THE PREVENTION OF STICKING IN BRIGHT-ANNEALING SHEET STEEL

A THESIS

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by

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Approved:

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Two major steel companies furnished information about the annealing practice and the methods used in preventing sticking in their plants, but it was confidential and could not be quoted in this work. The author wishes to express his thanks for this information even though he was unable to use it.

CONTENTS

INTRODUCTION

During the manufacture of bright-annealed sheet steel, the cold-rolled steel may either be cut into sheets of convenient size or produced as a continuous strip and wound into rolls. In either case rolling strains are set up in the metal, which must be partially or completely removed by annealing.

The bright-annealing of the sheets requires the utmost care to prevent discoloration of the metal by the formation of a light coat of oxide on the surface (1). A special technique has been developed and special equipment designed for carrying out this operation.

The essential equipment required consists of an annealing-box, and annealing furnace, and suitable devices for charging and withdrawing the box from the furnace.

The furnace, which may be fired by either coal or gas, is built in two parts, a combustion compartment and a heating chamber. A reducing atmosphere in the heating chamber is desired in order to prevent rapid oxidation which causes destruction of the annealing box. The heating chamber has an arched roof and is about 16 ft. long, 8 ft. wide, and 8 ft. high (2). The gas is admitted into flue-like passages at the closed end of the furnace and distributed along each side of the heating chamber.

(1) Shannon, Sheet Steel and Tin Plate, The Chemical Catalog Company, Inc., pp. 135, 139. (1930)
(2) J. M. Camp and C. B. Francis, The Making, Shaping, and Treating of Steel, Carnegie Steel Co., p. 960, (1925)

The annealing-box is made in two parts, a top and a bottom, from cast steel. The top is made slightly longer and wider than the largest sheet to be annealed, and it is in the form of a rectangular hood. The bottom is flat on its upper side and is several inches longer and wider than the top. Its outer edge is enclosed by a rim.

The annealing-box is charged as follows: The sheets are stacked on the bottom to a height of about five feet and then the top lowered over them; the crevice that is formed between the top and the rim of the bottom is filled with sand. This sand serves as a seal and prevents the sheets from coming into direct contact with air other than that already in the remaining space in the box. Only enough air is left in the box to oxidize the edges of the sheets and the surface of the top sheet of the stack.

The box, as charged, is pushed into the furnace, the doors closed, and the gas lighted. The temperature is allowed to rise until the outside of the box has reached the desired annealing temperature. It is necessary to maintain this temperature for six to eight hours until it is uniform at all points in the stack. The box is then withdrawn from the furnace and allowed to cool to about 200°F. before the top is removed. In cooling the gas inside the box contracts, thereby pulling fresh air into it through the sand seal causing further oxidation of the sheets. If this oxidation is found to be too great, the process may be changed slightly by keeping the box filled with a reducing gas during both heating and cooling cycles (3).

The process for bright-annealing rolls of sheet metal differs from the above only to the extent that, instead of stacking, the individual rolls are charged to the annealing box.

The temperatures used in bright annealing range from 1240°F. to 1600°F. The temperature most commonly used, 1400°F., is sufficient to eliminate, to a great extent, the stress and strain produced by rolling operations, and to develop the ductility almost to the maximum obtainable in the metal(4). Higher temperatures are found to promote excessive grain growth, which tends to weaken the sheet considerably as well as to roughen the surface in deep drawing. Lower temperatures do not sufficiently remove the rolling strains, nor do they impart sufficient softness and ductility to the metal(5).

One of the principal limitations of box-annealing is the tendency of the metal sheets to stick together at the annealing temperature. The pressure on the bottom sheets of a stack or near the center of the coil is great enough to cause the sheets to stick so tightly together that a sword is frequently necessary to loosen them(6). The pressure on the bottom sheets in a five foot stack is about 15 lb./sq.in. At times separation is impossible, making it necessary to discard the sheets as waste. Those that are separated may have their surface damaged so badly that they must either be discarded or sold as a lower grade product. The losses due to sticking may range

(3) Shannon: loc. cit., p. 141
(4) J. M. Camp and C. B. Francis: loc. cit., p. 963
(5) Ibid, p. 963
(6) Ibid, p. 963

up to 3%, or even more at higher temperatures(7). These losses increase the cost of producing bright-annealed sheets, and therefore open up a field for the development of suitable methods for preventing sticking.

One method reported for overcoming sticking calls for the coating of the sheets with a one percent solution of alkaline metal soaps(8) before they are placed in the annealing-box. At the annealing temperature, this separating medium is broken down, leaving an inert inorganic residue which is not harmful to the bright surface of the metal.

The purpose of this work has been to investigate the factors causing sticking and to find, if possible, a method for preventing it. The economic as well as the practical side has been considered in the selection of suitable materials. It is obvious that, to have any commercial value, the cost of treatment must be less than the value of the sheets lost due to sticking.

Because of the nature of this problem, the experimental work and the discussion is divided into two parts. Part I will embrace the studies of the factors effecting sticking and Part II will cover the prevention of sticking.

(7) Confidential communication
(8) S. H. Bobrov, U. S. Patent, No. 2,132,557 (Oct., 1938)

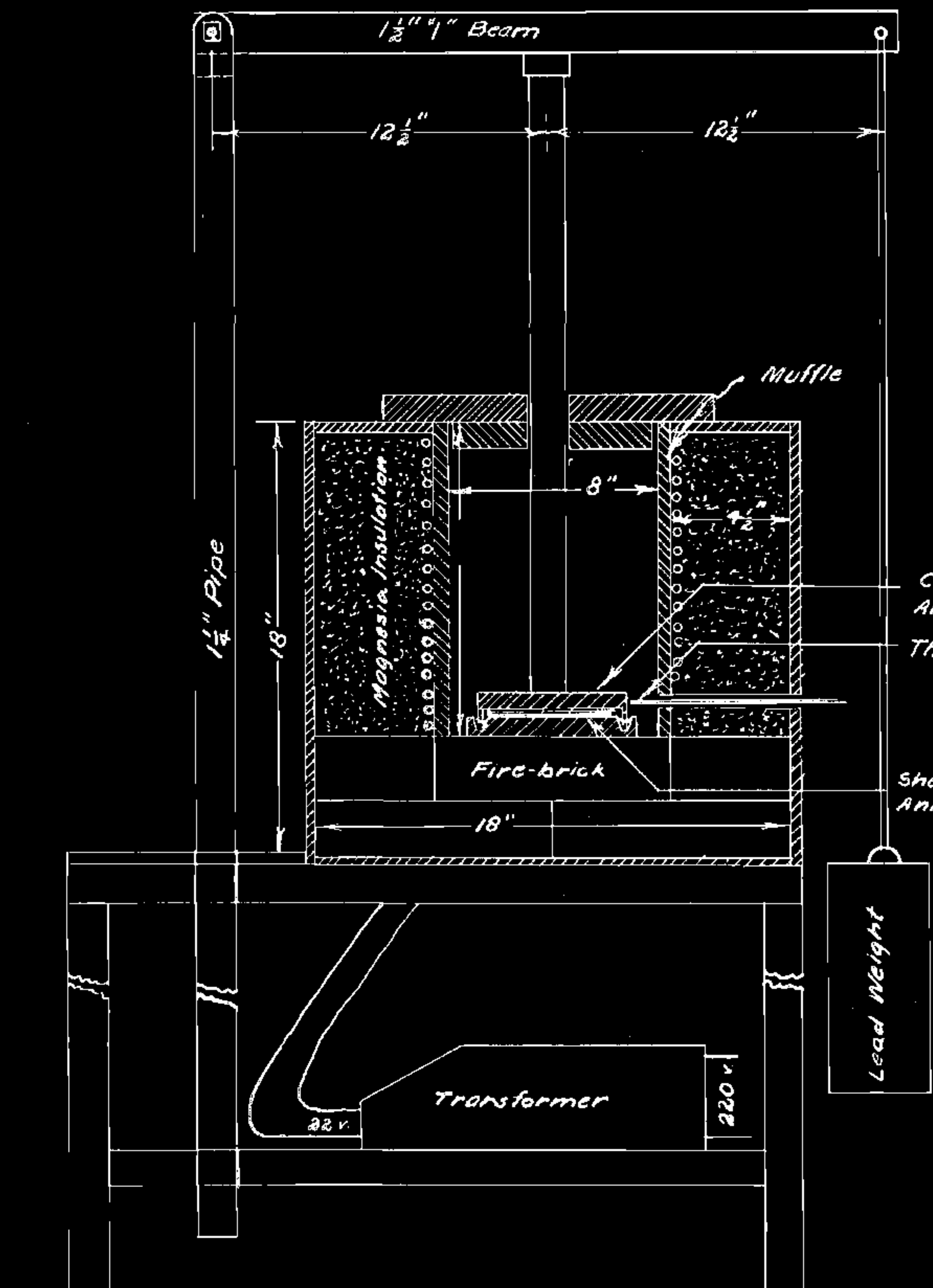
EQUIPMENT

The furnace used for the annealing was of the resistance type, rated at 3500 watts at 22 volts. The maximum temperature obtainable was about 1800°F. The muffle was a vertical cylinder with an opening at the top through which the annealing-box could be introduced. The furnace was insulated with 4½ inches of magnesia and its bottom made of two layers of fire-brick. The outside walls and top were made of "Transite" board.

The loading arrangement for applying pressure to the samples being processed was made up as follows: A piece of 1¼ inch pipe was set up in a vertical position and bolted to the table which supported the furnace. This pipe extended about 18 inches above the top of the furnace and was slotted at the top end. A 1½ inch "I" beam was pivoted in the slot and extended across the center of the muffle and several inches past the edge of the furnace. The shaft that lead down to the annealing-box contacted the beam at mid-point between the overhanging and pivoted ends. This arrangement gave a mechanical advantage of two, thus making the load on the shaft equal to twice the load applied to the end of the beam. Figures 1 and 2 show the furnace and loading arrangement as it appears during actual operation.

The annealing-box in which the sheets were processed was made of two circular pieces of cast iron. The cover of the box was 5½ inches in diameter and one inch thick with a rim 3/8 of an inch wide and 1/8 of an inch thick about the bottom edge. A shaft of one inch pipe was attached to the center of the

Fig. 1



1½" "I" Beam
12½"
12½"
Muffle
8"
4½"
Magnesia Insulation
1¼" Pipe
18"
Cast Iron
Annealing Box
Thermocouple
Fire-brick
Sheets being
Annealed
18"
Lead Weight
Transformer
22 v.
220 v.

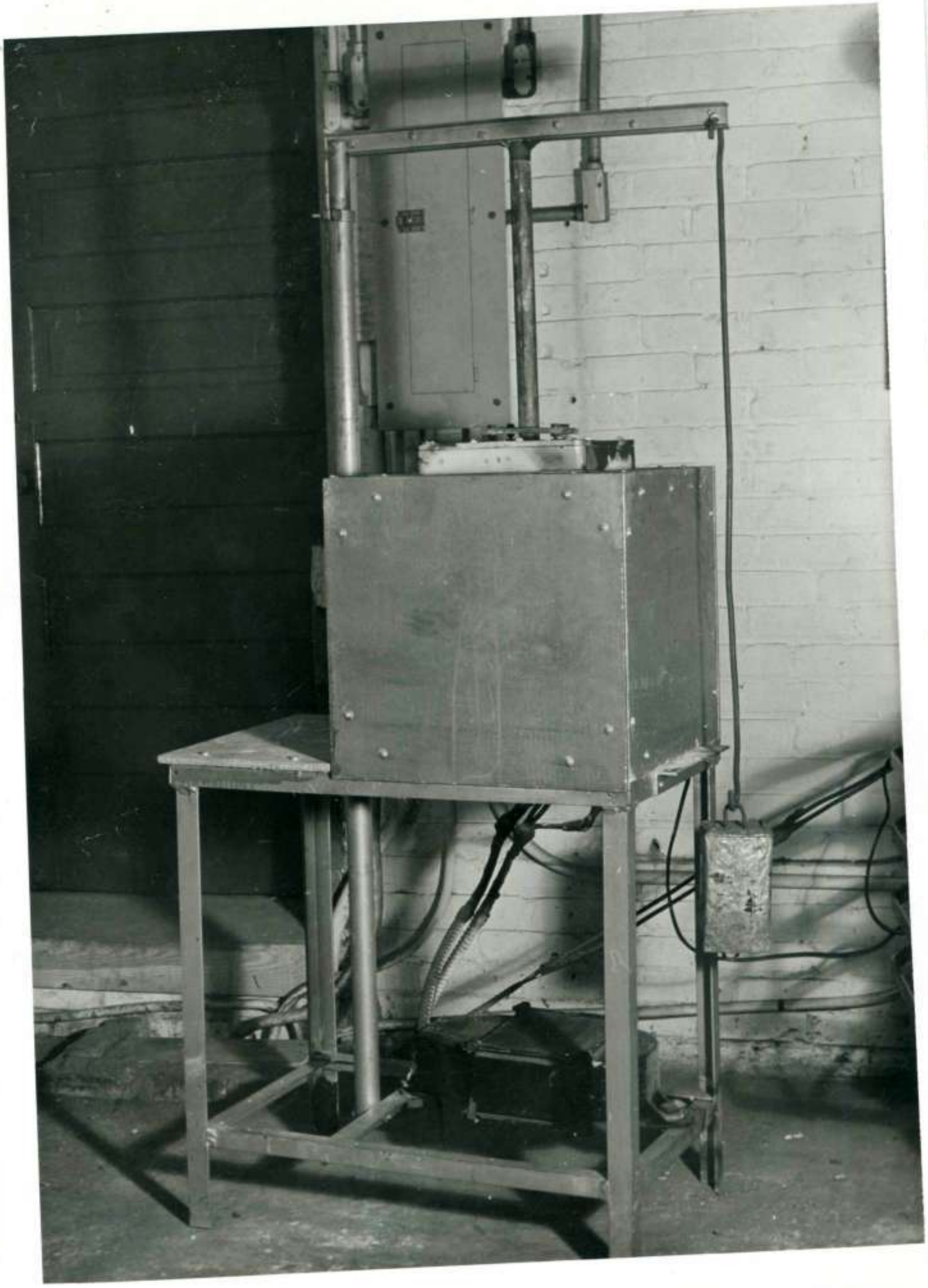


Figure 2.

Annealing-furnace in operation.

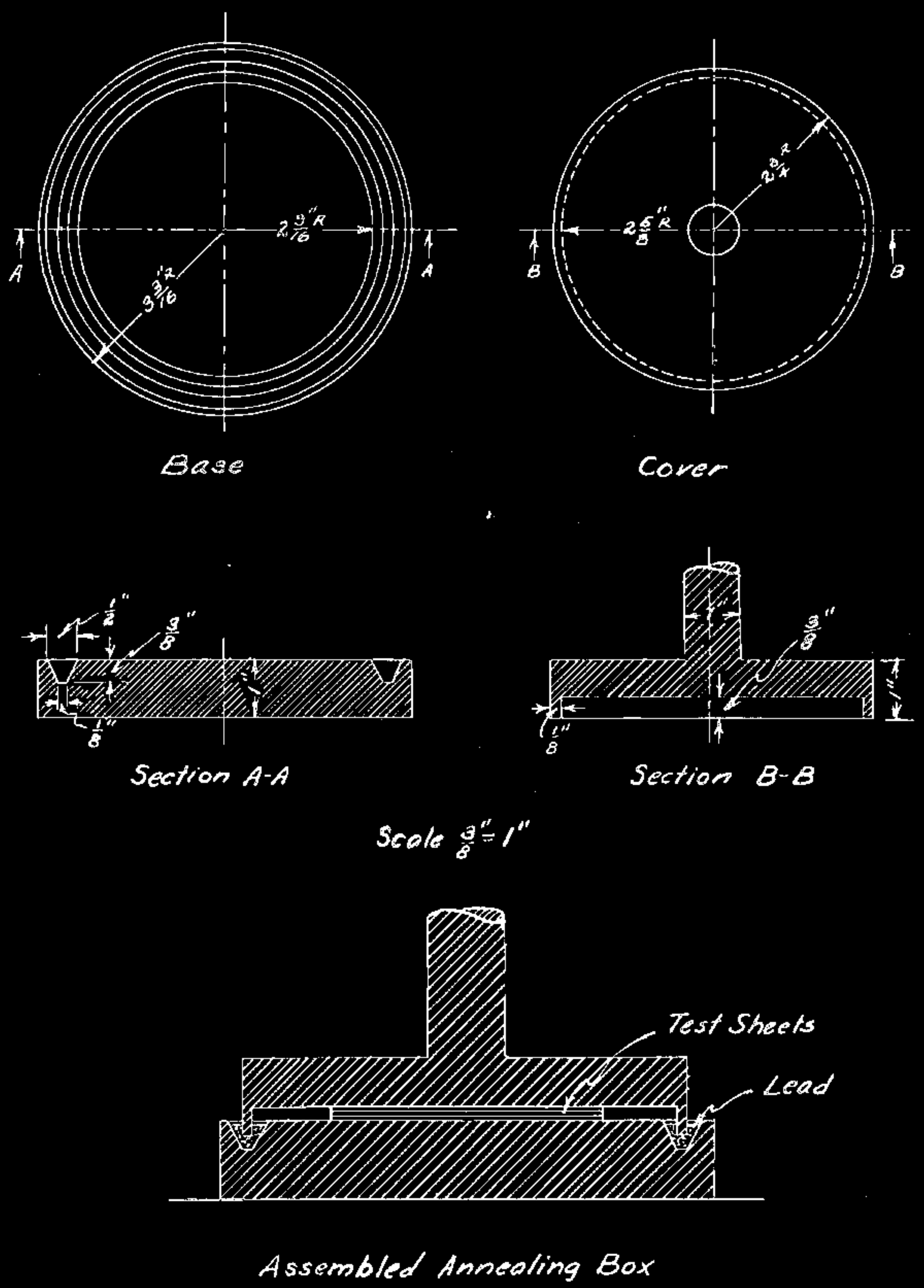

A
A
B
B
Base
Cover
Section A-A
Section B-B
Scale 3/8" = 1"
Test Sheets
Lead
Assembled Annealing Box

Fig. 3

cover for transmitting the lead from the beam to the samples. The bottom of the box was made of a $6\frac{1}{2}$ inch circular piece of cast iron with a "V" groove cut about the outer edge. When the box was assembled, the rim of the cover extended down into the groove of the bottom. When the two were fitted together, the box was made air-tight by filling the groove with lead. At the temperatures encountered in the furnace the lead became molten, thereby allowing the pressure to be applied directly to the sheets in the box. Details of the box are shown in Figure 3.

The temperature of the furnace was measured and controlled to within $\pm$ 5°F. by a Model 601, Wheelco "Capacitrol" controller, using a chromel-alumel thermocouple with compensating leads.

EXPERIMENTAL

PROCEDURE FOR A TYPICAL RUN

The sheet steel used in this work was cold-rolled No. 18 American wire gage sheet of the following analysis:

Open hearth, mechanical capped, deep drawing quality, 50% cold reduced.

Ladle Analysis

C	Mn	P	S	Si	Cu	
.06	.30	.019	.035			%

Check Analysis

C	Mn	P	S	Si	Cu	
.055	.25	.017	.032	.003	.050	%

The individual samples of sheet metal were 3 x 3½ inches, this being the largest size sample that could be used in the furnace and annealing-box.

In all runs three sheets of steel were used for a charge. The sheets were cleaned by washing with alcohol and then thoroughly dried.

The annealing-box was prepared for a run as follows: The bottom of the box was heated over a gas burner until it reached a temperature of about 500°F., and then set on a level surface in readiness for the lead to be poured into the "V" groove on its outer edge. Three used samples of sheet were placed in the bottom of the box and the "V" groove filled with molten lead. Before the metal solidified, the top was placed on the bottom with the rim extending down into the lead-filled groove. The rim displaced some of the lead and left the groove just full with the cover in place. The whole box was cooled to room temperature. The box was easily opened by lightly tapping on the bottom, leaving the lead from the groove adhering to the rim of the cover. The clean samples to be studied were then stacked on the bottom of the box and the cover replaced. The annealing-box with the samples was then ready to be charged to the furnace.

The furnace was heated to about 1800°F., and the annealing-box then carefully lowered into it. The cover was then put on the furnace, and the "I" beam lowered across the top of the shaft of the annealing-box. The desired weight was hung on the end of the beam, and the thermocouple inserted in the furnace. The controller was then set at the desired temperature for the run.

The temperature of the furnace usually dropped to about 1200°F. during loading and a time of about 1½ hours was required to reach the annealing temperature. When the temperature reached the required value the time was noted and the samples held at that temperature for a predetermined period. After the specified time had passed, the current was cut off, and the samples allowed to cool to below the melting point of the lead seal. This was necessary in order to make the top and bottom adhere to each other so that the box could be removed from the furnace intact.

The box was allowed to cool to room temperature before opening. This was necessary because there was danger of oxidation of the sheets if opened at too high a temperature. Opening the box involved tapping the bottom and breaking the lead seal.

This is the procedure for a typical run and it should be understood that the time, temperature, and pressure can be varied as desired. The method used for coating the sheets in preventing sticking will be given in the section devoted to discussion of the runs using the various suspensions.

PART I

INVESTIGATION OF FACTORS INFLUENCING STICKING

The three factors investigated were the pressure, the annealing temperature, and the length of time that the sheets were held at the annealing temperature.

Each of these three factors was varied and observations were made of the condition of the sheets after treatment.

A search of the literature disclosed the fact that 1400°F. is the temperature most commonly used(9), and for this reason it was chosen as the temperature at which to work.

First, the temperature and time were held constant at 1400°F. and one hour respectively and pressure varied from 10 lb./sq. in. to 25 lb./sq. in. At 25 lb./sq. in. a satisfactory degree of sticking was obtained. The pressure was then held constant at 25 lb./sq. in. for one hour, and the temperature varied. The temperature was lowered by 100 degree intervals until the sheets no longer stuck together. In this manner, the lower temperature limit of sticking was found to be 1100°F. at 25 lb./sq. in. for one hour. The temperature and pressure were next held constant at 1100°F. and 25 lb./sq. in. and runs were made for five and twenty hours. A run was also made at 25 lb./sq. in. and 1400°F. for three hours.

(9) J. M. Camp and C. B. Francis: loc. cit., p. 963

DATA AND RESULTS

Run No.	Temperature °F.	Time at Temperature	Pressure, lb./sq. in.	Results	Remarks
1	1400	1 hour	10	Sticking	Sheets oxidized near edges for about 3/16" and sticking occurred in these parts to a slight degree.
2	1400	1 hour	11	Sticking	Same as Run No. 1
3	1400	1 hour	12	Sticking	Same as Run No. 1
4	1400	1 hour	15	Sticking	Same as Run No. 1
5	1400	1 hour	16	Sticking	No oxidation on edges. Sticking was localized over an area of approximately .75 sq. in., and surface damaged in this area.
6	1400	1 hour	17	Sticking	Same as Run No. 5
7	1400	1 hour	18	Sticking	Same as Run No. 5
8	1400	1 hour	19	No Sticking	Localized pressure eliminated. No oxidation occurred on the edges of the sheets.
9	1400	1 hour	20	Sticking	Slight sticking in oxidized edges and at points near the center portions of sheet. Sheets easily separated.
10	1400	1 hour	21	Sticking	Pressure evenly distributed over surface. Slight sticking occurred but sheets easily separated.
11	1400	1 hour	22	Sticking	Sheets stuck tighter than in Run No. 10
12	1400	1 hour	23	Sticking	Sheets oxidized on edges but sticking occurred in center areas. More difficult to separate than in Run No. 11.
13	1400	1 hour	24	Sticking	Sheets bright and bending necessary to separate.
14	1400	1 hour	25	Sticking	Sheets bright and bending necessary to separate. The surface was badly damaged in separating.
15	1400	3 hours	25	Sticking	No apparent difference in the degree of sticking noted in this and a 1 hour run.
16	1300	1 hour	25	Sticking	Sticking uniform over sheet but sheets not stuck as tightly as at 1400°F.
17	1200	1 hour	25	Sticking	Sheets stuck together but easily separated.
18	1100	1 hour	25	No Sticking	Sheets in execellent condition.
19	1100	5 hours	25	No Sticking	Sheets in execellent condition with no sticking.
20	1100	20 hours	25	No Sticking	Sheets in execellent condition.

DISCUSSION OF RESULTS:

The pressure required to produce positive sticking on every run was 25 lb./sq. in.; sticking not being appreciable until a pressure of 20 lb./sq. in. was applied, and even at this pressure the sheets could easily be separated. They became more difficult to separate as the pressure was increased to 25 lb./sq. in.; at which point they were stuck together so tightly that destructive bending was necessary to separate them. These sheets were so badly damaged in separation that they would have no commercial value. It was for these reasons that 25 lb./sq. in. was chosen for testing the effectiveness of materials in preventing sticking. Even though this pressure is nearly twice the average pressure encountered in actual practice, localized pressure might easily reach this value in a stack of large sheets.

As the temperature was lowered from 1400°F. and the pressure held constant at 25 lb./sq. in., the degree of sticking became progressively less until at 1100°F. sticking no longer occurred. This was the lower temperature limit of sticking for 25 lb./sq. in., and it closely coincides with the lower temperature limit for box-annealing.

Runs made at 1100°F. in which the temperature was maintained first for five and then twenty hours, caused no sticking. A run held for one hour at a temperature which produced sticking exhibited sticking as severe as one held for three hours under the same conditions.

Since some of these runs were held for times far in excess of that used in practice and since extending the time produced no change, it is apparent that the time factor is a relatively unimportant one.

Figure 5 shows the polished edges of sheets that stuck together at 1400°F. and 25 lb./sq. in.

PART II

PREVENTION OF STICKING

SUSPENSIONS

The oil used for preparing the suspensions was a light colored mineral oil sold by The Texas Company under the name "Spindle Oil B". Its viscosity is 96-100 Saybolt seconds at 100°F. The solids used in the suspensions were as follows:

"BC Volclay", a grit-free colloidal bentonite, all finer than 5 microns with 90% 1 micron or finer.

"Argosite Clay", powdered bentonite, all particles 300 mesh and finer.

"Alberoyd Clay", airfloat powdered soapstone, 97% of which will pass through 300 mesh screen.

"Micatone B-1000", very finely divided white mica.

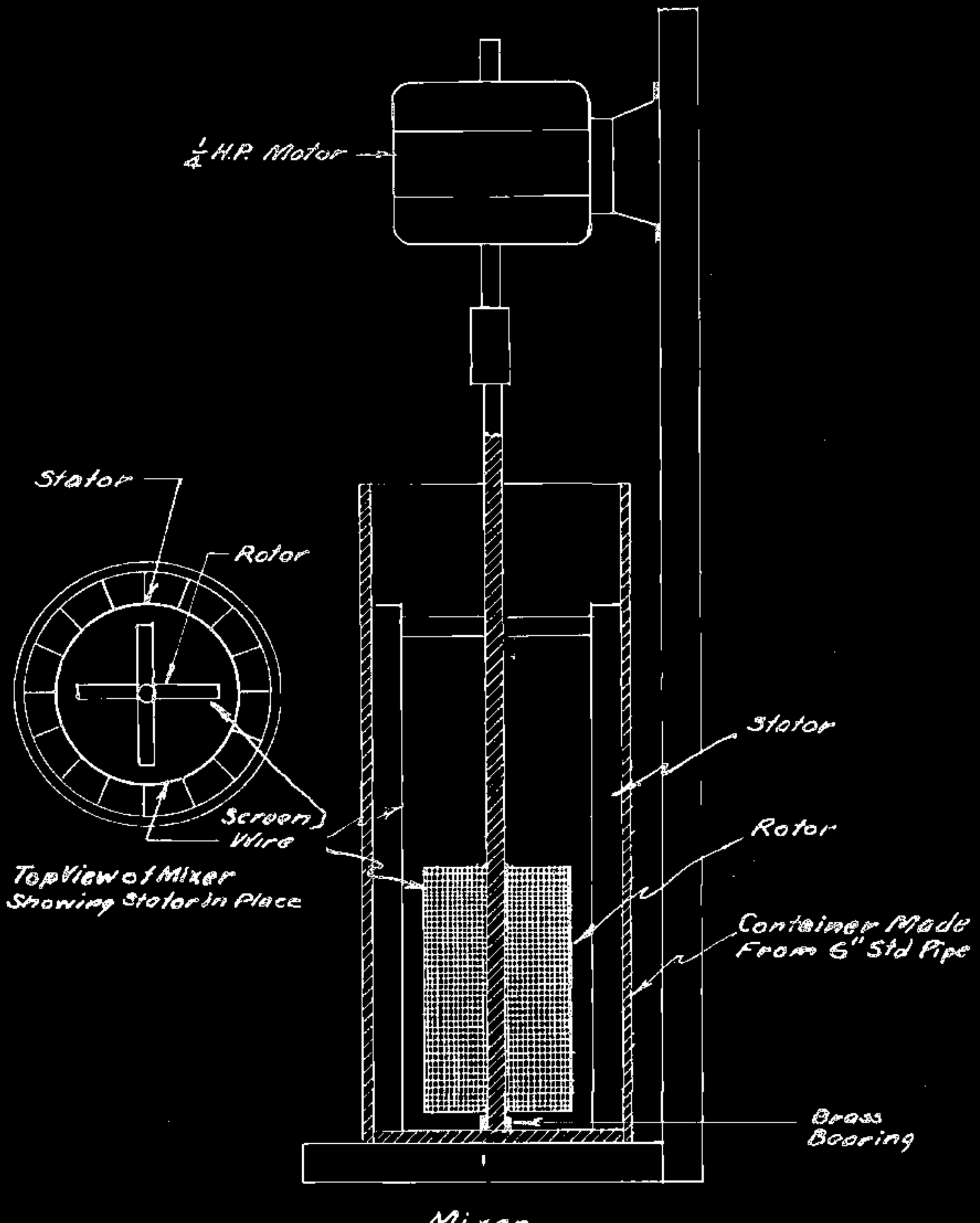
"Water-ground White Mica"

"Dry-ground Mica", No. 250 Grade.

"Oildag", a 10% suspension of colloidal graphite.

Magnesium oxide, 250 mesh and finer.

The suspensions were made by mixing, for 30 minutes, the various powders with oil by means of a special type mixer. The mixer consisted of a container made of a piece of six inch pipe, in which was placed a stator of the squirrel-cage type. The inside of the stator was covered with a layer of 16 mesh screen wire. The rotor was made by welding four pieces of 1/2 inch strap iron on a cold-rolled shaft in such a way that four rectangular openings 1-1/2 inches by 6 inches were formed at


1/4 H.P. Motor
Stator
Rotor
Screen Wire
Top View of Mixer Showing Stator In Place
Stator
Rotor
Container Made From 6" Std Pipe
Brass Bearing

Mixer

Fig. 4

right angles to each other. The openings were covered with screen wire, thus forming four blades on the end of the shaft. One end of the rotor was connected directly to a quarter horsepower motor and the other supported by a brass bearing in the bottom of the container. Figure 4 shows the mixer.

The first suspension prepared was from "BC Voloaly", which had the smallest particle size of all the powders used. In making the first suspension an effort was made to adjust the concentration so that there would be enough particles present to give a layer over the sheet one particle thick. Other suspensions of this powder were made by adding oil until one of approximately one-half the previous concentration resulted. Each of these suspensions was tried on the sheet metal in the furnace and the behavior of the various concentrations observed.

After a number of runs in the furnace, it was found that fairly low concentrations were successful in preventing sticking; hence it was obvious that high concentrations of the suspensoids need not be used.

The suspensoids were found to settle in a relatively short time, indicating that it might be advantageous to attempt to stabilize the suspensions. When these suspensions broke down, a layer of clear oil was formed on the surface. The mono-glyceride of coconut oil, a naptha soluble material, was tried as a suspending agent for those solids which showed promise in preventing sticking. It was found that, in order for the stabilizing effect to be noticeable, 6% mono-glyceride had to be present, and under these conditions the suspensoids settled in two days. This was an improvement as far as the permanence of the suspension was concerned, but it was found that the presence of the mono-glyceride caused the sheets to be very badly stained when coated with this suspension and annealed.

The sheets were coated by dipping. Since the suspensions were not stable, and since a clear layer of oil formed on the surface in settling, it is obvious that, in the industrial application of this method of coating sheets, continuous agitation of the suspension would be necessary; otherwise the clear layer would have a tendency to wash the suspended particles off of the sheet when it was withdrawn from the bath. The suspensions were thoroughly mixed and the time for a layer of clear oil about 1/16 of an inch thick to form on the surface was determined.

It was found in every case that the longest time was required for the clear layer to form on the lowest concentration of suspension. "Water-ground mica", "Micatone B-1000", "Dry-ground Mica", and magnesium oxide required longer settling times than the soapstone and the two grades of bentonite.

It was also noted that, when the suspensoids had settled out completely, the ease of resuspending was in the following order: All grades of mica, magnesium oxide, "BC Volclay", "Argosite Clay", and "Alberoyd Clay".

METHOD OF COATING SHEETS:

Three sheets were cleaned thoroughly by washing with alcohol and then dried. The sheets were dipped into the uniform suspension one at a time and then stacked together. Pressure was applied to the stack of sheets in order to force out the excess material between them. The top and bottom surfaces of the stack were wiped clean of any suspension that might be left on these surfaces. The samples were then placed in the annealing box and charged to the furnace as described in the section entitled "Procedure for a Typical Run."

In all runs the pressure was 25 lb./sq. in., and the time one hour. The temperature used was 1400°F. unless otherwise indicated. Duplicate runs were made with each suspension and triplicate runs with those suspensions that showed promise of being useful.

DATA AND RESULTS

Run No.	Material	Concentration of Suspension	Settling Time*	Temperature °F.	Time at the Temperature	Pressure, lb./sq. in.	Results	Remarks
21	Oil only	-----	-----	1400	1 hour	25	Sticking	Sheets bright and unstained but stuck so tightly together that bending necessary to separate. The surface was badly damaged in separating.
22 23	"BC Volclay" (Bentonite)	10.80%	94 sec.	1400	1 hour	25	Sticking	Slight sticking occurred at various points over the surface. The bentonite migrated to points of high concentration over sheet. The sticking was in the vicinity of these areas of concentration. Excess powder difficult to clean from the surface.
24 25	"	5.60%	120 sec.	1400	1 hour	25	Sticking	The surface was slightly roughened and easy to clean of the excess powder. The sheets easily separated.
26 27	"	2.72%	190 sec.	1400	1 hour	25	Sticking	Surface undamaged and sheets easily separated. The particles of Bentonite migrated to areas of concentration.
28 29	"Argosite Clay" (Bentonite) 300-mesh	9.76%	84 sec.	1400	1 hour	25	No Sticking	The surface was roughened and the clay was concentrated in small areas. The sheets very difficult to clean.
30 31	"	5.12%	95 sec.	1400	1 hour	25	Sticking	Slight sticking on opposite side of sheet from areas of high concentration of clay. The surface was slightly roughened and very difficult to clean
32 33	"	3.26%	160 sec.	1400	1 hour	25	No Sticking	The surface was undamaged and easily cleaned of the excess powder by brushing.
34 35	"	1.58%	570 sec.	1400	1 hour	25	Sticking	The surface was undamaged but the sheets were very difficult to separate.
36 37	"	3.26%	160 sec.	1600	1 hour	25	No Sticking	The sheets did not stick but were badly discolored.
38 39	"Argosite Clay" (Soapstone) 300-mesh	10.96%	52 sec.	1400	1 hour	25	No Sticking	The surface was undamaged but due to the clay being concentrated in small areas over the sheet they were difficult to clean.
40 41	"	6.16%	65 sec.	1400	1 hour	25	No Sticking	Again the clay concentrated in small areas making the sheets difficult to clean.
42 43	"	3.69%	90 sec.	1400	1 hour	25	No Sticking	The surface of the sheets was in excellent condition and was easily cleaned by light brushing.
44 45	"	1.85%	480 sec.	1400	1 hour	25	Sticking	The sheets were very difficult to separate and the surface damaged in separating.
46 47	"	3.69%	90 sec.	1600	1 hour	25	No Sticking	The surface was a dull brown color and in good condition except for this.

*The time for a layer of clear oil 1/16" thick to form on the top surface of the suspension.

DATA AND RESULTS (Cont'd.)

Run No.	Material	Concentration of Suspension	Settling Time*	Temperature °F.	Time at the Temperature	Pressure, lb./sq. in.	Results	Remarks
48	"Argosite Clay" plus 6% Mono-glyceride of Coconut Oil	3.26%	2 days	1400	1 hour	25	No Sticking	The surface of the sheets was stained a dull-brown.
49	"Alberoyd Clay" plus 6% Mono-glyceride of Coconut Oil	3.69%	2 days	1400	1 hour	25	No Sticking	The surface of the sheets was stained a dull-brown.
50 51	"Water-ground Mica"	7.00%	90 secs	1400	1 hour	25	No Sticking	The surface was in excellent condition and the residual mica powder could be removed easily.
52	"	3.5%	360 sec.	1400	1 hour	25	No Sticking	The results of this run are same as preceding run.
53 54	"	3.5%	"	1600	1 hour	25	No Sticking	The sheets were in excellent condition and the residual powder could be removed easily.
55 56	"Micatone B-1000" (White Mica)	3.50%	390 sec.	1400	1 hour	25	No Sticking	The sheets were in excellent condition and the residual powder could be removed easily.
57 58	"	3.50%	"	1600	1 hour	25	No Sticking	The surface of the sheets was in exeellent condition and the residual powder could be easily removed.
59 60	"Dry-ground Mica", No. 250 Grade	3.50%	330 sec.	1400	1 hour	25	No Sticking	The surface of the sheets was slightly roughened. The particles of mica appeared to have been forced into the surface of the metal.
61 62	"	3.50%	330 sec.	1600	1 hour	25	No Sticking	The surface of the sheets was rougher than in the run at 1400°F.
63 64	Ethylphenyl Stearic Acid	-----	------	1400	1 hour	25	Sticking	The sheets were stained very badly and bending the sheets was necessary in order to separate.
65 66	Phenoxyphenyl Stearic Acid	-----	------	1400	1 hour	25	Sticking	The sheets were stained very badly and bending the sheets was necessary in order to separate.
67	"Oildag" (Colloidal Graphite)	.0352 gm. / 100 gm. oil	----	1400	1 hour	25	Sticking	The sheets stuck together very tightly and were damaged in separating.
68	"	.0704 gm. / 100 gm. oil	------	1400	1 hour	25	Sticking	The sheets stuck together very tightly and were damaged in separating.
69	Magnesium Stearate	1% Solution		1400	1 hour	25	Sticking	Sticking appeared to be as bad as when oil alone was used to coat the sheets.

*The time for a layer of clear oil 1/16" thick to form on the top surface of the suspension.

DATA AND RESULTS (Cont'd)

Run No.	Material	Concentration of Suspension	Settling Time*	Temperature °F.	Time at the Temperature	Pressure, lb./sq. in.	Results	Remarks
70	Magnesium Stearate	3% Solution	----	1400	1 hour	25	Sticking	Sticking appeared to be as bad as when oil alone was used to coat the sheets.
71	Zinc Stearate	1% Solution	-----	1400	1 hour	25	Sticking	The sheets stuck so tightly that bending was necessary to separate them.
72	"	3% Solution	-----	1400	1 hour	25	Sticking	The sheets stuck so tightly that bending was necessary to separate them.
73 74	Magnesium Oxide, 250-M.	3.50%	205 sec.	1400	1 hour	25	No Sticking	The surface of the sheets was stained a light brown color.
75	"	1.75%	395 sec.	1400	1 hour	25	No Sticking	The surface of the sheets was stained a light straw color.

*The time for a layer of clear oil 1/16" thick to form on the top surface of the suspension.

DISCUSSION OF RESULTS:

Sheets Coated with Oil Alone

The sheets came from the furnace bright and unstained but were stuck together so tightly that bending and badly damaging of the surface was necessary to separate them. This run was a control and showed that the oil alone would not discolor the surface of the metal or prevent sticking, and hence any run in which sticking was prevented or discoloration of the metal occurred was due to substances added to the oil and not to the oil alone.

"BC Volclay" (Bentonite)

10.80% Suspension

The surface of the metal was bright, but slight sticking occurred at various points over the sheets. The bentonite was found to have migrated to various points over the surface and to have formed high concentrations of powder at these points. It is reasonable to believe that sticking occurs at points on the opposite side of the sheet from these areas of concentration, because of the localized pressure produced there. The excess powder, at these points of concentration, was very difficult to remove from the surface. It may be concluded that this concentration of suspension is too great to be of any use.

5.60% Suspension

Although the sheets stuck together, they were very easily separated and cleaned of the excess powder. The surface was found to be roughened slightly. This roughening of the surface, as well as the slight sticking, rendered this suspension of no value.

2.72% Suspension

The sheets were stuck slightly but could be easily separated. The powder residue was again found to be concentrated in several small areas over the sheet, but the surface remained undamaged. The sticking was great enough to preclude use.

"Argosite Clay", (300-mesh Bentonite)

9.76% Suspension

Slight sticking occurred and the surface was slightly roughened. The bentonite was found to be concentrated in small areas scattered over the surface of the metal and sticking appeared at points on the opposite side of the sheet from these areas. The surface was damaged in cleaning off the residual powder, as it was necessary to scrape the powder off in some places with a knife. The suspension is of no value because of the difficulty encountered in cleaning and the slight sticking.

5.12% Suspension

Again the bentonite became concentrated in small areas over the sheet and slight sticking occurred at points on the opposite side of the sheet from these areas. The surface was slightly roughened in places and the sheets were very difficult to clean. The concentration of the suspension appears to be still too high, as in the case of the preceding runs.

3.26% Suspension

No sticking occurred when this concentration was used. The surface was in good condition and easily cleaned of any residual powder by light brushing. The excess powder could not be detected on the surface until it was rubbed with a white cloth. There were no points of high concentration of powder. This suspension shows promise and would be recommended had not a cheaper one been found. Figure 6 shows the polished edges of sheets which were prevented from sticking.

1.58% Suspension

The residual powder appeared to be evenly distributed, but the suspension was not concentrated enough to give complete protection to the surface, hence sticking occurred in the unprotected portions. This run gives the limit below which the suspension will not prevent sticking.

3.26% Suspension, 1600°F.

Even though the sheets did not stick together, they were badly discolored. This discoloration was probably due to the bentonite breaking down at the higher temperature and oxidizing the sheet metal. This run shows that bentonite is of no value in higher temperature work.

"Alberoyd Clay", (300-mesh soapstone)

10.96% Suspension

No sticking occurred but the sheets were very difficult to clean of the excess soapstone. It was concentrated in small

Figure 5.

Edges of sheets stuck together at a pressure of 25 lb./sq. in. and a temperature of 1400°F. Note: Sticking along line near top of picture. (Magnification x 40)

Figure 6.

Edges of sheets which were prevented from sticking by use of 3.26% suspension of "Argosite Clay". (Magnification x 40) areas over the sheet, just as in the case of the higher concentrations of bentonite. The difficulty in cleaning excess powder from the surface makes this concentration of no value.

6.16% Suspension

The powder was concentrated in small areas over the sheet as in the case of the 10.96% suspension, but the number of these areas was considerably less. Even though there was no sticking at this concentration, the cleaning of the surface was too difficult to warrant its use.

3.69% Suspension

The sheets did not stick at all, could be easily cleaned by light brushing, and the surface was in excellent condition. The excess powder was just barely visible and evenly distributed over the surface of the metal. This concentration of suspensoid shows promise and it is cheaper than the 3.26% "Argosite Clay" suspension.

1.85% Suspension

The sheets stuck tightly together and were very difficult to separate. Their surfaces were bright but they were damaged slightly in separating. The concentration of the suspension is apparently below the limit to completely protect the sheets.

3.69% Suspension, 1600°F.

At this temperature sticking did not occur but the surface was badly discolored; this was probably due to the soapstone breaking down and oxidizing the surface. The surface was also slightly roughened by the soapstone. It may be concluded that "Alberoyd Clay" is of no use in higher temperature work.

"Argosite Clay", 300-mesh, 3.26% Suspension, 6% Mono-glyceride of Coconut Oil

Although no sticking occurred the sheets were badly stained to a dull brown color. This discoloration was not noted when the mono-glyceride was left out of the suspension so it can be assumed that the discoloration was due to the presence of the mono-glyceride and not to the bentonite. The mono-glyceride was a very satisfactory suspending agent, but the fact that it discolors the sheets will prevent its use in practice.

"Alberoyd Clay", 3.69% Suspension, 6% Mono-glyceride of Coconut Oil

The results of this run check the preceding run with "Argosite Clay" and the same conclusions can be drawn about the mono-glyceride.

"Water-ground Mica"

7.00% Suspension

No sticking occurred with this concentration and the surface was bright, undamaged, and easily cleaned of the residual powder left there. This suspension shows great promise.

3.50% Suspension

The sheets came from the furnace in excellent condition and no sticking occurred. It is believed that this suspension would serve satisfactorily in practice and that the mica concentration is close to the optimum. Only a small amount of residual powder was left on the surface and it could easily be cleaned by light brushing, making it satisfactory in all respects.

3.50% Suspension, 1600°F.

The surface was in excellent condition and no sticking whatsoever occurred. The sheets were also easy to clean of the residual powder. This material is found to be useful for all temperatures up to 1600°F., which is the highest temperature ever used in box-annealing.

"<u>Micatone B-1000</u>"

3.50% Suspension

The sheets were in excellent condition and no sticking occurred. The concentration is found to be very satisfactory for preventing sticking.

3.50% Suspension, 1600°F.

The sheets did not stick at this temperature and they were found to be in as good condition as sheets heated to only 1400°F. This material is found to be excellent for all annealing temperatures.

"<u>Dry-ground Mica</u>", <u>No</u>. <u>250</u> <u>grade</u>

3.50% Suspension

No sticking occurred at this concentration but the surface of the sheets was roughened to some extent. This roughening was probably due to particles of mica being pressed into the surface of the metal. The material is of no value because of the roughening effect.

3.50% Suspension, 1600°F.

The runs at this temperature check those at 1400°F., except for the fact that the surface was slightly rougher.

Ethylphenyl Stearic Acid

This material was applied full strength. The sheets stuck together as tightly as if nothing had been used on them. The surface of the metal was stained a very dark brown. This substance is of no use.

Phenoxyphenyl Stearic Acid

The runs with this material check the results obtained with Ethylphenyl Stearic Acid. These two compounds have recently been synthesized and have exhibited good temperature resistance, but 1400°F. is apparently too high for them.

"Oildag"

.0352 gm./100 gm. oil

The surfaces were bright but sticking occurred to such an extent that they were slightly damaged in separating. This suspension was of such a concentration that its cost was comparable to the other suitable materials.

.0704 gm./100 gm. oil

Sticking occurred and bending the sheets was necessary to separate them.

<u>Magnesium Stearate</u>

1.00% and 3.00% Solutions

Sticking occurred with both concentrations and the surface was bright but damaged. The sticking was about the same as if oil alone were used. A 1% solution is reported to work well, but it is found that even a 3% solution does not work under the conditions used in these tests(10).

<u>Zinc Stearate</u>

1.00% and 3.00% Solutions

The results of these runs check those for Magnesium Stearate.

<u>Magnesium Oxide, 250-mesh and finer</u>

3.50% and 1.75% Suspensions

With the higher concentration, no sticking occurred but the surface of the sheets was badly stained. With the lower concentration the same trouble was experienced, but not to as great an extent as with the 3.50% suspension.

(10) S. H. Bobrov: loc. cit.

APPENDIX

COST OF MATERIALS

Texaco Spindle Oil "B" .... 20.1¢/gal.*
The Texas Company,
Port Arthur, Texas

"Argosite Clay", 300-mesh bentonite .... 2.085¢/lb.**
United Clay Mines Corporation,
Trenton, N. J.

"Alberoyd Clay", 300-mesh soapstone .... 0.3¢/lb.**
Alberene Stone Corporation of Virginia,
Schuyler, Virginia

"Micatone B-1000", ... 5.0¢/lb.**
"Water-ground Mica", ... 5.5¢/lb.**
The English Mica Co.,
Spruce Pine, N. C.

* Tank car lots delivered in Atlanta, Georgia
** Ton lots delivered in Atlanta, Georgia

SAMPLE CALCULATION FOR COST OF SUSPENSIONS

"Argosite Clay", 3.26% by weight

Price: 2.085¢/lb.

Sp. Gr. of clay = 2.75   Sp. Gr. of Oil = 0.897

$\frac{3.26}{96.74}$ = .0337 lb. solid/lb. oil

.0337 x 8.34 x .897 = .252 lb. solid/gallon of oil

$\frac{.252 \times 1728}{2.75 \times 62.4}$ = 2.54 cu. in. of solid/ gallon of oil

$\frac{231 + 2.54}{231}$ = 1.01 gallons of suspension from 1 gallon of oil

Cost of solid/gal. oil = .252(2.085) = .525
Cost of oil per gallon = 20.1
Total Cost/gallon of oil = 20.625¢

Cost of Suspension/gallon = $\frac{20.625}{1.01}$ = 20.45¢

Cost of material to coat one ton of sheets:

Weight of suspension to coat one 3 x 3-1/2 in. sheet = .645 gms.

$\frac{.645 \times 144}{3 \times 3.5}$ = 8.84 gms./sq. ft. of sheet metal

Thickness of sheet = .04 inch.

$\frac{12(2000)}{.04(489)}$ = 1225 sq. ft. of sheet metal/ton.

$\frac{1225(8.84)}{453.6}$ = 23.91 lb. of suspension/ton of sheets.

Weight of suspension/gallon = $\frac{7.47 + .252}{1.01}$ = 7.63 lb./gallon

Cost of suspension for coating 1 ton = $\frac{23.91 \times 20.45}{7.63}$

= 64.2¢

SUMMARY OF COSTS

Material	Concentration of Suspension	Cost/gal. Cents	Cost/ton of sheets Cents
"Argosite Clay"	3.26%	20.45	64.2
"Albercyd Clay"	3.69%	20.08	63.0
"Micatone B-1000"	3.50%	21.34	66.8
"Water-ground Mica"	3.50%	21.47	67.2

CONCLUSIONS AND RECOMMENDATIONS

Only four of the materials studied in this investigation were suitable for use in the prevention of sticking. These materials were:

"Argosite Clay", 3.26% suspension

"Alberoyd Clay", 3.69% suspension

"Micatone B-1000", 3.5% suspension

"Water-ground Mica", 3.5% suspension

In arriving at a conclusion as to which of the materials is best, three factors are of prime importance: (1) the ease of maintaining a uniform suspension, (2) the cost of the suspension, and (3) the temperature range over which the material is useful.

"Alberoyd Clay" and "Argosite Clay" suspensions are both limited in use to temperatures of about 1400°F. and lower. The "Alberoyd Clay" suspension is slightly cheaper than the "Argosite Clay" suspension, but this slight difference is offset by the fact that the former settles more quickly and is more difficult to resuspend than the latter, hence would require a greater amount of agitation to maintain a uniform suspension. For these reasons both suspensions are recommended.

Both mica suspensions are useful up to 1600°F. No difference was noted in the suspending qualities of the two materials; hence, "Micatone B-1000" is recommended because it is cheaper than "Water-ground Mica".

Because of the difference in cost between the two clay suspensions and the mica suspensions, "Alberoyd Clay" or

"Argosite Clay" should be used for temperatures up to 1400°F. and "Micatone B-1000" from 1400°F. to 1600°F.

It is suggested that a cheaper method of coating the sheets might result from the use of rubber rolls instead of dipping. Such a method would have the advantage of giving a continuous process of coating sheets as well as supplying smaller quantities of suspensions to the surface of the metal. In using smaller quantities of suspensions on the surface of the sheets the concentration would probably have to be increased, thus lowering the cost of coating, since the cost of the solid material is negligible in comparison with the cost of the oil. Only one grade of oil was used in this work, but it is probable that a much cheaper oil would work.

Since the search for completely suitable suspending agent proved fruitless, further work could be done to improve the quality of the suspensions by finding such an agent, and thereby eliminating the cost of agitation. It is suggested also that work on this problem could be continued profitably by investigating the possibility of using a cheaper oil and other methods of applying the suspensions to the sheets.

CALIBRATION OF CONTROLLER

The thermocouple used with the controller was a chromel-alumel couple connected to the instrument with compensating leads.

The calibration was made against the melting points of the following materials:

Material	Melting Point, °F.
Tin	449.6
Bismuth	519.8
Lead	620.6
Zinc	786.2
Antimony	1166.0
Sodium Chloride	1479.2
Barium Chloride	1763.6
Copper	1981.4

The controller was calibrated by an indirect method in that a chromel-alumel thermocouple was calibrated, using a potentiometer set-up, and then using this thermocouple in the furnace along with that on the controller.

The procedure for the calibration was as follows: The thermocouple was placed in the molten material; the substance was then allowed to cool slowly and readings in millivolts were taken at 10 sec. intervals until the material had solidified. The temperature at which the readings in millivolts became constant was taken as the melting point of the material.

The calibrated thermocouple was then inserted in the furnace, along with the thermocouple from the controller, and the temperature was allowed to rise. In this manner, the controller was checked against the calibrated thermocouple, and found to be indicating the correct temperature.

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County of Fulton

State of Georgia

This is to certify that Thomas Jefferson Daniels is known to me personally and that he has appeared before me at Atlanta, Fulton County, Georgia on May 21, 1940 and has sworn that this paper, his thesis presented for a master's degree, entitled, "The Prevention Of Sticking In Bright-Annealing Sheet Steel," including the manuscript, blue prints, photographs, is an exact record of work originated and accomplished by the aforesaid Thomas Jefferson Daniels at the Georgia School of Technology beginning in October 1938 and completed April 30, 1940.

[redacted]

Sworn to before me at Atlanta,
Fulton County, Georgia, this
21 day of May, 1940.

[redacted]

Notary Public, Fulton County, Georgia.
My Commission Expires Feb. 5, 1944.