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THE EFFECT OF SMALL AMOUNTS OF
ANTIMONY ON THE TRANSFORMATION
AND PROPERTIES OF ALUMINUM

presented by

ARTHUR J. STONE

has been accepted towards fulfillment
of the requirements for

MS degree in MET. ENG.



Major professor

Date

May 27, 1947

THE EFFECT OF SMALL AMOUNTS OF ANTIMONY
ON THE TRANSFORMATION AND PROPERTIES OF
ALUMINUM

by

ARTHUR JAMES STONE

A THESIS

Submitted to the School of Graduate Studies of Michigan
State College of Agriculture and Applied Science
in partial fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

Department of Chemical and Metallurgical Engineering

1947

THESIS

TABLE OF CONTENTS

I.	Title Page	1
II.	Table of Contents	2
III.	Acknowledgement	3
IV.	Introduction	4
V.	An Investigation of the Equilibrium Diagram	
	General Theory	9
	Materials and Apparatus	13
	Thermocouple Calibration	16
	Procedure	22
	Tabulated Data	23
	Discussion	47
VI.	The Microstructure of the Alloys	
	General Theory	48
	Procedure	49
	Microstructure	50
	Discussion	58
VII.	Chemical Analysis	
	Introduction	60
	Procedure	61
	Tabulated Data	63
	Discussion	64
VIII.	Proposed Equilibrium Diagram	65
IX.	An Examination of the Physical Properties	
	Introduction	70
	Materials and Apparatus	71
	Procedure	72
	Physical Testing Data	74
	Discussion	75
X.	Conclusion	80
XI.	References	83

ACKNOWLEDGEMENT

The author is indebted to Mr. Robert L. Sweet and Mr. D.D. McGrady for their assistance, advice and guidance. The author also wishes to express his appreciation to Mr. H.L. Womochel for his advice, for the use of the Mechanical Engineering Research Chemistry Laboratory and for the machining of the physical test bars.

IV. INTRODUCTION

The effects of antimony upon the physical properties of aluminum and aluminum alloys are not generally known. However, it has been believed that the effects are detrimental. Small additions of antimony to aluminum-magnesium alloys were used at one time. At that time, it was claimed that the formation of antimony oxychloride caused good resistance to corrosion of these alloys, but it has been proven that aluminum-magnesium alloys have good corrosion resistance without the antimony additions. Additions of antimony are sometimes used in other aluminum alloys, but the reasons are seldom clear.

The main objective of the investigation was to be a study of the physical properties of additions of 1% or less antimony to aluminum. A survey of the literature for an appropriate equilibrium diagram was made. Upon discovering that there were several diagrams, none of which agreed, it was thought that an accurate equilibrium diagram would be a valuable addition to the literature and also would be necessary to the completion of the original investigation.

Of the several diagrams published, three are reproduced in this report, shown in figures 1, 2 and 3. Those reproduced are the equilibrium diagrams published by Dowdell, Jerabek, Forsyth, Green¹; Mondolfo²; and in the International Critical Tables³. Of those published,

FIG. 1

AL-36 DIAGRAM AS SHOWN IN METALLOGRAPHY
 BY DOWDEN, JERABEK, FORSYTH, GREEN.

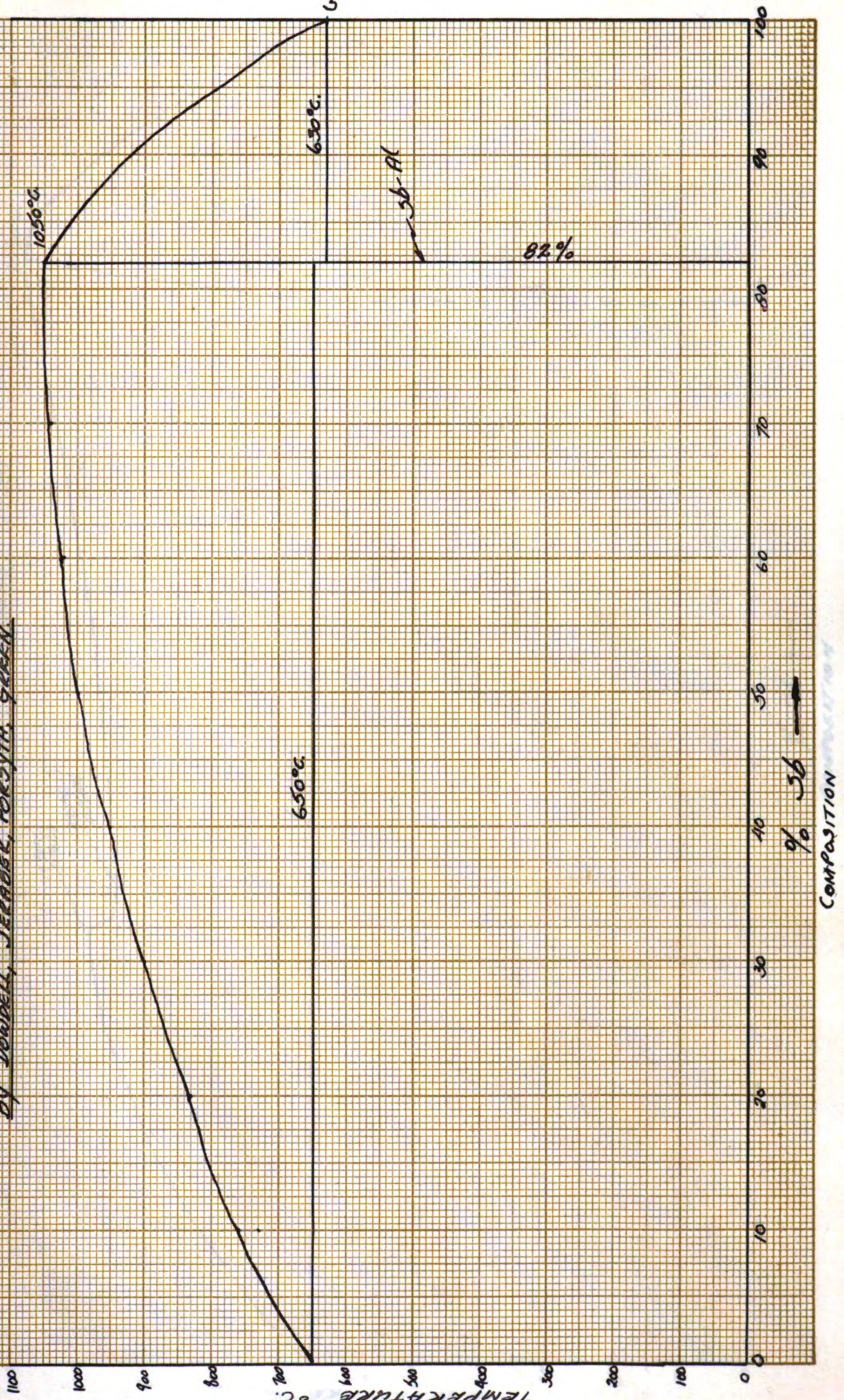


Fig. 2.

Al-36 Equilibrium Diagram As Shown
in the International Critical Tables. (1928)

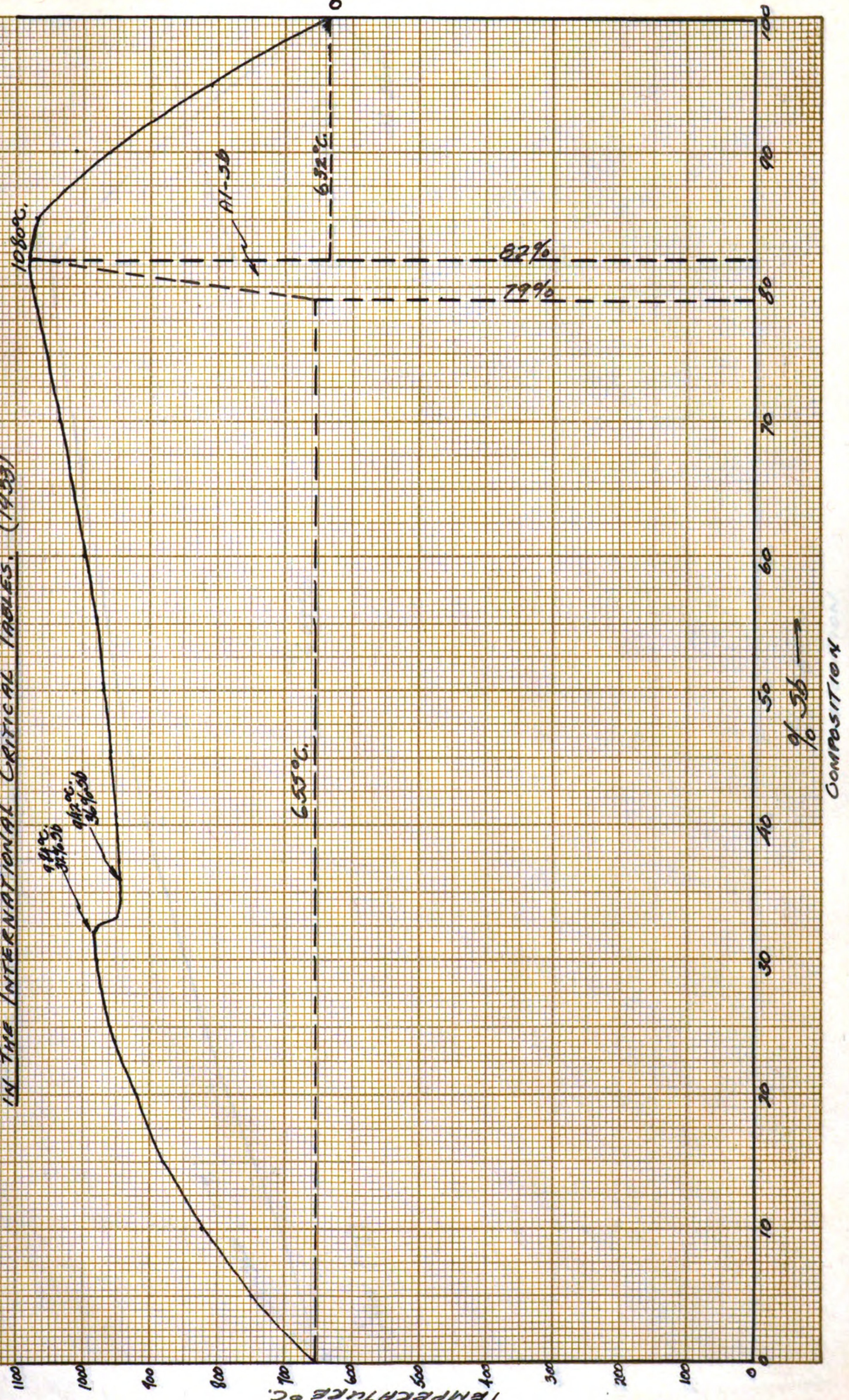
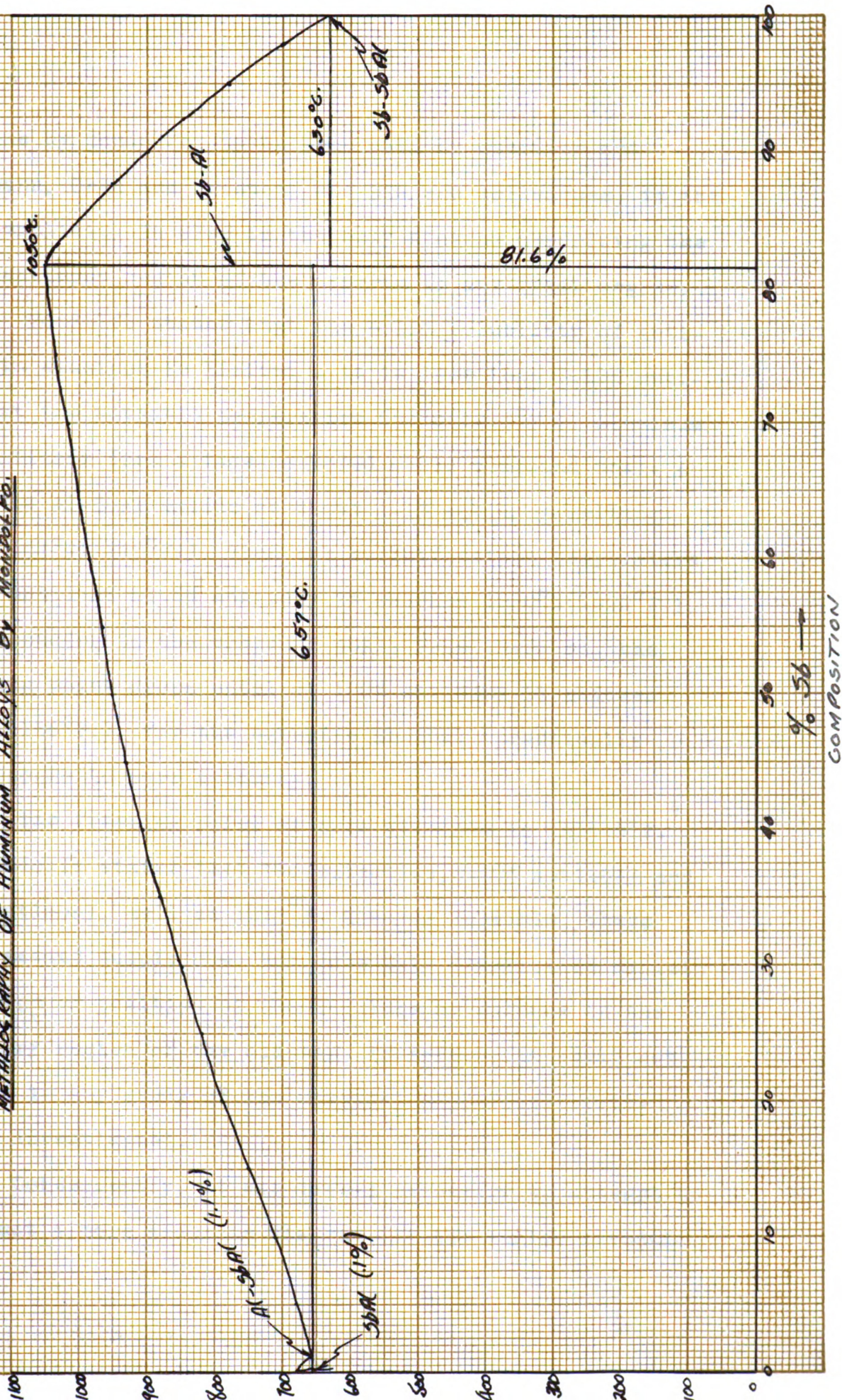


Fig. 3

Al-Sb EQUILIBRIUM DIAGRAM AS SHOWN IN
METALLOGRAPHY OF ALUMINUM ALLOYS BY MONROE ET AL.



the one by Mondolfo, taken from data by Guerther and Bergmann, Owen and Preston, and Dix and colleagues, is the most explicit. It also is the only diagram describing the 0-1% antimony region of the diagram to any great degree.

²
Mondolfo states that the solid solubility of antimony in aluminum is limited. It is less than 0.10% antimony at the eutectic temperature $657^{\circ}\text{C}(1215^{\circ}\text{F})$. Aluminum and antimony form a face-centered cubic compound SbAl . A eutectic Al-SbAl exists at about 1.1% antimony-melting point $657^{\circ}\text{C}(1215^{\circ}\text{F})$. Then the solidification temperature rises until at 81.6% antimony it reaches $1050^{\circ}\text{C}(1922^{\circ}\text{F})$, corresponding to the compound SbAl . From there on, the freezing point drops until at 100% antimony it reaches $630^{\circ}\text{C}(1166^{\circ}\text{F})$, for the eutectic Sb-SbAl practically corresponds to pure antimony.

⁴
The freezing point of pure aluminum is 1214.6°F . and Mondolfo states that two eutectics, 0.10% antimony and 1.1% antimony, melt at 1215°F . However possible, these facts seemed suspicious. Nor does this description explain the shape or position of the liquidus or solidus in the necessary region of the diagram. Therefore, it could not be used in any study of the physical properties of the alloys investigated.

Consequently, it was deemed necessary to attempt to construct a more accurate diagram for the region investigated.

V. AN INVESTIGATION OF THE EQUILIBRIUM DIAGRAM

GENERAL THEORY

Before outlining the specific properties which would tend to show what may be expected in the nature of an equilibrium diagram for additions of antimony to relatively pure aluminum, perhaps it would be well to repeat a few general characteristics of all additions of one metal to another to form alloys.

From the standpoint of crystal structure, solid solution alloys may be divided into two main classes, called, substitutional and interstitial types. The structure of any phase of the first may be viewed as though derived from a lattice of one of the constituents by replacing some of the atoms of this metal with atoms of the alloying metal. Therefore, as the composition is varied within the solubility limits of a given phase of a substitutional alloy, the variation takes place by the replacement of one kind of atom by the other. In interstitial alloys, one or more of the constituent atoms enter into the interstitial positions of another metal.

Since only four interstitial atoms namely hydrogen, carbon, nitrogen, and boron, are small enough to satisfy the conditions of an interstitial alloy⁵, the requirements for interstitial alloys will not be stated further.

⁶Carapella states that the extent to which atoms of solute replace atoms of solvent on the solvent lattice is termed solid solubility.

The type of crystal structure influences this factor. Complete solid solubility can only be expected with like crystal structure, provided all other factors are favorable. Metals with other crystal structures cannot form complete solid solutions with the solvent, for, by their very nature, they introduce at least one phase to the system that is not of the same crystal structure as the solvent.

Where the atomic sizes of the solvent and solute metals differ by less than 15%, the size factor is favorable^{5,6}. If the differences of atomic sizes exceed this limit the solubility is restricted. In fact, the greater the difference in size, the more restricted is the solubility if other factors are equal. Solute atoms which have atomic sizes just on the edge of the favorable zone tend to give erratic results. Moreover, if the atomic sizes differ by more than 8%, but still in the favorable zone, there is usually a minimum in the liquidus curves representing a definite tendency toward eutectic formation.

The more electropositive the solvent metal the more electronegative the solute metal, or visa versa, the greater the tendency to restrict solid solubility and to form intermetallic compounds⁶. The electronegative degree of metals in the periodic system of chemical compounds increases from left to right in any period and bottom to top in any group. That is, for wide ranges of composition, elements which alloy well lie near one another

in the electromotive series as well as having nearly equal radii.

A general trend, where size factor is favorable, is for solvent solutions to become more restricted as the valencies become more unequal⁶. Furthermore, the more unequal the valency factor, the steeper is the drop in both the liquidus and solidus curves.

As has been shown, crystallographic structure, size factor, the electronegative degree and the valencies of the metals in the alloy each tend to influence the limit of solid solubility, formation of intermetallic compounds and general shape of the liquidus and solidus in the equilibrium diagram.

Aluminum and antimony have very different crystal structure. Aluminum has a face-centered cubic lattice while antimony has a rhombohedral hexagonal. Thus aluminum and antimony cannot have much solubility.

The atomic diameters of aluminum and antimony vary by approximately 1%, antimony being slightly larger⁵. This factor does not restrict solubility nor does it cause a minimum in the liquidus curve.

Aluminum lies higher in the electromotive series than antimony. In fact, in the formation of intermetallic compounds, antimony is considered electronegative and aluminum electropositive⁵. This fact would tend to prevent solid solubility and cause the probability of the formation of intermetallic compounds.

Aluminum has a valence of three and antimony has a valence of three, four, or five, although three generally predominates. The fact that the valencies are generally the same favors formation of solid solution. Furthermore, this fact tends to flatten the liquidus and solidus curves.

With the foregoing facts in mind, a very restricted region of solid solubility, one or more intermetallic compounds, and a gradual slope of liquidus and solidus curves should be expected.

MATERIALS AND APPARATUS

A Hoskins Furnace, Type FA120, with a resistance type heating coil, having a capacity of 100 volts and 3.3 Amperes was used for melting the alloys. The aluminum used was from ingot 5 with impurities as shown in Table 1. The antimony added to the aluminum was listed as commercially pure. Since only tenths of a percent were added, it was thought that the small amounts of impurities would be negligible. An iron-constantan thermocouple of 24 B&S gauge wire was used, with melting ice in a vacuum flask as a cold-junction. A Leeds and Northrup "K" type potentiometer, with its galvanometer, light source and scale, was used to take the temperature readings. These readings were accurate to ± 0.01 mv. A single stop-watch was used for reading time and when once started was allowed to run to the completion of the test run. These time readings were accurate to the nearest 5 sec.

The rate of heating or cooling was controlled by the addition of external resistance and an ammeter and voltmeter were added as shown in Fig. 4, to the circuit to prevent overloading the furnace.

The melting was done in a sealed aluminum thin shell crucible, $1\frac{1}{4}$ " D X 4", as shown in Fig. 5.

TABLE I

COMPOSITION OF ALUMINUM USED
(Analyzed By Spectrographic Methods)
(By Percent)

<u>Ingot#</u>	<u>Cu</u>	<u>Zn</u>	<u>Fe</u>	<u>Ni</u>	<u>Mg</u>	<u>Si</u>	<u>Mn</u>	<u>Al</u>
1	.01	trace	.21	trace	.002	.13	trace	balance
2	"	"	.15	"	"	.12	"	"
3	"	"	.14	"	"	.12	"	"
4	"	"	.15	"	"	.12	"	"
5	"	"	.14	"	"	.14	"	"

TABLE II

ALLOY COMPOSITIONS
Impurities Subtracted from Aluminum
(By Weight)

<u>%Sb</u>	<u>Wt. Al</u> In Grams	<u>Wt. Sb</u> In Grams
.05	85.0112	.0425
.10	79.8475	.0798
.20	88.3837	.1768
.30	77.8697	.2336
.40	69.5252	.2771
.50	87.7758	.4389
.60	70.6842	.4241
.70	76.2426	.5337
.80	80.7050	.6456
.90	86.1890	.7757
1.00	92.9969	.9300
1.10	74.0836	.8149

TABLE IIIZINC ANALYSIS

Insoluble
in H_2SO_4 -, .02%
As - .000001
Pb - .005
Fe - .003
Total - .028001

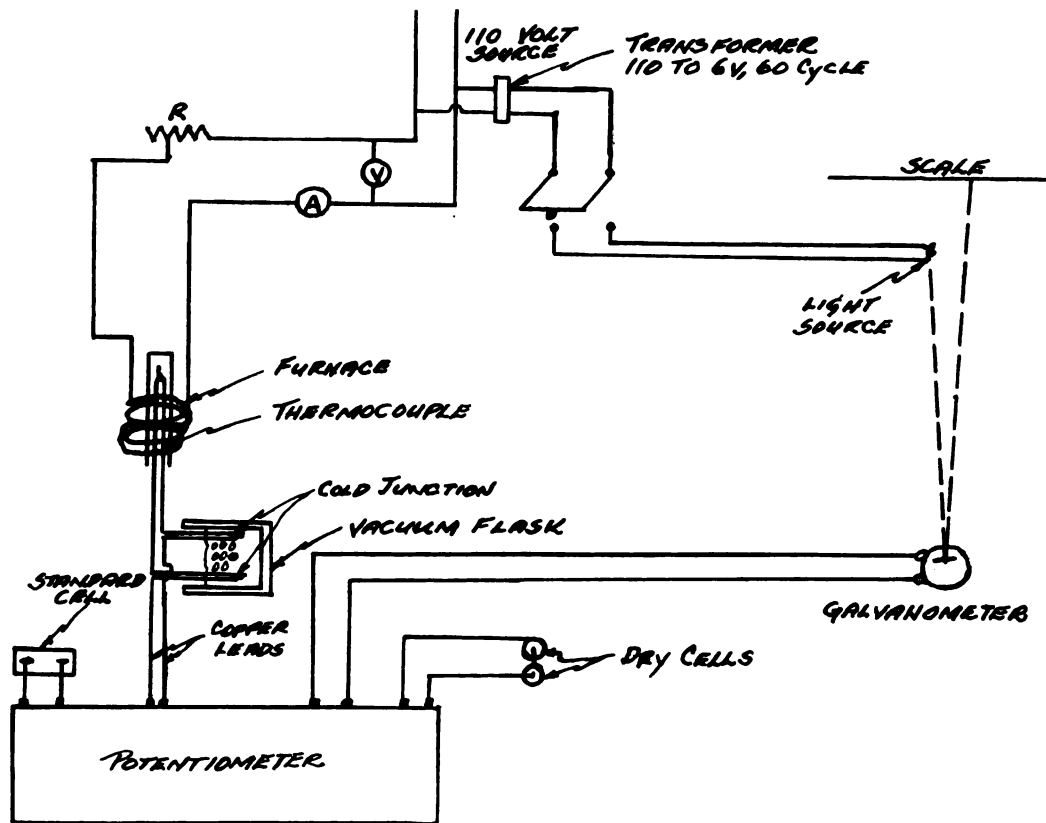


FIG. 4 WIRING DIAGRAM OF APPARATUS FOR SECTION 1.

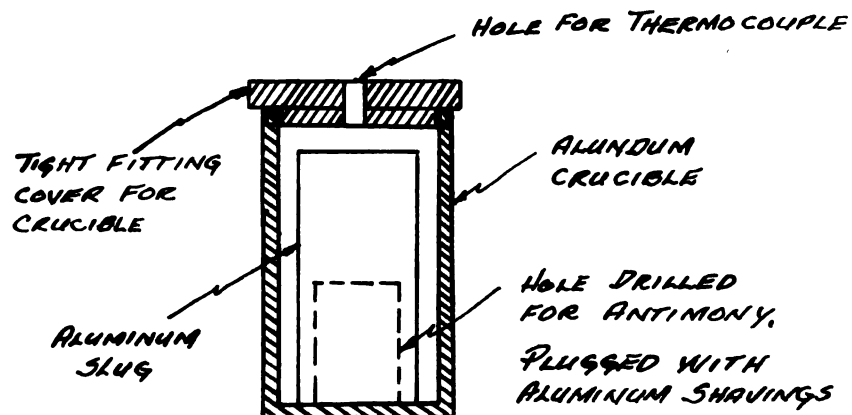


FIG. 5 DIAGRAM OF THE CRUCIBLE, SHOWING THE POSITION OF THE ALUMINUM AND ANTIMONY.

THERMOCOUPLE CALIBRATION

Before making any test runs for cooling curves, it was necessary to calibrate the iron-constantan thermocouple.

The most important temperature range for investigation of the alloys chosen was the range from 700°F. to 1400°F. Therefore, zinc with a melting point of 787°F. and pure aluminum with a melting point of 1214.6°F. were chosen to calibrate the thermocouple. The zinc analysis is shown in Table 3. The aluminum used was from ingot 5, with an analysis as shown in Table 1.

The cooling curves were made using the same procedure as that described in the following section. The results were as shown in Figures 6 and 7.

The thermocouple bead was covered with alundum cement. This covering was made as thin as possible to eliminate any hysteresis that might be caused by this covering. The thermocouple, with the exception of this bead, was covered with refractory tubing, commonly called "spagetti", two inches beyond the furnace. The wires were spread from there to the cold-junction to prevent any short to the system.

From the results shown it was agreed that the thermocouple was accurate within the probable error of the other equipment, which was less than .03%.

After calibrating the thermocouple, a heating and

cooling curve of the furnace were run. It was found that when holding the voltage of the furnace at 73 volts the temperature of 1000°F. could be held for an hour, once the temperature was reached.

It was decided, therefore, when running a heating curve, to maintain the voltage at 83-85 volts and when running a cooling curve, to maintain 63-65 volts.

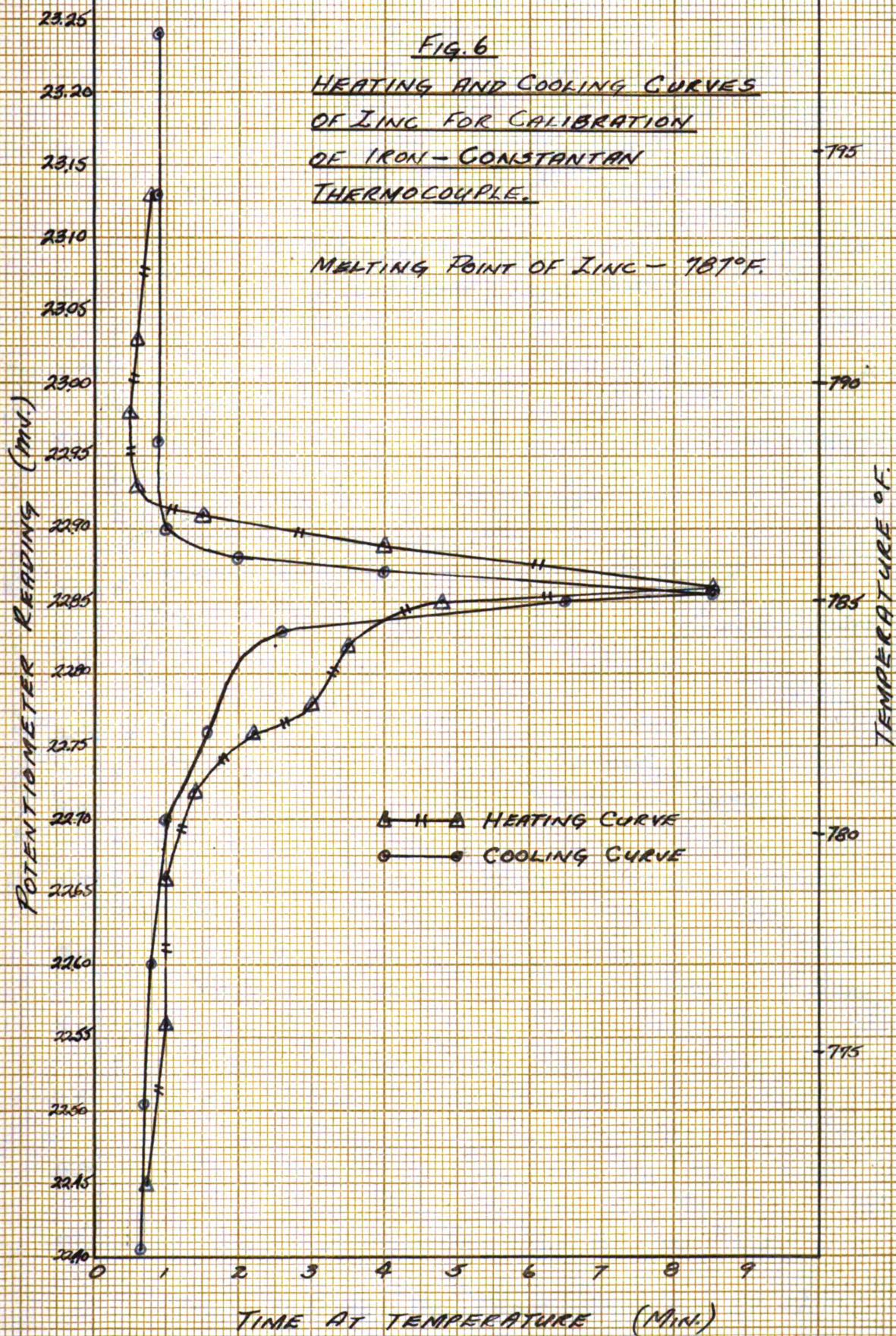
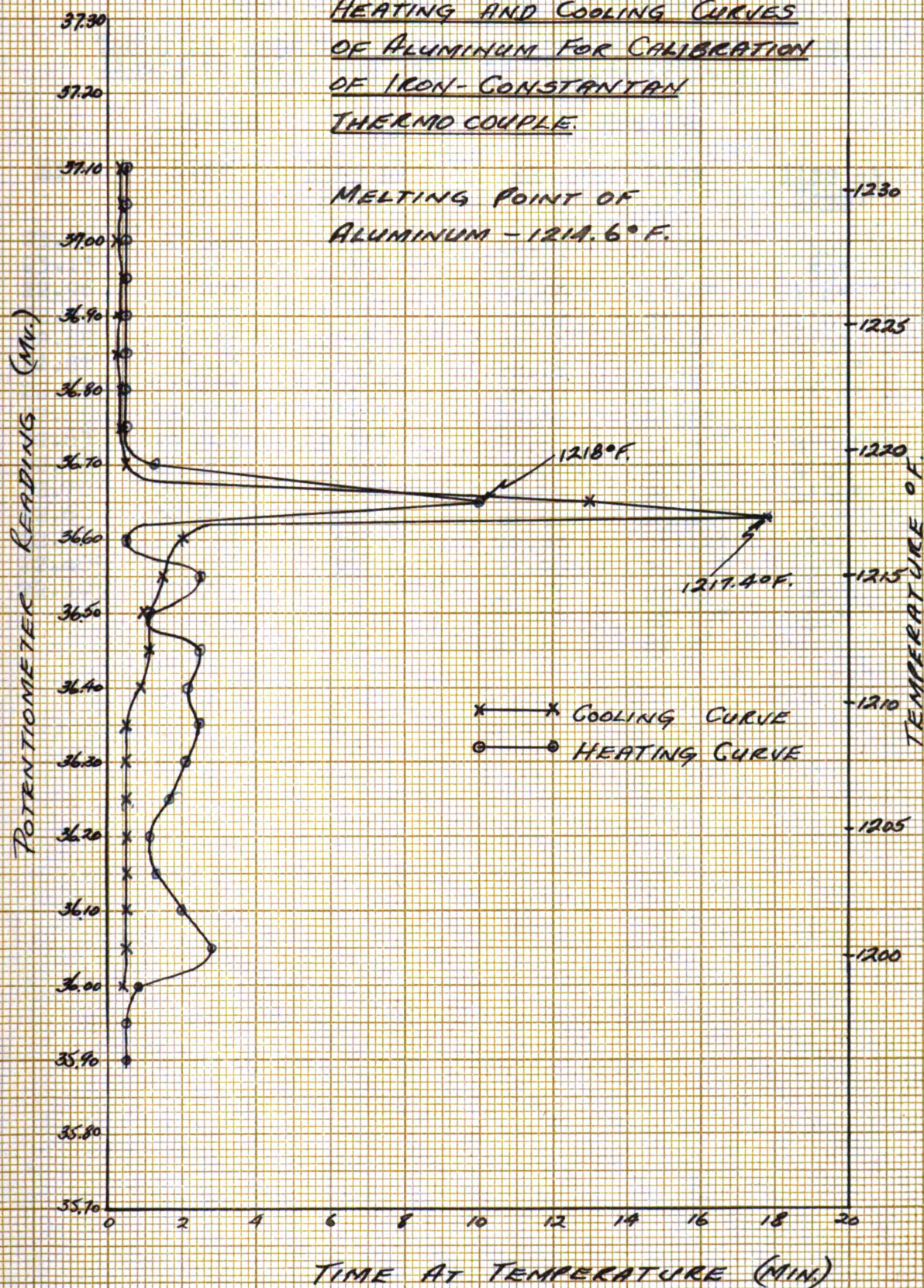


FIGURE 7
HEATING AND COOLING CURVES
OF ALUMINUM FOR CALIBRATION
OF IRON-CONSTANTAN
THERMO COUPLE.



DATA FOR THERMOCOUPLE CALIBRATIONTABLE IV Data for the transformation of zinc from liquid to solid and solid to liquid. (Figure 6)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
23.24	0'00"	55"	22.45	0'00"	50"
23.13	55"	55"	22.56	50"	55"
22.96	1'50"	55"	22.66	1'45"	1'0"
22.90	2'45"	1'0"	22.72	2'45"	1'0"
22.88	3'45"	2'0"	22.76	3'45"	1'0"
22.87	5'45"	4'0"	22.78	4'45"	1'20"
22.855	9'45"	8'35"	22.82	6'5"	2'10"
22.85	18'20"	6'30"	22.85	8'15"	3'25"
22.83	24'50"	2'35"	22.86	11'40"	8'35"
22.76	27'25"	1'35"	22.875	20'15"	4'0"
22.70	29'0"	1'0"	22.89	24'15"	1'30"
22.60	30'0"	50"	22.91	25'45"	30"
22.505	30'50"	35"	22.93	26'15"	35"
22.405	31'25"	35"	22.98	26'50"	37"
22.305	32'0"		23.03	27'27"	45"
			23.13	28'12"	50"
			23.23	29'2"	

TABLE V Data for the transformation of aluminum from liquid to solid and solid to liquid. (Figure 7)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
37.50	30'10"	20"	35.50	36'35"	25"
37.45	30'30"	20"	35.55	36'45"	10"
37.40	30'50"	20"	35.60	37'15"	30"
37.35	31'10"	15"	35.65	37'40"	25"
37.30	31'25"	20"	35.70	38'00"	20"
37.25	31'45"	20"	35.75	38'25"	25"
37.20	32'5"	15"	35.80	39'10"	45"
37.15	32'20"	20"	35.85	39'25"	15"
37.10	32'40"	20"	35.90	40'00"	35"
37.05	33'00"	15"	35.95	40'30"	30"
37.00	33'15"	15"	36.00	41'20"	50"
36.95	33'30"	30"	36.05	44'10"	2'50"
36.90	34'00"	15"	36.10	46'15"	2'5"
36.85	34'15"	15"	36.15	47'35"	1'20"
36.80	34'30"	25"	36.20	48'45"	1'10"
36.75	34'55"	25"	36.25	50'30"	1'45"
36.70	35'20"	35"	36.30	52'40"	2'10"
36.65	35'55"	17'40"	36.35	55'15"	2'35"
36.60	53'35"	2'0"	36.40	57'30"	2'15"
36.55	55'35"	1'30"	36.45	61'0"	2'30"
36.50	57'5"	1'0"	36.50	62'15"	1'15"
36.45	58'5"	1'10"	36.55	64'45"	2'30"
36.40	59'15"	50"	36.60	65'15"	30"
36.35	60'5"	30"	36.65	75'15"	10'0"
36.30	60'35"	30"	36.70	76'0"	1'45"
36.25	61'5"	30"	36.75	77'15"	25"
36.20	61'35"	25"	36.80	77'40"	25"
36.15	62'0"	25"	36.85	78'5"	25"
36.10	62'25"	20"	36.90	78'30"	25"
36.05	62'45"	15"	36.95	78'55"	25"
36.00	63'0"	15"	37.00	79'20"	20"
35.95	63'15"	15"	37.05	79'40"	25"
35.90	63'30"	15"	37.10	80'5"	20"
35.85	63'45"	15"	37.15	80'25"	20"
35.80	64'0"	20"	37.20	80'45"	15"
35.75	64'20"	15"	37.25	81'0"	20"
35.70	64'35"	15"	37.30	81'20"	15"
35.65	64'50"	15"	37.35	81'35"	20"
35.60	65'5"	10"	37.40	81'55"	20"
35.55	65'15"	10"	37.45	82'15"	20"
35.50	65'25"	10"	37.50	82'35"	20"
35.45	65'35"		37.55	82'55"	

PROCEDURE

The alloys used were made up on a weight percentage basis as shown in Table 2. The metals were placed in the crucible as shown in Fig.6. The crucible was covered with an alundum cover, placed in the Ni-Chrome wire carrier and the furnace sealed. After the metals had melted, the thermocouple was inserted, and the furnace resealed. The metal mixture was superheated to 1500°F. to assure as much diffusion as possible. A cooling curve was then run on the alloy, inserting a constant external resistance into the furnace circuit to cause the drop of any small increment of temperature to take place over a reasonably long period of time. After the cooling curve, a heating curve was run to determine the amount of hysteresis. The thermocouple was then withdrawn and the alloy allowed to cool in air.

A heating and cooling curve were run on all alloys. However, since there was such little hysteresis and no startling revelations other than that they paralleled one another and the cooling curve had fewer unreasonable fluctuations, the heating curves were omitted from the data.

TABULATED DATATABLE VI Data for the transformation of .05% antimony in aluminum. (Figure 8)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
37.00	0'00"	10"	35.50	9'00"	20"
36.95	10"	10"	35.55	9'20"	20"
36.90	20"	10"	35.60	9'40"	20"
36.85	30"	10"	35.65	10'00"	20"
36.80	40"	15"	35.70	10'20"	25"
36.75	55"	15"	35.75	10'45"	25"
36.70	1'10"	15"	35.80	11'10"	25"
36.65	11'20"	25"	35.85	11'35"	20"
36.60	22'45"	10'30"	35.90	11'55"	25"
36.55	27'15"	11'25"	35.95	12'20"	30"
36.50	30'20"	4'30"	36.00	12'50"	40"
36.45	33'0"	3'5"	36.05	13'30"	55"
36.40	34'55"	2'40"	36.10	14'25"	1'10"
36.35	36'15"	1'55"	36.15	15'35"	1'55"
36.30	37'25"	1'20"	36.20	17'30"	2'30"
36.25	38'10"	1'10"	36.25	20'0"	1'15"
36.20	38'55"	45"	36.30	21'15"	1'45"
36.15	39'30"	35"	36.35	23'0"	1'45"
36.10	40'15"	45"	36.40	24'45"	2'0"
36.05	40'35"	20"	36.45	26'45"	2'50"
36.00	41'10"	35"	36.50	29'35"	3'45"
35.95	41'50"	40"	36.55	33'20"	11'25"
35.90	42'55"	1'5"	36.60	44'45"	4'55"
35.85	43'25"	30"	36.65	49'40"	1'5"
35.80	43'45"	20"	36.70	50'45"	50"
35.75	44'5"	20"	36.75	51'35"	55"
35.70	44'25"	20"	36.80	52'30"	45"
35.65	44'45"	20"	36.85	53'15"	45"
35.60	45'5"	20"	36.90	54'0"	50"
35.55	45'20"	15"	36.95	54'50"	40"
35.50	45'35"	15"	37.00	55'30"	
35.45	45'55"				

TABLE VII Data for the transformation of .10%
antimony in aluminum. (Figure 9)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at</u> <u>Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at</u> <u>Temp.</u>
37.00	0'00"	20"	35.85	0'00"	1'5"
36.95	20"	25"	35.90	1'5"	1'15"
36.90	45"	25"	35.95	2'20"	1'0"
36.85	1'10"	25"	36.00	3'20"	50"
36.80	1'35"	25"	36.05	4'10"	50"
36.75	2'0"	25"	36.10	5'0"	55"
36.70	2'25"	25"	36.15	5'55"	1'15"
36.65	2'45"	30"	36.20	7'10"	1'15"
36.60	3'15"	25"	36.25	7'25"	2'30"
36.55	3'40"	25"	36.30	9'55"	2'15"
36.50	4'5"	25"	36.35	12'10"	3'55"
36.45	4'30"	35"	36.40	16'5"	3'50"
36.40	5'5"	55"	36.45	19'55"	4'55"
36.35	6'0"	30'30"	36.50	24'50"	3'5"
36.30	36'30"	3'45"	36.55	27'55"	50"
36.25	40'15"	1'20"	36.60	28'45"	25"
36.20	41'35"	1'20"	36.65	29'10"	35"
36.15	43'15"	1'10"	36.70	29'45"	15"
36.10	44'0"	1'15"	36.75	30'0"	15"
36.05	45'15"	35"	36.80	30'15"	35"
36.00	45'50"	30"	36.85	30'50"	35"
35.95	46'20"	35"	36.90	31'25"	35"
35.90	46'55"	25"	36.95	32'0"	30"
35.85	47'20"	25"	37.00	32'30"	30"
35.80	47'45"	30"	37.05	33'0"	
35.75	48'15"	30"			
35.70	48'45"				

TABLE VIII Data for the transformation of .20% antimony in aluminum. (Figure 10)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
37.50	0'00"	1'0"	35.50	2'7"	15"
37.45	1'0"	1'10"	35.55	2'22"	20"
37.40	2'10"	50"	35.60	2'42"	18"
37.35	3'0"	35"	35.65	2'60"	22"
37.30	3'35"	55"	35.70	3'22"	18"
37.25	4'30"	55"	35.75	3'40"	17"
37.20	5'25"	45"	35.80	3'57"	37"
37.15	6'10"	40"	35.85	4'30"	40"
37.10	6'50"	40"	35.90	5'10"	45"
37.05	7'30"	1'0"	35.95	5'55"	1'0"
37.00	8'30"	40"	36.00	6'55"	1'10"
36.95	9'10"	50"	36.05	8'5"	55"
36.90	10'0"	40"	36.10	9'0"	40"
36.85	10'40"	40"	36.15	9'40"	1'25"
36.80	11'20"	40"	36.20	11'5"	1'25"
36.75	12'0"	45"	36.25	12'30"	1'45"
36.70	12'45"	20"	36.30	14'15"	2'30"
36.65	13'5"	35"	36.35	16'45"	45"
36.60	13'40"	1'10"	36.40	17'30"	2'30"
36.55	14'50"	3'20"	36.45	20'0"	10'30"
36.50	18'10"	10'30"	36.50	30'50"	3'20"
36.50	18'10"	10'30"	36.50	30'50"	3'20"
36.45	28'40"	2'30"	36.55	34'10"	1'10"
36.40	31'10"	45"	36.60	35'20"	35"
36.35	31'55"	2'30"	36.65	35'55"	20"
36.30	34'25"	1'45"	36.70	36'15"	45"
36.25	36'10"	1'25"	36.75	37'0"	40"
36.20	37'35"	1'25"	36.80	37'40"	40"
36.15	39'0"	40"	36.85	38'20"	40"
36.10	39'40"	55"	36.90	39'0"	50"
36.05	40'35"	1'10"	36.95	39'50"	40"
36.00	41'45"	1'0"	37.00	40'30"	1'0"
35.95	42'45"	45"	37.05	41'30"	40"
35.90	43'30"	40"	37.10	42'10"	40"
35.85	44'10"	37"	37.15	42'50"	45"
35.80	44'47"	17"	37.20	43'35"	55"
35.75	45'04"	18"	37.25	44'30"	55"
35.70	45'22"	22"	37.30	45'25"	35"
35.65	45'44"	18"	37.35	46'0"	50"
35.60	46'2"	20"	37.40	46'50"	1'10"
35.55	46'22"	15"	37.45	48'0"	1'0"
35.50	46'37"		37.50	49'0"	

TABLE IX Data for the transformation of .30%
antimony in aluminum. (Figure 11)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at</u> <u>Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at</u> <u>Temp.</u>
37.00	0'00"	15"	35.50	1'15"	10"
36.95	15"	20"	35.55	1'25"	10"
36.90	35"	25"	35.60	1'35"	10"
36.85	1'0"	30"	35.65	1'45"	15"
36.80	1'30"	25"	35.70	2'0"	15"
36.75	1'55"	30"	35.75	2'15"	20"
36.70	2'25"	35"	35.80	2'35"	15"
36.65	3'0"	1'0"	35.85	2'50"	40"
36.60	4'0"	3'0"	35.90	3'30"	40"
36.55	7'0"	32'10"	35.95	4'10"	1'0"
36.50	39'10"	13'20"	36.00	5'10"	35"
36.45	52'30"	4'0"	36.05	5'45"	50"
36.40	56'30"	1'30"	36.10	6'35"	1'15"
36.35	58'0"	1'0"	36.15	7'50"	1'10"
36.30	59'0"	40"	36.20	9'0"	1'25"
36.25	59'40"	50"	36.25	10'25"	1'50"
36.20	60'30"	35"	36.30	12'15"	2'15"
36.15	61'5"	40"	36.35	14'30"	2'55"
36.10	61'45"	40"	36.40	17'25"	3'20"
36.05	62'25"	25"	36.45	20'45"	3'35"
36.00	62'50"	25"	36.50	24'20"	2'30"
35.95	63'15"	1'25"	36.55	26'50"	3'00"
35.90	64'40"	25"	36.60	29'50"	1'40"
35.85	65'5"	25"	36.65	31'30"	2'00"
35.80	65'30"	25"	36.70	33'30"	50"
35.75	65'55"	30"	36.75	34'20"	40"
35.70	66'25"	30"	36.80	35'0"	25"
35.65	67'0"	30"	36.85	35'25"	35"
35.60	67'25"	20"	36.90	36'0"	50"
35.55	67'45"	20"	36.95	36'50"	30"
35.50	68'5"		37.00	37'20"	

TABLE X Data for the transformation of .40% antimony in aluminum. (Figure 12)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
37.25	0'00"	30"	35.50	1'20"	10"
37.20	30"	25"	35.55	1'30"	30"
37.15	55"	25"	35.60	2'00"	10"
37.10	1'20"	35"	35.65	2'10"	15"
37.05	1'55"	35"	35.70	2'25"	20"
37.00	2'25"	30"	35.75	2'45"	15"
36.95	2'50"	25"	35.80	3'00"	25"
36.90	3'10"	20"	35.85	3'25"	30"
36.85	3'30"	20"	35.90	3'55"	35"
36.80	4'0"	30"	35.95	4'30"	1'15"
36.75	4'20"	20"	36.00	5'45"	1'35"
36.70	4'55"	35"	36.05	7'20"	2'10"
36.65	5'15"	20"	36.10	9'30"	2'00"
36.60	5'40"	3'00"	36.15	11'30"	1'20"
36.55	8'40"	3'00"	36.20	12'50"	1'30"
36.50	11'40"	19'20"	36.25	14'20"	1'45"
36.45	31'00"	4'5"	36.30	16'5"	2'10"
36.40	35'5"	1'50"	36.35	18'15"	2'35"
36.35	36'55"	1'5"	36.40	20'50"	3'10"
36.30	38'00"	40"	36.45	24'00"	3'45"
36.25	38'40"	50"	36.50	27'45"	3'15"
36.20	39'30"	30"	36.55	31'00"	2'00"
36.15	40'00"	30"	36.60	33'00"	1'50"
36.10	40'30"	25"	36.65	34'50"	1'35"
36.05	40'55"	1'00"	36.70	36'25"	1'35"
36.00	41'55"	25"	36.75	38'00"	1'10"
35.95	42'20"	20"	36.80	39'10"	1'15"
35.90	42'40"	20"	36.85	40'25"	1'5"
35.85	43'00"	20"	36.90	41'30"	1'5"
35.80	43'20"		36.95	42'35"	1'10"
			37.00	43'45"	45"
			37.05	44'30"	45"
			37.10	45'15"	45"
			37.15	46'00"	45"
			37.20	46'45"	35"
			37.25	47'20"	25"
			37.30	47'40"	10"
			37.35	47'50"	

TABLE XI Data for the transformation of .50% antimony in aluminum. (Figure 13)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
37.50	0'00"	10"	35.50	0'00"	15"
37.45	10"	15"	35.55	15"	15"
37.40	25"	15"	35.60	30"	10"
37.35	40"	15"	35.65	40"	15"
37.30	55"	15"	35.70	55"	20"
37.25	1'10"	20"	35.75	1'15"	20"
37.20	1'30"	20"	35.80	1'35"	30"
37.15	1'50"	20"	35.85	2'5"	45"
37.10	2'10"	15"	35.90	2'50"	1'10"
37.05	2'25"	25"	35.95	4'0"	1'30"
37.00	2'50"	20"	36.00	5'30"	45"
36.95	3'10"	20"	36.05	6'15"	20"
36.90	3'30"	30"	36.10	6'35"	1'0"
36.85	4'0"	35"	36.15	7'35"	2'5"
36.80	4'35"	50"	36.20	9'40"	1'55"
36.75	5'25"	1'15"	36.25	11'35"	2'30"
36.70	6'40"	2'10"	36.30	14'5"	2'40"
36.65	8'50"	13'25"	36.35	16'45"	3'0"
36.60	22'15"	14'5"	36.40	19'45"	3'20"
36.55	36'20"	3'10"	36.45	23'5"	4'50"
36.50	39'30"	2'20"	36.50	27'55"	8'50"
36.45	41'50"	1'45"	36.55	36'45"	7'15"
36.40	43'35"	1'10"	36.60	44'0"	2'20"
36.35	44'45"	55"	36.65	46'20"	45"
36.30	45'40"	55"	36.70	47'5"	55"
36.25	46'35"	25"	36.75	48'00"	1'0"
36.20	47'0"	35"	36.80	49'0"	1'0"
36.15	47'35"	30"	36.85	50'0"	1'10"
36.10	48'5"	30"	36.90	51'10"	1'15"
36.05	48'35"	30"	36.95	52'25"	1'10"
36.00	49'5"	25"	37.00	53'35"	1'10"
35.95	49'30"	30"	37.05	54'45"	1'40"
35.90	50'0"	30"	37.10	56'25"	1'10"
35.85	50'30"	50"	37.15	57'35"	1'25"
35.80	51'20"	25"	37.20	59'0"	1'15"
35.75	51'45"	20"	37.25	60'15"	45"
35.70	52'5"	15"	37.30	61'0"	30"
35.65	52'20"	15"	37.35	61'30"	15"
35.60	52'35"	15"	37.40	61'45"	10"
35.55	52'50"	15"	37.45	61'55"	10"
35.50	53'5"		37.50	62'5"	

TABLE XII Data for the transformation of .60% antimony in aluminum. (Figure 14)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
37.50	0'00"	10"	36.00	0'00"	1'5"
37.45	10"	15"	36.05	1'5"	1'10"
37.40	25"	15"	36.10	2'15"	2'5"
37.35	40"	20"	36.15	4'20"	2'40"
37.30	1'0"	20"	36.20	7'0"	1'45"
37.25	1'20"	25"	36.25	8'45"	2'0"
37.20	1'45"	25"	36.30	10'45"	1'30"
37.15	2'10"	20"	36.35	12'15"	2'0"
37.10	2'30"	25"	36.40	14'15"	3'15"
37.05	2'55"	30"	36.45	17'30"	3'10"
37.00	3'25"	25"	36.50	20'40"	5'15"
36.95	3'50"	20"	36.55	25'55"	5'55"
36.90	4'10"	35"	36.60	31'50"	6'40"
36.85	4'45"	25"	36.65	38'30"	2'40"
36.80	5'10"	35"	36.70	41'10"	1'25"
36.75	5'45"	35"	36.75	42'35"	1'40"
36.70	6'20"	3'30"	36.80	44'15"	1'15"
36.65	9'50"	16'55"	36.85	45'30"	1'0"
36.60	26'45"	10'25"	36.90	46'30"	1'20"
36.55	37'10"	4'0"	36.95	47'50"	1'0"
36.50	41'10"	3'0"	37.00	48'50"	1'15"
36.45	44'10"	2'10"	37.05	20'5"	1'5"
36.40	46'20"	2'0"	37.10	21'10"	1'5"
36.35	48'20"	1'5"	37.15	21'45"	35"
36.30	49'25"	50"	37.20	22'0"	15"
36.25	50'15"	45"	37.25	22'10"	10"
36.20	51'0"	40"	37.30	22'20"	10"
36.15	51'40"	35"	37.35	22'30"	10"
36.10	52'15"	35"	37.40	22'40"	10"
36.05	52'50"	30"	37.45	22'50"	15"
36.00	53'20"	40"	37.50	23'5"	
35.95	54'0"	45"			
35.90	54'45"	35"			
35.85	55'20"	25"			
35.80	55'45"	30"			
35.75	56'15"	25"			
35.70	56'40"	20"			
35.65	57'00"	15"			
35.60	57'15"	15"			
35.55	57'30"	15"			
35.50	57'45"				

TABLE XIII Data for the transformation of .70% antimony in aluminum. (Figure 15)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
37.50	4'25"	15"	35.50	0'00"	10"
37.45	4'40"	30"	35.55	10"	10"
37.40	5'10"	15"	35.60	20"	10"
37.35	5'25"	10"	35.65	30"	25"
37.30	5'35"	30"	35.70	55"	45"
37.25	6'5"	15"	35.75	1'40"	1'5"
37.20	6'20"	20"	35.80	2'45"	1'40"
37.15	6'40"	15"	35.85	4'25"	4'35"
37.10	6'55"	20"	35.90	9'0"	1'45"
37.05	7'15"	20"	35.95	10'45"	2'10"
37.00	7'35"	20"	36.00	12'55"	3'25"
36.95	7'55"	25"	36.05	15'20"	4'55"
36.90	8'20"	20"	36.10	20'15"	3'10"
36.85	8'40"	25"	36.15	23'25"	2'35"
36.80	9'5"	35"	36.20	26'00"	25"
36.75	9'40"	30"	36.25	26'25"	25"
36.70	10'10"	10"	36.30	33'40"	10'10"
36.65	10'20"	30"	36.35	43'50"	1'20"
36.60	10'50"	25"	36.40	45'10"	40"
36.55	11'15"	15"	36.45	45'50"	1'10"
36.50	11'40"	30"	36.50	47'00"	1'0"
36.45	12'10"	1'10"	36.55	48'00"	1'30"
36.40	13'20"	1'50"	36.60	49'30"	45"
36.35	15'10"	7'50"	36.65	50'15"	1'15"
36.30	23'0"	19'5"	36.70	51'30"	1'0"
36.25	42'5"	4'30"	36.75	52'30"	30"
36.20	46'35"	3'5"	36.80	53'00"	25"
36.15	49'40"	2'35"	36.85	53'25"	25"
36.10	52'15"	1'30"	36.90	53'50"	10"
36.05	53'45"	1'10"	36.95	54'00"	10"
36.00	54'55"	1'10"	37.00	54'10"	10"
35.95	56'5"	45"	37.05	54'20"	10"
35.90	56'50"	40"	37.10	54'30"	10"
35.85	57'30"	30"	37.15	54'40"	10"
35.80	58'0"	30"	37.20	54'50"	10"
35.75	58'30"	30"	37.25	55'00"	10"
35.70	59'0"	20"	37.30	55'10"	15"
35.65	59'20"	25"	37.35	55'25"	10"
35.60	59'45"	35"	37.40	55'35"	15"
35.55	60'20"	1'15"	37.45	55'50"	10"
35.50	61'35"		37.50	56'00"	

TABLE XIV Data for the transformation of .80% antimony in aluminum. (Figure 16)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
37.50	8'15"	20"	35.50	0'00"	25"
37.45	8'35"	30"	35.55	25"	30"
37.40	9'5"	20"	35.60	55"	25"
37.35	9'25"	25"	35.65	1'20"	30"
37.30	9'50"	25"	35.70	1'50"	30"
37.25	10'15"	25"	35.75	2'20"	30"
37.20	10'40"	25"	35.80	2'50"	35"
37.15	11'5"	25"	35.85	3'25"	45"
37.10	11'30"	25"	35.90	4'10"	1'00"
37.05	11'55"	25"	35.95	5'10"	1'10"
37.00	12'20"	30"	36.00	6'30"	1'20"
36.95	12'50"	25"	36.05	7'50"	2'30"
36.90	13'15"	25"	36.10	10'20"	2'00"
36.85	13'40"	30"	36.15	12'20"	1'50"
36.80	14'10"	30"	36.20	14'10"	1'55"
36.75	14'40"	40"	36.25	16'5"	2'10"
36.70	15'20"	55"	36.30	18'15"	2'30"
36.65	16'15"	1'45"	36.35	20'45"	3'30"
36.60	18'00"	2'5"	36.40	24'15"	4'45"
36.55	20'5"	16'40"	36.45	29'00"	6'50"
36.50	36'45"	11'00"	36.50	35'50"	6'35"
36.45	47'45"	3'35"	36.55	42'25"	2'5"
36.40	51'20"	2'00"	36.60	44'30"	50"
36.35	53'20"	1'25"	36.65	45'20"	1'25"
36.30	54'45"	1'00"	36.70	46'45"	1'20"
36.25	55'45"	45"	36.75	48'5"	1'20"
36.20	56'30"	40"	36.80	49'25"	1'00"
36.15	57'10"	30"	36.85	50'25"	1'10"
36.10	57'40"	35"	36.90	51'35"	1'15"
36.05	58'15"	25"	36.95	52'50"	1'10"
36.00	58'40"	25"	37.00	54'00"	1'15"
35.95	59'5"	25"	37.05	55'15"	1'10"
35.90	59'30"	25"	37.10	56'25"	1'20"
35.85	59'55"	30"	37.15	57'45"	1'15"
35.80	60'25"	1'15"	37.20	59'00"	1'10"
35.75	61'40"	10"	37.25	60'10"	10"
35.70	61'50"	25"	37.30	60'20"	10"
35.65	62'15"	15"	37.35	60'30"	10"
35.60	62'30"	15"	37.40	60'40"	15"
35.55	62'45"	15"	37.45	60'55"	10"
35.50	63'00"		37.50	61'5"	

TABLE XV Data for the transformation of .90% antimony in aluminum. (Figure 17)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time At Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time At Temp.</u>
37.50	6'5"	25"	35.50	0'00"	10"
37.45	6'30"	25"	35.55	10"	20"
37.40	6'55"	20"	35.60	30"	25"
37.35	7'15"	25"	35.65	55"	35"
37.30	7'40"	25"	35.70	1'30"	1'35"
37.25	8'5"	25"	35.75	3'5"	1'45"
37.20	8'30"	25"	35.80	4'50"	1'40"
37.15	8'55"	25"	35.85	6'30"	1'25"
37.10	9'20"	25"	35.90	7'55"	1'30"
37.05	9'45"	25"	35.95	9'25"	1'35"
37.00	10'10"	25"	36.00	11'00"	2'5"
36.95	10'35"	30"	36.05	13'5"	3'30"
36.90	11'5"	25"	36.10	16'35"	4'45"
36.85	11'30"	25"	36.15	21'20"	6'40"
36.80	11'55"	25"	36.20	28'00"	5'25"
36.75	12'20"	25"	36.25	33'25"	4'00"
36.70	12'45"	25"	36.30	37'25"	1'5"
36.65	13'10"	25"	36.35	38'30"	1'0"
36.60	13'35"	25"	36.40	39'30"	30"
36.55	14'0"	25"	36.45	40'00"	50"
36.50	14'25"	30"	36.50	40'50"	50"
36.45	14'50"	45"	36.55	41'40"	50"
36.40	15'40"	1'50"	36.60	42'30"	55"
36.35	17'30"	2'50"	36.65	43'25"	40"
36.30	20'20"	2'30"	36.70	44'5"	55"
36.25	33'10"	9'55"	36.75	45'00"	35"
36.20	41'50"	8'40"	36.80	45'35"	15"
36.15	46'35"	4'45"	36.85	45'50"	15"
36.10	49'50"	3'15"	36.90	46'5"	15"
36.05	52'30"	2'40"	36.95	46'20"	15"
36.00	54'40"	2'10"	37.00	46'35"	20"
35.95	57'0"	2'20"	37.05	46'55"	10"
35.90	59'15"	2'15"	37.10	47'05"	10"
35.85	61'25"	2'10"	37.15	47'15"	10"
35.80	63'5"	1'40"	37.20	47'25"	5"
35.75	64'15"	1'10"	37.25	47'30"	10"
35.70	65'5"	50"	37.30	47'40"	10"
35.65	65'50"	45"	37.35	47'50"	10"
35.60	66'25"	35"	37.40	48'00"	15"
35.55	67'0"	35"	37.45	48'15"	10"
35.50	67'35"		37.50	48'25"	

TABLE XVI Data for the transformation of 1.00%
antimony in aluminum. (Figure 18)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at</u> <u>Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at</u> <u>Temp.</u>
37.50	3'00"	20"	35.50	0'00"	10"
37.45	3'20"	20"	35.55	10"	20"
37.40	3'40"	20"	35.60	20"	15"
37.35	4'00"	20"	35.65	35"	15"
37.30	4'20"	20"	35.70	50"	20"
37.25	4'40"	15"	35.75	1'10"	15"
37.20	4'55"	20"	35.80	1'25"	25"
37.15	5'15"	20"	35.85	1'50"	30"
37.10	5'35"	20"	35.90	2'20"	40"
37.05	5'55"	20"	35.95	3'00"	55"
37.00	6'15"	15"	36.00	3'55"	1'15"
36.95	6'30"	20"	36.05	5'10"	1'15"
36.90	6'50"	25"	36.10	6'25"	1'15"
36.85	7'15"	20"	36.15	7'40"	1'30"
36.80	7'35"	30"	36.20	9'10"	1'45"
36.75	8'5"	45"	36.25	10'55"	2'5"
36.70	8'50"	55"	36.30	13'00"	3'0"
36.65	9'45"	55"	36.35	16'00"	3'40"
36.60	10'40"	1'20"	36.40	19'40"	5'20"
36.55	12'00"	2'20"	36.45	25'00"	6'00"
36.50	14'20"	16'15"	36.50	31'00"	3'5"
36.45	30'35"	3'25"	36.55	34'5"	1'15"
36.40	34'00"	3'25"	36.60	35'20"	55"
36.35	37'25"	2'0"	36.65	36'15"	55"
36.30	39'25"	1'30"	36.70	37'10"	50"
36.25	40'55"	1'5"	36.75	38'0"	1'10"
36.20	42'00"	55"	36.80	39'10"	45"
36.15	42'55"	35"	36.85	39'55"	50"
36.10	43'30"	30"	36.90	40'45"	45"
36.05	44'00"	25"	36.95	41'30"	55"
36.00	44'25"	20"	37.00	42'25"	55"
35.95	44'45"	20"	37.05	43'20"	40"
35.90	45'5"	20"	37.10	44'00"	50"
35.85	45'25"	15"	37.15	44'50"	45"
35.80	45'40"	15"	37.20	45'35"	50"
35.75	45'55"	15"	37.25	46'25"	50"
35.70	46'10"	15"	37.30	47'15"	50"
35.65	46'25"	15"	37.35	48'5"	50"
35.60	46'40"	20"	37.40	48'55"	40"
35.55	47'00"	20"	37.45	49'35"	45"
35.50	47'20"		37.50	50'20"	

TABLE XVIII Data for the transformation of 1.10% antimony in aluminum. (Figure 19)

<u>Cooling</u>			<u>Heating</u>		
<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>	<u>Mv.</u>	<u>Time</u>	<u>Time at Temp.</u>
36.65	0'00"	10"	35.50		
36.60	10"	30"	35.55	0'00"	1'10"
36.55	40"	1'20"	35.60	1'10"	1'5"
36.50	2'0"	3'30"	35.65	2'15"	1'0"
36.45	5'30"	3'55"	35.70	3'15"	1'5"
36.40	9'25"	3'15"	35.75	4'20"	1'5"
36.35	12'40"	3'10"	35.80	5'25"	1'15"
36.30	15'50"	2'15"	35.85	6'40"	1'10"
36.25	18'5"	3'10"	35.90	7'50"	1'25"
36.20	21'15"	2'45"	35.95	9'15"	1'20"
36.15	24'0"	2'45"	36.00	10'35"	1'45"
36.10	26'45"	2'35"	36.05	12'20"	1'50"
36.05	29'20"	2'5"	36.10	14'10"	2'10"
36.00	31'25"	1'25"	36.15	16'20"	2'20"
35.95	32'50"	1'0"	36.20	18'40"	3'30"
35.90	33'50"	55"	36.25	22'10"	3'25"
35.85	34'45"	55"	36.30	25'35"	5'20"
35.80	35'40"	50"	36.35	30'55"	3'20"
35.75	36'30"	40"	36.40	34'15"	3'55"
35.70	37'10"	35"	36.45	38'10"	2'25"
35.65	37'45"	30"	36.50	40'35"	1'25"
35.60	38'15"	40"	36.55	42'0"	1'5"
35.55	38'55"	25"	36.60	43'5"	1'0"
35.50	39'20"		36.65	44'5"	40"
			36.70	44'50"	35"
			36.75	45'25"	35"
			36.80	46'00"	25"
			36.85	46'25"	10"
			36.90	46'35"	10"
			36.95	46'45"	10"
			37.00	46'55"	

FIGURE 8

THE TRANSFORMATION OF THE
ALLOY CONTAINING .05% ANTIMONY
IN ALUMINUM UPON COOLING.

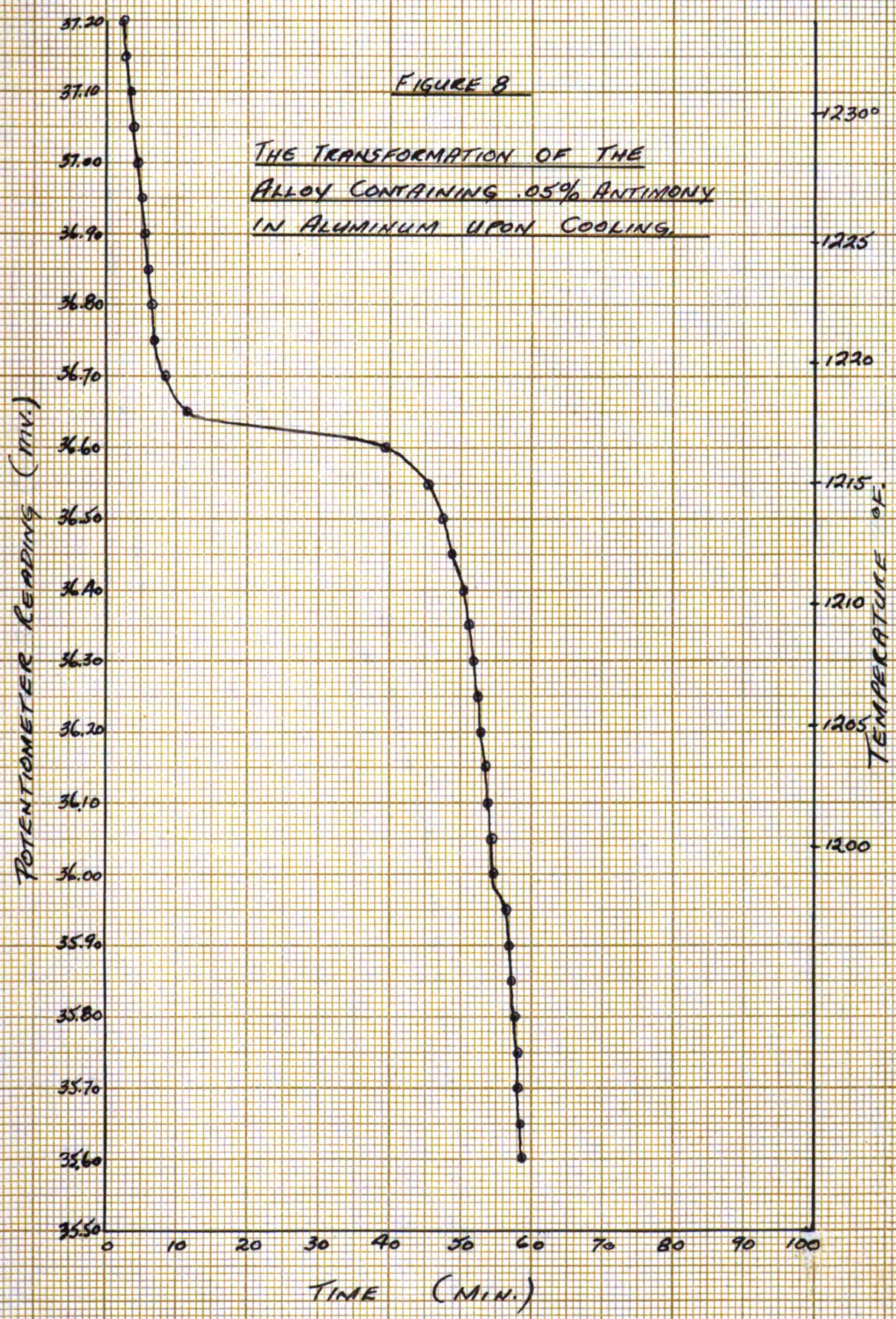


FIGURE 9

THE TRANSFORMATION OF THE
ALLOY CONTAINING .10% ANTIMONY
IN ALUMINUM UPON COOLING.

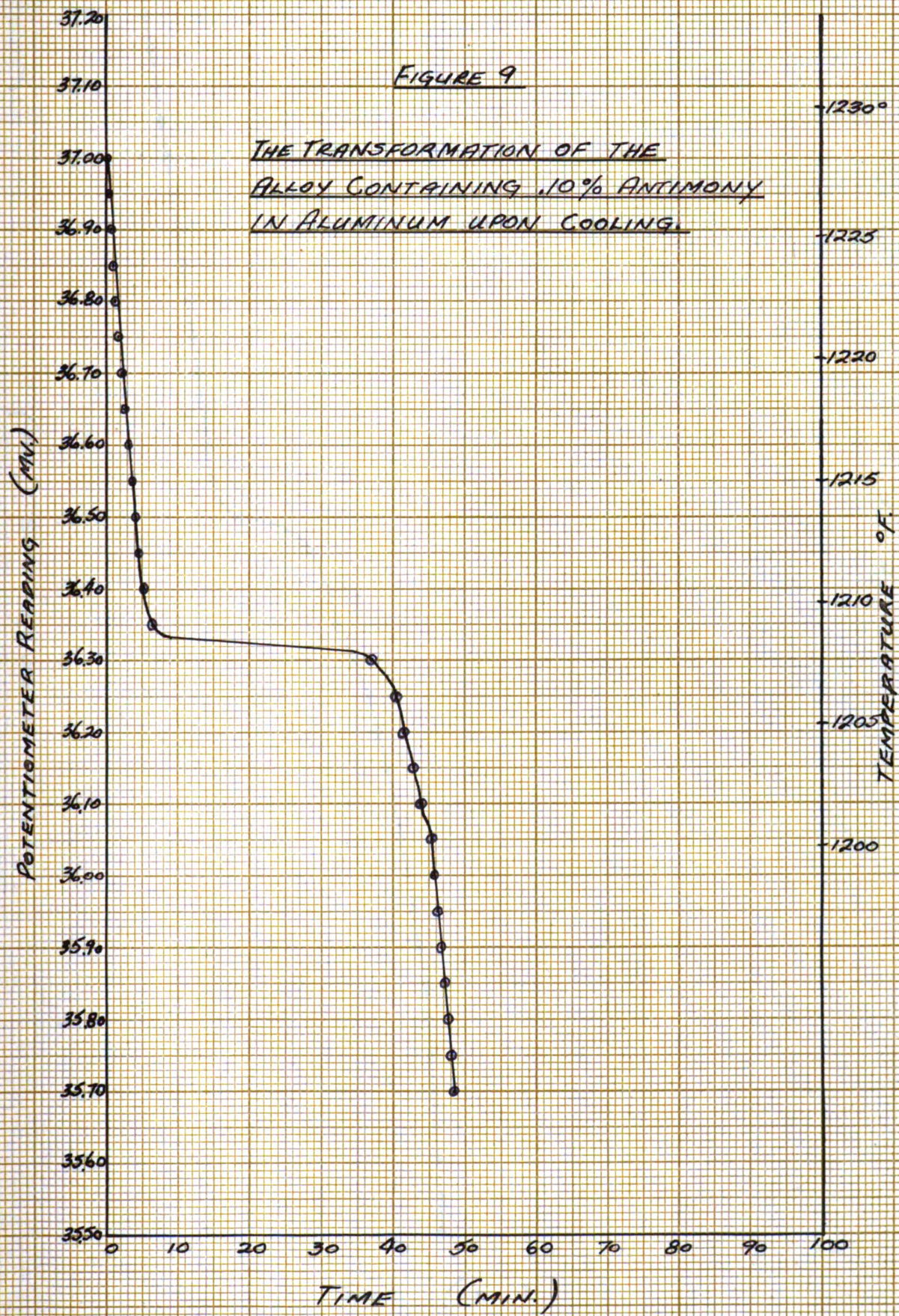


FIGURE 10

THE TRANSFORMATION OF THE
ALLOY CONTAINING .20% ANTIMONY
IN ALUMINUM UPON COOLING.

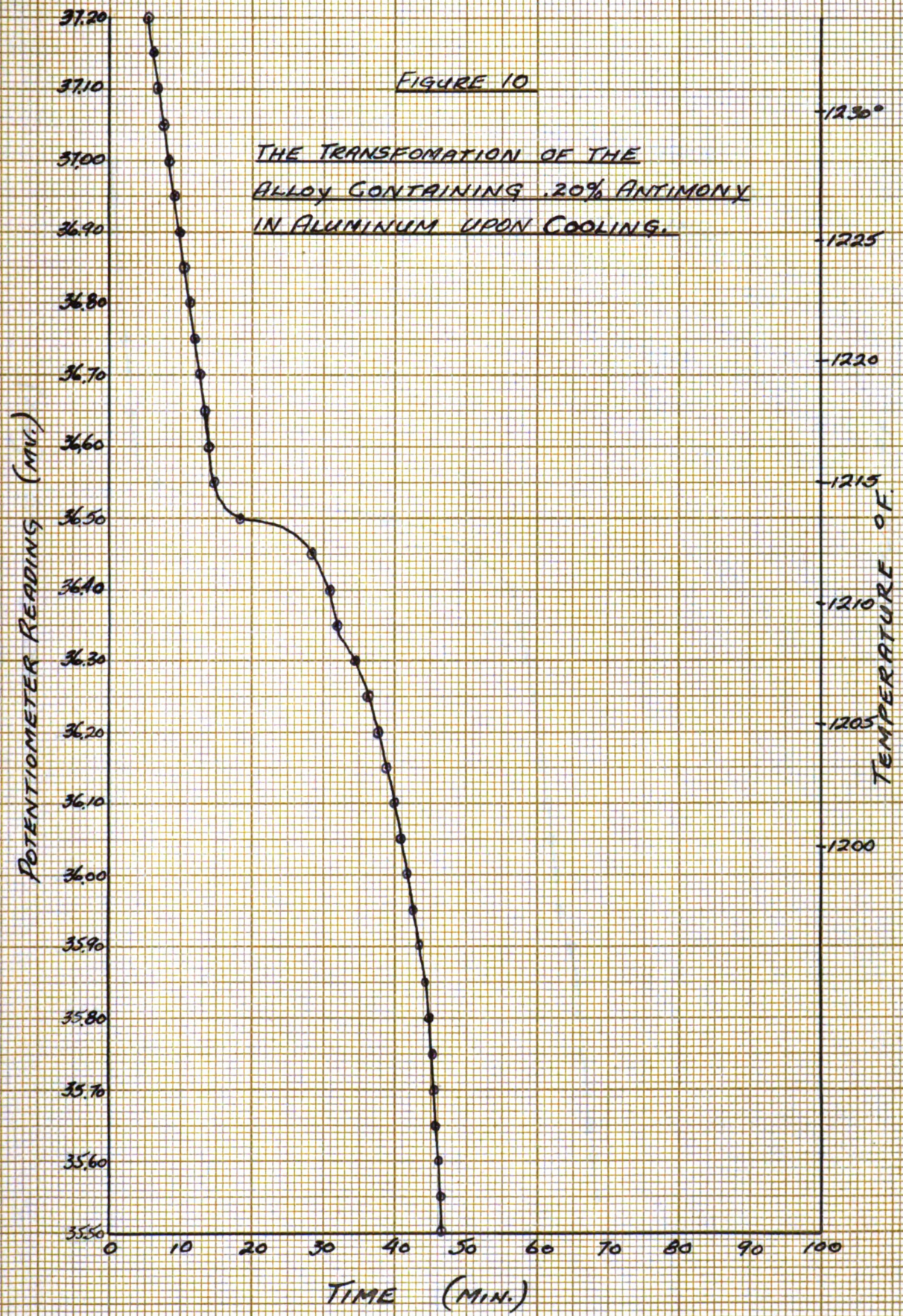


FIGURE 11

THE TRANSFORMATION OF THE
ALLOY CONTAINING 30% ANTIMONY
IN ALUMINUM UPON COOLING.

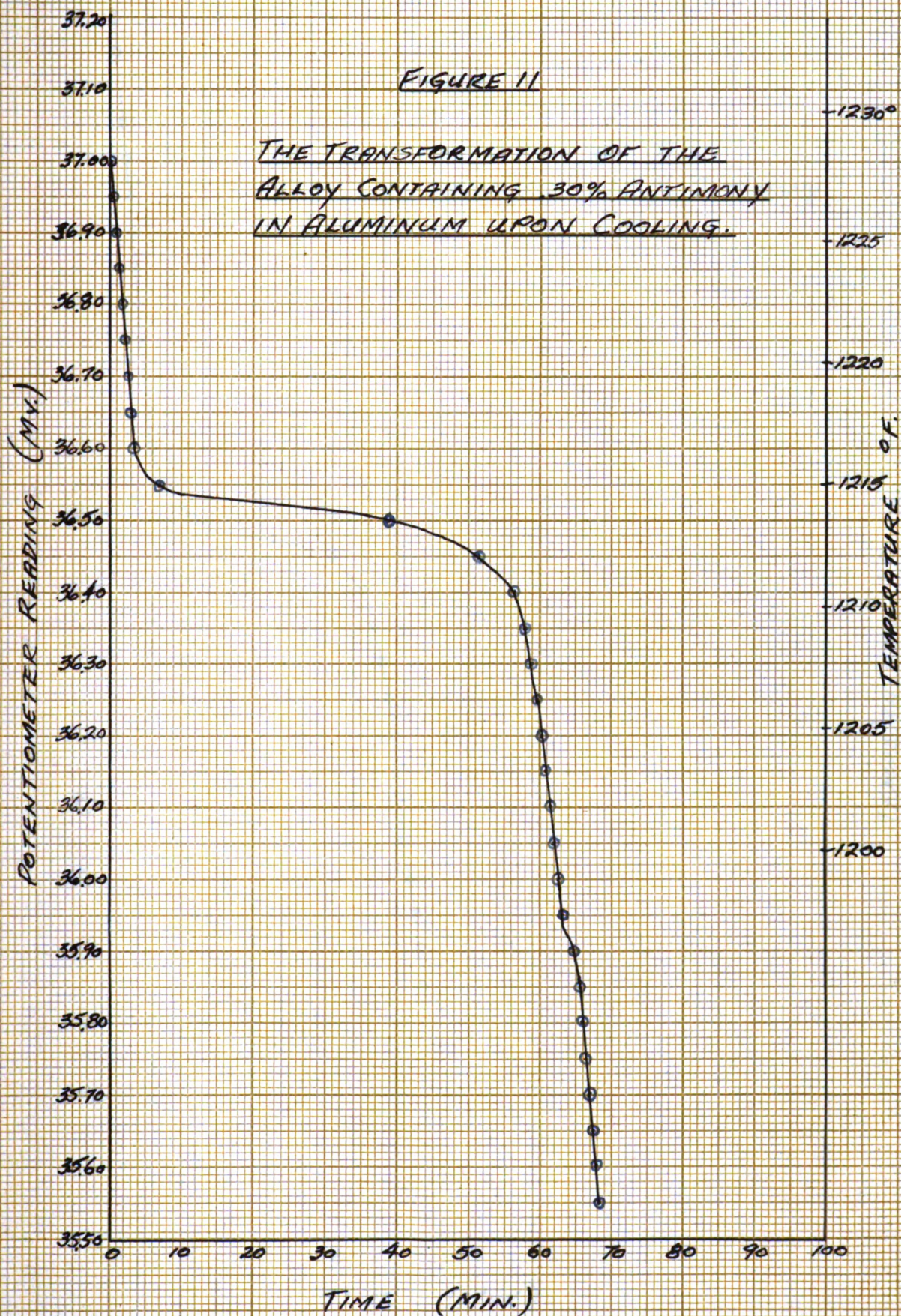


FIGURE 13

THE TRANSFORMATION OF THE
ALLOY CONTAINING .50% ANTIMONY
IN ALUMINUM UPON COOLING.

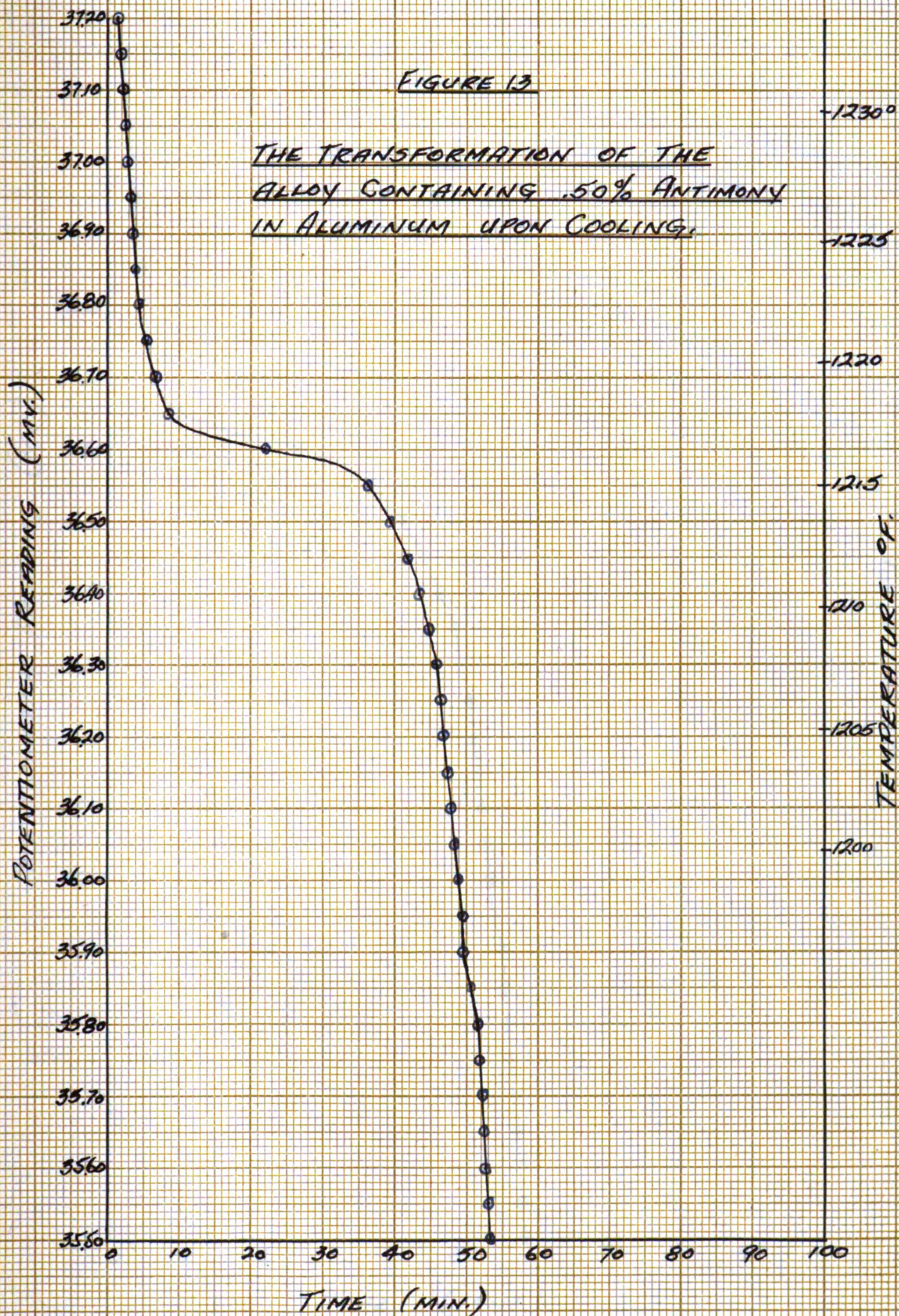
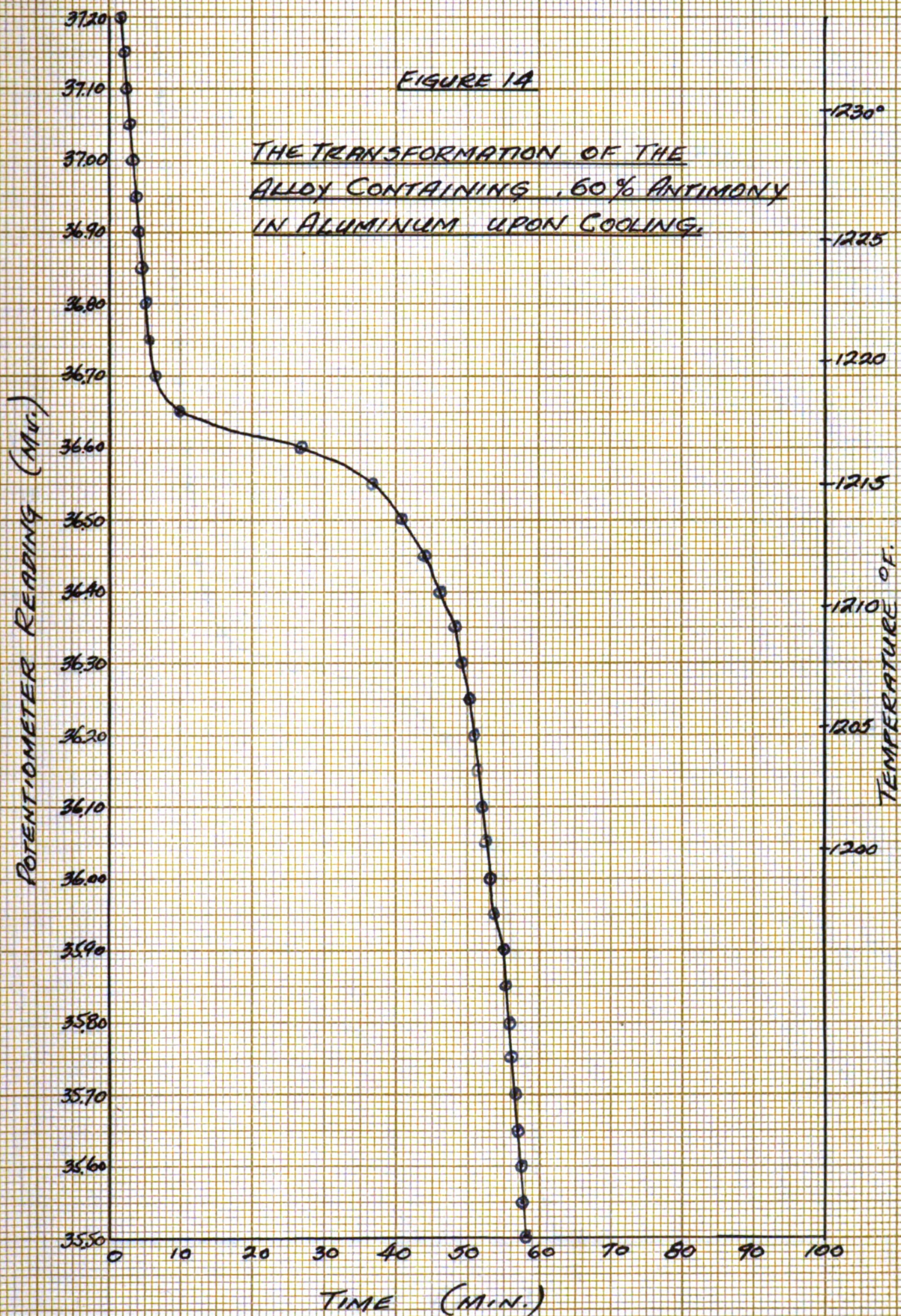


FIGURE 1A

THE TRANSFORMATION OF THE
ALLOY CONTAINING .60% ANTIMONY
IN ALUMINUM UPON COOLING.



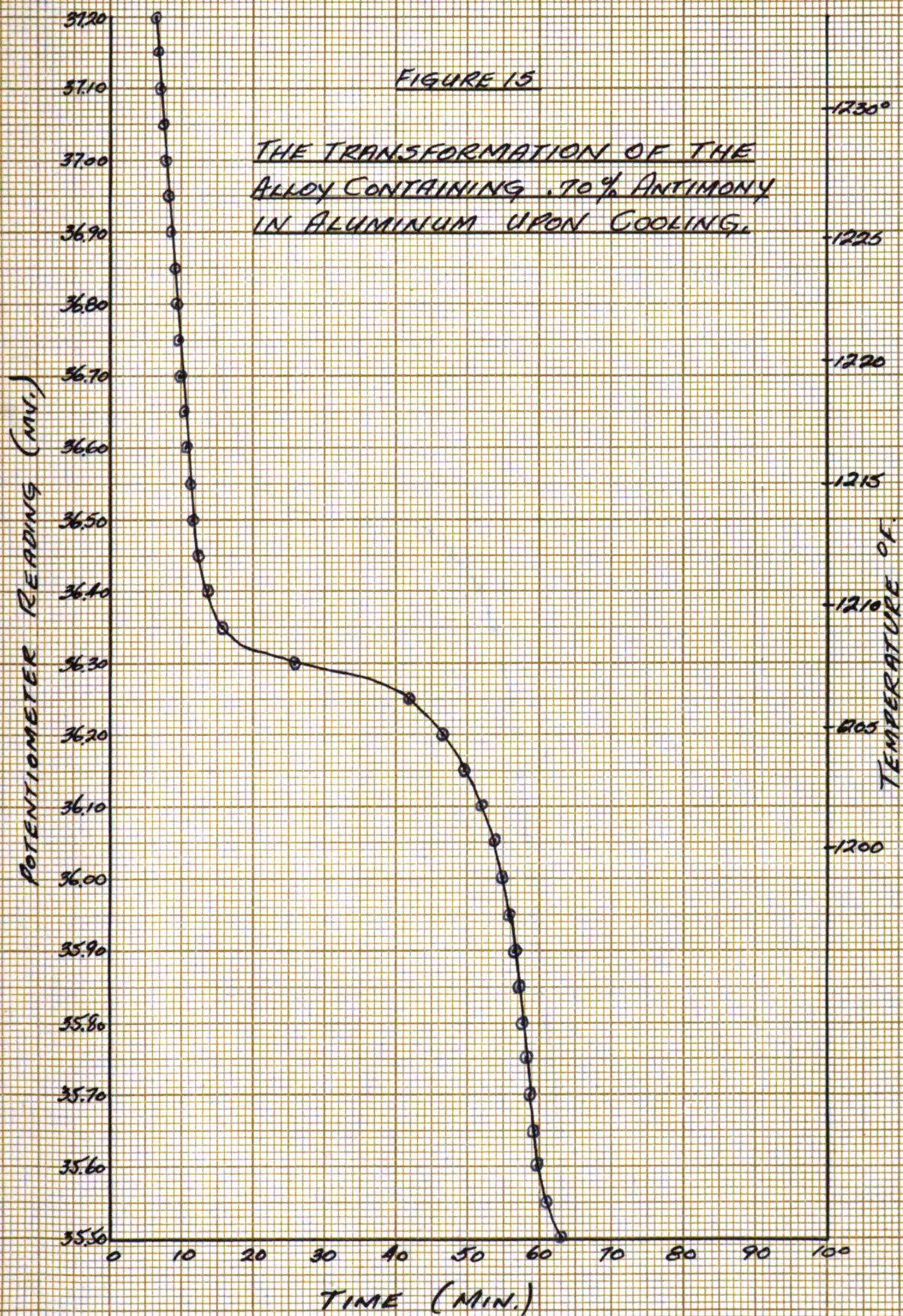
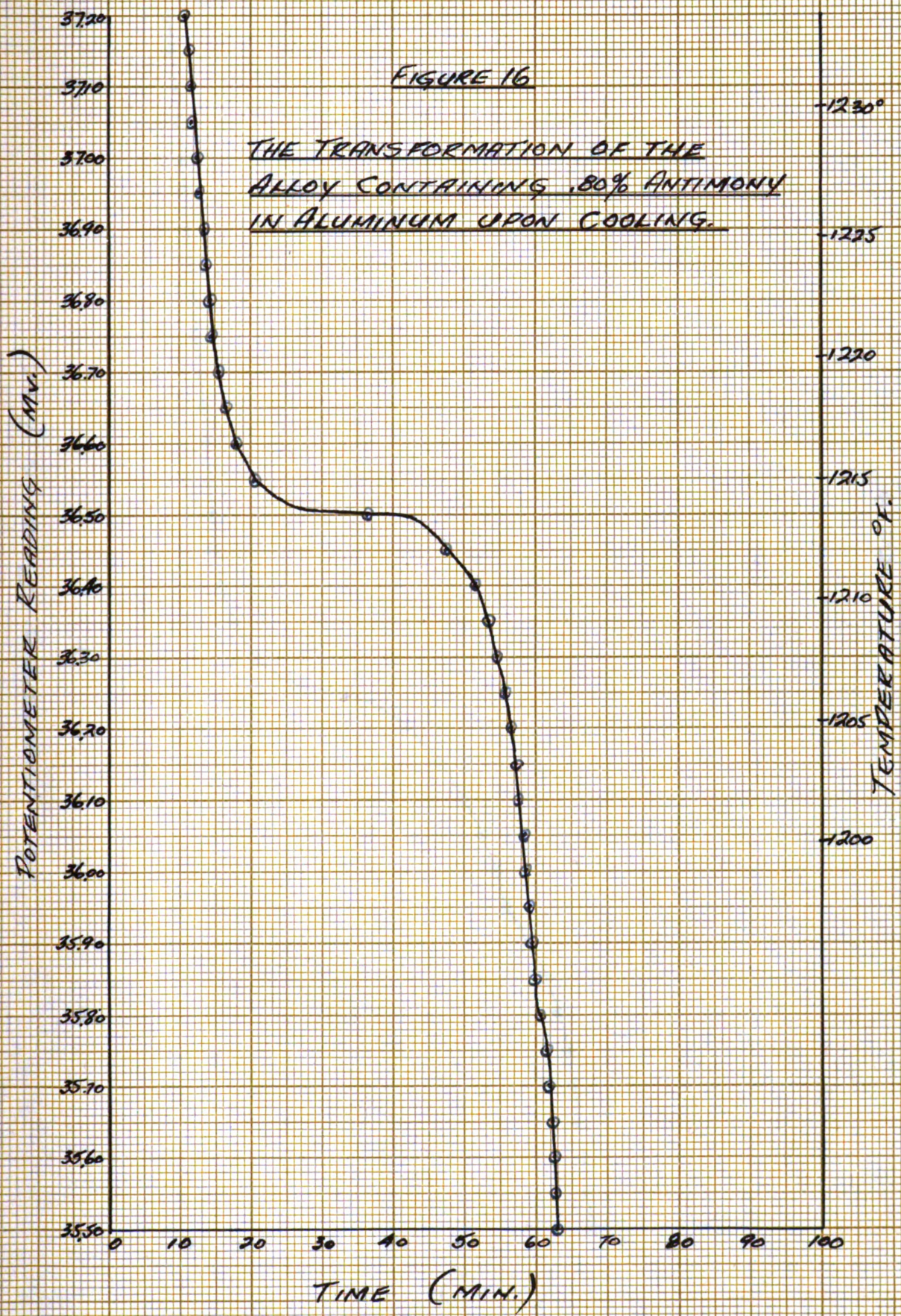


FIGURE 16

THE TRANSFORMATION OF THE
ALLOY CONTAINING .80% ANTIMONY
IN ALUMINUM UPON COOLING.



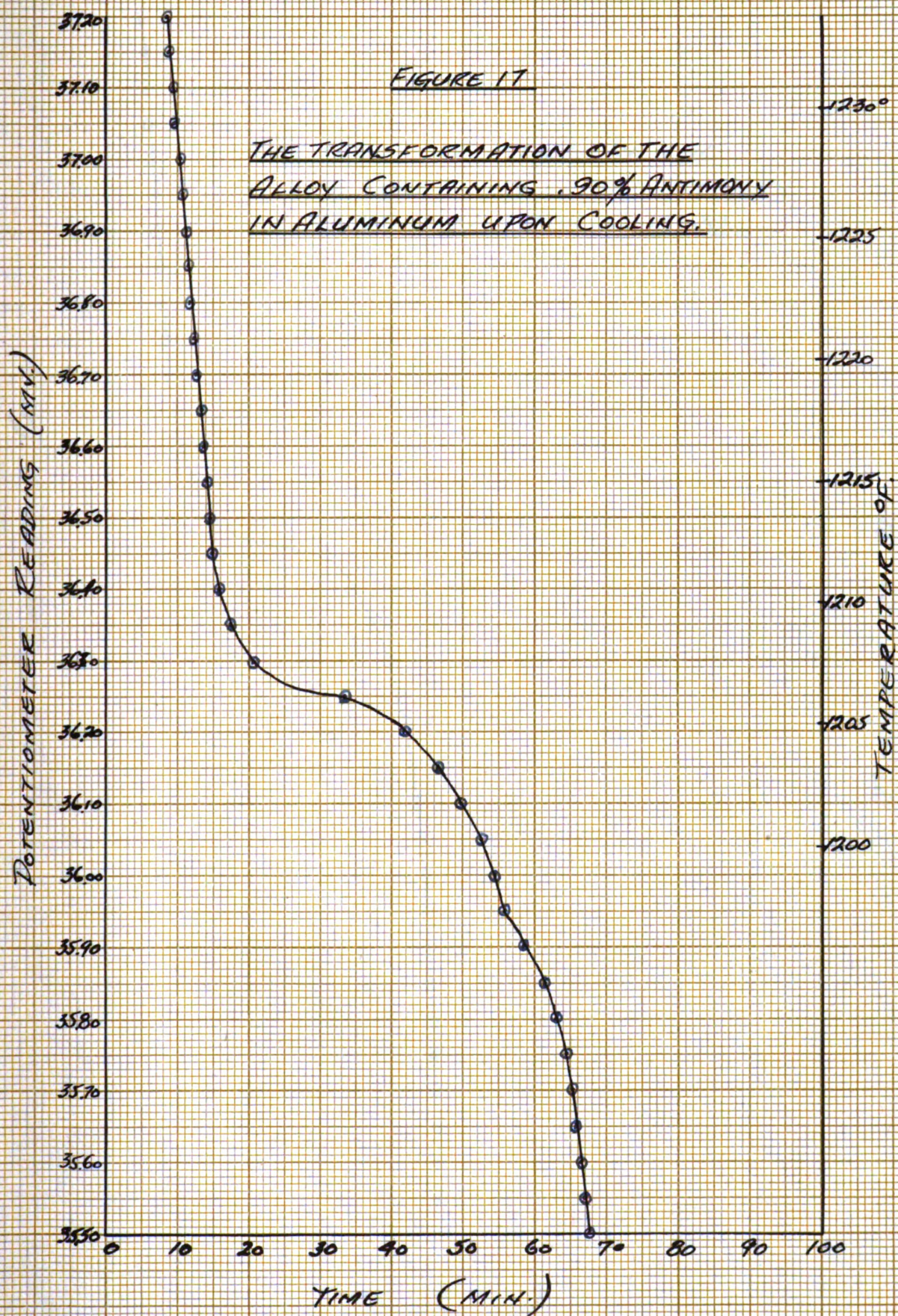
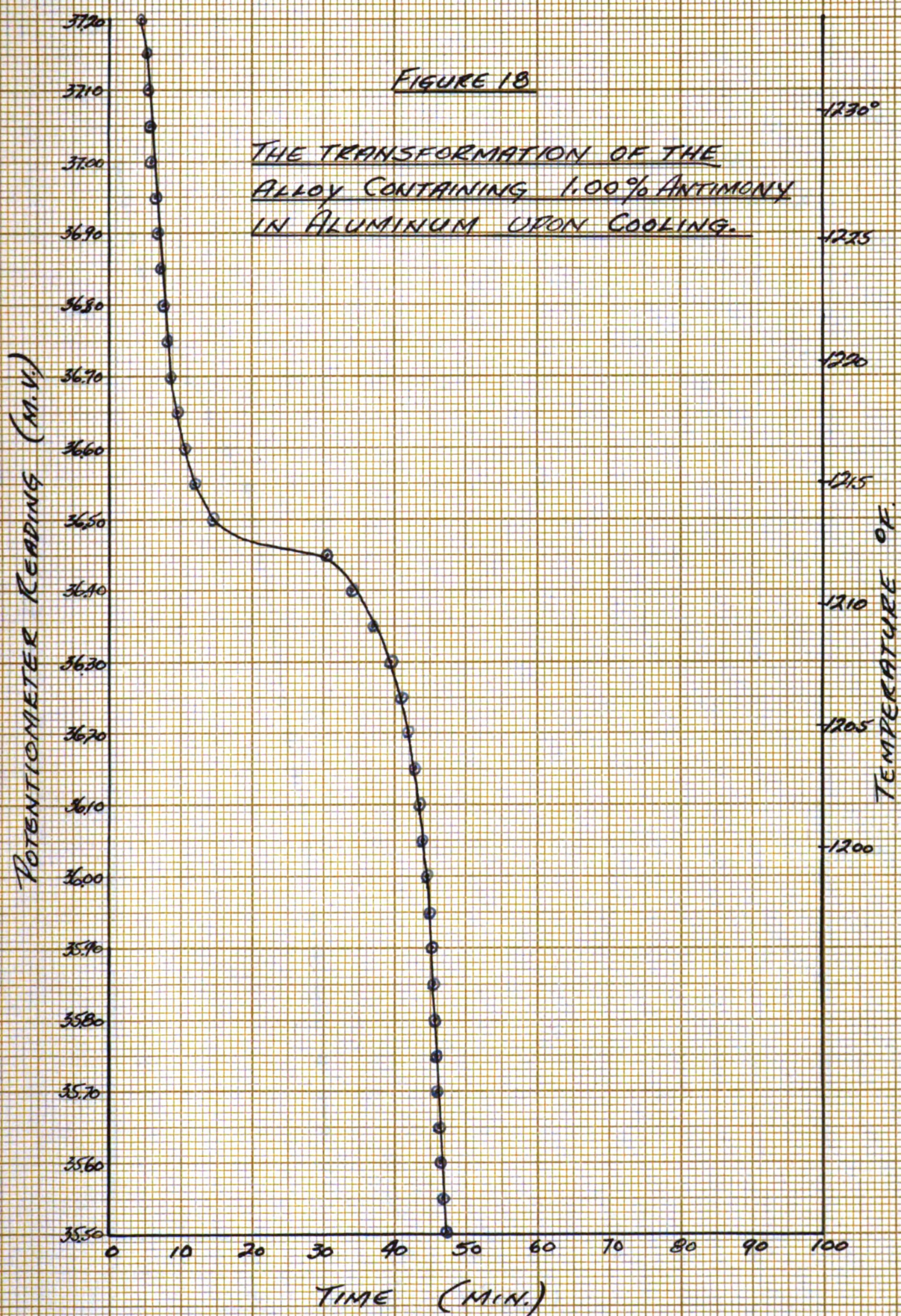
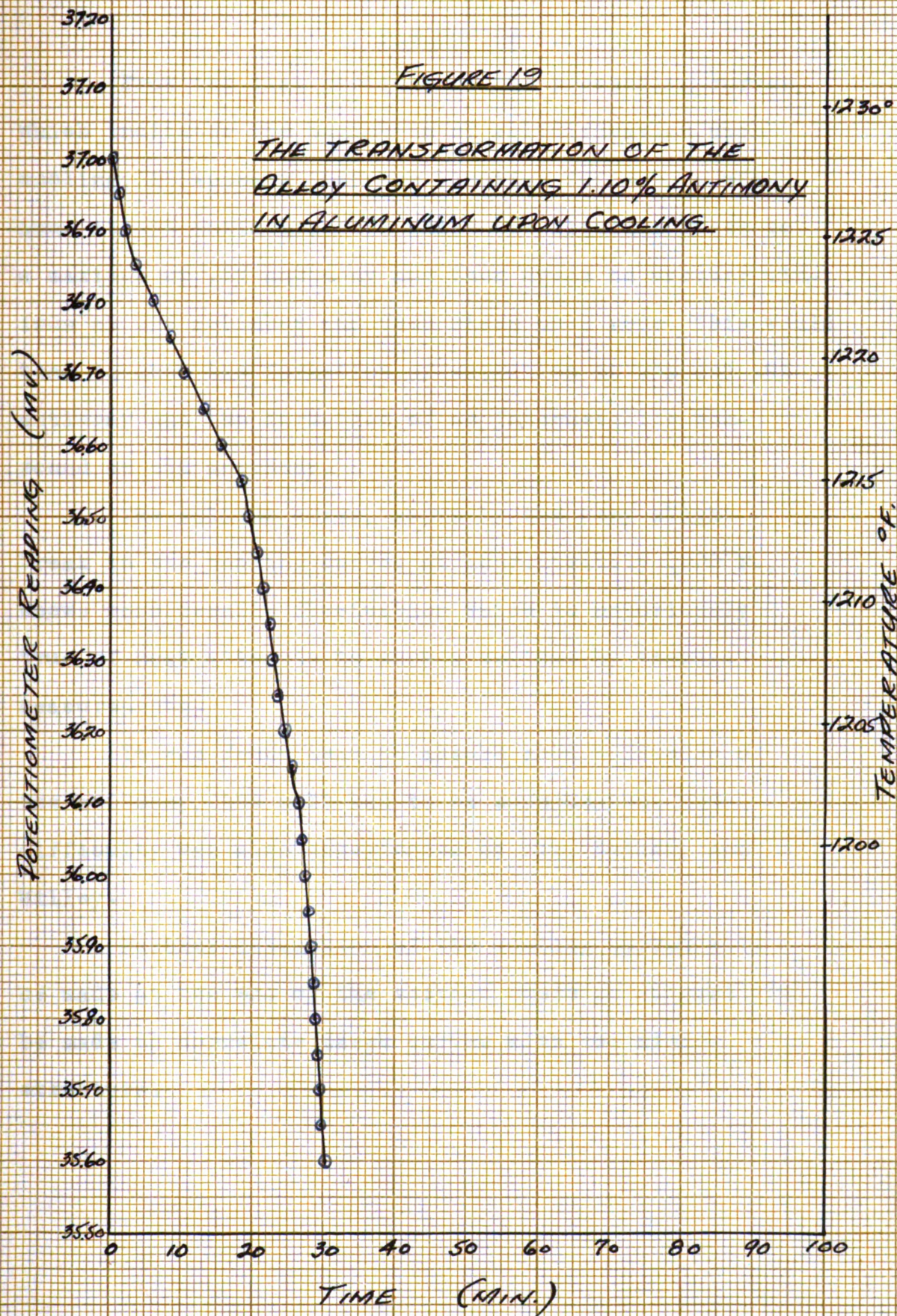


FIGURE 18

THE TRANSFORMATION OF THE
ALLOY CONTAINING 1.00% ANTIMONY
IN ALUMINUM UPON COOLING.





DISCUSSION

From the flat portions of the preceeding curves, which indicates the beginning of solidification upon cooling, it appears that the liquidus falls to a minimum at .3% antimony, from which the liquidus rises to a maximum at .6% antimony and then drops to another minimum at .9% antimony and then rises once more. This would tend to show the formation of eutectics at .3% and .9% antimony and the formation of an intermetallie compound at .6% antimony.

The solidus curve, shown by the very slight break in the cooling curves at approximately 1200°F., shows considerable fluctuation which is notoriously true of most attempts to show a solidus by thermal measurements.

It was found that to assure good diffusion of the antimony, the melt had to be superheated. This difficulty was also experienced by Dix, Keller, and Willey.

Further discussion of the cooling curves will be made at the end of the section where an attempt will be made to correlate these curves with the micro-structure.

VI. THE MICROSTRUCTURE OF THE ALLOYS

GENERAL THEORY

According to Dix, Keller and Willey⁷ the solid solubility of antimony in aluminum appears to be less than 0.10 per cent. Microscopic examination of this alloy showed particles of AlSb constituent out of solution.

These authors found that upon additions of higher percents of antimony, it was found that the eutectic concentration was approximately 1.1 per cent antimony. Although, at concentrations which should produce a eutectic both primary aluminum and primary AlSb occurred in the same field.

Since the metals used in this experiment were not pure, the microstructures were expected to be somewhat complex.

PROCEDURE

Samples were cut from the bottom portion of the air cooled ingots used for the cooling curves. These were filed flat, ground on a belt grinder, and polished successively on #1 paper, #0 paper, 320 wet wheel, 520 wet wheel and levigated alumina wet wheel. All finish polishing was done with re-levigated alumina on billiard cloth. A magnesia paste on a silk wheel was attempted but apparently the base wheel was of the wrong composition since the samples became badly corroded.

The samples were etched in a 10 percent NaOH solution at 160°F. for 5 seconds and then examined before being photographed.

MICROSTRUCTURE OF THE ALLOYS

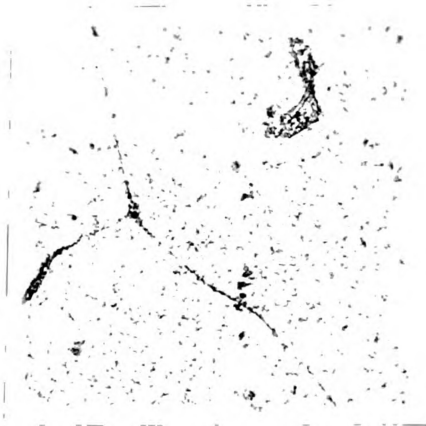


PHOTO #1 - Showing microstructure of aluminum- 0% antimony. Fe_2Al_7 present at upper right, FeSiAl_5 at grain boundary with eutectic Al-Si. 250X

PHOTO #2 - microstructure of .05% antimony alloy showing apparent increase in boundary eutectic. 250X

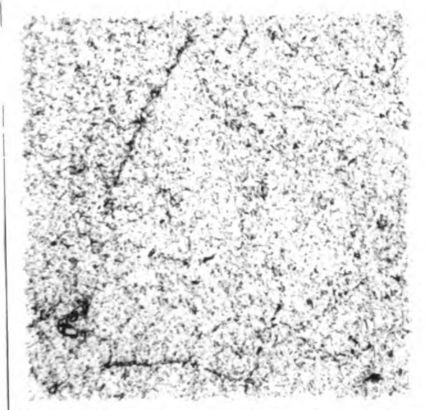
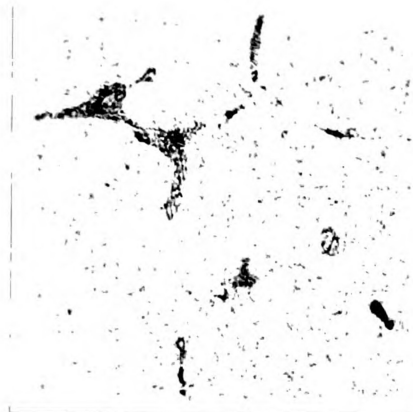


PHOTO #3 - microstructure of .1% antimony alloy showing apparent disappearance of majority of boundary constituents. 250X

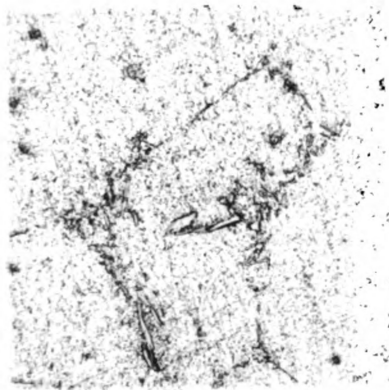


PHOTO #4 - Micro-structure of alloy containing .2% antimony. Two phases present but little eutectic. Predominantly impurity phases. 250X

PHOTO #5 - Micro-structure of alloy containing .3% antimony. 250X

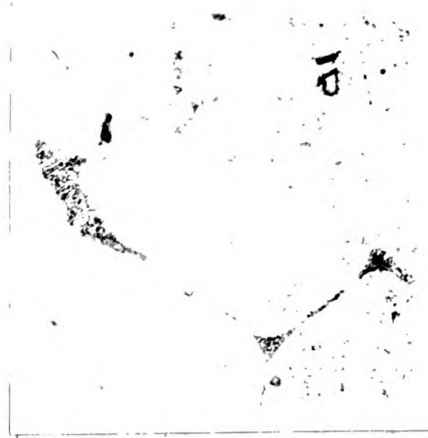


PHOTO #6 - Same as 5. Showing appearance of different phase (dark gray) lower left of photo. 500X

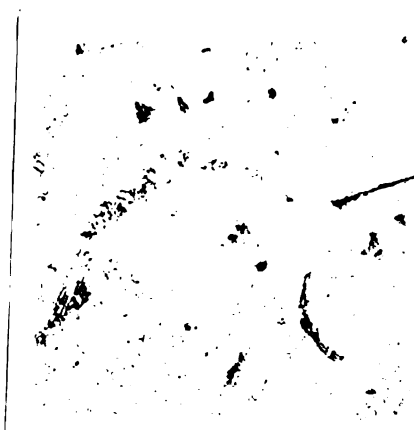


PHOTO #7 - Micro-structure of the alloy containing .4% anti-mony. 250X

PHOTO #8. Same as 7. Showing gray constituent in long angular form at lower right. 500X

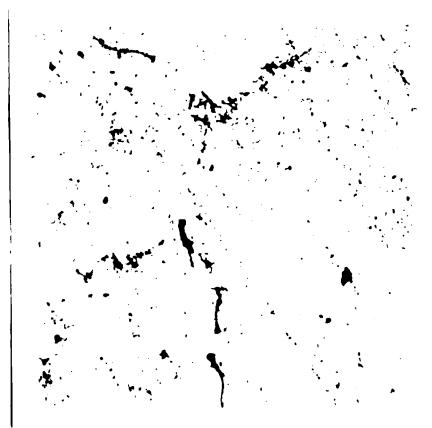
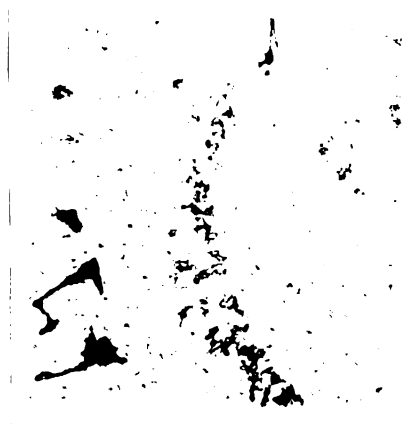


PHOTO #9 - Micro-structure of the alloy containing .5% anti-mony. Little eutectic present. 250X



PHOTO #10. Same as 9. Showing angular shapes of gray constituent, no eutectic. Appearance of black constituent in lower center. 500X

PHOTO #11 - Micro-structure of the alloy containing .6% anti-mony. Grain boundary constituents appear all black. 250X

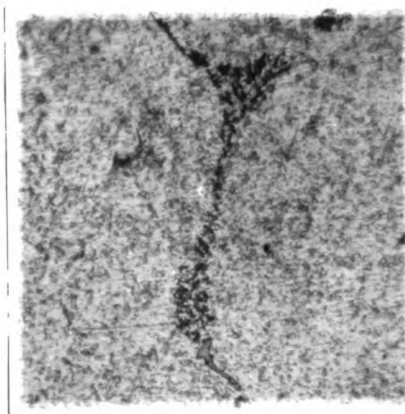
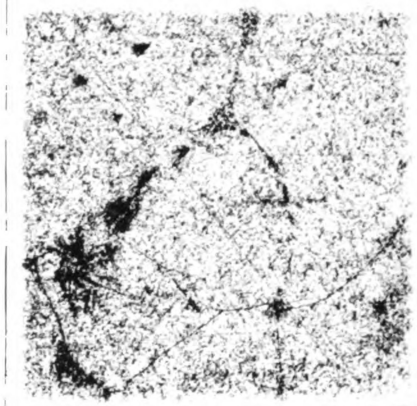


PHOTO #12. Same as 11. Showing a complex shape of black constituent. A particle of the gray constituent present in lower center. 500X

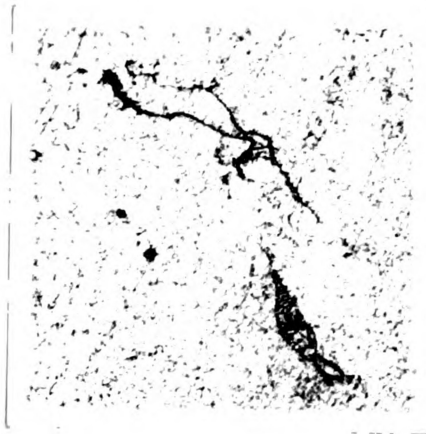


PHOTO #13 - Micro-
structure pf the alloy
containing .7% anti-
mony. Showing appearance
of a small amount of
eutectic. 250X

PHOTO #14 - Same as
13. Eutectic contain-
ing black constit-
uent. Gray constituent
still present. 500X

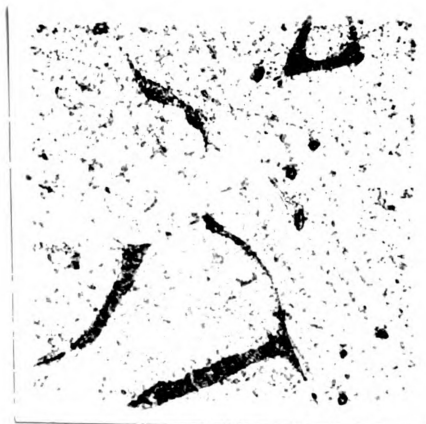
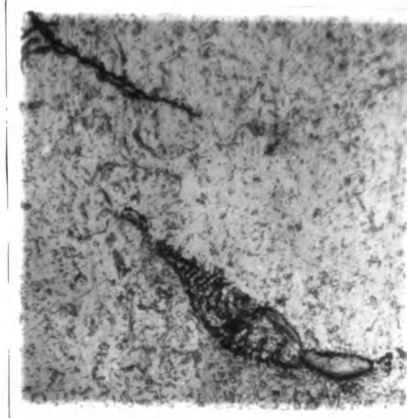


PHOTO #15 - Micro-
structure of the alloy
containing .8% anti-
mony. Predominantly
eutectic. 250X

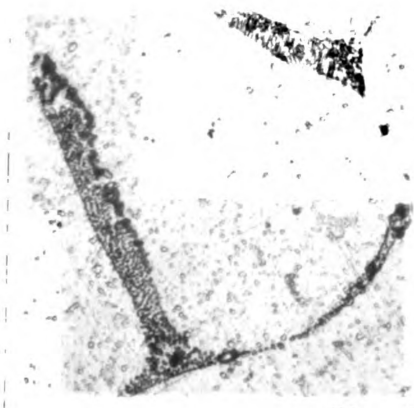


PHOTO #16 - Same as 15. Showing the eutectic. Appears to be slightly spherical and finely divided. Majority formed in grain boundaries. 500X

PHOTO #17 - Microstructure of alloy containing .9% antimony. 250X

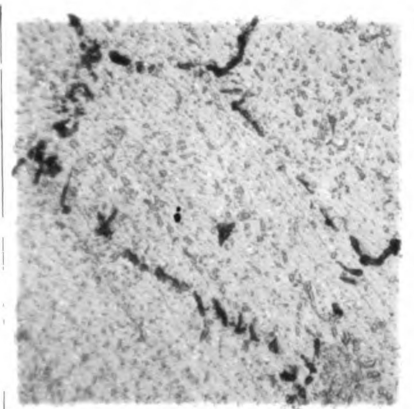
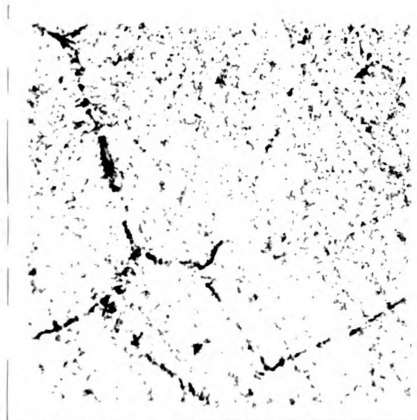


PHOTO #18 - Same as 17. Entirely dark constituent almost completely surrounding grain. No light gray particles apparent. 500X



PHOTO #19 - Micro-structure of alloy containing 1.0% antimony. Little eutectic present. 250X

PHOTO #20 - Same as 19. Showing long stringer-like appearance of a different light gray constituent and large massive black constituent. 500X

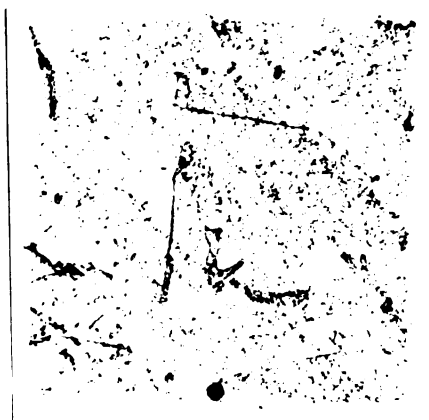


PHOTO #21 - Micro-structure of alloy containing 1.1% antimony. Shows little black constituent. Light gray constituent becoming more agglomerated. Not confined to grain boundary. 250X



PHOTO #22 - Micro-
structure of alloy
containing .9%
antimony. Showing
hard spherical
constituent of matrix
which has not been
identified. Also
shows black constituent
probably AlSb. 2000X

100
100

DISCUSSION

Dix, Keller and Willey⁷ found that a chill-cast alloy containing .1% antimony showed very small particles of AlSb constituent in the microstructure, and after various solution heat treatments, the alloy still showed particles of AlSb constituent out of solution.

The microstructure of the .1% antimony alloy which was air cooled for this investigation showed the boundary constituents largely in solution. Whereas the sample containing .05% antimony showed more grain boundary eutectic and constituents than the alloy containing no antimony.

Dix, Keller and Willey⁷ found that the examination of an alloy containing 1.04% antimony that had been cooled slowly through the freezing range in a hot graphite mold, showed the alloy to be hypoeutectic. Although the alloy did not show a true eutectic structure, a similar alloy of slightly higher concentration, 1.14% antimony showed a few particles of primary constituent, probably AlSb. They stated that it was apparently impossible to produce a uniform eutectic structure, since both primary aluminum and primary aluminum-antimony compound occurred in the same field.

However, the alloy containing .8% antimony used for this investigation shows a eutectic containing the black constituent which was probably AlSb. Undoubtedly, the slower cooling rate through the solidification

of this alloy aided in producing the eutectic. Dix, Keller, and Willey used a cooling rate approximately 6° per minute, whereas, for this investigation the melt containing .8% antimony was furnace cooled at a rate such that the transformation from liquid to solid took place in 17 minutes. Of course, the rate of cooling in a graphite mold could hardly be approximated with the facts which are known.

At .1% and 1.1% antimony, another constituent appears which had not been in the alloys of lower concentration. Also at 1.1% antimony, the black constituent begins to disappear.

Difficulty was experienced in assuring solution of the antimony upon melting. This difficulty was also experienced by others working with this alloy. Superheating to 1500° F. appeared to overcome this hazard. However, lower temperatures might possibly be used to obtain comparable results.

It is possible that the reactions shown to appear at .3%, .6% and .9% are the result of reactions of antimony with the impurities and aluminum rather than the antimony itself. Consequently, no effort was made to identify the constituents which appeared.

VII. CHEMICAL ANALYSIS

INTRODUCTION

It was thought, since antimony has a low vapor pressure, that some of the antimony might have been lost despite the precautions taken in melting.

Therefore, to determine whether there was any mechanical loss, spot analyses were run on two of the lower percentage alloys, one medium percentage alloy and one higher percentage alloy.

Since the impurities included iron, silicon, copper, magnesium, zinc, nickel and manganese, the hydrogen sulfide precipitation method was chosen .

Antimony sulfide is soluble in a NaOH-Na₂S mixture, whereas copper is not and this method of separation was used. All the other elements are separated by hydrogen sulfide precipitation from an acid solution .

PROCEDURE

Drillings were taken from test ingots containing .05%, .1%, .5%, and 1.1% antimony. The test ingots referred to were those used in running the cooling curves for the equilibrium diagram investigation. The sample was drilled from the cross section of the ingot exclusive of 1/8" on each side.

The method of analysis was as follows: A 2 gram sample of the alloy was attacked with 40 ml. of hydrochloric-nitric acid mixture, diluted to 150 ml. and concentrated until pasty to remove oxides of nitrogen and excess acid.

Since copper interfered with the method used,⁸ the copper was then removed. The acid mixture was treated with 3 to 5 grams of tartaric acid, then poured into the following mixture: 150 ml. of a mix of 60 grams of sodium sulfide with 40 grams of sodium hydroxide dissolved in 1000 ml., diluted to 300 ml. The mixture was warmed and the insoluble sulfides (copper and lead) allowed to settle out. Then the solution was filtered free of the precipitate and the latter washed.

The solution was then acidified with hydrochloric acid and a rapid current of hydrogen sulfide passed into the solution for 20 to 25 minutes. The solution was warmed and filtered on No. 541 Whatman paper, washing with acidified H₂S water.

The residue was extracted with hydrochloric

acid containing a little potassium chlorate. The solution was heated to boiling and .5 gram of potassium chlorate was added. The solution was boiled until colorless and another .5 gram of potassium chlorate added and the solution again boiled to very low bulk to remove excess chlorine and its oxides. The solution was diluted to 50 ml. and evaporated to low bulk once again.

Then 20 ml. of hydrochloric acid was added and the solution transferred to a 500 ml. conical flask and diluted to 200 ml. The solution was then cooled to room temperature and 4 grams of potassium iodide was added and the solution titrated with sodium thiosulfate solution, using fresh starch solution as indicated ⁹.

The sodium thiosulfate solution was standardized against electrolytic copper. One gram of copper = .9576 grams of antimony.

TABULATED DATATABLE XXI RESULTS OF CHEMICAL ANALYSIS

<u>%Antimony</u> <u>Added by</u> <u>weight</u>	<u>%Antimony</u> <u>by Analysis</u>
.05	.051
.10	.102
.50	.511
1.10	1.122

DISCUSSION

From the results obtained, antimony appears to be of slightly higher concentration than the amounts added to make up the heat. This amount, however, is approximately only 2%.

This loss of aluminum was probably due to the oxides of aluminum which were slagged off.

These results would tend to show that the loss of antimony was nil.

An attempt was made to separate copper by the use of cupferron. However, it was found to be very difficult to remove from the filtrate after filtering the copper salt out of the solution. Also, it was found that results seemed to be low. This was probably due to the fact that some of the antimony was occluded upon the surface of the copper salt.

VIII. PROPOSED EQUILIBRIUM DIAGRAM

From the small error in composition shown by the chemical analysis, no correction was made to the diagram resulting from the cooling curves.

Dix, Keller and Willey⁷ showed in their results that antimony was soluble in aluminum up to 0.1%. They also showed that a eutectic was formed at 1.1% antimony with a eutectic temperature at 657°C as shown in Figure 20. The aluminum and antimony used for their investigation was very pure and the aluminum had less than .03% impurities.

The diagram produced by this investigation (Figure 21) shows a minimum in the curve at .3% and .9% antimony with a maximum at .6% antimony. No attempt was made to show a definite solidus since any attempt to show the solidus curve by thermal measurements is notoriously inaccurate. The impurities of the aluminum used in this investigation were less pure than that used by Dix, Keller, and Willey. The impurities in the ingot used amounted to .292%. It is believed that the impurities had a definite effect upon the shape and form of the equilibrium diagram.

The eutectic was shifted to the left and the eutectic temperature was apparently lowered. There also was the formation of an intermetallic compound apparent at .6-.7% antimony. This was also evidenced by a greater tendency to cause piping upon cooling from the molten condition.

The sample containing .9% antimony was studied by microscope in an effort to determine whether the hard particle showing in the matrix of Photo #22 was caused by any technique in polishing. The sample was originally polished with alumina. The sample was repolished using magnesia, etched with 10% NaOH and the particles were still found to be present at 500X. The same sample was then repolished with Adolphe Beuhler #1563, chrome green compound, washed with soap and water, etched with 10% NaOH, swabbed with hot water and dried with C.P. ethel alcohol. The constituent was still present at 500X and at 2500X. However, the constituent was not identified.

The low points in the curve, found at .1% and .7% antimony were disregarded, since neither was borne out by the microstructure.

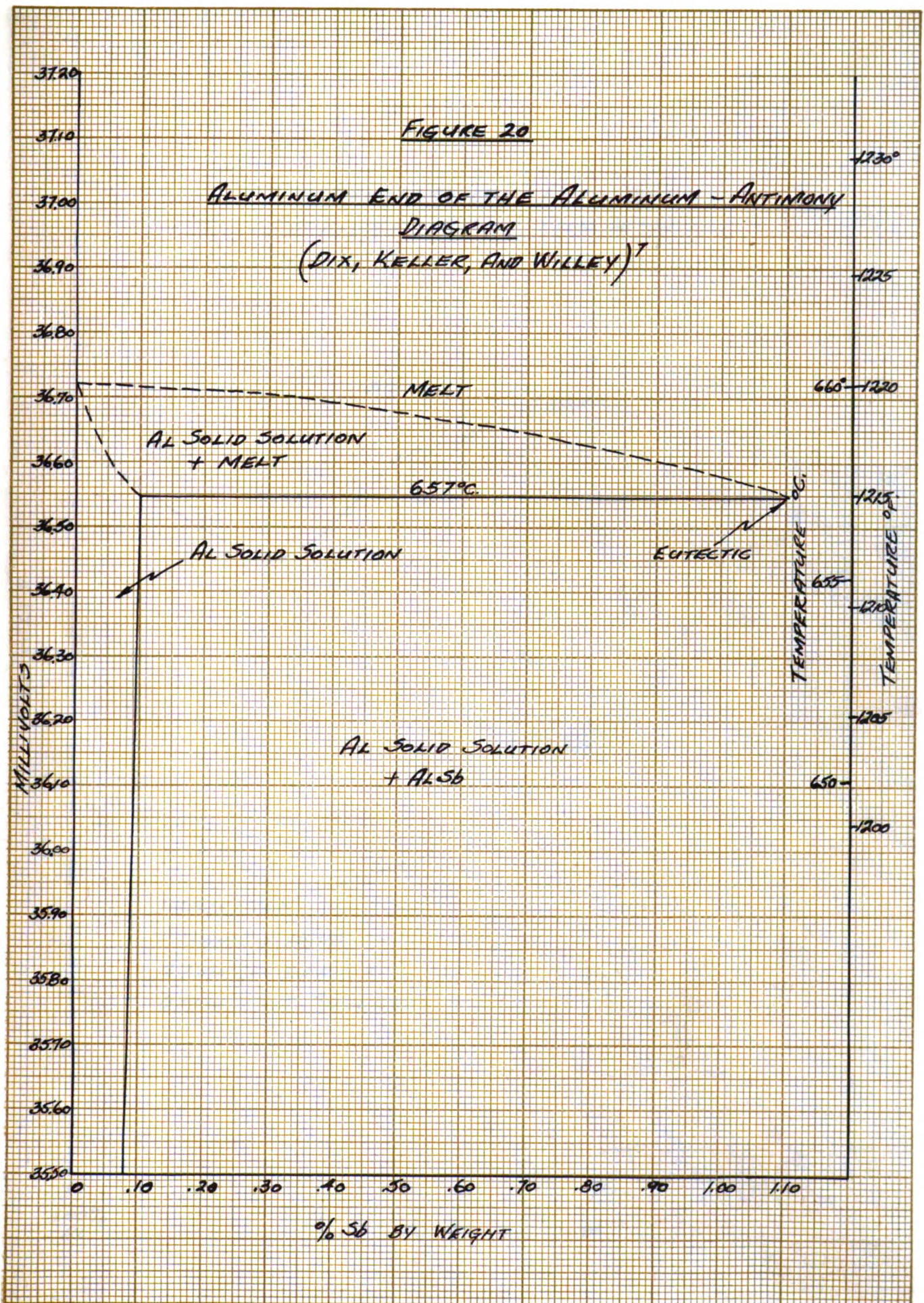


FIGURE 21

THE EQUILIBRIUM DIAGRAM
RECEIVED FROM THE DATA
OF THE COOLING CURVES.

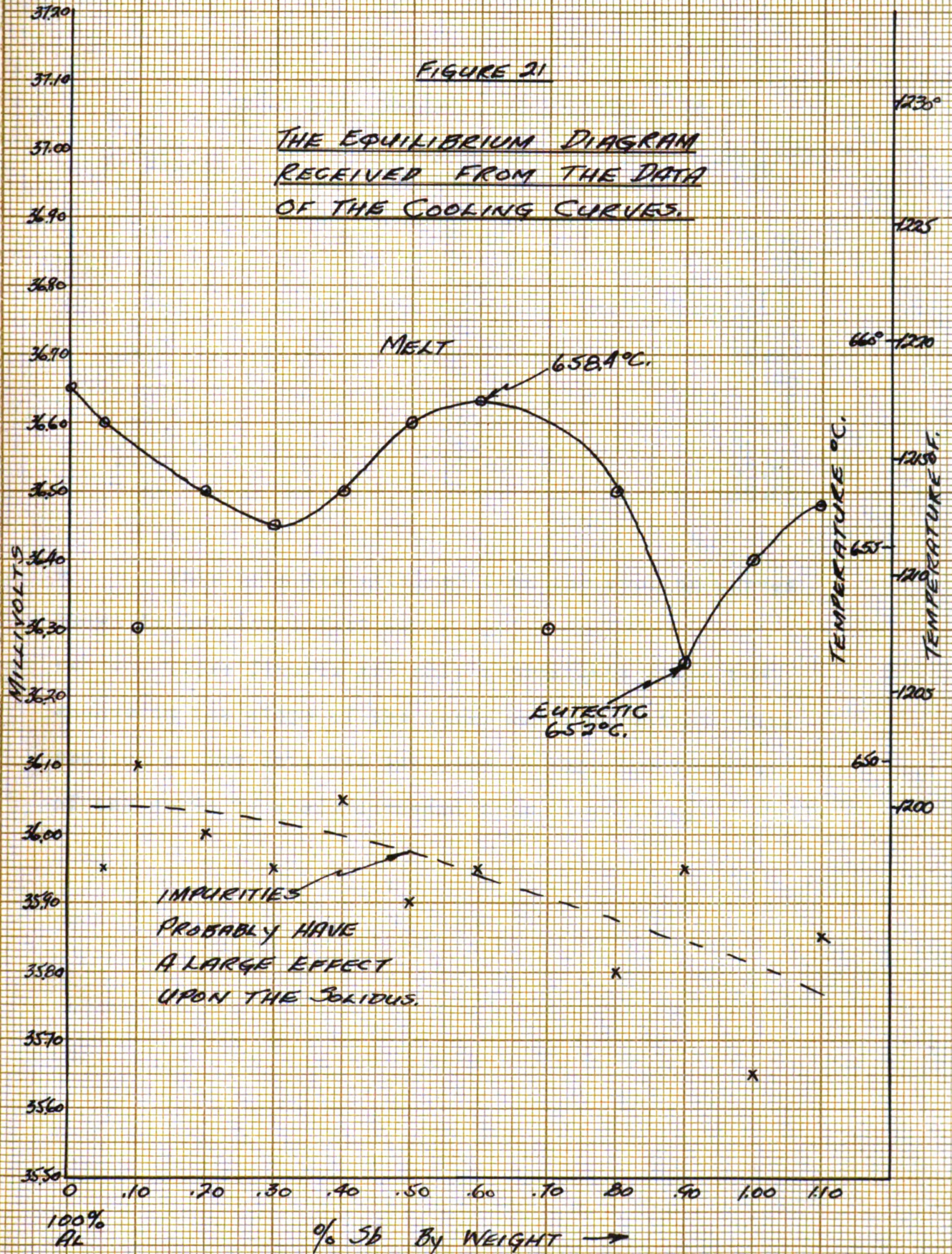


TABLE XIX EQUILIBRIUM DIAGRAM DATA **

<u>% Sb</u> By Weight	<u>Liquidus</u> (Mv.)	<u>Solidus</u> ** (Mv.)
0	36.65	
.05	36.60	35.95
.10	36.30	36.10
.20	36.50	36.00
.30	36.45	35.95
.40	36.50	36.05
.50	36.60	35.90
.60	36.63	35.95
.70	36.30	35.55
.80	36.50	35.80
.90	36.25	36.00
1.00	36.40	35.65
1.10	36.48	35.85

* This data was obtained from the cooling curves shown in Section I Part I.

** These figures are only approximate since the change in the slope of the curve was very slight. This section of the data was included to show the tendency of the curve to slope downward from 0% antimony.

IX. AN EXAMINATION OF THE PHYSICAL PROPERTIESINTRODUCTION

Annealed aluminum sheet of high purity has a tensile strength of 8,500 p.s.i. with an elongation of 60 per cent in 2 inches and exhibits a hardness of 16 Brinell. Of course, aluminum of purity such as was used in this investigation, and in the "as cast" condition, probably would have less strength and less elongation.

This investigation was not for the purpose of showing the effects of antimony upon all the physical properties of aluminum, but merely, to indicate the tendencies of any of the major properties to show a change. The properties investigated include tensile strength, elongation, reduction of area, and hardness.

MATERIALS AND APPARATUS

The melting was done in a Hoskins Electric Furnace of Type FHIO4 with a capacity of 15 volts and 125 amperes. The furnace was connected to a Kuhlman Transformer, 2K.W., 60 cycle, with a 110-120 volt primary and 17 volt secondary and to a variable resistance. (Figure 23)

Slugs were cut from aluminum ingot #5 (Table I) for the melt and the antimony was of the commercial type for technical use, as was used in the investigation of the equilibrium diagram.

Temperatures were measured with a Chromel-Alumel thermocouple of 16 B & S gauge wire and an L. & N. direct reading potentiometer with manual reference junction compensator.

The heats were melted in graphite crucibles and poured into permanent molds made of $\frac{3}{4}$ inch steel pipe which were split and wired together. (Figure 24) The bottom was a plug of alundum cement baked in at a temperature of 1400-1500°F. The mold had a small pouring basin of alundum which would be easily crushed if the longitudinal contraction was great enough. The bottom 2 inches of the mold were packed in a mixture of crushed refractory and asbestos as a precaution in case the mold should "bust out."

PROCEDURE

The aluminum slugs were weighed and a corresponding amount of antimony weighed out to make the necessary concentration. The aluminum was then placed in a graphite crucible and melted. The molten aluminum was poured into another graphite crucible containing the antimony and this second crucible returned to the furnace and the mixture superheated to 1500°F. (with the exception of one of two samples containing 1.0% antimony which was heated to 1300°F.)

The molten metal was then poured into the mold at a rate such that the basin was full at all times and the basin was left full after the mold was filled to allow some metal for contraction, in an effort to prevent the formation of pipes if possible. All the samples were poured into the same type of mold.

After being allowed to cool in the mold, one of the two bars of the .05% antimony composition was heated to 1200°F. and quenched in water.

The bars were then machined to the specifications shown in Figure 25.

The diameter of the bars was measured and a 2" gauge length punched on the bars. The bars were broken in a small hand operated Dillon tensile testing machine using serrated chucks. The bars were then put together and held in place in a vise while the elongation was measured. This was done by means of a pair of dividers

and a set of outside calipers accurate to $\pm .001$ inches. The diameter after rupture was measured by micrometer on one of the broken sections. Then approximately $\frac{3}{4}$ inch was ground from the portion of the tensile bar used in the chuck and a Brinell impression taken using the 500 kg. load.

TABLE XX PHYSICAL TESTING DATA

<u>Composition</u> <u>% Antimony</u>	<u>Tensile</u> <u>Strength</u>	<u>Elongation</u>	<u>Reduction</u> <u>of Area</u>	<u>Brinell</u> <u>Hardness</u>
0	5,620	*	*	24.9
.05	8,690	35.5	21.9	29.7
↓ .05	3,160	*	*	32.6
.10	7,900	30.9	26.1	28.4
.20	9,130	50.6	49.5	29.7
.30	8,800	*	*	29.7
.40	8,340	30.5	18.5	23.8
.50	8,490	35.3	29.6	21.8
.60	9,100	40.1	46.0	31.2
.70	9,710	50.9	64.5	31.2
.80	**	**	**	**
.90	9,500	55.9	64.8	29.7
1.00	9,170	39.3	31.9	29.7
↓↓ 1.00	11,140	51.9	56.5	29.7
1.10	7,150	27.4	18.8	24.9

* Broken outside 2" gauge marks.

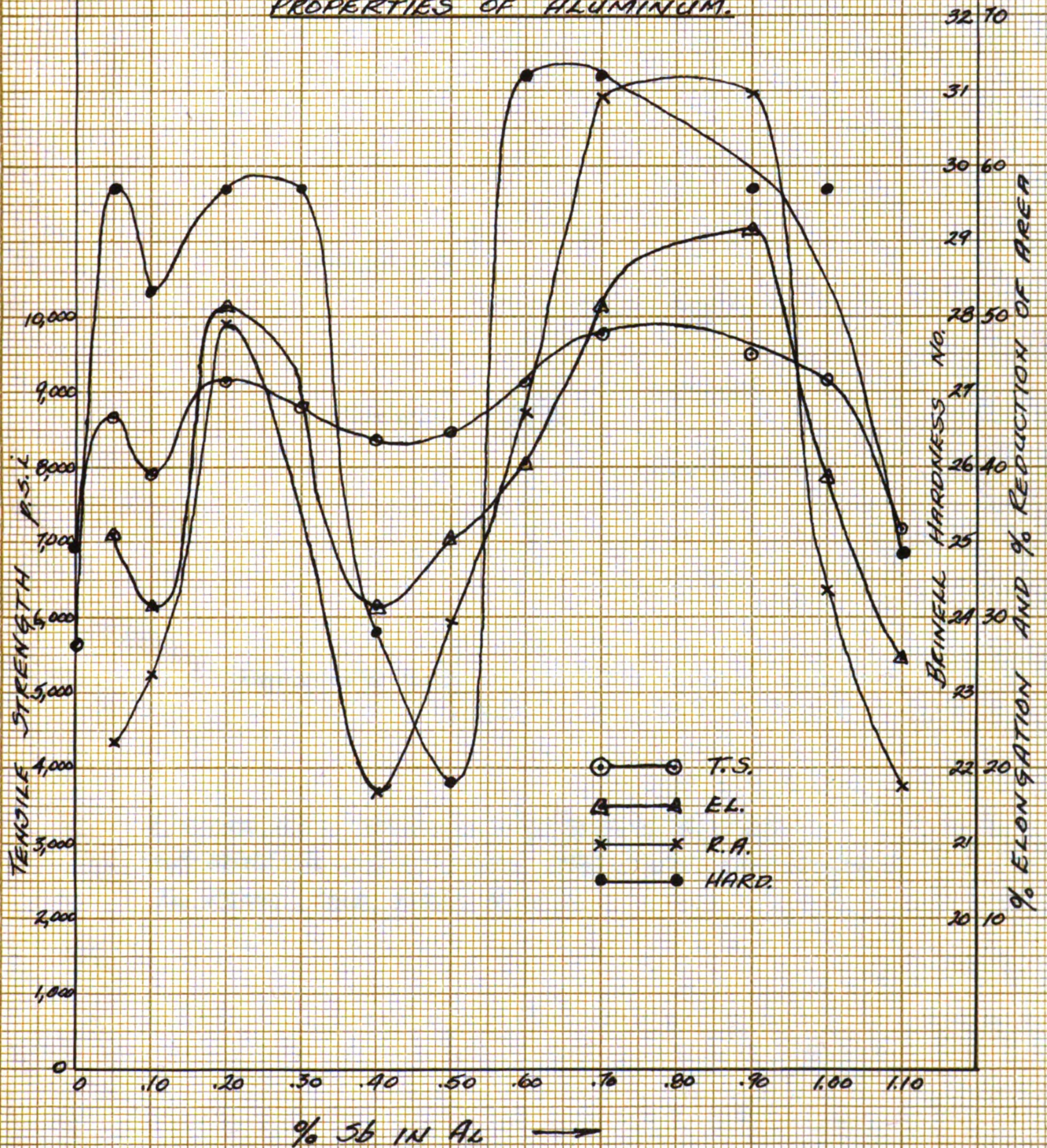
↓ Quenched from 1200°F. Aged at room temperature
for 15 days 2½ hours.

** Broke while machining.

↓↓ Heated to 1300°F. before pouring rather than
1500°F.

FIGURE 22

THE EFFECT OF SMALL ADDITIONS OF
ANTIMONY UPON THE PHYSICAL
PROPERTIES OF ALUMINUM.



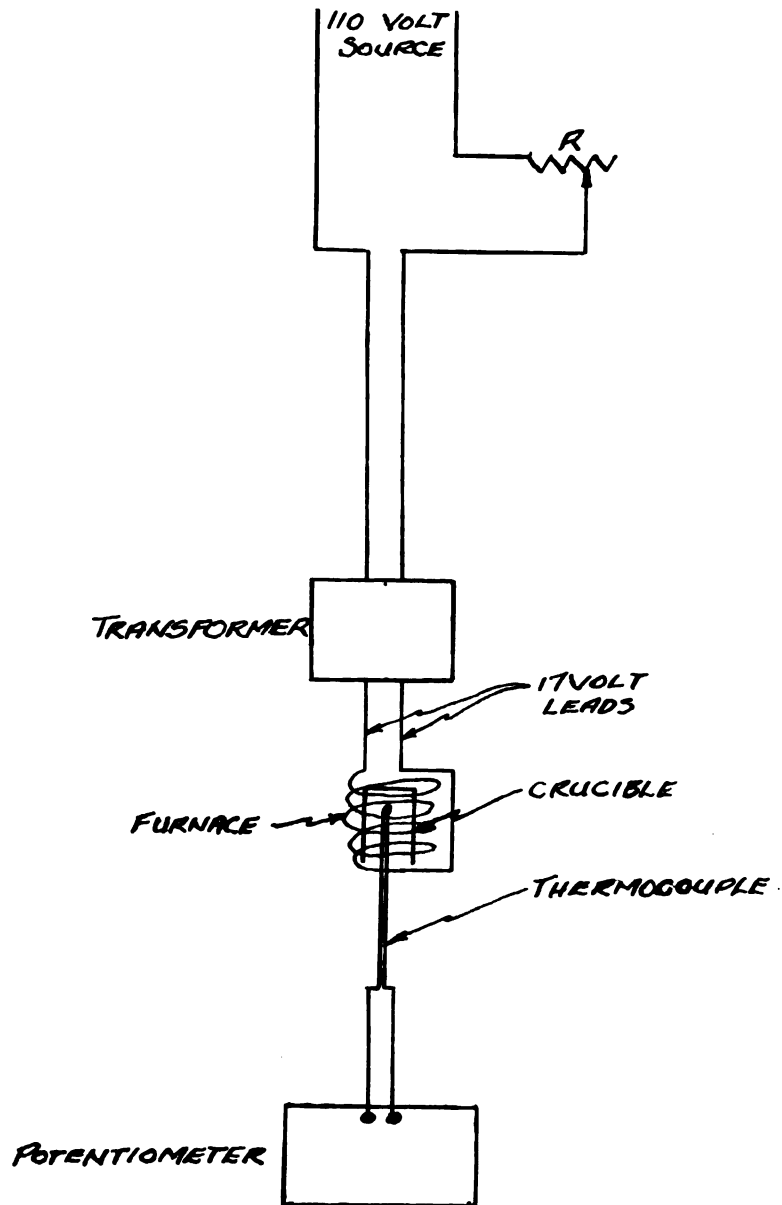


FIGURE 23 WIRING DIAGRAM OF THE
APPARATUS USED FOR MELTING THE METAL
TO POUR THE TEST BARS.

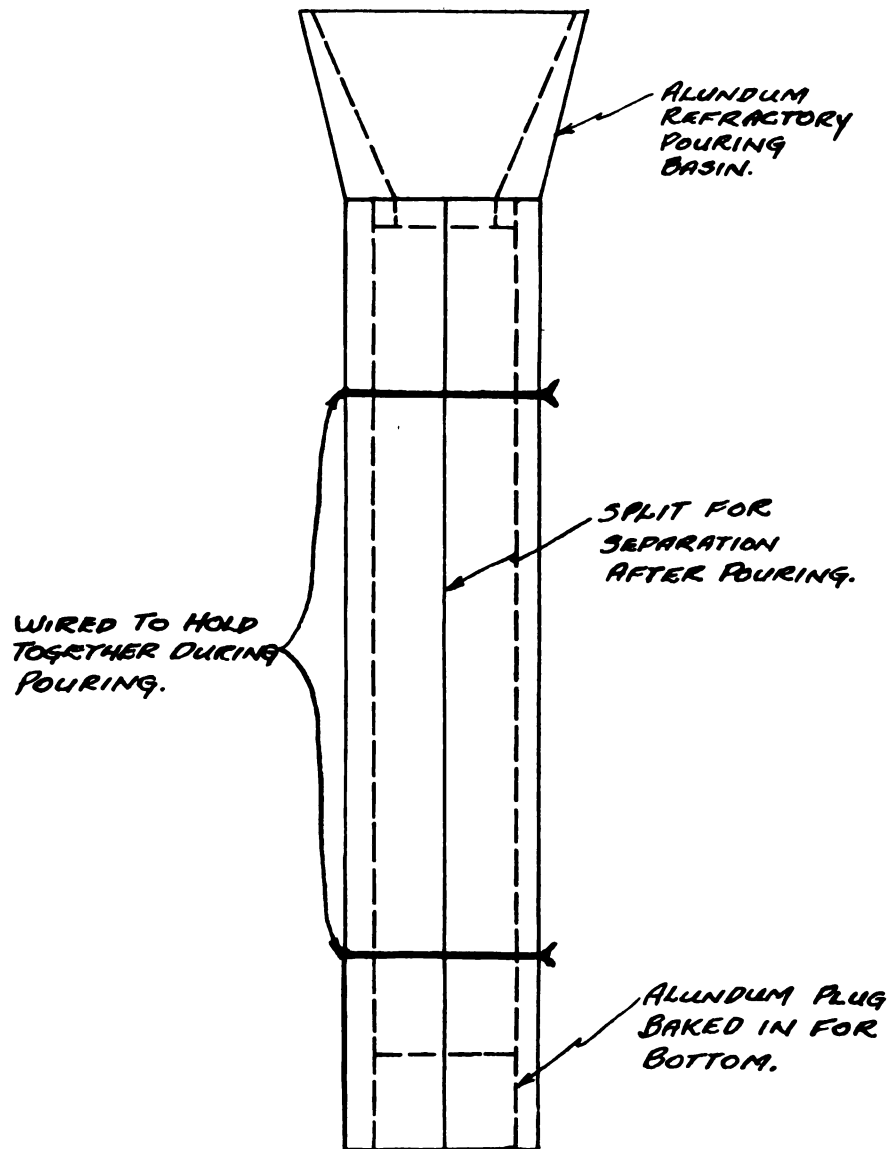


FIGURE 24 PERMANENT MOLD FOR TENSILE BARS.

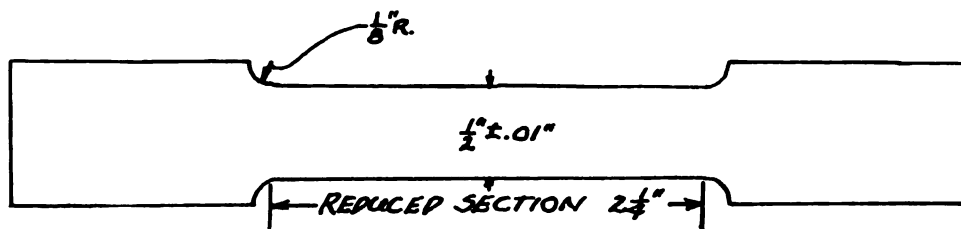


FIGURE 25 TENSILE BAR SPECIFICATIONS.

DISCUSSION

All the properties apparently follow the same general trend, i.e., where one has a minimum in the curve of properties vs. composition, the others do also.

At .1% antimony, the point at which all the boundary eutectics and compounds appear to be in solution, a minimum occurs in all the properties investigated. The greatest drops in properties occur at about .4% and 1.1% antimony. At .4% antimony the dark gray constituent has appeared in long angular form, and the equilibrium diagram shows the liquidus rising from the point at .3% antimony. At 1.1% antimony the light gray constituent appears in a long angular shape which is not confined to the grain boundaries. Apparently these two constituents are embrittling in their nature.

The properties appear to be at their maximum at .05%, .2%-.3%, and .6%-.9% antimony. At .05% antimony the boundary eutectic has increased slightly. At .2%-.3% antimony the dip occurs in the liquidus curve which may or may not be a eutectic. The microstructures show eutectic, but this may be predominately impurity phases. At .6%-.9% antimony the microstructure is predominately of the black phase which progresses toward a eutectic formation almost entirely occurring at the grain boundaries. Apparently this constituent is strengthening in its nature.

The tensile bar containing .05% antimony, which was quenched into water from 1200°F. and aged at room temperature

for 15 days and $2\frac{1}{2}$ hours, had very low tensile properties. Apparently this alloy is heat treatable, but the mechanism of aging proceeds rather slowly at room temperature and has a detrimental effect upon the tensile strength.

However, the tensile bar containing 1.0% antimony, which was poured at 1300°F. rather than 1500°F. , had very good properties and even better properties than the bar of the same composition which was poured from 1500°F. Apparently, the grain size was much smaller - which might be inferred from the appearance of the tensile bars after rupture. The bar poured from 1500°F. had a very pronounced ripple to its surface, while the bar poured from 1300°F. had a relatively smooth surface.

Three of the bars tested had imperfections, none of which caused failure. Apparently, this alloy is notch insensitive.

X. CONCLUSIONS

Antimony below 1% has very noticeable effects upon the physical properties in very definite ranges of composition, as shown by Figure 22. Therefore, any effort to strengthen aluminum with small amounts of antimony should be closely controlled.

The effects of small additions of antimony to aluminum are not entirely detrimental. Additions of .6% to .9% antimony give the maximum properties. Within this range of composition the tensile strength, ductility and hardness are all at a maximum.

The alloys appear to be notch insensitive and the tensile bars continued to show strength after they had split and cracked on their external surface.

Care must be exercised to assure complete diffusion or antimony may separate out in the melt. This was overcome by heating to 1500°F. However, heating to 1300°F. gives much better tensile properties.

The equilibrium diagram shows limited solubility below 0.1% antimony with the formation of at least one eutectic. Apparently, there is some change at 0.1% antimony, as shown by the cooling curves and the graph of properties vs. composition. However, the metallographic examination failed to corroborate this fact.

When viewing these facts, it would be well to bear in mind that all the changes in temperature and strength which are pointed out in this investigation

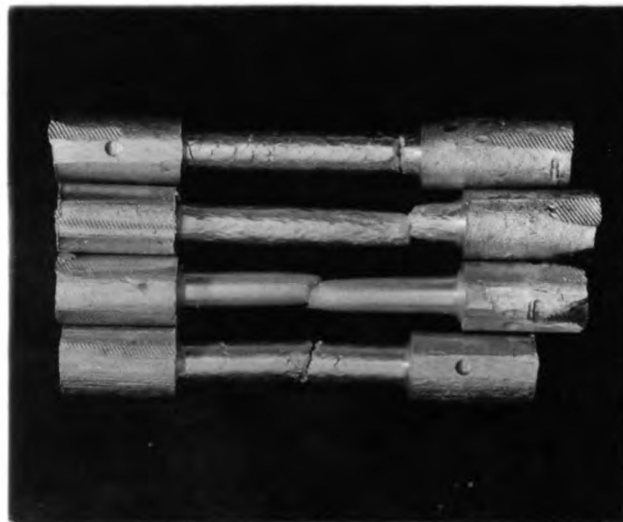
are rather small changes.

Further investigation undoubtedly should be carried out on these alloys. A few suggestions might be: additions to commercial alloys, condition after heat treatment, and other physical properties such as impact strength, fatigue strength, electrical resistance, effects on thermal expansion, and machineability. The equilibrium diagram in the region of 0.1% antimony also should be investigated further.



PHOTO#23

Top view of a sprue showing a particle of antimony which was forced there during solidification.



PHOTO# 24

A view of tensile bars containing (top to bottom) .4%, .7%, 1% (heated to 1300°F.) and 1.10% antimony, showing the surface variation after rupture and the relative elongation.

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