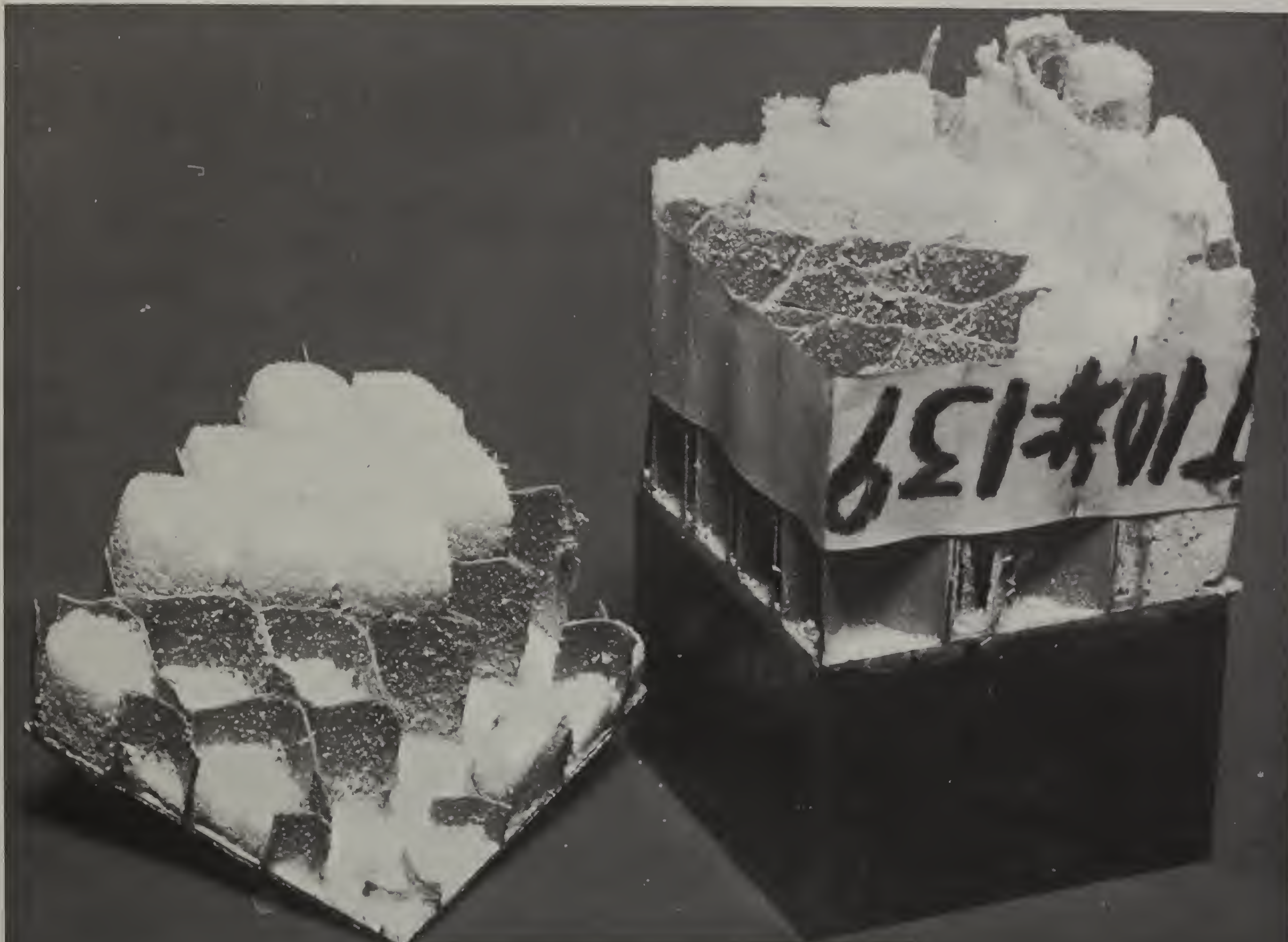


Figure 8. Flatwise Tensile Test - Core Failure with Spliced Specimen

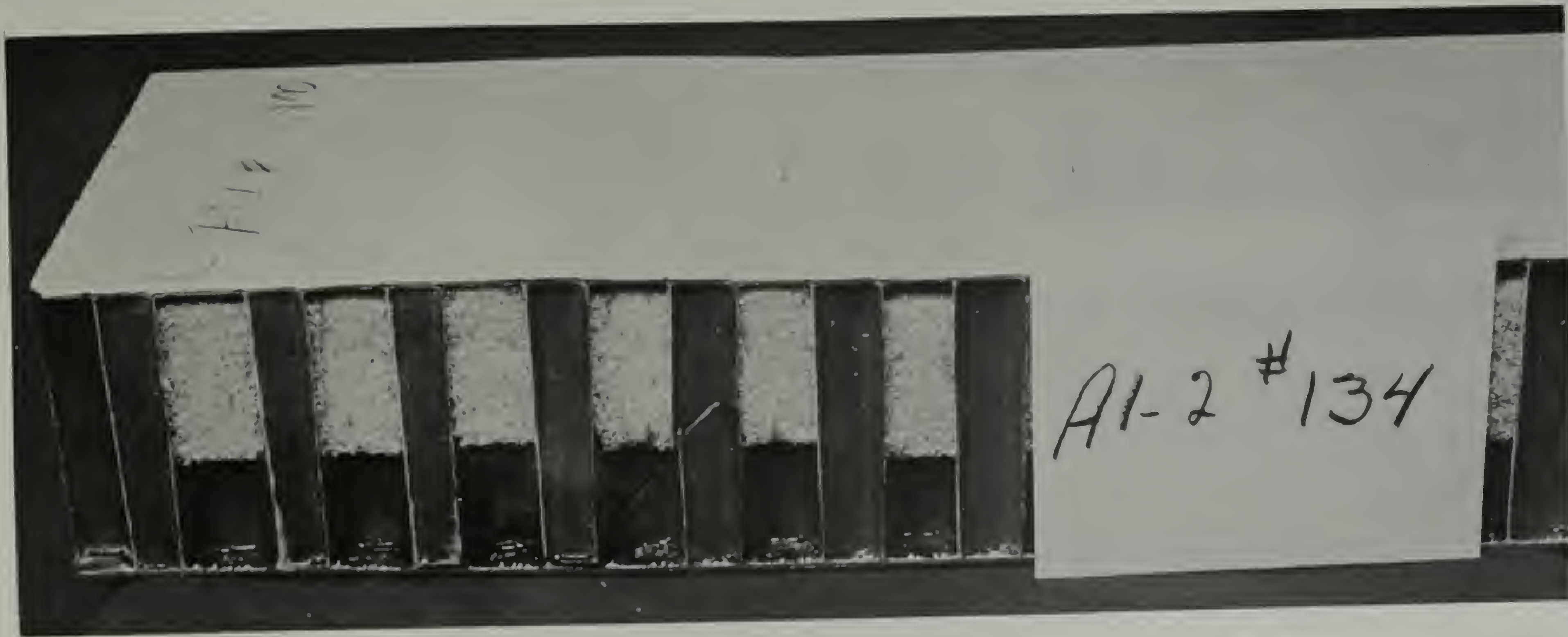


Figure 9. Flexural Shear Failure

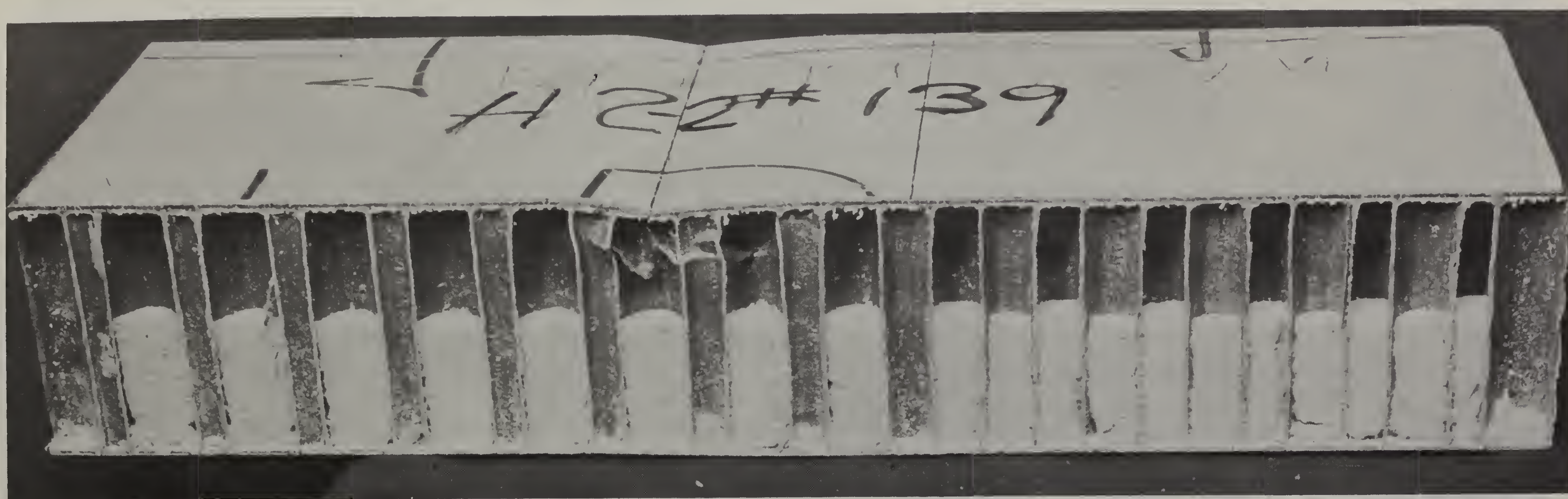


Figure 10. Flexural Humidity Specimen -
Core Crushing

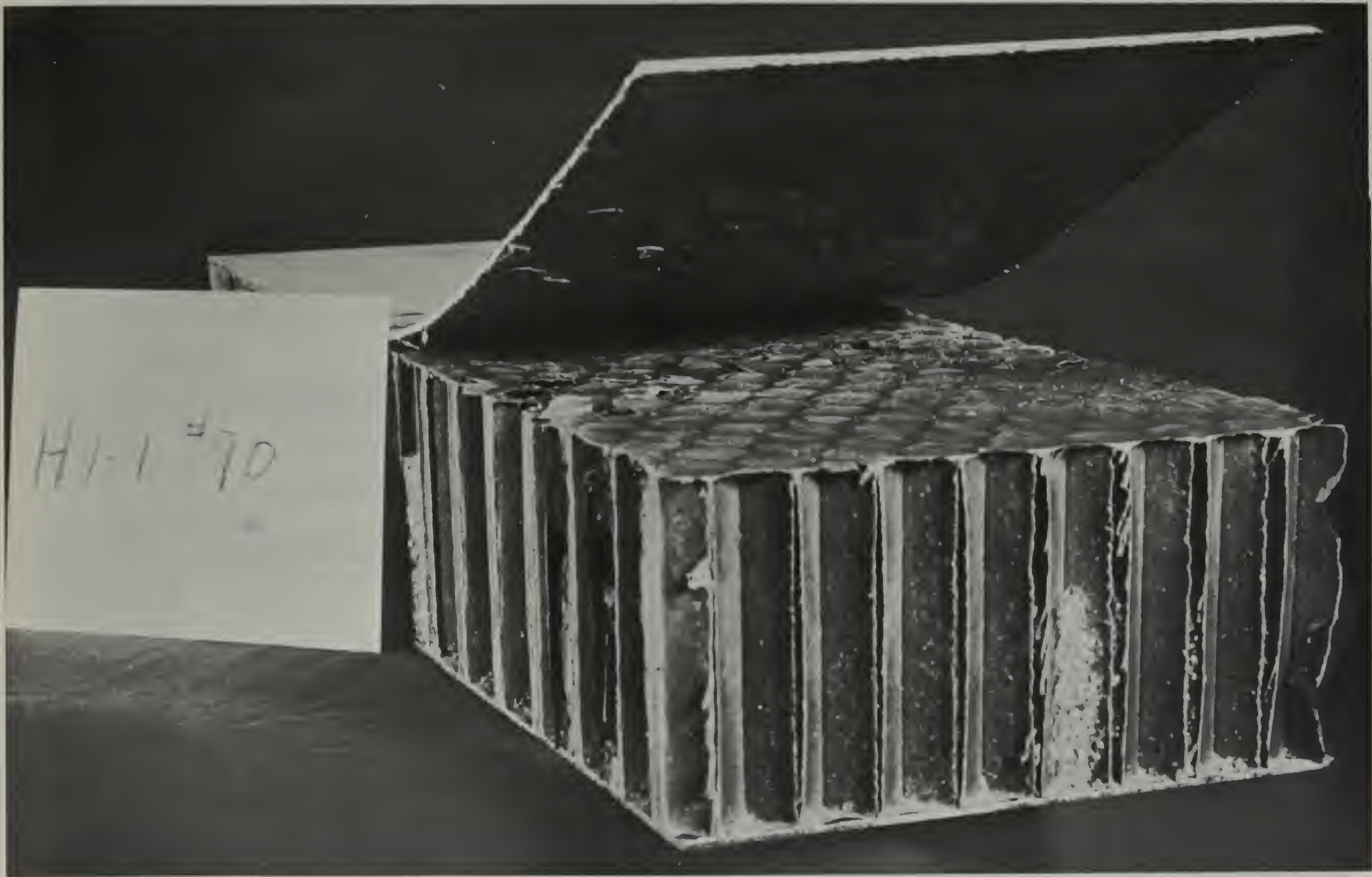


Figure 11. Flexural Humidity Specimen - Delamination Before Test

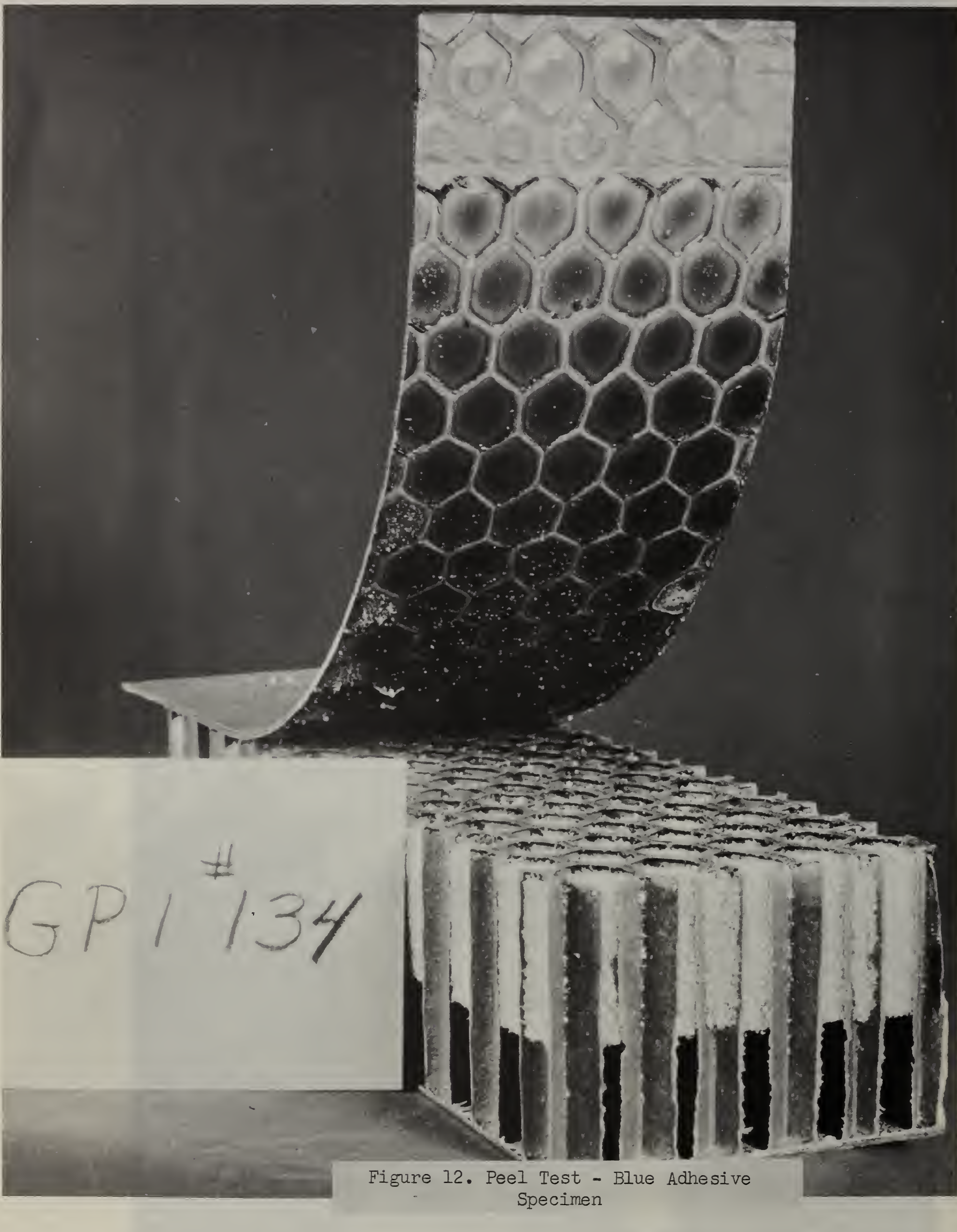
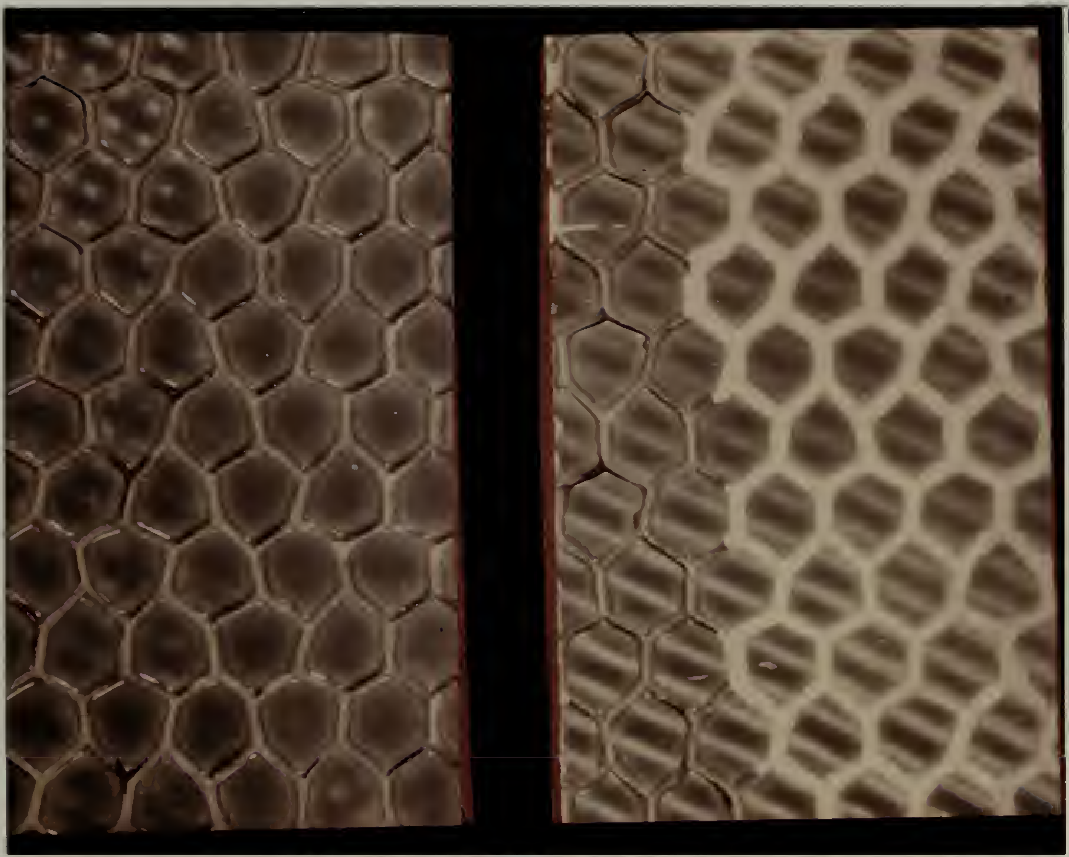


Figure 12. Peel Test - Blue Adhesive Specimen

Figure 13. Peel Test - Pink Adhesive Specimen with Splice

Figure 14. Peel Test - Yellow Adhesive
Specimen with Splice



a. Good bond   b. Poor bond

Figure 15. Skins of specimens from same sample (No. 137) before (a) and after (b) humidity test.

Figure 16. View (2X) of skin after humidity test showing change in the conversion coating under the edges of the paper honeycomb.

24 March 1970

TEST PLAN

SUBJECT: Test Plan for the Combined U.S. Army Natick Laboratories and Missouri Research Laboratories Testing of Honeycomb Sandwiched Panels

1. Reason for Testing:

Due to the considerable number of delaminated panels recently reported from Atlanta General Depot and as a result of decisions reached at a meeting held at MECOM on 20 March 1970 between Government representatives and Missouri Research Laboratory representatives, it was agreed that comparative tests be conducted on sample panels taken from containers in stock at Atlanta General Depot and from unassembled panels recently received at Missouri Research Laboratories.

2. Selection of Panels to be Tested:

a. Missouri Research Laboratories will obtain samples from three (3) unassembled panels of the size used for folding roofs or folding floors. Panels will be selected at random by DCAS personnel at Missouri Research Laboratories and will consist of three (3) panels, one each from three (3) shipments.

b. Government selection of Panels: Samples to be tested by the Government will be obtained from three (3) different Ward Containers now in stock at Atlanta General Depot. Sample will be taken from folding roof panel in either Serial No. 090 or 100 and will be representative of Ward Container Serial Numbers showing delaminations, although neither of these numbers have actually been reported as having delaminations. Samples will also be taken from folding roof panels from two Ward Containers in the range of 130 to 164 and will be representative of the range of serial numbers which have not yet shown delaminations.

3. Testing Responsibility:

Missouri Research Laboratory testing will be accomplished by independent laboratory of their choice. Government testing will be conducted by National Bureau of Standards.

4. Type of Testing:

Shear, tension, flexure and peel tests shall be conducted in accordance with MIL-STD-401B with the humidity and age tests in accordance with ZSMS 25, except as indicated hereinafter. In addition, each humidity and age test shall be followed by a flexure test on specimens of proper size cut from the humidity and age test specimens.

Test	Total Specimens	Specimens (per panel)	Specimen Size (inches)	Miscellaneous
(S) Shear	12	4	4-1/4 X 25-1/2	
(T) Tension	18	6	2 X 2	
(F) Flexure	12	4	4-1/4 X 12	Span length - 10 in
(P) Peel	18	6	3 X 10	
(H) Humidity	6	2	12 X 12	Test report per 4.4 of MIL-STD-401B
(A) Age	6	2	12 X 12	Test report per 4.4 of MIL-STD-401B. Aging temperature 175F $\pm$ 5F

All test reports, with the exception of the shear test report, may omit the data required by the first three sentences of paragraph 4.4 of MIL-STD-401B. However, all test reports shall include a description of the mode of failure during each test (adhesive, core, etc). Two samples shall be cut from each panel in accordance with Figure 1 with test specimens cut from each sample as indicated in Figure 2.

5. Identification of panel samples and specimens:

Each panel specimen shall be marked as shown on Figure 2. In addition, each Missouri Research Laboratory specimen shall contain the lot number and panel number while each specimen from Government panels shall be marked with the Ward Container serial number.

6. Verification Panel Sample:

A verification panel sample shall be cut from each panel. It shall be of the size shown on Figure 1. Verification samples taken at Atlanta General Depot shall be shipped to Missouri Research Laboratory, Attn: Mr. E. Randolph. Verification samples taken at Missouri Research Laboratories shall be shipped to National Bureau of Standards, Attn: Mr. T. Reichard.

7. Shipping Instructions for Panel Specimens:

All panel specimens taken at Atlanta General Depot shall be shipped to:

National Bureau of Standards
Attn: Structures Div.
(Mr. T. Reichard)
Gaithersburg, Maryland

8. Special Instructions:

Prior to cutting panel samples, tap test shall be conducted on areas to be cut out. Panel samples shall have no indication of delaminations. Exercise caution when cutting panel samples and specimens to keep heat and vibration to a minimum.

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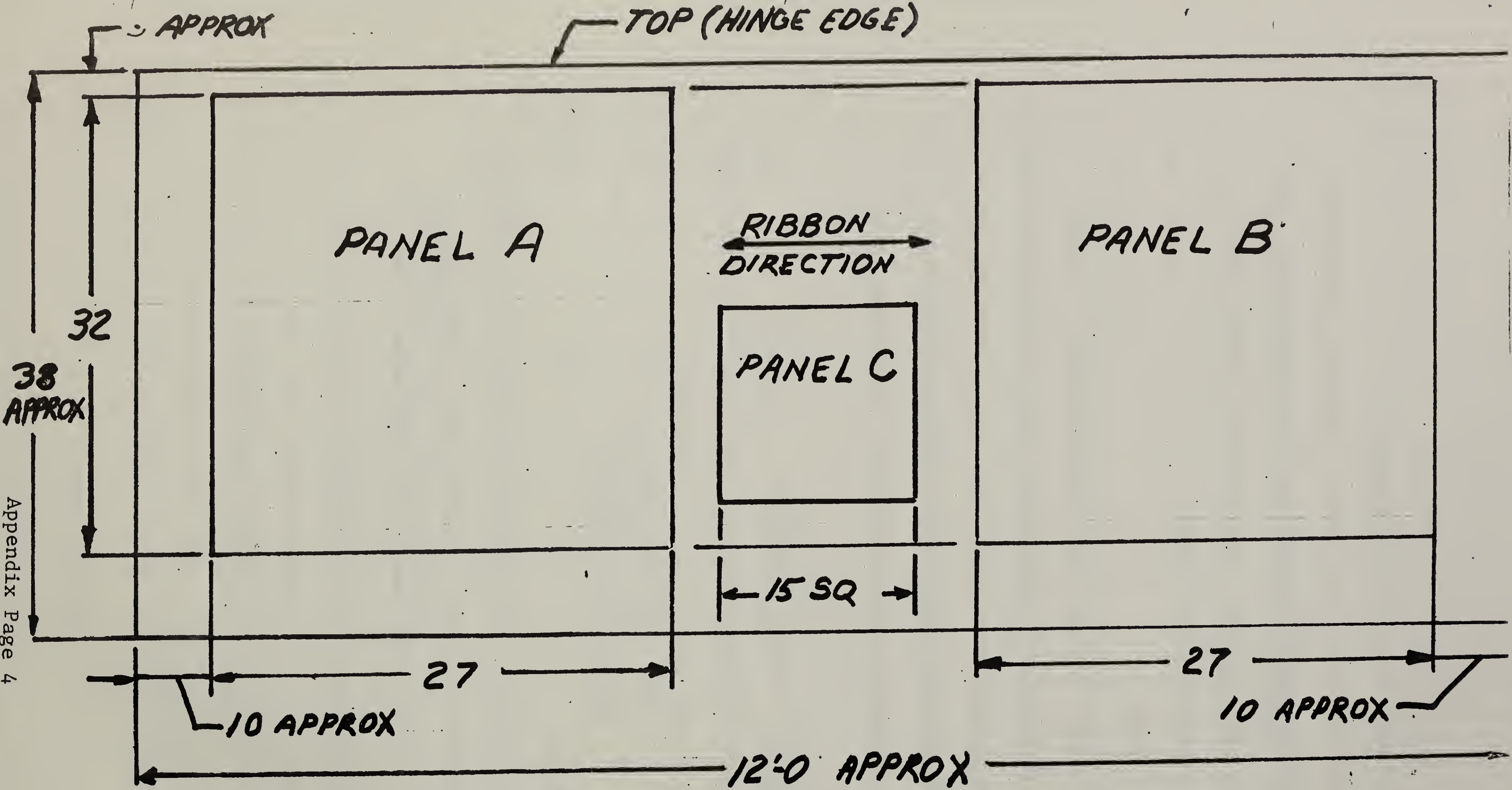
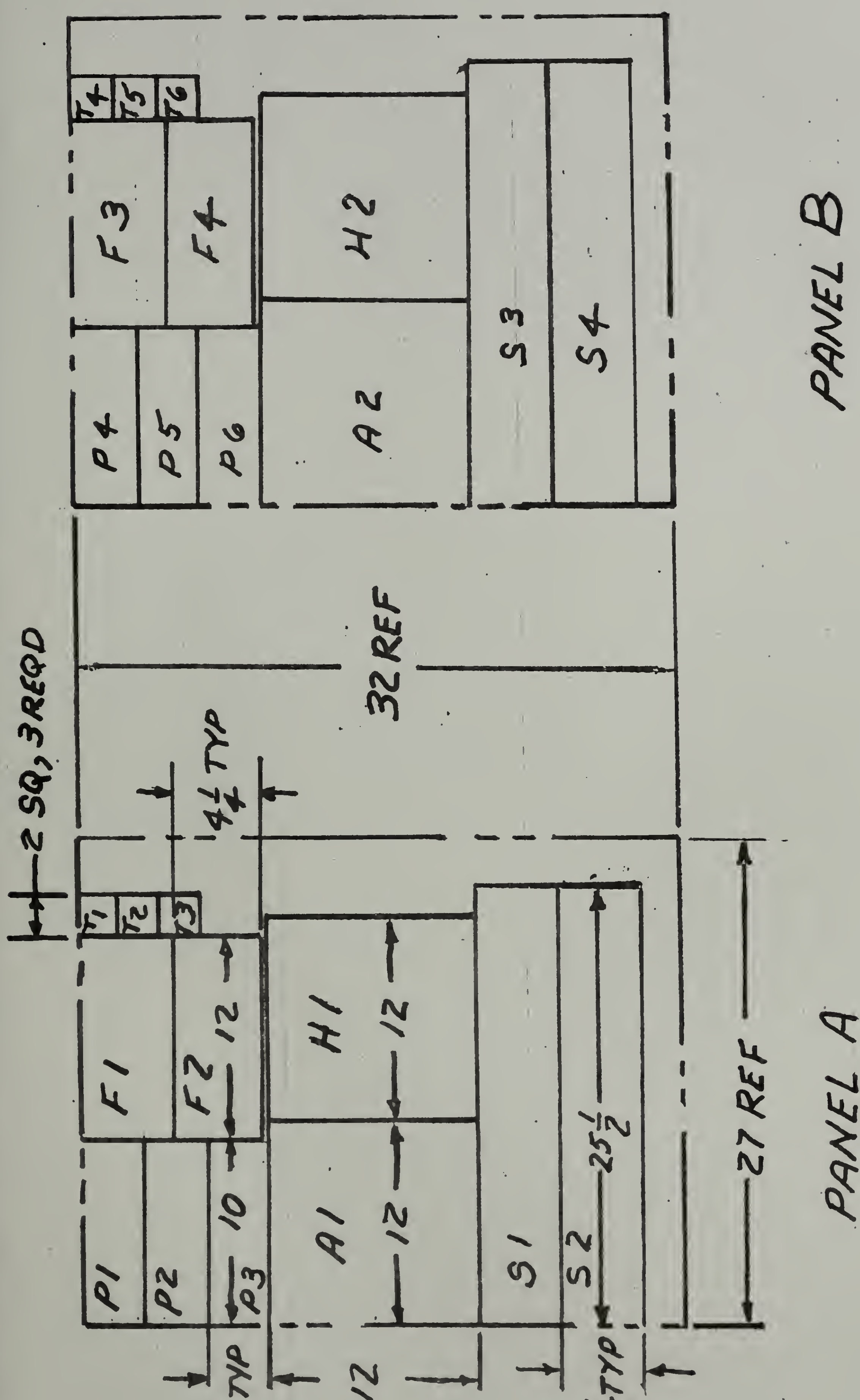


FOLDING ROOF

PANEL A&B ARE SAMPLES
PANEL C IS VERIFICATION SAMPLE

DIMS ARE IN INCHES

FIG 1



DIMS FOR PANEL B ARE IDENTICAL TO PANEL A

FIG 2

DIMS ARE IN INCHES

DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS