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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO

VINYL COATING OF GRAPHITE PLATES FOR ULTRASONIC INSPECTION

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VINYL COATING OF GRAPHITE PLATES FOR ULTRASONIC INSPECTION

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ABSTRACT

A process has been developed for application of a thin, adherent vinyl plastic coating to graphite plates to prevent absorption of "coupling fluids" used in ultrasonic inspection. The plates are preheated and dipped mechanically in a fluid plastisol, and the resulting coating is fused in an infra-red heater. No significant attenuation of ultrasonic impulse results from presence of the coating. After inspection, the vinyl sheath may be easily stripped from the plate.

ACKNOWLEDGMENT

Construction of the coating machine described in this report was done by Floyd Hutchinson of Zia Company, Los Alamos, New Mexico. Circuit design and location of control instrumentation in the circuitry was also done by Mr. Hutchinson, who was supplied with only the general specifications desired in the unit.

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Chapter 1

OBJECTIVES

In the use of ultrasonic inspection techniques to determine location and nature of flaws or discontinuities within a given part, "coupling fluids" are used to transmit signals from a transducer located adjacent to the part. Similarly, the signal is transmitted from the work piece through the fluid to a receiving instrument. Variations in signal transmission are indicative of the inner structural features of the work piece, and can be interpreted with a high degree of accuracy and reliability by a skilled operator. Inspection of porous parts by this process has not, in general, been possible because of the uncertainty of location of the fluid-part boundaries.

1.1 The Problem

The specific occasion for an attempt to develop a barrier coating for porous work parts was the requirement that certain graphite components be subjected to ultrasonic inspection at various stages during their fabrication. The preferred coupling fluids are low viscosity organic liquids. These materials readily penetrate the pores of graphite and are not easily removed. Two objections are obvious: loss of the inspection precision, and undue complication of the part production process.

A test of the utility of high viscosity fluids was made by evaluation of mercury as a coupling fluid. It was found not to wet plate surfaces adequately unless a film of alcohol was used to bridge the mercury-plate surface gap. This process had all the disadvantages of the low viscosity fluid technique and was complicated by the problems of safe handling of quantities of mercury. These factors resulted in a request for development of a plastic coating.

1.2 Coating Specifications

1.2.1 Material Properties

The chemical properties of a suitable coating material are not of particular importance except for resistance to the coupling fluid environment. Kerosene resistance was specifically requested. Reaction of the coating with the plate constituents, either during processing or in storage, obviously cannot be tolerated. The specifications for mechanical properties of a suitable coating are also not particularly critical. Dimensional stability under ambient thermal conditions is important; and resistance to impact and abrasion is desirable, although the plates themselves are not especially strong.

1.2.2 Coating to Plate Bonding

Adhesion of the coating to the work piece is of prime importance. Intimate contact of the coating with the plate must be first attained, and then retained, until the coating is removed. A simple test of the degree of contour matching required was made by sealing a section of plate inside an evacuated vinyl bag made from 0.004 inch thick film. Signal transmission was essentially zero. Apparently the bag "bridged" between high points of the rough graphite surface, leaving minute voids at the vinyl-graphite junction. Penetration of the surface pores of the plate is therefore essential. Air bubbles, blisters, etc., are not tolerable for the reason that signal distortion occurs whether the defect is in the coating or at the coating boundary. Although "adhesion" as described is vital to the success of the inspection, the coating must be capable of easy removal in order that it not interfere with other inspection operations or with subsequent processes of plate fabrication.

1.2.3 Dimensions

Plate dimensions vary with the application of the individual parts. A size range of 5 by 8 inches to 10 by 14 inches and a thickness range of 0.2 inch to 0.5 inch are the approximate limits originally involved. The coating must conform easily to the faces of parts and form a film a maximum of 0.030 inch thick on the faces. Edge thickness is not critical. The ideal thickness of coating over the faces was believed to be one as thin as possible consistent with maintenance of other properties. Uniformity of coating over the faces of any one plate, or from plate to plate, within 0.005 inch was set as an objective to eliminate frequent recalibration of inspection equipment. Presence of drip marks or "runs" on the faces has the same effect as the presence of internal flaws in the coating; signal distortion is the result. The occurrence of occasional small coating faults can be approved only if the affected area is smaller than the resolution demanded of the inspection process.

1.2.4 Application Conditions

Specifications relating to method of coating application to parts for inspection fall into two categories. Damage to the plates during coating is not permitted; and process time required should be small per unit for reasons of economy and to prevent interference with other work. The first requirement precludes use of solvent dip processes and requires that temperatures not in excess of 180°C be involved. The second requirement indicated immediately that a mechanized procedure be developed.

In the first test program alone, there are a large number of plates to be processed. The uncertainty as to whether "green" plates (baked, but not "graphitized") would require coating produced the temperature limitation because the baking temperature attained is 180°C and additional heat would cause decomposition of the furane resin binder. There was originally some possibility that plates would be inspected in the impregnated stage, where the identical situation exists. Multiple stage inspection by ultrasonic technique could also have resulted in as many as three coating operations per plate.

In addition to the complication of variations in plate size and thermal stability, the chemical composition of the plates with respect to various experimental fillers was to be intentionally controlled over a range sufficient to produce variation in heat capacity from piece to piece. Accordingly, a process which was not adversely affected by this factor was required.

1.3 Choice of Process

1.3.1 Advantages of Vinyl Coatings

A coating system based on a vinyl plastisol was chosen for evaluation for several reasons, the major incentive being considerable local experience with this type material and process. Limited experience with hot melt coatings of the polyethylene-paraffin-polyisobutylene type and the cellulose acetate butyrate type indicated that they would also be suitable, but would require more development time. Both these systems would present problems of fire hazard and emit noxious fumes. The vinyl system is capable of great versatility because in addition to its flexibility in formulation, it can be used with lower temperatures, involves no solvent or low temperature volatilization of ingredients, and presents no fire hazard or serious toxicological problems. Almost any vinyl plastisol system will meet the basic material specifications. Semi-automated vinyl coating processes are rather commonly used on an industrial scale with good results in terms of production rate and economy.

1.3.2 Limitations of the Vinyl Plastisol Process

The greatest uncertainty in choice of a vinyl system was in its sensitivity to factors which could prevent conformance to dimensional specifications. A major consideration is the increase in viscosity of plastisol formulations with age. A compound may double its initial viscosity in a week and redouble the initial viscosity in an additional two weeks. Frequently thixotropic characteristics develop with age. Coating thickness is a directly related function of viscosity.

Fusion time for a plastisol varies with the formulation, and physical properties vary tremendously with fusion temperatures and time at temperature. Age of a plastisol also affects the fusion conditions required to develop any given minimum physical specification.

Solution of the problem of attaining and maintaining intimate physical contact with the work plate surface while producing a coating which could be easily stripped was also doubtful. There is no great difficulty in achieving either of these objectives independently; however, the two specifications are nearly incompatible.

Chapter 2

COATING

A large number of plates have been successfully coated on a test production basis. No coating has failed to meet specifications. The present process has been found much less efficient for application of coatings to plates which have not been graphitized. Early adoption of an inspection process which includes ultrasonic testing of graphitized plates only in a semi-machined stage eliminated the necessity of additional process development work.

2.1 Process Cycle

In brief, the process cycle involves the following steps: (1) fixing the work pieces in jigs, (2) preheating the plate, (3) dipping the plate into plastisol, (4) fusing the coating, (5) cooling, (6) removing the jig, (7) inspecting, and (8) coating of jig contact points. The over-all time required per plate for steps (1) through (4) is 5 to 6 minutes. A preheat time of 180 seconds, a 2 second dwell in the tank during the dip cycle, and a fusion time of 22 seconds are controlled by interval timers. Speeds of dipping and withdrawal are controlled by a combination of rheostats and gear reduction boxes. Twenty plates can be processed in approximately 2 hours operating time. The coated plates are then allowed to cool overnight as a matter of convenience. (They could be handled within a half-hour, if necessary; and in emergencies, they could be water cooled by immersion and handled in 5 minutes.) After cooling, the plates are removed from the jigs, and jig contact points are coated with "Spray-O-Namel."

This material is tentatively identified as an acrylic emulsion packaged with aerosol propellant by the Illinois Bronze Powder Co., Chicago, Ill., as product number K-225. After monitoring, the coated plates are packed for delivery to inspection.

2.2 Equipment

2.2.1 Vacuum De-aerator

A large scale unit for de-aeration of freshly prepared plastisol was designed and constructed. Details of construction are shown on LASL Drawing 42Y-44660, sheets D-1 through D-3. Essentially, this item is a large vacuum chamber which contains a series of sloping plate baffles arranged at a slight angle from horizontal. Material introduced at the top of the unit flows over the baffles in a thin film, allowing entrapped air to be easily and rapidly removed. By use of this equipment, a major source of bubbles and blisters in the coatings has been eliminated. The storage tank capacity of the de-aerator is about 8 gallons.

2.2.2 Plate Mounting Jigs

The work pieces are mounted in especially designed clamping jigs which make contact with plate edges on two points at each of two diagonally opposed corners. The use of diagonal mounts allows free drainage of surplus plastisol from plate edges during withdrawal of the part from the dip tank. This, in turn, alleviates, or eliminates, the problem of "runs" across the faces of the plates. One set of contacts on each jig is fixed; the other is spring mounted so that installation of a part in the fixture is quite simple unless there is large variation in the size of consecutive plates. The clamps are shown in several positions in the accompanying illustrations. Plastisol build-up on the jigs is a problem. After 3 to 5 cycles, they are placed in an oven to fuse the vinyl material and make stripping easy. The high thermal conductivity of the jigs, compared to that of the plates, prevents complete fusion of the coating in normal processing. Materials used in construction of the jigs are dural and stainless steel for parts which are in regular contact with the plastisol.

2.2.3 The Coating Machine

The machine which controls the mechanics of the coating operation consists of two basic elements: dipping apparatus and conveyor-oven. Figures 1, 2, and 3 show the entire unit from three points of view. As will be explained in Chapter 3, the unit would not be exactly ideal for production work on the cycle now in use. The present model is the product of changes which were made in the course of the development program.

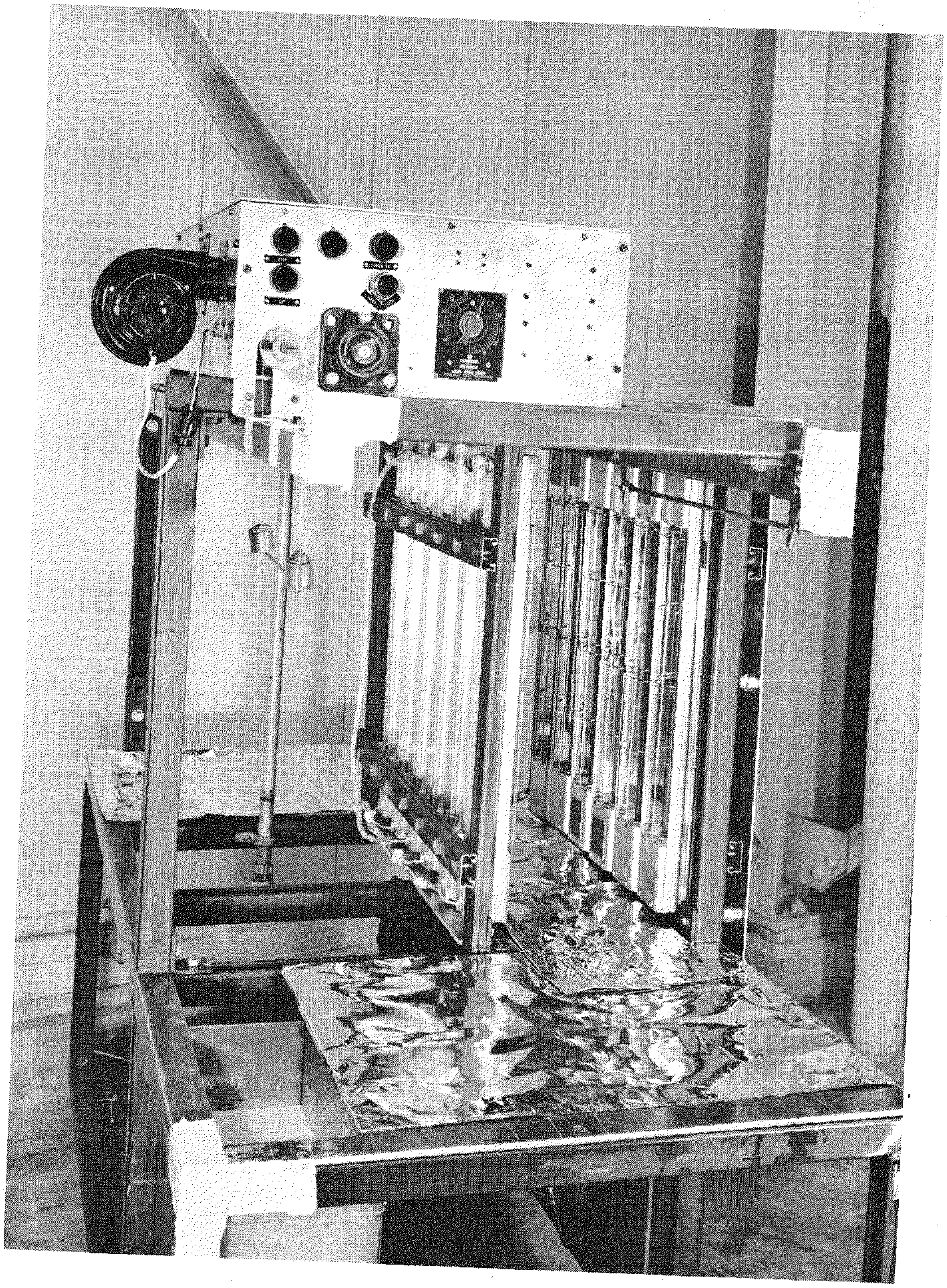

Fig. 1. Coating machine, dipping end.

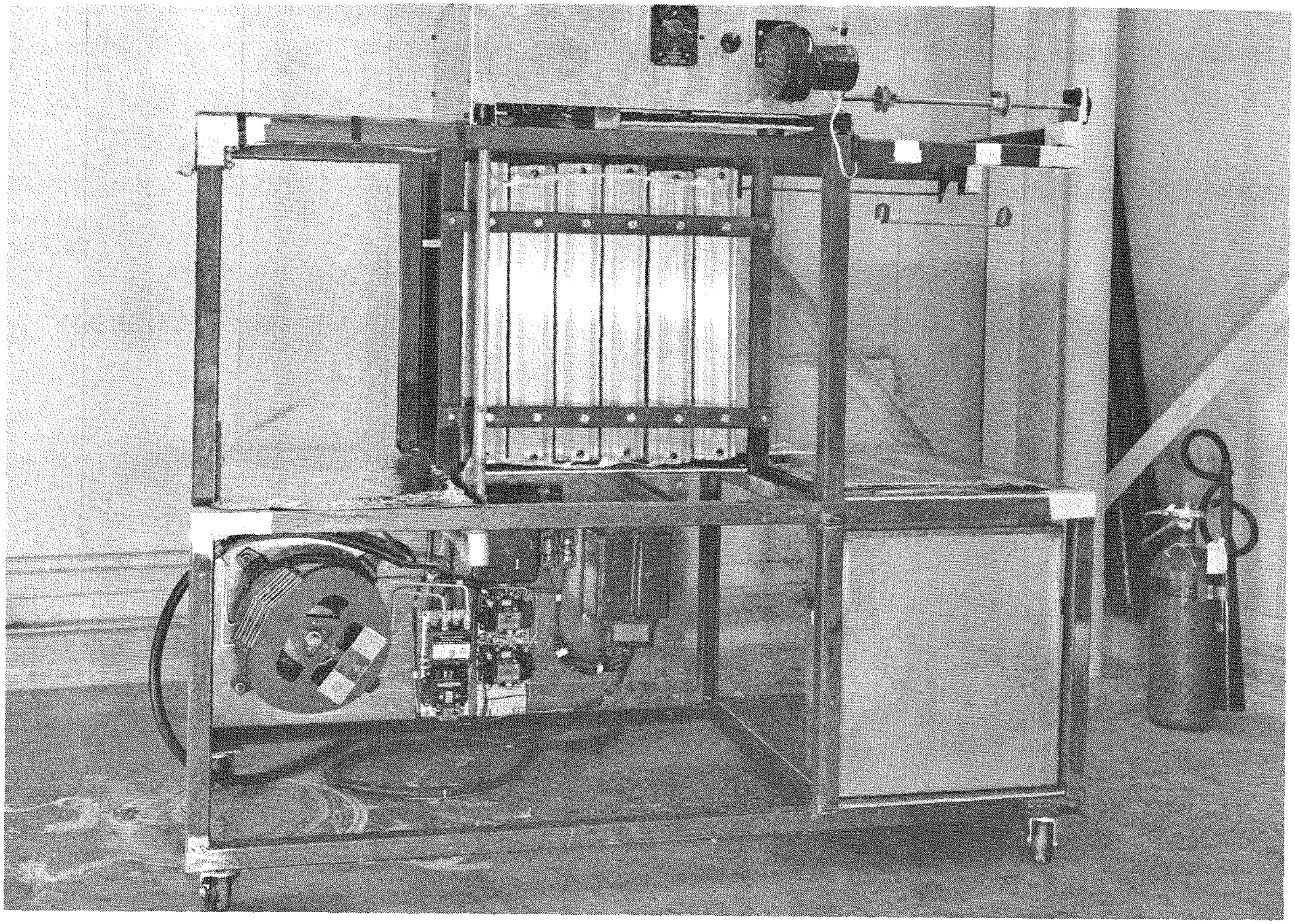

Fig. 2. Coating machine, rear view.

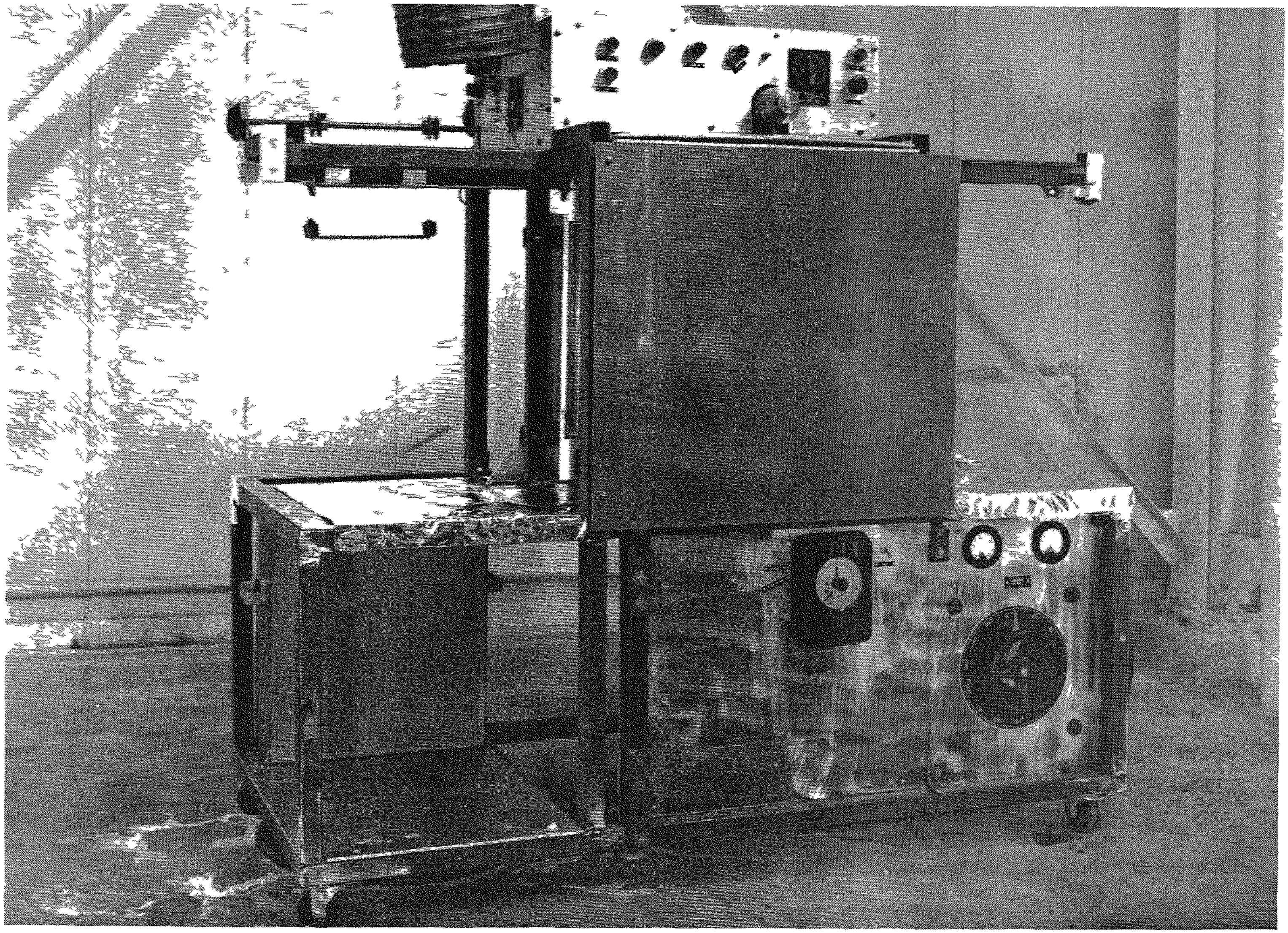

Fig. 3. Coating machine, front view.

2.2.3.1 Dipping Apparatus

Figure 4 shows a close-up view of the dipping apparatus with a test plate suspended in position to be lowered into the tank of plastisol. The entire dipping operational sequence, between installation of the plate on the dip rack and its removal, is machine controlled. Limit switches control rack travel. Figure 5 shows a close-up view of the dip control panel area. Provision has been made for selection of either semi-automatic operation or manual "inching" control. Time of dwell in the tank is variable. The initial rate of withdrawal after dwell is also variable by means of a rheostat. The time interval during which the reduced rate of withdrawal is effective is also variable. The blower in the foreground of the photograph serves to cool the electrical circuitry and is not otherwise a part of the operation. Speeds of operation are: down-travel - 65 inches/minute; initial withdrawal rate - 37 inches/minute; return is completed at a rate of 83 inches/minute. The over-all time cycle is 50 seconds, including the brief dwell time in the tank and electrical relay operation. Figure 6 shows the plate at its terminal position after dipping and prior to transfer to the conveyor rack for fusion.

2.2.3.2 Conveyor-Oven

Preheat and fusion stages of the coating operation are conducted in the same portion of the machine. A motor-driven, reversible conveyor rack operates through the heat zone. The heaters, of which there are 12, are mounted vertically, side by side in opposed banks separated by

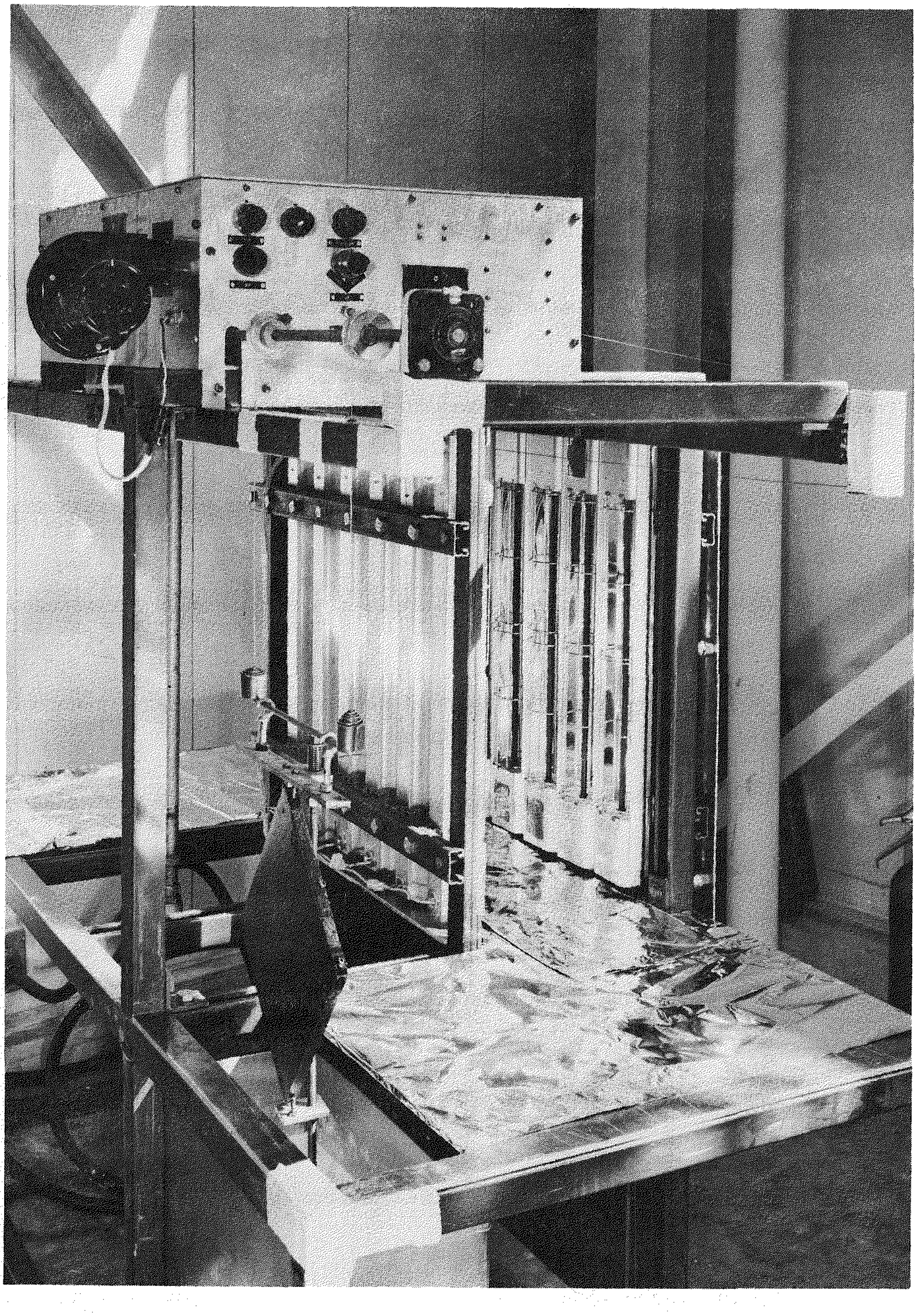

Fig. 4. Test plate mounted for dipping.

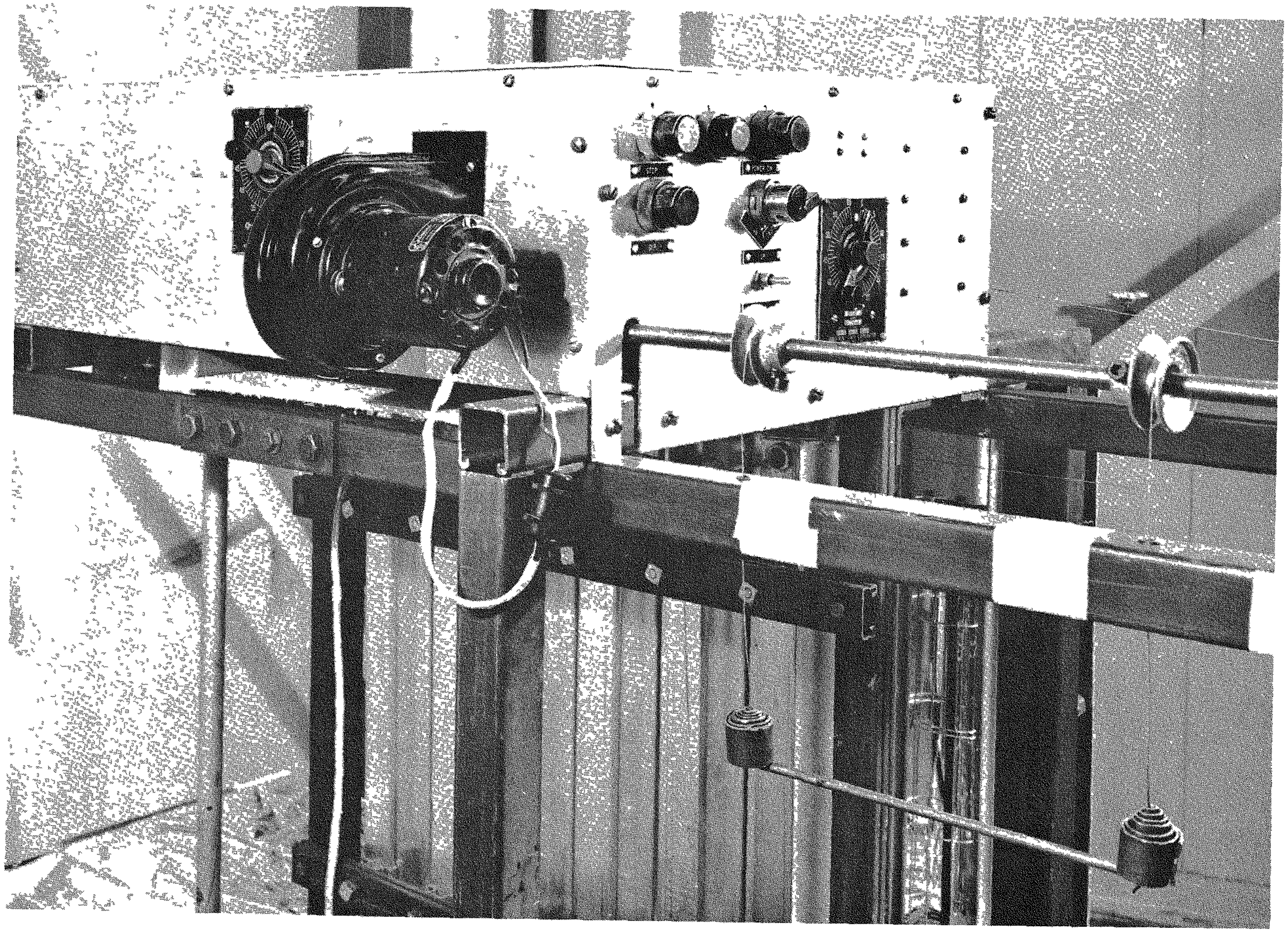

Fig. 5. Dipping stage controls.

approximately 12 inches. The mounting is constructed to allow adjustment of this distance. The heater units are Chromolox Radiant Units, Catalog No. 2083 B, supplied by E. L. Wiegand Co., Pittsburgh, Penna. They are wired in parallel. Heating rating is 800 watts at 240 volts. The double bank is presently operated at 40 amperes, with input controlled by means of a large transformer. The particular model of heater selected by the manufacturer is for use in vinyl baking and fusing operations because of its high output in the far infra-red range. A specific advantage is that the radiation is "color-blind," and heating rates are not affected materially by the type of surface exposed. No shielding, other than that required for operator protection, is used to prevent convection or conduction losses.

Figure 7 shows a test plate mounted on the conveyor rack ready to be passed through the heating zone. Figure 8 shows the control instrumentation on the heater circuit. Power input to the heaters is fully controlled, and, in addition, there is a proportioning control to allow power input to be cycled off-and-on on a time percentage basis. The cycle is 60 seconds. This instrument is currently set to supply power 100 percent of the time.

The conveyor control system is shown in Figure 9. Power transmission to the conveyor rack is by means of drum-cable drive. Limit switches control travel and position the rack and its load centrally in the heat zone where an interval timer controls dwell. The circuit is arranged for semi-automatic operation or "inching." Conveyor transit speed is 130 inches/minute.

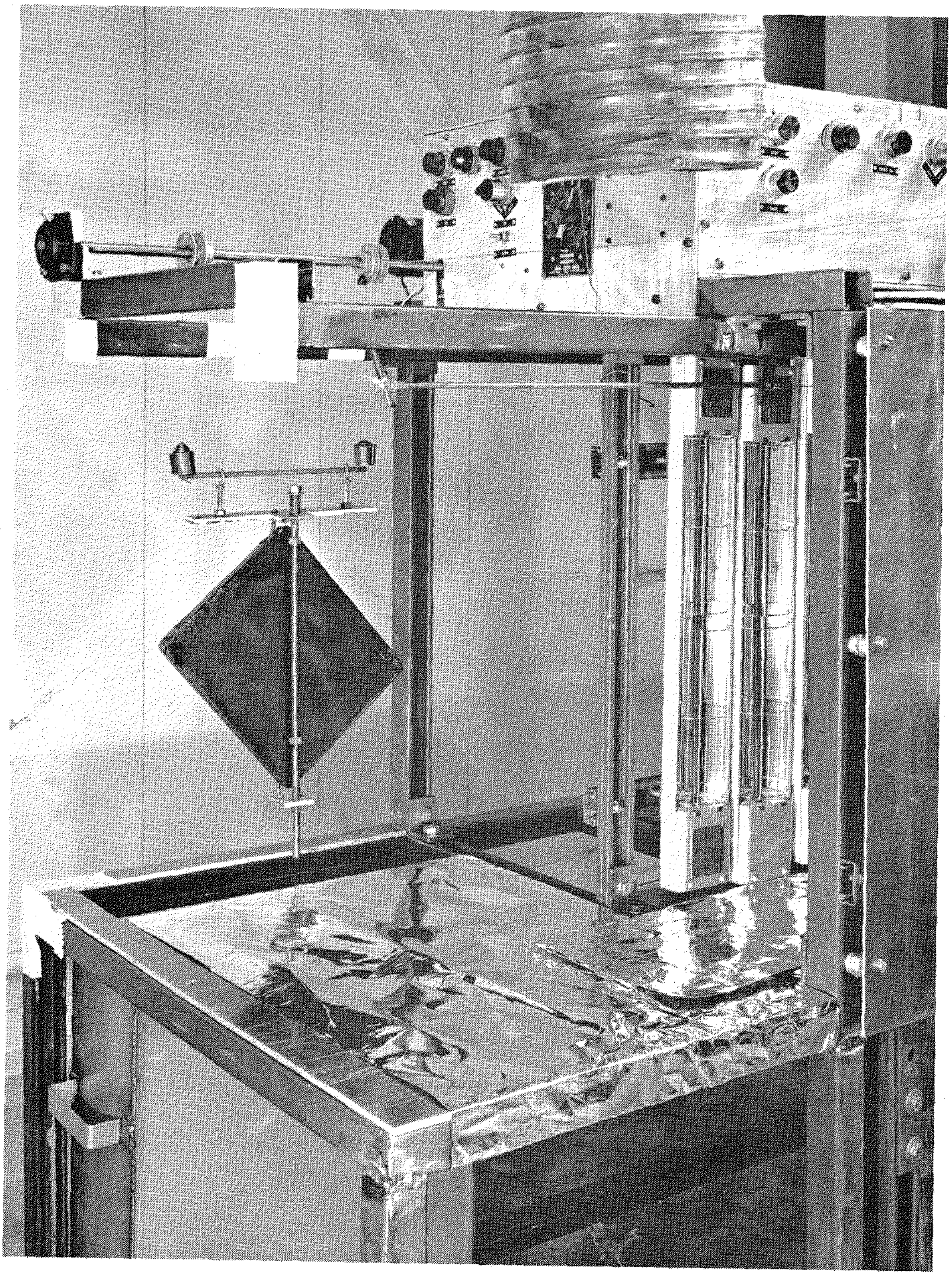

Fig. 6. Dipped plate.

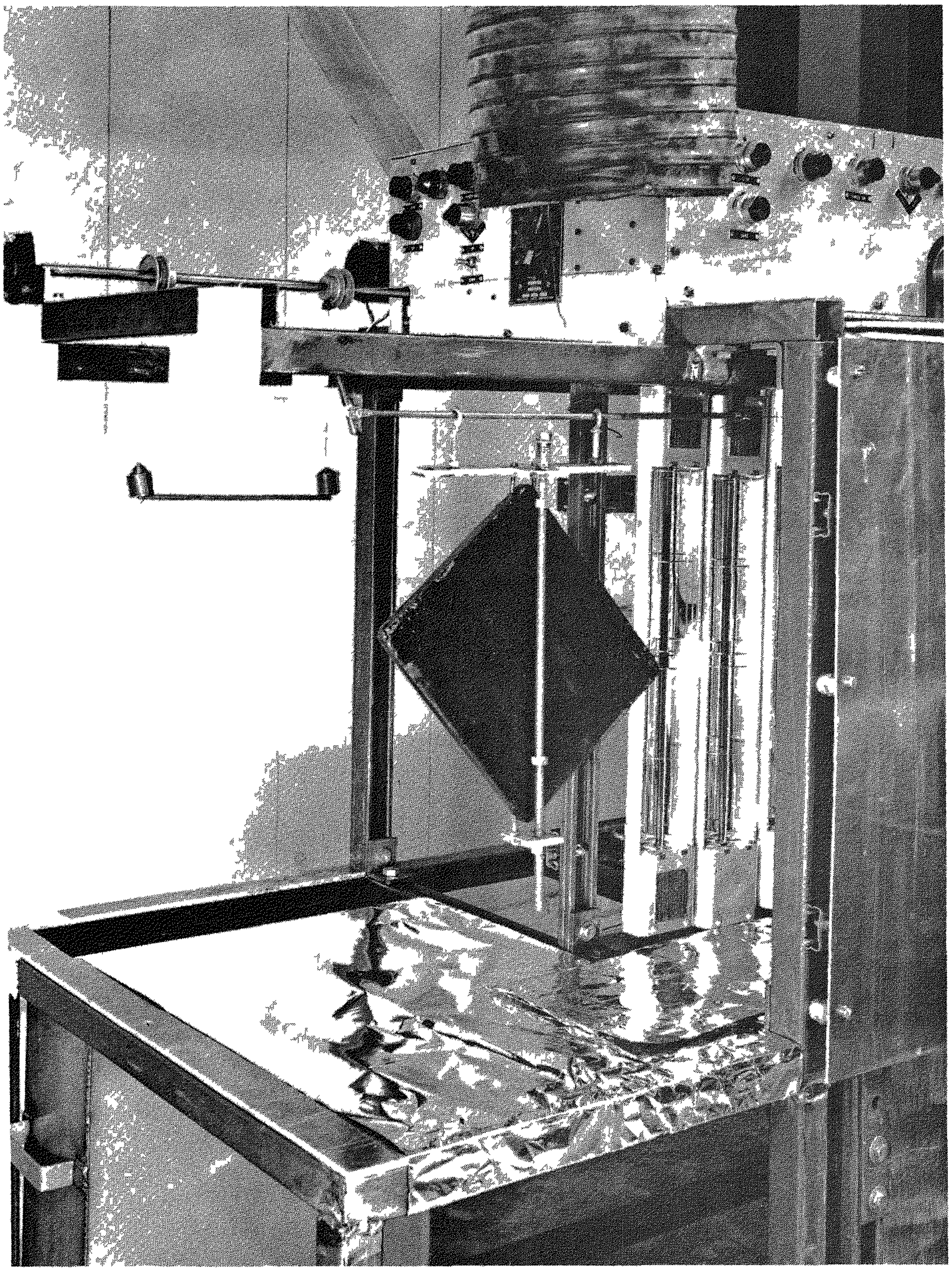

Fig. 7. Plate mounted for fusion of coating.

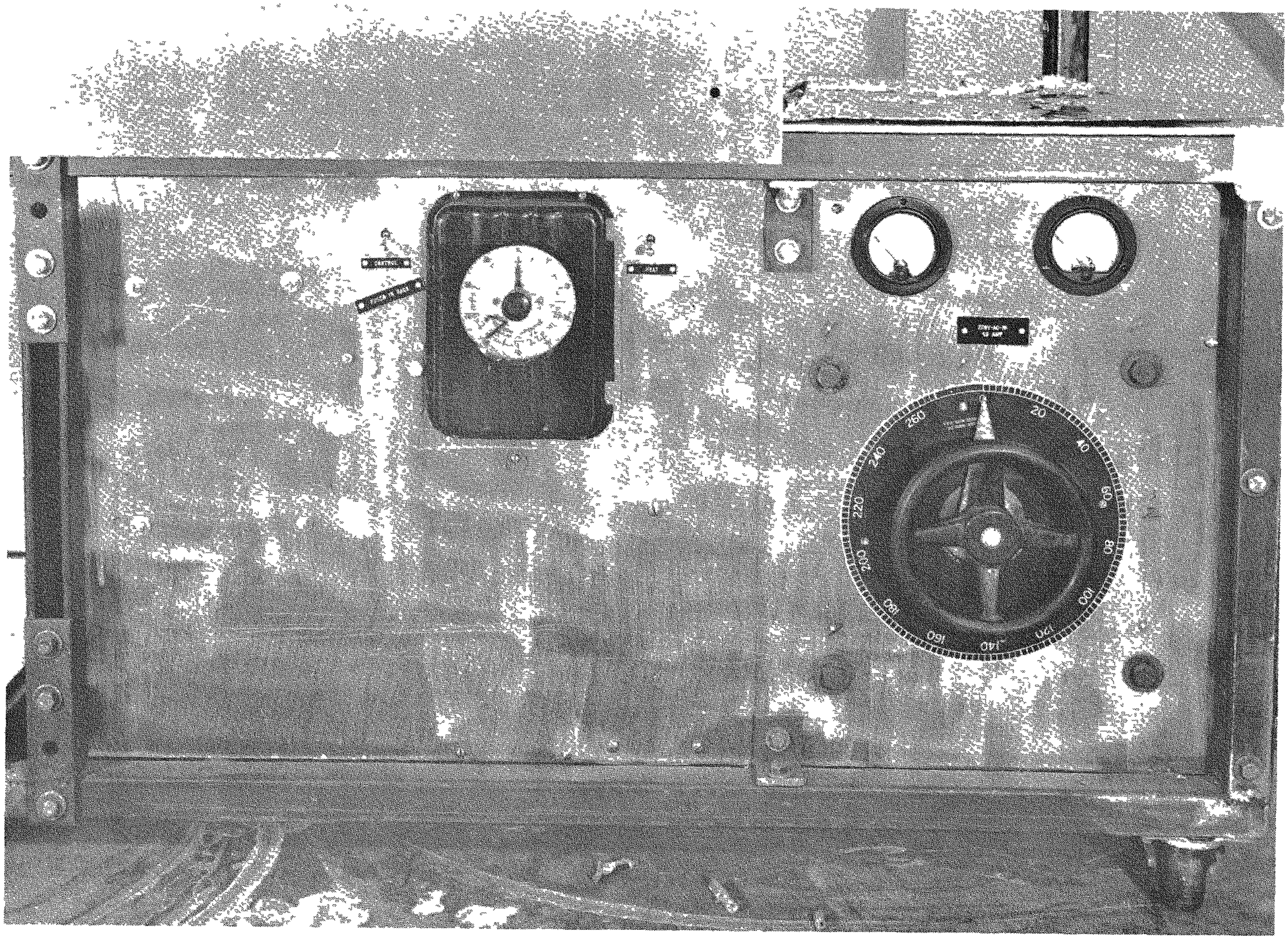

Fig. 8. Heater controls.

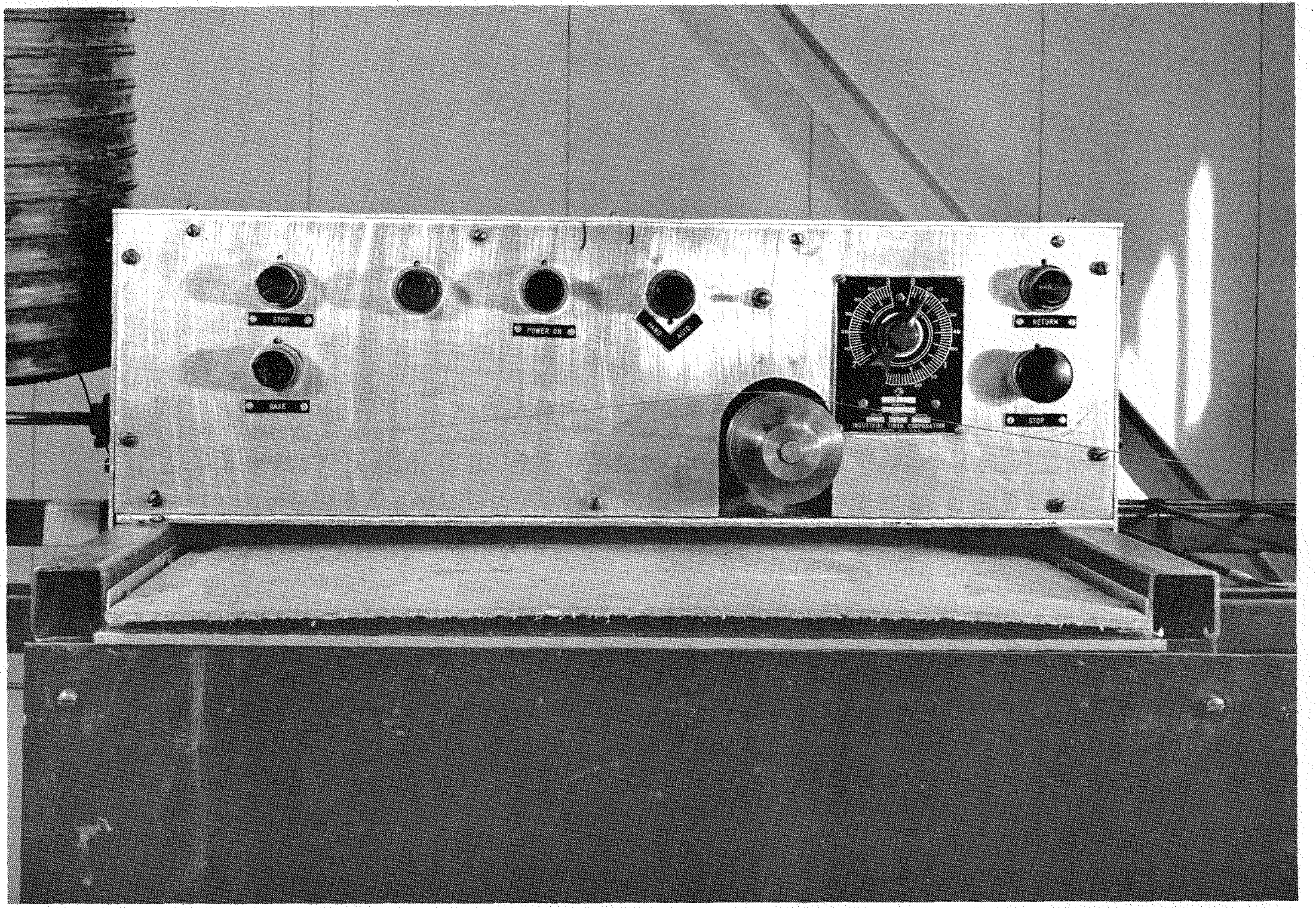

Fig. 9. Conveyor controls.

Figure 10 shows a plate in the heating zone at the point where the timed dwell period occurs. The plate in Figure 11 is at the terminal position of conveyor travel at the end of the fusion cycle. This spot is adjacent to cooling racks built over the end of the machine base.

2.3 Plastisol Composition

A vinyl plastisol is a suspension of finely divided polyvinylchloride in an organic plasticizer mixture which, when subjected to heat, becomes a homogeneous system. Specific plasticizers are usually selected to impart specific properties to the finished, fused compound. Tensile strength, brittle point, solvent resistance, elastic modulus, elongation, etc., are controlled in this way, as well as by choice of the base vinyl chloride resin. Heat and light stability are achieved by the addition of "stabilizers" which usually are capable of reacting with hydrogen chloride produced in the degradation of vinyl chloride. (Free HCl catalyzes vinyl degradation and must be removed from the system in some manner.) Freshly prepared plastisols have only the outer portions of the individual vinyl particles affected by the plasticizer system present. Solvation proceeds with age, and the viscosity of the mixture rises. Limited control of this phenomenon is possible by use of plasticizers which have low solvating power at temperatures below those used for fusion. Plasticizers which have this property are seldom capable of producing desirable end properties alone and generally require higher fusion temperatures for the finished compound.

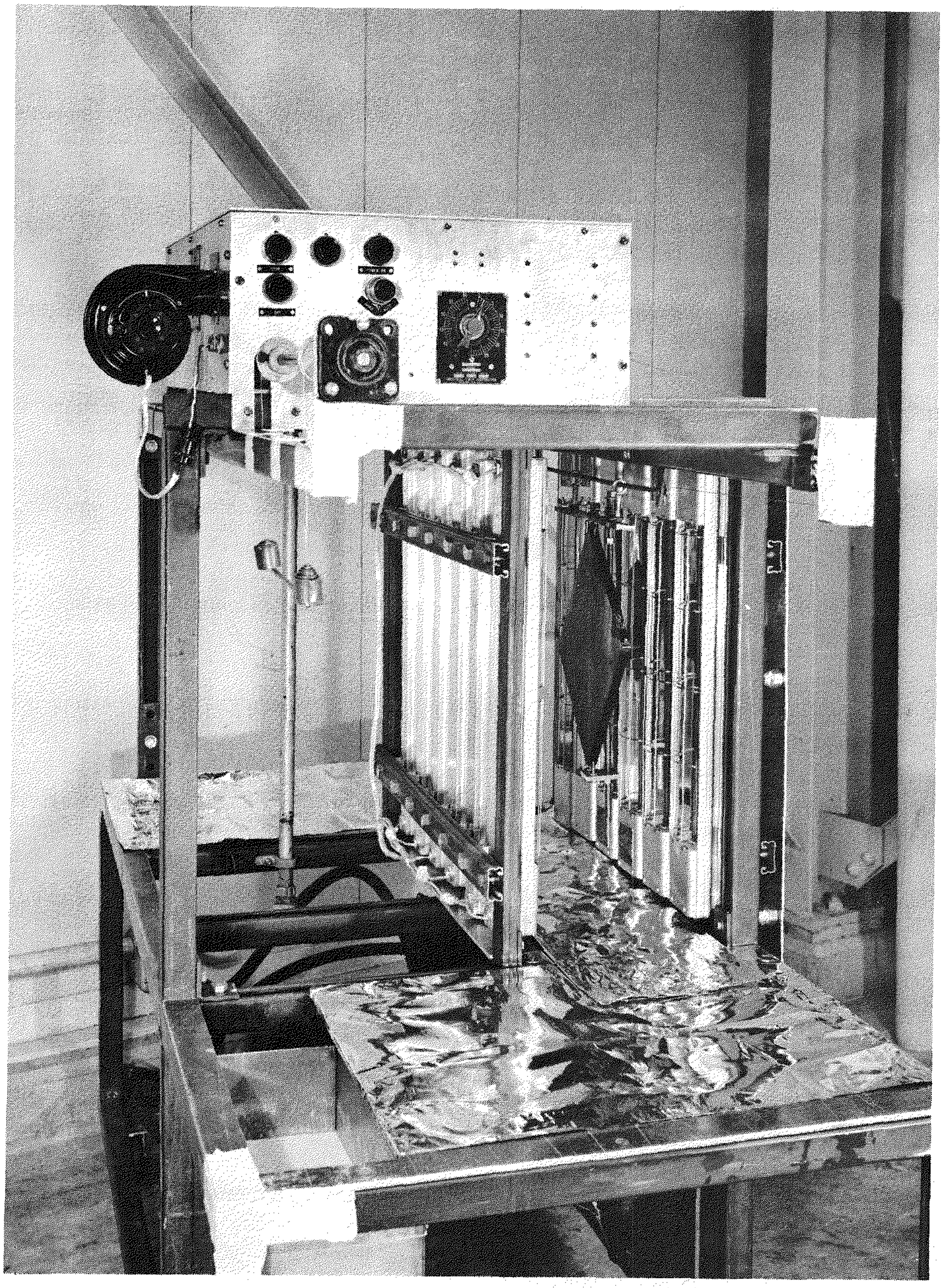

Fig. 10. Plate inside heating zone.

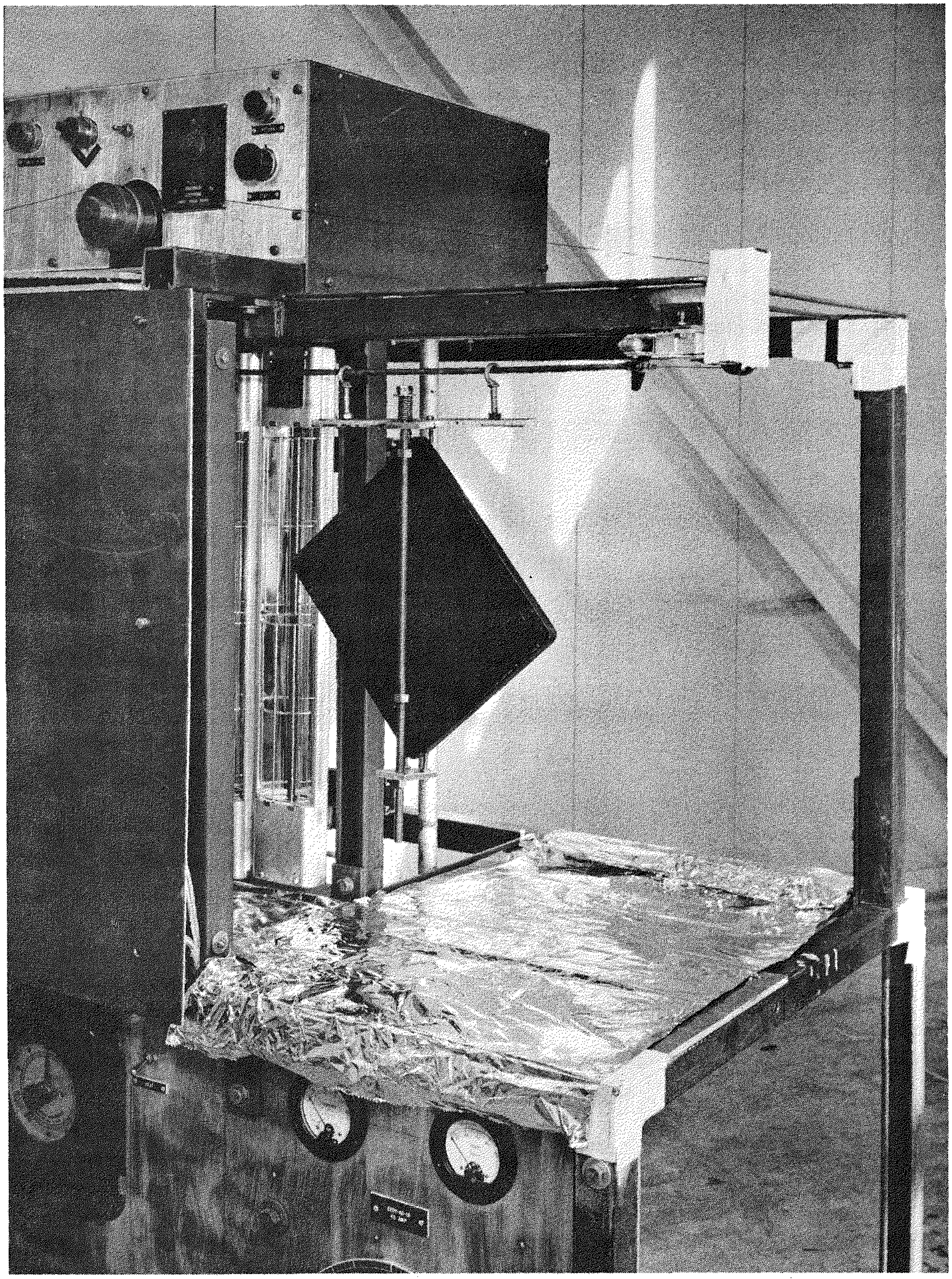

Fig. 11. Coated plate at completion of the process.

2.3.1 Coating Compound Formula

The formulation for coating is as follows (proportions given are by weight):

TABLE I

COATING COMPOUND FORMULA

Ingredient	Parts
1. Geon 121	100
2. Flexol DOP	90
3. DIDA	25
4. Flexol 4GO	20
5. Paraplex G-60	10
6. Barinac	4

(1) Geon 121 is a high molecular weight vinyl chloride polymer of minus 200 mesh size especially developed for use as a plastisol base. The manufacturer is B. F. Goodrich Chemical Co., Cleveland (15), Ohio.

(2) Flexol DOP is a primary plasticizer manufactured by Carbide and Carbon Chemical Co., Division of Union Carbide and Carbon Corporation, New York (17), N. Y. Chemically, it is di(2-ethylhexyl) phthalate.

(3) DIDA, obtained from Monsanto Chemical Company, Organic Chemicals Division, St. Louis, Mo., is diisodecyl adipate. Its function in the compound is to serve as a partial viscosity control agent.

(4) Flexol 4GO is another Carbide and Carbon product included here as an aid to increase low temperature flexibility, reduce compound viscosity, and control viscosity rise with age. The chemical identification is polyethylene glycol di(2-ethyl hexoate).

(5) Paraplex G-60 is a polyester type plasticizer which serves to increase hardness and abrasion resistance of the fused compound. It also acts as a light and heat stabilizer to some extent, without adversely affecting the transparency of the compound. The Resinous Products Division of Rohm and Haas Co., Philadelphia (5), Penna., is the source for this material.

(6) Barinac (barium ricinoleate) is the primary system stabilizer. It is supplied by the National Lead Co., New York (6), N. Y.

2.3.2 Compound Preparation

The materials listed in Table I are blended in an Ajax heavy duty mixer. The preferred order of addition is (3), (2), (4), (5), (6), (1), with reference to the order in which the ingredients were tabulated. Make-up time is about one hour for a 12 gallon batch. The majority of the time is consumed in addition of the Geon and Barinac; all other components are liquids. To prevent "speckling" of the coating caused by agglomeration of the resin or stabilizer, these materials are "dusted" into the mixing vessel in increments. Mixing is continued for at least 2 hours after the last addition. De-aeration may be done any time prior to use, but is normally completed within a day or two. De-aeration time per batch is of the order of 90 minutes at 0.5 inch mercury pressure. The plastisol is then stored for 7 to 10 days before use in order to allow time for the initial viscosity increase rate to level off. Two weeks' storage is a reasonable maximum if coating thickness control is to be maintained without adjustment of the coating machine controls.

A batch of material is sufficient to fill the dip tank on the machine once and to replenish the supply as material is withdrawn during a week of normal operation. The tank is drained and cleaned weekly or after 60 to 120 plates have been processed. Two factors influence the life of a batch of material: increase in viscosity caused by thermal abuse and contamination of the plastisol by spalling of graphite. Increased coating thickness results from the first factor; contaminated coatings result from the second.

2.4 Coating Process Evaluation

Inspection records for coatings applied to graphitized and semi-machined plates show no rejects among the last large batch of plates processed. In recent attempts to coat "green" plates, a reject (re-run) rate of 30 percent was experienced, and no plate was coated to conform fully to the specifications with respect to bubbles and blisters.

Earlier in the program "green" plates were coated successfully to all specifications. It is believed that these plates had undergone a more complete polymerization of the furane binder, or had at least been processed at higher temperatures than the later ones. Another factor affecting the apparent difficulty in duplication of experimental results is that the same plates were reprocessed many times during tests on compounding and machine adjustment. The binder may have been freed of its volatile content in this manner. No other explanation for this discrepancy is offered.

Coating thickness control has been good on the basis of occasional spot checks. Average thickness over one face of a plate is about 0.012 inch, with a range of no more than 0.005 inch. Average thickness variation between faces of an individual plate is negligible, and variation in average thickness from plate to plate is within 0.005 inch.

Transmission of ultrasonic impulses through a coated plate is in the range of 68 to 75 percent of the original signal strength at the frequencies used. This is the same range found for uncoated plates. Coating thickness variations of the magnitudes noted above do not affect the signal strength.

Chapter 3

EXPERIMENTAL BACKGROUND

Because the development of this coating process was undertaken with specific objectives in view, investigations of variable factors and their effects on the properties of the coating were limited. No attempt was made to provide the best possible coating system or to develop the best possible equipment. Such investigatory work as was done was for the sole purpose of providing a process capable of meeting the original specifications in as short a time as possible. Changes made in coating formulation and equipment design or control features are more properly assigned to the category of "trouble shooting" than that of "research."

3.1 Equipment Development

Problems in development of suitable equipment were confined largely to the coating machine and the clamp jigs used to mount the work plates. The de-aerator design is not particularly complicated, and there was no trouble experienced with its operation other than with minor relocation of vacuum line connections.

3.1.1 Jig Design

Several models of clamp jigs to hold plates were constructed and tested. Point contacts, ball contacts, and combinations of other geometries were tested. All early model clamps were designed to position the plate edges parallel to the tank sides. Trouble with "runs" down the faces of the plates was repeatedly encountered whether the clamp cross bar was immersed in plastisol or mounted so as not to enter the liquid. It was eventually decided that the real source of the difficulty was pick-up of a pool of ungelled plastisol on the upper edge of the plate. Slight vibrations in the withdrawal mechanism caused this puddle of material to spill and form random "run" marks on the faces. The change to the diagonal plate mounting system prevented accumulation of these pools.

3.1.2 Coating Machine Features

The concept of the coating machine was originally based on attempts to use a simplified process of coating which did not involve preheating the plate. For this reason, only one conveyor-oven assembly was included. Previous to assignment of this specific problem, all vinyl plastisol coating or mandrel forming work done locally had been based on use of mechanical convection ovens. Early phases of development work were also conducted with ovens as the source of heat.

3.1.2.1 Heaters

A crude trial of the efficiency of infra-red heat was made with open, ceramic base, resistance heating coils. As expected, efficiency was quite high. Surface temperatures of 200°C were attained in less than 2 minutes, compared to 15 to 30 minutes required by the ovens. In the tests, plates were passed into the heat zone from above and withdrawn by reversing the movement. The lower edge of the work piece was overheated, while the upper edge remained too cool. On the basis of this evaluation, the Chromolox heaters were ordered and the horizontal conveyor travel adopted. The decision to pass plates completely through the hot zone was made to assure equal exposure of leading and trailing edges. Use of doors, asbestos curtains, etc., around the heaters was considered and purposely abandoned in order to allow free access to the heaters and simplify construction.

3.1.2.2 Conveyor Speed

No study of the effect of conveyor speed was made. Space considerations favored use of a timed dwell in the heat zone rather than an attempt to control plate temperature by control of over-all plate transit time.

3.1.2.3 Heater Control

Most infra-red heating systems used commercially are controlled by proportional timers, which cost considerably less than variable output transformers where large current capacities are involved. Both types of controls were included in order to gain maximum precision of plate temperature control. Experimental work showed that the best reproducibility was attained by proper coordination of voltage input to the heaters and dwell timer setting. It is believed that the combination of proportional time controllers with long banks of heaters served by a variable speed conveyor would prove more economical in a unit where space is not a design limitation. There is no reason to believe that control precision would suffer.

3.1.2.4 Dipping Controls

Ability to produce uniformity in coating thickness and flaw-free coatings is dependent to a great extent on the speed control exerted on plate movement during the dipping operation. The cycle must be considered in three stages if efficiency of the process is a factor: (a) immersion, (b) withdrawal to expose upper surfaces, and (c) completion of withdrawal. The immersion rate is least important; any speed slow enough to avoid splashing is adequate. The third stage must be regulated with relation to plastisol viscosity. Observation of flow of plastisol on the face of a plate in this stage is the basis for adjustment. Speed of the plate's upward movement must be less than the downward plastisol flow rate. This relationship allows surplus coating material to drain freely from the plate surface (with its adherent layer of gelled plastisol) and prevents "runs" from starting on the faces of the plate.

The critical step in the dipping process is the initial rate of upward movement. Until all surfaces which form contact angles of more than 90° with the plastisol surface are free, the probability of pulling surplus material from the tank is high. The diagonal mounting of plates to preclude 180° contact angles is a necessity, but angles at edges of plates are in the range 120 to 160°. To allow drainage from the large contact angle edges, plate movement must again be slower than the drainage rate. This rate is dependent on plastisol viscosity and also on contact angle. Observation of results obtained at different speeds leads to correct adjustment of the machine speed. The timed, rheostat-controlled slow-down feature was a late modification of the dip cycle control system made after investigation of the effect of cycle stage speeds on coating quality.

3.2 Vinyl Compound Development

Because the original hope with which the development program was started was to avoid a preheat stage, all early work was done with high viscosity plastisol systems. The theory was to dip unheated plates, allow drainage time adequate for a minimum gradient in film thickness to be attained from top to bottom of the plate, and then fuse the coating. Obvious advantages are elimination of both the preheat stage and the necessity for precision dipping cycle controls. A less obvious advantage is the lengthening of plastisol life by virtue of not having thermally abused the tank contents. Extremely unreliable duplication of coating thickness values was the result of all tests made on high viscosity plastisols, including those which contained gelling agents such as metallic stearates.

Introduction of a preheat operation made necessary a search for a low viscosity system which possessed good aging characteristics when subjected to repeated thermal abuse. Meanwhile, a series of experiments related to use of low preheat and fusion temperatures was conducted without any degree of success. At this point, a high temperature preheat was agreed to be essential to rapid process development, and the need for a stable low viscosity system became even greater. Accordingly, a basic set of compound ingredients was established as standard and the effect of additions evaluated. The basic compound is shown in Table II.

TABLE II

EXPERIMENTAL COMPOUND

Ingredient	Parts by Weight
Geon 121	100
Paraplex G-60	40
Test Plasticizer	70
Test Stabilizer	4

Thermal abuse was provided by repeated dipping of dural bars heated to 180°C into small volumes of the test compounds. Comparative performance data are recorded in Table III. Actual viscosities were measured with a Brookfield rotational viscometer. An additional measurement of viscosity change with storage after thermal exposure is also reported. Data represents duplicate and triplicate tests.

TABLE III

TEST COMPOUND VISCOSITY CHANGES

Test Plasticizer	Test Stabilizer	Initial Viscosity (Centipoises)	Percent Change after Thermal Abuse	Percent Change on 6-Day Storage
DIDA	Dyphos(1)	810	+226	+1
DIDP(2)	Dyphos	1750	+317	Decrease
DOP	Dyphos	1560	+305	+29
DIDA	Barinac	710	+192	+98

(1) Dyphos is dibasic lead phosphite manufactured by the National Lead Co.

(2) DIDP is Monsanto Chemical Co. diisodecyl phthalate.

NOTE: All other compounding ingredients have previously been identified.

An intensive investigation of systems in which DIDA was thoroughly tested as a primary plasticizer showed uniformly poor results. Barinac was used as the companion stabilizer. Major problems were film thickness control and poor fusion characteristics at reasonable working temperatures.

Reference to other time tested, locally prepared plastisol formulations led to gradual changes in composition until a satisfactory compound was developed. The coating machine was completed, and tests were made under simulated production conditions as the composition was modified. This permitted coordination of machine adjustments with compound formulation.

In response to requests for improved ease of stripping coatings from plates, an addition of 0.5 part by weight of Dow-Corning Silicone Mold Release Fluid (DCMRF) was made to the basic vinyl compound listed in Table I. A slight decrease in adhesion was exhibited by coatings made from the modified formulation; however, the failure of adhesion of some of the revised coatings in routine handling also resulted. Shear failure occurred at the coating-plate interface when the exterior of a coated plate was rubbed against another. A few failures occurred during removal of the plates from the coating fixtures. Areas where no bond existed were not capable of being inspected ultrasonically. Failures of this type can be easily detected by their cloudy appearance. Good bonding results in a high degree of transparency and plate surface details are subject to visual inspection through the coating. Part numbers inscribed on the corners of the plates are visible and legible through the coating. The use of the DCMRF addition was discontinued after one set of 20 plates had been coated.

3.3 Effects of Processing Variables

The previous discussion has included information relative to the effects of some process variables on coating quality. A more important factor is the relationship of preheating time and temperature with bubbles, blisters, and coating thickness. Preheating of work pieces causes plastisol adjacent to the plate to fuse or gel to an extent dependent on the temperature and heat capacity of the part. The mechanism is by acceleration of the solvation process in the regions of plastisol near the heated part. The process of thickness build-up can be allowed to go to completion by use of a prolonged time dwell in the dip tank, or it can be stopped by withdrawal of the plate. Both approaches were evaluated.

Although unfused coatings have an unflawed appearance if applied with a low degree of preheating of work plates, fused coatings blistered with nearly 100 percent frequency. A theory which seems to explain this action adequately is that the poor thermal conductivity of the graphite plates and their surface porosity act jointly: Preheating affects only the surface area. The dipping operation then cools the plate. Complete fusion of the coating requires re-heating of the coating and the plate surface to 150 to 180°C. The heat dissipation into the plate expands the air in the pores, creates pressure, and the soft, hot coating is expanded locally to form blisters. A related phenomenon is the overheating of "green" or impregnated plates to cause decomposition of furane resin to gaseous products, which also create blisters.

The result of examination of the factors just detailed was adoption of a preheat stage more drastic than the subsequent fusion stage. The basis for the apparent reversal of the original plan was the premise that blistering and bubbling, if due to the causes theorized, would be eliminated: High temperature preheating followed immediately by a fast dip cycle would deposit a thin coating. The plate surface would be at its highest temperature, and decomposition products or air in pores would both be in their most expanded state. As heat dissipated into the plastisol, the gases would cool and contract, and no pressure would be available to form blisters. If conditions were judged correctly, the coating layer adjacent to the plate would fuse immediately, and the outer layers could be fused on a short time cycle without overheating of the plate proper.

Practice confirmed the theory, and practical operating conditions were established. Actually, plates coming from the dip cycle appear to be equipped with fused coatings. The plastisol layer is 80 to 90 percent transparent, and the fusion stage, as now used in processing, is for the most part a safety factor. It assures completion of fusion, development of high tear strength, and maximum resistance to chemicals and abrasion.

Chapter 4

CONCLUSIONS

A process for application of vinyl plastic coatings to porous graphite plates has been developed. The process permits rapid and convenient ultrasonic inspection of the plates. Although the development program was not accompanied by a thorough investigation of possible variations in material compounding or equipment design, a large number of important factors were discovered and their general action and interaction observed and controlled.

The use of the coating material and equipment to allow extension of the ultrasonic inspection process to other plate-shaped, porous articles is self-evident. The only prerequisite is that the items be sufficiently heat-resistant to withstand coating conditions. The potential field for use of the technique described includes graphite and ceramic rocket components and unsintered powdered metal parts. Minor modifications of the base vinyl formulation and the processing equipment would further expand the usefulness of the process to include articles having more complex geometry than that of the plates involved in the present program.