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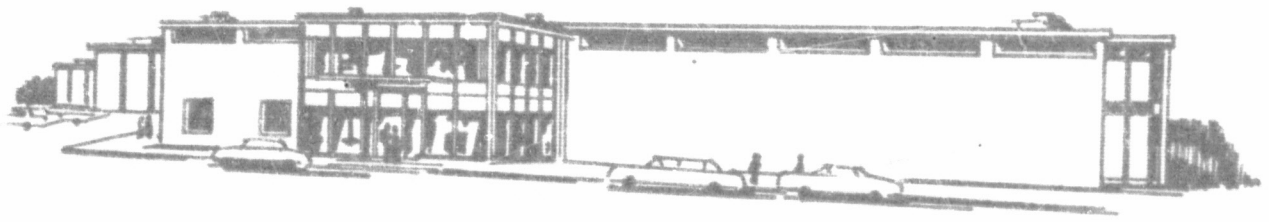
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September 5, 1963

FINAL REPORT

HEAT STABILIZATION STUDY

OF

ADVANCED MATERIALS

Mod 3

CONTRACT NAS5-2834

MODIFICATION 3

SUBMITTED TO

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GODDARD SPACE FLIGHT CENTER
GREENBELT, MARYLAND

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TABLE OF CONTENTS

		Page
I.	INTRODUCTION AND SUMMARY	1
II.	HEAT TREATMENT OF AS-RECEIVED MATERIAL	3
	A. Objective	3
	B. Test Description	4
	C. Test Apparatus	10
	D. Comparison of Brands of Polypropylene	10
	E. Conclusions	17
III.	TEST OF HEAT TREATMENT DURATION EFFECTS	19
	A. Objective	19
	B. Test Description	19
	C. Discussion	20
	D. Conclusion	20
IV.	OTHER HEAT TREATMENT TECHNIQUES	22
	A. Objectives	22
	B. Test Description	22
	C. Conclusions	27
V.	SHRINKAGE TESTS OF LAMINATED POLYPROPYLENE	28
	A. Objective	28
	B. Test Description	28
	C. Conclusions	36
VI.	SHRINK TESTING OF MILLED LAMINATES	37
	A. Objective	37
	B. Test Description	37

VII. MATERIAL TENSILE STRENGTH 56

A. Objective 56

B. Test Description 56

C. Discussion 57

D. Conclusions 65

VIII. OPTICAL PROPERTIES OF POLYPROPYLENE 66

A. Objective 66

B. Test Description 66

C. Discussion 67

D. Conclusions 68

IX. HEAT TREATMENT AND SHRINKAGE OF MYLAR AND MYLAR LAMINATES 70

A. Objective 70

B. Test Description 70

C. Discussion 71

D. Conclusions 74

X. OPTICAL PROPERTIES OF MYLAR 74

A. Test Objectives 74

B. Test Description 74

XI. COLUMN TESTING 75

A. Objective 75

B. Test Description 75

C. Test Apparatus 77

D. Discussion 77

E. Conclusions 88

XII. SPHERE BUCKLING TESTS 89

A. Objective 89

B. Test Description 89

C. Test Apparatus 92

D. Results 92

E. Discussion 94

XIII. SOLAR DISTORTION OF SPHERES 95

A. Objective 95

B. Analysis 96

LIST OF TABLES

		Page
TABLE I	Polypropylene Tensile Strength	12
TABLE II	One-Sided, Chemically Milled, Laminate Shrinkage	55
TABLE III	Spectrophotometer Transmission Values for Polypropylene and Mylar	69
TABLE IV	Percent Shrinkage of Mylar and X-15 Material as a Function of Shrinkage Exposure Duration	73
TABLE V	Echo A-12 Material (Sequential Tests on One Sphere)	92
TABLE VI	Chemically Milled (70 Percent) Material 1.0 Mil Aluminum, 0.6 Mil Polypropylene	93
TABLE VII	Sphere Shrinkage Calculation	101

LIST OF FIGURES

Figure No.		Page
1	Test Set Up	7
2	Measuring Process	9
3	Thermal Shrinkage Vs. Shrink Test Temperatures for Various Heat Treatment Temperatures	13
4	Thermal Shrinkage Vs. Shrink Test Temperatures for Various Heat Treatment Temperatures	14
5	Thermal Shrinkage Vs. Shrink Test Temperatures for Various Heat Treatment Temperatures	15
6	Thermal Shrinkage Vs. Shrink Test Temperatures for Various Heat Treatment Temperatures	16
7	Melting Polypropylene	18
8	Percent Shrinkage as a Function of Heat Treatment Duration	21
9	Laboratory Oven Testing	23
10	Condition of Polypropylene After Oven Heat Treatment	25
11	Delamination During Shrinkage	30
12	Percent Shrinkage of X-32F Material Laminated on Both Sides	32
13	Percent Shrinkage of X-32F Material with Laminate Etched from One Side	33
14	Percent Shrinkage of X-32F Material For Various Combinations of Aluminum Etching	34
15	Printing Resist Pattern with 50 Inch Silk Screen	43

Figure		Page
16	Silk Screening Process Completed	44
17	Continuous Etcher Used in Milking Process	45
18	Percent Shrinkage of 70% Milled Material for Various Shrink Test Temperatures	46
19	Percent Shrinkage of 48% Milled Material for Various Shrink Test Temperatures	47
20	Percent Shrinkage of 70% Milled Material for Various Shrink Test Temperatures	48
21	Percent Shrinkage of 48% Milled Material for Various Shrink Test Temperatures	49
22	Percent Shrinkage of 70% Milled Material for Various Shrink Test Temperatures	50
23	Percent Shrinkage of 70% Milled Material for Various Shrink Test Temperatures	51
24	Percent Shrinkage of 70% Milled Material for Various Shrink Test Temperatures	52
25	Percent Shrinkage of 48% Milled Material for Various Shrink Test Temperatures	53
26	Room Temperature Tensile strength as a Function of Heat Treatment Temperatures	58
27	Room Temperature Tensile Strength as a Function of Heat Treatment Duration	59
28	Polypropylene Strength as a Function of Heat Treatment Duration	60
29	Tensile Strengths of Heat Treated Laminate as a Function of Heat Treatment Temperature (TD)	61

Figure		Page
30	Tensile Strengths of Heat Treated Laminates as a Function of Heat Treatment Temperatures (MD)	62
31	Percent Shrinkage of Mylar as a Function of Shrink Test Temperature	72
32	Buckling Load Vs. Aluminum Thickness 1 1/2 Inches Diameter X 5 Inch Long Columns	79
33	Buckling Load Vs. Percent Aluminum Removed 1 1/2 Inch Diameter X 5 Inch Long Columns	80
34	Buckling Load Vs. Aluminum Thickness 1 1/2 Inch Diameter X 5 Inch Long Non Milled Material Columns	81
35	Buckling Load Ratio Milled Material Compared to Non-Milled Material Column Tests	82
36	Strength to Weight Ratios - 90% Milled Laminate	83
37	Column Loading Test Fixture	84
38	Typical Buckled Column	85
39	Polypropylene (0.6 Mil) Column Prior to Loading	86
40	Polypropylene Column After Loading	87
41	30 Inch Sphere Test Fixture - Sphere With Slight Superpressure	90
42	30 Inch Sphere After Buckling Test	91
43	Sketch of Physical Situation	98
44	Sphere Flattening as a Function of Percent Shrinkage	102

I. INTRODUCTION AND SUMMARY

To improve Echo type satellites over the present configuration which utilizes a laminate of aluminum-Mylar-aluminum, it has been suggested that the spheres be manufactured of a laminate of polypropylene to aluminum (the final report for NAS5-1190 discusses development of such a material). Following lamination, the aluminum would be printed with a pattern so that chemical milling could be utilized to remove upwards of 90% of the aluminum laminate. The advantages of using an aluminum-polypropylene chemically milled material are: greater strength-to-weight ratio, and a lower effective modulus of elasticity. These two advantages lead to a lower weight sphere for a given diameter with higher relative rigidity and greater ease of packing relative to a sphere made of Echo II material. However, polypropylene has a tendency to shrink when exposed to heat. Such a tendency to shrink would cause distortions in a sphere and attendant degradation of the radio frequency reflecting characteristics of the sphere made from a chemically milled laminate. This is undesirable.

To eliminate or reduce the shrinkage in polypropylene film, attention to several factors in addition to simple shrinkage elimination is required. That is, whatever technique is adopted for eliminating or reducing shrinkage must consider the commercial feasibility of treating the polypropylene under production conditions at a minimum cost and a minimum hazard to operating personnel. In addition, the shrinkage elimination technique must not degrade the

mechanical strength of the material nor the bond between the laminate and the polypropylene. Moreover, the infrared and solar transmittance of the polypropylene film should not be degraded to a point at which solar energy causes the material to reach thermal equilibrium at high temperature, since this would cause the sphere to shrink excessively. This study program investigated the shrinkage characteristics of polypropylene films and determined a shrinkage reduction technique for films and film laminates while simultaneously considering the factors mentioned. In addition, an analysis of the shrinkage of a sphere, cylinder buckling tests of laminates, buckling tests of small spheres were conducted and some Mylar shrinkage data are presented as a part of this report.

The results of the test program which are the subject of this report indicate that Union Carbide biaxially oriented polypropylene is the best from the point of view of minimum shrinkage and maximum retention of basic material strength for any given heat treatment temperature. Heat treatment of Union Carbide polypropylene in a glycerine bath at 160 C for 1 minute provides the most economical heat treatment to obtain minimum shrinkage. Tensile strength of this material falls off rapidly at 165 or 170 C and heat treatment above 160 C is therefore, not recommended. Optical tests of the transmission of polypropylene indicate little variation between the untreated material and the treated material, thereby indicating that the heat treatment technique does not affect the average optical transmission properties of the polypropylene.

Glycerine, the heat treatment vehicle, is readily adaptable to presently available advanced material production equipment. It is an inexpensive, easily handled, non-hazardous, and non-toxic chemical and therefore, appears to be ideally suited for the heat treatment.

Heat treating methods have been developed to reduce shrinkage of polypropylene laminates to as low as 2% (for a hot spot temperature of 100° C). The processes make fabrication of large spheres for satellite application possible.

Rigidity tests of cylinders show that heat treatment of the material does not affect the buckling strength of the laminate.

One highly important by-product of this study program has been the demonstration of the commercial feasibility of chemically milling 90% patterns in aluminum foil as thick as 2 mils. Moreover, success was obtained on a laboratory or experimental scale in the chemical milling of 4 mil aluminum laminates in a 90% pattern. Precise milling of laminates using printed patterns has been demonstrated to be possible for lines as narrow as .004 inches and for a foil thickness of .002 inches. This work was carried out to advance and supplement the work done for Advanced Materials.

II. HEAT TREATMENT OF AS-RECEIVED MATERIAL

A. Objective

The purpose of this portion of the test program was to establish the parameters which provide a minimum shrinkage of as-received polypropylene upon re-exposure to heat energy.

B. Test Description

The method used for heat treatment of all as-received material was identical. A deep-fat fryer was filled with glycerine and the thermostatic control was set for the desired heat treatment temperature. The temperature of the bath was allowed to stabilize at the desired temperature and was checked periodically by means of a laboratory thermometer to be certain that the temperature at which heat treatment was being conducted was as desired. The value of the temperature of the glycerine bath was at all times within plus or minus one degree Centigrade of the desired temperature.

Material was cut from a roll and placed in 7 inch diameter embroidery hoops. These hoops consist of two concentric aluminum rings lined with a thin strip of cork. When the hoops are pressed together, the cork is compressed between the two hoops. This provides a clamping force to hold material firmly in the hoops. The material was laid over the inner hoop and the outer hoop was pressed on to the inner hoop, and in the process the material was drawn taut within the hoop system. Where wrinkles in the material appeared, the material was tightened so that the polypropylene was stretched and had the appearance of a taut membrane.

After the material was mounted in the hoops, it was then marked so that measurements could be made of the shrinkage. Moreover, observation also could be made of distortion of the

material during heat treatment caused by uneven stretching of the material in the hoops. A stiff template, 3 inches square, was laid on the hooped material and its outline traced by means of a marking pen. The template was removed and locating marks were placed on the material so that measurements would be made in identical places after each test. The sample was identified by code and the machine direction was noted by means of an inked arrow. A total of ten samples were exposed and the results averaged for each shrinkage data point plotted on shrinkage test graphs.

At the outset of the heat treatment experiments, it was noted that the polypropylene would shrink while the hoops would expand. The result of these opposing forces caused the polypropylene to pull loose from the hoops or the hoops to pop apart during heat treatment. To prevent the slippage of material or popping of the hoops, special clamps were attached to the hoops and then the hooped material was lowered into the glycerine heat treatment bath for 15 seconds.

Upon removal from the glycerine bath, the samples were dipped in a room temperature water bath to remove excess glycerine and to cool the hoops. The three inch square pattern which had been marked on the polypropylene was then observed for distortion. In those cases where distortion was noted, the samples were assumed to be unevenly stressed in the hoop.

These samples were discarded and a new sample was mounted in a hoop and heat treated so that in all cases ten samples were obtained. The samples were also examined for evidence of material slippage from within the hoops. If slippage was noted, these samples also were discarded and replaced by fresh material. (Fig. 1 shows the test setup and also an example of material slippage from a hoop following heat treatment.)

After a sample had been heat treated at a given temperature and for a given duration and had been rinsed in the water bath, the samples were carefully removed from the hoops. Initially, 0.3 mil polypropylene (manufacturer - Moplefane) was mounted in hoops and subjected to heat treatment. This cast polypropylene was too thin to withstand the forces imposed by thermal expansion and contraction. As a result, whenever this film was subjected to heat treatment, it would snap within the hoop as soon as it was dipped into the heat treatment solution. Because this material was so difficult to handle in its as-received condition, further tests were cancelled to avoid delaying program completion.

When the samples had been removed from the hoops, they were carefully laid on cotton cloths and wiped clean. Then, the samples were dipped in a solution of water and isopropanol, and the samples were again wiped dry with cotton cloths. At this point, measurements of sample shrinkage were made in the

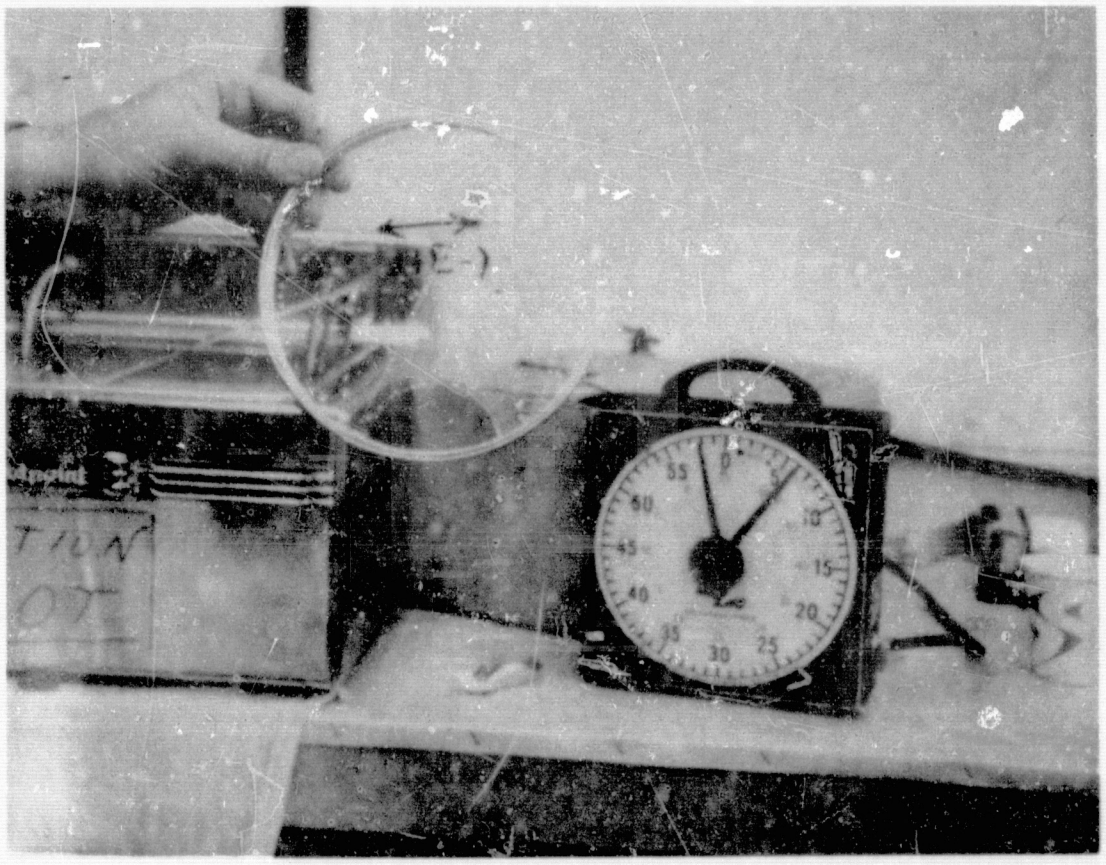


Figure 1

Test Setup

machine and transverse directions. These measurements were recorded to be used later as the base lengths from which shrinkage was determined. A steel rule which had 1/64 inch divisions was used to make measurements. To minimize the error in measurement due to wrinkled samples, the heat treated material was held in both hands and placed over the measuring rule which was lying on the table. Sufficient force was applied to the material to assure that there were no material undulations and that the material was not being stretched significantly. In the case of 0.5 mil polypropylene, calculations showed that it would be necessary to apply about 3/4 pounds of force per inch of material width to stretch the material outside of measuring accuracy. No such force was necessary to smooth out the material to make measurements (See Fig. 2 for a photograph of the measuring process.)

Prelminimary experiments indicated that identical results were obtained for the percent of shrinkage when shrinkage tests were performed in either of two ways: 1.) for each shrinkage test temperature a new sample was exposed, or 2.) for each shrinkage test temperature, the same sample was exposed. Of course when either a new heat treatment temperature or heat treatment duration was used, new samples had to be prepared. Since the second test method required the least time, it was used for gathering shrinkage data.

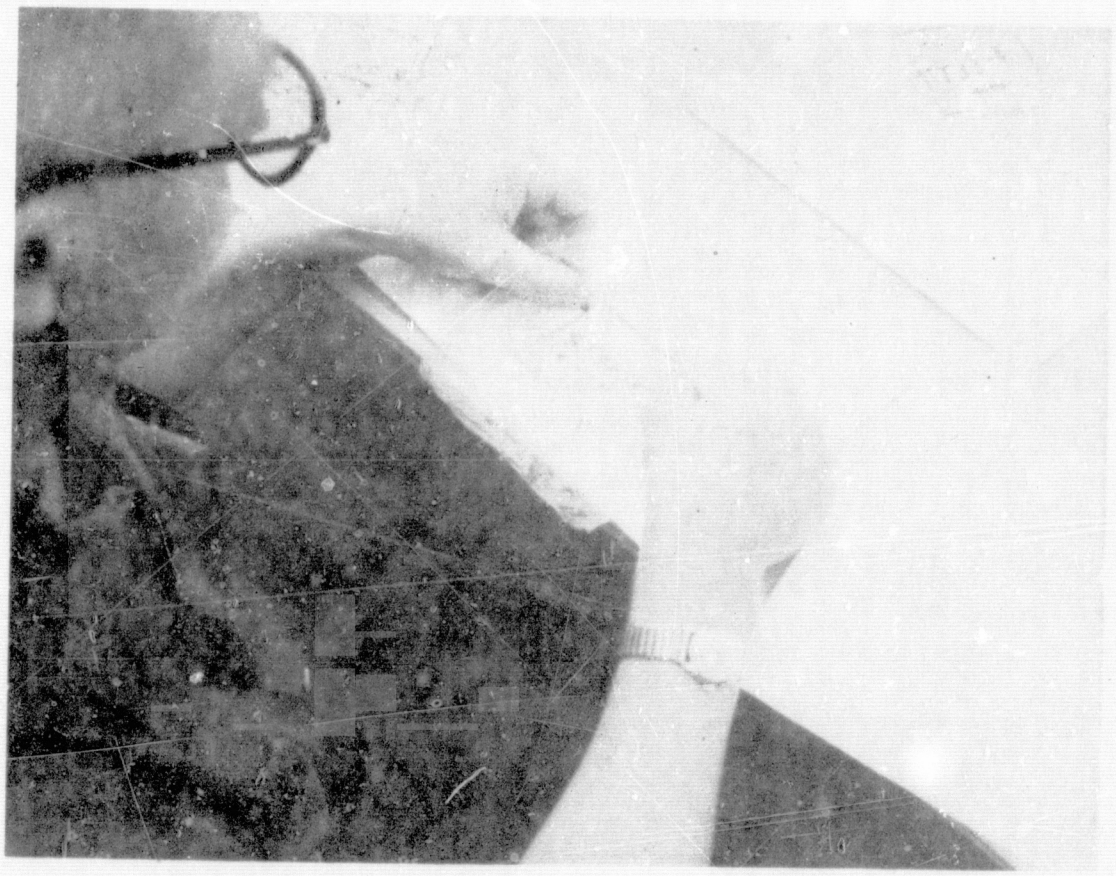


Figure 2

Measuring Process

C. Test Apparatus

1. Deep fat fryer with automatic thermostatic control of liquid temperature.
2. Standard photographic timing clock.
3. A set of 7 inch embroidery hoops.
4. Glycerine.
5. Thermometer graduated in degrees Centigrade by one degree steps.
6. Marking template (3 inches x 3 inches).
7. Steel measuring rule with 1/64 inch increments.
8. Marking pen.
9. Pail of water.
10. Cotton cloths.
11. Pail of water - isopropanol mixture.
12. Spring-loaded clothes pins.
13. Sharp knife, (Exacto).
14. Blank data sheets.

D. Comparison of Brands of Polypropylene

Extensively tested materials included the following:

1. Hercules Powder Co. biaxially oriented polypropylene 0.5 mil thick.
2. Union Carbide Plastics Company biaxially oriented polypropylene 0.6 mil thick.

At the outset, the vendors whose material was expected to be tested were (1) Hercules Powder Co., (2) Kordite, (3) Monti-

catini, (4) Olin-Mathieson, and (5) Union Carbide Plastics Co. Montecatini material was 0.3 mil cast polypropylene which was found difficult to handle during the shrinkage test process and was discounted for study for that reason. The handling difficulties resulted from the material's poor basic strength characteristic. Olin-Mathieson polypropylene melted and fused together at temperatures as low as 150 C and also exhibited characteristics opposite to that of other films during the heat treatment. That is, rather than tightening in the hoops during heat treating, the Olin-Mathieson film (a cast film) expanded. Because of these two conditions this film was rejected for extensive testing.

Data obtained after shrinkage tests were begun indicated that the two most promising sources of polypropylene were Union Carbide Plastics Company and Hercules Powder Co. This conclusion was based on comparisons of tensile strengths. Table I compares the tensile strengths of various brands of polypropylene. Of the films listed, it may be seen that Union Carbide Plastics Co. and Hercules Powder Co. polypropylene have comparable tensile strengths at room temperature and at -70 F. Moreover, these tensile strengths are higher than those obtained for other brands. For this reason, Union Carbide material was compared only with Hercules Powder Co. material, since high tensile strength is important to the application of this film.

Figures 3, 4, 5 and 6 show the percentage of shrinkage

TABLE I

POLYPROPYLENE TENSILE STRENGTHS

MANUFACTURER	TYPE OF FILM	TENSILE STRENGTH			
		Room Temperature		-70 F Temperature	
		MD (psi)	TD (psi)	MD (psi)	TD (psi)
Cryovac Corp.	Oriented	19,320	32,800	24,680	34,080
Olin Mathieson	Cast	9,066	3,782	12,480	9,653
Union Carbide	Oriented	23,440	25,840	31,400	33,400
Montecani	Oriented	19,100	27,860	22,140	42,540
Moplefane	Oriented	16,920	39,280	19,160	39,800
Hercules Powder Co.	Oriented	33,560	29,500	37,800	37,600

NOTE: Average of five Instron tests.

THERMAL SHRINKAGE VS. SHRINK TEST TEMPERATURE
FOR VARIOUS HEAT TREATMENT TEMPERATURES

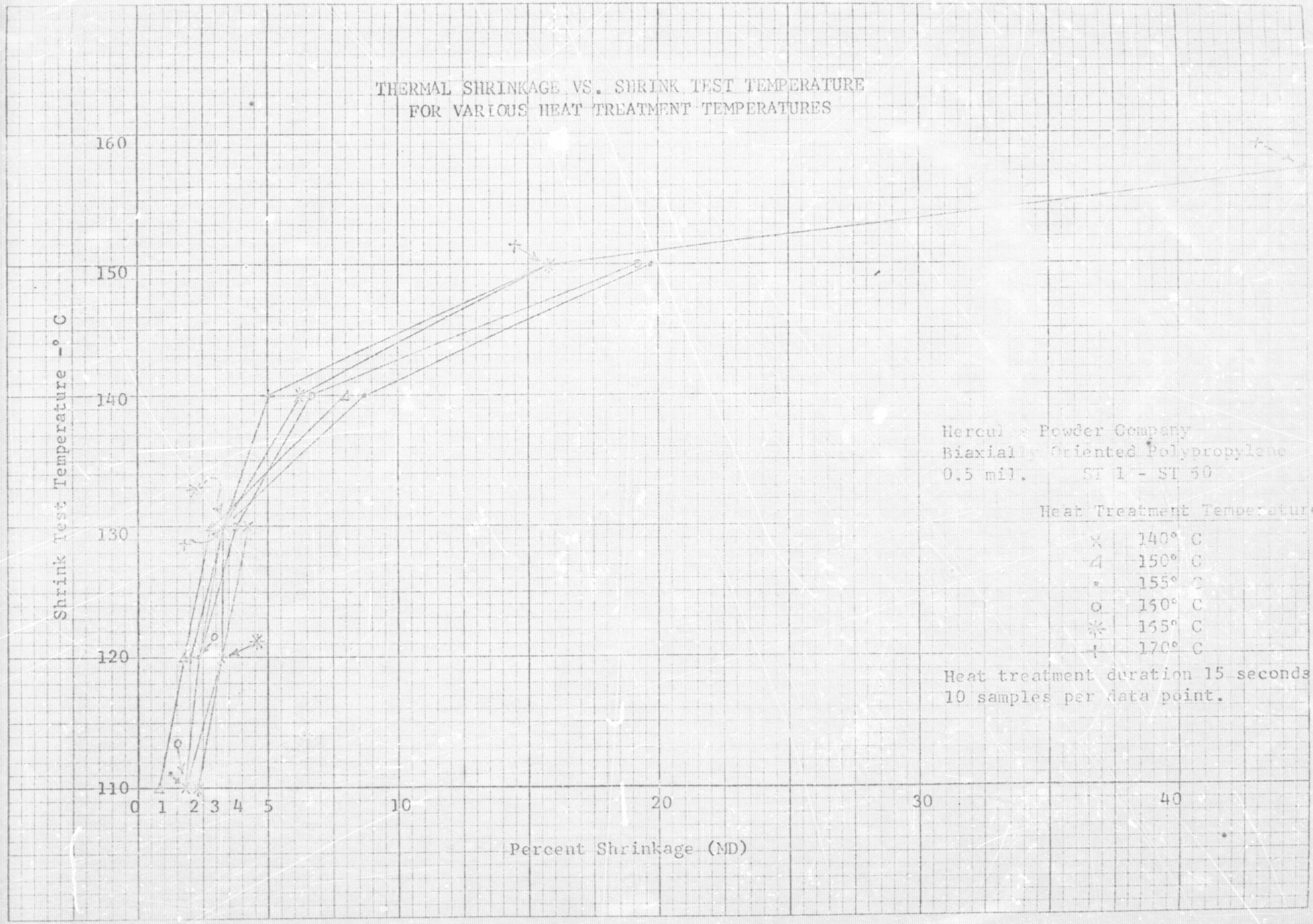


FIGURE 3

THERMAL SHRINKAGE VS. SHRINK TEST TEMPERATURE
FOR VARIOUS HEAT TREATMENT TEMPERATURES

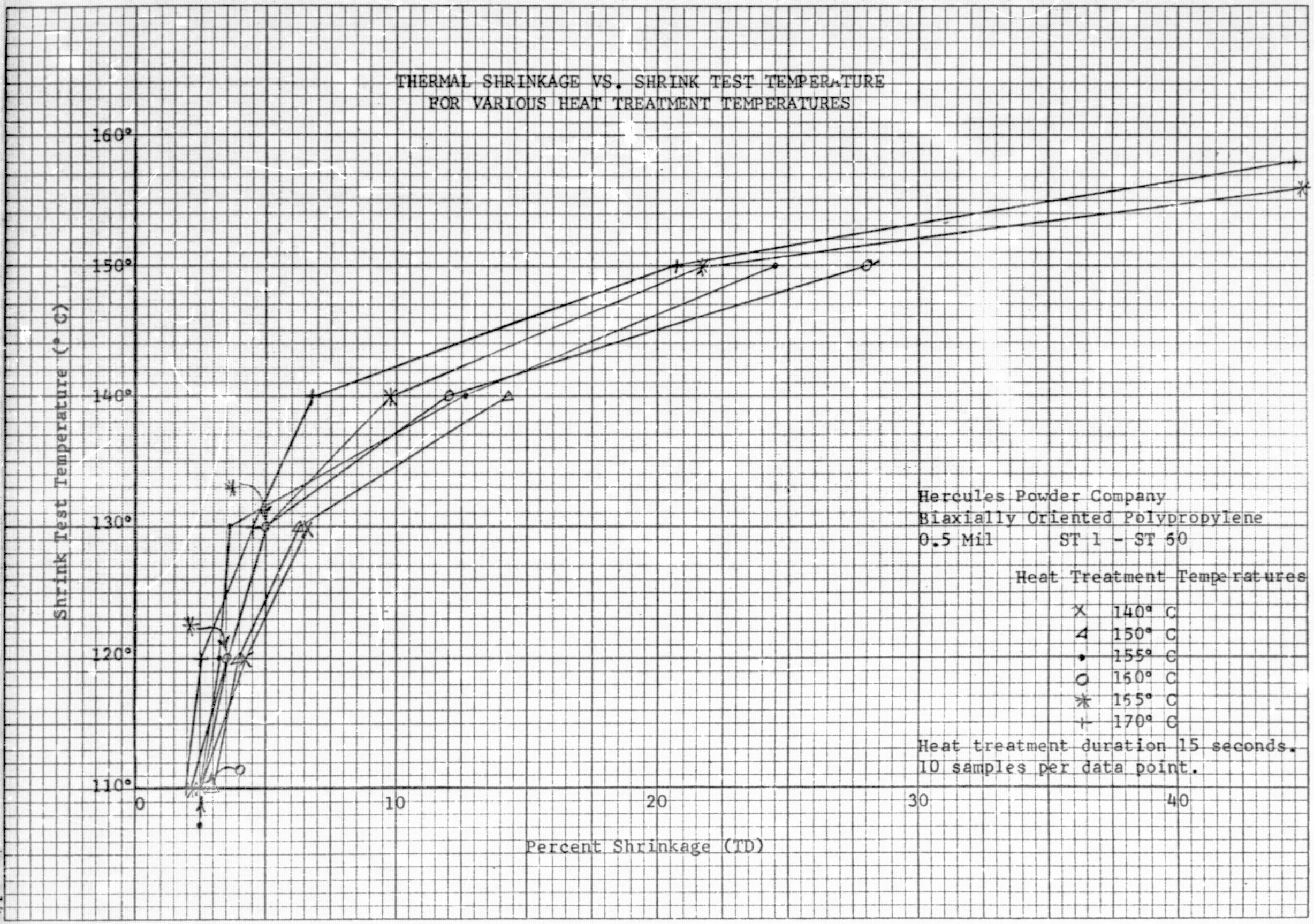


FIGURE 4

THERMAL SHRINKAGE VS. SHRINK TEST TEMPERATURE
FOR VARIOUS HEAT TREATMENT TEMPERATURES

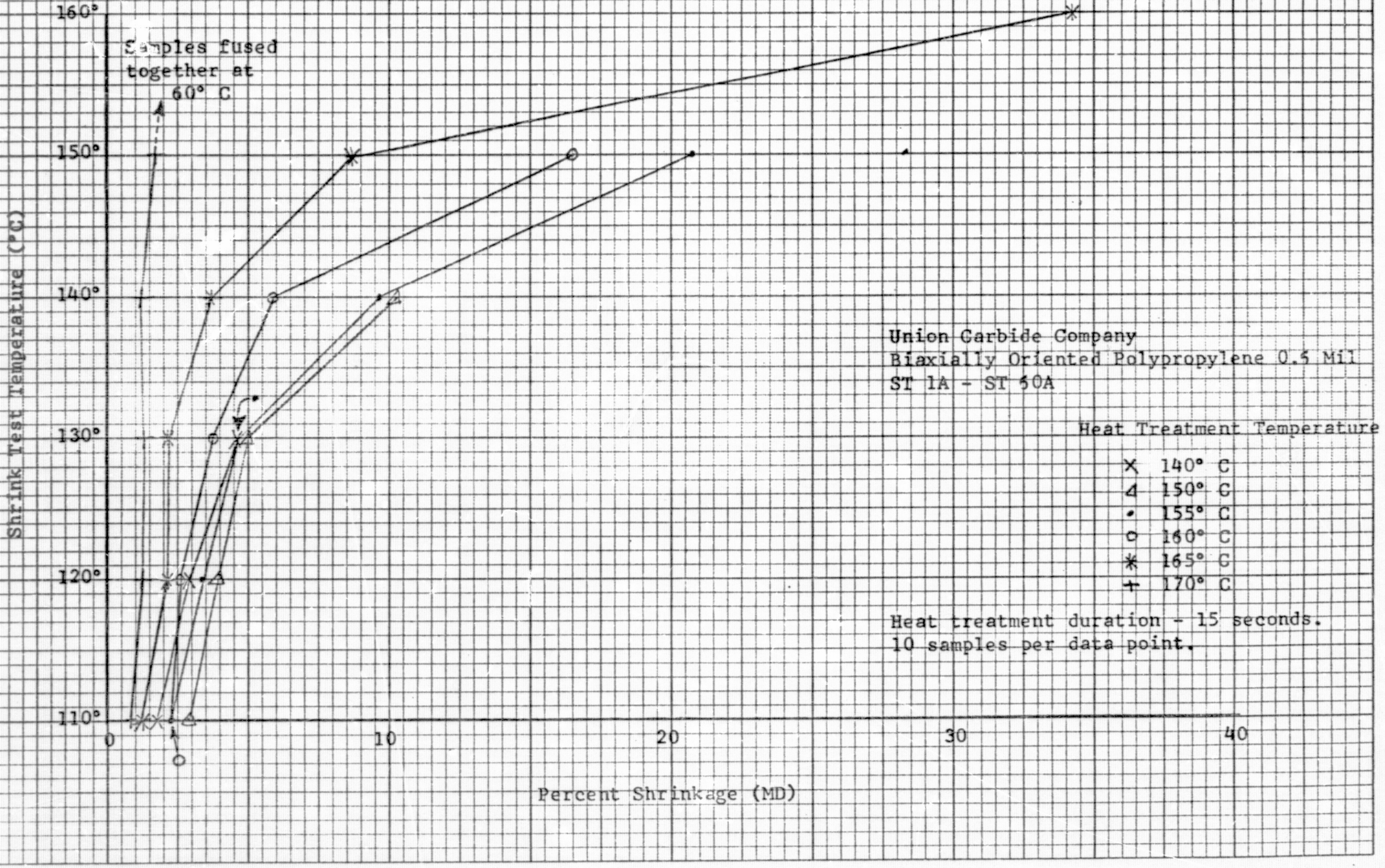


FIGURE 5

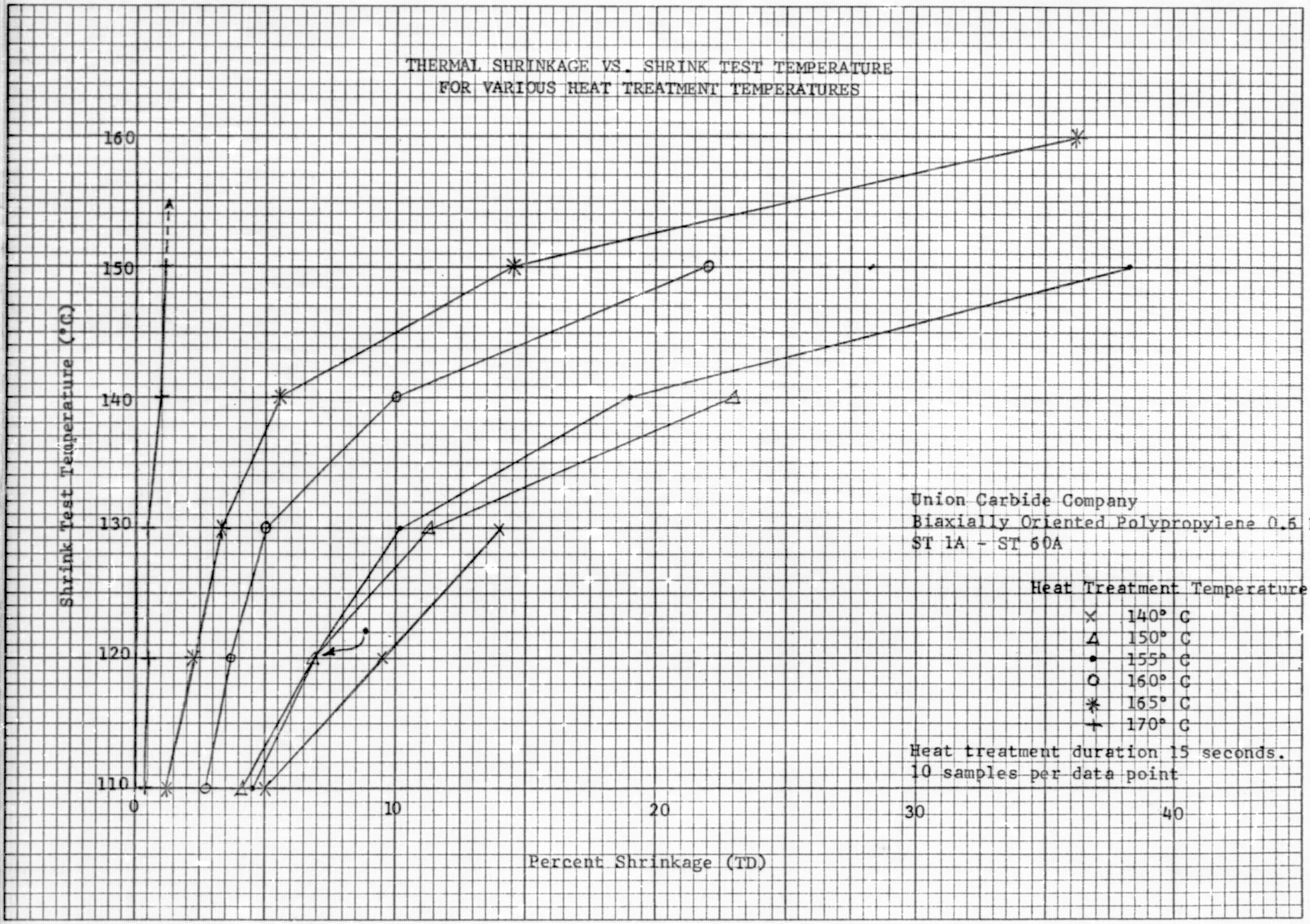

THERMAL SHRINKAGE VS. SHRINK TEST TEMPERATURE
FOR VARIOUS HEAT TREATMENT TEMPERATURES
Shrink Test Temperature (°C)
160
150
140
130
120
110
0
10
20
30
40
Percent Shrinkage (TD)
Union Carbide Company
Biaxially Oriented Polypropylene 0.6 Mil
ST 1A - ST 60A
Heat Treatment Temperature
140° C
150° C
155° C
160° C
165° C
170° C
Heat treatment duration 15 seconds.
10 samples per data point

FIGURE 6

versus temperature for Hercules Powder and Union Carbide material. In addition, both films have data presented for shrinkage in the machine and transverse directions for various heat treatment temperatures. The Union Carbide film showed a steady decrease in shrinkage percentage for increases in the heat treatment temperature, whereas the Hercules Powder material did not show as large a reduction in shrinkage for higher heat treatment temperatures.

Above 170 C the polypropylene melted. Figure 7 shows what happened to the films when they melted in hoops. This Figure shows a sample of Olin-Mathieson film which melted at less than 170 C; specifically, it melted at 155 C.

E. Conclusions

1. Union Carbide Plastics Co. biaxially oriented polypropylene has a lower shrinkage for a given heat treatment temperature than does Hercules Powder Co. polypropylene.
2. Although Figures 3, 4, 5 and 6 indicate that minimum shrinkage occurs for heat treatment temperatures of 170 C, tests of the tensile strength of the material show that tensile strength drops off rapidly at 170 C. Therefore, although 170 C yields a minimum of shrinkage, some lower heat treatment temperature is dictated by the loss of material strength as caused by heat treatment temperature. This maximum heat treatment temperature is discussed later in this report.



Figure 7

Melting Polypropylene

III. TEST OF HEAT TREATMENT DURATION EFFECTS

A. Objective

Purpose of this section of the test program was to determine the appropriate duration of heat treatment to yield minimum shrinkage of as-received material when re-exposed to heat energy.

B. Test Description

The tests for duration of heat treatment were conducted in exactly the same manner as other shrinkage tests with the exception that duration was changed. In tests of the as-received material (described in Section II) the polypropylene was heat treated for 15 seconds in the interest of saving time in testing. This meant that the hooped material was dipped in the hot glycerine bath for a period of 15 seconds and then withdrawn. In this section of the test program, the hooped material was dipped in the hot glycerine for periods of 15 seconds, 1 minute, 3 minutes, 5 minutes and 10 minutes, and at the completion of the appropriate heat treatment duration, the sample was withdrawn, cleaned and then later re-exposed to heating. Following the second heating in the oil bath, the materials were cleaned and measurements were made of the shrinkage.

All heat treating was performed on Union Carbide biaxially oriented polypropylene (0.6 mil) at 160 C. Union Carbide was

chosen for further testing based on the results obtained in the 15 second shrinkage tests of Union Carbide and Hercules Powder films. The 160 C was chosen because of data obtained in another section of this program which suggested this temperature. Each recorded data point represents an average of the shrinkage of ten samples which have been subjected to shrinkage tests.

Apparatus used to conduct this section of the program were identical to those described in Section II.

C. Discussion

The results of the tests are shown graphically on Figure 8. In general the data are consistent and show a trend of decreasing percent of shrinkage with increasing heat treatment duration. For durations above three minutes, and in the higher shrinkage test temperatures of 140 and 150 C, there appears to be an increase followed by a decrease of percent shrinkage. These reversals of a trend may be real. On the other hand, they may be due to errors in the test method. However, it is not anticipated that temperatures of the order of 140 or 150 C will be experienced in space. Therefore, those anomalous bulges in the curves are of academic interest only and do not affect the conclusions of this report.

D. Conclusions

1. A heat treatment duration of one minute is the optimum

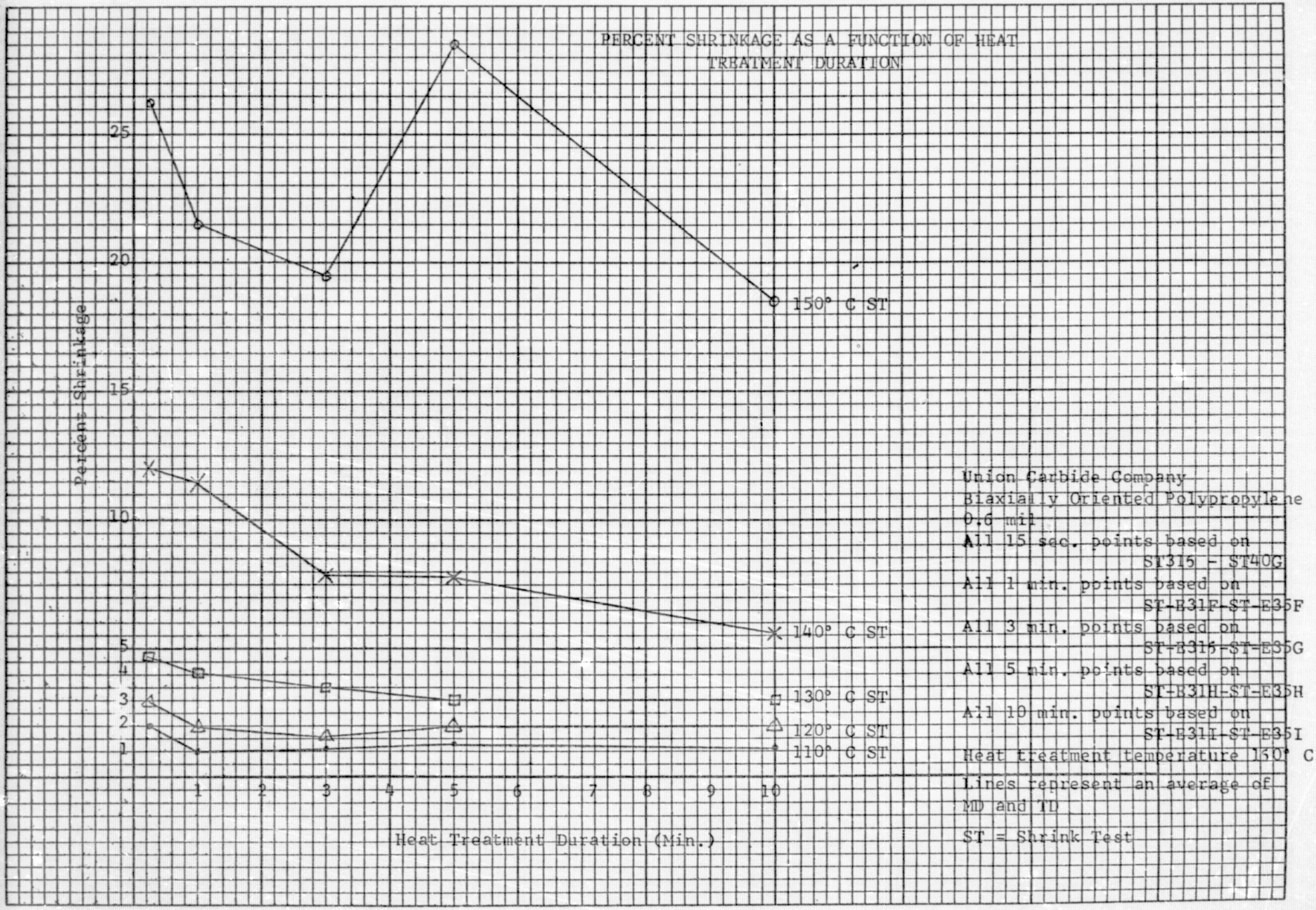

PERCENT SHRINKAGE AS A FUNCTION OF HEAT TREATMENT DURATION
Percent Shrinkage
Heat Treatment Duration (Min.)
150° C ST
140° C ST
130° C ST
120° C ST
110° C ST
Union Carbide Company
Biaxially Oriented Polypropylene
0.6 mil
All 15 sec. points based on ST315 - ST40G
All 1 min. points based on ST-E31F-ST-E35F
All 3 min. points based on ST-E31G-ST-E35G
All 5 min. points based on ST-E31H-ST-E35H
All 10 min. points based on ST-E31I-ST-E35I
Heat treatment temperature 130° C
Lines represent an average of MD and TD
ST = Shrink Test

FIGURE 8

exposure time for minimum film shrinkage.

IV. OTHER HEAT TREATING TECHNIQUES

A. Objective

Purpose of this section of the test program was to determine the most economical mode and the fastest mode of heat treating a laminate material.

B. Test Descriptions and Results

One mode of heat treatment which was considered was that of placing a roll of laminate in an oven and soaking it at a specified temperature for a long period of time. This has several advantages, it would be easy to do, inexpensive and rapid. However, certain difficulties arise in the oven heat treating of polypropylene.

Thirty-six samples of X-32 material were heat treated at 165 C in a laboratory oven, "Precision Scientific, Model 524". Temperatures within the oven were checked against readings taken through a vent in the oven with a glass centigrade mercury-type thermometer. The samples were clamped to an oven rack with clothes pins and suspended in the oven (See Figure 9). This was repeated until all samples were heat treated. Heat treatment duration was one hour to correspond to the need for a long period of oven soaking in a roll of laminate. When the pieces were removed from the oven, an oily sticky substance was noted on the samples. The oven was then cleaned thoroughly of

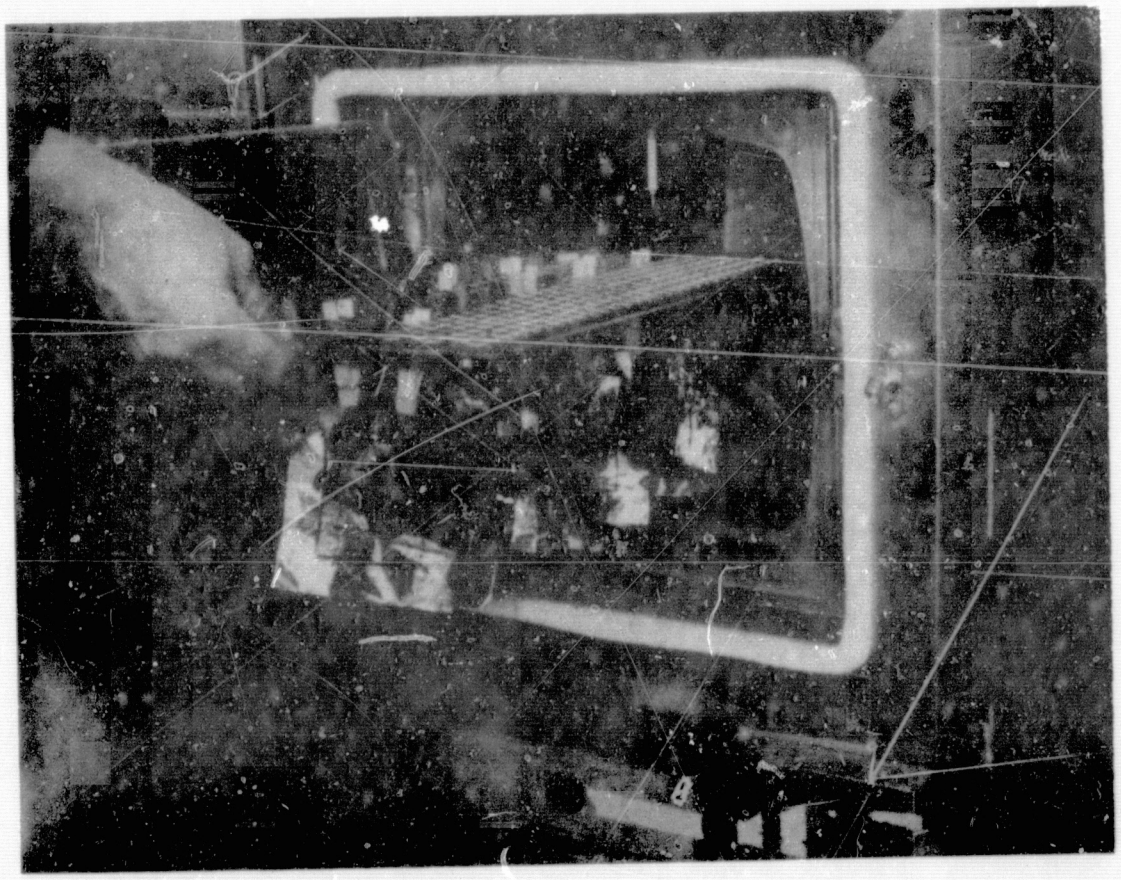


Figure 9

Laboratory Oven Testing

all resins that might have been left in the oven from other uses and then six new samples were placed in the oven for one hour. At the completion of this test, a great amount of smoke and vapor escaped from the oven when it was opened and this smoke had a pungent odor. At this point, aluminum was removed from a sample and the plastic was tested with the infrared spectrometer to ascertain whether the material was truly polypropylene. Positive identification was made of Union Carbide polypropylene.

The test was continued with the assumption that oxidation was causing the smoke odor. New samples were placed in the laboratory oven with a vacuum of 10 inches of mercury within the oven. The oven was set for a temperature of 165 C. After one hour, the samples were removed. Some smoke was noted when the oven was opened. Two samples were placed in a caustic soda solution and the aluminum was removed. The polypropylene exposed after chemical milling was in fragments. Figure 10 shows the condition of the polypropylene after oven heat treatment and removal of the aluminum. On the basis of this test oxidation was determined to cause the film deterioration.

Because rolls of laminate would require long periods of soaking to allow heat treatment of the inner layers of the laminate, the oven method of laminate heat treatment is not advisable. The long period of heat treatment would certainly

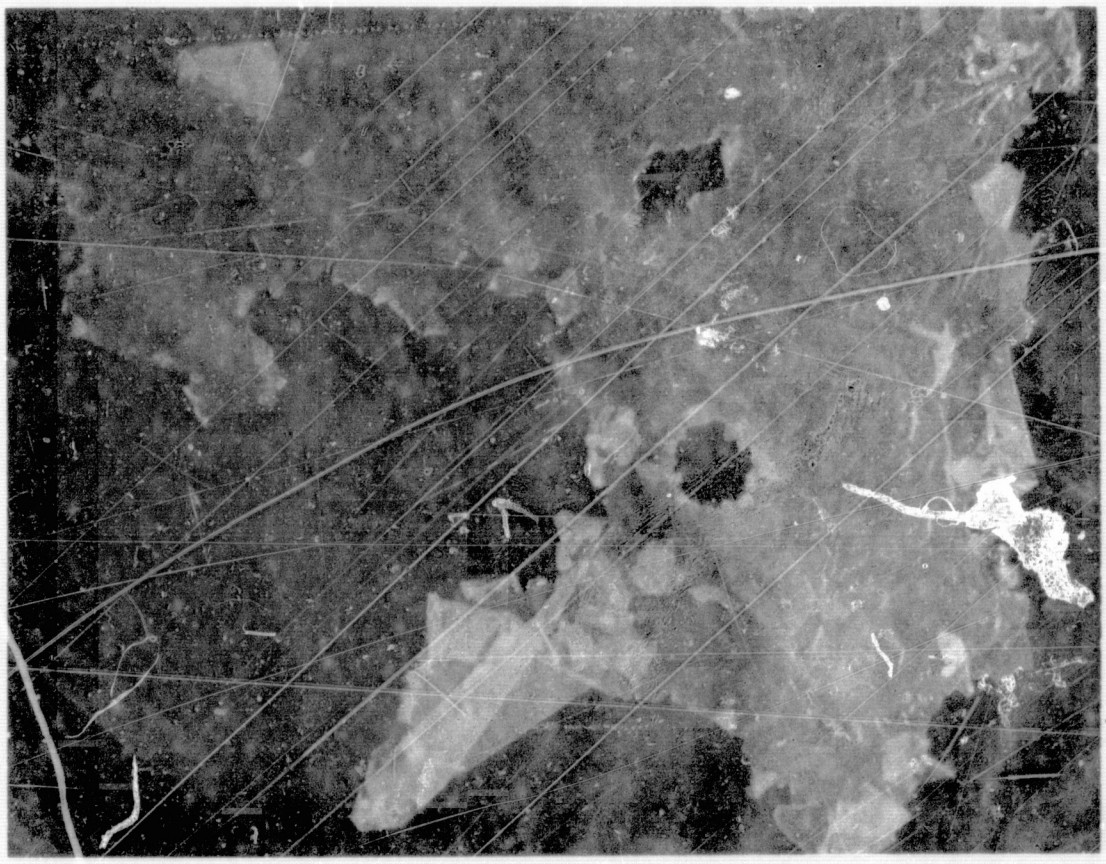


Figure 10

Condition of Polypropylene After Oven Heat Treatment

cause the outer layers of the laminate roll to become oxidized and deteriorate while the inner layers might not deteriorate.

In yet another attempt to heat treat the laminate, samples of X-32 (laminated on both sides) were passed through a laminator under pressure and high temperature. The results of this test were not satisfactory for heat treatment since material that passed under the center of the laminator roll was heat treated at a higher t mperature than that which passed under the ends of the laminator roll. This reflects the condition that the temperature of the laminator roll is highest at its center and this temperature drops off as the end of the roll is approached. As a consequence of the temperature variation (which does not affect laminations) across the width of the laminate, inflatables fabricated from this material would exhibit less shrinkage at the center of the gores than the edges. This non-uniform shrinkage would introduce a distorting parameter to an Echo-type sphere exposed to solar radiation. Because of this non-uniform heat treatment caused by the physical limitations of the laminator, this form of heat treatment was not pursued further.

Glass beads, such as those used by an optometrist for heating eyeglass temples, were considered. However, holding a large vat of tiny glass beads at a constant temperature during passage of a laminate would be difficult since the beads cannot be stirred like a liquid.

Liquids or solutions other than glycerine were considered as possible heat treatment baths. By using the Handbook of Physics and Chemistry, the boiling point and flash point of various compounds and mixtures were determined. None of the chemicals examined incorporated all of the attractive features of glycerine. In the great majority of cases the flash point of the liquid was too low (use hazard) as was the boiling point (loss of utility). In most cases where the boiling point was high, the compound was either expensive, scarce or was toxic to some degree.

Glycerine with its boiling point of 290 C and its flash point of 225 C is an ideal heat treatment bath. There is no danger of toxicity, or irritation to the human skin. Glycerine is inexpensive, easy to handle, and easy to remove from a laminate material. The solution to use for removing glycerine is a mixture of water and isopropanol. Moreover, present laminate handling equipment can be readily adapted to provide a tank of glycerine through which the laminate must pass for heat treatment and in addition, a wash tank of water-isopropanol is feasible as a part of the process of heat treating polypropylene laminates.

C. Conclusions

1. Oxidation occurs when polypropylene laminates are heat treated in a hot air oven, therefore, oven heat treating is not advisable.

2. Axial variations in the temperature of laminator roll leads to non-uniform heat treatment of material. Therefore, laminator heat treating is not advisable.

3. Glycerine is the safest, most economical and easiest fluid to use for heat treatment.

V. SHRINKAGE TESTS OF LAMINATED POLYPROPYLENE

A. Objective

The purpose of this portion of the test program was to determine the effect of heat treatment on laminates of polypropylene and aluminum. Heat treatment was conducted on one-sided aluminum laminates of polypropylene.

B. Test Description

Although the as-received shrinkage test indicated that 170 C was an ideal shrinkage test temperature, it was necessary to consider the effect of high heat treatment temperatures on polypropylene-aluminum laminates to ascertain whether any delamination occurred due to the heat treatment procedure. Moreover, it was necessary to determine whether the glycerine bath had any effect on the film during heat treatment and whether it was necessary to laminate both sides of the polypropylene for plastic protection during heat treatment.

Materials tested in this section of the test program were treated in glycerine in the same manner as the as-received material, except that the embroidery hoops were not used to

provide resistance to shrinking. The bond between the aluminum and the polypropylene served this function in a uniform manner. After heat treatment, the samples were cleaned and re-exposed to elevated temperatures in the glycerine. The 3 inch by 3 inch square template measuring figure was measured following shrinkage testing and results recorded.

Initially, X-32 material laminated on both sides was heat treated in glycerine at 165 C. The material was then re-exposed to elevated temperatures in the glycerine bath. No measurable shrinkage was noted after completion of the tests.

Following this series of tests, another group of 10 identical samples was heat treated in glycerine at 165 C. (Polypropylene used in X-32 material is Union Carbide Plastic Company oriented polypropylene). After heat treatment, the aluminum was removed from one side of the X-32 material by use of a caustic soda chemical milling process. When these samples were then subjected to shrinkage test temperature of 140 C some delamination was noted on some of the samples and at 150 C this delamination was pronounced. Figure 11 is a photograph which shows the delamination experienced during shrinkage testing. The photograph shows a sample with delaminations circled. For samples exposed to 160 C no delamination occurred on re-exposure to heat.

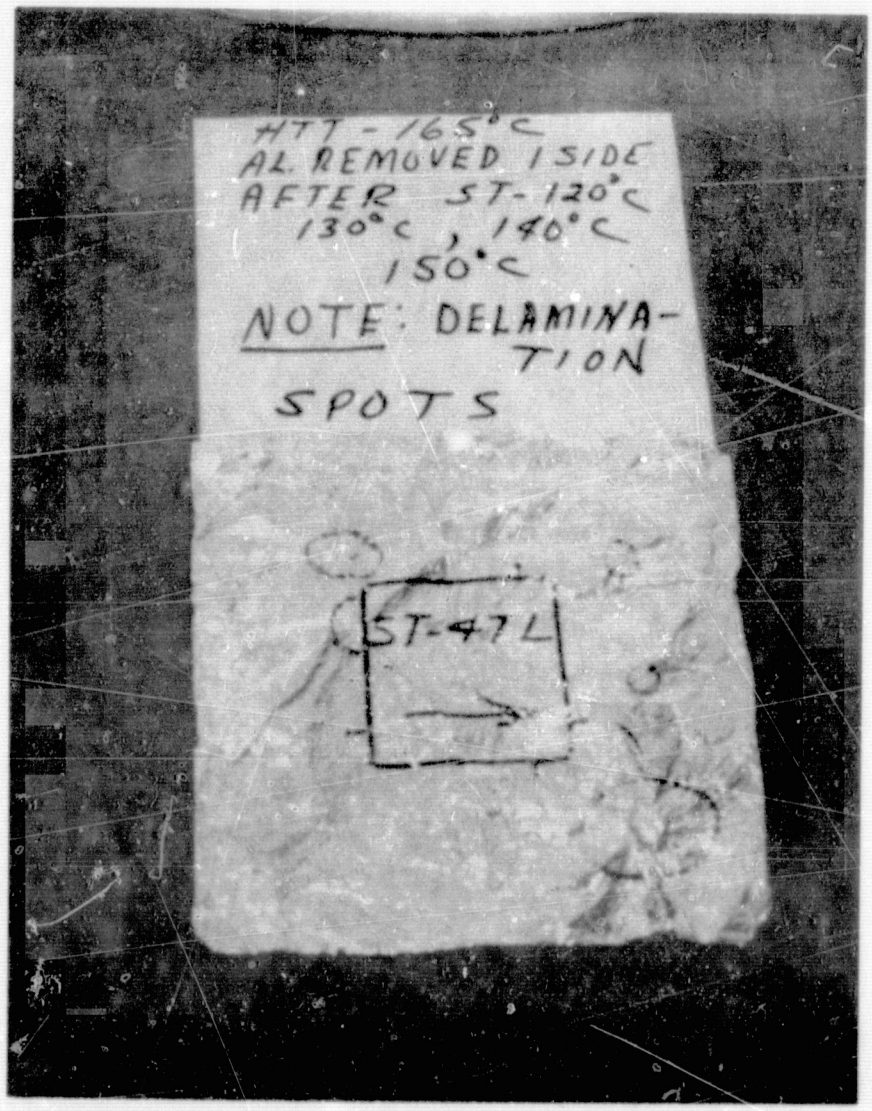


Figure 11

Delamination During Shrinkage

Figures 12, 13, and 14 present summaries of the amount of shrinkage for X-32 material laminated on two sides, X-32 material laminated one side, and X-32F material with various combinations of aluminum siding, respectively. The percentages of shrinkage shown in Figures 12 and 13 are very small and may exist largely due to errors in measurement with the 1/64 inch scale, although each data point represents the average shrinkage of 10 different samples. However, on the other hand, these two figures indicate that the aluminum provides support for the polypropylene so that the polypropylene is uniformly stressed during heat treatment.

Another portion of the investigation of the effect of heat treatment on aluminum-polypropylene laminates was to determine whether curling occurred when the laminate was subjected to heat treatment. X-32 material laminated on one side was used to determine the amount of curling caused by heat treatment in glycerine. Samples of X-32 material were cut and during the process of cutting, curling occurred to varying degrees with different samples. The samples cut were 7 inches square and the amount of curl they exhibited after cutting and before heat treatment ranged from slight curling of the cut edge to a nearly completely rolled sample. Subjecting these samples to heat treatment and examination of the samples following heat treatment demonstrated that curl was not induced by the heat treatment. All curl in the samples

PERCENT SHRINKAGE OF X32F MATERIAL
LAMINATED ON BOTH SIDES

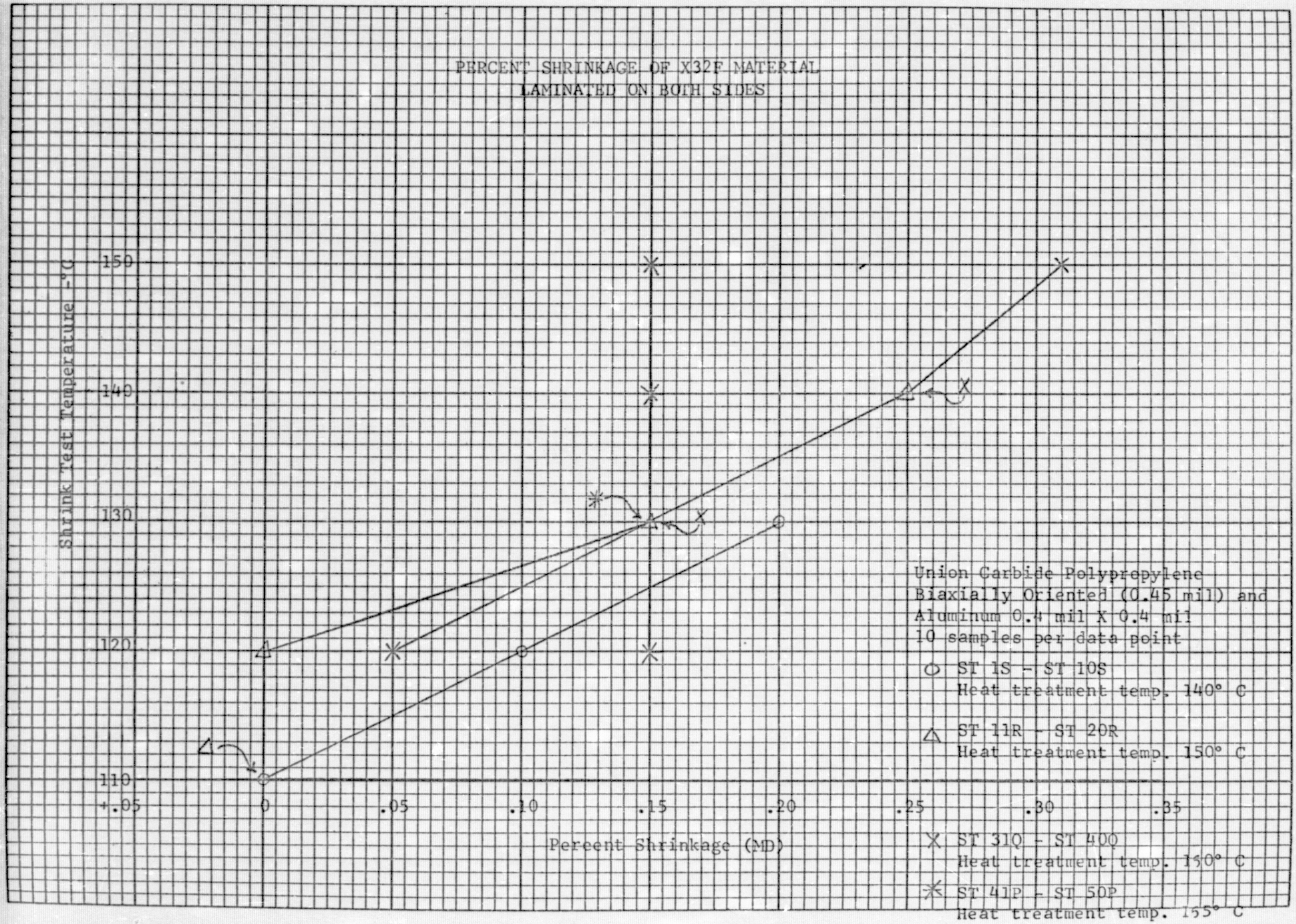


FIGURE 12

PERCENT SHRINKAGE OF X32F MATERIAL
WITH LAMINATE ETCHED FROM ONE SIDE

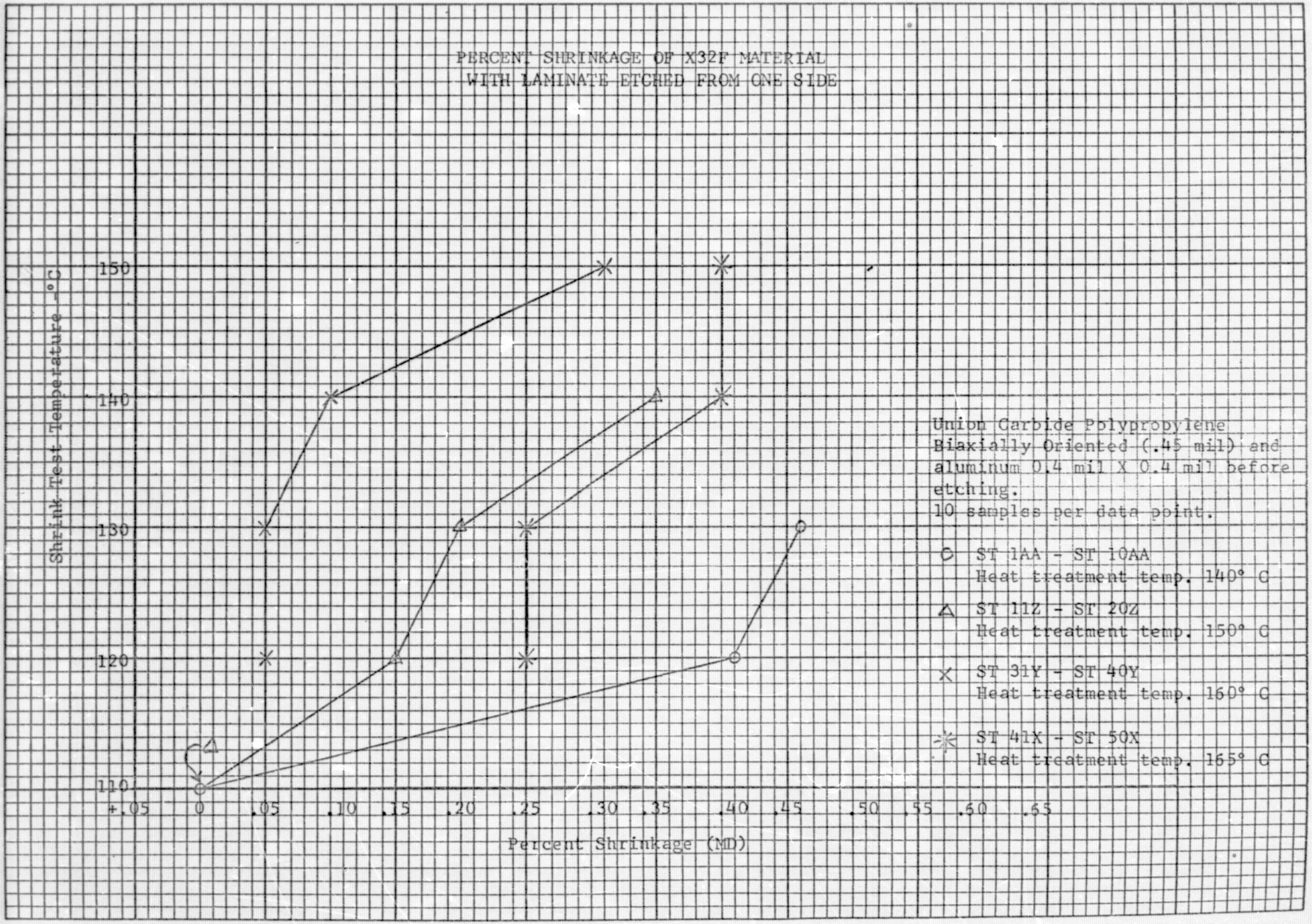


FIGURE 13

PERCENT SHRINKAGE OF X32F MATERIAL
FOR VARIOUS COMBINATIONS OF ALUMINUM ETCHING

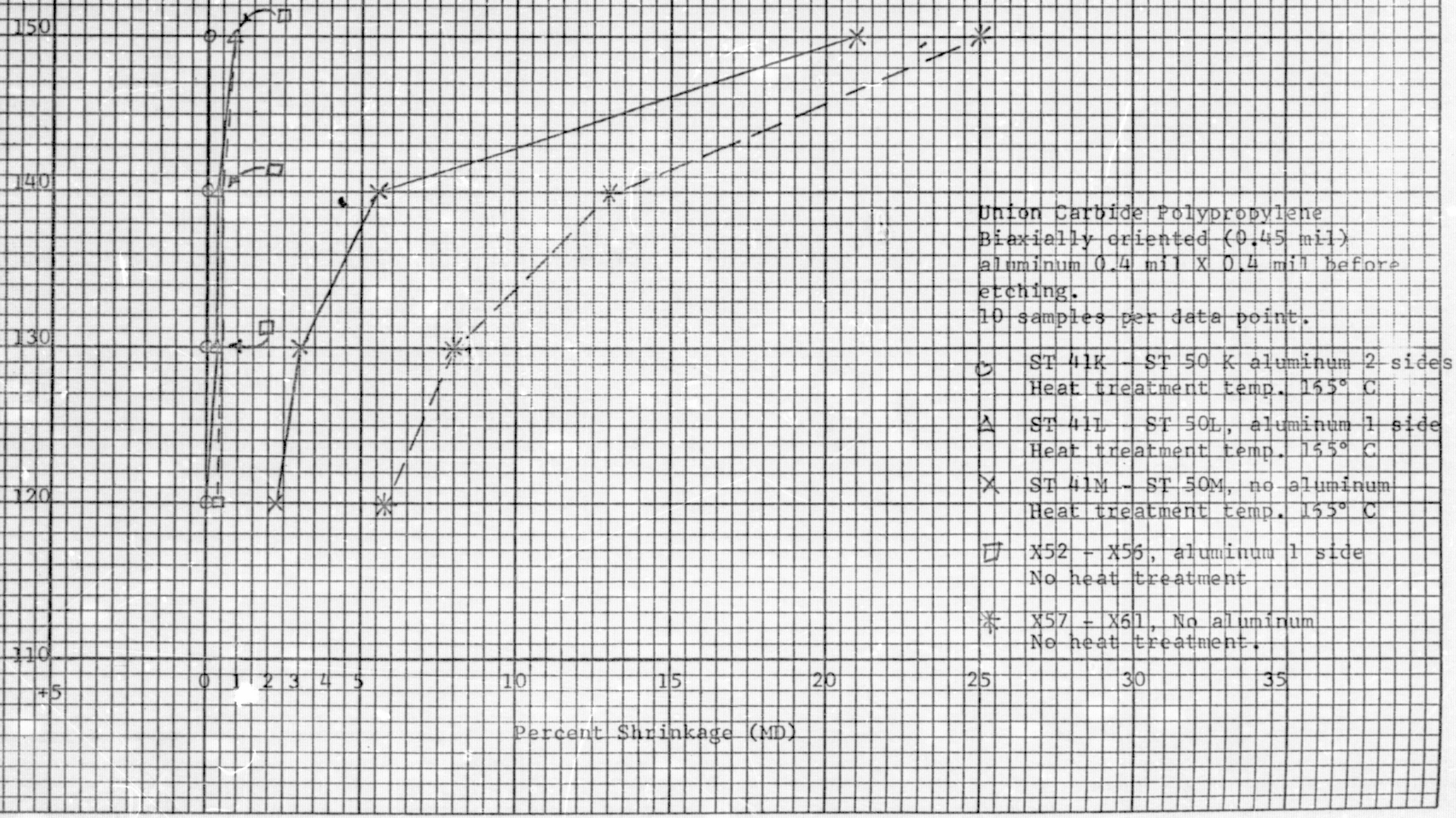


FIGURE 14

was a result of the cutting process and all curling took place along cut edges.

Further tests of the effect of heat treatment on laminated materials (other than X-32 laminates) were conducted to determine the extent of delamination along cut edges of the material. At one point during the heat treatment of one-sided laminates, material was subjected to 160 C heat treating and serious delamination occurred along the edges of the material. Further investigation showed that along those edges which were cut by scissors or a knife, delamination originated at the intersection of the cuts and proceeded away from such an intersection. The extent of delamination was a function of the heat treatment temperature and the duration of heat treatment; the higher the heat treatment temperature, the greater the delamination for a given heat time. For a laminate of 0.6 mil polypropylene and 2.0 mil aluminum, delamination first occurred at 130 C. This delamination took the form of a curling of the polypropylene away from the aluminum. For a laminate of 0.6 mil polypropylene and 4-mil aluminum, delamination occurred in the same manner and to approximately the same degree; that is, delamination began at a corner or intersection of two cuts and was first noticed at a heat treatment temperature of 130 C with the most serious delamination occurring at 160 C.

However, no delamination was seen to occur along the

edges of the aluminum-Mylar laminate which represented the sides of a roll of material. Since the roll of polypropylene used in fabricating the laminate was narrower than the roll of aluminum to which it was laminated, a strip of bare aluminum resulted along the side of the laminate. This edge did not show delamination during heat treatment at any heat treatment temperature or duration. Moreover, laminates of aluminum-polypropylene-aluminum could be cut carelessly and the material exposed to 160 C heat treatment with no resulting delamination.

C. Conclusions

1. Non-milled two-sided and one-sided aluminum polypropylene laminates exhibit little shrinkage.
2. Heat treatment of laminates should be conducted at less than 165 C to avoid laminate delamination on re-exposure to heat energy.
3. A laminated aluminum layer provides uniform support for polypropylene during heat treatment.
4. Amount of curling of laminates is a function of cutting and not of heat treatment.
5. Two-sided laminates of aluminum-polypropylene do not delaminate at 160 C heat treatment temperature.

VI. SHRINK TESTING OF MILLED LAMINATES

A. Objective

The purpose of this section of the test program was to heat-treat and shrink-test chemically milled laminates (hexagonal pattern) fabricated from .6 mil biaxially oriented polypropylene and various aluminum foil thicknesses. Shrinkage variation as a function of percent of material milled, thickness of aluminum laminate and heat treatment temperature is presented.

B. Test Description

Heat treatment of chemically milled laminates was accomplished prior to resist printing and chemical milling. The laminate (originally a three layer laminate of 0.2 mil aluminum, 0.6 mil polypropylene and another aluminum layer) was dipped in the hot glycerine heat treatment bath for a period of 1 minute. The three-layer laminate was used to prevent glycerine from touching the polypropylene during heat treatment. Then, the heat treated material was thoroughly cleaned with a 25 per cent solution of water and isopropanol. Because of the presence of aluminum laminate, it was unnecessary to support the material in hoops as the aluminum provided the support for the polypropylene during heat treatment. Following heat treatment and cleaning, the laminates were printed with the hexagonal pattern so that chemical

milling could be accomplished. Chemical milling produced a one sided laminate of the percent opening printed.

At this point in this test program significant effort had to be expended to chemically mill the laminate. It was possible to purchase material which was chemically milled to G. T. Schjeldahl specifications. However, it was felt that the need to demonstrate commercial feasibility of the production of chemically milled laminates (90% milled) was of importance to further developments of advanced materials.

Therefore, a program was undertaken to learn the technique of chemically milling 90% patterns on a commercial scale for thick aluminum foils. The first problem to overcome was that of a choice of resist. The resist is the paint-like material which is printed in a hexagonal pattern on the aluminum so that during chemical milling the chemicals do not attack that portion of the aluminum underneath the printed hexagonal pattern. After chemical milling, the resist is removed by means of a solvent.

The first resist used was EY-214 Menasha Printing Resist. Ninety percent patterns were printed by means of a silk screen pattern on 2-mil and 4-mil aluminum laminates. The etchant used was a 25 percent solution of sodium hydroxide and the method of chemical milling was the dip process. The duration of chemical milling was about 40 minutes and at the end of that period of time the resist had deteriorated while the

chemical milling was incomplete. Following this, varying percentage solutions of sodium hydroxide were used with Menasha Printing Resist to attempt to determine the best approach to chemical milling heavy laminates with 90 percent mil patterns. The results were negative since the Menasha Printing Resist, in all cases, deteriorated prior to completion of chemical milling of the pattern desired.

Further tests of chemical milling of the laminate were conducted with a 90 percent pattern, dip process and various sodium hydroxide solutions. However, the resist used was Nasdar Screen Process Ink No. 211. Despite baking of the samples in an oven for 30 minutes at 95 C in an effort ot strengthen the resist, the chemical milling proved unsatisfactory. In some spots the solution would eat through and undercut the pattern and in other spots fine lines were obtained. No consistency was obtainable by this method.

The next step was to change the etchant to ferric chloride. A 90 percent pattern was printed on 2-mil aluminum that had not been heat treated and a dip process was used for the milling method. The results of these experiments showed that less time was required for chemical milling and were encouraging since the 90 percent pattern was being milled completely prior to destruction of the resist (Menasha Printing Resist). The best sample obtained was one in which a heavy coating of resist was applied and the resist was allowed to dry overnight prior

to milling.

The next major change made was to change the chemical milling process from a dip process to a spray etching process. Spray etching was accomplished in a small spray etcher with samples about 8 inches square. Because spray etching chemically mills the laminate much more rapidly than the dip process, it was necessary to monitor the etching process very closely. The spray etcher was tried on several samples with caustic soda but unsatisfactory chemical milling resulted. Ferric chloride was then tried as the etchant and considerably better results were obtained. The Naz-Dar Screen Process Ink No. 211 was used in the spray etching experiments and printed laminates were baked in a laboratory oven for 2 hours at 100 C. The baking of the 211 Naz-Dar Ink and the spray etcher in combination with the ferric chloride etchant provided the best samples of all at that point.

To improve the milling process further, the resist was changed again. The resist selected was Naz-Dar Screen Process Ink No. 205 which is a specially designed ink for use with silk screen patterns. The blue ink had one drawback -- when printing through a given size pattern on a silk screen the blue ink yielded a thicker line than did the other inks in the printing process. However, the chemical milling process was markedly improved by the use of this ink. Moreover, it was discovered that baking was not necessary to provide a good

pattern.

After establishing the proper ink to use, the proper solution to use, the chemical milling process to use (spray etching), the time of etching and the temperature of the etchant in this experimental scale spray etcher, a continuous spray etcher was obtained and attempts were made to spray-etch 90 percent pattern on a continuous roll basis. The use of this etcher made it possible to chemically mill both sides of the laminate simultaneously, a process that was not possible previously and which therefore caused several difficulties. Initially, for the purposes of testing, samples were placed on a stationary rack rather than on a belt and samples were moved about by hand during the etching process. This resulted in some good samples - others failed due to inadequate silk screen printing and faulty maneuvering of the sample during the etching process. Although the use of ferric chloride provided a rapid chemical milling process and sodium hydroxide had proven to be unworkable in the laboratory scale etcher, it was decided that caustic soda should be tried in the continuous etcher. Unexpectedly, the caustic soda solution provided a much better chemically milled laminate than did the ferric chloride solution. However, the process proceeded at a slower rate. With the discovery that caustic soda produced better chemical milling, experiments were made in the continuous etcher to determine the etchant temperature,

the solution percentage and the time of etching so that the best possible chemically milled material could be produced. After considerable experimentation, the proper solution temperature, (40.5 C) and the proper solution strength (25 percent caustic soda) were established.

Figures 15, 16 and 17 show the method of pattern printing, a printed pattern and the continuous etcher, respectively.

Samples of 48 percent milled material and 90 percent milled material* were subjected to heat treatment prior to milling and to shrinkage tests following milling. Aluminum thicknesses tested were 0.2 mil, 0.6 mil, and 1.0 mil. Heat treatment temperatures were 160 C and heat treatment durations were one minute. In addition, samples of .6 mil aluminum, 70 percent milled patterns were heat treated at 150 C and 140 C. Results of these shrinkage tests are shown in Figures 18 through 25.

*Although the large silk screen pattern (12 inches by 48 inches) was theoretically 90% open, measurements of the rib and flat dimensions of the hexagons revealed that actual percent milling was about 70%. The cause of the loss of open area was the resist used -- it spread on printing. For the cylinder buckling tests a small silk screen was used for printing and these samples were milled to about 90%.

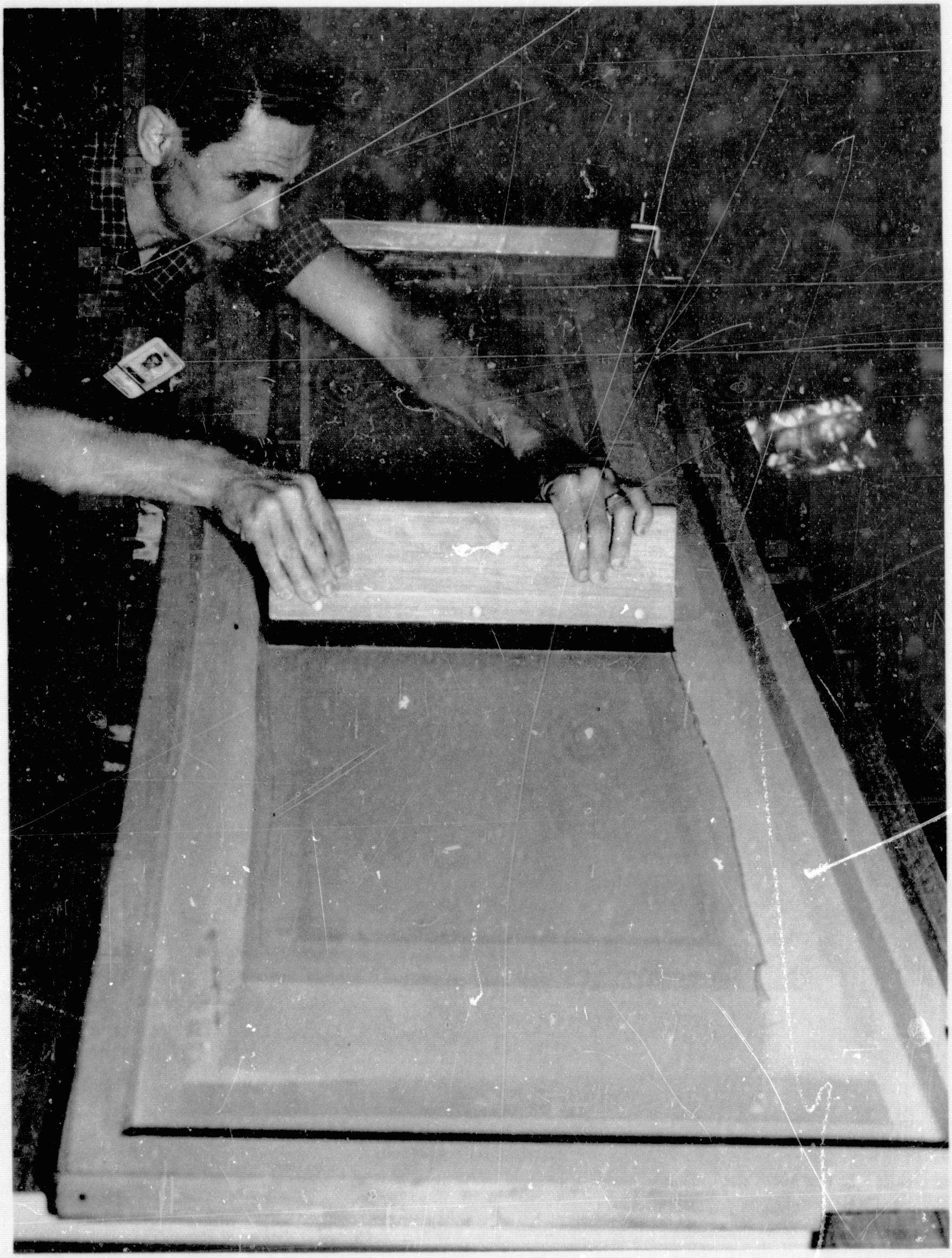
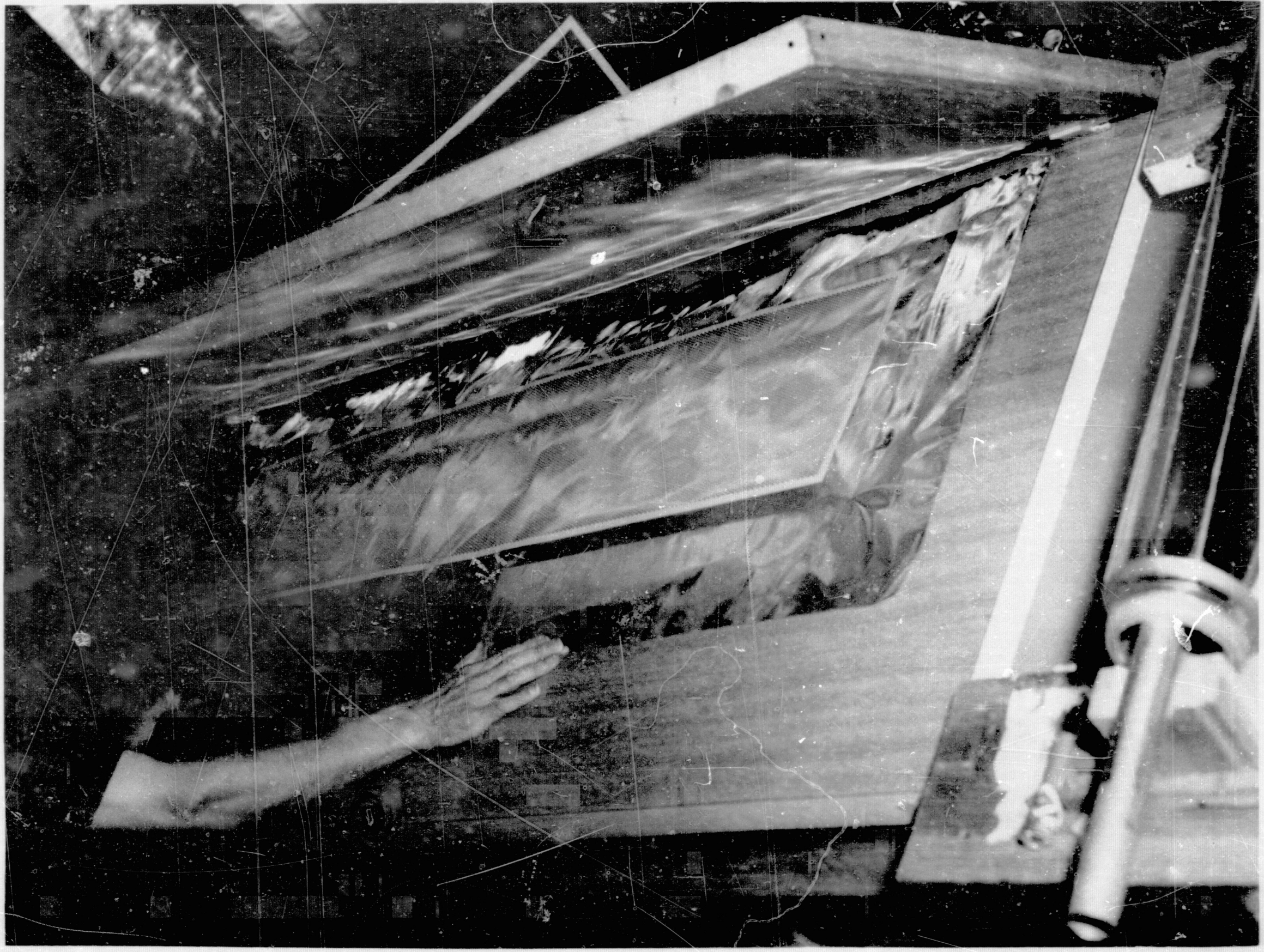


FIGURE 15 - PRINTING RESIST PATTERN WITH 50 INCH SILK SCREEN



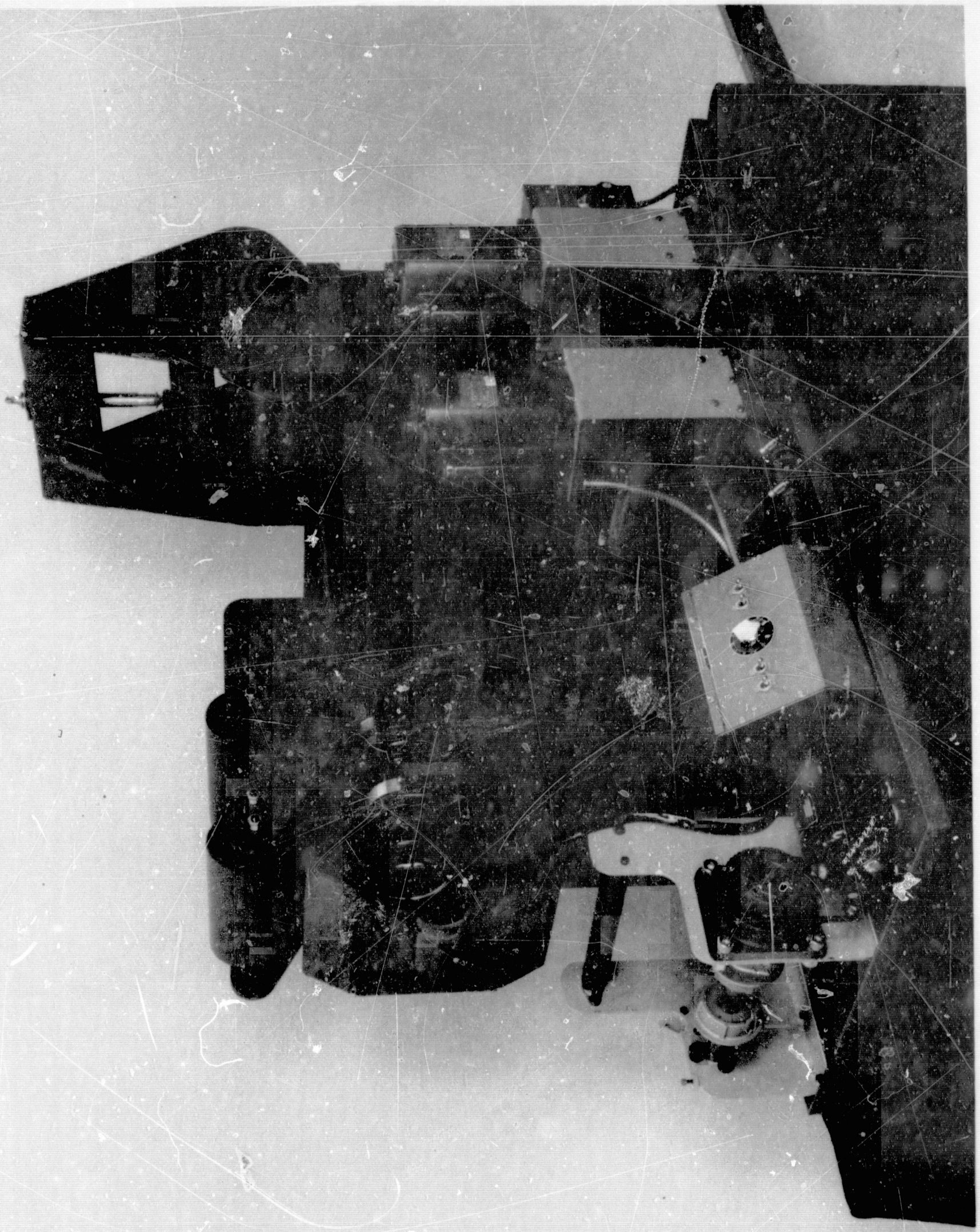


FIGURE 17 - CONTINUOUS ETCHER USED IN MILKING PROCESS

PERCENT SHRINKAGE OF 70% MILLED MATERIAL
FOR VARIOUS SHRINK TEST TEMPERATURES

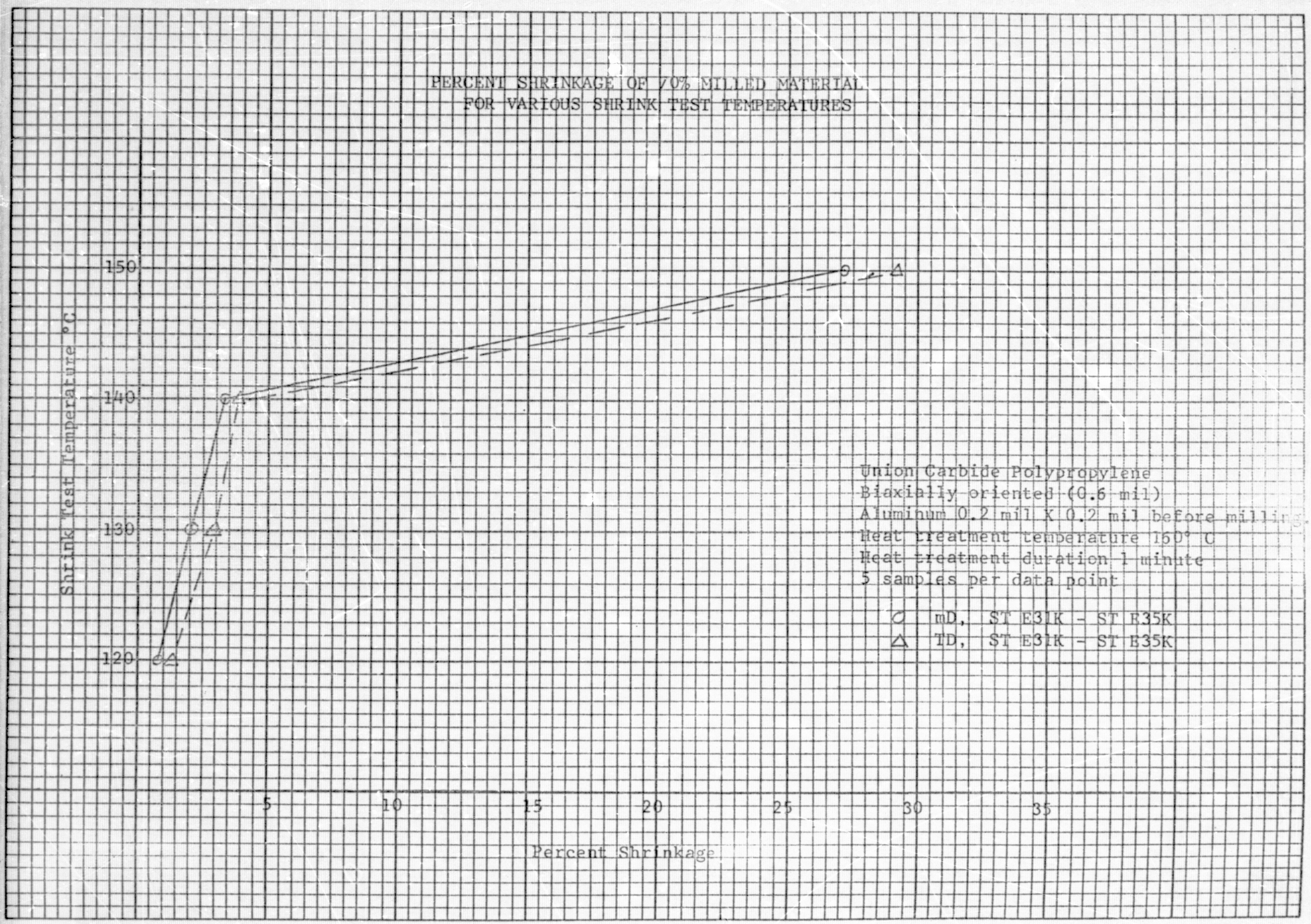


FIGURE 18

PERCENT SHRINKAGE OF 48% MILLED MATERIAL
FOR VARIOUS SHRINK TEST TEMPERATURES

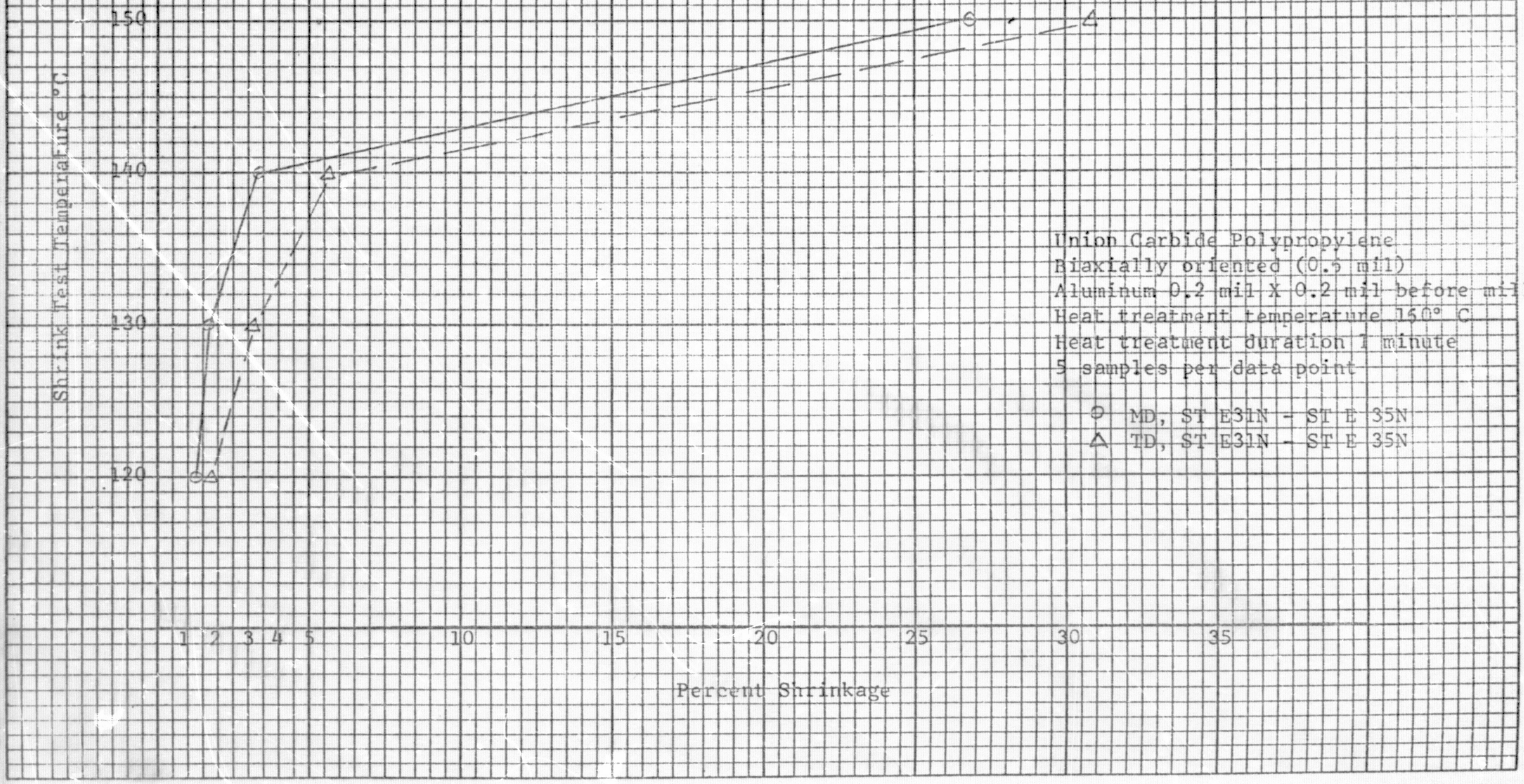


FIGURE 19

PERCENT SHRINKAGE OF 70% MILLED MATERIAL
FOR VARIOUS SHRINK TEST TEMPERATURES

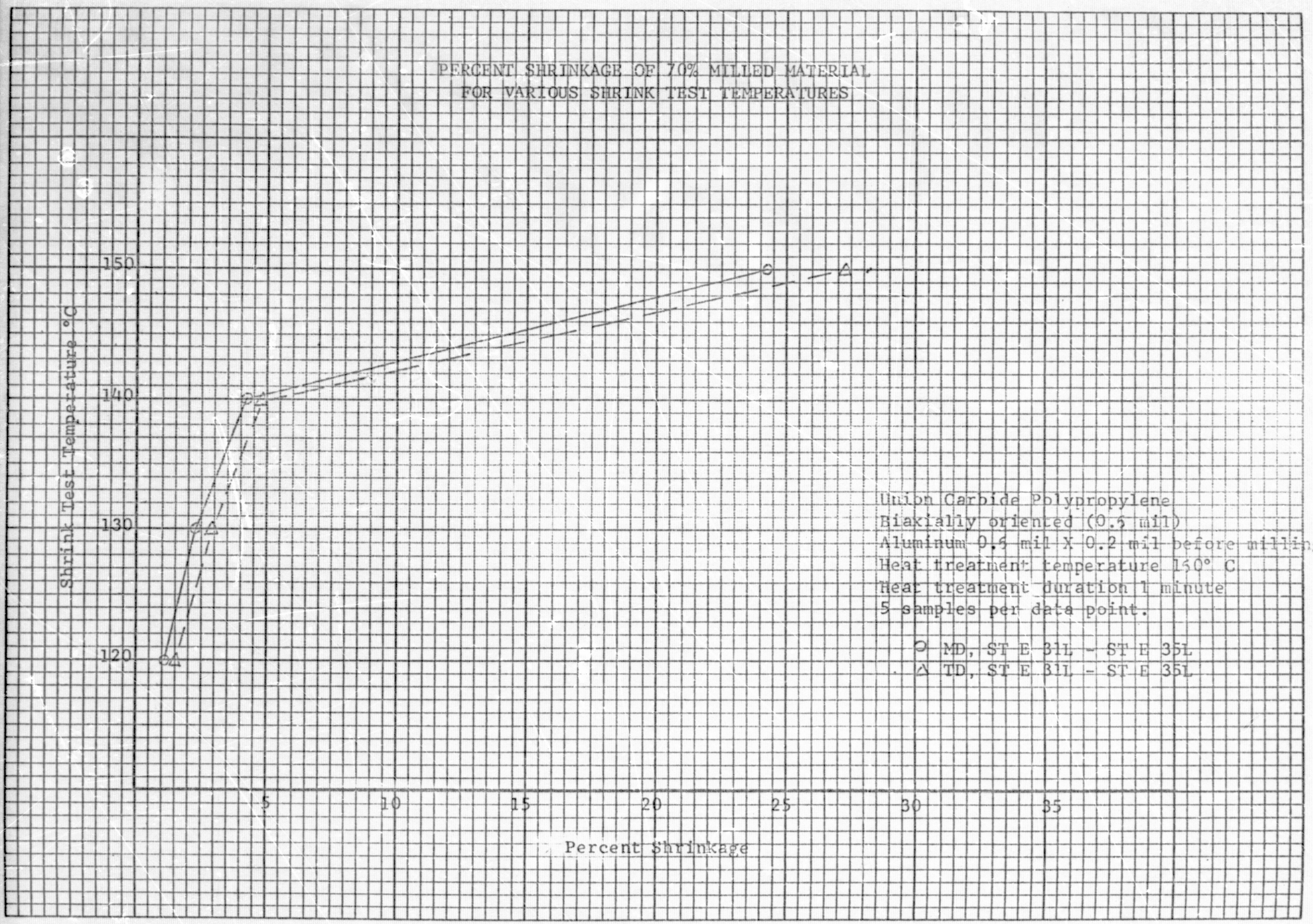


FIGURE 20

PERCENT SHRINKAGE OF 48% MILLED MATERIAL
FOR VARIOUS SHRINK TEST TEMPERATURES

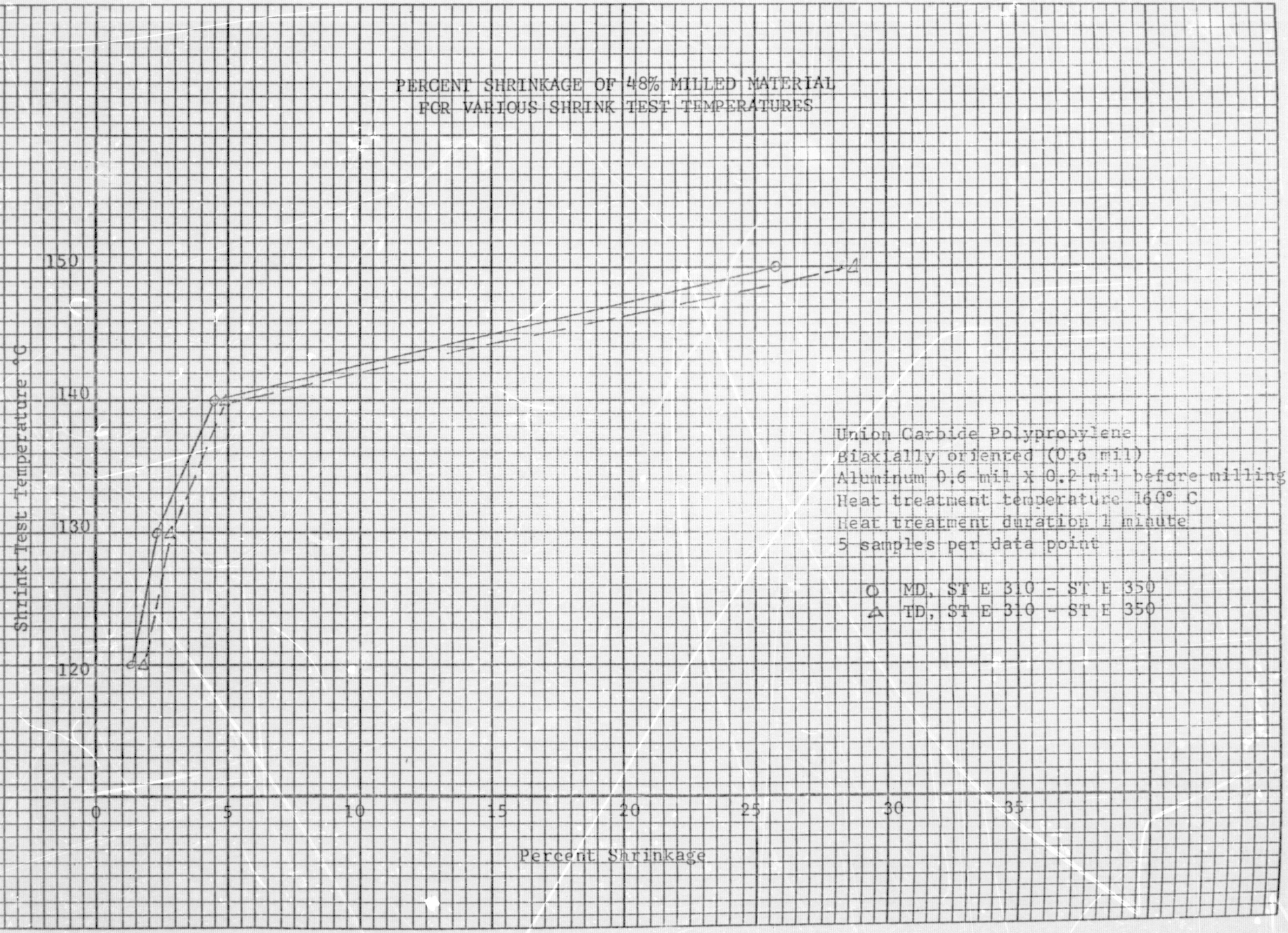


FIGURE 21

PERCENT SHRINKAGE OF 70% MILLED MATERIAL
FOR VARIOUS SHRINK TEST TEMPERATURES

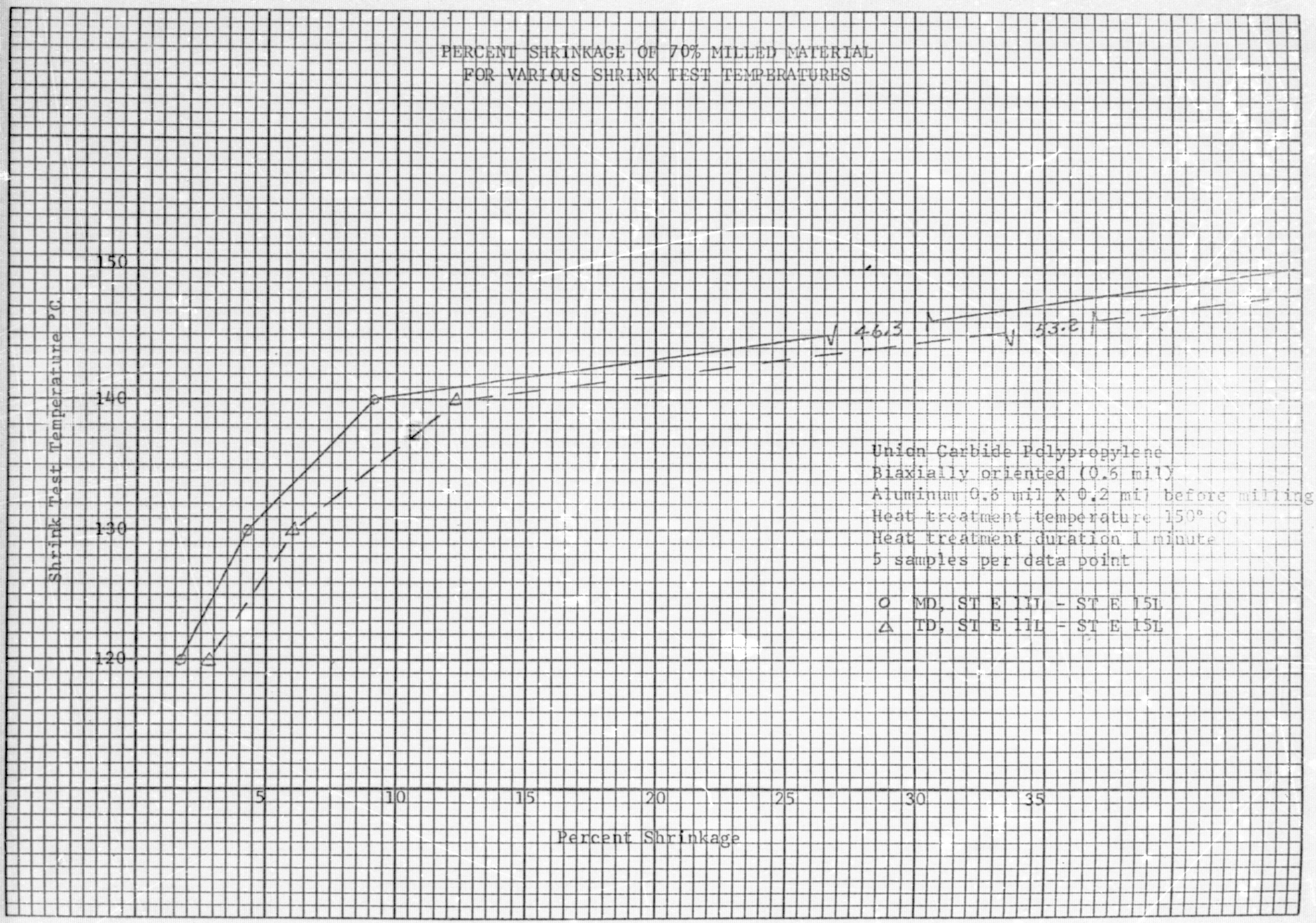


FIGURE 22

PERCENT SHRINKAGE OF 70% MILLED MATERIAL
FOR VARIOUS SHRINK TEST TEMPERATURES

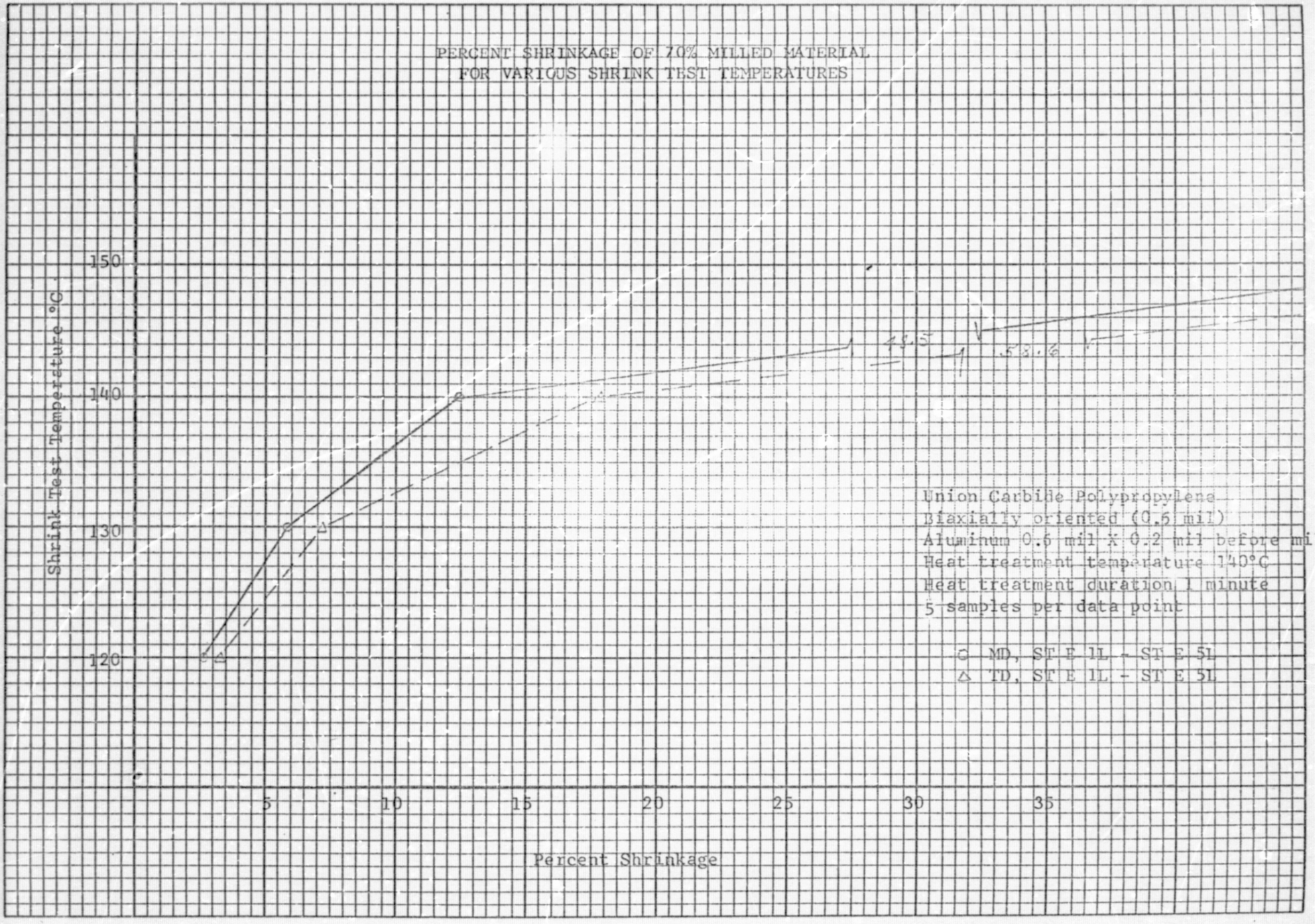
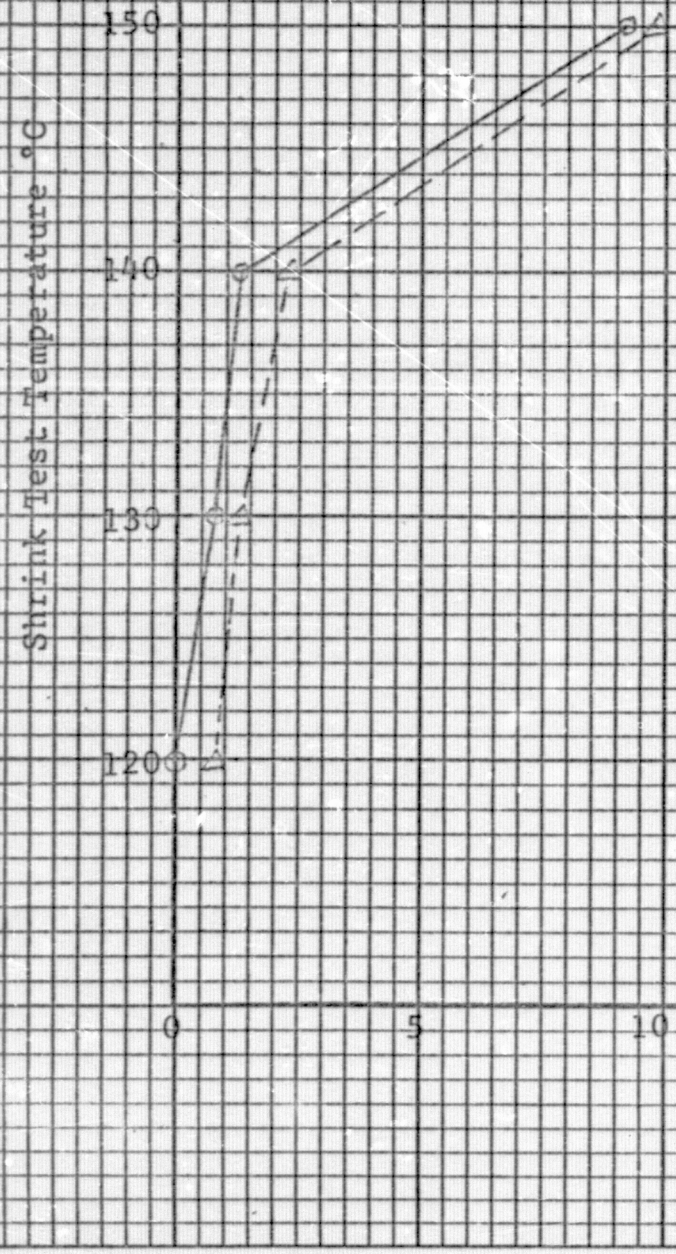


FIGURE 23

PERCENT SHRINKAGE OF 70% MILLED MATERIAL
FOR VARIOUS SHRINK TEST TEMPERATURES

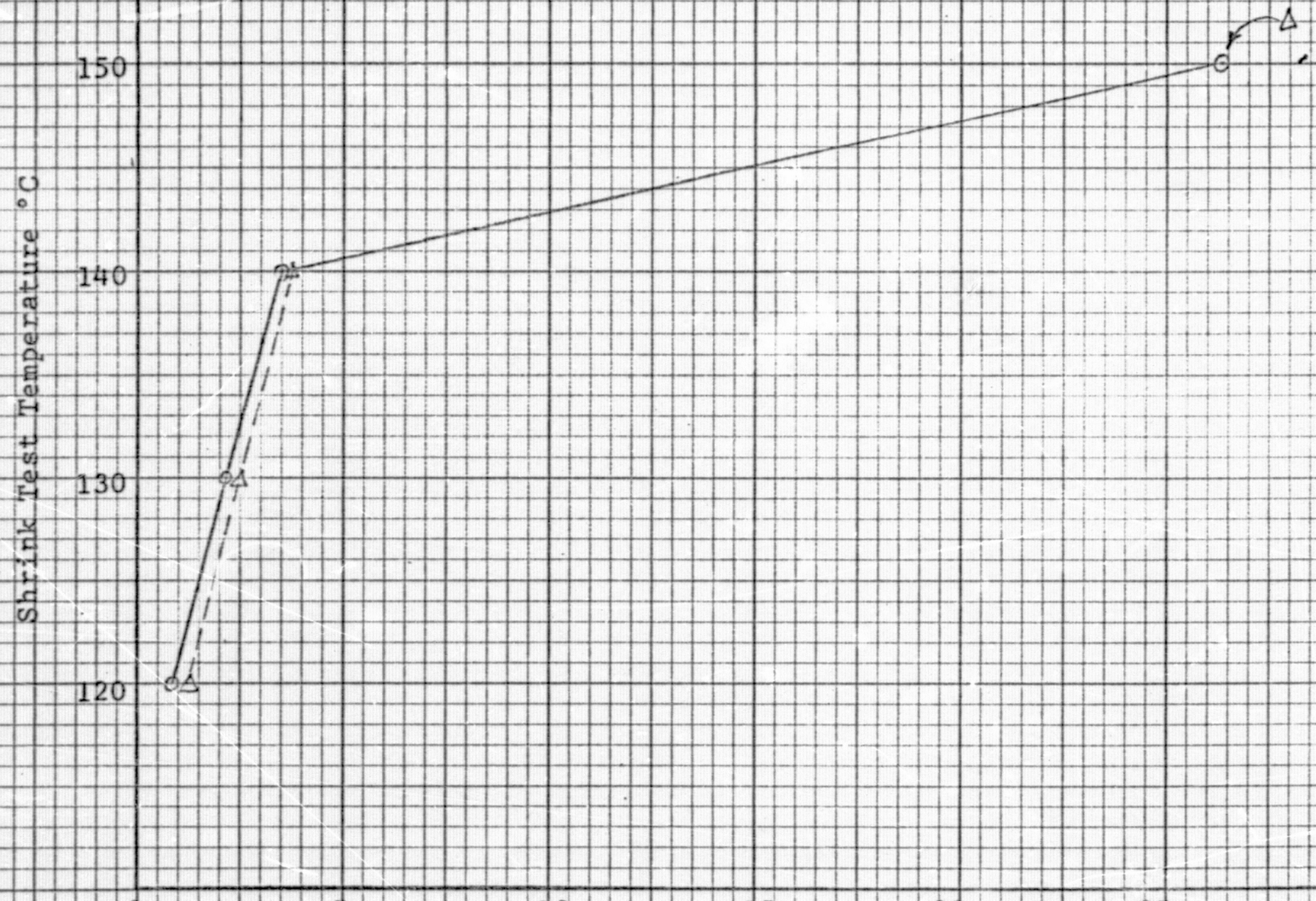


Union Carbide Polypropylene
Biaxially oriented (0.6 mil)
Aluminum 1.0 mil X 0.2 mil before milling
Heat treatment temperature 150°C
Heat treatment duration 1 minute.
5 samples per data point.

○ MD, ST E 31M - ST E 35M
△ TD, ST E 31M - ST E 35M

FIGURE 24

PERCENT SHRINKAGE OF 48% MILLED MATERIAL
FOR VARIOUS SHRINK TEST TEMPERATURES



Union Carbide polypropylene
Biaxially oriented (0.5 mil)
Aluminum 1.0 mil X 0.2 mil before milling
Heat treatment temperature 150° C
Heat treatment duration 1 minute
5 samples per data point

○ MD, ST R 31P - ST E 35P
△ TD, ST R 31P - ST E 35P

FIGURE 25

Puzzling results are obtained as can be seen by comparing the figures. For instance, at a given shrinkage test temperature, the percentage shrinkage is lowest for the 0.2 mil aluminum, higher for the 0.6 mil aluminum and then lower for the 1 mil aluminum. This phenomenon is evident for either 70 percent or 48 percent material. Moreover, for a given thickness of aluminum laminate, the percentage of shrinkage is greater at a given shrinkage test temperature for the 48 percent milled material as compared to the 70 percent milled material. On the other hand, plain polypropylene exhibits a shrinkage percentage greater than that for any of the milled materials at any given shrinkage test temperature.

Table II presents values of shrinkage taken from Figures 5, 18, 19, 20, 21, 24 and 25. Without extensive investigation and experimentation, it is possible at this point to hypothesize that there is a complicated relation between the effective force exerted by the shrinking polypropylene and the buckling strength of the aluminum pattern such as to cause the variations noted.

Measurements of percentage shrinkage also reflect, in part, the actual shrinkage to be expected in an orbiting sphere. That is, shrinkage was measured with the milled material in its buckled condition with no significant attempts being made to flatten the material. By not smoothing the material, unusual test results could have been obtained. However, had the

TABLE II

ONE SIDED, CHEMICALLY MILLED, LAMINATE SHRINKAGE

Aluminum Thickness (inches)	Shrinkage Temperature (C)					
	120 C			140 C		
Percent Milled	100%	70%	48%	100%	70%	48%
.0002	2.5	.8	1.2	6.0	3.4	3.4
.006	2.5	1.0	1.4	6.0	4.3	4.5
.0010	2.5	0.0	.9	6.0	1.3	3.5

Heat Treatment Temperature 160 C

material been flattened prior to the measurement, straining of the material beyond the accuracy of measurement of the experiment would have been necessary. Such a straining of the material being tested would have resulted in erroneous and lower-than-actual values of percent of shrinkage. Therefore, an "eye" fit of a least-squares curve to the data points may be a better approximation to true shrinkage trends than the curves shown in the figures.

VII. MATERIAL TENSILE STRENGTH

A. Objective

The purpose of this section of the test program was to compare the tensile strengths of the materials before and after heat treatment and after varying heat treatment duration. Both as-received and chemically milled materials were processed and tested.

B. Test Description

As-received material was heat treated in the manner described under shrinkage test of as-received material. Chemically milled material, on the other hand, was dipped in the heat treatment bath prior to removal of aluminum from the two-sided aluminum laminate from the polypropylene. The glycerine was cleaned from the laminate surface, a hexagonal material pattern was printed on the aluminum laminate and the material was then chemically milled prior to tensile tests being per-

formed. The tensile tests were preformed on one sided laminates of the percent opening printed.

For each data point plotted, five tensile tests were performed for the machine direction and five tensile tests were performed for the transverse direction of the material undergoing examination.

In addition to tensile strength tests of as-received material and chemically milled material, impact tests were conducted on as-received material. To conduct the impact test, the Instron equipment was operated at a cross head speed of 15 inches per minute in order to simulate impact loading conditions.

C. Discussion

All the materials tested for this section of the program were made from 0.6 mil biaxially oriented Union Carbide polypropylene with or without aluminum laminates.

The results of the tests are shown in Figures 26 through 30. Figure 26 shows that above 160 C of the tensile strength of as-received polypropylene drops off quite rapidly to a low of about 8000 PSI for heat treatment temperature of 170 C. These data indicate that heat treatment temperatures less than 170 C are desirable for this material.

Figure 27 presents the tensile strength of as-received polypropylene which has been heat treated at 160 C for various

ROOM TEMPERATURE TENSILE STRENGTH AS
A FUNCTION OF HEAT TREATMENT TEMPERATURE

Union Carbide Polypropylene
as received.
Biaxially oriented

× MD
○ TD

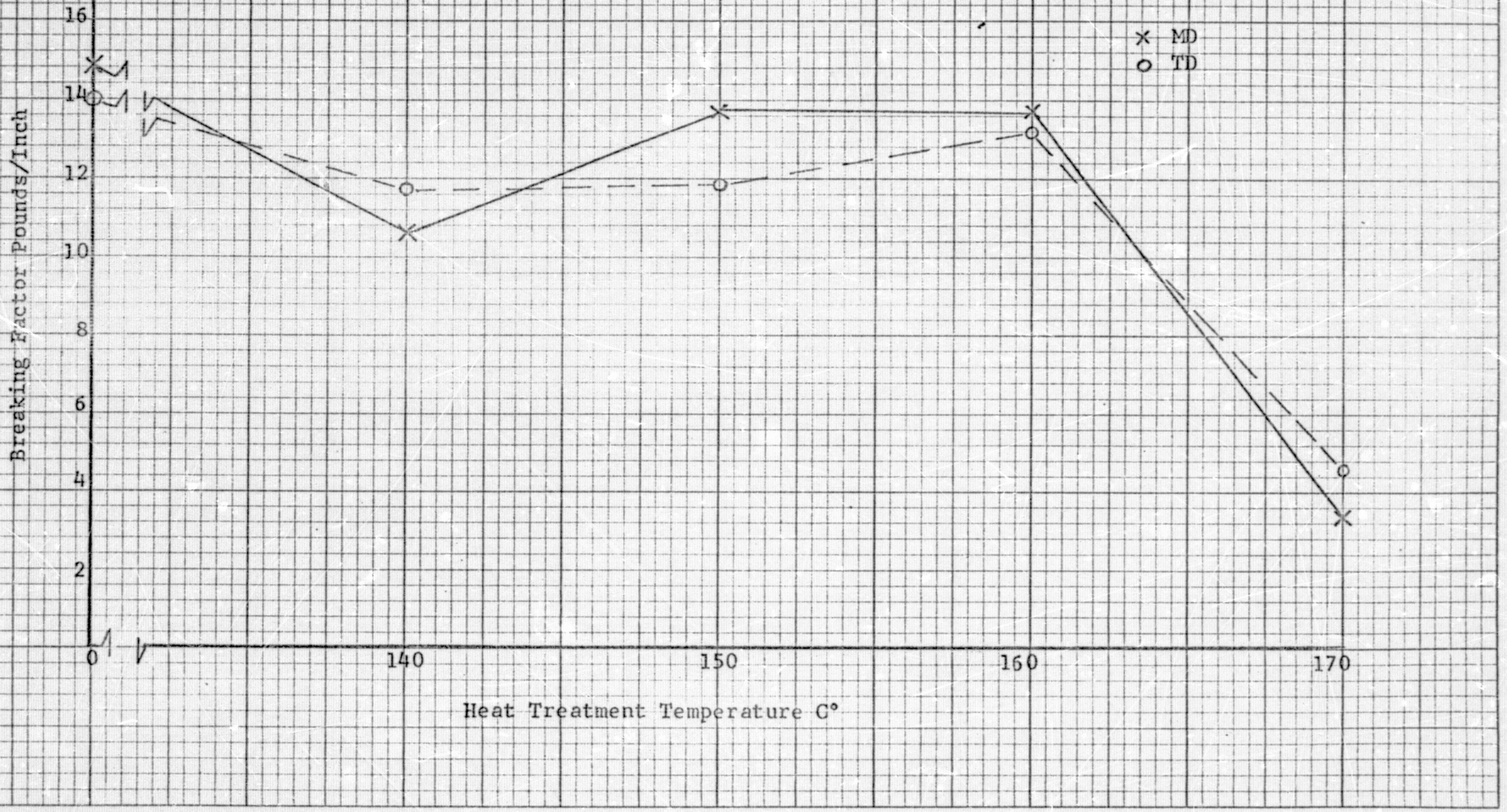
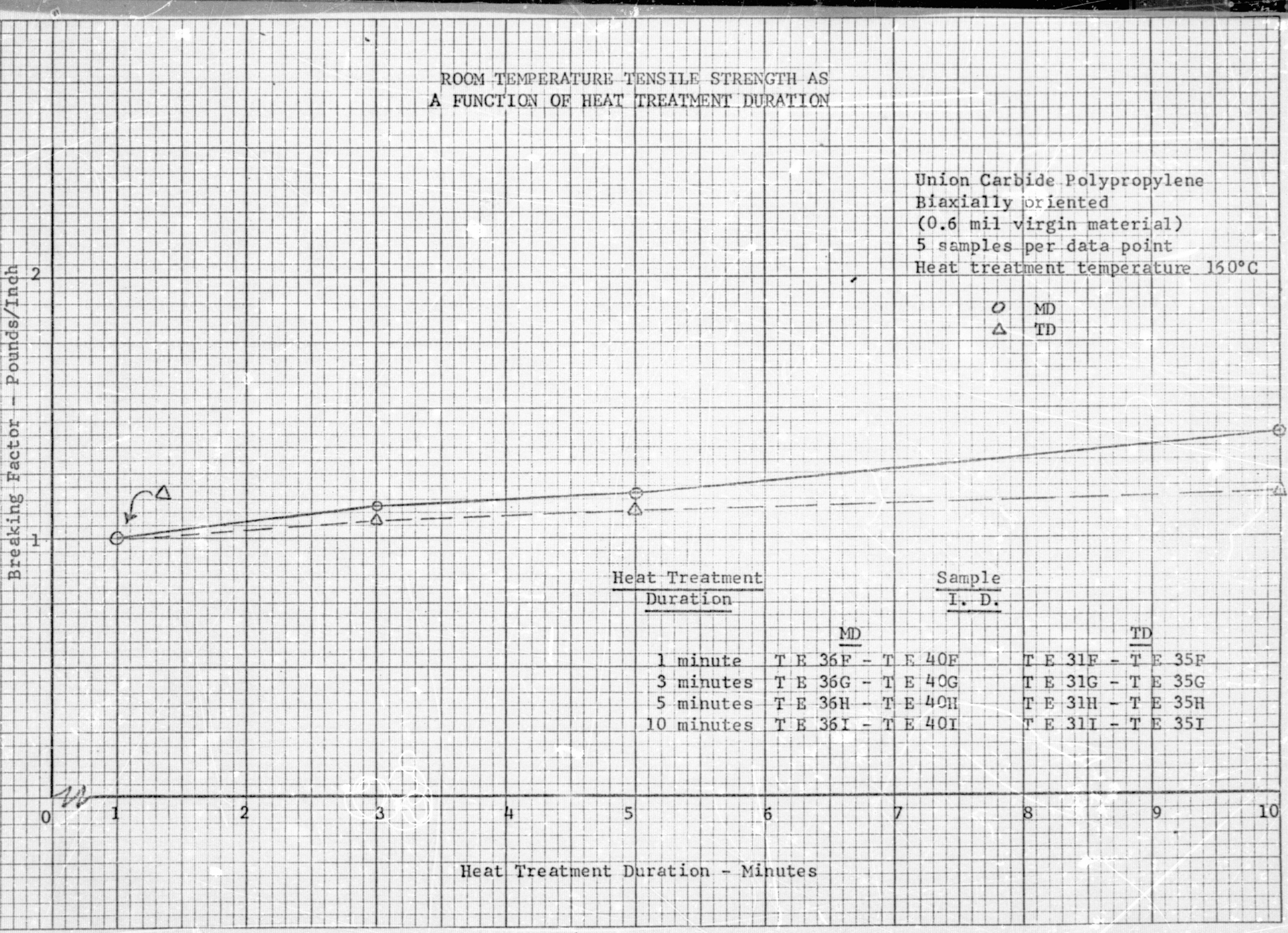


FIGURE 26

ROOM TEMPERATURE TENSILE STRENGTH AS A FUNCTION OF HEAT TREATMENT DURATION

Union Carbide Polypropylene
Biaxially oriented
(0.6 mil virgin material)
5 samples per data point
Heat treatment temperature 160°C

○ MD
△ TD

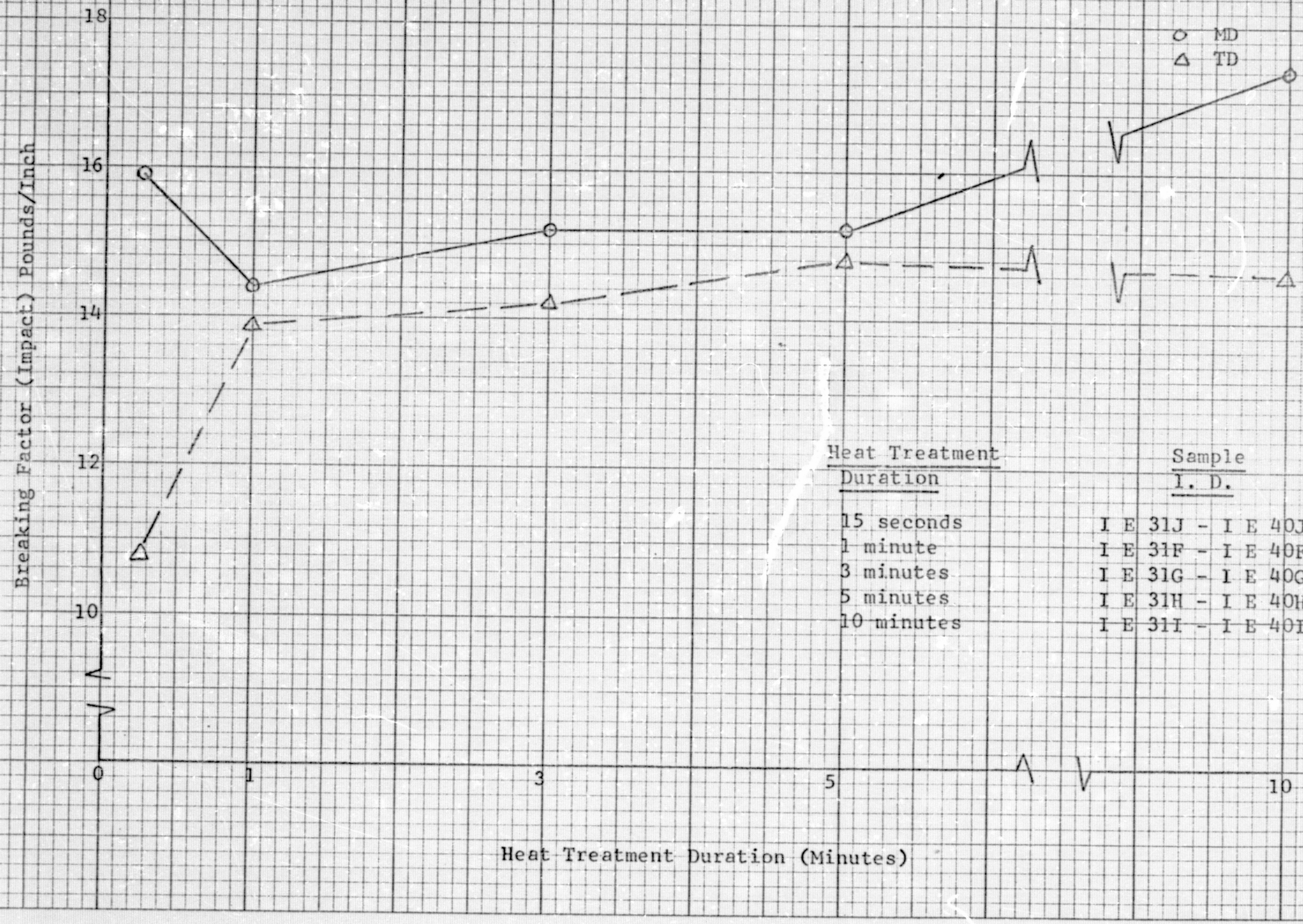


Heat Treatment Duration	Sample I. D. MD	Sample I. D. TD
1 minute	T E 36F - T E 40F	T E 31F - T E 35F
3 minutes	T E 36G - T E 40G	T E 31G - T E 35G
5 minutes	T E 36H - T E 40H	T E 31H - T E 35H
10 minutes	T E 36I - T E 40I	T E 31I - T E 35I

FIGURE 27

POLYPROPYLENE STRENGTH AS A FUNCTION
OF HEAT TREATMENT DURATION

Union Carbide Polypropylene
Biaxially oriented (0.5 mil)
5 samples per data point
Heat treatment temp. 150°C



Heat Treatment Duration	Sample I. D.
15 seconds	I E 31J - I E 40J
1 minute	I E 31F - I E 40F
3 minutes	I E 31G - I E 40G
5 minutes	I E 31H - I E 40H
10 minutes	I E 31I - I E 40I

FIGURE 28

TENSILE STRENGTHS OF HEAT TREATED LAMINATES
AS A FUNCTION OF HEAT TREATMENT TEMPERATURE (TD)

70% milled
Union Carbide polypropylene
Biaxially oriented (0.5 mil)
Aluminum (before milling)

O	0.2 mil X 0.2 mil
Δ	0.6 mil X 0.2 mil
X	1.0 mil X 0.2 mil

5 samples per data point

Breaking Factor Pounds/Inch

20
18
16
14
12
10
8
6
4
2

140
150
160
170

Heat Treatment Temperature C°

Heat Treatment Temperature	0.2 mil Aluminum	0.6 mil Aluminum	1.0 mil Aluminum
No HT	T E.0K - T E.4K	T E.5L - T E.9L	T E.0M - T E.4M
140°C	T E1K - T E5K	T E6L - T E/0L	T E1M - T E5M
150°C	T E11K - T E15K	T E 16L - T E 20L	T E11M - T E 15M
160°C	T E31K - T E35K	T E31L - T E35L	T E31M - T E 35M
165°C	T E41K - T E45K	T E41L - T E45L	T E41M - T E45M
170°C	T E51K - TE55K	T E51L - T E55L	T E51M - T E55M

FIGURE 29

TENSILE STRENGTH OF HEAT TREATED LAMINATES
AS A FUNCTION OF HEAT TREATMENT TEMPERATURES (MD)

70% milled
Union Carbide polypropylene
Biaxially oriented (0.5 mil)
Aluminum (before milling)

○ 0.2 mil X 0.2 mil
△ 0.6 mil X 0.2 mil
✕ 1.0 mil X 0.2 mil
--- No data available

Breaking Factor Pounds/Inch

20
18
16
14
12
10
8
6
4
2
0

Heat Treatment Temperature	0.2 mil Aluminum	0.6 mil Aluminum	1.0 mil Aluminum
No HT	T E.5K - T E.9K	T E.0L - T E.4L	T E.5M - T E.9M
140°C	T E6K - T $10K	T E1L - T E5L	T E6M - T E10M
150°C	T E16K - T E20K	T E11L - T E15L	T E16M - T E20M
160°C	T E36K - T E40K	T E36L - T E40L	
165°C	T E46K - T E50K	T E46L - T E50L	T E46M - T E50M
170°C	T E56K - T E60K	T E56L - T E60L	T E56M - T E60M

140 150 160 170

Heat Treatment Temperature C°

FIGURE 30

durations. Because shrinkage has been shown to be minimized by a heat treatment duration of one minute, the data plotted on Figure 27 are related to strength values determined at one minute heat treatment duration. That is, for both the machine and transverse directions the values of breaking factors at each heat treatment duration were divided by their respective values at the heat treatment duration of one minute. As a result, the normalized breaking factors plotted then begin at a value of one for a heat treatment duration of one minute and vary as the breaking factor varies. In the general trend shown in Figure 27, the tensile strength of the as-received polypropylene appears to gain strength as the heat treatment duration is increased. This phenomenon may be caused not by a fundamental change in the strength of the material, but rather by a change in the effective cross-section of material being tested in the Instron tester. Although the as-received material is suspended in embroidery hoops and theoretically does not shrink during heat treatment, some shrinkage probably occurs during heat treatment. As a result, a given sheet of film effectively will be of smaller sheet dimensions but of greater material thickness. Thus, when a sample of polypropylene is cut from this treated material for tensile testing, Instron tests show increasing loading necessary to fracture the sample (for increasing heat treatment duration). Another cause of the apparent increase in strength may be

further orienting of the film during heat treatment.

Figure 28 presents the breaking factor in impact for as-received material as a function of the duration of heat treatment. This graph also shows an increase in strength as the heat treatment duration is lengthened. For the same reasons as were previously presented, this is an apparent increase in material strength. The important point to note, however, is that the treatment of as-received polypropylene in glycerine at high temperatures for long durations does not affect the strength of the polypropylene.

Figures 28 and 29 present the tensile strength of chemically milled heat-treated laminates as functions of heat treatment temperature. Variations in the thickness of the aluminum laminate are presented as parameters of the curves. As explained previously, these materials were heat treated in glycerine baths prior to chemical milling. The polypropylene had an aluminum laminate bonded to each side during heat treatment. Thus, the laminate provided a physical barrier to the glycerine to prevent contact between the polypropylene and glycerine, and the aluminum laminate provided a uniform support for the polypropylene during heat treatment. Figures 28 and 29 indicate that the lack of contact between the polypropylene and the glycerine during heat treatment and the uniform support provided by the aluminum during heat treatment do not materially affect the strength of the polypropylene in tensile tests.

This statement is based on a comparison of the breaking factor for Figures 28 and 29 and Figure 26 (as-received material) for heat treatment temperatures of 150 C and higher. Although Figures 28 and 29 indicated higher strength at 150 C compared to Figure 26, the strength of the milled material at 170 C is about 1/2 that of the material shown on Figure 26 for the same temperature. It should be pointed out here, however, that this apparent difference could be caused by the as-received material having shrunk and become thicker for a given width of material than the same width of the aluminum laminate sample.

D. Conclusions

1. Heat treatment temperatures for polypropylene should be less than 170 C as determined by tensile strength tests. This does not preclude lower heat treatment temperatures as suggested by other tests in this study program.
2. Heat treatment duration does not affect the strength of polypropylene adversely.
3. Heat treatment duration does not affect impact strength of polypropylene adversely.
4. Heat treatment of polypropylene whether in the as-received or two-sided laminated condition makes little if any difference in the tensile strengths of the material.
5. Heat treatment of polypropylene should be conducted on a two-sided laminate to obtain the highest uniformity of

support given to the polypropylene during heat treatment and to minimize or eliminate the contact of the glycerine bath with the polypropylene film.

VIII. OPTICAL PROPERTIES OF POLYPROPYLENE

A. Objective

The objective of this portion of the test program was to determine the effect of heat treatment temperature and heat treatment duration on infrared and solar transmittance of polypropylene. Polypropylene from two different manufacturers and with and without Corona treatment was analyzed on a Perkin-Elmer model 137 Spectrophotometer and a Beckman BK2A Spectrophotometer.

B. Test Description

As-received material was heat treated in the manner utilized in shrinkage testing. Following heat treatment of the as-received material in glycerine, the glycerine was washed away with a solution of isopropanol and water so that optical measurements which were to be made would not be affected. In one case, however, the glycerine was only washed off with water. In all cases, the material being tested was allowed to dry thoroughly before it was subjected to the optical tests.

Sections of the heat treated polypropylene were cut from the heat treated sample and tested in the Beckman BK2A Spectro-

photometer and a Perkin-Elmer model 137 Spectrophotometer. From the Spectrophotometer transmission reading of the sample, the ratio of transmitted to total energy for the sample was compared to the curve of black body radiation. By integrating this comparison the infrared and solar transmissions of the sample was obtained.

C. Discussion

Materials tested included the following:

1. Union Carbide biaxially oriented polypropylene 0.6-mil thick without Corona treatment.
2. Union Carbide biaxially oriented polypropylene 0.6-mil thick Corona treated one side.
3. Union Carbide biaxially ariented polypropylene 0.6-mil thick Corona treated both sides.
4. Hercules Powder Co. biaxially oriented polypropylene 0.5-mil thick Corona treated one side.

In all cases of varying heat treatment temperature, heat treatment duration, material thicknesses, material vendors, material surface treatments, results are in the range of 79 percent to 84 percent in the infrared transmission region. For the case of the as-received polypropylene which was cleaned only in water after the glycerine heat treatment, the infrared transmission was 76 percent while another sample of the same material which had the glycerine cleaned by use of isopropanol solution showed an infrared transmission

value of 79 percent -- not a significant difference.

The solar transmission of the same materials was calculated for Union Carbide biaxially oriented polypropylene 0.6-mil thick. Corona treated both sides, and the value obtained was 90.9 percent. Calculations for the other samples of polypropylene were not made since the raw data from which the calculations are made is very nearly that used for the infrared transmission calculations. This was estimated by overlaying data sheets on a vertical light table. Since all of the infrated transmission values are within 5 percent of one another, it was concluded that the solar transmission values would all be about 90 percent. Table 3 shows the results of the calculations made.

D. Conclusions

1. Glycerine heat treatment of as-received polypropylene does not significantly affect its solar or infrared transmission properties relative to the untreated material.
2. Variations in solar and infrared transmission characteristics of Hercules Powder Co. and Union Carbide polypropylene are not significant.
3. Small variations in polypropylene thickness have little if any effect on solar and infrared transmission values.
4. The duration of heat treatment has little or no effect on the value of solar or infrared transmission characteristics of polypropylene.

TABLE III

SPECTROPHOTOMETER TRANSMISSION VALUES FOR POLYPROPYLENE AND MYLAR

MATERIAL	HEAT TREATMENT TEMPERATURE	HEAT TREATMENT DURATION	SOLAR* TRANSMISSION	INFRARED TRANSMISSION
UC - 3	No Heat Treatment			82.3
(.0006 in.)	140 C	15 Sec.		82.3
	150 C	15 Sec.		84.1
	160 C	15 Sec.		84.0
	170 C	15 Sec.		79.6
UC - 4	160 C	15 Sec.		79.0
(.0006 in.)				
UC - 5	160 C	10 Min.	90.9	82.6
(.0006 in.)				
HE - 4	160 C	15 Sec.		82.6
(.0005 in.)	160 C	5 min.		82.4
	160 C	10 min.		81.5
Mylar	No Heat Treatment	---	83.7	48.7
	160 C	15 Sec.	83.7	48.7
(.0005 in.)	160 C	5 Min.	83.7	48.7
	160 C	10 Min.	83.7	48.7

* Where values are not presented for τ_s, it was assumed that their magnitude was that of UC-5; 90.9 percent.

CODE:

UC - Union Carbide Plastics Co.

HE - Hercules Powder Co.

3 - Not Corona Treated

4 - Corona Treated One Side

5 - Corona Treated Both Sides

5. The solar and infrared transmission characteristics of polypropylene is not affected significantly by the presence or absence of Corona treating of one or both sides of the film.

6. Because the presence of glycerine may lower the transmission characteristics of polypropylene, it is important that polypropylene be heat treated under cover of two-sided aluminum laminates to prevent accidental carrying of glycerine into space where lowered transmission characteristics would increase local temperatures of the material.

IX. HEAT TREATMENT AND SHRINKAGE OF MYLAR AND MYLAR LAMINATES

A. Objective

The objective of this section of the report was to determine shrinkage which occurred in Mylar after Mylar had been subjected to varying degrees of heat treatment temperature.

B. Test Description

Shrinkage tests of as-received 35 gage Mylar were conducted in exactly the same manner as those for polypropylene. The Mylar was mounted in embroidery hoops and exposed to the glycerine bath for the heat treatment portion of the cycle. Following this step the Mylar was cleaned with isopropanol and water solution and the re-exposed to shrink test temperatures and measurements were made of the shrinkage of a three-inch square measuring pattern.

C. Discussion

Results of the tests are presented on Figure 31. It may be noted that for decreasing degrees of heat treatment the amount of shrinkage experienced in Mylar at any given shrink-test temperature generally decreases. This suggests that Mylar without any heat treatment may undergo the least amount of shrinkage. This hypothesis is partially confirmed by the results listed on Table 4.

Table 4 presents shrinkage values for as-received 35 gage Mylar and X-15 material after exposure to a shrinkage test temperature of 90 C without prior heat treatment. Although this test was conducted in a hot air oven and is at a lower shrink test temperature, it is believed that this table lends further weight to the hypothesis that no heat treatment of Mylar provides a minimum of shrinkage of Mylar on re-exposure to heat energy.

X-15 material, it should be noted shrinks less (approximately 1/2) than does 35 gage Mylar for comparable exposure times in the oven with the exception of the figures for six days exposure. However, these figures may be misleading since the amount of measured shrinkage in Mylar is extremely small. This fact suggests that measurements are difficult to make and the figures presented may actually be averages of measuring errors made. Norwithstanding the fact that measuring errors may cause the variations in percentage shrinkage in

PERCENT SHRINKAGE OF MYLAR AS A FUNCTION
OF SHRINK TEST TEMPERATURE

0.35 mil Mylar film
3 samples per data point
X31-X33 200°C
X34 - X36 210°C
X37 - X39 220°C

○ TD -200°C
△ MD -210°C
□ MD -220°C
✳ TD -200°C
+ TD -210°C
• TD -220°C



FIGURE 31

ABLE IV

.CENT RINKA F MYLAR AND X MATERIAL

S A FU ION (HRINKAGE EXP E DURATION

INKAGE E)SURE RATION (H)	SHRINKAGE (PERCENT)			
	MYLAR (.0C in.)		X-15	
	MD	T	MD	TD
4	.2	.4	.4	0
8	.4	.6	.2	.2
12	.4	.6	.6	.2
16	.4	.4	.4	.2
20	.4	.6	.2	.2
24	.4	.6	.4	.2
144	.5	.7	.6	.4

No He eatment.

Shrin kage Obtained in Ove t 90 C.

Table 4, it is obvious that the shrinkage of Mylar is quite small when compared with that of polypropylene at any given shrinkage test temperature.

D. Conclusions

1. Mylar should not be heat treated to minimize shrinkage on exposure to heat energy.

X. OPTICAL PROPERTIES OF MYLAR

A. Test Objective

The objective of this portion of the test program was to determine the infrared and solar spectrophotometer transmission characteristics of Mylar which was both heat treated and not been heat treated to determine the effect of heat treatment on these characteristics.

B. Test Descriptions

Samples of 35 gage Mylar were heat treated in glycerine in the standard manner. Heat treatment durations varied in the following manner: 15 seconds, 5 minutes, and 10 minutes. In addition, one sample was not heat treated.

The results of the tests are shown on Table 3. Mylar has an infrared transmission value of 48.7 percent for no heat treatment or for heat treatment of any duration up to 10 minutes. Its solar transmission value is 83.7 percent under the same parametric variations.

A comparison of Mylar with polypropylene on the basis of infrared and solar transmission values shows that Mylar is less transparent than polypropylene. However, the implied higher temperatures of Mylar caused by these lower transmission values, may be offset by this significantly lower shrinkage value that Mylar exhibits when compared with polypropylene. Therefore, Mylar may be the more appropriate material for use in low-shrinkage space inflatables whose characteristics must be high radio frequency reflectivity and long life.

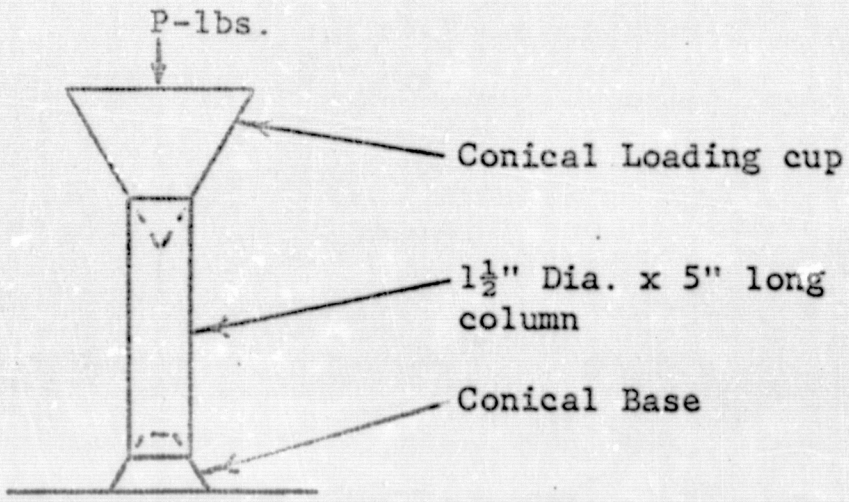
XI. COLUMN TESTING

A. Objective

The purpose of the test program was to compare chemically milled (hexagonal pattern) laminates fabricated with 0.6 mil biaxially oriented polypropylene and various aluminum foil thicknesses. Both heat treated and non-heat treated material were tested for further comparisons.

B. Test Description

1 1/2 inch diameter by 5 inch long cylinders were formed from the laminate material and tested as follows:



The column is set vertically on a conical base and loaded with a conical cup which is slowly filled with steel shot. The conical base and load cup provide an excellent loading device since they automatically compensate for irregularities in cutting, therefore eccentric loading is minimized. The nature of the column failure is such that the column will support a load up to a critical one. At that point, the column is unstable and a small additional weight will cause the column to buckle. Loading with steel shot provides a very accurate method for determining the critical load of the material since each pellet is a small fraction of a gram. The critical load can be determined within a very few grams in nearly all cases. It is relatively easy to determine whether the column is loaded axially because failure will occur as bending rather than yielding of the material in one plane.

The number of tests conducted is dependent upon the consistency of results. In some cases, 3 tests may be adequate; in others 8 to 10 tests may be required. In all cases, skill and judgement must be exercised. Haste, or non-skilled personnel can affect the results to a considerable extent.

In this portion of the test program, samples of 90 percent material were produced and tested. This contrasts with all other portions of the test program in which the maximum percent milled was 77 percent.

C. Test Apparatus

1. Conical Load Cup
2. Conical base
3. Steel shot (balloon ballast)
4. Ringstand with ring
5. Gram scale
6. Camera

D. Discussion

The materials tested included the following:

(All were one sided laminates)

1. 2.0 mil aluminum

   90 percent milled and non-milled

2. 1.0 mil aluminum

   90 percent milled and non-milled

3. 0.6 mil aluminum

   90 percent milled and non-milled

4. 0.2 mil aluminum

   90 percent milled and non-milled

5. 0.6 mil aluminum

   48 percent milled

6. 1.0 mil aluminum

   48 percent milled

7. 0.6 mil polypropylene

   both heat treated and non-heat treated.

POOR QUALITY IN ORIGINAL DOCUMENT

All aluminum laminates had 0.6 mil Union Carbide Plastic Co. biaxially oriented polypropylene as the plastic.

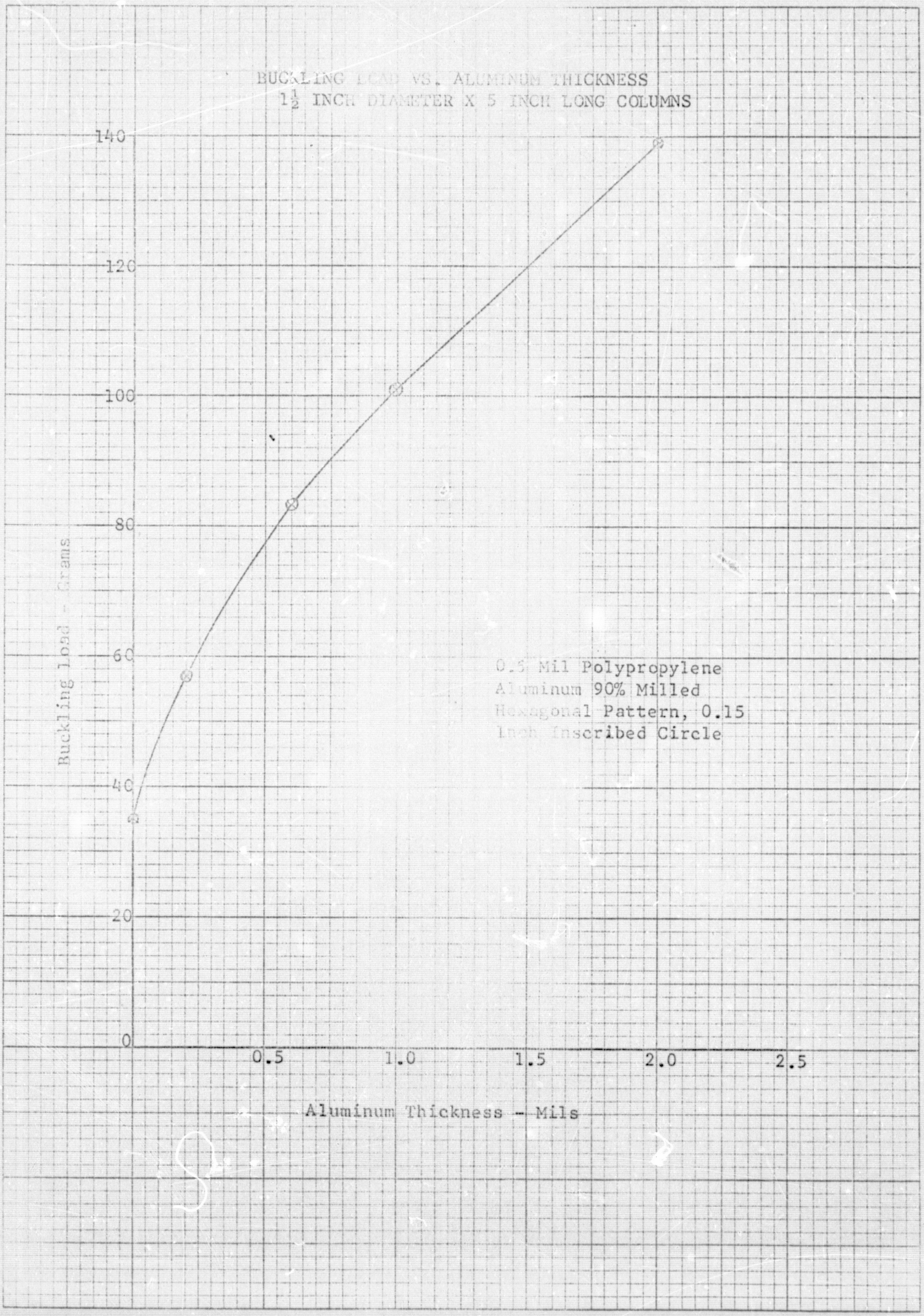
The results of the tests are shown graphically in figures 32 through 36 and pictorially in Figures 37 through 40. In general, the data are consistent and exhibit the trends expected. For a given plastic the load capacity of the material decreases as the fraction of aluminum removed increases or as the aluminum thickness decreases.

Laminates with heat treated and non-heat treated polypropylene were tested and compared. It was concluded that heat treating has no significant effect on the material strength in buckling.

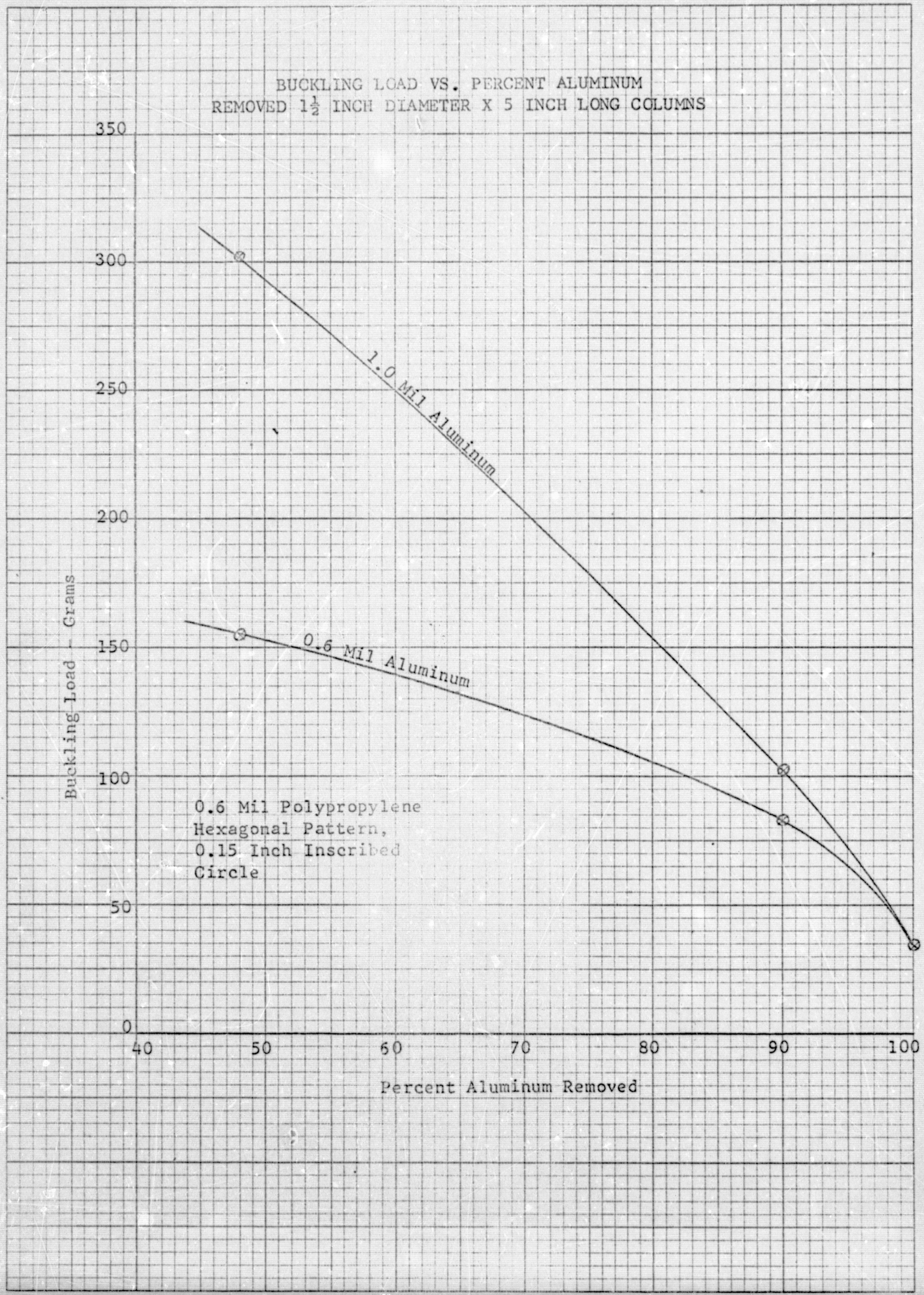
Figures 32 through 34 are self explanatory. Figure 35 seems strange, but it is reasonable. Polypropylene is the factor which determines the shape of the curve. With thin aluminum percent removed the strength of the polypropylene is more significant than with thick aluminum. For the non-milled material, the polypropylene strength is not significant for any of the materials tested. Therefore, as the thickness of aluminum is decreased, the ratio of load capacity will approach 1 (zero aluminum thickness).

An adjusted curve is also shown on Figure 35. It was difficult to obtain smooth test specimens with the extremely thin non-milled foil laminate. As a result, it is believed that failure occurred prematurely. Test experience has shown that

BUCKLING LOAD VS. ALUMINUM THICKNESS
$1\frac{1}{2}$ INCH DIAMETER X 5 INCH LONG COLUMNS



BUCKLING LOAD VS. PERCENT ALUMINUM
REMOVED 1½ INCH DIAMETER X 5 INCH LONG COLUMNS



BUCKLING LOAD VS. ALUMINUM THICKNESS
$1\frac{1}{2}$ INCH DIAMETER X 5 INCH LONG COLUMNS
NON-MILLED MATERIAL

Buckling Load - Grams X 10^{-1}

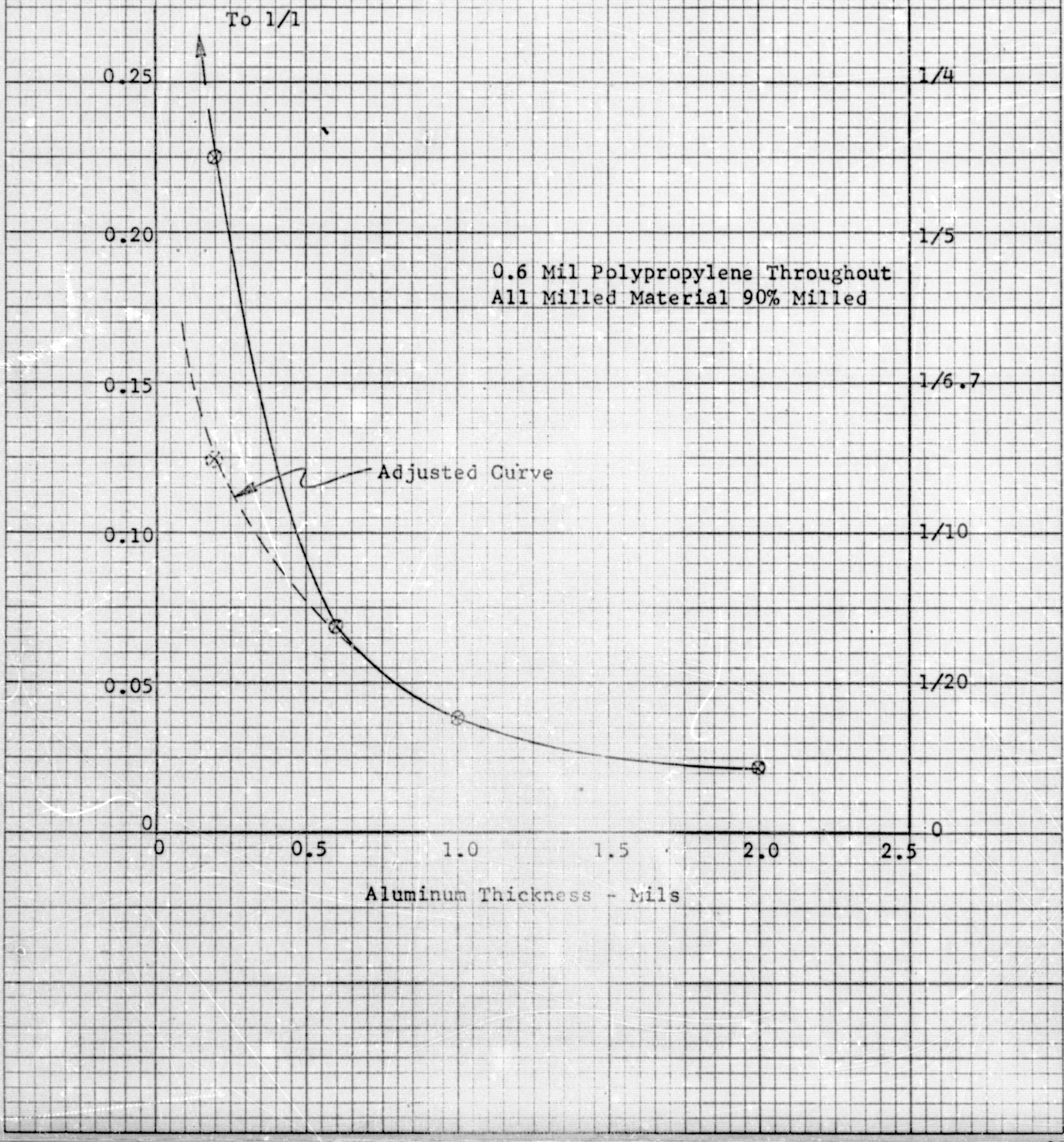
700
600
500
400
300
200
100
90
80
70
60
50
40
30
20
10
9
8
7
6
5
4
3
2

Aluminum-Polypropylene Laminate, 0.6 Mil Poly-Propylene

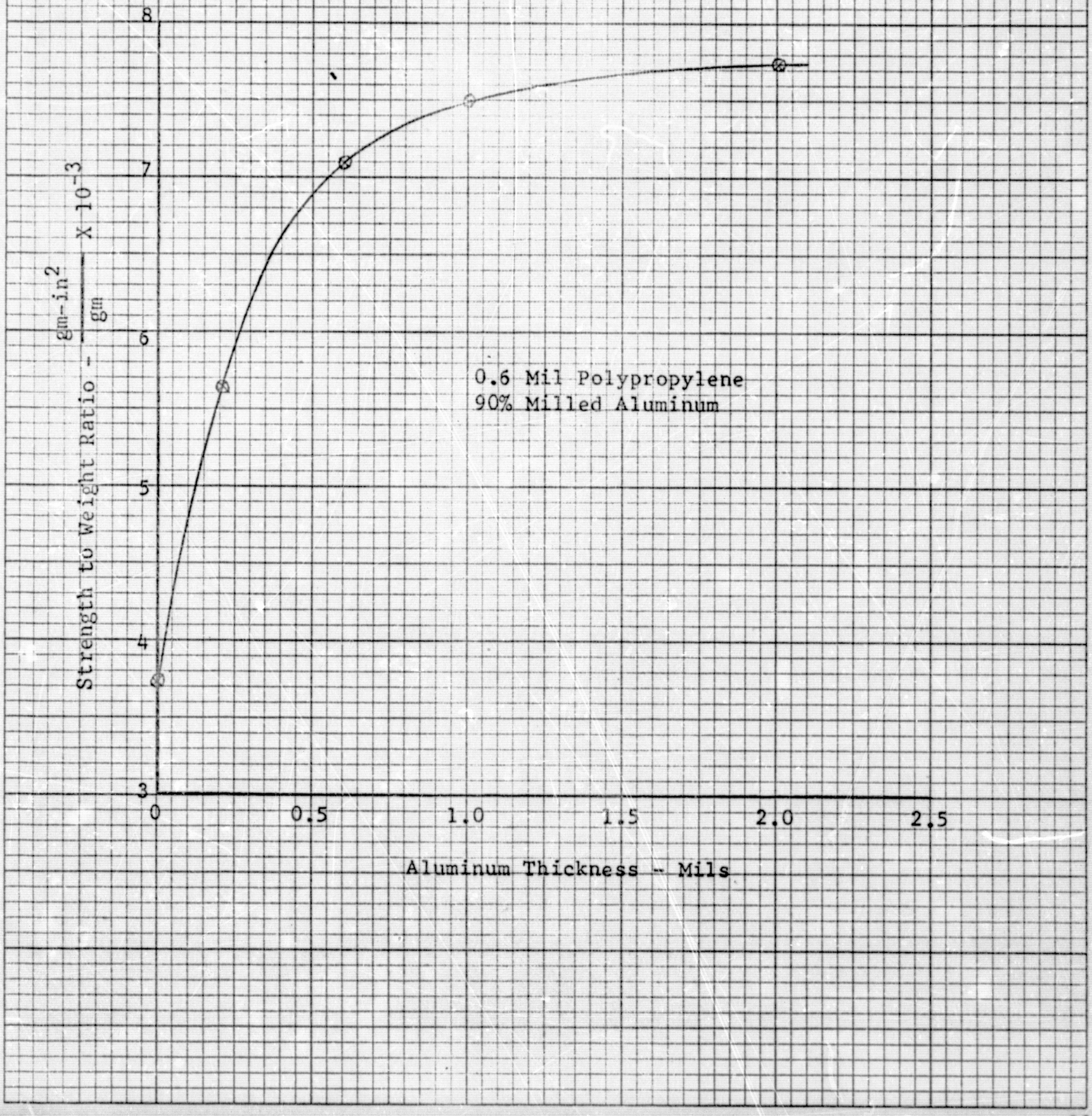
0 0.5 1.0 1.5 2.0 2.5

Aluminum Thickness - Mils

BUCKLING LOAD RATIO
MILLED MATERIAL COMPARED TO
NON-MILLED MATERIAL
COLUMN TESTS



STRENGTH TO WEIGHT RATIOS
90 PERCENT MILLED LAMINATES



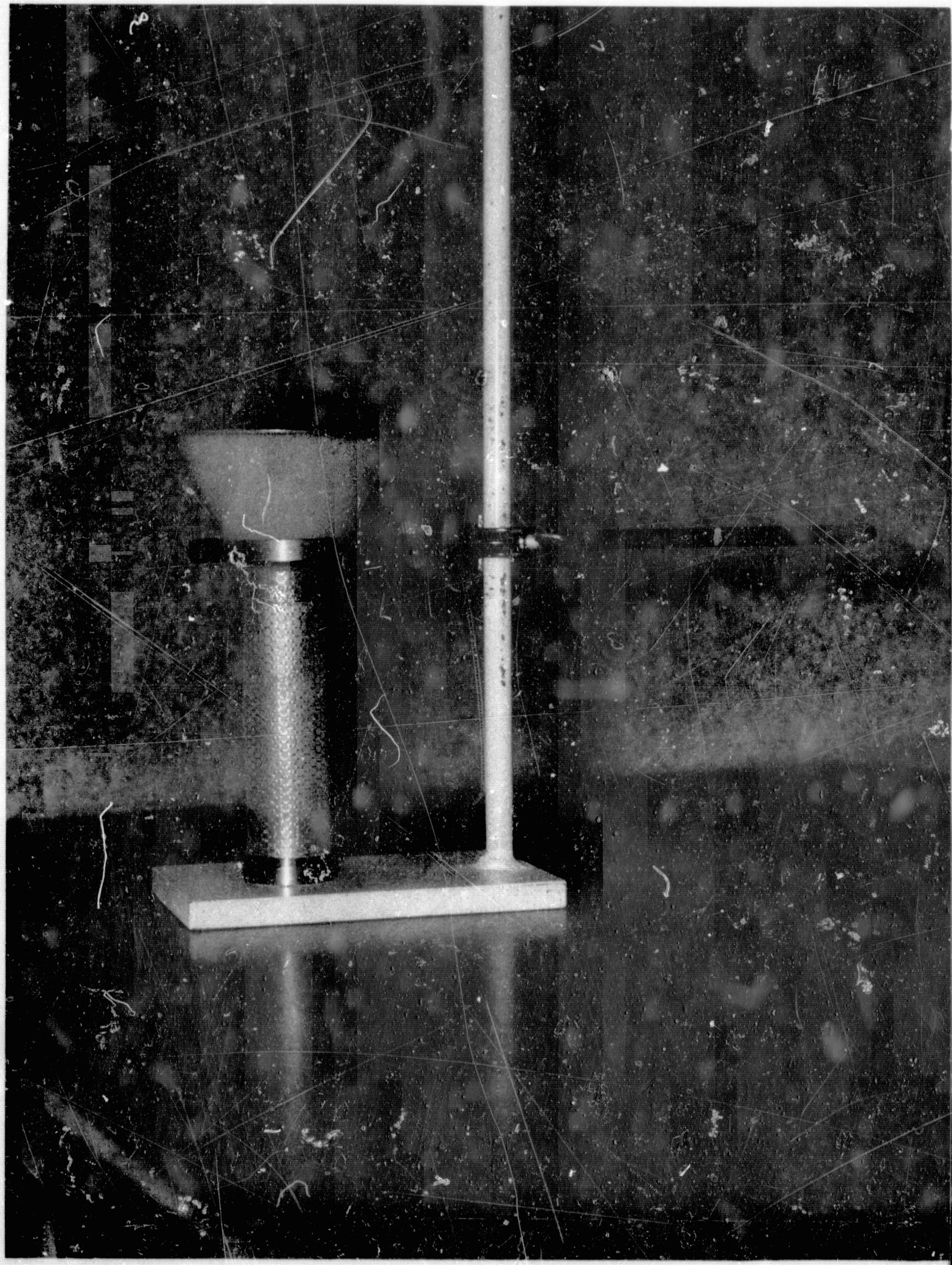


FIGURE 37 -COLUMN LOADING TEST FIXTURE

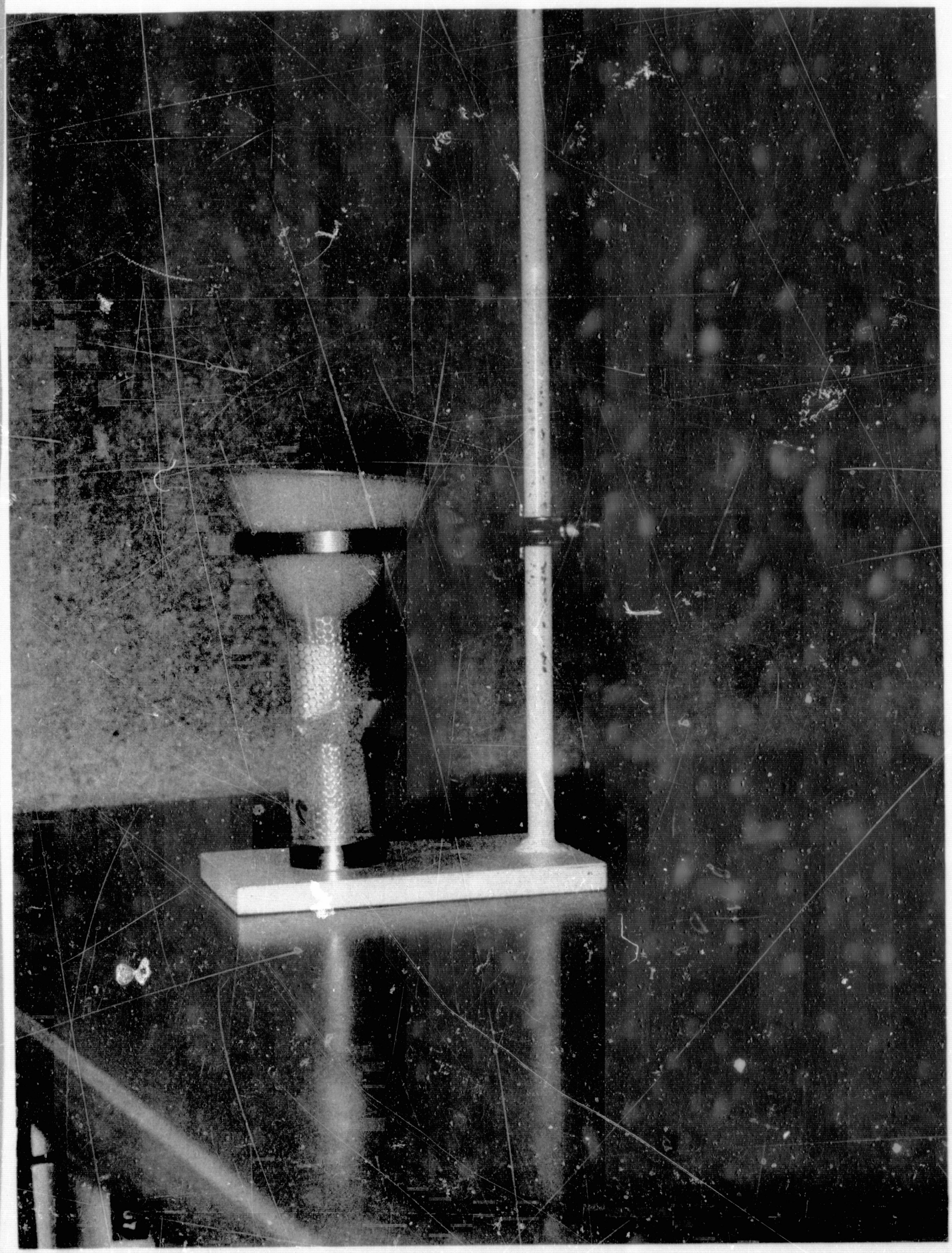


FIGURE 38 - TYPICAL BUCKLED COLUMN

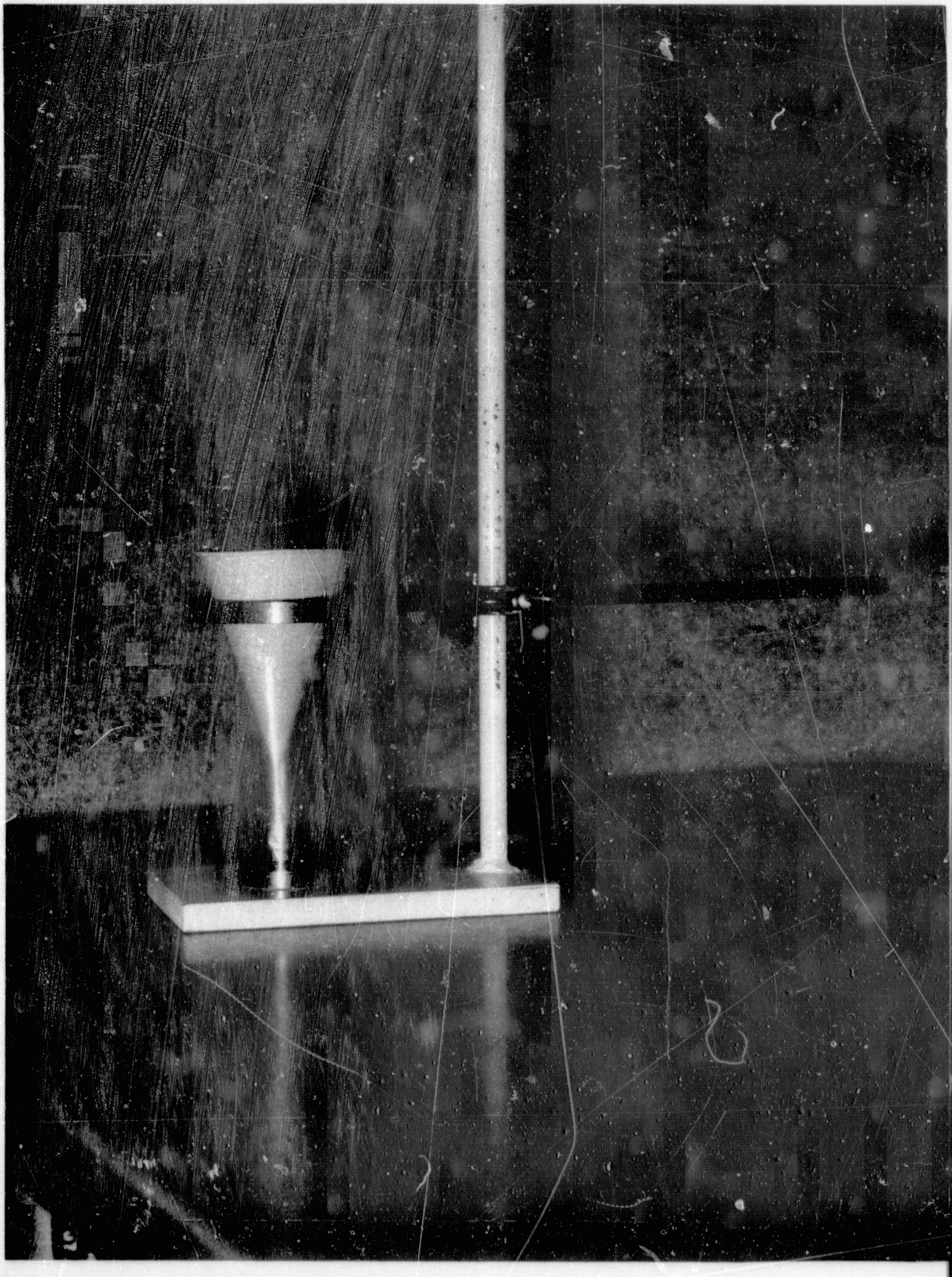


FIGURE 39 - POLYPROPYLENE (0.6 mil) COLUNN PRIOR TO LOADING

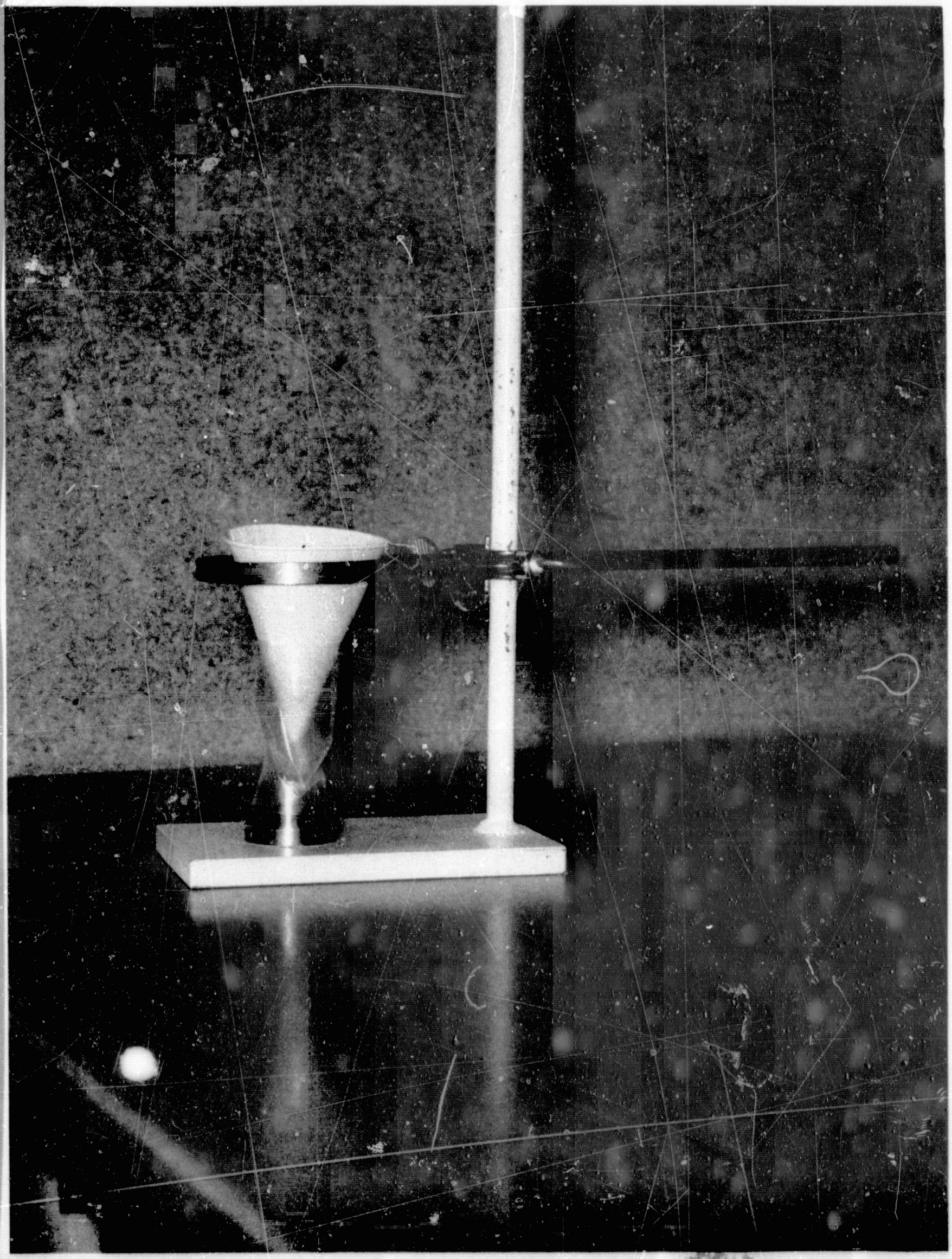


FIGURE 40 - POLYPROPYLENE COLUMN AFTER LOADING

the value should be approximately that shown by the adjusted curve.

Figure 36 shows the strength-to-weight ratios for the materials tested. The curve shows that thin foils are not efficient. It indicates that thin plastics and thick foils are the most desirable.

The same relations hold true for the 48 percent milled material. A laminate with 1.0 mil aluminum has a strength-to-weight ratio of 9.2 X 10^3 gm.in^2/gm while a laminate with 0.6 mil aluminum has a strength-to-weight ratio of 7 X 10^3 gm.in^2/gm.

E. Conclusions

1. As the fraction of aluminum removed is increased, the laminate strength decreases at a high rate.
2. As the thickness of aluminum is decreased the laminate strength decreases rapidly.
3. Heat treating the polypropylene has no significant effect on the material strength.
4. Because of high absolute strengths and high strength-to-weight ratios, thick foils and thin plastics are the most suitable for the proposed space application.

XII. SPHERE BUCKLING TESTS

A. Objective

The purpose of the test program was to measure the pressure required to cause deformation or buckling of a thin-shell sphere subjected to uniform external pressure. Chemically milled material was tested as well as Echo A-12 material which was used as the reference material.

B. Test Description

A sphere 30 inches in diameter with 18 gores, and 1/2 inch wide sealing tapes was fabricated from Echo A-12 material and chemically milled material. Two outlets were installed in the sphere. One outlet opened directly into the sphere, the other extended to the center of the sphere. The purpose of the two outlets was to allow pressure measurements to be made some distance from the inflation inlet.

The sphere was placed in a 24-inch inside diameter ring with the purpose of preventing deformation of the sphere at its support (see Figures 41 and 42). This left the upper 4/5 of the sphere unsupported and available for observation.

During testing, the sphere was partially inflated with helium to offset the material weight in the upper half of the sphere. The sphere was then pressurized with a small, low volume, low pressure pump. At a predetermined stress level pressurization was stopped and the sphere was slowly returned

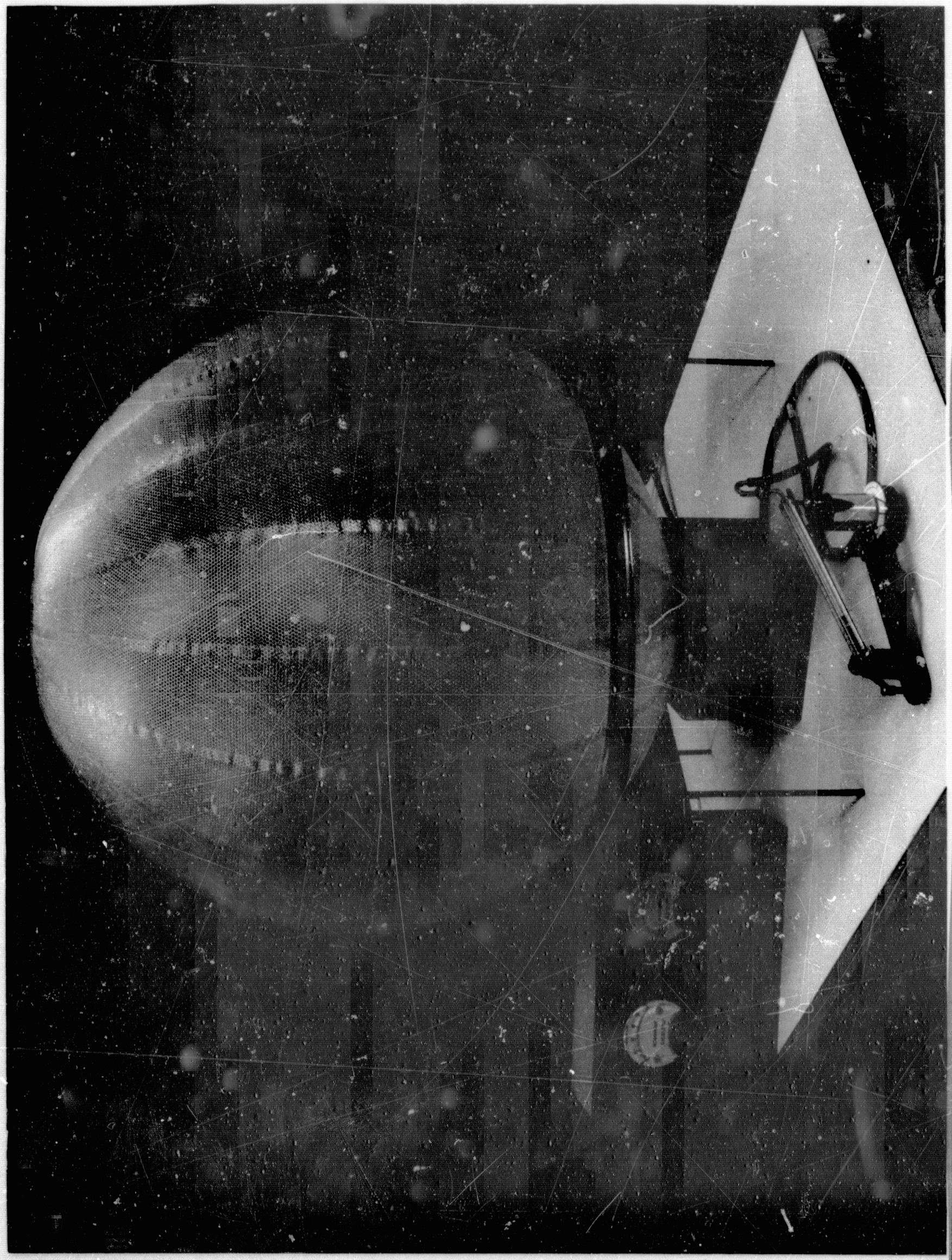


FIGURE 41 - 30 INCH SPHERE TEST FIXTURE - SPHERE WITH SLIGHT SUPERPRESSURE

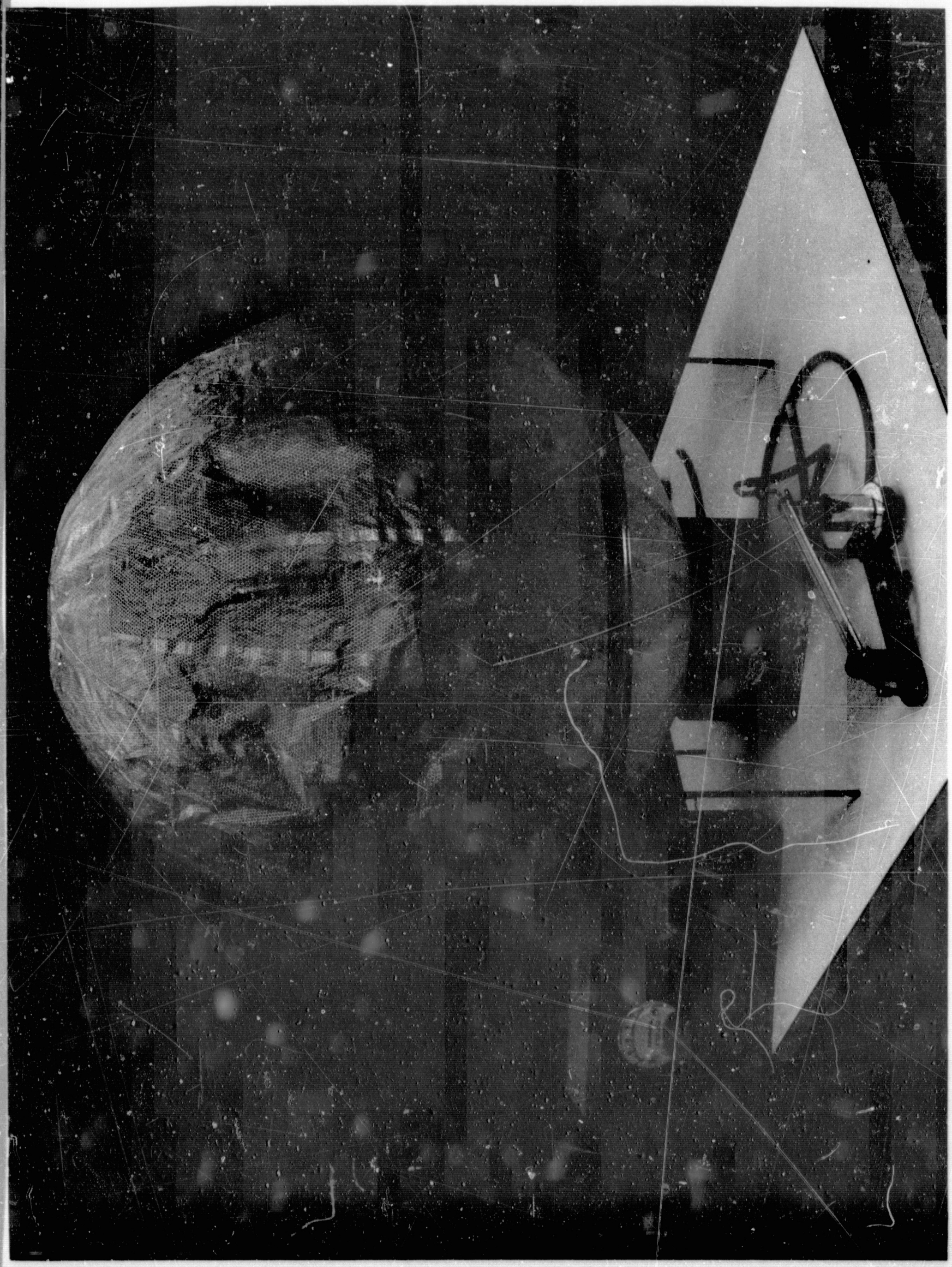


FIGURE 42 - 30 INCH SPHERE AFTER BUCKLING TEST

to zero differential pressure.

After equilibrium was reached, the sphere was slowly evacuated. During evacuation, the differential pressure was monitored with an inclined manometer capable of measuring pressure increments of 0.001 inches water. Visual observations were made continuously during the evacuation.

C. Test Apparatus

1. Helium
2. Air Pump (Vacuum and pressure)
3. Inclined Manometer (0.001 inch water sensitivity)
4. Pressure
5. Camera

D. Results

1. Table V, Echo A-12 Material (Sequential tests on one sphere).

Pressure difference (dP) in inches of water

Prestress*	Dimples (Negative Pressure)	Buckles** (Negative Pressure)
10 (.27)	0.028	0.048
3 (.08)	0.030	0.040
5 (.13)	0.020	0.025
12 (.32)	0.038	0.048
18 (.48)	0.040	0.050

*Values in parenthesis are stress levels in lb/in of material width.

**Also maximum differential pressure reached.

2. Table VI, Chemically milled (70 percent) material 1.0 mil aluminum, 0.6 mil polypropylene

Pressure difference in inches of water

Prestress*	Dimples**	Buckles*** (Negative Pressure)
0.5 (.013)		0.007
1.0 (.027)		0.007
1.5 (.040)		0.005
2.0 (.054)		0.006
3.5 (.094)		0.007
5.5 (.148)		0.006
7.5 (.202)		0.006

*Values in parenthesis are stress levels in lb/in of material width.

**Not able to determine or observe difference between dimples or buckles.

***Also maximum pressure reached.

E. Discussion

Observation of sphere buckling must be defined to some extent because the surface, after stress relieving, is not smooth but dimpled. The surface irregularities are a result of the number of tapes (18) required for sphericity. Since the tape area is a double thickness of material, the tape stress is only 1/2 the gore stress. Therefore, a high internal pressure is required before the foil in the tapes is strain-hardened. At pressures required for gore strain the tapes are in an elastic state and with subsequent stress relief the tapes probably return to zero stress while the gore does not and dimples form.

During the reversal (evacuation) of pressure with Echo A-12 material small deformations about 2 inches in diameter appeared first. With continued evacuation larger deformations would appear which were 5 to 6 inches in diameter. At this point, the maximum negative internal pressure would be attained. Further evacuation only caused more deformations to appear.

The data in Table V show that strain-hardening of the material is effective. The first test pressure was adequate for strain-hardening the foil, but the second and third were not. The buckling pressure differentials are successively lower in the second and third tests than in the first. When the material was again strain-hardened, by exceeding the previous

maximum stress, the buckling pressure was again the same as for the first test.

Dimples were not noticeable during the testing of the chemically milled material. The manner of surface deformation was different from the Echo A-12 material in that the gores would flatten, then buckle inward in an elliptical form approximately 5 inches by 6 inches.

Test results for a chemically milled material are shown in Table VI. The material tested was a 1.0 mil aluminum foil 70 percent milled, laminated to a 0.6 mil polypropylene. No difference in buckling pressure as a function of prestress was noted; probably because of the small cross section of aluminum.

In all tests, the 24 inch diameter ring provided adequate support for the sphere. In most cases the buckles were likely to form at any point within the middle two-thirds of the sphere where the gores were not reinforced by the tapes.

XIII. SOLAR DISTORTION OF SPHERES

A. Objective

The purpose of this section of the test program was to analyze the extent of shrinkage that a satellite balloon would experience during orbiting. The shrinkage could lead to distortion of the spherical shape and thereby diminish the buckling strength of the sphere. The radio frequency

energy reflection characteristics of the sphere could also be deteriorated by such shrinkage.

B. Analysis

NOMENCLATURE

C_c	Semi circumference of circle (in)
C_E	Semi circumference of Ellipse (in)
p%	Percent of shrinkage (percent)
λ	Ratio of smaller axis to larger axis of a Ellipse (in)
a	Radius of sphere and larger axis of Ellipse (in)
b	Smaller axis of Ellipse (in)
δ	Flattening of Sphere (b-a)
φ	Polar angle & parameter (deg)
Δs	Magnitude of shrinkage (in)
$\Delta c = C_c - C_E$	Difference in arc-length between circle and Ellipse (in)
H_s	Hot spot radius (in)
ρ	Radius of curvature of deformed body (in)

It is readily apparent that the parts of the balloon directly opposite the sun will be affected to the greatest extent by the solar energy. The assumption of uniform shrinkage over the hemisphere with attendent uniform shrinkage of the sphere and no distortion must be excluded as a possibility. Actually, the shrinkage will be non-uniform

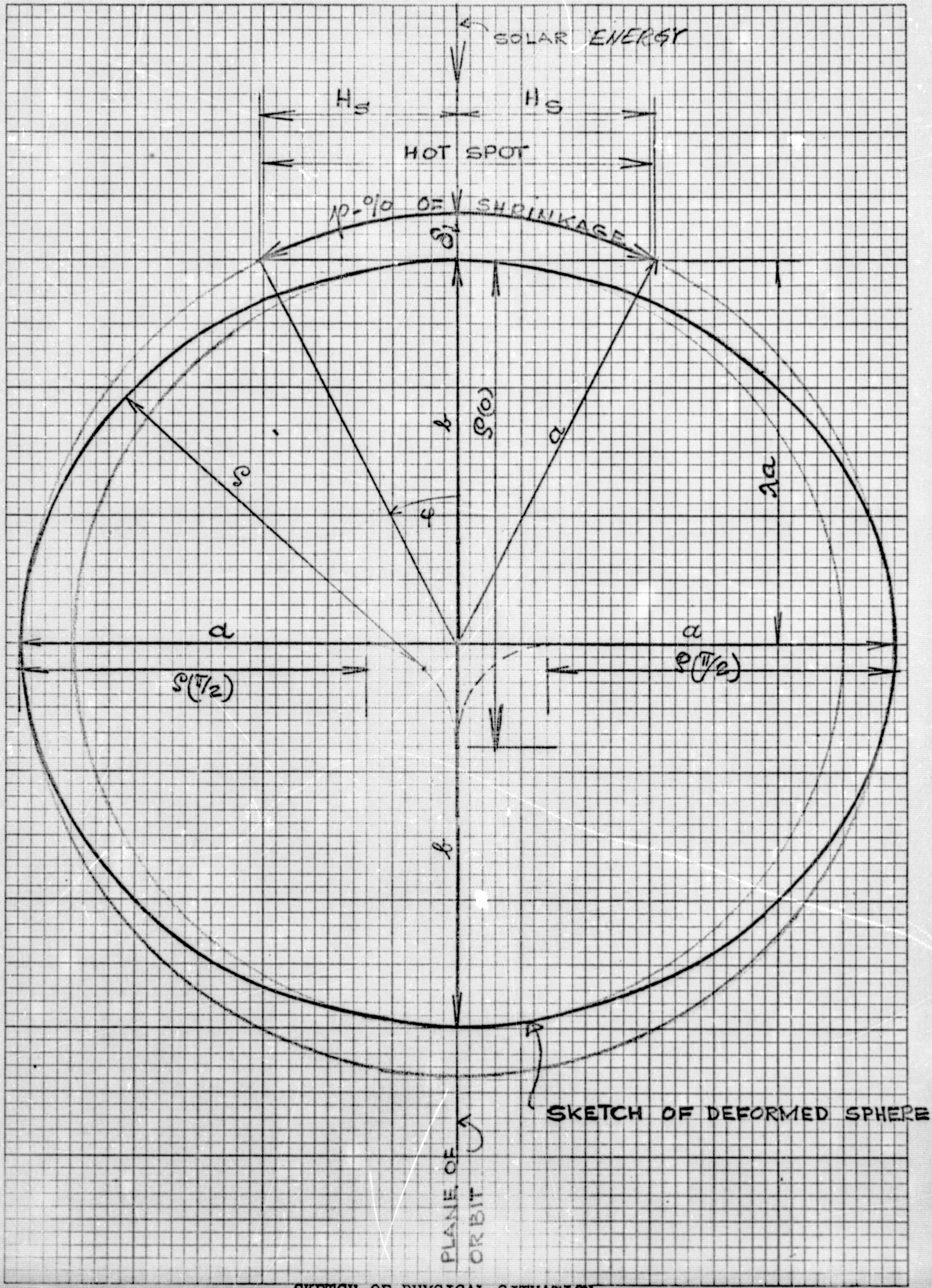
and will be governed by the angle between the normal to the sphere's surface and the solar energy impinging locally. This means that maximum shrinkage occurs at that point which is closest to the sun and minimum shrinkage occurs at the great circle of the sphere which is normal to the direction of propagation of the solar energy. For the sake of analysis, all shrinkage will be assumed to be uniform.

The approach taken to this analysis is that the surface of the balloon perpendicular to the plane of the orbit will be flattened. Mathematically speaking, the assumption of an affine transformation of a circle will be used. By making this assumption, the uniform hot-spot shrinkage may be "spread out" over the full hemisphere so that maximum and minimum shrinkages occur where expected. That is, the maximum shrinkage will be placed at the nearest point of the sphere and the minimum shrinkage will be placed at the great circle on the sphere which is normal to the direction of propagation of solar energy.

Referring to Figure 43 an analysis of the shrinkage may be made. The area of shrinkage shown is the hot-spot which is anticipated in a sphere orbiting in space. This section of the sphere will shrink p percent and is related to the polar angle φ as follows:

Length of semi-circle = $C_c = \pi a$

Length of arc = $2 a \varphi$



SKETCH OF PHYSICAL SITUATION

p % Shrinkage of this arc = $\frac{p}{100}$ 2 aφ = Δ s

$C_c - \Delta s$ = Length of semi-ellipse = C_E

$C_E = a\left[E\ \sqrt{1-\lambda^2},\ \pi/2\right]$ (Elliptical Integral of the second kind.)

The equation of the ellipse in parametric form is:

$X = -a \sin \varphi$ $\quad Y = \lambda a \cos \varphi$ $\quad b = \lambda a$

For $\varphi = 0$ $\quad Y = b$

For $\varphi = \pi/2$ $\quad X = a$

Given the foregoing relation, a more simple and accurate computation can be made by reversing the problem. Assuming that the radius of this sphere, a, always remains the length of the semi-axis of the deformed Ellipse, then the other axis of the ellipse will be called b.

Equations for affine transformations representing this situation are:

$\lambda = b/a$ $\quad \delta = a - b$ = Deformation of Circle

λ is the ratio by which the ordinate of the circle will be reduced.

$\delta = a - \lambda a = a\ (1-\lambda)$

$\cos \varphi = \frac{a\lambda}{a} = \lambda$ $\quad \varphi$ = ARC COS (λ)

S = a sine φ

$C_E = a\left[E\ (\sqrt{1-\lambda^2},\ \pi/2\right]$

This expression represents the arc-length of the semi-ellipse and can be computed by using the elliptic integral of second kind.

For geometric compatibility

$$C_c - C_E = D = \frac{P}{S} = p\,\frac{2a}{100}$$

This condition is fulfilled only for certain P versus Q and for certain values of the affine transformation λ. Calculations to determine the conditions of geometric compatibility are shown in Table VII and first approximations to compatibility are circled. A further graphical interpolation was made and the results are shown in Figure 44 as the relationship between spere flattening and the percent shrinkage expected.

TABLE VII

SPHERE SHRINKAGE CALCULATION

	1 Degree	2 Radians	3 --- Sin	4 --- Cos	5 H	6 2 a	7 b = a	8 = a - b	9 C_E	10 O	11 S For 1%	12 S For 2%	13 S For 3%	14 S For 4%	15 S For 5%
1	1	.0175	.01745	.99985	114.13	228.27	809.88	.10	2544.53	.17	.283	.5655	.8482	1.1309	
2	2	.0349	.03490	.99939	28.27	56.55	809.50	.50	2543.88	.82	.566	1.1310	1.6964	2.2618	
3	3	.0524	.05234	.99863	42.40	84.82	808.90	1.10	2542.91	1.79	.8482	1.16965	2.5446	3.3927	
4	4	.0698	.06976	.99756	56.51	113.09	808.0	2.00	2541.62	3.08	1.1309	2.262	3.3928	4.5236	
5	5	.0873	.08716	.99619	70.60	141.37	806.9	3.10	2539.84	4.86		2.827	4.2410	5.6545	
6	6	.1047	.10453	.99452	84.67	169.64	805.6	4.40	2537.73	6.97		5.937	5.089	6.7854	8.481
7	7	.1222	.12187	.99255	98.71	197.91	804.0	6.00	2535.30	9.40			7.916	7.9160	9.896
8	8	.1396	.13917	.99027	112.73	226.19	802.1	7.90	2532.38	12.32				9.048	11.309
9	9	.1571	.15643	.98769	126.71	254.46	800.0	10.00	2528.98	15.72					12.723
10	10	.1745	.17365	.98481	140.66	282.73	797.70	12.30	2525.42	19.28					14.100
11	11	.1920	.19081	.98161	154.56	311.01	795.10	14.90	2521.37	23.33					
12	12	.2094	.20791	.97815	168.41	339.28	792.30	17.70	2517.00	27.70					
13	13	.2269	.22495	.97437	182.21	367.55	789.20	20.80	2512.13	32.57					
14	14	.2443	.24192	.97030	195.96	395.83	785.90	24.10	2507.11	37.59					
15	15	.2618	.25882	965.93	209.64	424.10	782.40	27.60	2501.60	43.10					
16	16	.2793	.27564	.96126	223.27	452.37	778.60	31.40	2495.60	49.10					
17	17	.2967	.29237	.95630	236.82	480.65	774.60	35.40	2489.45	55.25					
18	18	.3142	.30902	.95106	250.31	508.92	770.4	39.60	2482.81	61.89					
19	19	.3316	.32557	.94552	263.71	537.19	765.9	44.10	2475.84	68.86					
20	20	.3491	c34202	.93969	277.04	566.47	761.1	48.90	2468.56	76.14					

NOTE: Col. 10 shows the difference between $a - C_E = D$
a = 67.50 ft. = 810 inch.
ax = 2544.7 inches = C_c
Closed framed numbers are the values at which S D.
S = D condition of geometric compatibility
Columns 5 through 20 in inches.

TABLE VII

SPHERE SHRINKAGE CALCULATION

7	8	9	10	11 S For 1%	12 S For 2%	13 S For 3%	14 S For 4%	15 S For 5%
b = a	= a - b	C_E	O					
809.88	.10	2544.53	.17	.283	.5655	.8482	1.1309	
809.50	.50	2543.88	.82	.566	1.1310	1.6964	2.2618	
808.90	1.10	2542.91	1.79	.8482	1.16965	2.5446	3.3927	
808.0	2.00	2541.62	3.08	1.1309	2.262	3.3928	4.5236	
806.9	3.10	2539.84	4.86		2.827	4.2410	5.6545	
805.6	4.40	2537.73	6.97		5.937	5.089	6.7854	8.481
804.0	6.00	2535.30	9.40			7.916	7.9160	9.896
802.1	7.90	2532.38	12.32				9.048	11.309
800.0	10.00	2528.98	15.72					12.723
797.70	12.30	2525.42	19.28					14.100
795.10	14.90	2521.37	23.33					
792.30	17.70	2517.00	27.70					
789.20	20.80	2512.13	32.57					
785.90	24.10	2507.11	37.59					
782.40	27.60	2501.60	43.10					
778.60	31.40	2495.60	49.10					
774.60	35.40	2489.45	55.25					
770.4	39.60	2482.81	61.89					
765.9	44.10	2475.84	68.86					
761.1	48.90	2468.56	76.14					

D

S D.

TABLE VII

SPHERE SHRINKAGE CALCULATION

9	10	11	12	13	14	15	16	17	18	19	20
C_E	0	S For 1%	S For 2%	S For 3%	S For 4%	S For 5%	S For 6%	S For 7%	S For 8%	S For 9%	S For 10%
644.53	.17	.283	.5655	.8482	1.1309						2.827
643.88	.82	.566	1.1310	1.6964	2.2618						5.655
642.91	1.79	.8482	1.16965	2.5446	3.3927						8.482
641.62	3.08	1.1309	2.262	3.3928	4.5236						11.309
639.84	4.86		2.827	4.2410	5.6545						14.14
637.73	6.97		5.937	5.089	6.7854	8.481					16.96
635.30	9.40			7.916	7.9160	9.896	11.87				19.79
632.38	12.32				9.048	11.309	13.571				22.62
628.98	15.72					12.723	15.268	17.81			25.45
625.42	19.28					14.100	16.964	19.791	22.618		28.27
621.37	23.33						18.660	21.77	24.881		31.10
617.00	27.70							23.750	27.142	30.535	33.93
612.13	32.57							25.730	29.400	33.079	36.76
607.11	37.59								31.670	35.625	39.58
601.60	43.10									38.170	42.41
495.60	49.10									40.710	45.74
489.45	55.25										48.07
482.81	61.89										50.90
475.84	68.86										53.72
468.56	76.14										56.65

18 S For 8%	19 S For 9%	20 S For 10%
		2.827
		5.655
		8.482
		11.309
		14.14
		16.96
		19.79
		22.62
		25.45
22.618		28.27
24.881		31.10
27.142	30.535	33.93
29.400	33.079	36.76
31.670	35.625	39.58
	38.170	42.41
	40.710	45.74
		48.07
		50.90
		53.72
		56.65

SPHERE FLATTENING AS A FUNCTION
OF PERCENT SHRINKAGE

P	δ
0	0
1	.25
2	.96
3	1.21
4	2.85
5	6.61

