Study of the Use of Vapor and Vacuum Techniques for the Development of High Strength Filamentary Materials

Summary Technical Report
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by
William A. Robba
Pyrogenics, Inc.

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ABSTRACT

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The process investigated involves the deposition of pyrolytic graphite thin films on liquid metal substrates as a means of producing continuous high strength filaments. No evidence of reaction of the liquid metal with the graphite was found at temperatures as high as 2160°C.

Experimental vapor deposition apparatus was designed to produce films under controlled conditions and in sufficient quantities for evaluation. Parameters varied in the study included time, temperature, pressure and flow. Filaments were screened by testing tensile strength in a simple test rig. Specimens were then examined metallographically. The target objective of 100,000 psi tensile strength was reached when an as-deposited filament 2.0_{μ} thick reached 120,000 psi tensile strength.

This work is, in part, a continuation of that reported previously in Reference #1.

Key Words: Filaments, Graphite, Vapor Deposition, Graphite Filaments.

Ref. 1. Summary Technical Report "A Study of the Use of Vapor and Vacuum Deposition Technique For The Development of High Strength Filamentary Materials, May 21, 1964 - May 21, 1965.

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I. INTRODUCTION

This is a Summary Technical Report for Contract NASW-1201 entitled "A Study of the Use of Vapor and Vacuum Deposition Techniques for the Development of High Strength Filamentary Materials", covering the period May 22nd, 1965 through May 21st, 1966. The purpose of this contract is to conduct an investigation on methods of producing high strength, continuous length graphite filaments using vapor deposition techniques.

Graphite was chosen as the candidate material since theoretically graphite has the highest elastic modulus (144×10^6) of any material known.

Since substrate surface, nucleation mechanism and growth stresses affect structure the general approach undertaken was to deposit on a liquid to insure a smooth imperfection-free surface to initiate the reaction.

II. EXPERIMENTAL PROCEDURE

a. Deposition Apparatus

The original deposition apparatus (Ref. 1) consisted of a graphite heating element which was used to melt the liquid metal in a depression in the element. This unit was contained in a 100 mm diameter quartz envelope and powered by a 10 volt 5 kw variac regulated transformer. The relatively low power capacity of the supply limited sample size and radiation losses made temperature control extremely difficult. It was decided to go to an induction power supply of 20 kw capacity to permit more experimental flexibility. Accordingly, a vertical apparatus similar to the resistance heated unit was designed and built. This unit is shown in Figure 1. The envelope consists of a 50 mm quartz tube. Tantalum radiation shields were used around the four sides of the round graphite crucible susceptor. Coupling of the 450 kc output of the RF induction generator was directly to the crucible. The cylindrical graphite rod stock had a depression milled in it for the gold sample. The first successful deposition of a graphite film on gold was achieved in this setup. Maximum temperature attainable was 2200°C.

Initial experiments with the unit proved cumbersome to set up and disassemble. The tantalum shields became embrittled after several runs and were difficult to position on the tungsten pins used to hold

them in place. The tantalum was replaced with graphite foil which was easier to position, but was still rather clumsy. It was therefore decided to redesign the entire unit. A horizontal setup was laid out, still utilizing a 50 mm quartz tube for an envelope. Proper alignment of the brass end caps, collars and "O" ring seals was obtained by mounting the assembly on a one meter optical bench. The sliding clamps used with the bench permitted assembly and disassembly without breakage or chipping of the quartz, or readjustment of the optical pyrometer. A tantalum shield was attached to the brass cap on the uncooled end of the setup to protect the brass cap and "O" ring from radiation emanating from the sight hole in the susceptor. A quartz window mounted on the cap permitted a direct line of sight to the interior of the hot zone. The exhaust end of the unit is water cooled since the gases leaving the unit are hot. A step-down transformer was used with the RF generator to reduce the voltage across the work coil. This was necessary to minimize arcing and ionization within the quartz tube, since the experimental program called for pressures in the range 0.1 torr to 700 torr and ionization was encountered with a direct coupled coil at the low pressure end of the program. The copper work coil consisted of six close wound turns of 1/4 inch tubing. The coils contact the quartz and aid in keeping the envelope cool.

Process gases are individually metered and then mixed prior to admission to the deposition chamber. The mixed gases enter at the uncooled sight port end of the unit, thus keeping the quartz optical flat clean during a run. After leaving the chamber, the exhaust gases pass through a cooling coil, a small filter, a throttling valve and then to the vacuum pump. A flow diagram of the system is shown in Figure 2.

An important part of the deposition apparatus is the horizontal deposition unit. It was necessary that the unit meet the following requirements:

a. Reach temperatures of 2200°C.

b. Minimum of outgassing and contamination (i.e. no lampblack or graphite felt insulation near hot zone).

c. Be capable of producing filaments 1-1/2 inches long.

d. Permit no contact between the hot zone and the quartz envelope.

e. Have a relatively low thermal inertia so that many experiments can be run in a reasonable time.

f. Be easy to assemble and disassemble with a minimum of maintenance.

To meet these requirements, an induction crucible design developed by Pyrogenics to reach temperatures of 3400°C was utilized. The design was modified to be used in a horizontal position and to contain a rectangular graphite deposition boat. To obtain a uniform temperature the

unit was designed so that the RF field coupled with the inner surface of the susceptor, thus heating the graphite boat by radiation. With the exception of the graphite boat, all components of the heating unit are made of Pyroid. The overall design is illustrated in Figure 3. Two one-half inch thick pyrolytic graphite discs are used to support the chamber and keep it from touching the quartz envelope. The discs are close fitted to the envelope, so that the process gases are forced to pass through the deposition unit rather than around it. This also prevents the source gases from cracking and depositing graphite on the outside of the chamber or on the quartz tube. Press fitted to the discs are Pyroid tubes around one inch long. Both tubes and stand off discs are oriented to prevent axial heat flow and minimize RF coupling. The main chamber is cylindrical and measures 2.375 inches long by 1.250 inches O.D. and 0.750 I.D. The A-B planes of the Pyroid are oriented to follow the contour of the cylinder on all surfaces. A removable cap is press fitted into the inlet end of the cylindrical tube to permit insertion of the graphite deposition boat. The Pyroid chamber thus serves as an insulating chamber with a hole in each end to permit a through flow of process gases. A series of slots are cut in the cylindrical chamber from each end to a depth which leaves the inner 1/32 inch layer intact. The RF field thus couples with this layer only with the outer layers acting as highly efficient thermal shields.

Yet, the whole chamber is one integral unit. There has been no outgassing, or pitting in the unit in over six months of use. Typical performance during temperature calibration runs gave an internal temperature of 2200°C with an outside chamber temperature of 850°C at a power of approximately 5 KW. Figure 4 is a photograph of the deposition chamber. A picture of the deposition apparatus showing arrangement of the equipment is shown in Figure 5.

b. Crucible Design

A major problem in the development of this technique has been the design of a suitable container for the liquid metal. Earlier work with platinum indicated that the production of a smooth shiny film required that there be no reaction between the liquid metal and graphite or, for that matter, any of the other materials in contact with the metal, since they tend to crystallize out on the surface and interfere with the growth of the critical first layer of the pyrolytic graphite film. Thus, though platinum is an ideal metal from the viewpoint of melting point and vapor pressure, its ability to dissolve graphite in the molten state precluded its use as a deposition surface. Gold was investigated and metallographic sections of films deposited on molten gold indicated no reaction or dissolution had occurred. As a corollary to this, the molten gold does not wet graphite and thus a pronounced meniscus is formed.

It is difficult therefore, in a small scale laboratory equipment to produce flat films. Original circular crucibles with circular milled depressions produced hemispherical films which were impossible to section and prepare for tensile testing. Milling of a square depression improved the situation to some extent, but curvature was still excessive. To overcome this problem as well as to increase the quantity of material produced in a run, it was decided to increase the size of the crucible. For this purpose the deposition chamber described in the previous section was designed. A semi-cylindrical graphite boat was designed in which a rectangular channel was milled 0.375 inches wide x 1.5 inches long x 0.125 inches deep. The central portion of the molten gold charge in this design was flat enough for proper testing, though it was necessary to cut out the section for testing. This operation must be studied in greater detail to determine whether it is in fact producing edge defects and notches. Figure 6a is a sketch of the boat described above. The curved projection shown at one end of the boat is used for reading the black body temperature inside the deposition chamber during a run. Another graphite crucible design that has been used with some success is shown in Figure 6b. In this design long narrow grooves are machined in the boat, so that edge trimming is not required, and as many as three filaments are produced simultaneously. However, in this design, due to the narrowness of the slot, the curvature is very pronounced across the width of the

filament though along the length it is quite flat. A method of testing such filaments has been developed which is described in the following section.

c. Tensile Test Method

The filaments were tested in tension in an autographically recording tensile machine at a strain rate of 5×10^{-4} sec^{-1}. In order to eliminate or reduce any deformation of the specimens while mounting them in the tensile machine, a rig similar to the one designed by Lawley and Schuster (1962) for thin films was constructed. The specimens could be easily mounted in this rig without any deformation prior to mounting in the tensile machine, and consequently handling of the specimen was reduced to a minimum. A schematic diagram of the rig is shown in Figure 7. The lower half of the specimen grips were first mounted in the holder (A) by means of the two spring loaded set screws. In the construction of the rig two lines parallel to the side of the grips were accurately scribed on the upper faces of the grips. The tensile specimens were laid along the two scribe marks, so ensuring accurate alignment of the specimens and grips. The top halves of the grips were then tightened down on the specimen by means of the four Allen Screws (B). It was later found convenient to simply epoxy the films onto the bottom halves of the grips, so doing away with the two top halves. The holder was mounted on the retracting mechanism which was in turn

mounted to the base of the tensometer. In order to aid the alignment of the specimen, two further drive mechanisms were attached to the holder. These allowed it to be moved in two directions at right angles (either along the tensile axis or at right angles to it). For the sake of simplicity, these are not included in Figure 7.

Accurate alignment of the specimen was obtained by lining up three scribe marks, one on each of the brackets, and one on the tensile machine base. The above procedure ensured accurate alignment of the specimen in the tensile axis. The holder was finally removed by unloading the spring-loaded screws and withdrawing with the retracting mechanism. No load was applied to the specimen, when the top grip was released from the holder, since the machine was previously calibrated for zero load with the top grip attached to the load cell. By this method a total of seventeen specimens were tested. The actual experimental arrangement is shown in Figures 8a and 8b.

With the new deposition apparatus set up midway through the effort and producing films under closely controlled conditions, it was found that a large number of filaments were breaking in the grips or failing prior to the application of a reasonable load. At first, it was thought that this was due to deposition parameters or edge defects, but the percentage of failures was too high in light of the fact that every so often strengths of over 30,000 psi would be obtained. It

became evident that the mounting rig was not satisfactory for filaments under 25_{μ} in thickness. It was decided therefore to try a method used successfully in testing the tensile strength of boron filaments. (Ref. 2). This method would also permit testing a larger number of filaments in a shorter time than the previous approach. Accordingly, a series of aluminum tabs were cut and the filaments were fastened on to them with fast curing epoxy. Filaments with curved cross sections could be used by putting a drop of epoxy on the tab first, and then placing filament end on top followed by another dab of epoxy. Two hardened steel hooks were used on the load train of the tensile tester and the tabs slipped on from a piece of cardboard as described in the prior references. Though this rig prevented measuring modulus of elasticity over 10 million psi (the modulus of the rig) it did alleviate the alignment problem and the yield of successful tests was increased considerably. The weight of one of the aluminum tabs was 0.17 grams. This dead weight was neglected in arriving at the ultimate tensile strength since the amount was less than .1% of the average breaking stress.

Ref. 2. "A Survey of Test Procedures for Evaluating Short Brittle Fibers"- S. Schulman and J. Epting, ASD-TDR-63-92, May 1963.

d. Metallographic Analysis

The microstructure of pyrolytic graphite is a function of the substrate surface condition, temperature of deposition, partial pressure of the hydrocarbon gas and post deposition treatment. It has been established in prior tests with bulk pyrolytic graphite that microstructure has a definite effect on tensile strength. Surface roughness results in structural defects known as nodules. In a nodule the planes of pyrolytic graphite are tilted and the intersection between the tilted planes and those parallel to the deposition surface represent a natural cleavage plane. A crack will propagate through or initiate from this area which acts as a stress concentration. Samples that have been tested in tension are placed in clear plastic potting compound. A small section of the hardened plastic which contains the specimen is then placed in a second cup of compound so that the fracture surface is perpendicular to the surface to be polished. In this manner, an area very close to the fracture surface is examined in the metallograph. Examination of the fracture surface itself is performed prior to potting in plastic by mounting the sample in a micrometer stage of a metallurgical microscope.

The procedure for polishing is relatively simple, utilizing 240, 320, 400 and 600 grit silicon carbide paper with water for rough grinding. Polishing is done with 6 micron, 1 micron and 1/4 micron diamond compounds on Buehler Met cloth.

Pyrolytic graphite filaments were produced on liquid gold, polished pyrolytic graphite substrates and some of the latter were annealed at 3000°C. Microstructural specimens were prepared by the method outlined above. Figures 9a, b and c are typical of the structures observed in the three types of filaments. A comparison of Figures 9a and b reveals obvious differences in the microstructures of these two classes. The liquid gold substrate produced a much finer structure which appears continuously nucleated and even though five times thicker than the solid substrate film, showed no signs of coarse growth cones or nodules. The heat treated filaments, Figure 9c, show no microstructure at all, which is typical of stress annealed pyrolytic graphite (Ref. 3).

The metallographic preparation of samples polished at the fracture surface also serves in determining filament thickness and width. A metallurgical microscope with a filar eyepiece is used for this purpose. At 800 magnification, a full revolution of the micrometer dial equals 0.0005 inches. The dial is divided into 100 divisions so that thickness down to .125$_{\mu}$ can be measured. A precision ruled glass reticle is used to calibrate the filar eyepiece.

Ref. 3. Klein, C.A., Straub, W.D. & Diefendorf, R.J., Phys. Rev. 125 No. 2 468 (1962)

III. RESULTS & DISCUSSION

The experimental deposition program was laid out to explore the effects of temperature, pressure, flow rate and time on the deposited film. The range of parameters investigated is listed in Table I. As the program progressed, it became apparent that the range of some of the parameters would have to be narrowed considerably as long as liquid gold is used as the substrate material. The melting point of gold is 1063°C and its boiling point is 2966°C. At 1869°C the vapor pressure is 1 mm. It was found that operation above 1850°C at the lower pressure range resulted in excessive evaporation of gold. Operation in pure methane at pressure ranging from 1 - 20 mm did not produce films that could be removed from the gold without breakage. The films had a rather dull wrinkled and mottled appearance and were extremely brittle. With a diluent gas at a pressure of 600 mm, films were produced which could be separated from the gold and successfully mounted for test.

An early sample made under these conditions was sent for X-ray analysis of the c/2 spacing and the 1/2 width angles of the distribution curve representing the misalignment of the graphite crystallites from an in line parallel orientation. The results indicated that though the film macroscopically looked perfect, the film was, in fact, quite disordered

with a c/2 spacing of 3.404 Å and a 1/2 width tilt angle of 2°. For example, X-ray studies of stress annealed pyrolytic graphite indicate a c/2 spacing of 3.35 Å which is essentially that of a single crystal. Misorientation is 1/2° indicating less than one in 1,000 crystallites at 90°. It thus appears that stress annealing of the films produced on the liquid substrate should yield a much higher degree of perfection, since normally deposited pyrolytic graphite has a half width angle of 6° - 20°. The mirror-like appearance of the pyrolytic film indicates that on a macroscopic level there were no observable flaws or imperfections.

Table II lists runs #6 through #17 which were made with the resistance heated deposition unit. These runs established that a film could be deposited on a liquid metal, but quality and reproducibility were poor due to power supply limitations. In this series, both gold and copper were used as substrate materials, but the film produced on gold was smoother. This probably is due to the lower vapor pressure of gold at the deposition temperatures. Table III lists runs #18 through #65. These deposition runs were made in a vertical induction heated unit. Due to the small size of the deposition unit and difficulty with the positioning device on the tensile rig, only three samples were tested successfully. These gave tensile values of 29,170, 38,100 and 31,500 psi respectively. Since these values are only slightly

higher than bulk pyrolytic graphite (i.e. 12,000 to 20,000 psi), it was obvious that a better method of production and testing had to be devised. The horizontal unit was fabricated for deposition and the aluminum tab method was used for tensile testing. Runs #66 through #99 are not listed, since these were made in checking out the new unit. It was established again that low total pressure in the deposition chamber did not work well due to excessive evaporation of the liquid metal. The diamond wheel method of cutting the film was also abandoned as being too time consuming,and cleaving with a razor blade has been used since. Table IV lists runs #100 through #172 which were made in the horizontal unit during the last quarter of the program. Three observations can be made from studying the data in Table IV and comparing it with the rather scanty data presented in Tables II and III. Film thickness under 1 mil and width in excess of 40 mils appears to contribute to higher strength. The filament width effect is probably related to edge defects, since the narrower the width the more likely that a break or crack will occur during the slitting operation. Film thickness is also important for the same reason. The thinner the film, the easier a clean cut can be made, since all the films produced to date (with the exception of the heat treated type) are brittle and therefore, susceptible to micro cracks and notches. In addition, and of equal

importance, is the fact that the thinner the film the less the number of defects that can occur. This effect has been definitely established for filaments and whiskers of circular cross section. A thickness and area correlation is evident as indicated in Figures 10 and 11. The effect on strength is quite apparent below 0.2 mil thickness.

The third observation is that of the relation of hydrocarbon flow rate and partial pressure to film strength. The nucleation and growth mechanism of the film on the liquid metal is not yet understood. Measurements made after numerous experimental runs indicate that the deposition rate on the liquid metal ranges from 20 to 40 mils an hour (i.e. 0.3 to 0.8 mils/min.). Polished pyrolytic graphite plates placed in the susceptor under identical conditionsproduced deposition rates of 5 to 10 mils per hour which is considered a normal rate for deposition of soot free material. None of the specimens removed from the liquid metal showed any signs of sooting or nodular soot pockets characteristics of high pressure gas phase nucleated deposits.

However, the strength of such rapidly produced films (above 30 mils) per hour or 0.5 mil/min.) is not good, which infers that there is considerable misorientation in the film. In fact, visual inspection of film produced in this manner indicated a dull wrinkled appearance, and photomicrographs showed a highly regenerative structure. It is apparent that

the films produced at low hydrocarbon partial pressures are superior to those produced at higher pressures. Tensile strengths above 50,000 psi were obtained when methane flow (and correspondingly partial pressure) dropped to 0.5 CFH ($\sim$0.25 LPM) or less. Though the deposition rate drops and the film is much thinner, it is evident that the slower surface nucleation mechanism produces a more defect-free film than the higher rate combined surface and gas phase nucleation type. The three effects of film thickness, film width and nucleation mechanism are all probably interrelated, but further work is required before this can be clarified. The concept of surface versus gas phase nucleation has been discussed at length in the literature on vapor deposition, single crystal and thin film growth. Young (Ref. 4) and Diefendorf (Ref. 5) have attempted to apply these theories to the growth of pyrolytic graphite.

It is also to be noted that the highest strength filaments produced to date (117,000 psi and 121,500 psi) were produced in run #164 and #167. Run #167 produced the thinnest film up to that time (i.e. 0.075 mils $\sim$2.0$_\mu$) and since the run duration was two minutes,

Ref. 4 Young, D.A. - Discovery 44-48 December (1965)

Ref. 5 Diefendorf, R.J. - Journal de Chemie Physique 57 815.21 (1960)

the lowest deposition rate. The deposition temperature range 1560 - 1680°C is responsible for the low deposition rate, but the effect, if any, of the lower deposition temperature on strength is not known. A second lower range methane flow meter will be installed to permit accurate flow measurement down to 0.01 CFH, so that the temperature effect and partial pressure effect may be separated.

IV CONCLUSIONS

The construction of a versatile film deposition chamber where process variables can be accurately controlled has indicated that film thickness, deposition rate and pressure play an important part in determining filament strength over 50,000 psi. Insufficient data exists to determine the relation of these process variablesto one another but it is obvious that they are not independent.

The method of preparing specimens for testing is also important. Cleaving a thin brittle film is apparently better than cutting it and in either case, it is imperative that the film width be sufficient compared to its thickness to prevent crack propagation normal to the cut surface.

Finally, alignment of the specimen in tensile strength determination is vital to a successful test. It has been found that it is more important to permit self alignment during the test, than it is to attempt to establish the alignment prior to the test in a device which holds the filament rigidly during the test. In general, the feasibility of producing a thin film of pyrolytic graphite on a liquid metal has been established. The choice of the substrate metal is limited to those metals which do not react with graphite when in the molten state at the desired deposition temperature. Other methods of filament

production, such as iridium foil or polished pyrolytic graphite have not proved as simple or as good as the liquid metal approach, and it appears that of all the methods investigated that one should be the most amenable to quantity production of filaments on a continuous basis.

V FUTURE WORK

The first objective of a tensile strength of over 100,000 psi on an untreated pyrolytic graphite film grown on a liquid metal has been achieved. It is now necessary to determine accurately, the relation of the process parameters which permitted the increase from 30,000 psi to over 100,000 psi and then use this as a base for increasing strength by another factor of three or more.

Accurate flow measurement below 1 CFH is essential for the small deposition unit now in use and a second meter will be installed for hydrocarbon flow control down to 0.01 CFH.

Methods of overcoming edge defects will be studied by investigating two new approaches. The first will involve deposition boat redesign in a manner to permit filament separation at the desired width by natural cleavage rather than by mechanical means. The second will consist of recoating a separated filament with the objective of encasing it in a second film that theoretically will have no edge.

Once an adequate base strength has been established with as-deposited filaments, the effects of temperature cycling and stress annealing will be investigated. It is anticipated that large increases in strength and modulus will be achieved by this method when the

proper base material has been produced. It is felt that the key to truly high strength filaments lies in eliminating the microscopic defects and crystallite misalignment which exist in the as-deposited films. The fracture surface must be studied at high magnification to determine if possible, the type of fracture, and how it initiates. In line with this, the tensile test rig must be improved to give strain and modulus values as strength increases. Modulus values should approach 50 million, if strength values reach 300,000 psi.

Finally, if the second plateau can be reached, methods of producing such filaments in quantity and on a continuous basis will be outlined, since the usefulness of such filaments can only be achieved, if they can be used in quantity as light weight reinforcements, properly bonded in plastic and metal composite structures.

TABLE I

Parameter	Range
Temperature	1200° - $2100^{\circ}C$
Pressure	2 mm - 700 mm
Flow Rate CH_4	0.5 - 4 C.F.H
Flow Rate Argon	0 - 10 C.F.H
Time	30 Sec. - 5 Min.

TABLE II

Resistance Element
Film Deposition

Run #	Temp. oC	Sys Press mm	Flow CFH A	Flow CFH Ch4	Time Min.	Remarks
6	1345 to 1420	10	-	-	45	Gold melted 1005°C correction ~ 60°C excessive Au evaporation rough film produced.
7	1255 to 1280	20	-	-	45	Note "wrinkling" of Au surface poor film produced.
8	2130 to 2110	350	7	8	15	Use argon atmosphere to reduce Au evap. Note evap. stops when ch_4 introduced removable film produced.
9	1745 to 2110	480	1	1	10	Used copper - erratic pressure - poor deposit.
10	1920 to 1930	560	1.2	.8	30	
11	1790 to 1785	570	10	0.5	5	Film adhering to gold.
12	1750 50 1805	570	10	.8	5	
13	1815 to 1785	620	10	1.4	10	Film fractured on cooling.
14	1850 to 1815	630	10	1.1	15	Specimen removed but not tested.
15	1900 to 1810	640	10	1.2	15	Specimen removed but not tested.
16	1890 to 1810	635	10	1.3	15	Specimen removed - Slit on diamond wheel-broke in mounting.
17	1960 to 1810	635	10	1.2	15	1st specimen tested in tensile rig.

TABLE III

Deposition in Vertical Induction Set-Up

Run #	Temp. oC	Sys Press mm	Flow CFH A	Flow CFH Ch4	Time Min.	Remarks
18	1840 to 1835	630	10	1.4	5	
19	1900 to 1830	632	10	1.4	5	Specimen broke away from substrate
20	1850 to 1865	635	10	1.3	5	Specimen shattered on cutting.
21	1825 to 1870	635	10	1.5	5	
22	1825 to 1860	30	-	1.5	5	
23	1850 to 1865	630	10	1.4	3	Broke on removal.
24	1850 to 1830	630	10	1.3	3	
25	1840	635	10	1.4	3	
26	1875	630	10	1.4	3	
27	1860 to 1825	635	10	1.3	5	
28	1860 to 1825	635	10	1.3	1	Specimen Shattered.
29	1810 to 1850	630	10	1.3	1.5	Specimen flaked off.
30	2000 to 1930	640	10	1.4	3	Substrate balled up.

TABLE III

(Continued)

Run #	Temp. oC	Sys Press mm	Flow CFH A	Flow CFH Ch4	Time Min.	Remarks
31						Leak
32	1900 to 1940	650	10	1.3	3	Diamond wheel shattered during cutting.
33	1930 to 1980	630	10	1.3	3	
34	1950 to 1940	640	10	1.2	3	
35						Lost Vacuum
36	1900 to 1930	640 660	10	1.4	3	
37	2030 to 1995	640	10	1.3	3	
38	2000 to 2080	640	10	1.3	3	
39	2040	640	10	1.4	5	
40	2060	640	10	1.3	5	
41						Lost Vacuum
42	1850	600	10	1.4	1	Lost control of Gas flow.
43						
44	1980 to 2120	640	10	1.3	2	
45	2000 to 2030	640	10	1.4	2	

TABLE III

(Continued)

Run #	Temp. oC	Sys Press mm	Flow CFH A	Flow CFH Ch4	Time Min.	Remarks
46	1920 to 1970	630	10	1.5	3	
47	1970	640	10	1.5	2.5	
48	2020	640	10	1.4	2.5	
49	2020	640	10	1.3	1	
50	1960 to 2000	640	10	1.4	1	
51	1960 to 2020	640	10	1.3	1	
52	2100 to 2130	640	10	1.3	1	
53	2030 to 2060	650	9	1.4	1	
54	2060 to 2090	630	10	1.4	1	
55	2130 to 2150	640	10	1.4	1	
56	2040 to 2100	630	10	1.3	1.5	
57	2000 to 2030	640	10	1.4	1	
8	1960 to 2010	640	10	1.5	1	

TABLE III

(Continued)

Run #	Temp. oC	Sys Press mm	Flow CFH A	Flow CFH Ch4	Time Min.	Remarks
59	1970 to 2040	640	10	1.3	1.5	
60	2080 to 2120	640	10	1.5	1.5	
61	1970 to 2120	650	10	1.35	2	
62	2000 to 2130	640	10	1.4	2	
63	2000 to 1980	640	10	1.45	2	
64	2000 to 2040	640	10	1.5	2	
65	2020 to 2040	640	10	1.45	2	

66 Thru 99 Parametric runs to study process variables samples not tested.

TABLE IV

Horizontal Tray - Induction Unit

Run #	Temp. oC	Time Min	Sys Press mm	Flow CFH A	Flow CFH Ch4	t m.1	w m.1	Cross Sec. 10-6 in 2	L in	Stress lbs.	Uts KPSI
100	2130 2000	2	640	10	1.0	-	-	-	-	-	-
101	1900 1860	2	640	10	1.0	1.1	48	52.8	.09	1.6	30.7
102	1900 1880	2	650	5	1.0	1.3	82	106.6	.160	0.95	9.0
103	1850 1880	2	650	5	0.25	.90	40	36.0	.08	1.2	33.3
104	1800 1840	2	640	5	0.25	.86	57	48.4	.176	3.4	69.6
158	1700 1780	2	640	5	0.25	.22	74.9	16.4	0.1	1.62	98.6
164	1560 1680	2	640	5	0.5	.11	41.2	4.94	0.1	0.58	117
167	1750 1710	2	640	5	0.5	.075	52.6	3.95	0.1	0.49	121.5
168	1670 1710	2	640	5	0.25	.25	68.8	17.2	0.1	0.45	26.5
169	1680 1750	2	640	5	0.5	.18	46.9	8.45	0.1	0.71	84.1
170	1700 1780	2	640	5	0.25	.29	75.3	21	0.1	1.06	50.5
172	1630 1670	3	640	5	0.31	.10	56.1	5.6	0.1	0.5	89.5

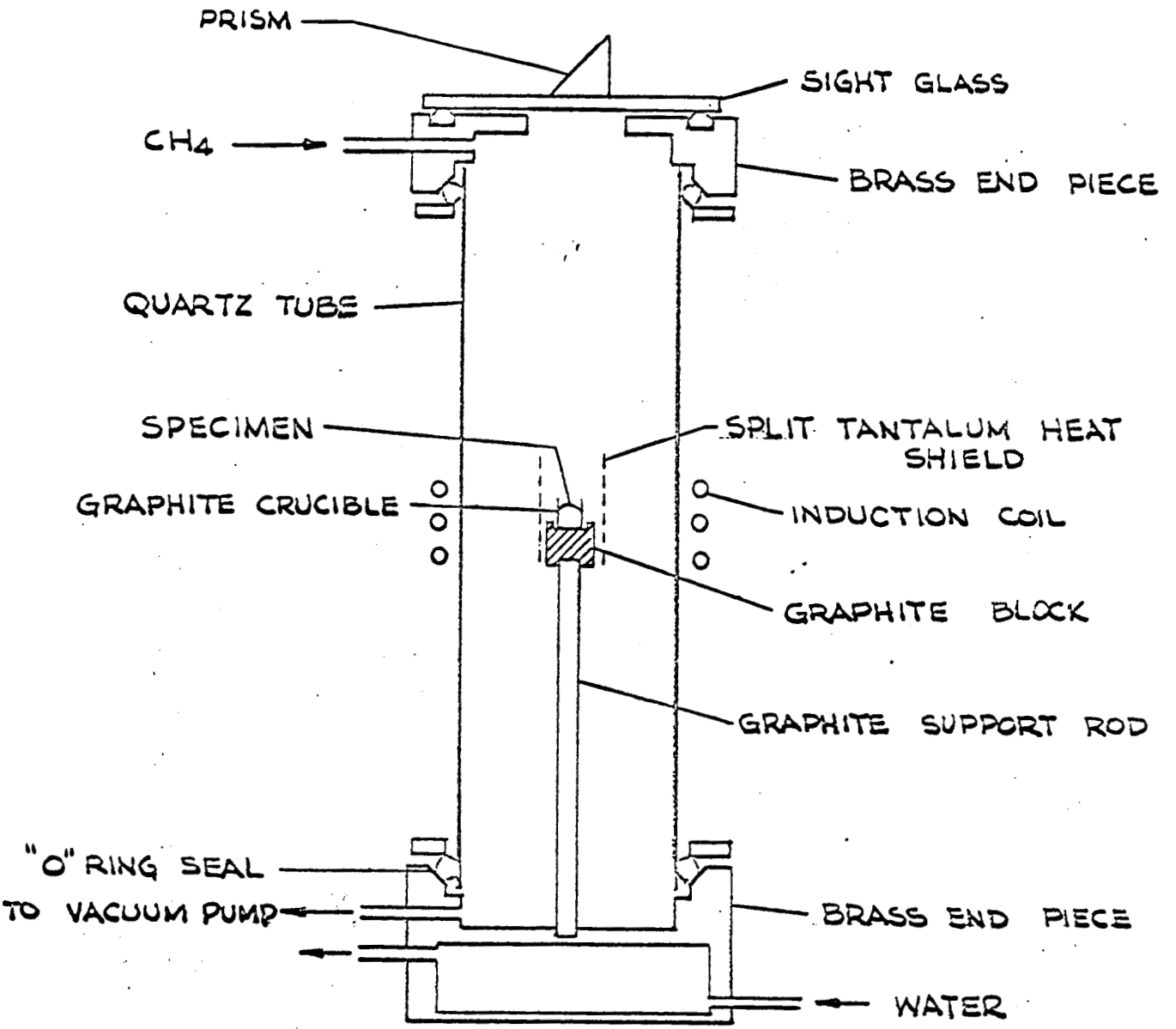


FIG. 1 SCHEMATIC DRAWING OF DEPOSITION UNIT USING INDUCTION HEATING

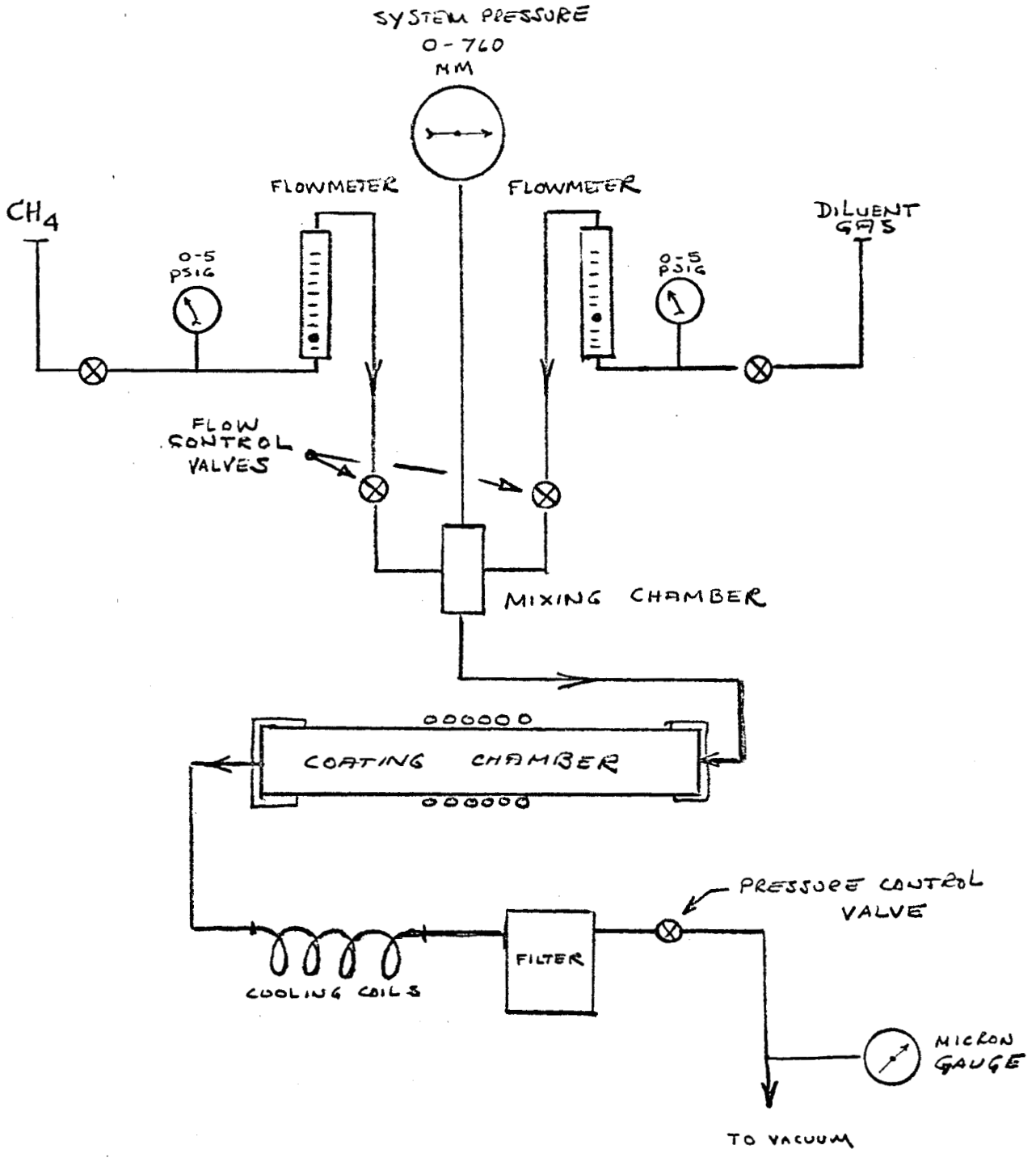


FIG. 2 FLOW DIAGRAM FOR HORIZONTAL UNIT

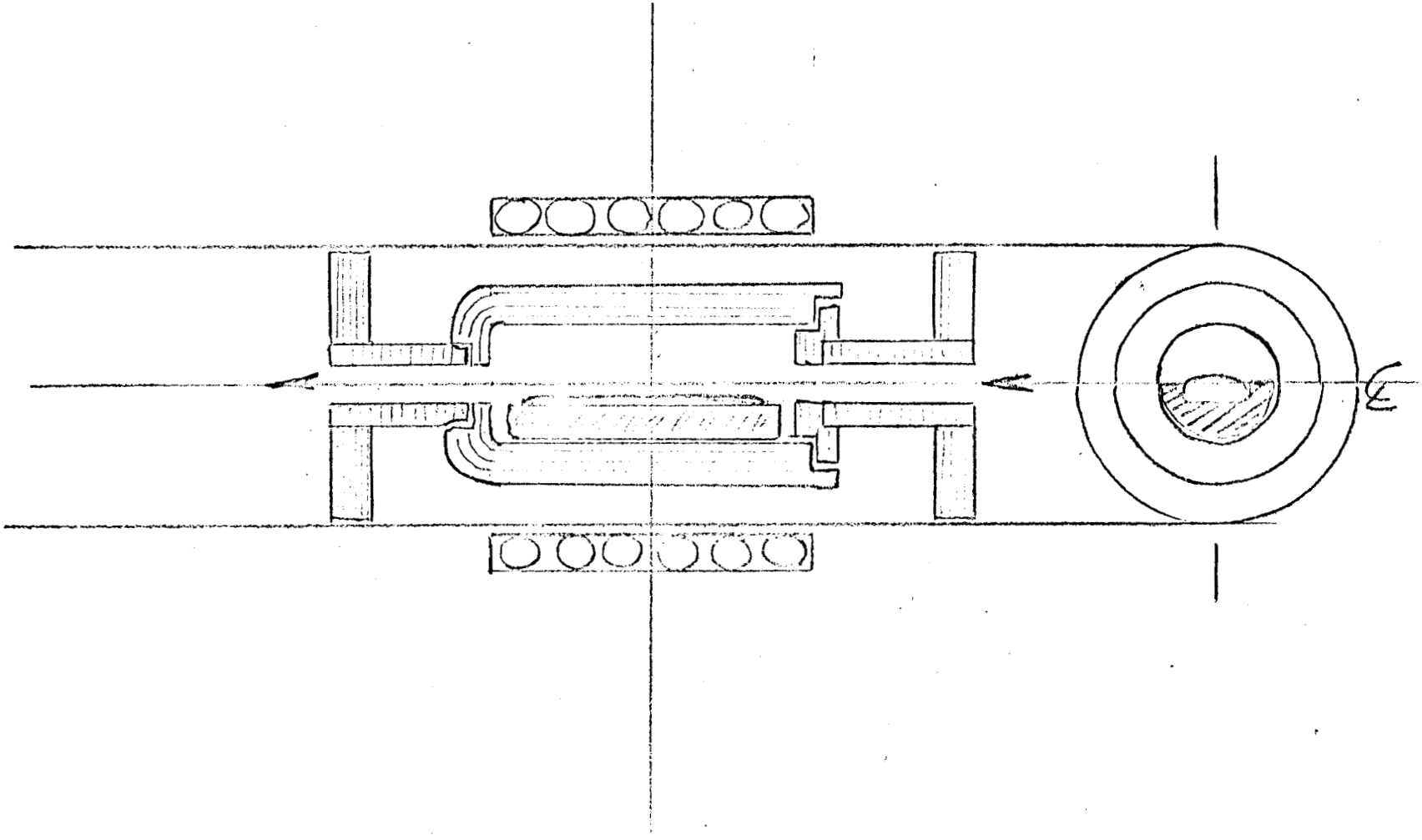


FIG. 3
DEPOSITION CHAMBER

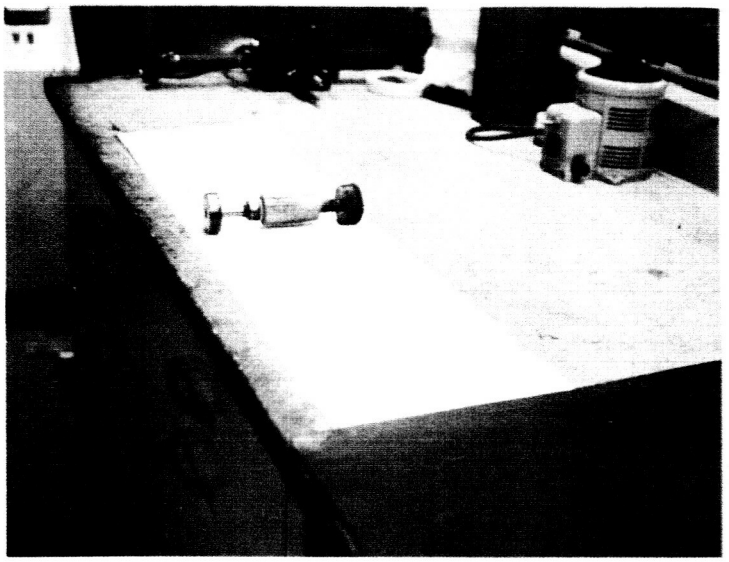


FIG. 4

PHOTOGRAPH OF DEPOSITION CHAMBER

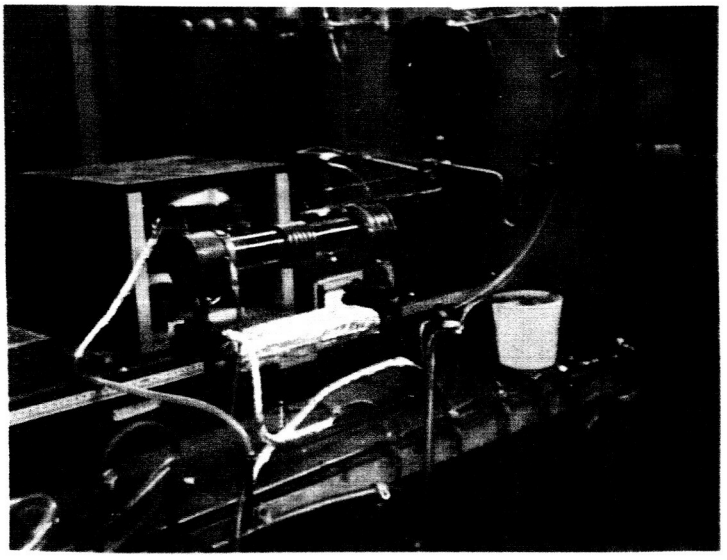
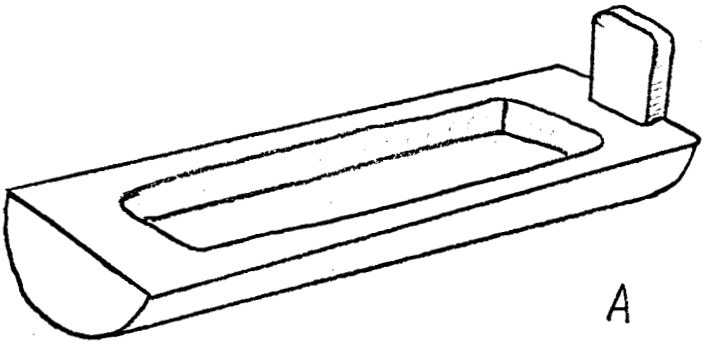


FIG. 5

OVERALL LAYOUT OF EQUIPMENT



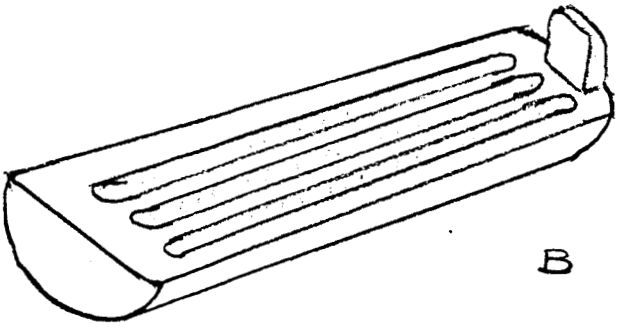


FIG. 6 GRAPHITE DEPOSITION BOATS

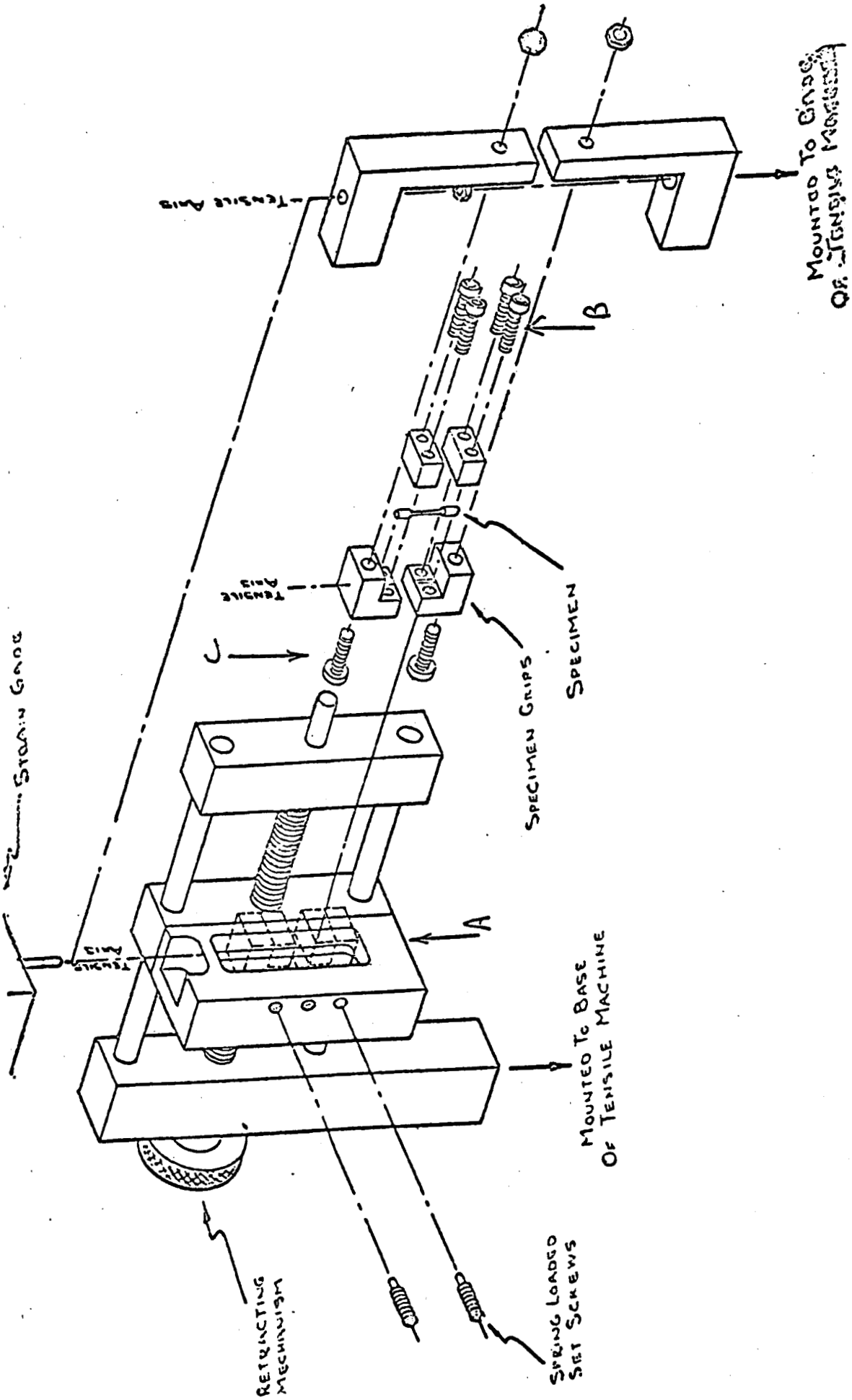


FIGURE 7 SCHEMATIC DRAWING OF MOUNTING RIG FOR P.G. FILAMENTS

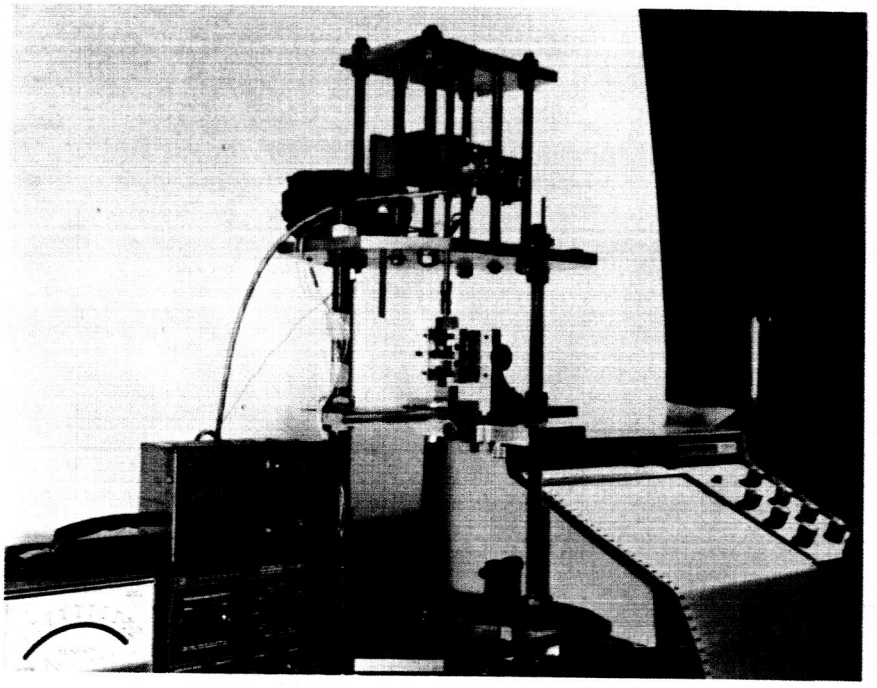


FIGURE 8a. PHOTOGRAPH OF TENSILE MACHINE AND AUXILIARY EQUIPMENT

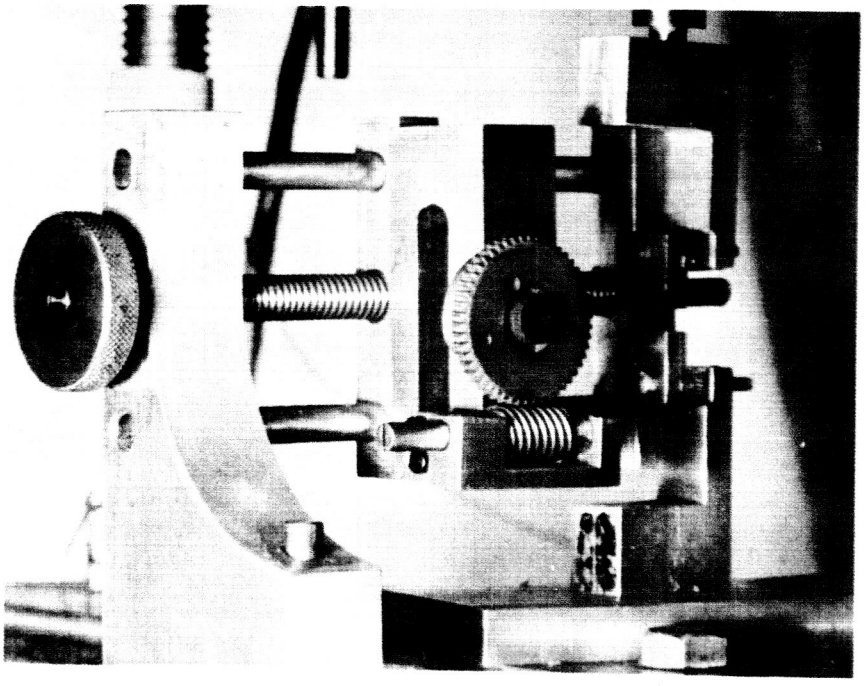
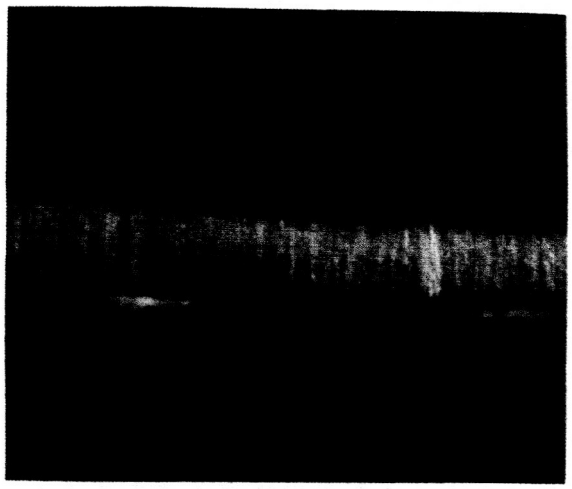
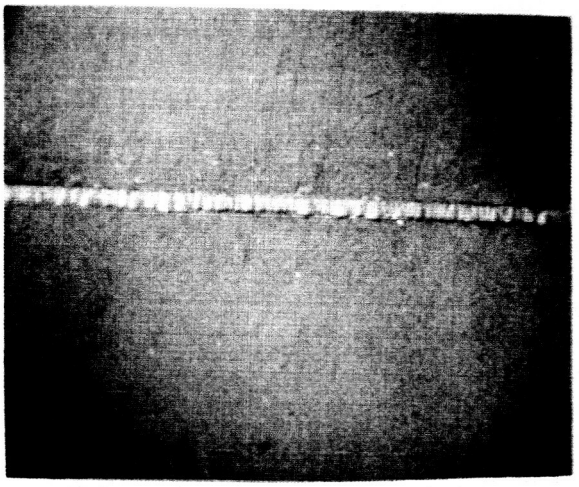


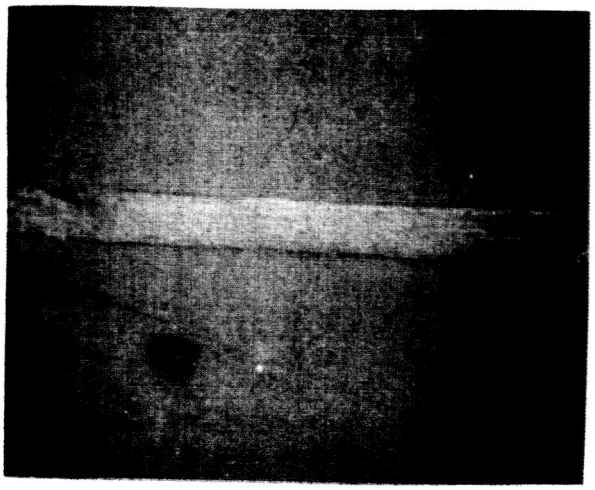
FIGURE 8b. CLOSEUP OF MOUNTING RIG, WITH SPECIMEN AND GRIPS IN POSITION, AND THE HOLDER WITHDRAWN



(a) x400



(b) x 400



(c) x400

FIGURE 9 TYPICAL MICROSTRUCTURES OF P. G. FILAMENTS
a. Deposited on Liquid Gold Substrate
b. Deposited on P. G. Substrate
c. Heat Treated Filament

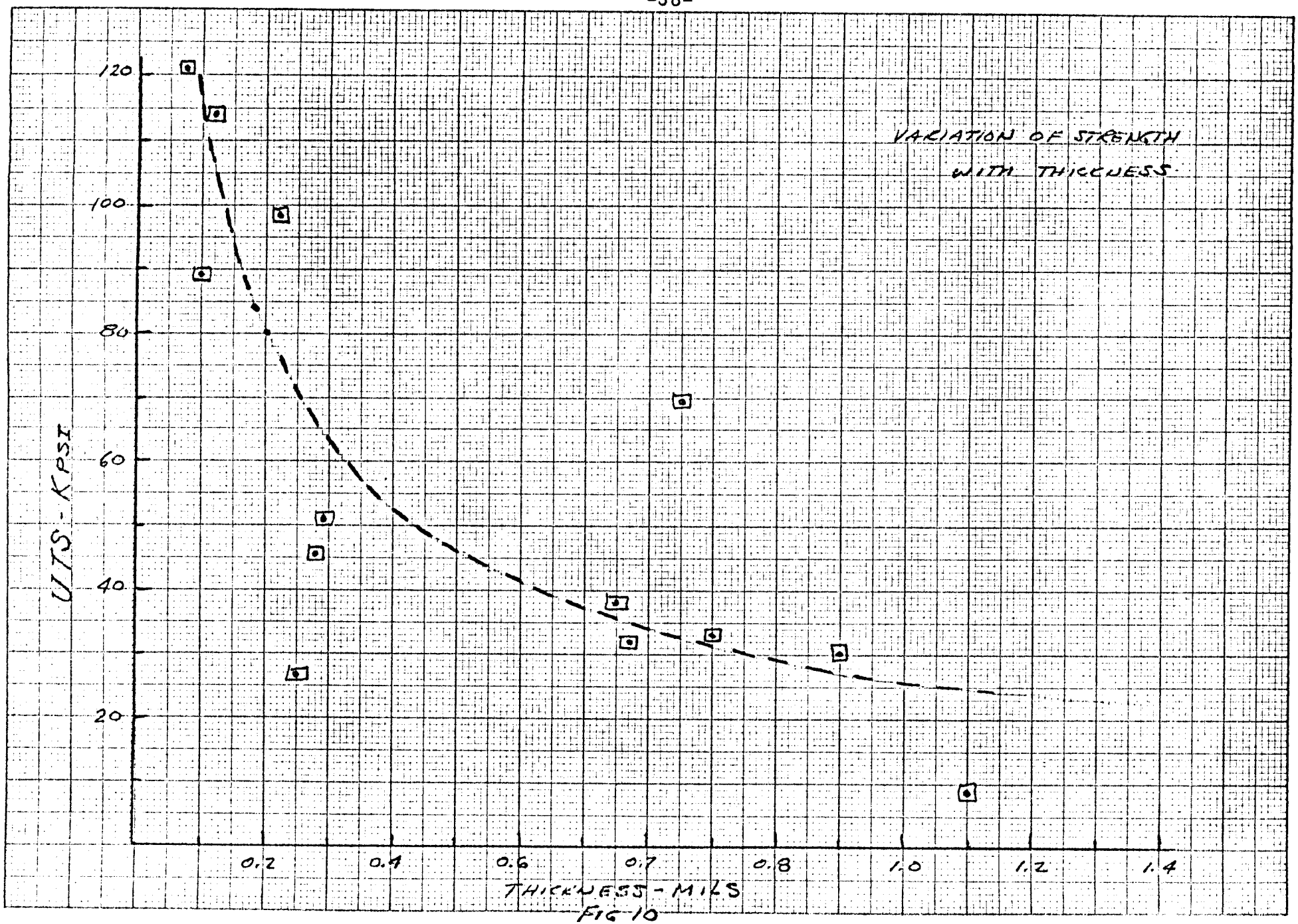

VARIATION OF STRENGTH
WITH THICKNESS
UTS - KPSI
120
100
80
60
40
20
0.2
0.4
0.6
0.7
0.8
1.0
1.2
1.4
THICKNESS - MILS

FIG 10

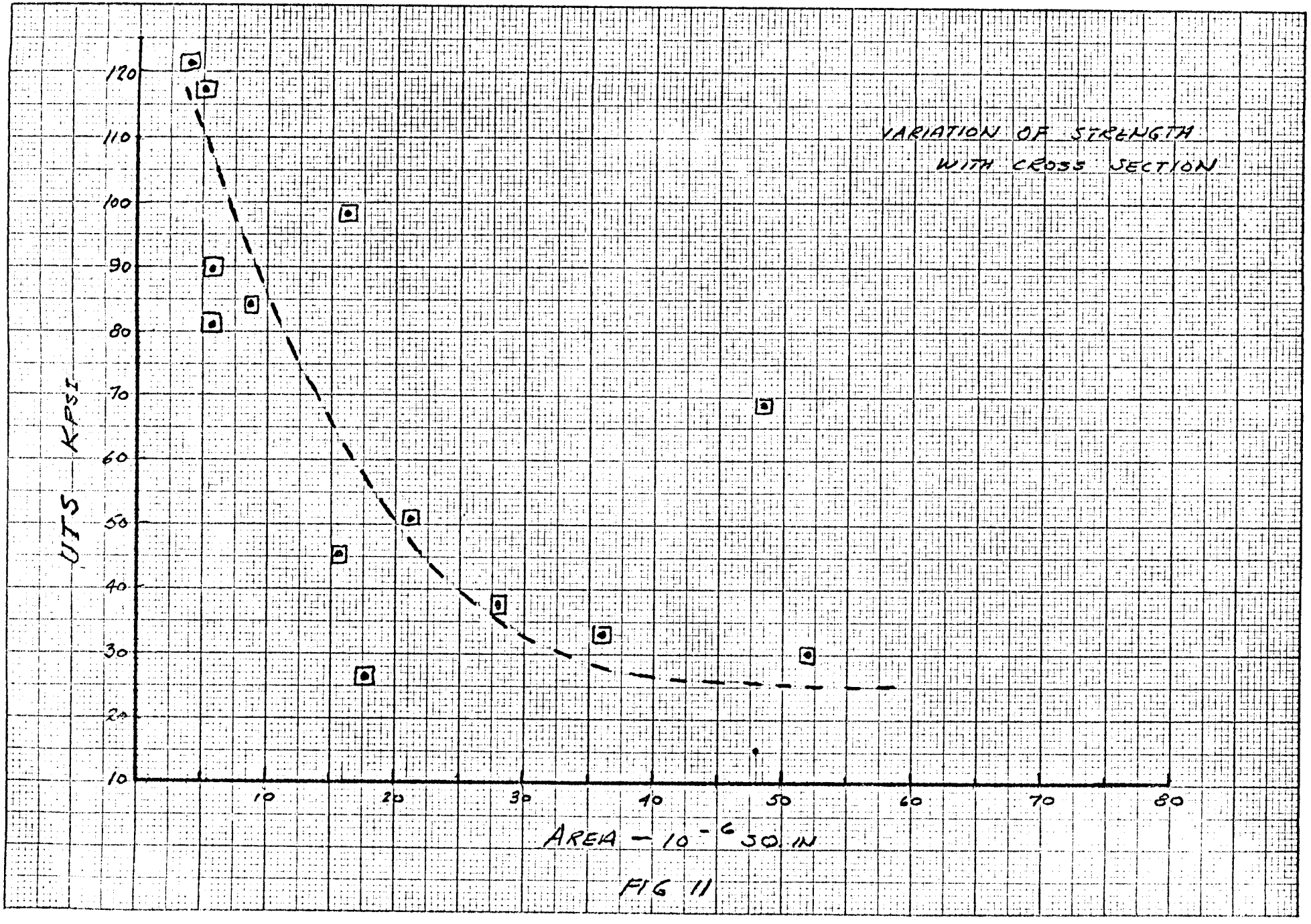

VARIATION OF STRENGTH
WITH CROSS SECTION
UTS KPSI
120
110
100
90
80
70
60
50
40
30
20
10
10
20
30
40
50
60
70
80
AREA — 10⁻⁶ SQ. IN

FIG 11