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UNCLASSIFIED

Quarterly Progress Report No. 5
On the Measurement of the Physical and Chemical Properties of the Sodium-Potassium Alloy

C. T. Ewing, H. B. Atkinson, Jr. and R. R. Miller

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P. Borgstrom, Superintendent, Chemistry Division

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NAVAL RESEARCH LABORATORY
CAPTAIN H. A. SCHADE, USN, DIRECTOR
WASHINGTON, D.C.

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(A) Mine Safety Appliances Company, Callery, Pa.
Preliminary Report No. 5 "Corrosion of Certain Metals by Potassium - Sodium" by R. E. Lee, dated 1 October 1947.

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ABSTRACT

The properties of the alloys of potassium and sodium are under study. Physical and chemical properties, together with measurements of heat-transfer coefficients on an engineering scale, are being investigated. The status of active property measurements and all measured results obtained since the last Quarterly Report, C-3152 are presented. Included are recent results on viscosity, density, and corrosion. Where results are given on the property, a description of the current apparatus and methods is also presented. Changes in both apparatus and methods are necessary as dictated by experience.

AUTHORIZATION

This problem was initiated upon the request of the Bureau of Ships Project 990/46 and was assigned NRL Problem Number C01-06.

STATEMENT OF PROBLEM

The physical and chemical properties of liquid metals, together with measurements of heat transfer coefficients on an engineering scale, are being investigated. The investigation to date has been concerned with the alkali metals.

PROPERTY MEASUREMENTS

The present Quarterly Report combines the results obtained by the Laboratory and contractors with the Laboratory since the preceding Quarterly Report, (1) and covers the period from July 1 to October 1, 1947. Included as an Appendix is a report by Mine Safety Appliances Company, "Preliminary Report No. 5, Corrosion of Certain Metals by Potassium-Sodium".

PURIFICATION

Purification studies have been somewhat neglected during the last three months in an effort to speed up some of the urgent property measurements. Nevertheless, studies regarding the effectiveness of alloy scavangers, centrifuging, stripping columns, etc., are still active. Methods for the determination of traces of oxygen in covering gases are still being tested, and preliminary experiments indicate that the oxygen content of the nitrogen, passed at a reasonable rate through the standard purification train, is much less than 0.01 percent by weight.

The desirability of the metal still over the glass still for distillation of the metals was adequately covered in the preceding report, for nothing further has been learned regarding the action of the metal vapor on glass. Both nickel and stainless steel stills have been constructed for the high-vacuum distillation work, and both types have been used successfully. Pressures attainable with these stills were always less than 0.1 micron-- and usually below the scale range of the McLeod gage (.01 micron). Vacuum tight nickel welds were obtained with pure nickel and rod and oxy-acetylene welding. The stills were always pickled by recognized pickling procedures.

A diagram of the nickel still now being used for distillation work is presented as Plate III. The type of metal to glass seal for best results was for a time a source of concern. Three types

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(1) Ewing, C.T; Hartman, R.S.; and Atkinson, H.B.Jr., NRL Report C-3152, "Quarterly Progress Report No. 4 on the Measurement of the Physical & Chemical Properties of the Sodium-Potassium Alloy", July 1947.

of seals have been employed: (1) copper housekeeper metal to pyrex, (2) covar metal to pyrex, and (3) metal to glass ground joint (spherical). The type of seal for connection to vacuum where no application of heat is required was not critical. However, the joint through which the condensed metal had to pass was subjected to rough temperature treatment. The copper housekeeper seals -- nickel plated -- were used successfully for distillation with the stainless steel still; but breakage due to temperature shock from hot metal slugs passing through during distillation and from operations involved in cleaning out the still, prohibited efficient use. The same difficulties were experienced with covar seals. A ground metal to glass joint has given best results and is being used at present. The metal male portion of the joint is welded directly to the still and is machined with a projecting tube that extends down inside the glass tube to prevent contamination of the effluent metals by the sealing compound. The joint is sealed with a high temperature silicone compound and secured with a standard clamp. The application of the spherical joints, then, provided tight seals and some flexability for the heavy stills when attached to the glass system. The metal filling tube of the still is equipped with a standard tapered metal joint, for ease of introduction of the metal. The sintered glass filter is placed between the still and the receiver to filter out any scale or other solid impurities present in the metals. The temperature of the filter was not such as to invoke action of the metals on the glass.

VISCOSITY

Known Facts Bearing on Viscosity

Surface Tension and Effective Head - As was shown in the discussion of viscosity in NRL Report C-3105, the major obstacle in measurements with the Ostwald viscometer was the influence of surface tension on the hydrostatic head. An accurate value for the head at any position of the menisci between the two extremes normally could be determined by noting the actual balanced hydrostatic pressure as registered by an attached manometer. With the metals, however, which presumably have relatively high surface tensions, changes in curvature of menisci as the moving menisci come to a stationary equilibrium can result in appreciable error. This was demonstrated in an attempt to measure alloy heads using the Fenske viscometer described in NRL Report C-3105. With such a small measuring bulb (3cc volume) and receiving bulb (3cm diameter), the head value as observed with the attached manometer was continually fluctuating up and down. Change in curvature of meniscus as equilibrium was reached, resulted in an increase in head allowing the liquid to move down and repeat the cycle, giving rise to the fluctuating head. The fluctuations were observed to be greatest when the meniscus of the top measuring bulb was in the smaller diameters. Obviously, no accurate head values could be obtained under these conditions. Every precaution was taken to assure that the fluctuations did not result from temperature changes in the limbs of the manometer and viscometer.

The solution of these difficulties lay in the use of a modified viscometer with a larger measuring bulb and a larger receiving bulb. The viscometer used for the present measurements had a measuring bulb with 30 cc volume and main diameter of 5 cm, and a receiving bulb of 7 cm diameter. With the large diameters, the shape of the meniscus had little effect on head values, and the heads were readily measurable. The shape of the measuring bulb was such that the top meniscus was in the smaller diameters at the measuring marks for only a very small fraction of the total time.

To determine the mean effective head at any temperature it was necessary to (1) note the actual hydrostatic pressure, as registered by the attached manometer, corresponding with successive positions of the menisci between the upper and lower extremes, and (2) note the times required for these positions to be reached when the liquid ran out under its own head. Now, from a plot of the experimental heads at successive positions of the falling menisci against the fraction of total time required to reach each position during the run, a mean hydrostatic head was determined by graphical integration. The mean head as obtained differed from that calculated from the differences of level at mean time due to surface effects.

Calibration and Kinetic Energy - It has been shown in preceding reports that for a viscometer of the capillary type, the Poiseuille equation for absolute viscosity including the correction for any gain in kinetic energy is:

$$\eta = \frac{\pi P t r^4}{8 V l} - \frac{m V d}{8 \pi l t}$$

η - absolute viscosity
P - mean effective pressure
t - time
V - volume
r - capillary radius
l - capillary length
m - kinetic energy constant
d - density

Now, when relative measurements are to be made by calibration of the viscometer with a fluid of known viscosity, all of the terms in the equation above which are constant for a given viscometer can be determined experimentally. Grouping all these constant terms, the equation then reduces to:

$$\eta = APt - Bd/t$$

To determine the viscometer constants A and B, a graphical method was employed. The time of flow and the mean effective head was determined for water at several temperatures. Then by plotting η/Pt against d/Pt^2, a straight line was obtained, the intercept and the slope of which determine the values of A and B, respectively. The absolute viscosity of the alloy at any temperature was then calculable from a knowledge of its density, the time of transpiration, and the mean effective pressure. An apparent advantage in the above method of calibration is that any expansion in the pyrex viscometer, though slight, will be included in the empirical constants.

Methods

Apparatus - The modified viscometer, the attached glass system, and the temperature bath are schematically presented in Plate IV. In line with standard practices, the complete viscometer unit was portable, designed for operation as a closed system and for direct distillation of the metal into the receiving bulb. Therefore, the elaborate attachments to the viscometer required under the old procedure (NRL Report C-3105) were no longer necessary. Octoil-S, with extremely low vapor pressure, was used as the manometer fluid, to allow evacuation of the manometer arms. Manometer readings were made with a cathetometer, with the manometer inclosed in a liquid bath with stirrer and insulated from heat.

The temperature bath was a standard assembly; silicone fluid in a large unsilvered dewar, electrically heated and controlled. Sensitive temperature control was provided by a mercury thermoregulator and a super-sensitive relay in conjunction with a 500 watt heater and a variac. A number of regulators were set for different temperatures over the required range, to avoid resetting a single regulator after each run. A variable high-speed stirrer provided adequate circulation of the fluid. This bath would control to ± .03°C at 50°C and ± .2°C at 200°C.

Temperatures were noted with a Rubicon high-precision, type B, potentiometer using a five junction iron-constantan thermel. The calibration of this thermel against standard temperature reference points was described in NRL Report C-3105, p. 13. The thermel was further calibrated against two Bureau of Standards couples to 200°C. The thermel is considered accurate to ± .05°C to 100°C and ± .3°C to 200°C.

Operation - The portability of the viscometer unit facilitated the operations involved in cleaning and introducing the metals. The apparatus assembled for the distillation work was described in the preceding section on purification. The filling arm of the cleaned viscometer was sealed directly to the arm of the still below the sintered glass filter, such that the receiving bulb of the viscometer served as the receiver of the still. Preceding the distillation, the viscometer and the still were heated to at least 250°C under high vacuum. The distillation of each pure metal was performed in a nickel still, and no distillation was made unless the initial pressure was less than .1 micron. The desired volume of metal having been introduced into the viscometer and the filling arm sealed off under a slight negative pressure of nitrogen, the viscometer unit was ready for removal to the temperature bath and subsequent viscosity determinations.

Manipulation of the liquid into the measuring bulb was accomplished by manipulating the vacuum seal stopcocks (A) and (B), while alternately cooling and heating the bulb (C). Then to observe the time of transpiration between the two marks on the measuring bulb, it was necessary only to open the stopcock at (B) to equalize the pressure of gas in the two limbs of the viscometer.

Discussion of Results

This report presents viscosity measurements on sodium and potassium metals from their melting points to 200°C. (Tables I-II). It is believed that the metals used in these determinations were purer than any that have been prepared at the Laboratory. Each metal, as removed from the viscometer, analyzed within the accuracy of the analytical methods to 100 percent purity.

The precautions adopted in the design of the present viscometer avoid the possibility of appreciable error from either surface tension or drainage effects. Any error due to retention of liquid on the surface of the measuring bulb tends to be less, due to the relatively small surface per unit volume. While no adherence of the sodium to the walls of the measuring bulb was observed, a thin layer of potassium was noted to stick at the very top and at the very bottom of the bulb. This caused some difficulty in observation of the flow of liquid past the measuring marks, but the effect of volume change on viscosity results would be insignificant. The large measuring bulb also tends to minimize surface effects due to the relatively small ratio of circumference to surface area on the face of the meniscus. The procedure for determining a mean-effective-head which includes surface effects has been discussed. For any one temperature, the determination of approximately ten heads between the two extremes was found adequate to obtain an accurate value of the mean-head. For the abbreviated temperature range, actually only one mean-head was required for each metal. Variation of mean-head in terms of metal height was found experimentally to be practically negligible, and, too, could be calculated knowing the diameter of the lower receiving bulb, the density change, and the approximate volume of metal introduced. The determinations of two or more mean-heads over the temperature range were made merely to establish the accuracy of the head value.

The method for calibration of the viscometer by preliminary experiments with water has already been described. The calibration constants, determined by graphical solution of water results, were $A = 3.792 \times 10^{-9}$ and $B = .135$ using cgs units. The accuracy of these constants and subsequent viscosity values for the metals, necessarily depended on the preciseness of the water determinations. An indication of the accuracy of the water measurements may be obtained by a comparison of calculated viscosities for the water data against corresponding observed values, where the maximum deviations amounts to only 0.2 percent. The rate of flow with the fluid alloys was sufficiently high as to make the kinetic energy correction appreciable. However, the correction term for even the more fluid potassium amounts to only 4 percent of the viscosity value at 200°C. The overall accuracy of the reported viscosity results is believed to be better than ± 1 percent. However, the effect of possible attack on the glass capillary during cleaning out operations cannot be overlooked. At the conclusion of alloy determinations with the viscometer, a check calibration with water will be made.

The viscosity values from Tables I-II are plotted in Plate I, for comparison with the experimental values reported by Chiong.(2) The viscosity values for sodium show excellent agreement with those reported by Chiong. The values for potassium, however, agree with Chiong's data at 70°C but diverge as the temperature is increased, differing by as much as 10 percent at 200°C. There is at present no explanation for this difference. Andrade(3) has advanced a formula to give the variation of the viscosity of a liquid with temperature. Chiong, in fitting this equation to his experimental data for potassium, found a maximum deviation of only 0.59 percent for potassium. However, our experimental data for potassium fits an equation of the type advanced by Andrade equally well. The densities used by Ch'ong in the calculation of absolute viscosities are not too well defined, but apparently could not differ more than 0.2 percent from the densities used in our calculations.

The present measurements are not conclusive enough to predict possible error in application of the approximate mixture law with Chiong's data, reported in NRL Report C-3105. However, the results reported for potassium in this report and for the alloys in NRL Report C-3105 indicate, with reservation, that the values for potassium and for the high potassium alloys by the mixture law may be high by as much as 15 percent at 400°C and 20 percent at 700°C. The measurements now underway on metal alloys covering the composition range should clarify viscosity in the lower temperature range, and it is hoped that an accurate mixture law can be presented soon. The large nickel viscometer has been reassembled with a smaller capillary, and viscosity values for the metals up to 700°C will be redetermined.

CONCLUSIONS

The present measurements throw unexpected uncertainty on the potassium values reported by Chiong. Thus the application of the approximate mixture law reported in NRL Report C-3105 to Chiong's extended data may be off in the high potassium range by as much as 20 percent at 700°C.

Barring difficulties, viscosity measurements accurate to better than ± 1 percent up to 200°C will be made with the pyrex viscometer on alloys covering the composition range. A thorough investigation of mixture laws will be made, and their application to the

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(2) Y. S. Chiong, Proc. Roy. Soc. London, A 157, 264 (1936)
(3) E. N. Andrade, Da Cl, Phil. Mag., Ser. 7, 17, 698(March 1934)Part II

present work will be presented.

The large nickel viscometer has been reassembled with a smaller nickel capillary. As soon as possible, results will be obtained on the liquid metals up to 700°C.

DENSITY

Known Facts Bearing on Density

Density values for the composition-temperature range have been reported by MSA. The direct bearing of density on the precision of property measurements and heat transfer data justifies every effort to better establish the validity of the existing data. To this end, determinations were made at the Laboratory with quartz dilatometers in the lower temperature range and show reasonable agreement with the MSA dilatometer values.

Methods

Apparatus - The quartz dilatometer which was assembled in a constant-temperature furnace for density determination, is shown in Plate V. In all, two quartz dilatometers were used, the bulb of each having a volume of approximately 33 cc and the capillary approximately 0.25 cc per cm of length. Obviously, any error due to shape of meniscus or to expansion of quartz was insignificant. The furnace, which was previously described under Viscosity in NRL Report C-3105, was here suitable for temperature control to better than ± 0.5°C. Temperature readings were noted with a Leeds and Northrup, type K, potentiometer using a Pt-PtRh thermocouple. The calibration of this couple (previously used in viscosity determinations) was rechecked by additional calibrations over the temperature range against a Bureau of Standards couple. It can be conservatively stated that for these measurements, the temperatures were controlled and known to better than ± 0.5°C.

All weighings were carried out on a heavy beam balance accurate to ±5milligrams.

Operation - Each dilatometer was calibrated with fresh triple distilled mercury. At least three fillings of mercury were used for each dilatometer, calibrating three overlapping sections of the capillary.

Before introduction of the liquid metal or alloy, the usual precautions were taken to thoroughly clean the quartz surfaces, finally preheating to 300°C under vacuum. The metal in each case was distilled from a stainless steel still through a metal-to-glass seal directly into the dilatometer, and the dilatometer fused off under a slight negative pressure of nitrogen. Then to make a density determination, it was necessary merely to allow the furnace to reach equilibrium at the desired temperature and to note with a cathetometer the height of the meniscus relative to a reference mark.

During the filling of the dilatometer, small globules of the alloy were found to adhere to the surfaces of the tube extending from the capillary. To eliminate appreciable error from this source, the net weight of the metal was determined at the conclusion of the runs. The dilatometer was broken off in the capillary immediately above the calibrated zone, and the weight of the lower portion noted before and after removal of the alloy. The microstopcock at the top of the dilatometer was used to introduce a positive pressure of nitrogen for the breaking operation.

Experimental Results

This report presents dilatometric measurements for pure sodium and potassium, and for two alloys (2.9 and 13.9 atom-percent potassium) which are listed in Tables III-V. Each composition value represents an analysis on the metal as removed from the dilatometer at the conclusion of the measurements.

CONCLUSIONS

The values presented tend to substantiate the low temperature determinations made by MSA, when all results are compared on isothermal plots of density against composition. However, an extrapolation of NRL data for pure sodium would give a value for density at the melting point of .927 as compared to .938 by Hackspill which was used by MSA in the extrapolation of the sodium buoyancy data. There still exists some discrepances in the dilatometric data, but additional measurements on three or four alloys should clarify density in the lower temperature range.

Density values as obtained at MSA by buoyancy measurements with a copper plunger (NRL Report P-3010) would appear to be in error by the amount of the surface tension force acting on the support wire, which could amount to as much as 1 percent at the higher temperatures. Rough surface tension corrections (using extrapolated values for surface tensions) when applied to the buoyancy data for the pure metals result in new density values which fall in line with those reported by E. Rinck.(4) It is anticipated that surface tension corrections to existing buoyancy data, above and below the wetting point, will clarify some of the discrepances in the data, - at least in the potassium-rich range. However, at present there are no surface tension values for the alloys except through mixture laws, and only questionable values for the pure metals.

If possible, a complete study of density and comprehensive curves will be presented in the next report.

SURFACE TENSION

Unforeseen difficulties arising in surface tension determin-

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(4) E. Rinck, Comptes Rendus, 189, 39-41 (1929)

ations with the apparatus described in the preceding report have prevented any accurate measurements to date. However, preliminary measurements indicate that the surface tension of the metals could be much lower than indicated by questionable literature values. Surface tension measurements are in progress.

SPECIFIC HEAT

Specific heat measurements are being made at the Laboratory with the modified calorimetric method described in the preceding Report. Measurements have been made on the pure metals and on one alloy to approximately 700°C. Those measurements will be completed in the near future and the data reported in the next Quarterly Report.

WETTING TEMPERATURES

Wetting determinations have been delayed in favor of other property measurements and to allow progress in purification methods.

CONCLUSIONS

A study of the properties of the sodium-potassium alloy is being continued.

TABLE I

VISCOSITY OF SODIUM

Temperature °C	Density gm/cc	Absolute Viscosity (centipoises)
103.7	.925	.692
103.7	.925	.692
121.6	.922	.628
121.6	.922	.624
121.6	.922	.624
147.0	.916	.552
147.0	.916	.552
167.6	.911	.509
167.6	.911	.509
192.7	.905	.465
192.7	.905	.479
192.7	.905	.478

TABLE XI

VISCOSITY OF POTASSIUM

Temperature °C	Density gm/cc	Absolute Viscosity (centipoises)
69.3	.827	.521
69.3	.827	.519
69.3	.827	.519
103.7	.819	.436
103.7	.819	.430
121.5	.815	.397
121.5	.815	.399
147.0	.809	.358
147.0	.809	.356
167.4	.804	.334
167.4	.804	.334
192.7	.798	.307
192.7	.798	.307

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TABLE III

DENSITY OF POTASSIUM

(100.0 Percent Potassium)

Temperature °C	Density grm/cc	Temperature °C	Density grm/cc	Temperature °C	Density grm/cc
90.5	.8204	144.5	.8089	214.0	.7937
105.0	.8182	164.5	.8044	228.0	.7908
128.0	.8133	188.0	.7997	241.5	.7869
		200.5	.7975	259.5	.7836

TABLE IV

DENSITY OF SODIUM - POTASSIUM ALLOY

(4.8 Weight-Percent Potassium)
(2.9 Atom-Percent Potassium)

Temperature °C	Density grm/cc	Temperature °C	Density grm/cc	Temperature °C	Density grm/cc
149.0	.9120	137.0	.9150	263.0	.8848
175.5	.9053	160.5	.9095	274.5	.8824
197.5	.9012	184.5	.9039	288.0	.8788
208.0	.8982	219.0	.8966	243.5	.8838
124.0	.9170	235.5	.8909	206.0	.8976
		249.5	.8869		

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TABLE V

DENSITY OF SODIUM - POTASSIUM ALLOY

(21.5 Weight-Percent Potassium)
(13.9 Atom-Percent Potassium)

Temperature °C	Density grm/cc	Temperature °C	Density grm/cc	Temperature °C	Density grm/cc
54.0	.9203	167.5	.8917	263.5	.8679
73.0	.9147	187.0	.8872	246.5	.8739
89.0	.9119	173.5	.8909	220.5	.8793
105.5	.9063	184.5	.8872	196.0	.8842
124.0	.9014	210.0	.8812	172.5	.8879
140.0	.8983	234.0	.8751	152.0	.8940
149.5	.8950	256.5	.8693	127.0	.9006
		281.5	.8648	102.5	.9058

TABLE VI

DENSITY OF SODIUM

(100.0 Percent Sodium)

Temperature °C	Density grm/cc	Temperature °C	Density grm/cc	Temperature °C	Density grm/cc
110.0	.9233	194.5	.9053	246.0	.8931
140.0	.9177	220.0	.8994	233.0	.8957
164.0	.9125	245.5	.8931	205.5	.9013
185.5	.9075	121.0	.9218	188.0	.9053
206.0	.9027	130.0	.9203	159.5	.9113
235.0	.8959	141.0	.9164	140.0	.9159
261.5	.8899	132.5	.9196	123.5	.9211
150.0	.9150	126.5	.9206	114.0	.9221
170.0	.9106	118.0	.9218	107.5	.9246

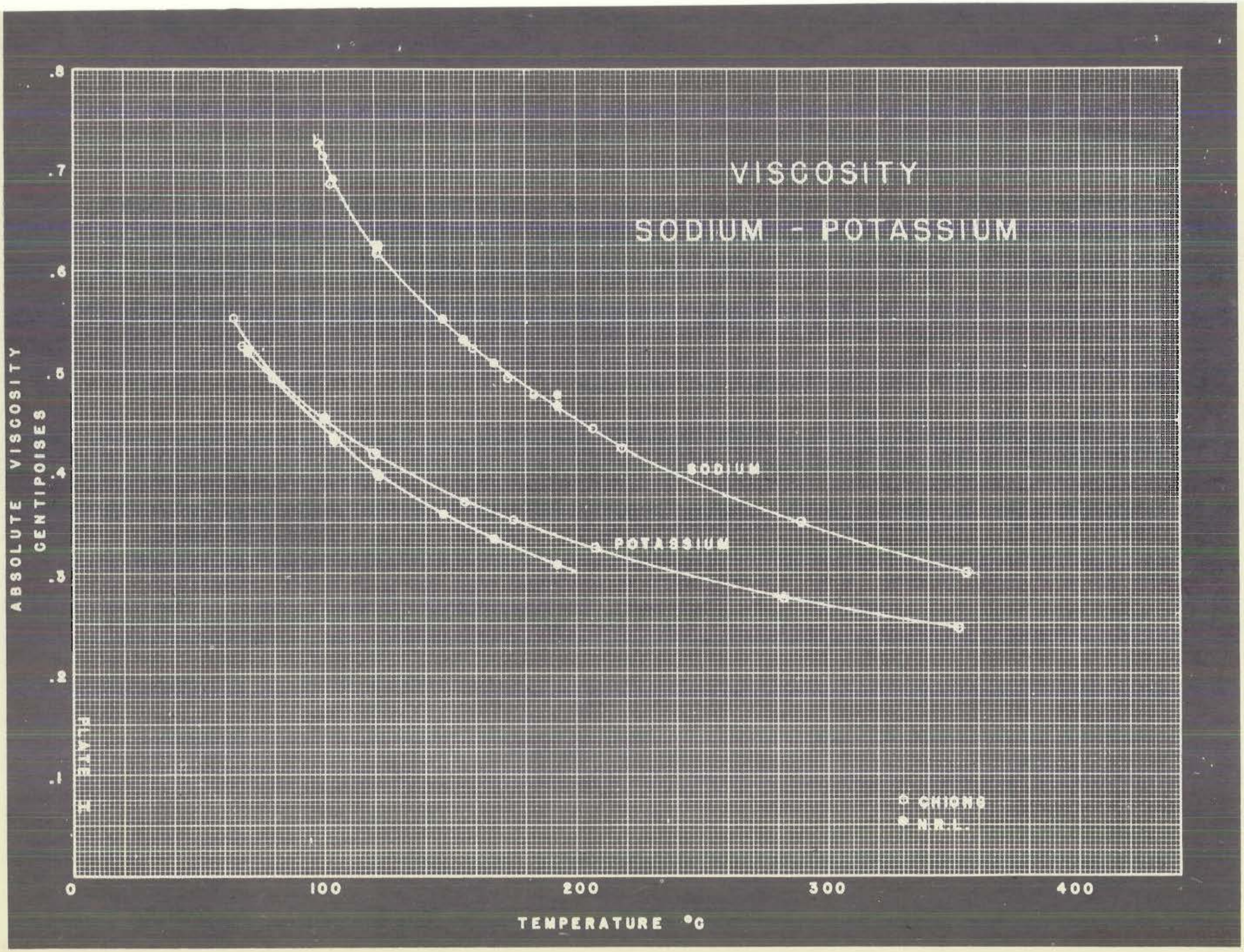
VISCOSITY
SODIUM - POTASSIUM
ABSOLUTE VISCOSITY CENTIPOISES
TEMPERATURE °C
SODIUM
POTASSIUM
○ CHIONG
● N.R.L.
.8
.7
.6
.5
.4
.3
.2
.1
0
100
200
300
400

PLATE II

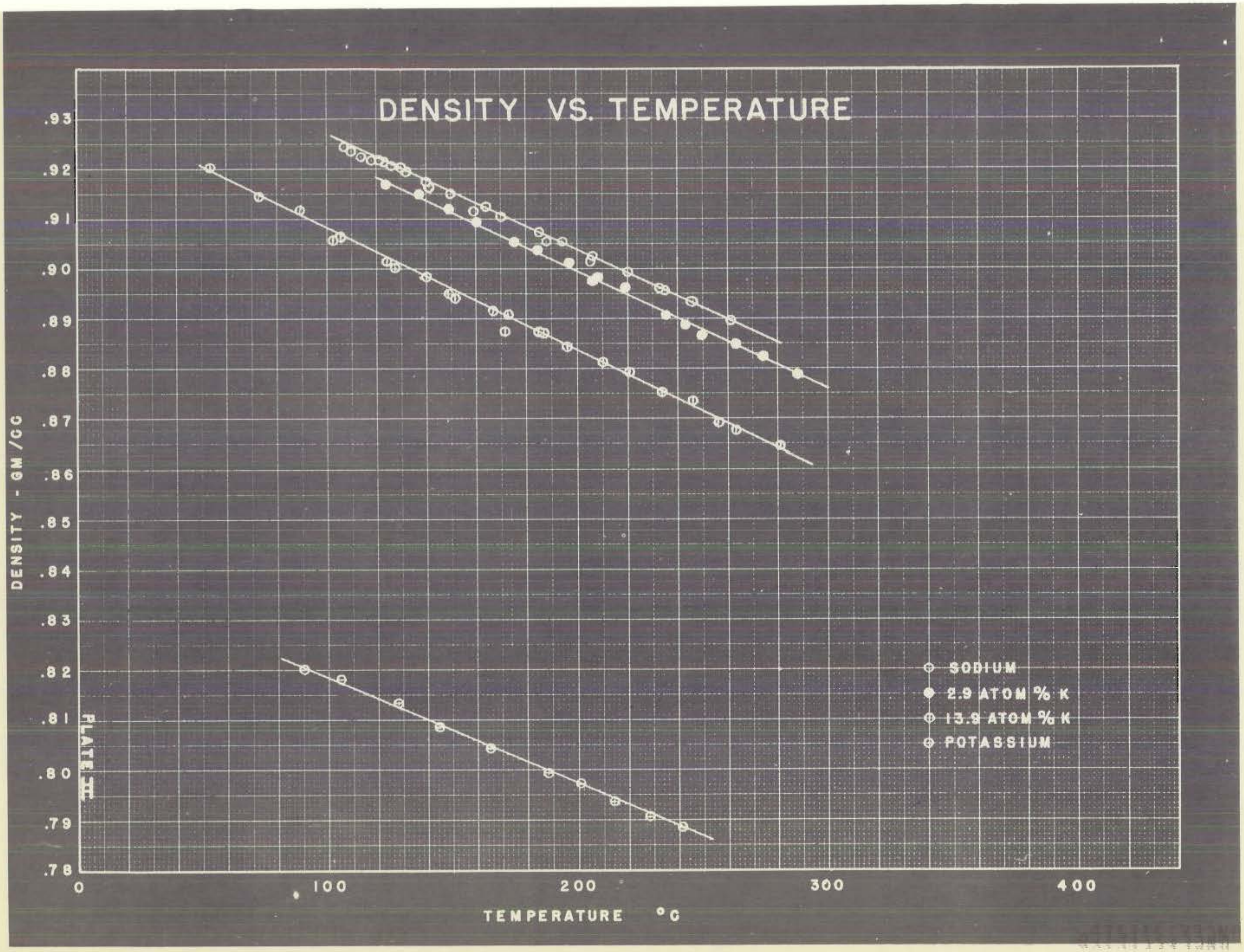
DENSITY VS. TEMPERATURE
DENSITY - GM /CC
.93
.92
.91
.90
.89
.88
.87
.86
.85
.84
.83
.82
.81
.80
.79
.78
0
100
200
300
400
TEMPERATURE °C
⊖ SODIUM
● 2.9 ATOM % K
⊕ 13.9 ATOM % K
⊖ POTASSIUM

PLATE III

METAL STILL

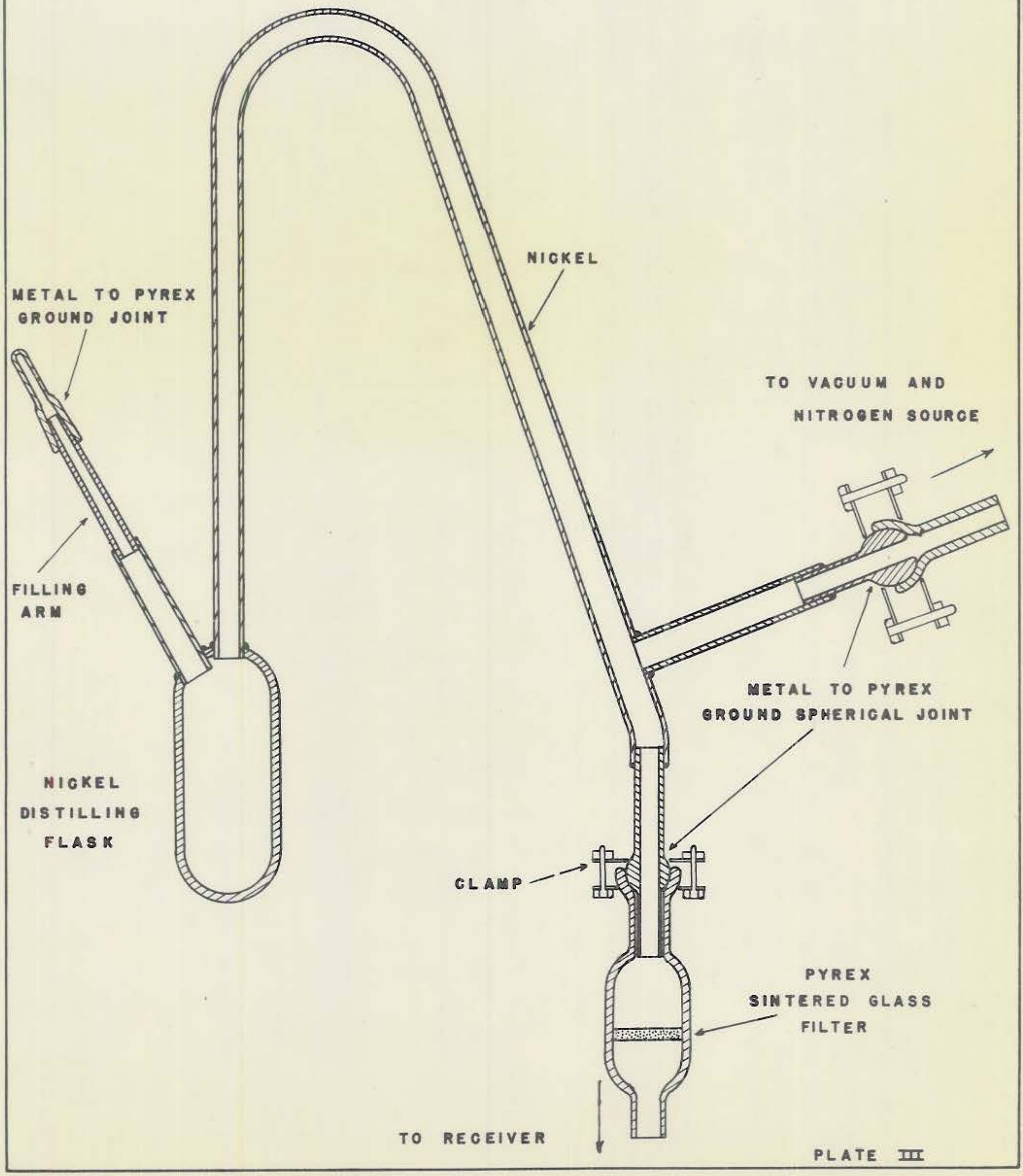

PLATE III

VISCOSITY APPARATUS

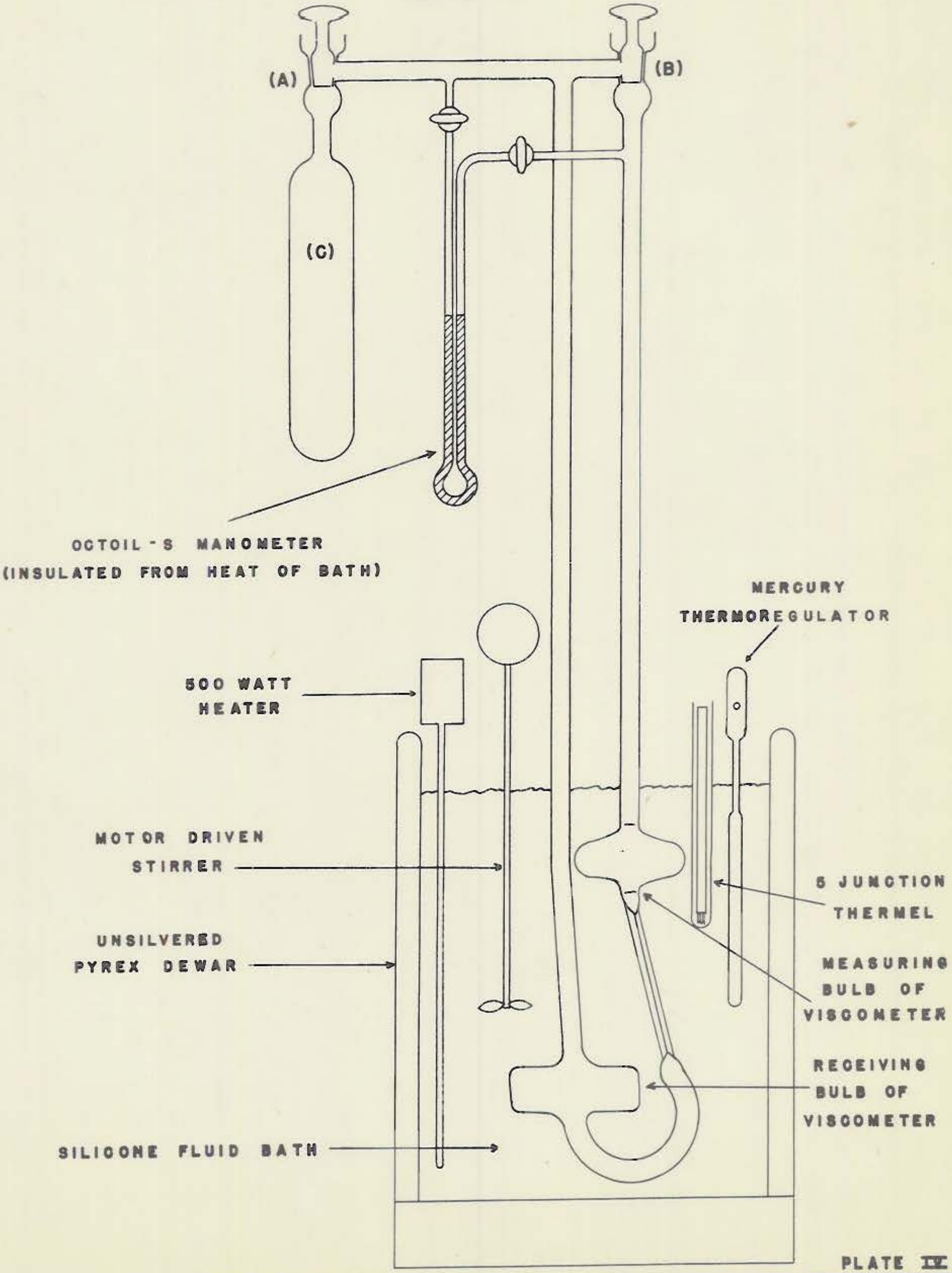

PLATE IV

DILATOMETER

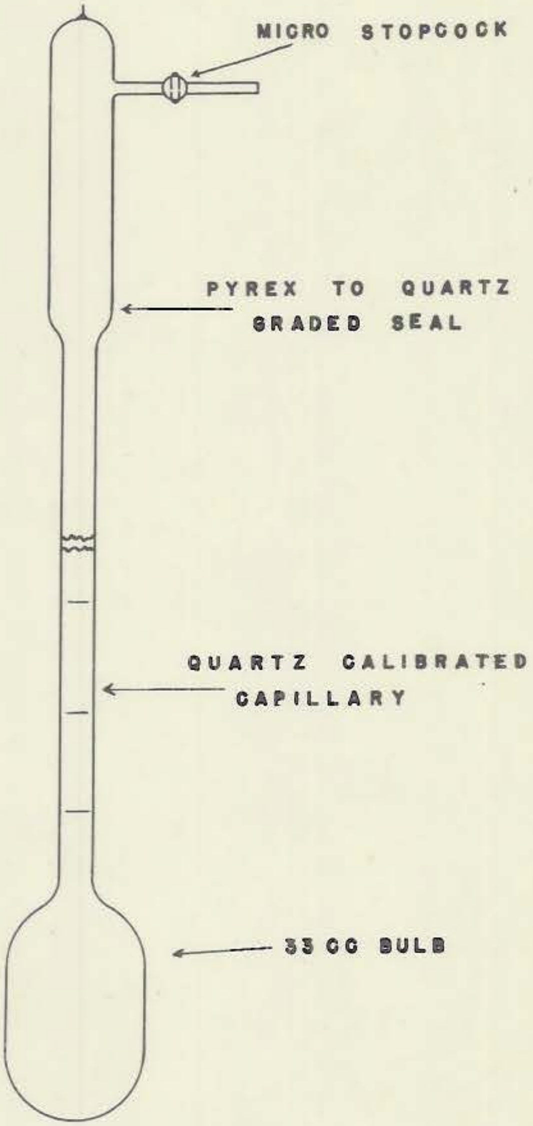

PLATE V

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Preliminary Report No. 5

CORROSION OF CERTAIN METALS BY POTASSIUM-SODIUM

by

R. E. Lee

MINE SAFETY APPLIANCES CO.
Callery, Pa.

October 1, 1947

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Weight Changes

Examination of Specimens

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2. Type 304 vs. KNa 44.
3. Nickel A vs. KNa 44.
4. Inconel vs. KNa 44.
5. Type 310 vs. KNa 44.
6. Type 304 vs. Na
7. Type 304 vs. K
8. SAE 1020 vs. Na

CONCLUSIONS

ABSTRACT

The following materials were tested for resistance to attack by Na, K, and KNa at 1400°-1600°F: mild steels, nickel, Inconel, Type 304, and Type 310. The mild steels were unsatisfactory under the conditions of test. The other materials showed little solution but certain other undesirable conditions were noted.

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INTRODUCTION

Authorization

The investigation reported here was authorized under Task Order I of Contract N6ori-146.

Statement of Problem

While potassium and sodium have been handled in large quantities for many years, no quantitative information is available indicating the stability of materials of construction to the action of these metals or their mixtures.

Accepted corrosion testing procedures are lacking for the case in question. Accordingly, a simple procedure was adopted for this study; it is believed that this testing procedure is so strenuous that various materials can be roughly classified as to their resistance to attack by the alkali metals.

The materials indicated by the procedure of this report to withstand attack by potassium and sodium will be subjected to more refined procedures of test. The results of further corrosion tests on the more resistant materials will be issued in the future as a supplement to this report.

Acknowledgement

The examinations of the test specimens were made by Mr. C. N. Emmanuel under the supervision of Mr. H. D. Newell, Chief Metallurgist, of the Babcock and Wilcox Tube Company.

METHODS

General

The use of liquid potassium, sodium or potassium-sodium alloys at high temperatures requires the use of piping, tanks, valves, pumps, gages, flowmeters, etc. constructed of materials that have a sufficiently long useful life. As far as can be determined, there is no published information on the solution of commonly used materials in the alkali metals at high temperatures.

The Mine Safety Appliances Co. has been distilling potassium and potassium-sodium alloys for a number of years in batch retorts as well as in continuous stills at atmospheric pressure. While corrosion in these units is not strictly applicable to the present problem, it is thought worth while to present here some information which has been accumulated in production of KNa alloys. The batch retorts used were modeled after a glass distilling flask, being constructed of Type 316 plate of 1/4" thickness. The neck of the retort was Type 316 seamless 6" I.P.S. tubing while the side arm (condenser) was usually Type 304 seamless 2" I.P.S. tubing. These retorts were used in reducing potassium chloride with sodium, and the corrosion is due to chloride as well as alkali metal. The batch operation entailed great changes in temperature; cracking of the side of the retort frequently occurred approximately at the liquid level. The retorts were grossly overheated at times. Sections from one of the retorts were taken from the side and from the condenser tube. External corrosion is not pertinent to the problem of this report.

Fig. 1 is a photograph of the samples as taken. Fig. 2 is a photograph of a section of the condenser tube showing a black (carburized area) on the I.D.

One half of the I.D. of the tube was covered with rust tubercles and showed signs of some pitting attack. Upon pickling, a black streak, 3/4" wide, was observed to extend the length of the tube. The opposite half of the bore appeared singularly clean as though the fluid carried by the tube had occupied only the bottom portion.

One of the plate sections was approximately 6" square x 1/4", the outside of which was covered with a blue-black oxide which was dotted with weld spatter. There were four actual cracks on this surface ranging from 1/4" - 2-1/2" long which, however, did not penetrate through the wall.

Except for size, the other section was identical in appearance. About midway on the long side was a failure 4" long and extending through the wall. This section also contained an arc weld at one end running parallel to the short side.

Hardness measurements made on the condenser tube are listed below, but since the values are in accord with ASTM Specification A-269-44T (R_b 90 max.), no special significance is attached to them.

Rockwell B	Average
75,76,75,73,77,73,76	75

The hardness values of a slice cut from one of the plate sections are shown in Fig. 3, however the average of these readings, excluding the two on the weld and the 58 value next to the fracture, is 69.9 Rockwell B. Since hardness of the stainless alloys is not particularly sensitive to ordinary heat treating temperatures, this rather low value is indicative of exposure to extremely high temperatures and may be due to grain coarsening.

Chemical analyses made on drillings taken from the kettle section and from the condenser tube yielded the following results:

	Kettle	Tube
Carbon	.08	.07
Chromium	17.53	18.61
Nickel	13.13	10.40
Molybdenum	2.10	nil

The above indicates the kettle to be a stainless steel alloy of Type 316 whereas the tube is stainless Type 304.

The general structure of the tube is represented by Fig. 4 which reveals an ASTM grain size of 3-4.

Fig. 5 depicts the extensive grain boundary precipitation which is indicative of a sojourn at a temperature of 1100-1300 F. sometime during the service life of the tubs. It is this condition which sensitizes the material to intercrystalline attack when exposed to corrosive media. The results of such an exposure are illustrated in Fig. 6 which shows the intergranular separation on the O.D. of the tube which follows chromium carbide precipitation.

Fig. 7 and Fig. 8 show bore carburization of the condenser tube at the black streak shown in Fig. 2. Carbon analyses yielded the following:

At streak	Edge of streak	Opposite streak
0.228%	0.124%	0.07%

This bore carburization is unexplainable at this time. The only carbonaceous material in the system was the minute quantity of oil occasionally occurring on the sodium used.

Fig. 9 shows the interface between the parent metal and one of the welds found in the tube. Fig. 10 indicates the general appearance of the structure which is definitely coarse-grained, ASTM grain size 1. The junction between the arc weld and the parent metal is illustrated in Fig. 11.

The preceding information does not apply to the problem of this report but is indicative of what may be expected under such strenuous operating conditions.

Apparatus.

Since these corrosion tests were to give information to determine which materials were most resistant to attack by K, Na, and KNa, it was decided to measure, if possible, the solution of the metals in KNa at the boiling point of the KNa at atmospheric pressure. Fig. 12 is a diagrammatic sketch of the apparatus used; all portions of the apparatus, as well as the test pieces, were of the same metal in order to avoid possible electrolytic effects. The boiler consisted of a 17" length of 2" I.P.S. tubing, with top and bottom plate welded with proper welding rod. The top plate was

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fitted with an inlet tube for nitrogen and a thermocouple well. The top plate also was fitted with a hook to hold a specimen in the vapor phase.

The boiler was heated by an insulated electrical muffle unit, with the power input controlled by a variable transformer to keep the KNa boiling at such a rate that the vapor almost reached the top plate of the boiler.

It was found necessary to bubble N_2 slowly into the Kna to prevent superheating. Without the introduction of the N_2, superheating as much as 200 F degrees occurred, followed by flash boiling which would shake the entire apparatus.

Fig. 13 is a photograph of a complete apparatus during a test.

This apparatus was used for the corrosion tests since it was desired to study the effect at as high a temperature as possible without pressure, i.e. the boiling point. Since both liquid and vapor phases of the alkali metal were present, specimens were used to show the differences, if any, between the condensing vapor and the liquid phase.

The liquid phase sample was a section of 1/2" I.P.S. tubing x 2-1/2" long which was dropped in the bottom of the boiler before the top plate was welded on. The vapor phase sample was a section of 1/2" I.P.S. tubing x 11" long. In every case a section of the tubing between the samples was reserved as a blank.

The apparatus and samples were given a mild cleaning before use; this consisted of washing with soap and water, rinsing, drying and a mild buffing.

After the apparatus was assembled with the test pieces in place, the system was filled with the alkali metal to the level indicated in Fig. 12, by forcing the alloy from a container into the boiler by way of the nitrogen inlet. Heat was then applied and the temperature maintained at the boiling point for the indicated time.

The alkali metal undoubtedly contained some oxides; before use, however, the alkali metal was distilled under commercial conditions. All N_2 used had been passed over Cu turnings at 800°F and dried with activated alumina.

On the conclusion of the test the apparatus and contents were cooled to room temperature or below. The top plate of the boiler was sawed off as shown in Fig. 14 and the test samples removed in air as quickly as possible; the samples were immersed immediately in a large quantity of water. On removal from the water, the specimens were washed with distilled water and dried.

RESULTS

Weight Changes

Tests were run on various metals using KNa, Na, or K. The loss in weight was determined by weighing before and after to the closest tenth of a gram.

Table I gives results of the weight changes.

Examination of specimens.

1. Wrought Iron vs. KNa 44. The parts of the apparatus for the 100 hour and the 500 hour tests are shown in Fig. 14 and Fig. 15 respectively.

Chemical analyses of the component parts of the 500 hour test as given below indicate that the test pieces were wrought iron while the/boiler and nitrogen tube were Bessemer grade steel.

	Blank	500 hrs. liquid	500 hrs. vapor	Boiler	N_2 tube
Carbon	.05	0.066	0.058	0.13	0.10
Manganese	.04	0.05	0.03	0.41	0.35
Phosphorous	.126	0.11	not run	0.102	0.091
Nitrogen	.003	0.001	nil	not run	not run

It is unfortunate that dissimilar metals were used in this case but it is felt that the dissimilarity did not account for the great corrosion.

Macroscopic examination of the samples showed an obvious decrease in wall thickness between the blank and the 500 hour liquid phase sample. This penetration is given in the table below with wall measurements on the remainder of the apparatus. The surfaces of both liquid samples were smooth and shiny in appearance indicating that the corrosion was general rather than selective. Not only the liquid phase samples, but also all parts of the apparatus which had been below the liquid level, were smooth and shiny in appearance.

This smooth appearance changed abruptly at the liquid level where a coarsely-crystalline silvery deposit was evidenced for about 2" on all exposed surfaces.

Fig. 14 shows the appearance of the test specimens, nitrogen tubes, and thermocouple well of the 100 hour test while Fig. 15 shows parts of the 500 hour apparatus. The crystalline deposit mentioned above is clearly evident in these photographs and can be seen to be considerably heavier in the longer test.

Fig. 16 shows a portion of the I.D. of the 500 hour retort at the liquid level. Immediately above the liquid level, the I.D. of the retort was quite deeply marked with numerous pits. Included in this area and adhering to the bore was the deposit mentioned previously.

Wall dimensions of the samples and boiler were;

Sample	Wall thickness	Average Loss
Blank	0.105" - 0.107"	---
100 hrs. vapor	0.102" - 0.106"	.002"
500 hrs. vapor	0.102" - 0.104"	.003"
100 hrs. liquid	0.101" - 0.103"	.004"
500 hrs. liquid	0.085" - 0.093"	.017"
Boiler (Top)	0.151	---
Boiler (6" from top)	0.137	0.014
Boiler (Bottom)	0.104	0.033

Vickers hardness tests (20 kg) were made on all of the samples and the 500 hr. boiler:

Sample	V H N	Average
Blank	167, 162, 167	165
100 hrs. vapor	88.6, 85.9, 81.6	85.4
500 hrs. vapor	79.5, 82.6, 82.6, 83.6	82.8
100 hrs. liquid	77.9, 77.9, 78.1	78.0
500 hrs. liquid	70.6, 69.2, 67.2	69.0
Boiler (Top)		R_b 68.0
Boiler (Bottom)		R_b 34.0

Ductility was measured by flattening tests and is expressed as the minimum distance between opposite walls at the time the first failure was noticed on the sample.

Sample	Distance at failure
Blank	11/32"
100 hrs. vapor	1/2"
500 hrs. vapor	7/16"
100 hrs. liquid	3/8"
500 hrs. liquid	1/4"

Samples from the lower ends of the retort and nitrogen tube were pickled in hot 1:1 hydrochloric acid and are shown in Fig. 16. It may be seen that the grains are extremely large, 1/8 to 1/4". An attempt was made to reproduce this grain size by heating a piece cut from the relatively fine grained top portion of the boiler at 2000°F for 5 hours. This heating did not give discernible grains. Additional heating of the same sample to 2200°F for 3 hours produced grains which were barely visible to the naked eye. While these heating times were not sufficiently long to allow equilibrium grain size to be reached, it is generally agreed that the major amount of grain growth for a given temperature takes place in a matter of a few hours of exposure to that temperature.

Examination of the microstructure of the samples showed no evidence of KNa or N_2 diffusion. Fig. 17 shows the structure of the blank, depicting the slag streamers and practically pure alpha ferrite grains. ASTM grain size of the blank was 3-4, while the grain size of the 500 hour liquid sample was 2-3 indicating some grain growth. Further growth could have been halted by the restraining action of the slag fibers.

Fig. 18 shows the structure of the upper end of the boiler; Fig. 19, of the structure of the bottom of the boiler, shows the very large grain size and almost complete decarburization which took place.

2. Type 304 vs. KNa 44. The apparatus used in this and subsequent tests was the same as described under the wrought iron except that all materials were of the same composition.

All test pieces had a dull gray appearance but suffered no noticeable decrease in wall thickness. The vapor samples of both the 100 hour and the 500 hour tests showed a coating of an unidentified crystalline deposit; this coating occurred on the lower 1/2 of the I.D. on the 100 hr. sample and on the lower 2" of both I.D. and O.D. of the 500 hour sample.

The results of hardness and ductility tests are given below:

	Vickers Hardness No.	Ductility
Blank	330	1/16"
100 hours vapor	240	when pressed flat, the ends split
100 hours liquid	191	when pressed flat, the ends split
500 hours vapor	199	1/16"
500 hours liquid	162	1/32"

The unusually high hardness of the blank is accounted for by the fact that the material was in a considerably strained condition.

Analysis of the samples gave the following composition:

Carbon	0.074
Manganese	0.58
Silicon	0.48
Chromium	19.02
Nickel	10.26

Fig. 20 shows the microstructure of the Type 304 stainless steel blank. A high degree of strain is clearly indicated by the slip lines. The grain size was unchanged by the test, remaining constant at ASTM grain size of 4-5 for blank and both test samples.

Fig. 21 shows a constituent representative of that found in several Type 304 specimens. In this case, the constituent was unidentified but was believed to be a diffusion of the KNa into the stainless steel. A similar condition existed in a later test of Type 304 and was identified as a diffusion product. Details of this diffusion are included later in this report.

3. <u>Nickel A vs. KNa 44.</u> Macroscopic examination revealed no significant differences between the blank and the specimens. All surfaces were bright and shiny and with the exception of the 500 hour vapor sample, no change was noticed. The lower 2" of both I.D. and O.D. of the vapor phase sample showed a light deposit of finely divided metallic particles. The ductility of the samples was unimpaired in that the specimens were pressed flat without cracking.

Micro-examination of the nickel blank and test samples showed no change except slight grain growth. The ASTM grain size of the blank was 4-6, which increased to 3-4 in the case of the 500 hour specimens. Fig. 22 shows the micro-structure of the blank which, with the exception of grain size, is representative also of the test samples. There was no evidence of diffusion by the KNa alloy into the nickel.

4. Inconel vs. KNa 44. Slight pitting of all surfaces in contact with the KNa was shown by macro-examination. This was true of those portions below the liquid level and those in contact with the vapors. The pitting was particularly evident on the vapor test specimens which had been in contact with KNa vapors over approximately 1/2 their lengths. The upper portion which extended beyond the confines of the furnace was relatively unchanged while the lower section was marked with numerous small pits.

Flattening tests were made on the blank and test specimens as a measure of the ductility. No embrittlement took place as was shown by the fact that all samples were pressed flat with no failure.

The only change occurring in the micro-structure was grain growth from ASTM grain size of 6-8 to one of 2-4 in the 500 hour samples. Fig. 23 is representative of the inconel blank with a grain size of 7-8.

5. Type 310 vs. KNa 44. The hardness and ductility are given below:

	V H N	Ductility
Blank	373	pressed flat
100 hours vapor	169	Pressed flat; ends slightly split 1/8"
100 hours liquid	165	
500 hours vapor	211	Broke in half @ 5/16"
500 hours liquid	195	Broke in half @ 5/16"

The intermetallic compound FeCr, commonly designated as sigma phase, was developed in all of the specimens. Fig. 24 shows the original conditions. Fig. 25 shows the presence of the sigma phase. At the temperatures at which the test was conducted, sigma is rapidly produced. Its presence seemed to promote a certain amount of embrittlement as indicated by the ductility tests on the 500 hour samples. The increase in hardness of the 500 hour samples over the 100 hour samples is believed due to the increasing amounts of the hard and brittle constituent - sigma phase.

Production of sigma is most rapid in the temperature range of 1400 - 1600°F although little or none is produced during exposures of three hours or less. At lower temperatures, growth of FeCr is quite slow; 1000 hours exposure at 1000°F produces no noticeable amounts of it. This constituent can be re-dissolved by heating specimens above 1950°F. Production of sigma phase can be retarded by a preliminary treatment to enlarge the grain size. This would consist of heating 2-3 hours at 2300°F and quenching. This large grain size, however, may entail introduction of other undesirable characteristics such as brittleness due to the excessive grain size.

6. Type 304 vs. Na. This test was run at a higher temperature than the previous ones due to the fact that the atmospheric boiling point of Na is approximately 1600°F as compared to 1450°F for the KNa 44. In this case, due to the low corrosion rate, the 100 hour test was omitted and only the 500 hour test run.

Macroscopically, both the liquid and vapor phase samples had a dull grey appearance with no evidence of pitting or deposition. This unit leaked and, consequently, was undoubtedly over-heated.

The micro-structure of the blank is shown in Fig. 26. Fig. 27 shows the structure of the vapor sample and the diffusion attack on the steel. The same condition is shown in Fig. 28 except that the sample was etched to show that the attack was intergranular. These diffusion products were identified by X-ray diffraction methods as Na_2O. This diffusion penetrated to a depth of 0.015 - 0.025".

Vickers hardness tests were run on the vapor sample, one measurement being taken on the portion where the diffusion occurred, and another taken on the core of the metal. These were, respectively, VHN 430-435 and VHN 175.

A ductility measurement by flattening was run on the vapor sample. Splitting occurred with a distance of 7/16" between walls. This embrittlement was, doubtless, due to the deep penetration of the diffusion product Na_2O.

7. Type 304 vs. K. The temperature at which this test was run was somewhat lower than previous ones, the boiling point of potassium being 1400° F.

The parts of the apparatus and test specimens had a dull grey appearance and there was no evidence of any corrosive action or deposition.

Micro-examination indicated that both liquid and vapor samples suffered diffusion attack similar in appearance to that noted in the previous test. This diffusive action is clearly shown in Fig. 29. In this case the diffusion products could not be identified by X-ray diffraction. The depth of the attack was somewhat less than in the case of the sodium, penetration occurring to a depth of only 0.002 - 0.005".

Vickers hardness tests on the vapor sample gave values approximately the same as in the previous tests for the core of the metal; VHN - 175. Hardness values of the case, VHN 276, were somewhat lower than those on the case of the sodium test due to the lighter penetration of the diffusion products. Ductility of the vapor sample was measured by flattening test giving a value of 1/4".

8. SAE 1020 Steel vs. Na. This 1600°F test was run for 500 hours with macro-examination showing corrosion and deposition similar to, but somewhat less than, that observed in the wrought iron tests. The liquid sample was quite smooth indicating that the corrosion was uniform and not selective. No pitting or deposition was evident on this sample. The vapor phase sample showed a deposit of an unidentified crystalline substance on the lower 1½" of the I.D. and O.D. A similar deposit occurred on all exposed surfaces of the remainder of the apparatus at and immediately above the liquid level.

Fig. 30 shows the microstructure of the blank; Fig. 31 that of the liquid sample, and Fig. 32 that of the vapor phase sample.

Extreme grain growth along with almost complete decarburization had taken place; no diffusion attack was noticed. The extreme softness of the specimens made it impossible to properly prepare them for microstructure examination.

CONCLUSIONS

Recommendations as to choice of materials for high temperature systems utilizing Na, K, or KNa cannot be made until further tests, now in progress, are concluded.

TABLE I

	Materials	Time hrs.	Phase	Alkali Metal	Penetration mg/cm^2/hr	Penetration in/yr
1.	Wrought Iron	100	L	KNa-44	-0.1307	-0.058
	"	"	V	"	deposit	
	"	500	L	"	-0.2018	-0.090
	"	"	V	"	deposit	
2.	Type 304	100	L	"	+0.0810	+0.035
	"	"	V	"	+0.0031	+0.001
	"	500	L	"	-0.0029	-0.001
	"	"	V	"	+0.0053	+0.002
3.	Nickel A	100	L	"	Nil	Nil
	"	"	V	"	"	"
	"	500	L	"	-0.0054	-0.002
	"	"	V	"	+0.0068	+0.003
4.	Inconel	100	L	"	-0.0405	-0.016
	"	"	V	"	-0.0123	-0.005
	"	500	L	"	+0.0108	+0.004
	"	"	V	"	-0.0006	Nil
5.	Type 310	100	L	"	Nil	Nil
	"	"	V	"	+0.0080	+0.003
	"	500	L	"	+0.0027	+0.001
	"	"	V	"	+0.0025	+0.001
6.	Type 304	500	L	Na	+0.0041	+0.002
	"	"	V	"	+0.0034	+0.001
7.	Type 304	500	L	K	+0.0070	+0.003
	"	"	V	"	+0.0013	+0.001
8.	SAE 1020	500	L	Na	-0.0213	-0.009
	"	"	V	"	+0.0258	+0.011

Penetrations of less than 0.0005 in/yr considered "nil".

TABLE OF ILLUSTRATIONS

FIG. 1 P-2363 Samples of distilling retort as taken.
2 P-2367 Condenser tube showing black streak (carburized area) on I.D.
3 P-2362 Hardness distribution on slice from kettle section.
4 8654 General structure of condenser tube.
5 8655 Grain boundary precipitation of carbides in condenser tube.
6 8658 Intergranular attack on O.D. of condenser tube.
7 8659 Carburized case 0.004-0.006" adjacent to black streak on tube.
8 8656 Pearlitic type carburization on bore of tube containing welds.
9 8657 Interface of weld metal and tube.
10 8671 General structure of kettle section.
11 8685 Junction of weld and parent metal of distilling kettle
12 Sketch Corrosion test apparatus
13 101 Corrosion test.
14 P-2398 Wrought iron 100 hour samples and apparatus
15 P-2400 Wrought iron 500 hour samples and apparatus
16 P-2414 Exaggerated grain growth on boiler and nitrogen tubes.
17 8847 Representative structure of wrought iron blank.
18 8846 Structure at top of boiler.
19 8845 Structure representing lower portion of boiler and nitrogen tube.
20 9075 Type 304 blank showing strained condition.
21 9069 Unidentified constituent found in several Type 304 specimens.
22 9076 Original structure of the nickel.
23 9074 Original structure of the inconel.
24 9073 Type 310 blank showing strained condition.
25 9085 Sigma phase and carbides in Type 310.
26 0066 Type 304 blank.
27 0013 Type 304 - structure of Na vapor sample showing diffusion attack.
28 0122 Same as Fig. 27 but etched to show intergranular attack.
29 0065 Type 304 - structure of K vapor sample showing diffusion attack.
30 0064 SAE 1020 blank.
31 0063 SAE 1020 Na liquid sample.
32 0067 SAE 1020 Na vapor sample.

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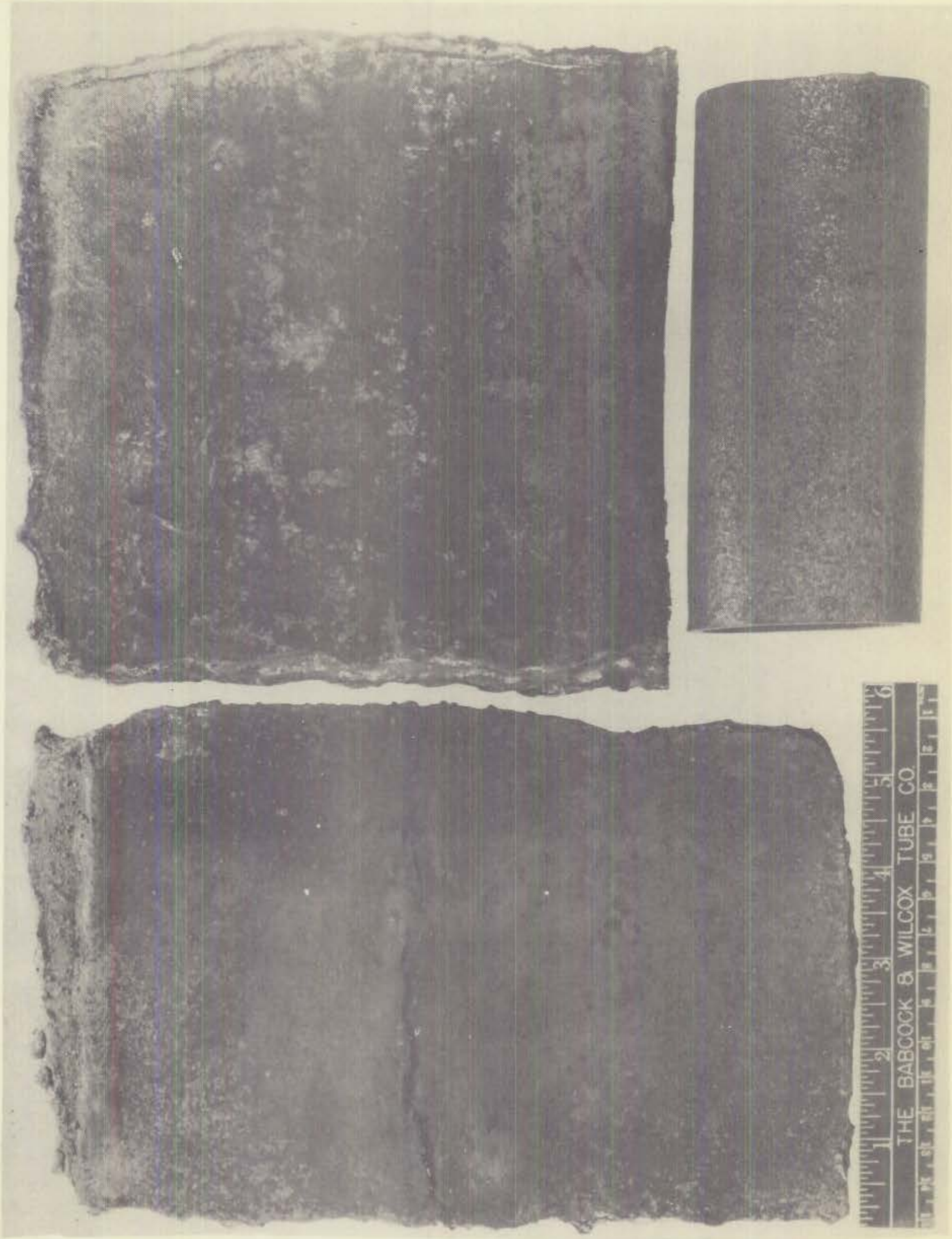

FIGURE 1 (P-2363)

SAMPLES OF DISTILLING
RETORT AS TAKEN.

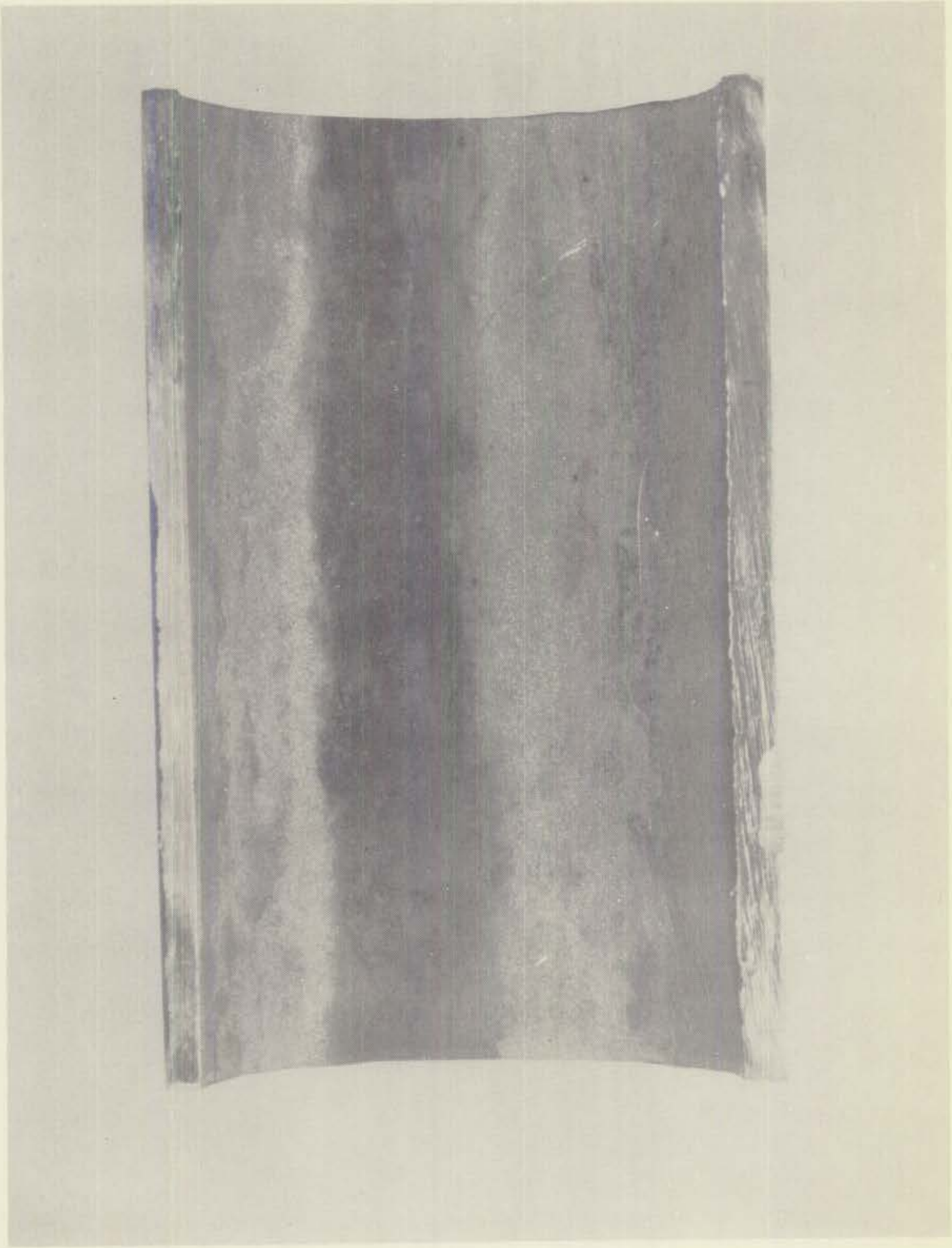

FIGURE 2 (P-2367)

CONDENSER TUBE SHOWING BLACK STREAK (CARBURIZED AREA) ON I. D.

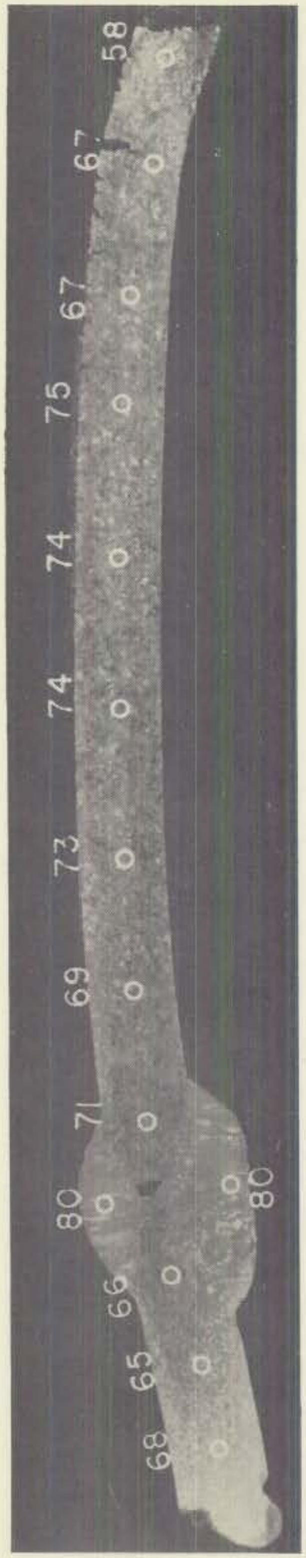

FIGURE 3 (P-2362)

ROCKWELL B HARDNESS DISTRIBUTION ON SLICE CUT FROM DISTILLING KETTLE SECTION.

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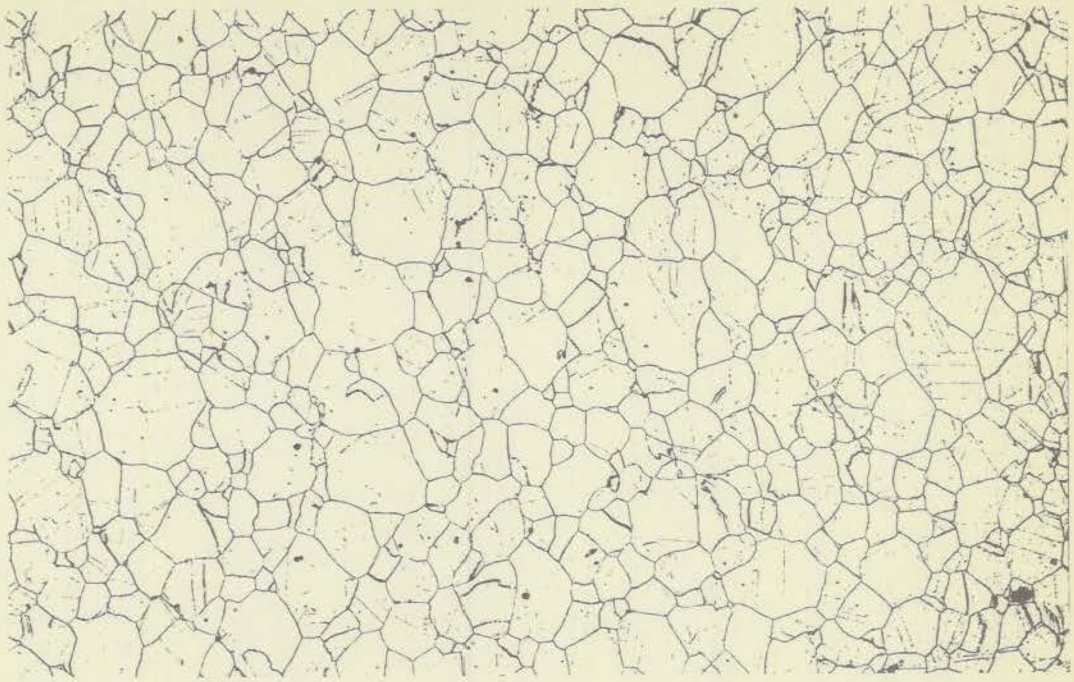
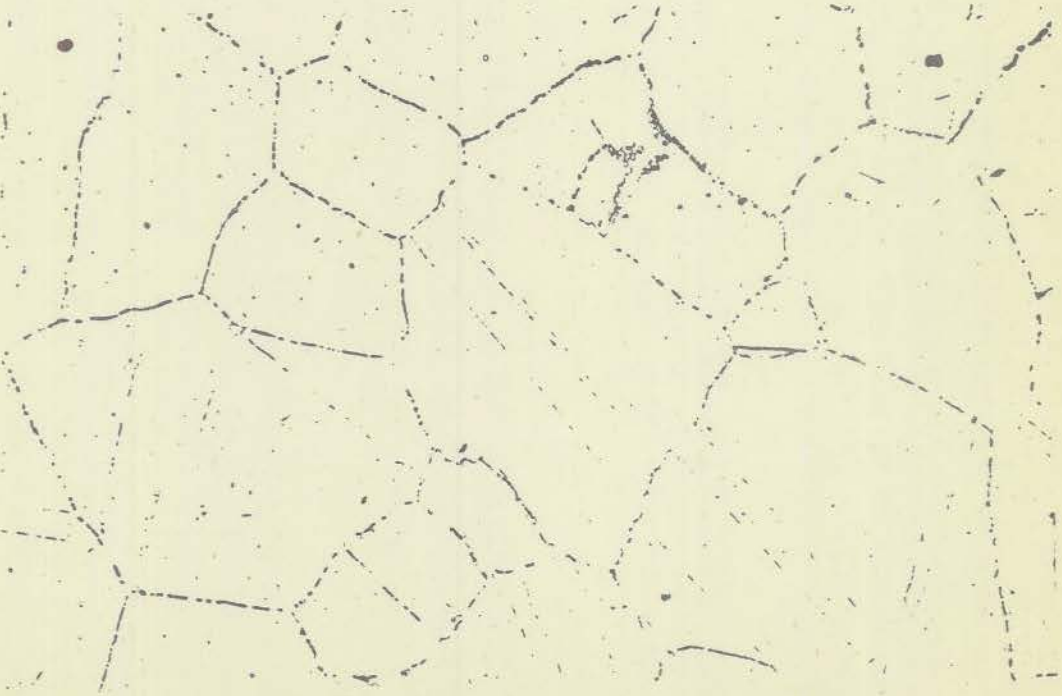


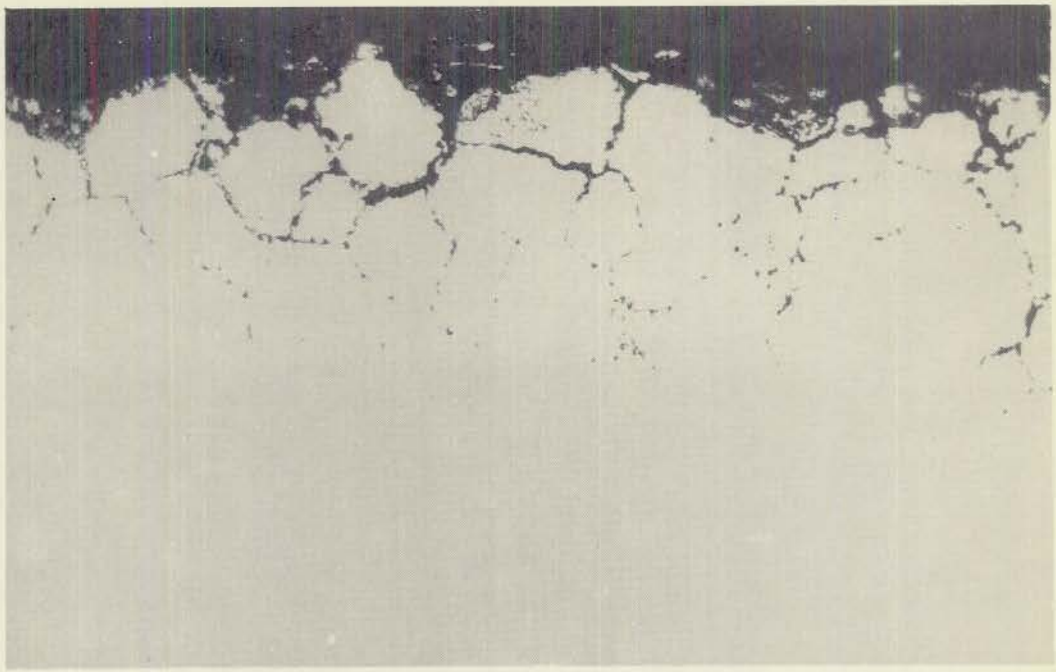
X 100 ETCHANT: ELECTROLYTIC OXALIC ACID

FIGURE 4 (8654) GENERAL STRUCTURE OF CONDENSER TUBE.



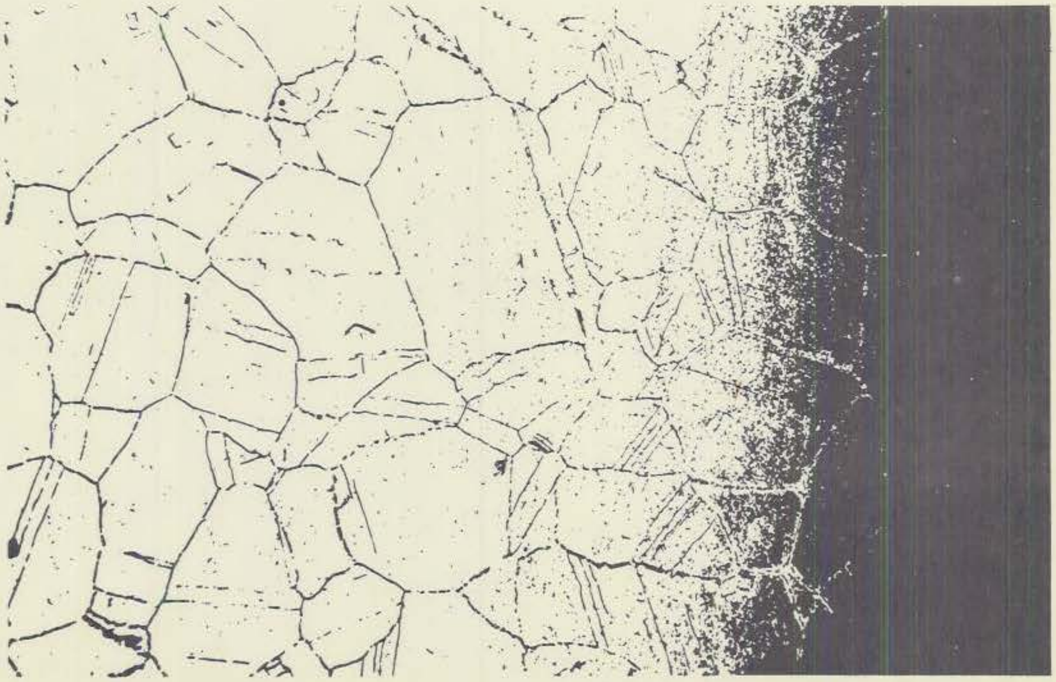
X 500 ETCHANT: ELECTROLYTIC OXALIC ACID

FIGURE 5 (8655) NOTE GRAIN BOUNDARY PRECIPITATION OF CARBIDES IN CONDENSER TUBE.



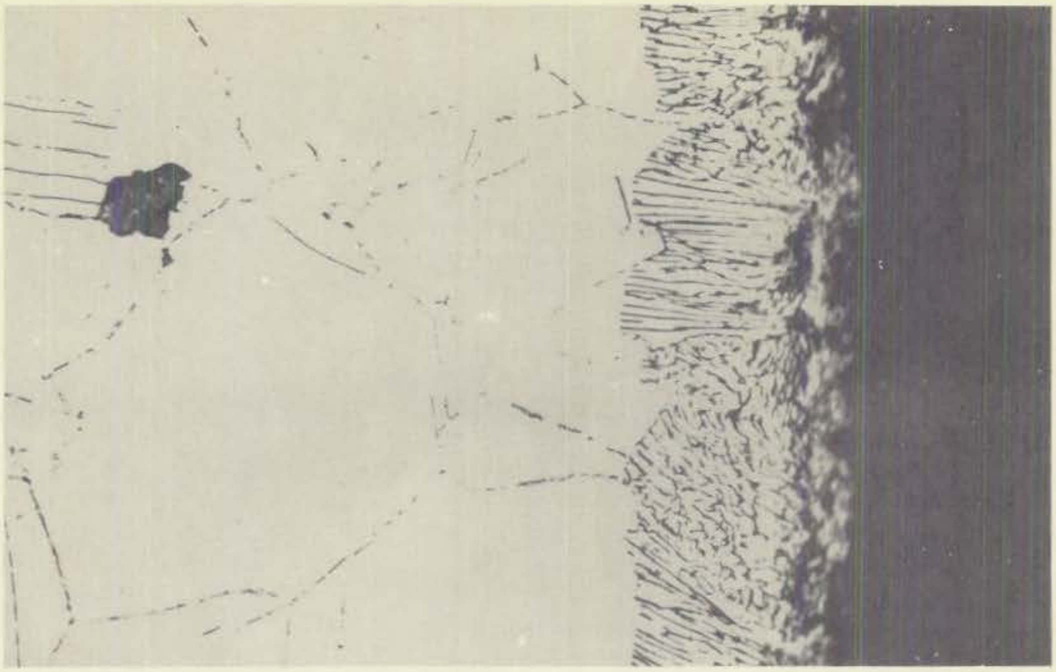
X 250 UNETCHED

FIGURE 6 (8658) INTERGRANULAR ATTACK ON O.D. OF CONDENSER TUBE.



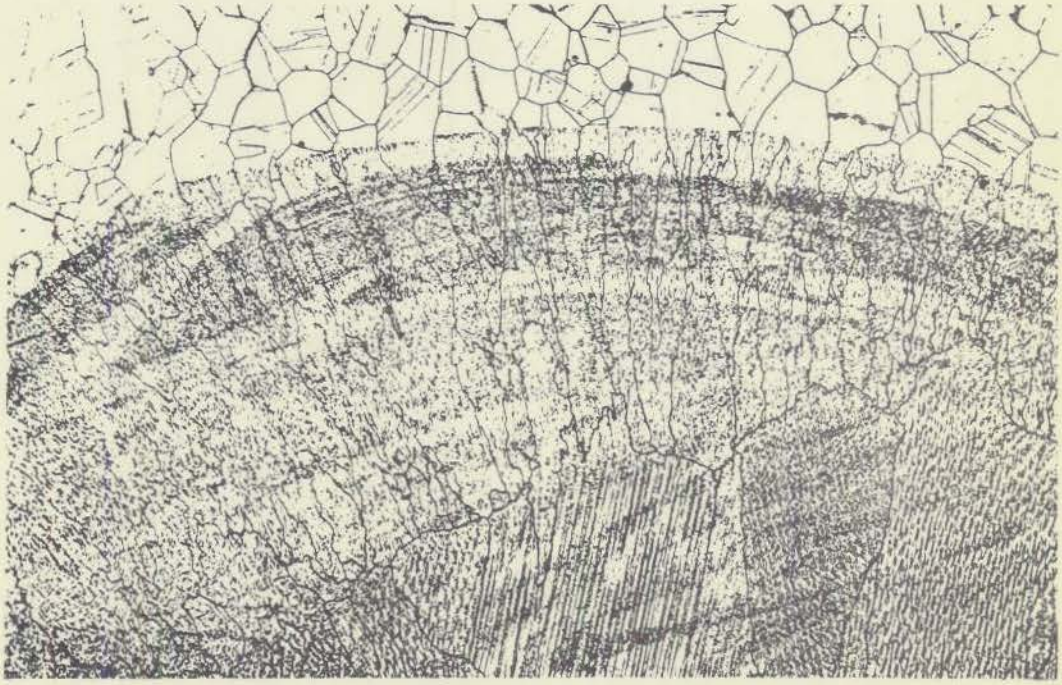
X 250 ETCHANT: ELECTROLYTIC OXALIC ACID

FIGURE 7 (8659) CARBURIZED CASE .004-.006" FOUND ADJACENT TO BLACK STREAK ON I.D. OF TUBE.



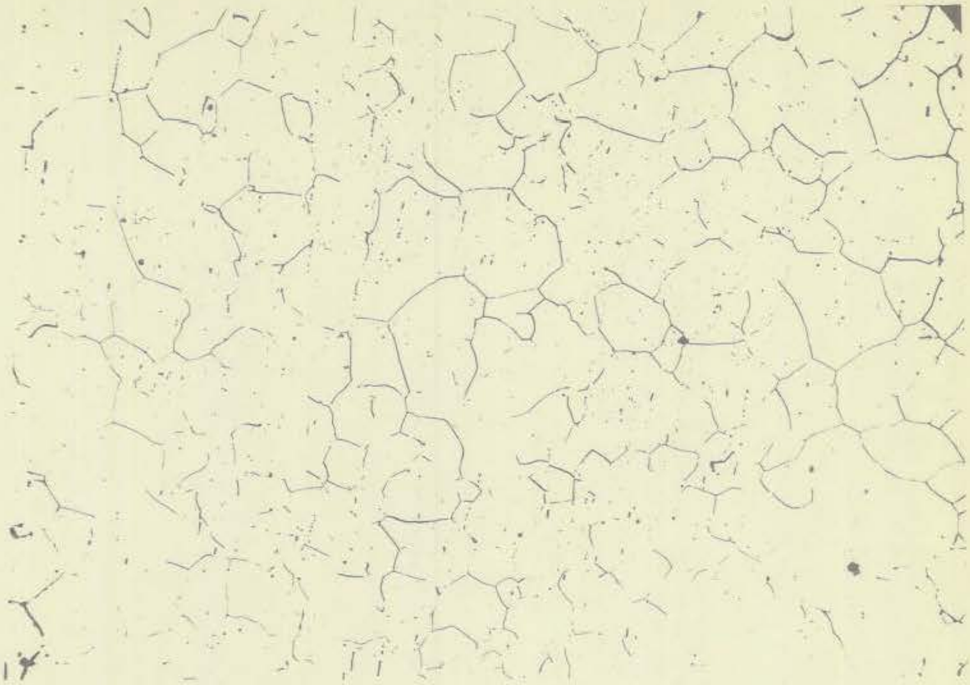
X 500 ETCHANT: ELECTROLYTIC OXALIC ACID

FIGURE 8 (8656) PEARLITIC TYPE CARBURIZATION FOUND ONLY ON BORE OF TUBE CONTAINING WELDS.



X 100 ETCHANT: ELECTROLYTIC OXALIC ACID

FIGURE 9 (8657) INTERFACE OF WELD METAL AND TUBE.

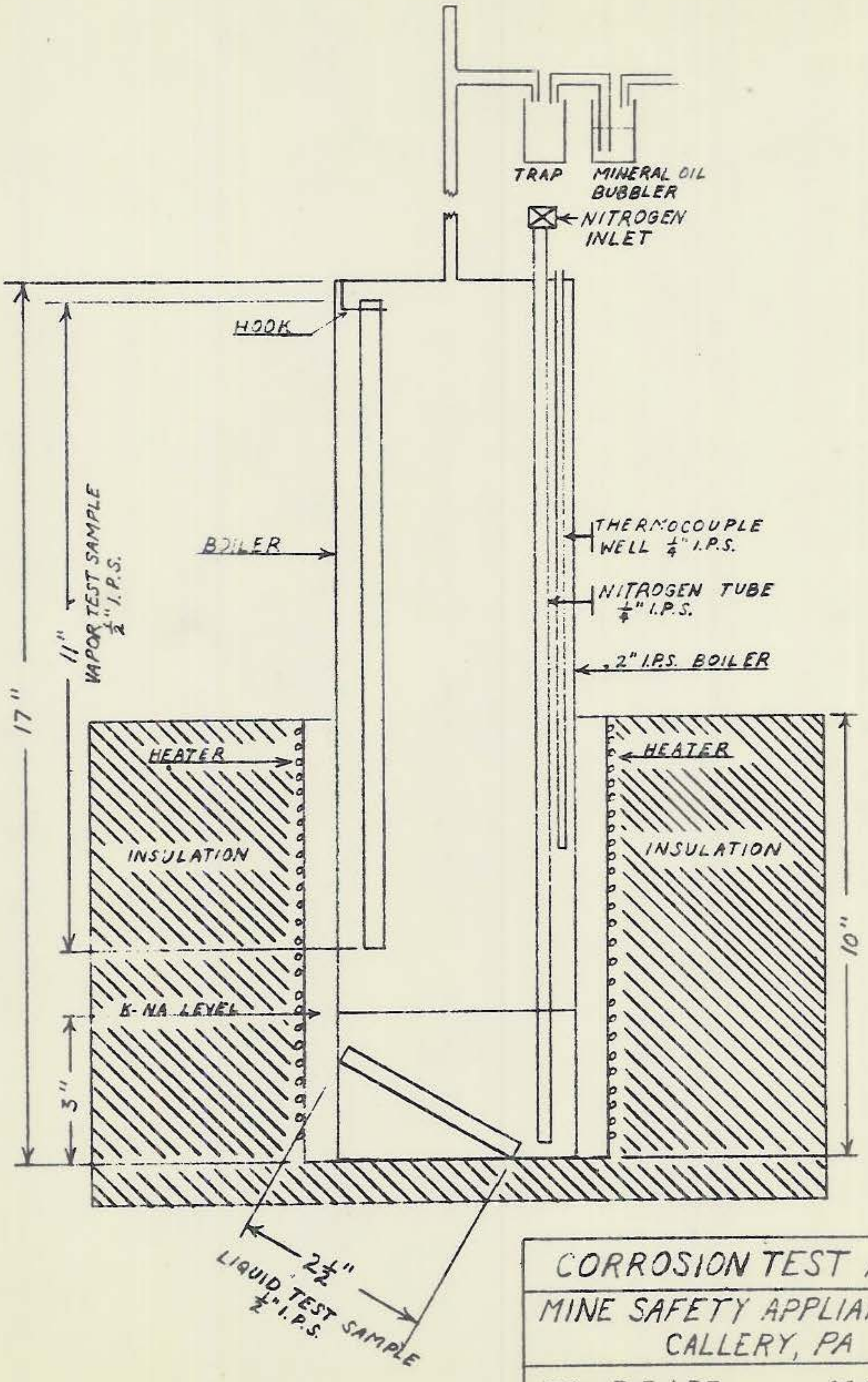


X 50 ETCHANT: ELECTROLYTIC OXALIC ACID

FIGURE 10 (8671) GENERAL STRUCTURE OF KETTLE SECTION

X 50 ETCHANT: ELECTROLYTIC OXALIC ACID

FIGURE 11 (8685) JUNCTION OF WELD AND PARENT METAL OF DISTILLING KETTLE.



CORROSION TEST APPARATUS

MINE SAFETY APPLIANCES CO.
CALLERY, PA

DR.- R.E. LEE
10-1-47

SCALE-NONE
FIG.-12

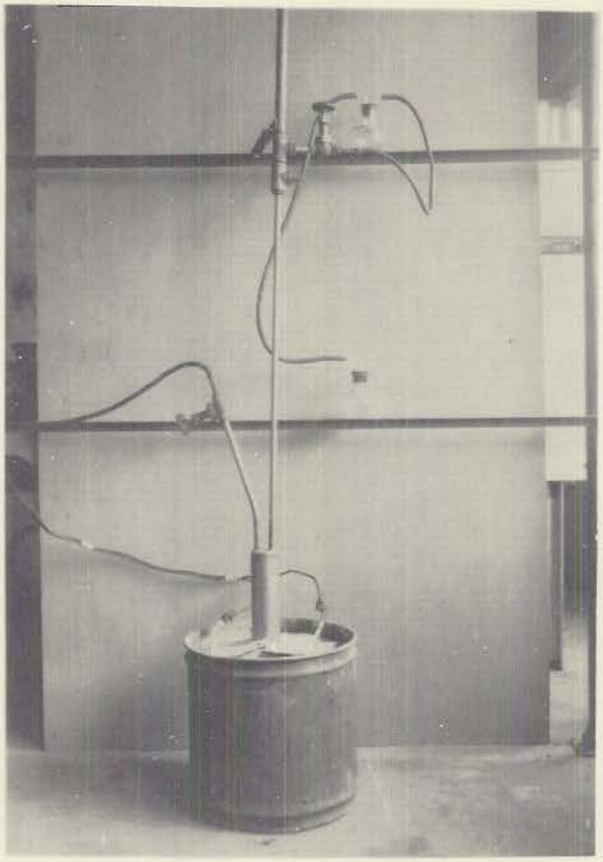


FIGURE 13 (101) CORROSION TEST

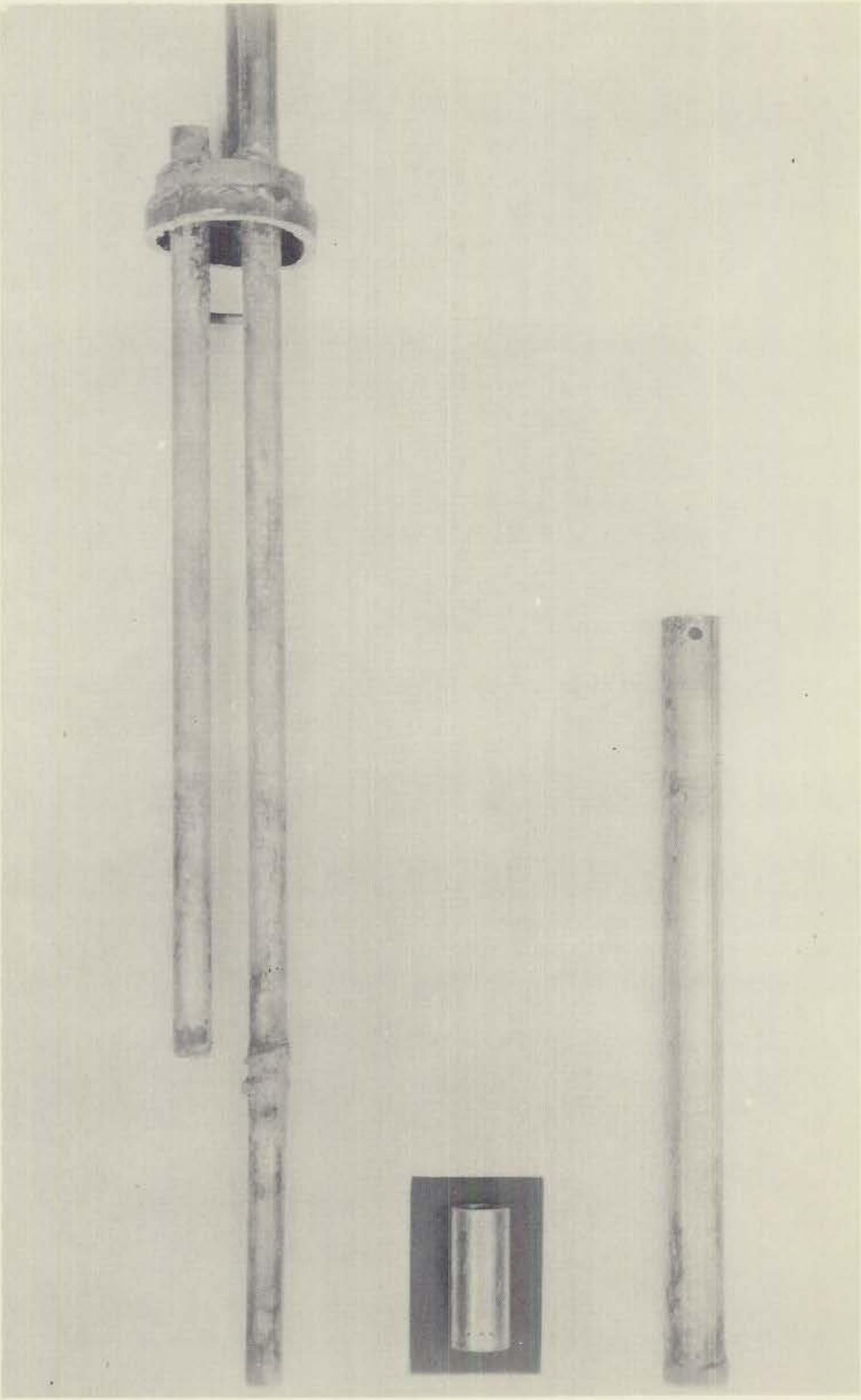


FIGURE 14 (P-2398)

WROUGHT IRON 100 HOUR SAMPLES AND APPARATUS.

UNCLASSIFIED

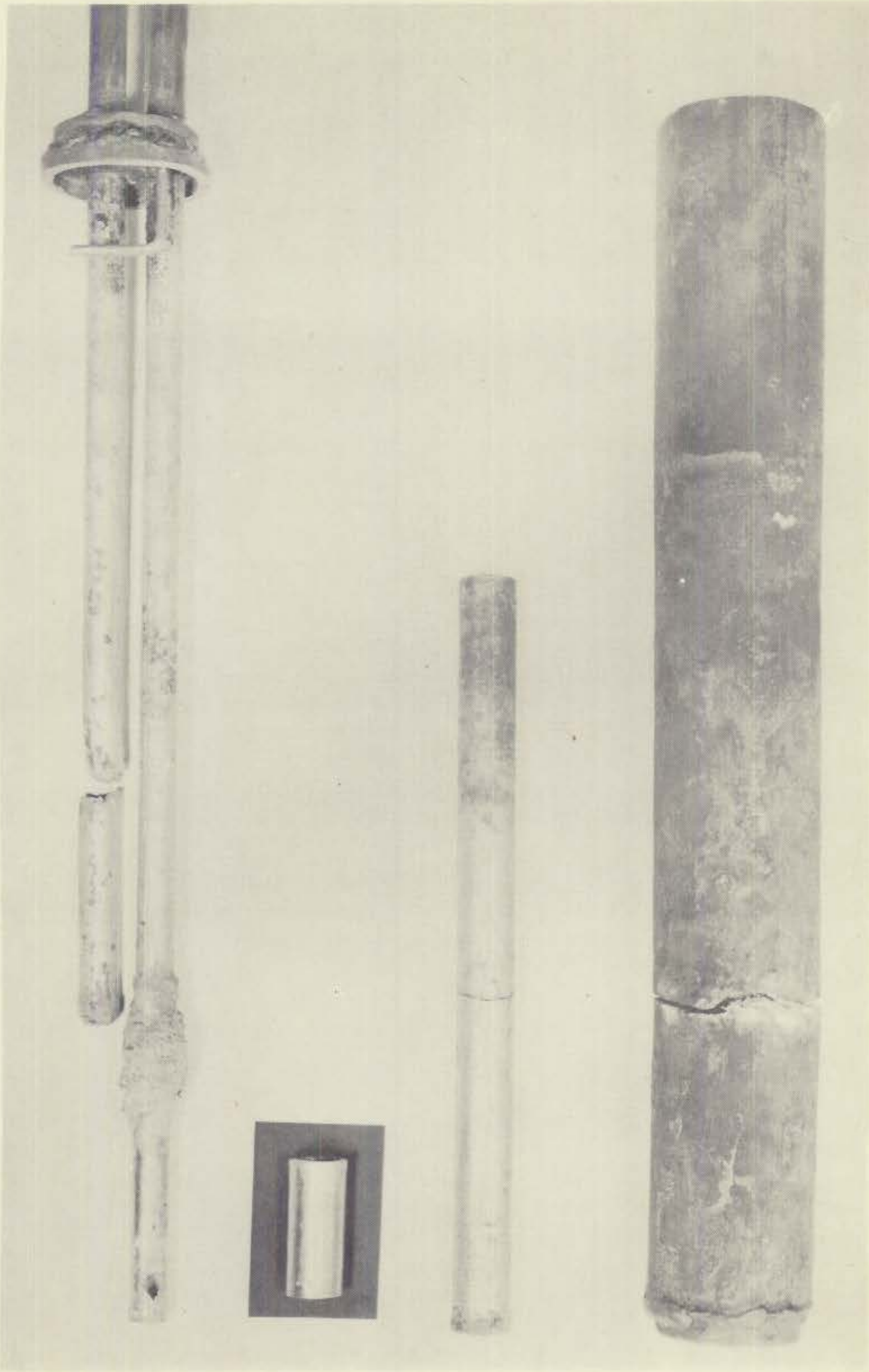


FIGURE 15 (P-2400)

WROUGHT IRON 500 HOUR SAMPLES
AND APPARATUS.

FIGURE 16 (P-2414)

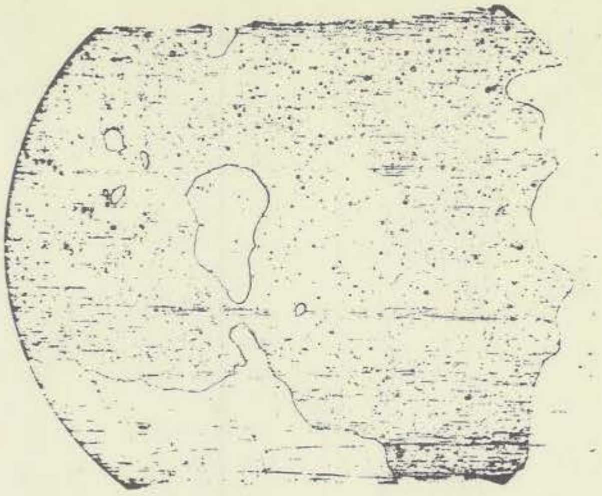
EXAGGERATED GRAIN GROWTH ON BOTTOM PORTION OF RETORT AND NITROGEN TUBES IN WROUGHT IRON TEST.

X 100 ETCHANT: NITAL

FIGURE 17 (8847) REPRESENTATIVE STRUCTURE OF WROUGHT IRON BLANK.

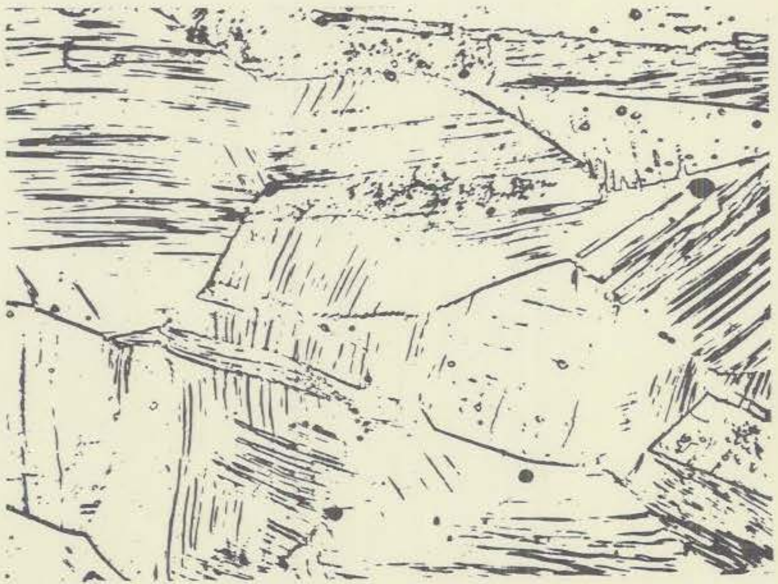
X 100 ETCHANT: NITAL

FIGURE 18 (8846) STRUCTURE AT TOP OF BOILER WHICH IS IDENTICAL WITH TOP OF N_2 TUBE.



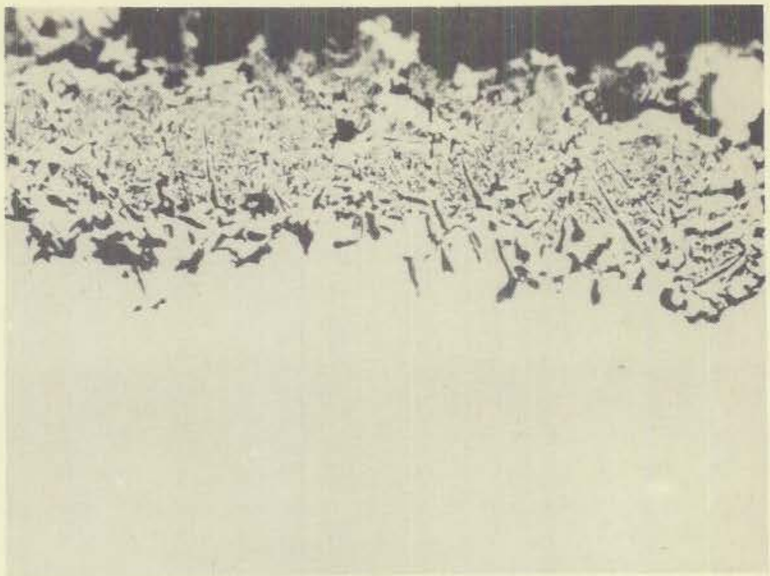
X 25 ETCHANT: NITAL

FIGURE 19 (8845) STRUCTURE REPRESENTING LOWER PORTION OF BOILER AND NITROGEN TUBE.



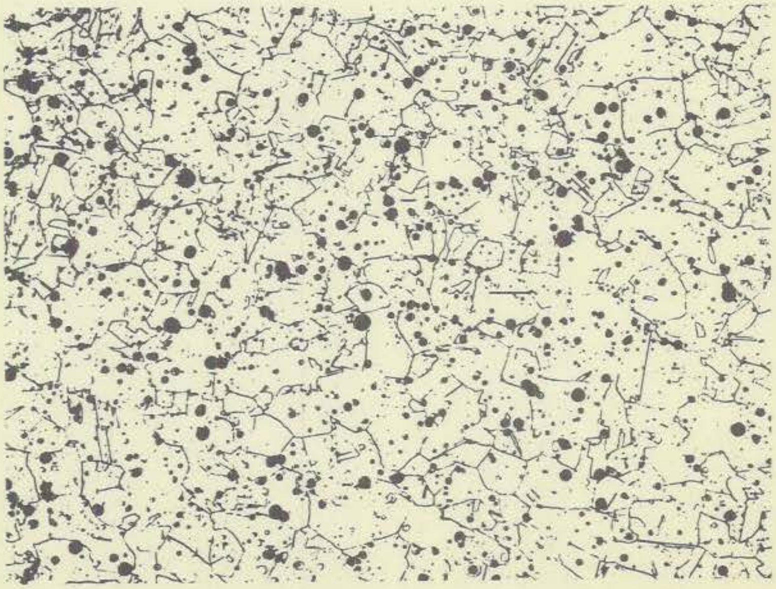
X 250 ETCHANT: OXALIC ACID

FIGURE 20 (9075) TYPE 304 BLANK SHOWING STRAINED CONDITION.



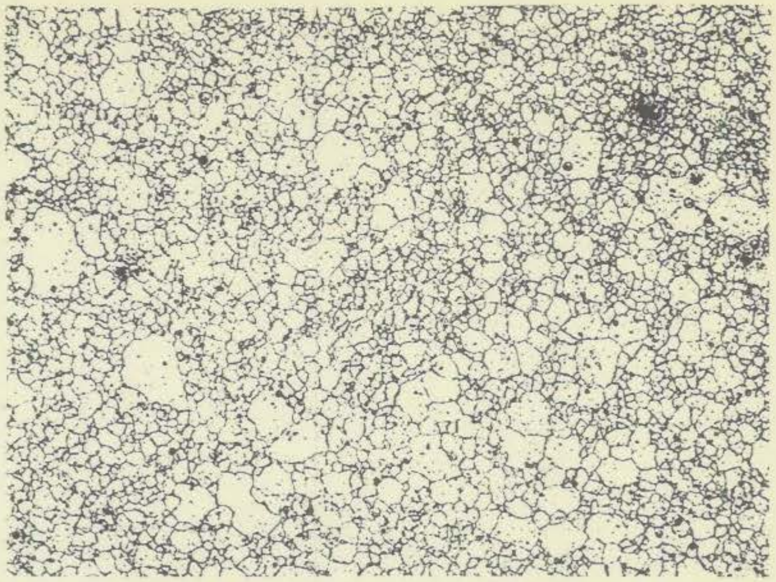
X 500 UNETCHED

FIGURE 21 (9069) UNIDENTIFIED CONSTITUENT FOUND IN SEVERAL TYPE 304 SPECIMENS.



X 100 ETCHANT: ACETIC + NITRIC ACID

FIGURE 22 (9076) ORIGINAL STRUCTURE OF THE NICKEL. GRAIN SIZE 4-6.



X 100 ETCHANT: ACETIC + NITRIC ACID

FIGURE 23 (9074) ORIGINAL STRUCTURE OF THE INCONEL. GRAIN SIZE 7-8.

X 100 ETCHANT: OXALIC ACID

FIGURE 24 (9073) TYPE 310 BLANK SHOWING STRAINED CONDITION.

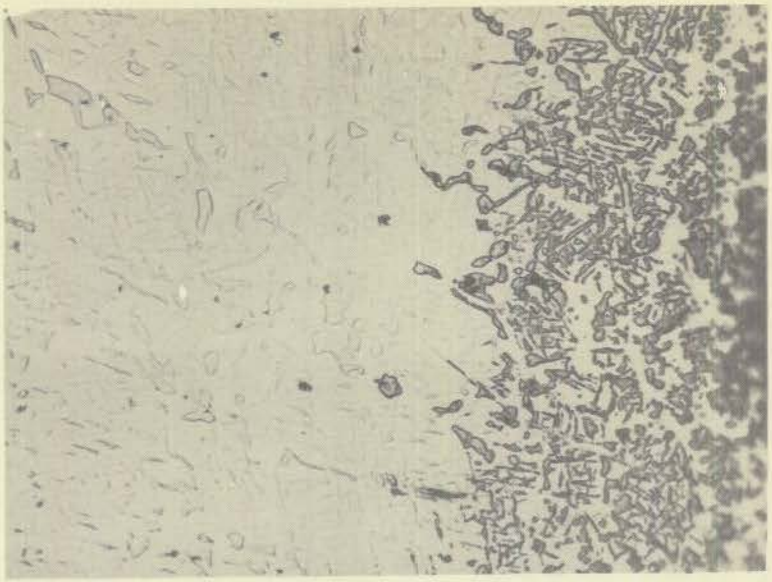
UNCLASSIFIED

X 250 ETCHANT: AQUA REGIA

FIGURE 25 (9085) SIGMA PHASE AND CARBIDES IN TYPE 310.

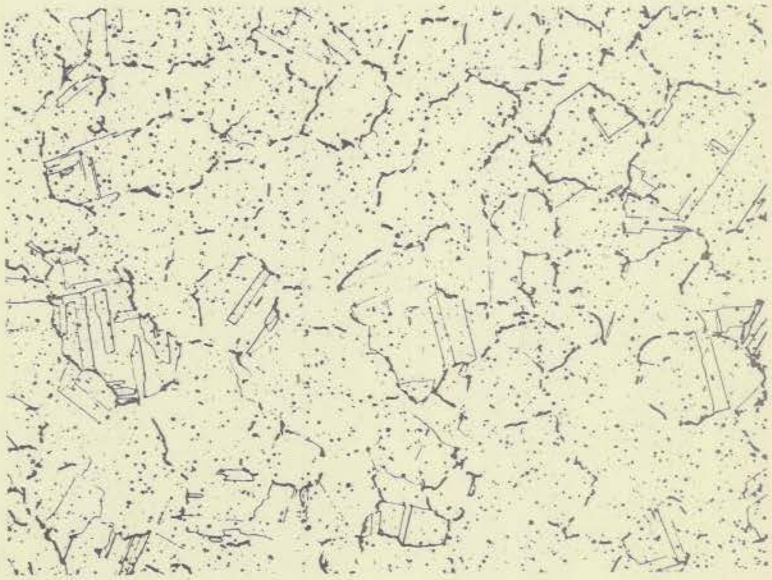
X 100 AQUA REGIA

FIGURE 26 (0066) TYPE 304 BLANK.



X 500 UNETCHED

FIGURE 27 (0013) TYPE 304 - STRUCTURE OF Na. VAPOR SAMPLE SHOWING DIFFUSION ATTACK.

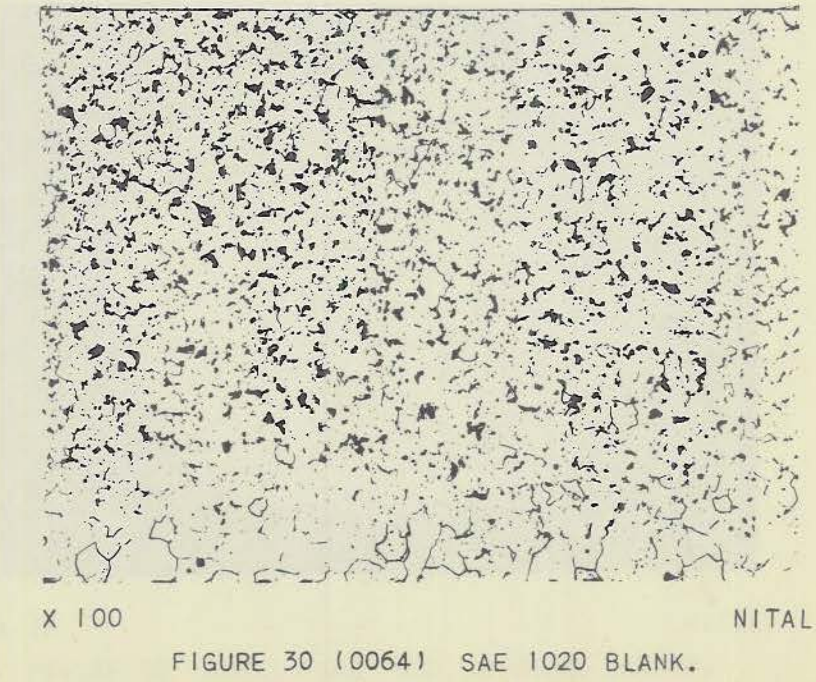


X 100 CHROMIC ACID

FIGURE 28 (0122) SAME AS FIG. 27 BUT ETCHED TO SHOW INTERGRANULAR ATTACK BY Na_2O.

X 500 AQUA REGIA

FIGURE 29 (0065) TYPE 304 - STRUCTURE OF K VAPOR SAMPLE SHOWING DIFFUSION ATTACK.



X 100 NITAL

FIGURE 30 (0064) SAE 1020 BLANK.