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UNION CARBIDE CORPORATION

OLEFINS DIVISION

Research and Development Department
South Charleston 3, West Virginia

MONTHLY REPORT

Subject: Program III - New Business Hydrocarbon Resins
Evaluation of Soft Hydrocarbon Resins as Binders for Coalinga Asbestos

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Summary

Material in the 3100-3900 psi range of flexural strength which has possible applications in the building industry has been produced by pressing and curing blends of soft hydrocarbon resins and Coalinga asbestos. With both soft resins evaluated, U-311 Prepolymer and one produced from "C" Cut of Unsaturate Oil, better results were obtained when blends were used containing less than two parts of asbestos to one of resin. Forming pressures in the 1500-2600 psi range generally made stronger material than did 1000 psi. Curing for 120 hours gave only 25% better strength than a 10-hour cure. No significant loss of strength resulted from 3-day water-immersion of cured material.

Pressing produced slightly better material than extrusion, for 3 of 4 blends compared. Both methods gave values above 3600 psi; the high value obtained by extrusion was with a 1:1 blend of U-311, and the high value by pressing was with a 1:1 blend of "C" Cut soft resin.

The various blends tested required different pressures to begin extruding; strength improvement of up to 57% for one blend and less for others was obtained by extruding at pressures 15 to 70% above the minimum pressure for extrusion. Elevated temperatures were definitely less satisfactory than room temperature for extrusion. Slightly better results were obtained by double extrusion than by single, by oven-drying of asbestos before mixing, and by more thorough mixing.

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Introduction

An earlier monthly report[1] described the initial work done in the investigation of soft hydrocarbon resins from Unsaturate Oil (including Prepolymers[2]) as binding agents for Coalinga asbestos. The object is to find a relatively low-cost binder which might be substituted for some applications for trimethylolphenol, the 16 cent/pound resin now used by the U. C. Nuclear Division in combination with its asbestos to produce F-100 building material. F-100 can be extruded in the form of pipe or extruded and calendered into sheets for use as building panels. It has superior flexural strength (~11,000 psi). We do not anticipate that our hydrocarbon resins would compete with F-100 in uses where maximum strength is essential. There are, however, probable applications in the building business for a material with a flexural strength of 3000-5000 psi if it can be produced at enough lower cost than F-100 and at lower cost than currently used materials. Examples are "asphalt" floor tile, backing for vinyl floor tile, and insulation board used in the walls of frame houses.

In the early work with our hydrocarbon resins, it was found that three of them produced materials which tested above 2500 psi in flexural strength when blended in 2:1 ratio of asbestos:resin[1]. In these experiments the test specimens were formed by pressing the blends at about 1000 psi in a laboratory press to a thickness of 1/8" and then cutting strips 1/4" wide. Then the test strips made from soft resins were thermopolymerized by baking at temperatures of 150° to 250°C for periods of 2 to 64 hours. The cured strips were finally evaluated as to flexural strength and modulus by measurements on an Instron Testing Instrument followed by appropriate calculations, as described in the earlier report.

Continuing work with soft resins[3] has mostly employed extrusion rather than pressing in an attempt to produce improved material. This approach was based on the experience at the Nuclear Division's Sterling Forest Laboratory with several binders evaluated there.

Additional Work on Pressed Material

Additional Curing Experiments

It had already been demonstrated that strength increases with curing time of up to 64 hours in air[1]. More recent experiments involving the materials made from the two types of soft resin have shown that 120-hour curing yields a small additional strength increase. The two materials showed increases of 27.5% and 25.0% for 120 hours over 10 hours, but most of this appears to have been gained by 64 hours, and probably well before this point (Table I). In fact, it seems clear from the rapid increase in strength with curing time up to 10 hours and the much slower increase up to the 64- and 120-hour points that a marked tendency to begin levelling off in strength must occur by 16 to 24 hours. There seemed to be no point in

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[1] 855-2307-6275, May 31, 1963, "Investigation of Hydrocarbon Resins as Binding Agents for Coalinga Asbestos".

[2] For convenience and brevity, in this work the term "soft hydrocarbon resins" is used broadly, to include our Prepolymers.

[3] Evaluation of selected hard hydrocarbon resins has also been made. This work is discussed in a report which follows.

getting further data toward plotting such a curve, since there would be a slightly different one for each particular material. However, any soft resin from Unsaturate Oil could be expected to attain probably 80-90% of its maximum strength in a cure time of less than 24 hours[4]. Modulus values (a measure of rigidity) also tended to go up, but the pattern is not very consistent.

Another point of investigation has shown that approximately a 25% loss in strength resulted from storing test strips exposed to the atmosphere for 18 days before curing (Table I). This is believed to have resulted from the evaporation of some of the more volatile components.

This is consistent with an observation noted earlier[1] that stronger material is produced by curing at 200°C preceded by 4 hours at 150° than by curing at 250° preceded by 4 hours divided between 150° and 200°. Still further support for this explanation is provided by data from Run 1930-86-N6. In the earlier report the flexural strength is given as 2170 psi; this is the average of five results. Actually, it has been subsequently noted that the specimens involved were cured (nitrogen atmosphere) at two separate times and can be denoted an A and a B set. The B set - two results averaging 3000 psi - was cured as planned: 4 hours at 200°C and 6 hours at 250°. In the case of the A set, the oven temperature was unintentionally allowed to get above 240° for 11 minutes early in the initial curing period, and the resulting three tests averaged only 1620 psi. (The modulus value was not affected much: 1,090,000 psi for B and 920,000 for A.)

Effect of Forming Pressure

In the work reported earlier, nearly all the pressed specimens had been compacted at a pressure of about 700 to 1000 psi. After the extrusion experiments had revealed a rather noticeable effect of pressure on strength of specimens produced, limited experiments on two blends were run with pressed material. These were a 2:1 blend of asbestos: "C" Cut resin (1961-78-1 and -7 thru -11) and a 1.5:1 blend of U-311 (1961-78-4 thru -6).

As can be seen from the data on these runs (Table II), pressures above 1000 psi produced stronger specimens in the case of both materials. With 1.5:1 U-311, strength apparently continued to increase up to the highest pressure used, 2600 psi. However, in the case of 2:1 "C" Cut resin, where a larger number of pressures were tried in the 1000-2600 psi range, strength was noticeably improved at 1450 and 1740 psi over 1000; but then it dropped off sharply for 2050 and 2350 psi, and appeared to be better again at 2600 psi. Flexural modulus values tended to vary in the same direction as strength values, but generally seemed to be less affected by forming pressure.

In this connection it should be noted that the very first test strips made (1/16" thick, out of 2:1 asbestos: U-311) had been pressed at 3000 psi;

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[1] 855-2307-6275, May 31, 1963, "Investigation of Hydrocarbon Resins as Binding Agents for Coalinga Asbestos".

[4] Except for a very few experiments which are noted otherwise, all the rest of our work with blends of asbestos and soft resins has employed 4 hours' baking at 150°C followed by 10 hours at 200° as "standard" curing conditions.

and their strength after a 10-hour cure tested 4120 psi, in comparison with 2630 psi for the best such material pressed at 1000 psi (1/8" thick then and since, for testing reasons)[1] . Because the Sterling Forest experience with their type of material had been that higher compacting pressures were detrimental to strength of product, we had decided to standardize our pressing conditions at 1000 psi. We also concluded that the 3000-psi specimens were stronger because, being only half as thick, they had experienced greater oxidation during curing. Now we believe the evidence indicates that the greater strength of these earliest strips was due more to the higher forming pressure.

Results with Different Blends in Pressed Specimens

Earlier work with pressed material had employed only 2:1 blends of asbestos:resin. After the extrusion program showed improved strength for 1.5:1 and 1:1 blends of asbestos: U-311 Prepolymer, some 1.5:1 specimens of U-311 and some 1:1 of both U-311 and "C" Cut soft resin were formed by pressing. Only 1000 psi was used for the latter two blends, but 1000 to 2600 psi for the 1.5:1 U-311, as reported above. Considering only the specimens made at 1000 psi (Tables XIII, XIV, XV) two comparisons can be made. In the case of U-311, flexural strength was improved as asbestos was increased: 2240, 2470, and 2630 psi for 1:1, 1.5:1, and 2:1. However, in the case of the "C" Cut resin, the flexural strength for 1:1 was much greater than for 2:1, 3920 psi over 2610.

As in the case of the pressure variable, the effect of varying the proportions of asbestos to resin has not been investigated thoroughly. However, it appears that further improvement can be expected if further work with soft resins as binders for asbestos is indicated.

Effect of Exposure to Water

Exposure to water does not appear to affect the structure of cured asbestos-hydrocarbon resin blends or to be very detrimental to their flexural strength and modulus, on the basis of limited tests. Five representative types of pressed mixtures showed an average loss 22% in strength and an average decrease of 5% in modulus as a result of 70 hours' immersion in water at room temperature (Table III). Three of these types of material, when allowed to dry in air for four hours following the three-day water immersion, recovered to within 10% of their original strength on the average. A fourth tested 6% stronger than originally, but the result was not included in the average because it was based on a single break. Flexural modulus did not seem to change much upon the re-drying.

It was found that one of the above types, the 1:1 blend of asbestos U-311, absorbed 1.6% of its weight in water during the three-day immersion. After having been allowed to dry in air for two days, it had lost half of this water and was back to 100.8% of its original weight. In a separate 6-hour water-immersion test, the same type of material was found to have a weight gain of 0.3%.

[1] 855-2307-6275, May 31, 1963, "Investigation of Hydrocarbon Resins as Binding Agents for Coalinga Asbestos".

Extrusion Experiments

Extrusion Equipment

The extrusion equipment consists essentially of a 5-inch-tall stainless steel cylinder with an inside diameter of ~2-1/8 inches in which a 5-1/2-inch-tall solid brass piston is pressed downward by the use of a small hydraulic press (Carver Laboratory Press, Model B). The cylinder, which has a wall thickness of 15/16 inches, is open at top and bottom and has a 1" threaded side opening centered ~7/8 inch above the bottom. Either a solid plug or a brass extrusion nozzle can be screwed into this side opening. A 1/2"-thick solid brass disk closes the bottom opening for extrusion out the side; or a similar disk with a 1-inch threaded hole can be used and the cylinder so placed in the press that downward extrusion is possible. There is a thermocouple well the length of the cylinder, parallel to and 3/8" from the inside wall. An electric heating jacket can be placed around the cylinder.

To date two brass extrusion nozzles have been used. One has a rectangular opening 1/8" by 1/4"; the other has a round opening 0.218" in diameter, so that its cross section is slightly larger than that of the other nozzle. The "land" (straight section of the nozzle opening) is about 1/4" deep, and back of this the opening is tapered out for about an inch on all sides at a 20-30° angle. A mold was made so that round strips could be pressed into the same cross-sectional shape and dimensions as material extruded through the rectangular opening. This equipment consists of two 11/16" x 4" x 6-1/2" solid stainless steel plates which can be bolted together and an insert plate 1/8" thick with four 1/4" x 5" slots cut out.

Physical Property Measurements - Rod-Shaped Test Pieces

As mentioned in the Introduction, flexural strength and modulus in our initial work were based on test results on an Instron Testing Instrument, with calculations made according to standard formulas[1]. These formulas, however, apply to flat test specimens of rectangular cross section.

In the course of later work, described in this current report, some rod-shaped specimens were extruded because of certain advantages in using a round nozzle. However, no standard formulas for this shape of test specimen could be found, either in appropriate reference books or by consultation with several qualified persons.

Apparently there are good reasons why cylindrical test specimens are not normally used. In a flexural strength test, it is the thin bottom layer of the test piece which largely determines at what load the piece will snap. When the cross section of the beam (or strip) being tested is round rather than flat, the critical bottom layer is a much smaller fraction of the whole piece;

[1] 855-2307-6275, May 31, 1963, "Investigation of Hydrocarbon Resins as Binding Agents for Coalinga Asbestos".

therefore any lack of uniformity in strength which may occur in this layer will be exaggerated by the calculation, and there is likely to be wider scatter in results. (As a matter of fact, wide scatter did show up in the tests made on our cylindrical test specimens).

So that our rod-shaped material could be evaluated, however, tests and calculations were made. We used an equation for flexural strength (psi) derived by Mr. J. V. Petrone of Chemicals R and D Department:

$$S = \frac{P_2\, l}{\pi r^3}$$

where P_2 = force at break, pounds
l = length of span, inches
r = radius of rod, inches.

An equation was used for flexural modulus (psi) which was based on assumptions thought to be theoretically sound:

$$E = \frac{P_1\, l^3}{6\sqrt{3}\,\pi\, \delta\, r^4}$$

where P_1 = force at linear portion of stress/strain curve, pounds
δ = deflection of load P_1, inches
(l and r same as above).

Comparison of Extrusion and Pressing

Generally speaking, the extrusion of blends of soft hydrocarbon resins with asbestos did not provide the looked-for large increase in strength of final cured material over that obtained by pressing. Of four blends compared, only the 1:1 blend containing U-311 Prepolymer had better flexural strength in extruded material than in pressed. The best results obtained by extruding the other three (the 1.5:1 and 2:1 blends of U-311 and the 2:1 of the "C" Cut resin) were about 10% lower than those from pressed samples (Table IV). In all four cases the extruded material was less flexible than the pressed. The comparison between extrusion and pressing had not been thoroughly investigated. In certain areas more work should be done as applications are suggested.

On the basis of experiments done so far certain broad conclusions can be made, however. If a particular blend is too dry (too rich in asbestos) to extrude satisfactorily at a rate of several inches per minute, stronger material is produced by pressing. On the other hand, it appears that when blends do have good flow characteristics, extrusion produces essentially as good or better material. As might have been expected, a blend containing the less viscous resin ("C" Cut resin, 2:1 blend) was extruded with better results than the same blend of the more viscous one (U-311, 2:1). (Obviously, many other variables are significant; these are discussed in the following sections.)

Extrusion of Different Asbestos-Resin Blends

After the new laboratory extrusion equipment had been tested with a hand-mixed 1:1 batch of asbestos U-311, milled 2:1 blends were run using both U-311 and the "C" Cut soft resin (Tables XIV and XV, 1961-47 and -48; some of these results have been mentioned above). This material was somewhat rough along the edges unless extremely slow extrusion rates were used (<0.5 inches/minute). Also, as already stated, it did not test as strong after curing as pressed material. Elevated temperatures were next used with the 2:1 "C" Cut blend in an attempt at improvement; but, while the material extruded faster and smoother, its flexural strength was down by a factor of 2 to 4. (A fuller treatment of this 1961-51 series is in a later section).

The next approach was to try blends containing less asbestos so as to improve the flow characteristics. Two series of runs (1961-63 and -64) employed 1:1 and 1.5:1 proportions of asbestos U-311. Extrusion was again done at room temperature.

Much better results were obtained than with the drier 2:1 blend. Extrusion was faster, the material was smoother, and greatly improved flexural strength and modulus values were achieved (strength increased and modulus decreased). A summary of these results is presented in Table V.

To evaluate certain variables, some of these runs were made through a round nozzle instead of the rectangular one, and some were done in a downward direction (same direction as the piston travel) rather than out the side. The effect of these variables is discussed later, as is a comparison of single and double extrusion.

A second series of room-temperature extrusion runs was next made using a 2:1 blend containing our "C" Cut soft resin (1961-67). More specimens were produced than in the first series with this blend, since the effect of various pressures and of double extrusion was being studied. These specimens had flexural strength values of 2850 to 3330 psi (Table V); however, extrusion rates were all less than 2.2 inches a minute with a rate of 0.7 or less required to achieve really smooth samples and the best strengths.

In the course of the experiments done to compare different resin blends and methods of forming final test material, it was necessary to seek the best conditions and techniques of operation and to study the effects of several variables involved. Discussions of these studies follow.

Effect of Extrusion Pressure

It was found, as expected, that each particular blend of asbestos resin required a certain pressure in our particular equipment before it would begin extruding. What was of interest to investigate was the effect of pressure increase over this "starting" pressure on the surface characteristics of the extruded strip, on extrusion rate, and on the strength and rigidity of the final cured material.

Of primary interest is the effect of extrusion pressure on strength of product, shown graphically by the slope of the lines in Figure 1. A distinct tendency toward strength improvement is seen for the 1:1 asbestos, U-311 blend when extrusion is done above minimum pressure and rate. This is believed to be a direct effect of pressure, either by compaction or by improved wetting of asbestos by hydrocarbon, or both. The same relationship between forming pressure and strength of product has been seen to exist in the case of pressed material. For the other three blends used, however, there appears to be no significant change in strength with increasing extrusion pressure.

As might be expected, higher rates of extrusion accompany higher pressures. However, there was also a consistent tendency for the material to be less smooth (Tables XIII, XIV, and XV).

Effect of Double Extrusion

When a batch of asbestos–resin blend was extruded a second time, extrusion rate was usually somewhat faster and product strength improved by several per cent. In the case of the stiffer (2:1) blends extrusion rate for the second extrusion tended to be about the same or even slightly slower than the first pass had been at the same pressure; however, with the other blends (1.5:1 and 1:1) the rate was usually 40 to >100% faster. Strength of a cured specimen producedby double extrusion, all other conditions being the same, was nearly always improved, usually by 3 to 10 per cent. Modulus value (rigidity) was also increased, but to a slightly less degree. Comparisons made between single and double extrusion for a variety of blends and extrusion conditions are shown in Table VI. In each comparison, all other variables were the same. (When double extrusion was to be done, the first pass was made at a higher pressure and rate, with little attention to the smoothness of the material extruded).

Effect of Extrusion at Elevated Temperatures

For blends of asbestos and soft hydrocarbon resin, extrusion at room temperature produces a definitely stronger material than does elevated-temperature extrusion. Experimentation was done with only one blend, 2:1 asbestos "C" Cut resin, with strength reduction of 43 to 58 per cent in extrusions up to 105°C, as shown in Table VII. As would be expected, much lower pressures were required to accomplish extrusion at the higher temperatures. Because U-311 (as well as other prepolymers or soft resins made from Unsaturate Oil) does not differ greatly in composition from the resin used in this test, there was no basis for expecting anything but poorer performance with elevated-temperature extrusion.

Effect of Drying Asbestos Before Blending

When any material is to be used as a binder, it is important to achieve the best possible bonding to the filler material. In the case of our hydrocarbon resins, which are hydrophobic, the possibility had to be considered that adsorbed water on the asbestos might be detrimental to adhesion of the resin.

A blend of 2:1 asbestos "C" Cut resin was prepared using asbestos which had just been oven-dried at 180°C and 20 mm Hg pressure for one hour. Sets of extruded (1961-67) and of pressed (1961-78) specimens were made, some in each case under conditions which were identical with those for specimens made using non-dried asbestos. Since only two such comparisons can be made which are considered reasonably reliable (Table VIII), it is not possible to be conclusive, but there is sufficient evidence to decide that no great improvement in strength is achieved by the pre-drying. Unfortunately, certain other comparisons had to be ruled out because one or the other of two sets of samples had to be rejected as being obviously faulty (1961-67-8 and 1961-48-2, the latter rejected; 1961-67-1-64 and 1961-45-64, the former rejected). The weight loss of the asbestos during the oven-drying was 1.2%

Effect of Thorough Mixing

While no real study of mixing has been made, certain observations can be made. The brief series of runs which was made to test the extrusion equipment (1961-46) employed a hand-mixed batch of 1:1 asbestos U-311 Prepolymer. A later series (1961-63) used a milled batch of the same blend. When corresponding runs and specimens from these two series are compared (Table IX), it is seen that (1) the hand-mixed batch required a considerably lower extrusion pressure (350 to ~600 psi) than did the more-vigorously-mixed milled batch (870 to 1740 psi); but (2) milled material produced better test specimens - stronger by 4 and 10%, and more flexible by 12 and 16%.

Other Extrusion Variables Investigated

In the course of the search for a high-quality extruded asbestos-resin material, it was necessary to devise methods and equipment which would best do the job in our laboratory extruder.

Three items investigated were: (1) extrusion out the bottom instead of the side of the extruder; (2) use of a round nozzle in place of the rectangular one; and (3) use of a rectangular mold during curing, both for round and rectangular strips.

1. Downward Extrusion

It was most convenient to extrude test strips in a horizontal direction, using the laboratory extruder as designed. However, as much of the material so produced seemed to be not smooth enough, the possibility had to be considered that the flow of material was being adversely affected by a 90° change in direction (downward travel of the piston, and horizontal extrusion). A brass plug fitting the bottom of the cylinder was then threaded to accomodate the extrusion nozzles. Part of the 1961-63 series of runs and all of 1961-64 were extruded downward.

On the basis of outward appearance, there was only very slight improvement in the material, if any. On the basis of flexural strength of cured material, there was an indicated improvement of only about 5%; modulus seemed unaffected. Values are shown in Table X.

As a result of this experiment and considering that no marked improvement was achieved by downward extrusion, it was decided to use the more convenient horizontal method in later experiments.

2. Use of Round Nozzle

After several series of runs had been made employing a rectangular nozzle, a round one was fabricated in the hope of producing smoother test strips at an improved extrusion rate. Not only were corners eliminated in the round nozzle, but it was easier to have the recommended taper machined behind the "land" (straight section) in this shape of nozzle. First use of this piece of equipment with blends of asbestos and soft resins was in Runs 1961-63 and -64.

In most cases there was no noticeable improvement in the appearance of strips extruded through the round nozzle over those extruded through the rectangular one at the same pressure; in a few cases there was slight improvement. Some advantage was gained by the fact that considerably higher rates usually occurred at equal pressures (10 to 100%faster).

Evaluation of the effect of extrusion through a round nozzle on flexural strength and modulus was uncertain. As discussed earlier, in the section entitled "Physical Property Measurements - Rod-Shaped Test Pieces", no standard formulas could be found for these physical property values, but equations were derived for calculating them from the test results. However, there was wide scatter in the results obtained in our work with cylindrical test specimens, probably for the reasons discussed in the earlier section. Examination reveals frequent occurrence of quite scattered results. In the graphs in Figure 1 showing the effect of extrusion pressure on strength, every graph which involves test results on rod-shaped pieces has a noticeable departure in slope from what appears to be the usual pattern. In the results presented in Table XI, no consistent correlation can be found between the flexural strength values for test specimens of round and of rectangular cross sections; and it is the values for round pieces which fail to fit an established pattern even among themselves. Further, if one examines the

individual test results which are averaged to arrive at the values shown in the table for cylindrical pieces, the tendency toward wide scatter is apparent. However, it is of interest to note that the correlation between values for rod-shaped specimens and those for flat ones does average 1.01, thus giving support to the validity of the formula used in the calculations.

The same scatter does not appear in the modulus values of cylindrical pieces. This is not surprising, since the modulus expresses a load/deflection relationship before failure occurs in the test piece. However, the modulus results obtained were quite consistently low by about 20-30% in comparison with those for flat specimens.

Accordingly, it seemed necessary to decide that we could not rely on the flexural strength and modulus values obtained for cylindrical pieces, even though they were extruded faster and more satisfactorily in many cases.

3. Use of Rectangular Mold During Curing

The original purpose of the rectangular mold was to press material extruded through the round nozzle into the flat shape regularly used in physical property tests. This provision was felt to be necessary because, as stated earlier, no standard formula had been found for calculating flexural strength and modulus values for cylindrical test specimens, and because of the possibility (which proved to be the case) that results for these specimens could not be consistently correlated with results on flat strips.

To evaluate the effect of this procedure, representative samples of material which had been extruded through the round nozzle in the 1961-63 and 1961-64 series were pressed gently by hand into the rectangular slots and the flat top piece clamped in place. Curing at the standard conditions was done with the assembly in the clamped position. As can be seen in Table XII, these pieces quite consistently tested 7 to 18% stronger than specimens of the same blends which had been extruded through the rectangular nozzle at the same pressure and temperature. Flexural modulus was increased on an average of 4%, but the effect on modulus varied more from one test to another.

There is more than one possible explanation for the observed effects. Regardless of the reason, however, it seemed clear that reliable test results would probably not be obtained by making test specimens in this manner. That is, the results could not be compared directly with those for specimens extruded as flat strips. Furthermore, in the final analysis it was easier to extrude through the rectangular nozzle, despite the additional care and time required, than to have to press cylindrical test pieces into the mold.

One question which naturally arises is whether the use of a mold during curing has any effect on the physical properties of the test material produced. This question had been anticipated, and three different blends had

been used to make a comparison between strips extruded through the rectangular nozzle and cured in the mold, and those cured between flat stainless steel plates in our usual manner. The results shown in Table XII seem to indicate that flexural strength is not affected significantly; modulus appeared to be influenced more, but not in any consistent manner in the three comparison tests.

Applying these results to those of the immediately preceding tests, one can reasonably conclude that the approximately 10% strength increase observed there was due to some cause other than mold-curing. The most likely reason seems to be either (1) an effect of a smoother extrusion by the use of the round nozzle or (2) an improvement in the character of the material as a result of having been pressed into the mold. Possibly the latter is accomplished either because surface imperfections are eliminated or because additional compacting takes place. In any case, the small improvement was not worth pursuing at this point in our work, and it seemed clear that we should not use material which had been extruded through a round nozzle in the same evaluation with any extruded through a rectangular nozzle.

The results obtained do suggest that perhaps calendering under the proper conditions following extrusion might improve our material.

Program

Any future work will depend on applications which may be uncovered for this type of material. Samples can be made available to any Corporation personnel concerned with building material requirements and specifications.

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Attachments: 15 Tables, 1 Figure

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Index:

Unsaturate oil	1
Hydrocarbon resins	0, 1, 2
Prepolymers	X
Asbestos	0, 1
Thermopolymerization	X
Curing	X
Oxidation	X
Flexural strength	X
Flexural modulus	X
Building materials	6, 8
Extrusion	X
Water-resistance	X

TABLE I

FLEXURAL STRENGTH AND MODULUS OF PRESSED MATERIAL MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Effect of Curing Time and of Air Storage Before Curing

Blend[1]	Run No.	Property	Value after final curing time of:[2] 10 hours	64 hours	120 hours
C Cut, 2:1	1961-38, -45	Strength (psi)	2,870	3,490	3,660
		Modulus (psi)	790,000	640,000	790,000
U-311, 2:1	1961-24, -44	Strength (psi)	2,630	2,210[3]	3,290
		Modulus (psi)	640,000	560,000	940,000

Stored in Air 18 days Before Curing

C Cut, 2:1	1961-45A	Strength (psi)	2,080
		Modulus (psi)	670,000
U-311, 2:1	1961-44A	Strength (psi)	2,050
		Modulus (psi)	540,000

1 Type of resin, and ratio of asbestos to resin. All specimens were 1/8" thick test strips formed by pressing at 700-1000 psi at room temperature.

2 Curing time shown was at 200°C. It was preceded by 4 hours at 150°C.

3 As stated in previous report, this value appears low; the additional results seem to substantiate the conclusion. These specimens were layered, probably as a result of poor forming.

TABLE II

FLEXURAL STRENGTH AND MODULUS OF PRESSED MATERIAL MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Effect of Forming Pressure

Resin	Ratio, Asbestos: Resin	Run No. 1961-	Pressure[1] (psi)	Flexural Strength (psi)	Flexural Modulus (psi)
U-311	1.5:1	78-4	1000	2470	600,000
		78-5	1500	2860	600,000
		78-6	2600	3170	650,000
C Cut	2:1	78-1	1000	2610	650,000
		78-7	1450	3580	1,000,000
		78-8	1740	3140	900,000
		78-9	2050	2280	790,000
		78-10	2350	2260	800,000
		78-11	2600	2830	1,060,000

[1] Room temperature.

TABLE III

FLEXURAL STRENGTH AND MODULUS OF MATERIAL

MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Effect of Water Exposure on Cured Material

Blend[1]	Pressure[2] (psi)	Run No. 1961-	Property[3]	Original Value (psi)	After 70 Hours in H_2O[4] Value (psi)	% of Original	After 70 Hours in H_2O Plus 4-Hour Air-Drying Value (psi)	% of Original
C Cut, 2:1	700-1000	38	S	2870	2360	82.2	--	--
			E	790,000	720,000	91.1	--	--
C Cut, 1:1	1000	78-2	S	3920	2810	71.7	2890	73.7
			E	740,000	670,000	90.1	700,000	94.6
U-311, 1:1	1000	78-3	S	2240	1700	75.9	2400	107.1
			E	440,000	460,000	104.6	460,000	104.6
U-311, 1.5:1	1000	78-4	S	2470	2150	87.0	2230	90.3
			E	600,000	570,000	95.0	470,000[5]	78.3[5]
U-311, 1.5:1	2600	78-6	S	3170	2270	71.6	3370[6]	106.3[6]
			E	650,000	610,000	93.8	670,000[6]	103.1[6]
Average.	--	---	S	--	--	77.7	--	90.4[7]
			E	--	--	94.9	--	92.5[7]

---

1 Type of resin, and ratio of asbestos to resin.

2 All pressed at room temperature.

3 S = flexural strength. E = flexural modulus.

4 Tests were made within 10 minutes after removal of specimens from water.

5 Results appear low without apparent reason.

6 Value based on a single test.

7 Average based on results for three blends; values for last specimen excluded.

TABLE IV

FLEXURAL STRENGTH AND MODULUS OF MATERIAL

MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Comparison of Extrusion and Pressing

(Best sets of specimens for each blend of material)[1]

		Pressed Specimens			Extruded Specimens		
Resin	Ratio Asbestos:Resin	Forming Pressure psi	Strength psi	Modulus psi	Extrusion Pressure psi	Strength psi	Modulus psi
U-311	2:1	700-1000[2]	2630	640,000	3200	2310	860,000
	1.5:1	2600	3170	650,000	1740	2840	730,000
	1:1	1000[2]	2240	440,000	1450	3670	610,000
C Cut	2:1	1450	3580	1,000,000	3780	3330	1,040,000
	1:1	1000[2]	3920	740,000	--	--	--

[1] In all cases these specimens were pressed or extruded at room temperature.

[2] Only pressure used. For all other blends several pressures were tried, and value shown is the one which produced the strongest specimens.

TABLE V

FLEXURAL STRENGTH AND MODULUS OF EXTRUDED MATERIAL

MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Comparison of Different Blends[1]

Resin Type, and Ratio, Asbestos:Resin	Run Numbers 1961-	Extrusion Pressure psi	Extrusion Rate in./min.	Strength psi	Modulus psi
U-311, 1:1					
Best single set	63-14	1450	10.7	3670	610,000
Average of all sets	63-5 thru -8 and -12 thru -15	870-1450	0.8-10.7	3280	600,000
U-311, 1.5:1					
Best single set	64-13	1740	2.2	2840	730,000
Average of all sets	64-12 thru -16	1310-2330	0.9-8.2	2730	730,000
U-311, 2:1					
Best single set	47-2	3200	0.2	2310	860,000
Average of all sets	47-2 and -3	3200-4400	0.2-0.5	2305	830,000
C Cut, 2:1					
Best single set	67-7	3780	0.6	3330	1,040,000
Average of all sets	67-6 thru -11	3260-6100	0.2-2.2	3.20	1,040,000

[1] In all cases specimens were formed by double extrusion at room temperature through a rectangular nozzle. First pass was at higher pressures and rates without regard to smoothness.

TABLE VI

FLEXURAL STRENGTH AND MODULUS OF EXTRUDED MATERIAL

MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Effect of Double Extrusion

Blend[1]	Run Number 1961-	Direction of Extrusion[2]	Pressure psi	Double or Single	Rate in./min.	Strength Value psi	Strength Factor[3] D/S	Modulus Value psi	Modulus Factor,[3] D/S
U-311, 1:1	63-8	Horizontal	1740	Double	46.0	3390	1.04	700,000	1.32
	63-3	Horizontal	1740	Single	17.5	3260		530,000	
U-311, 1.5:1	64-13, -16(avg)	Downward	1470	Double	2.0	2840	1.01	745,000	1.01
	64-1	Downward	1470	Single	1.1	2800		740,000	
U-311, 1.5:1	64-14	Downward	2040	Double	4.4	2510	1.03	700,000	0.97
	64-2	Downward	2040	Single	2.4	2440		720,000	
U-311, 1.5:1	64-15	Downward	2320	Double	8.2	2780	1.18	760,000	1.12
	64-3	Downward	2320	Single	4.6	2360		680,000	
U-311, 2:1	47-3	Horizontal	4400	Double	0.5	2300	1.24	800,000	0.95
	47-1	Horizontal	4400	Single	0.5	1860		840,000	
C Cut, 2:1	67-6	Horizontal	3490	Double	0.4	3300	1.02	1,100,000	1.09
	67-2	Horizontal	3490	Single	0.7	3220		1,010,000	
C Cut, 2:1	67-7	Horizontal	3780	Double	0.6	3330	1.10	1,040,000	1.02
	67-4	Horizontal	3780	Single	1.5	3020		1,020,000	
Average							1.09		1.07

[1] Type of resin and ratio of asbestos to resin.

[2] Downward is same direction as piston travel. All specimens in these sets were extruded at room temperature through a rectangular nozzle.

[3] Value for double ÷ value for single.

TABLE VII

FLEXURAL STRENGTH AND MODULUS OF EXTRUDED MATERIAL

MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Effect of Extrusion at Elevated Temperatures

(Best specimen shown for each temperature)

Resin	Ratio Asbestos: Resin	Temperature °C	Run No. 1961-	Pressure psi	Rate in./min.	Strength psi	Modulus psi
C Cut	2:1	~25	48-1	5800	0.1	2680	920,000
C Cut	2:1	50	51-1-2	1310	2.4	1520	790,000
C Cut	2:1	75	51-1-5	1160	5.4	1160	710,000
C Cut	2:1	105	51-1-11	1740-2910	120.	1520	780,000

NOTE: All specimens were formed by a single extrusion through a rectangular nozzle in the side position.

TABLE VIII

FLEXURAL STRENGTH AND MODULUS OF MATERIAL

MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Effect of Drying Asbestos Before Blending

Asbestos Used	Run No. 1961-	Strength psi	Ratio of Strength, dried/not dried	Modulus psi	Ratio of Modulus, dried/not dried
		Room-temperature single extrusion through rectangular nozzle at 5800 psi:			
Dried	67-5	3210	1.20	1,080,000	1.07
Not Dried	48-1	2680		920,000	
		Room-temperature pressing at ~1000 psi:			
Dried	78-1	2610	0.91	655,000	0.83
Not Dried	-38	2870		790,000	

NOTE: All specimens made from 2:1 blends of asbestos:C Cut soft resin.

TABLE IX

FLEXURAL STRENGTH AND MODULUS OF MATERIAL

MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Effect of Thoroughness of Mixing

How Mixed	Run Nos. 1961-	Pressure psi	Rate in./min.	Flexural Strength psi	Ratio of Strength, Milled/ Hand	Flexural Modulus psix10^3	Ratio of Modulus, Milled/ Hand
			Single Extrusion:				
Milled	63-1, 2, 3	870-1740	2.2-17.5	3330[1]	1.10	590[1]	0.84
Hand	46-1	350	>2.[2]	3020	--	700	
			Double Extrusion:				
Milled	63-6, 7, 8	870-1740	1.1-46.0	3260[1]	1.04	670[1]	0.88
Hand	46-2	~500-600[2]	>6.[2]	3140	--	760	

[1] Average of three runs.

[2] Estimated; not measured.

NOTE: All specimens made by room-temperature extrusion of 1:1 blends of asbestos:U-311 Prepolymer through a rectangular nozzle.

TABLE X

FLEXURAL STRENGTH AND MODULUS OF EXTRUDED MATERIAL MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Downward Extrusion Versus Horizontal

(All specimens made from 1:1 asbestos:U-311
by a double extrusion at room temperature through a rectangular nozzle)

Direction of Extrusion*	Run No. 1961-	Press. psi	Rate in./min	Flexural Strength psi	Ratio of Strength ↓/↳	Flexural Modulus psi x 10^3	Ratio of Modulus ↓/↳
↓	63-12, -15 (avg)	870	0.8	3055	1.02	675	1.01
↳	63-6	870	1.1	2990		670	
↓	63-13	1160	3.4	3700	1.24[1]	700	1.11
↳	63-5	1160	4.7	2970[1]		630	
↓	63-14	1450	10.7	3670	1.08	610	0.97
↳	63-7	1450	12.3	3400		630	
Average of all 3 ratios					1.11		1.03
Average of two ratios (1961-63-5 rejected)					1.05		0.99

[1] These values questionable because result from Run 1961-63-5 seems definitely out of line.

* Piston travel is downward in all cases.

TABLE XI

FLEXURAL STRENGTH AND MODULUS OF EXTRUDED MATERIAL

MADE FROM ASBESTOS AND SOFT HYDROCARBON RESINS

Round Extrusion Nozzle Versus Rectangular

Nozzle Shape	Run No. 1961-	Blend[1]	Extrusion			Flexural Strength		Flexural Modulus	
			Single or Double	Pressure psi	Rate in./min.	Strength psi	Ratio,[2] ○/□	Modulus psix10^3	Ratio, ○/□
Round	63-11	1:1	Double	1450	19.5	3720	1.09	500	0.79
Rect.	63-7	1:1	Double	1450	12.3	3400		630	
Round	63-18	1:1	Double	1160	3.7	3240	0.88	500	0.71
Rect.	63-13	1:1	Double	1160	3.4	3700		700	
Round	63-10	1:1	Double	725	0.5	2620	0.88	510	0.76
Rect.	63-6	1:1	Double	870	1.1	2990		670	
Round	64-7	1.5:1	Single	2620	18.0	2850	1.14	560	0.77
Rect.	64-4	1.5:1	Single	2620	9.5	2500		730	
Round	64-10	1.5:1	Double	1450	1.8	2880	1.05	500	0.70
Rect.	Avg. 64-12 and 13	1.5:1	Double	1525	1.6	2750		715	
Average of five ratios							1.01		0.75

[1] All specimens made by room-temperature extrusion of blends of asbestos and U-311 Prepolymer.

[2] Two additional ratios, not presented because the "round" value in each case was extremely low, were 0.50 (1963-16/avg. of 1963-12 and 15) and 0.44 (1963-19/1963-14).

TABLE XII

FLEXURAL STRENGTH AND MODULUS OF EXTRUDED MATERIAL

MADE FROM ASBESTOS AND HYDROCARBON RESINS

Comparison of Curing in a Mold with Curing Between Flat Plates

Blend[1]	Run No. 1961-	Extrusion Conditions[2] Pressure psi	Rate in./min.	Nozzle Shape	How Cured[3]	Strength Value psi	Strength Ratio, Mold/Plates	Modulus Value psi	Modulus Ratio, Mold/Plates
A. Study of Specimens Extruded in Rod Shape, Pressed into Flat Mold for Curing:									
U-311, 1:1	66-1	870	1.7	Round	Mold	3700	1.10	670,000	1.11
	63-2, -6 (avg)	870	1.7	Rect.	Plates	3175		635,000	
U-311, 1:1	66-2	1450	19.5	Round	Mold	4010	1.18	720,000	1.14
	63-7	1450	12.3	Rect.	Plates	3400		630,000	
U-311, 1.5:1	66-3	1450	1.1	Round	Mold	3190	1.18	680,000	0.95
	64-5	1450	0.5	Rect.	Plates	2700		720,000	
U-311, 1.5:1	66-4	1450	1.9	Round	Mold	2940	1.07	680,000	0.95
	64-12, -13 (avg)	1525	1.6	Rect.	Plates	2755		715,000	
Average							1.13		1.04
B. Study of Specimens Extruded in Flat Shape, Some Cured in Mold and Some Between Plates									
U-311, 1:1	46-2F	350	--	Flat	Mold	3440	1.09	650,000	0.86
	46-2	~500	--	Flat	Plates	3140		760,000	
U-311, 2:1	47-3M	4400	0.5	Flat	Mold	2280	0.98	630,000	0.79
	47-3	4400	0.5	Flat	Plates	2300		800,000	
C Cut, 2:1	48-1M	5800	0.1	Flat	Mold	3030	1.03	1,150,000	1.25
	48-1	5800	0.1	Flat	Plates	2680		920,000	
Average							1.03		0.97

[1] Type of resin, and ratio of asbestos to resin.
[2] All extrusions at room temperature.
[3] All cured flat, whether in mold or between plates.

TABLE XIII

EVALUATION OF SOFT HYDROCARBON RESINS AS BINDERS FOR COALINGA ASBESTOS

Conditions of Preparation and Physical Property Tests of Material

Made from 1:1 Blend of Asbestos and U-311 Prepolymer

Blend	Sample No.	Extrusion Conditions					Appearance of Extruded Specimen	Curing Conditions[b] Hours at 160°C	Curing Conditions[b] Hours at 200°C	Curing Conditions[b] Other	Physical Properties[c] Flexural Strength, psi	Physical Properties[c] Flexural Modulus, psi	Remarks
		Single (1) or Double (2)	Nozzle Used, Direction[a]	Temp. °C	Pressure psi	Rate in./min.							
U-311, 1:1	1961-46-1	1	□ →	RT	350	–	Smooth	4	10		3020	700,000	Mixed by hand only.
	-2	2	↓	↓	350	–	"	↓	↓		3140	760,000	All others milled.
	-2F	2	↓	↓	500-600?	(Fast)	Less smooth	↓	↓	5 hrs 250° in pressure mold	3440	650,000	↓
U-311, 1:1	1961-63-1	1	□ →	RT	1160	2.5	Smooth	4	10		3370	650,000	
	-2	1	↓	↓	870	2.2	"	↓	↓		3360	600,000	
	-3	1	↓	↓	1740	17.5	"	↓	↓		3260	730,000	
	-4	1	↓	↓	1740	3.2 d	"	↓	↓		3100	660,000	
	-5	2	↓	↓	1160	4.7	"	↓	↓		2970	630,000	
	-6	2	↓	↓	870	1.1	Very smooth	↓	↓		2990	670,000	
	-7	2	↓	↓	1450	12.3	Smooth	↓	↓		3400	630,000	
	-8	2	↓	↓	1740	46.0	"	↓	↓		3350	700,000	
	-66-1	2	○ →	↓	870	1.7	"	↓	↓	e	3700	670,000	
	-2	2	○ →	↓	1450	19.5	Less smooth	↓	↓	e	4010	720,000	
-25-	-63-12	2	□ ↓	↓	870	0.8	Very smooth	↓	↓		3030	700,000	
	-13	2	↓	↓	1160	3.4	Smooth	↓	↓		3700	700,000	
	-14	2	↓	↓	1450	10.7	Less smooth	↓	↓		3670	610,000	
	-15	2	↓	↓	870	0.8	Very smooth	↓	↓		3080	650,000	
	-63-10	2	○ →	↓	725	0.5	" "	↓	↓		2620	570,000	
	-11	2	○ →	↓	1450	19.5	Less smooth	↓	↓		3720	500,000	
	-63-16	2	○ ↓	↓	870	1.8	Sl. rough	↓	↓		1520	280,000	
	-17	2	↓	↓	725	0.6	Smooth	↓	↓		2060	480,000	
	-18	2	↓	↓	1160	3.7	Less smooth	↓	↓		3240	580,000	
	-19	2	↓	↓	1450	2.7 d	" "	↓	↓		1610	420,000	
U-311, 1:1	1961-78-3	(Pressed, not extruded)	↓	RT	1000	–	Very smooth	4	10		2240	440,000	
	-3W	↓	↓	↓	↓	–	"	↓	↓		1700	460,000	f
	-3WD	↓	↓	↓	↓	–	"	↓	↓		2400	460,000	g

a Direction of piston travel was downward in all cases.

b All specimens were cured in air, between two ¼" S.S. plates in convection furnace. (Except those cured in mold. Cf. Note e.)

c Values given are averages of 3-6 tests — more than 3 when necessary to obtain reasonable agreement.

d Slow, probably because last of charge.

e Pressed gently into rectangular mold for curing.

f Tested immediately after 70 hours' immersion in water.

g Tested after above immersion followed by 4 hours' drying.

6435

TABLE XIV

EVALUATION OF SOFT HYDROCARBON RESINS AS BINDERS FOR COALINGA ASBESTOS

Conditions of Preparation and Physical Property Tests of Material

Made from 2:1 and 1.5 Blends of Asbestos and U-311 Prepolymer

Blend	Sample No.	Extrusion Conditions: Single(1) or Double(2)	Nozzle Used, Direction[a]	Temp. °C	Pressure PSI	Rate In./Min.	Appearance of Extruded Specimen	Curing Conditions: Hours at 150°C	Hours at 200°C	Other	Physical Properties: Flexural Strength, PSI	Flexural Modulus, PSI	Remarks
U-311, 2:1	1961-47-1	1	□ →	RT	4400	0.5	Smooth,	4	10		1860	840,000	Medium brown.
	-2	2			3200	0.2	but slightly				2310	860,000	" "
	-3	2			4400	0.5	rough edges.				2300	800,000	" "
	-3M	2			4400	0.5				M Mold Pressure A	2280	630,000	Almost black.
U-311, 1.5:1	1961-64-1	1	□ ↓	RT	1740	1.1	Smooth	4	10		2800	740,000	
	-2	1			2040	2.4	"				2440	720,000	
	-3	1			2330	4.6	"				2360	680,000	
	-4	1			2620	9.3	"				2500	730,000	
	-5	1			1450	0.5	Very smooth				2700	720,000	
	-7	1	○ ↓		2620	18.0	Smooth				2850	560,000	
	-66-3	1	○ ↓		1450	1.1	"			d	3190	680,000	
	-64-12	2	□ ↓		1310	0.9	"				2670	700,000	
	-13	2			1740	2.2	"				2840	730,000	
	-14	2			2040	4.4	"				2510	700,000	
	-15	2			2330	8.2	"				2780	760,000	
	-16	2			1740	1.8	"				2530	760,000	
	-66-4	2	○ ↓		1450	1.9	"			d	2940	680,000	
	-64-9	2			1160	0.5	"				3440	570,000	
	-10	2			1450	1.8	"				2880	500,000	
	-11	2			2620	3.9	"				2700	490,000	
U-311, 1.5:1	1961-78-4	(Pressed, not extruded)		RT	1000	-	Very smooth	4	10		2470	600,000	
	-4W					-	"				2150	570,000	e
	-4WD					-	"				2330	470,000	f
	-5				1500	-	"				2860	600,000	
	-6				2600	-	"				3170	650,000	
	-6W					-	"				2270	610,000	e
	-6WD					-	"				3370	670,000	f (only one result)

[a] Direction of piston travel was downward in all cases.

[b] All specimens were cured in air, between two 1/8" S.S. plates in convection furnace. (Except those cured in mold. Cf. Note d.)

[c] Values given are averages of 3-6 tests — more than 3 when necessary to obtain reasonable agreement.

[d] Pressed gently into rectangular mold for curing.

[e] Tested immediately after 70 hours' immersion in water.

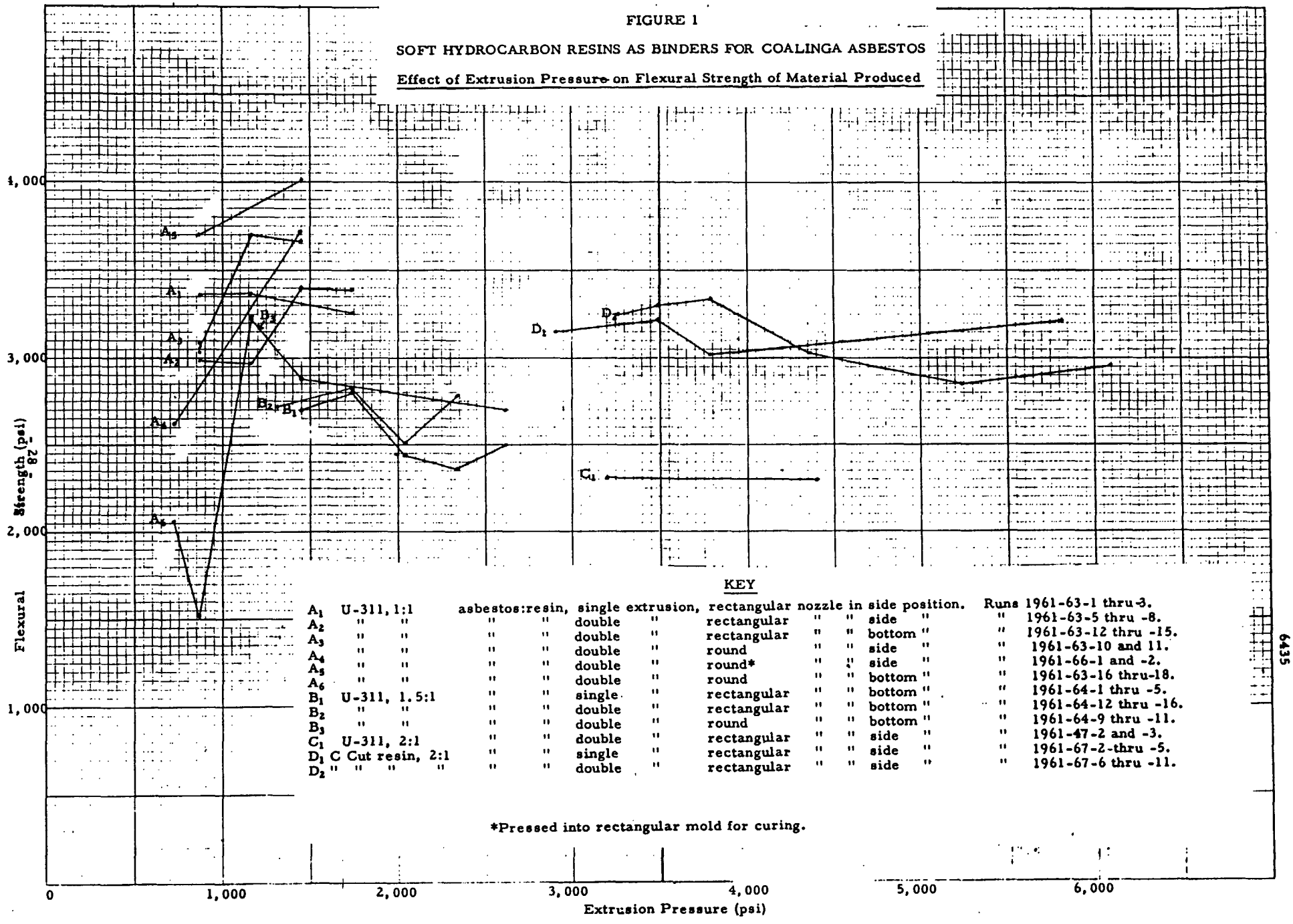
[f] Tested after above immersion followed by 4 hours' drying.

6435

FIGURE 1

SOFT HYDROCARBON RESINS AS BINDERS FOR COALINGA ASBESTOS

Effect of Extrusion Pressure on Flexural Strength of Material Produced



KEY

A_1	U-311, 1:1	asbestos:resin,	single	extrusion,	rectangular	nozzle in	side	position.	Runs	1961-63-1 thru -3.
A_2	" "	" "	double	"	rectangular	" "	bottom	"	"	1961-63-5 thru -8.
A_3	" "	" "	double	"	round	" "	side	"	"	1961-63-12 thru -15.
A_4	" "	" "	double	"	round*	" "	side	"	"	1961-63-10 and 11.
A_5	" "	" "	double	"	round	" "	bottom	"	"	1961-66-1 and -2.
A_6	" "	" "	double	"	rectangular	" "	bottom	"	"	1961-63-16 thru-18.
B_1	U-311, 1.5:1	" "	single	"	rectangular	" "	bottom	"	"	1961-64-1 thru -5.
B_2	" "	" "	double	"	rectangular	" "	bottom	"	"	1961-64-12 thru -16.
B_3	" "	" "	double	"	round	" "	bottom	"	"	1961-64-9 thru -11.
C_1	U-311, 2:1	" "	double	"	rectangular	" "	side	"	"	1961-47-2 and -3.
D_1	C Cut resin, 2:1	" "	single	"	rectangular	" "	side	"	"	1961-67-2-thru -5.
D_2	" " "	" "	double	"	rectangular	" "	side	"	"	1961-67-6 thru -11.

*Pressed into rectangular mold for curing.

6435