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THE EFFECT OF A STATIC ELECTRIC FIELD ON THE OXIDATION OF CERTAIN METALS

G. WM. LAWLESS

C. A. LOMBARD

TECHNICAL REPORT AFML-TR-65-412

FEBRUARY 1966

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FOREWORD

This report was prepared by the Physical Metallurgy Branch, Metals and Ceramics Division, Air Force Materials Laboratory. It was initiated under Project No. 7312 "Metal Surface Deterioration and Protection," Task No. 731202 "Metal Surface Deterioration," and administered by the Air Force Materials Laboratory, Research and Technology Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, G. Wm. Lawless and Carl A. Lombard, Project Engineers.

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The authors express their appreciation to Messrs. O. O. Srp and P. L. Faust for their guidance.

This technical report has been reviewed and is approved.



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ABSTRACT

The effects of an externally impressed, static electric field on the oxidation behavior of nickel, type 304 stainless steel, and molybdenum were investigated. While no effect was found for nickel, the oxidation rates of the stainless steel and molybdenum were markedly suppressed by the electric field within the range of field strengths investigated. Significantly, the suppressive effect was found to occur regardless of the direction of the electric field. In general, the theory of K. Hauffe on the "Effect of Electric Field on the Oxidation of Metals" was supported by the experimental results.

27 p 20 ref 17 fig

(20 pp) (17 fig.) (1 tbls.) (20 ref.)

TABLE OF CONTENTS

I.	INTRODUCTION	1
II.	THEORY	2
III.	SPECIMEN MATERIALS AND PRETREATMENT	4
IV.	EXPERIMENTATION	5
V.	EXPERIMENTAL RESULTS	9
VI.	DISCUSSION AND CONCLUSIONS	19
	REFERENCES	20

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ILLUSTRATIONS

FIGURE	PAGE
1. Concentration Gradient and Space Charge According to Hauffe and Ilschner	2
2. Schematic Representation of the Positions of Electron Levels in Metal According to Cabrera and Mott	2
3. Approximately Space-Charge-Free Oxide Layer According to Hauffe	3
4. Experimental Configuration of Microbalance	6
5. Specimen Configuration in the Platinum Electrode	7
6. Gas Train	8
7. DC Power Supply	8
8. 304 Stainless Steel Oxidized in an Electric Field at 1140°C (0 Volts, ±45 Volts)	10
9. 304 Stainless Steel Oxidized in an Electric Field at 1140°C (0 Volts, ±150 Volts)	11
10. 304 Stainless Steel Oxidized in an Electric Field at 1140°C (0 Volts, - 300 Volts)	12
11. 304 Stainless Steel Oxidized in an Electric Field at 1140°C (+150 Volts)	13
12. Comparison of Oxidation Data Without Electric Field 1140° - 1150 °C	14
13. Percent Weight Lost of Molybdenum vs Voltage at 700°C for One Hour	16
14. Comparison of Nickel Data at Approximately 1050°C	17
15. Nickel Oxidized in an Electric Field at 1095°C (0 Volts, ±49 Volts)	17
16. Nickel Oxidized in an Electric Field at 1043°C (0 Volts, ±195 Volts)	18
17. Schematic Representation of a Saddle Jump of Metal Ions	19

SECTION I

INTRODUCTION

High temperature metallic oxidation has assumed a prominent position in the current trend toward the use of engineering metals at higher and higher temperatures. A better understanding of the oxidation phenomenon is fundamental to the development of a means for preventing or mitigating its destructive effects. While weight gain versus time-at-temperature studies are essential, they often prove inadequate for a complete theoretical and kinetic analysis of the overall oxidation reaction. Hence new approaches which involve an external variable are desirable and worthy of investigation.

Current theory indicates that investigative techniques based upon the electrical characteristics of oxide film formation should prove useful for interpretation of metal oxidation phenomena. A consideration of basic corrosion precepts and the postulation that ionic diffusion through the oxide scale is the controlling factor, indicates that the presence of an external electric field should measurably affect the high temperature oxidation of metals. Furthermore, the effects of controlled variations and directions of the applied field should establish a clearer concept of the oxidation mechanism.

As reasonable as this may seem, evidence is available to indicate that external electric fields have no effect on metallic oxidation. Cismaru and Cismaru (Reference 1), studying zinc at 400°C, reported no change in the oxidation rate with an applied potential. Uhlig and Brenner (Reference 2), working with copper, also reported no change in the rate of oxidation with an impressed electric field. However, a point of difference arises in that both these investigations utilized a parallel plate type capacitor arrangement with air or oxygen as the dielectric. Jorgensen (Reference 3), who has done work on silicon and zinc, claimed that the greatest share of the potential drop is across dielectric, and little or no field effect is felt across the specimen material, particularly across the

metal/oxide interface. His results on zinc, using a direct contact type of arrangement, differ from those of Cismaru and Cismaru, and his earlier work on silicon also demonstrates that orientation and strength of the applied field can have a direct inhibiting effect on the rate of metallic oxidation (Reference 4). Schein, Le Boucher and Lacombe (Reference 5) report similar effects on their investigation on the oxidation of iron in an electric field. Kramer (Reference 6) studied molybdenum, and his results, later confirmed in the Physical Metallurgy Branch, Air Force Materials Laboratory (MAMP), (Reference 7), support the work of Jorgensen and Schein, et al. Recent efforts in the Air Force Flight Dynamics Laboratory, Wright-Patterson Air Force Base, Ohio (References 8 and 9) on 347 stainless steel and titanium showed a significant reduction in the oxidation rate of the stainless steel when under the influence of a static electric field; the results on titanium were inconclusive.

Based on the above work, and stimulated by the need for new techniques to study oxidation mechanisms, an effort to investigate more thoroughly the effect, if any, of static electric fields on high temperature metal oxidation was undertaken. Nickel, molybdenum and type 304 stainless steel were selected for study as representing a fair cross-section of metals of prime current interest to the Air Force, and as having some comparative data available in the literature. Caplan and Cohen (Reference 10), Radavich (Reference 11) and Gulbransen and Andrew (Reference 12) have published conventional oxidation data on stainless steels, with the latter reporting results on type 304. Gulbransen and Andrew (Reference 13), Sartell and Li (Reference 14), and Lombard (Reference 15) have reported conventional nickel oxidation data, and results on molybdenum oxidation are obtainable from the work of Jones, et al. (Reference 16) and Gulbransen, et al. (Reference 17). These available references on oxidation data served as good experimental check points.

SECTION II THEORY

A detailed discussion on the theory of the effect of electric fields on metal oxidation is not included in this report since most of the theory generated on this subject can be found in two books - Van Bueren's Imperfections in Crystals, (Reference 18), and Karl Hauffe's Oxidation of Metals, (Reference 19). Both books do an excellent job of discussing the theory and in referencing other reports dealing with electric field effects. Some of the major points of the theory as discussed by Karl Hauffe are presented here.

What is considered to be the generally valid variation of the defect concentration as it occurs during oxidation is given in Figure 1. (Reference 19). Figure 1 shows the local variation of the concentration of the ions in interstitial lattice positions in an n type oxide layer with a homogeneous electrochemical field according to Hauffe and Ilschner's concentration gradient and space charge.

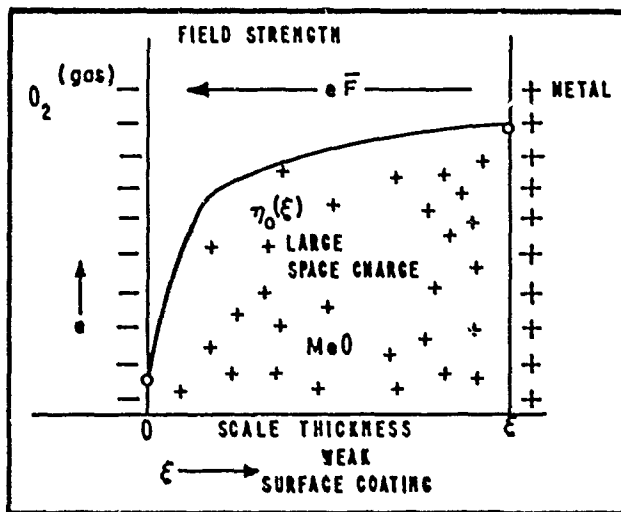


Figure 1. Concentration Gradient and Space Charge According to Hauffe and Ilschner

Figure 2 is a schematic representation of the positions of electron levels in the metal, oxide, and adsorbed or chemisorbed oxygen, according to Cabrera and Mott (Reference 20). Figure 2a represents the electron levels in the metal, in the oxide, and in the adsorbed oxygen before the electron transfer. Figure 2b qualitatively shows the variation of the energy terms in the stationary state after setting up an electron equilibrium without including transport processes for ions.

As shown for the tarnishing system seen in Figure 2 electrons leave the metal as a result of thermal emission and enter the oxide layer, migrating toward the outer boundary layer to the energetically more favorable chemisorbed oxygen until the homogeneous field thus set up produces a stationary equilibrium (Figure 2a). Since supposedly no significant space charges are present, an equal number of charges should be found on both sides of the oxide layer--negative exterior and positive interior (simplified model of a condenser, see Figure 3) (Reference 19).

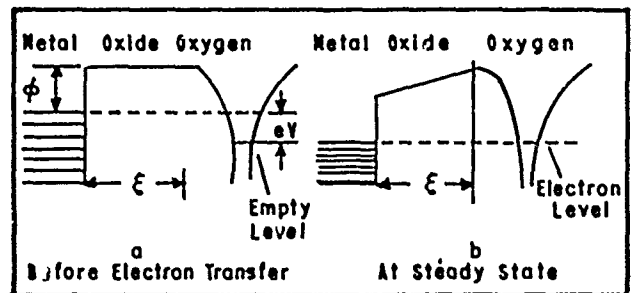


Figure 2. Schematic Representation of the Positions of Electron Levels in Metal According to Cabrera and Mott

Figure 3 depicts an approximately space-charge-free oxide layer with only surface charges (analogous to a condenser) formed during oxidation. $\bar{F} = V/\xi$, where V represents the potential difference between the charges at the interfaces, which is determined by the energy-level difference.

The potential difference V prevailing between the phase boundaries is just equal to the difference between the two energy terms for the metal and the chemisorbed oxygen, and these in turn are determined by the quantity

of charge. The theory generated by Hauffe, Cabrera and Mott and others might not agree on minor details, but does however agree that an electric field should affect the rate of oxidation.

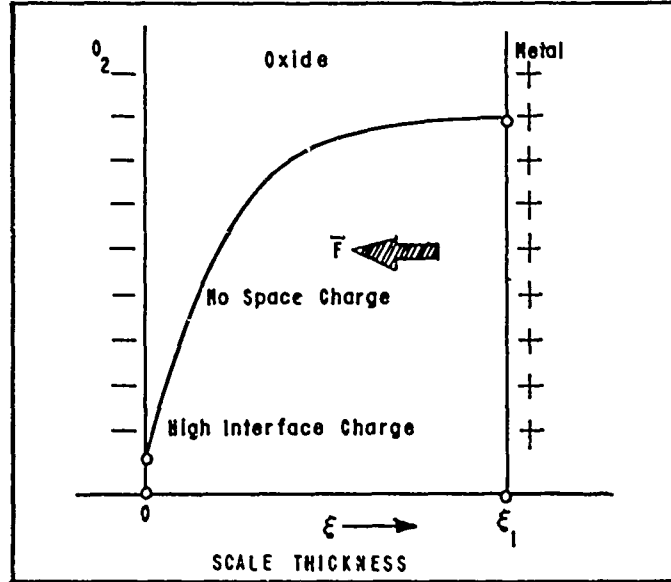


Figure 3. Approximately Space-Charge-Free Oxide Layer According to Hauffe

SECTION III

SPECIMEN MATERIALS AND PRETREATMENT

NICKEL

Spectrographic standard nickel rod (Johnson and Matthey), 0.5 cm in diameter and 1.9 cm long were used in the investigation. Spectrographic analysis of this material was as follows:

Fe less than 5 ppm
 Si less than 3 ppm
 Al less than 2 ppm
 Ca less than 1 ppm
 Mg less than 1 ppm
 Cu less than 1 ppm
 Ag less than 1 ppm

Surface preparation was performed immediately before use and consisted successively of light abrasion through No. 600 grit paper, an acetone soak, a one minute electropolish in 1 part H_2SO_4 /7 parts ethanol, distilled water soak, another one minute electropolish in 10 percent HCl in ethanol, a distilled water rinse, and soaking in warm ethanol.

MOLYBDENUM

Ground molybdenum rod, 0.318 cm in diameter and 3.81 cm long obtained from

Sylvania lot No. Mo-126 was used. One end of the specimens was tapered 60° for geometric and electrical accommodations. The taper allowed for mounting in platinum coated 1/8 in. hollow Al_2O_3 rods. The platinized alumina rods eliminated extraneous electric fields and their effects on the molybdenum specimens. Pretreatment of the molybdenum specimens consisted of solvent cleaning.

STAINLESS STEEL

A commercial type 304 stainless steel rod, 0.798 cm in diameter and 3.81 cm long was used. Analysis of this specimen material was as follows:

0.35 percent Si	9.42 percent Ni
1.67 percent Mn	19.42 percent Cr
0.02 percent S	0.30 percent Mo
0.03 percent C	0.17 percent Cu
0.03 percent V	0.06 percent Co
	Bal - Fe

Surface preparation consisted successively of light abrasion through No. 600 grit paper, a water rinse, a one minute etch in 2 percent HF/48 percent HNO_3 aqueous solution, a second water rinse and an acetone rinse.

SECTION IV

EXPERIMENTATION

NICKEL AND STAINLESS STEEL

The investigation of the nickel and stainless steel specimens was performed using a modified continuous weighing microbalance/double furnace assembly. For these two materials, the basic experimental procedure was to record the weight change of the specimens continuously while they were being subjected to constant conditions of temperature, air flow, and oxygen partial pressure, and an applied electric field. This was done to enable the authors to compare the results of this investigation with conventional oxidation data.

Figure 4 illustrates the experimental configuration used in this investigation. An insulated lead was passed through the air inlet valve of the balance, point A₁, and subsequently fastened to an electrically isolated stand on the base plate of the balance. Electrical connection from the stand to the platinum suspension wire was via a one mil, 24K gold, lower galvanometer suspension coil. This permitted free movement of the specimen suspension assembly without introducing extraneous vertical vector components. Thus electrical connection was achieved without distortion of balance readout. The entire arrangement was isolated from the balance and from electrical ground by a platinum tipped glass rod which replaced the left hand balance pan. This completed one leg of the electrical circuit. The other leg consisted of a platinum wire entering the assembly at the air outlet port of the furnace (point A₂, in Figure 4), and connected to a cylindrical platinum mesh screen which concentrically surrounded the specimen. The platinum screen and the specimen thus served as the two electrodes for the electric field. With this arrangement positive or negative fields could be conveniently applied to the specimen with assurance of uniformity and precision. Careful centering of the specimen, both axially and longitudinally, within the platinum electrode minimized inhomogeneities in the impressed electric field. The

electric field was applied normal to the surface of the specimen as shown in Figure 5. The cylindrical platinum electrode was 15 cm (L₂) long by 5 cm (D₂) in diameter.

The right-hand balance pan assembly was identical to the left-hand side with the exception of the electric field arrangement (Figure 4). A platinum counterweight on a platinum support wire was suspended in the second furnace. This arrangement minimized thermal fluctuations and buoyancy effects. It was found that the balance was precise to about twice its rated sensitivity, which was 0.1 mg. Geometric considerations and the possibility of electric field shorting made it impractical to place the specimen in a hot furnace.

The specimens were first placed into position (stainless steel or nickel) at room temperature. The furnace was raised into position and sealed, then a gas flow of two parts argon to one part hydrogen was introduced to prevent oxidation until the desired temperature was obtained (1140°C for stainless steel, 1040° and 1095°C for nickel). The time required to reach temperatures was about five hours. An electrical potential was then applied and a dried, metered air flow at 4 cu ft/hr was introduced to replace the Ar/H₂ mixture (Figure 6). Voltage potentials of ±45 volts, ±150 volts and -300 volts were applied before oxidation began. Previous work strongly indicated that the measures taken to insure complete electrical isolation and grounding were essential to the clear definition of the electric field and avoidance of extraneous electrical effects. The dc power supply arrangement (Figure 7) allowed for the variance of the voltage and the changing of direction of current flow and also permitted a continuous monitoring of the applied voltage and current. Temperature was measured with certified thermocouples, good to one degree at 1000°C, and continuously recorded. The temperature was controlled to within ±2°C of the desired temperature over a 24-hour period.

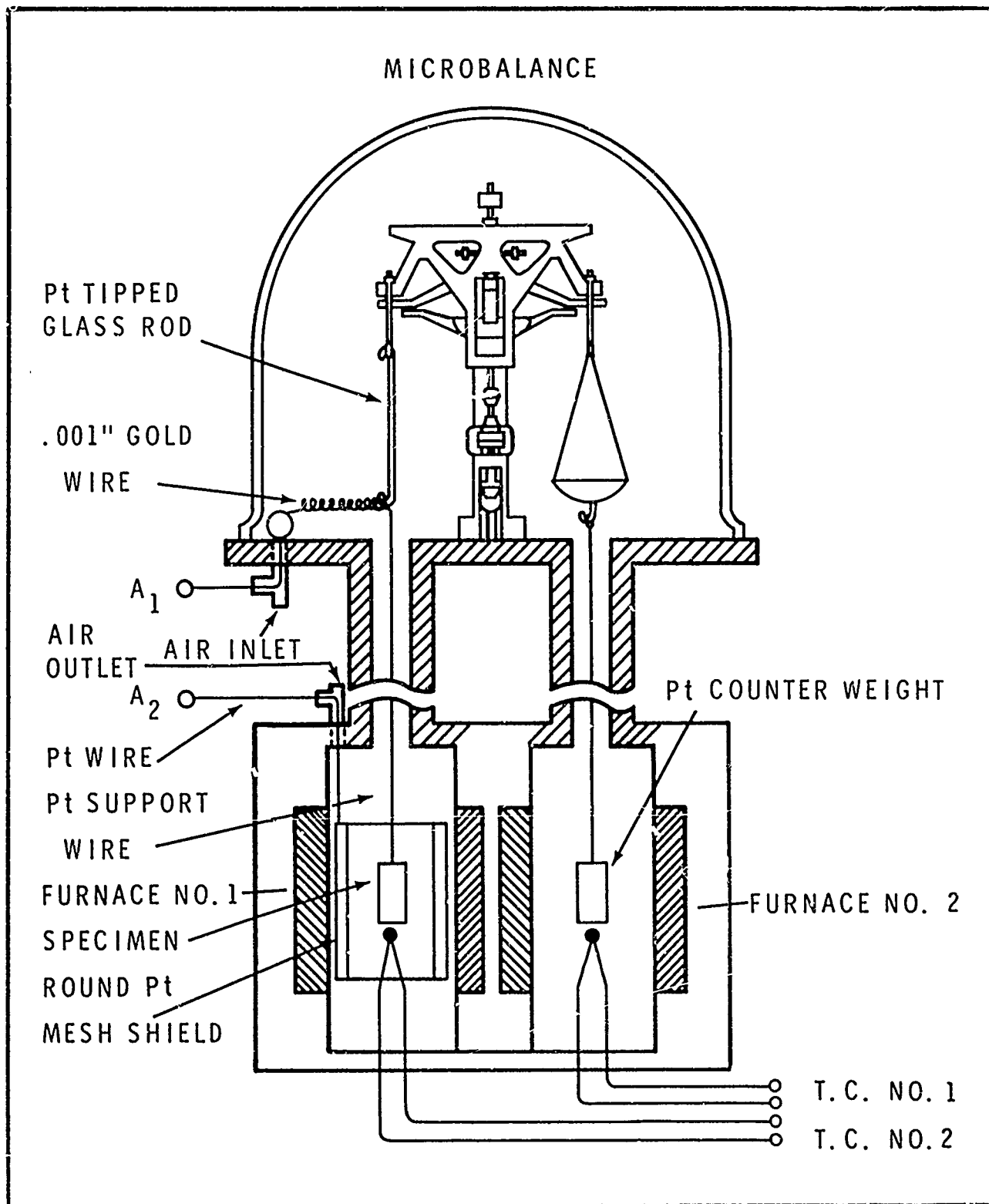


Figure 4. Experimental Configuration of Microbalance

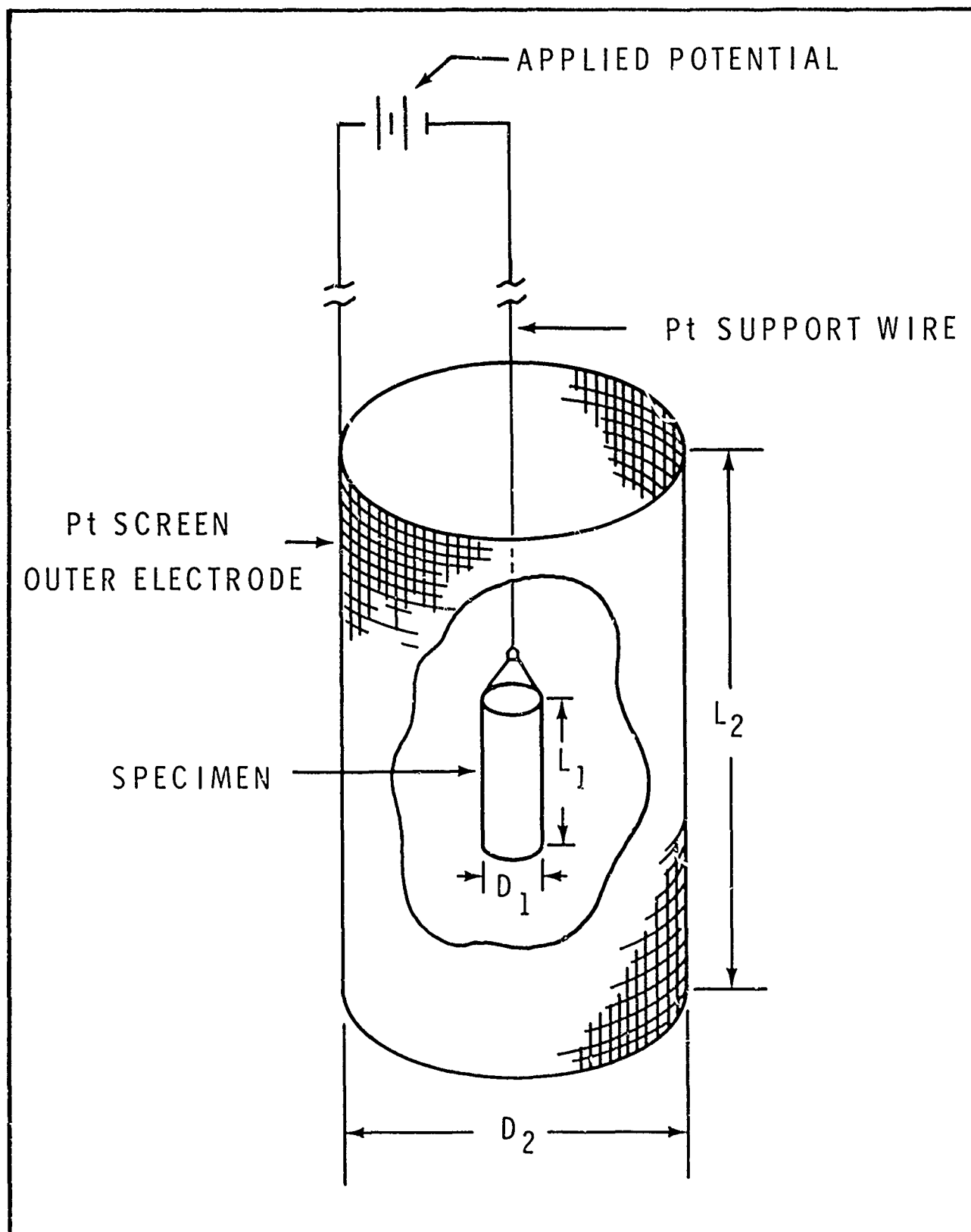


Figure 5. Specimen Configuration in the Platinum Electrode

MOLYBDENUM

Because of the high volatility of molybdenum trioxide the thermo-gravimetric balance assembly was not used in this portion of the experiment. The tapered molybdenum specimens were aligned vertically between two small diameter platinum coated alumina tubes containing the electrical lead and the recording thermocouple. The recording thermocouple was in the lower alumina tube and the electrical lead wire was contained in the upper tube. The furnace containing a charged platinum wire mesh screen and heated to temperature was lowered into place over the mounted specimen. The wire mesh screen concentrically surrounded the specimen and served as the other electrode making the final geometric configuration very much like that of Figure 5. The specimens were oxidized for one hour under the influence of the electric field at a temperature of 700°C in dry air flowing at a rate of 4 cu ft/hr. The oxidized specimens were then immersed in molten NaOH for 10 to 12 seconds to remove the oxide scale and weighed. Separate tests had shown that the molten salt removed less

then 0.5 percent of the base metal when left in the molten NaOH for one minute. Final weighings of the cleaned specimens were made to determine the percent of weight lost. Temperature deviation was less than 10°C over a 24-hour period with one-hour fluctuations well within 5°C.

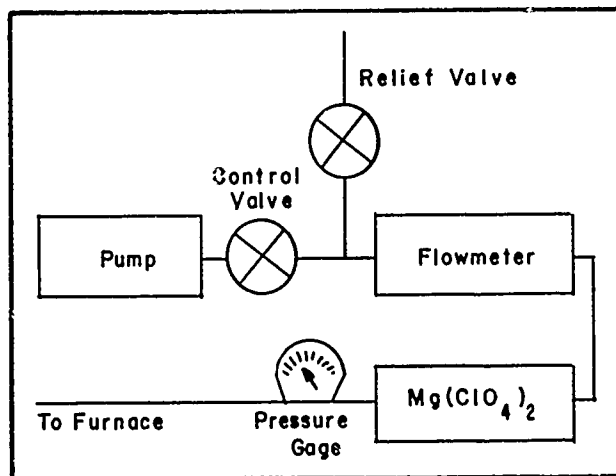


Figure 6. Gas Train

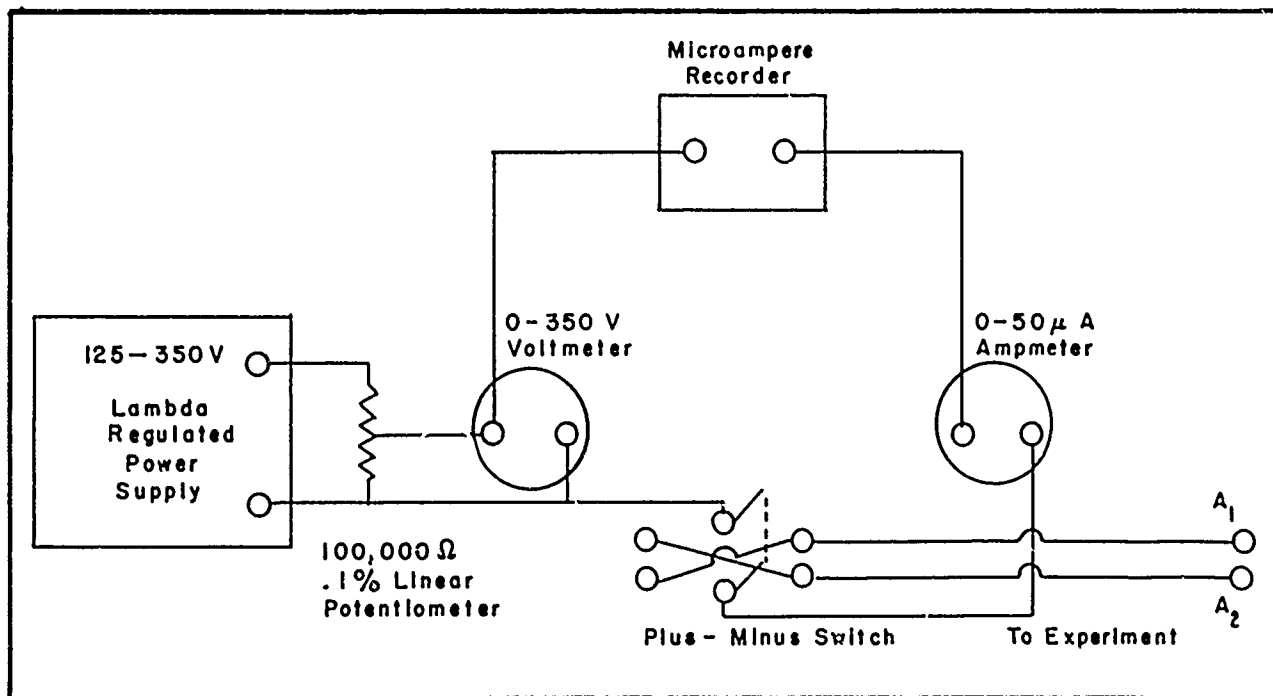


Figure 7. DC Power Supply

SECTION V

EXPERIMENTAL RESULTS

STAINLESS STEEL

Results of this work clearly indicate that the presence of an electric field does inhibit the rate of oxidation of type 304 stainless steel, which might be true for stainless steel in general. Figures 8, 9, and 10 show the resultant inhibited rate of oxidation of type 304 stainless steel with an impressed electric field. It appears that the stronger the field the greater the effect, and that the sign or direction of field has no real distinction. It should also be noted that the electric field could be removed at any time during the experiment with no change in oxidation. Figure 11 shows a reproducibility check at 150 volts with good agreement obtained between the two runs.

Good agreement was not noted however when comparing the authors' conventional oxidation data for type 304 stainless steel with that available from the literature. Figure 12 shows conventional oxidation curves obtained during this investigation compared to those reported by Gulbransen and Andrew (Reference 12). A number of factors probably contribute to this disparity. First of all, the experimental test temperatures used are high for stainless steel (within 300°C of the melting point). Further, the H₂/Ar "anneal" utilized herein may have produced an effect on the metal's oxidation characteristics. Another factor of influence may be the composition differences of the type 304 stainless steels being compared. Table I shows the nominal composition for type 304 stainless steels and the actual analyses of the steels used in this and the referenced investigations.

Radavich (Reference 11) reported the effect of silicon content on the oxidation characteristics of 16 percent Cr, 10 percent Ni and the balance Fe stainless steels. His results show a marked passivation tendency as the silicon content increases up to about 3.5 percent at 600° to 1000°C. His results were substantiated by MAMP using the second heat of steel of higher silicon content as

shown in Table. I. Figure 12 also shows the effects of the higher silicon content of the second heat.

Difficulties in reproducing oxidation data from a multicomponent material such as stainless steel might be expected. In any event the important factor here is the unequivocal evidence that there is a substantial dipolar effect on the oxidation of a low silicon type 304 stainless steel in the presence of an electric field.

MOLYBDENUM

Several field conditions were investigated to study the electric field influence on molybdenum oxidation. Results, reported as percent weight lost vs voltage after one hour at 700°C, are presented in Figure 13. From this data it can be deduced that the presence of an electric field will either inhibit or accelerate the oxidation of molybdenum, depending on the strength of the field. There also seems to be a slight difference in the effect dependent on the direction of the applied potential. For positive potentials (specimen is positive) the oxidation rate increases up to about 80 volts; beyond this, the rate decreases. For negative potentials (specimen is negative) the peak value is nearer to 100 volts with the rate decreasing thereafter.

NICKEL

Electric field influence on the oxidation of nickel was studied at 1040°C (± 195 volts) and 1095°C (± 49 volts). Conventional oxidation data were also taken. Unlike molybdenum and 304 stainless steel, nickel exhibited no anomalous effect when oxidized under the influence of an electric field. Figure 14 shows the authors' electric field oxidation data as agreeing well with the conventional (no electric field) oxidation data of Sartell and Li (Reference 14) and

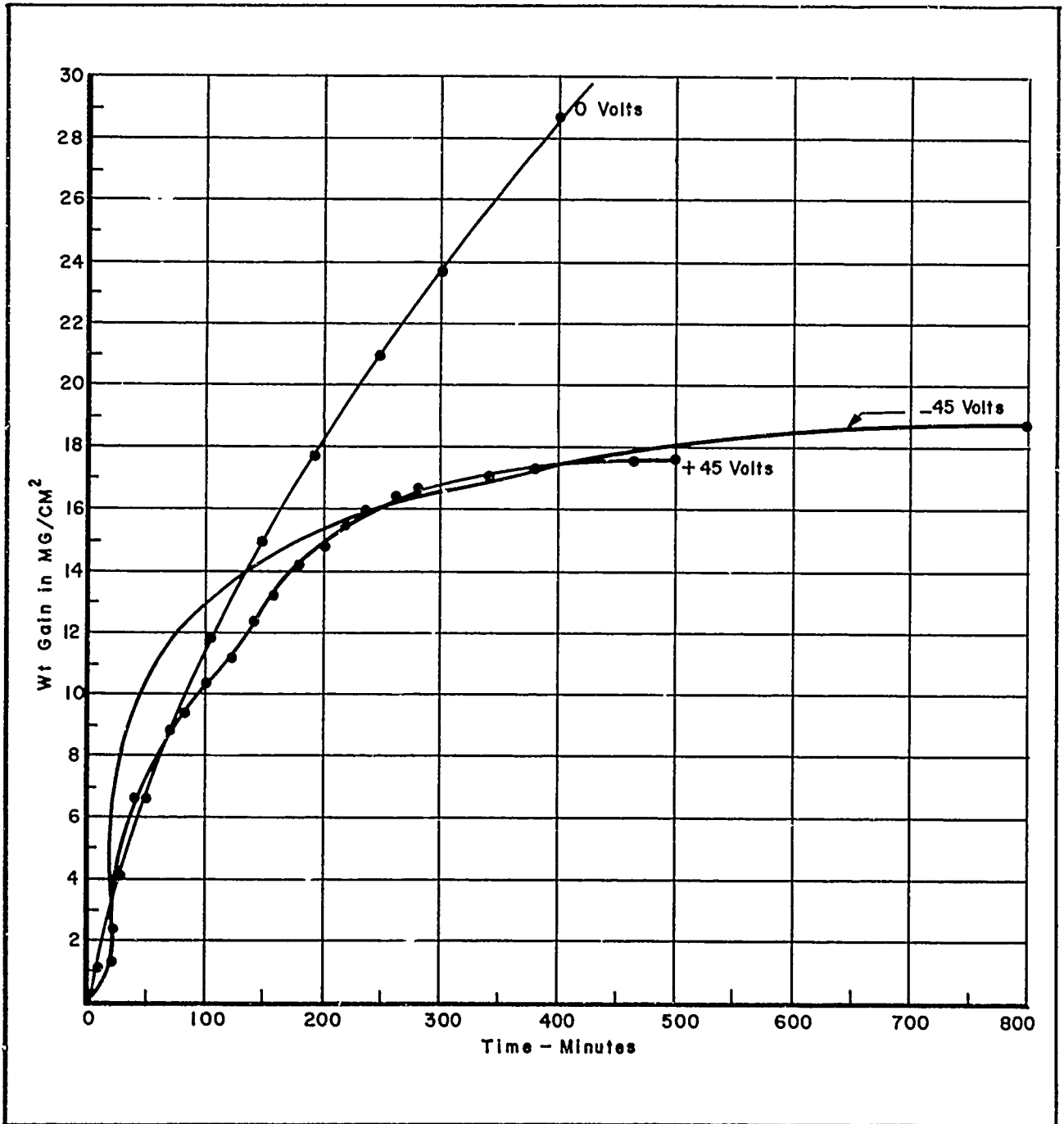


Figure 8. 304 Stainless Steel Oxidized in an Electric Field at 1140°C (0 Volts, ±45 Volts)

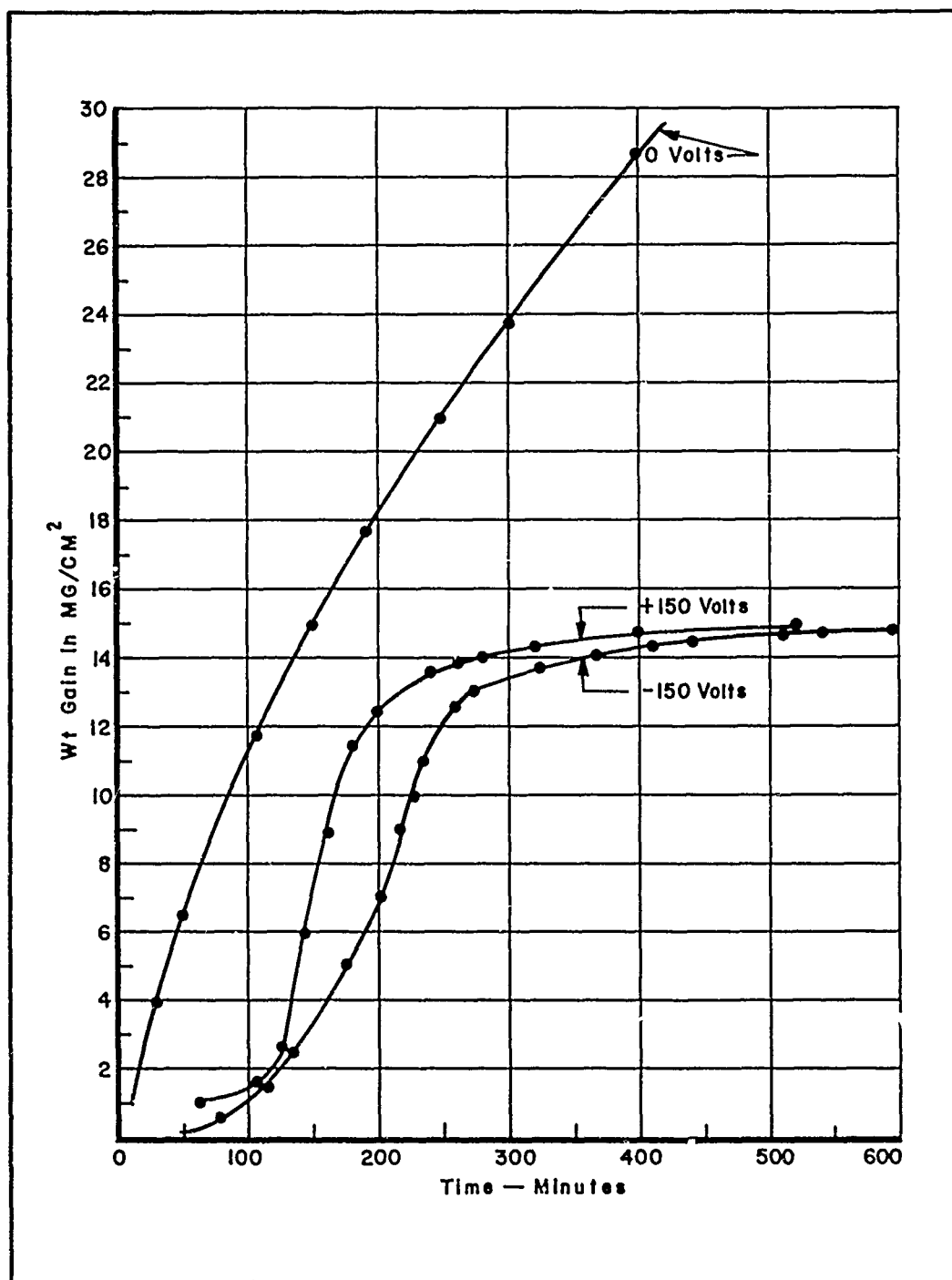


Figure 9. 304 Stainless Steel Oxidized in an Electric Field at 1140°C (0 Volts, ±150 Volts)

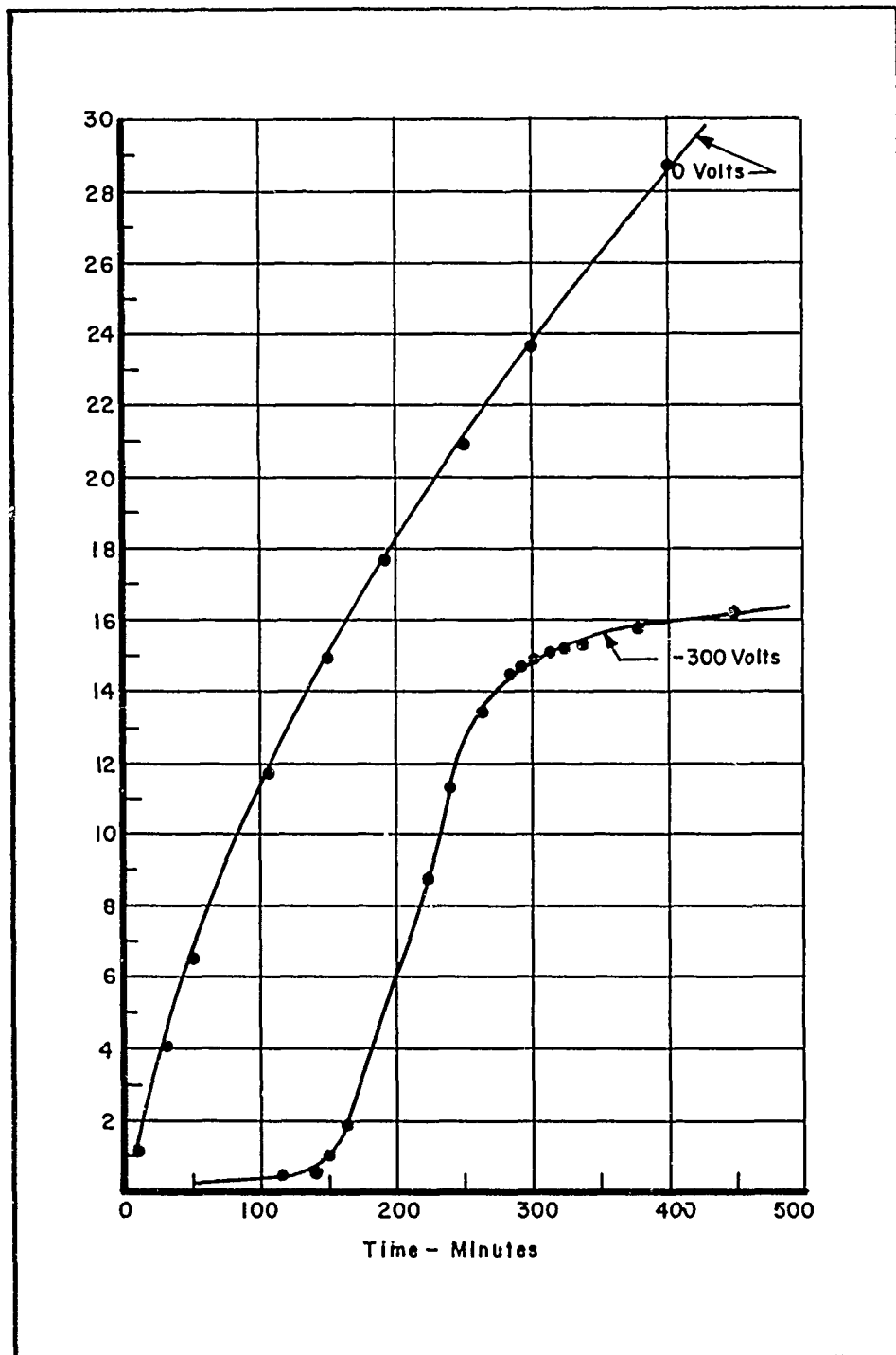


Figure 10. 304 Stainless Steel Oxidized in an Electric Field at 1140°C (0 Volts, - 300 Volts)

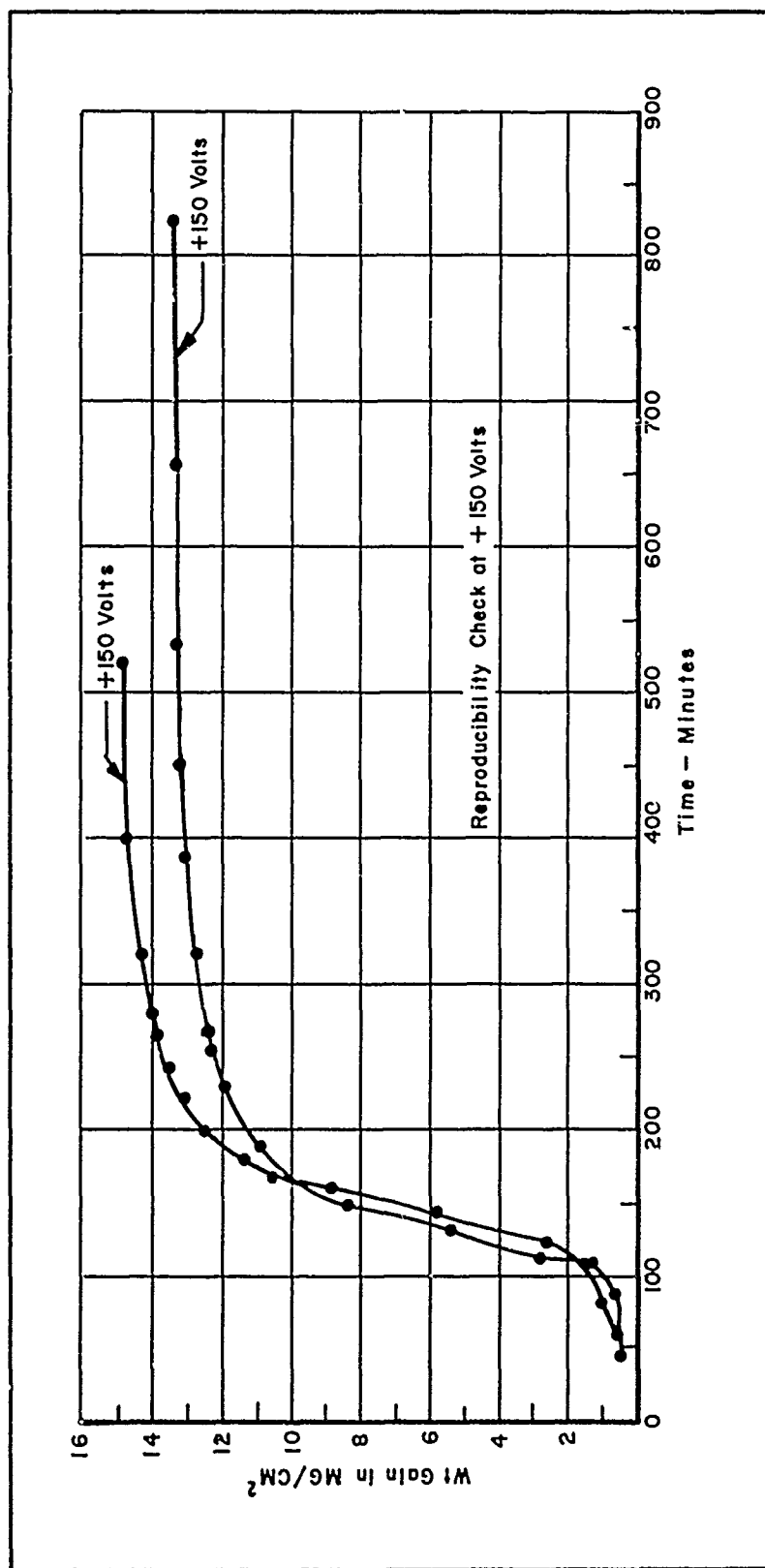


Figure 11. 304 Stainless Steel Oxidized in an Electric Field at 1140°C (+150 Volts)

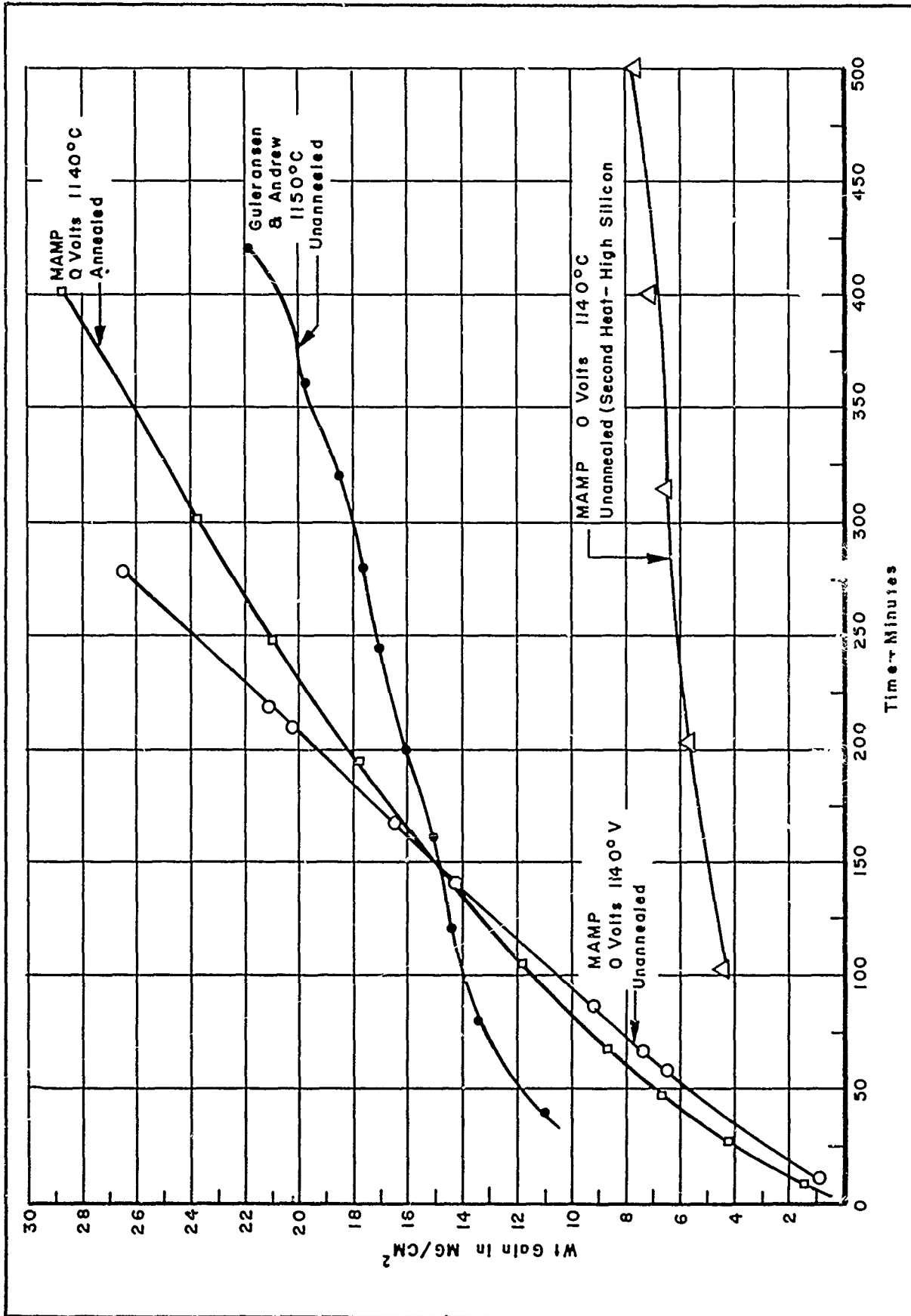


Figure 12. Comparison of Oxidation Data Without Electric Field 1140° - 1150°C

TABLE I
 STAINLESS STEEL ANALYSIS
 PERCENT ELEMENT PRESENT

Investigator	Cr	Ni	Mn	P	N	C	Fe	Si	S	Mo	Cu	V	Co
Caplan and Cohen (Ref. 10)	18-20	8-12	2.00 (max)	0.045 (max)		0.08 (max)	Ba1	1.00 (max)	0.03 (max)	—	—	—	—
Gulbransen and Andrew (Ref. 12)	17.7	8.34	1.11	0.02	0.16	0.11	Ba1	—	—	—	—	—	—
MAMP (First Heat)	19.42	9.42	1.67	—	—	0.03	Ba1	0.35	0.020	0.3	0.17	0.03	0.06
MAMP (Second Heat)	17.8	8.24	1.56	0.025	—	0.09	Ba1	0.6	0.23	—	—	—	—

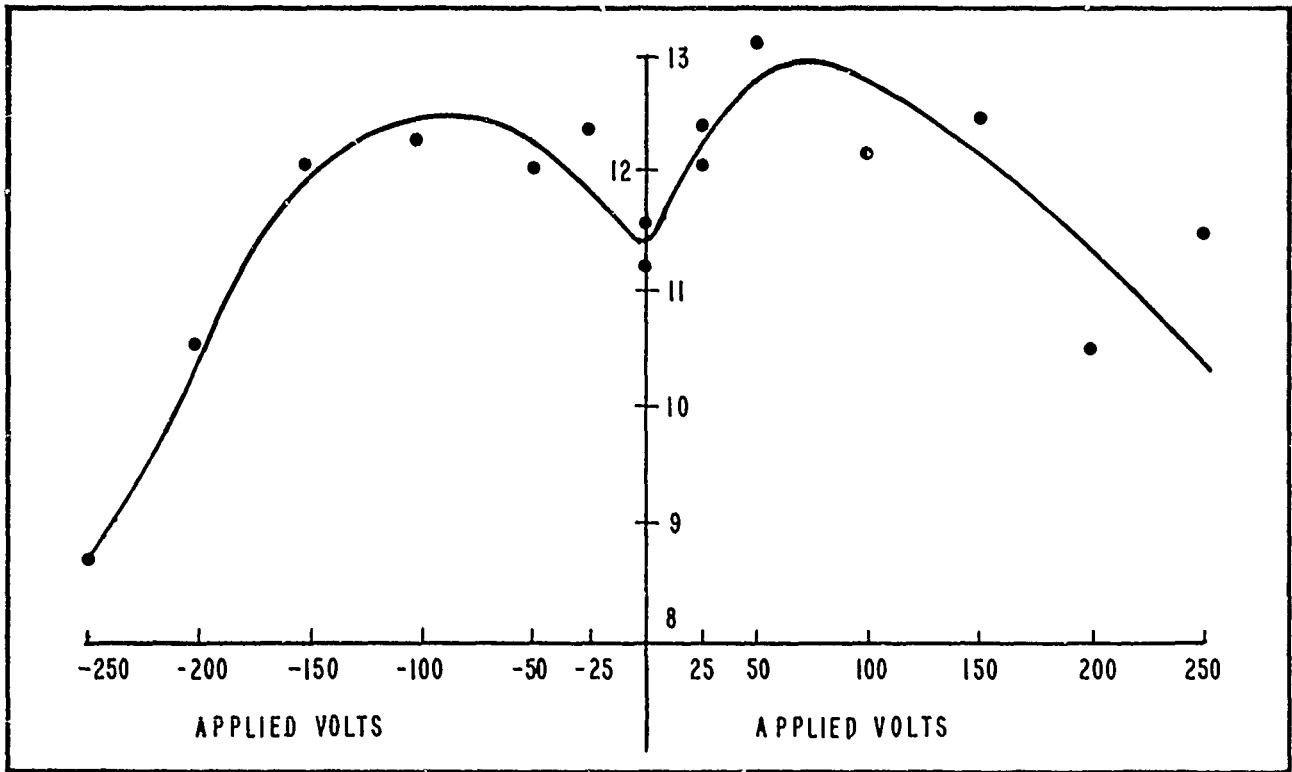


Figure 13. Percent Weight Lost of Molybdenum vs Voltage at 700°C for one Hour

Lombard (Reference 15). Figures 15 and 16 show the resultant oxidation curves obtained under applied potentials of ± 49 volts and ± 195 volts. Comparison with the conventional oxidation curves shows little difference, and the parabolic rate constants from

the conventional oxidation curves agree within 5 percent of Sartell and Li at the same temperature. Crystallographic and metallographic examination exhibited no systematic variation in structure or habit of the oxide layer.

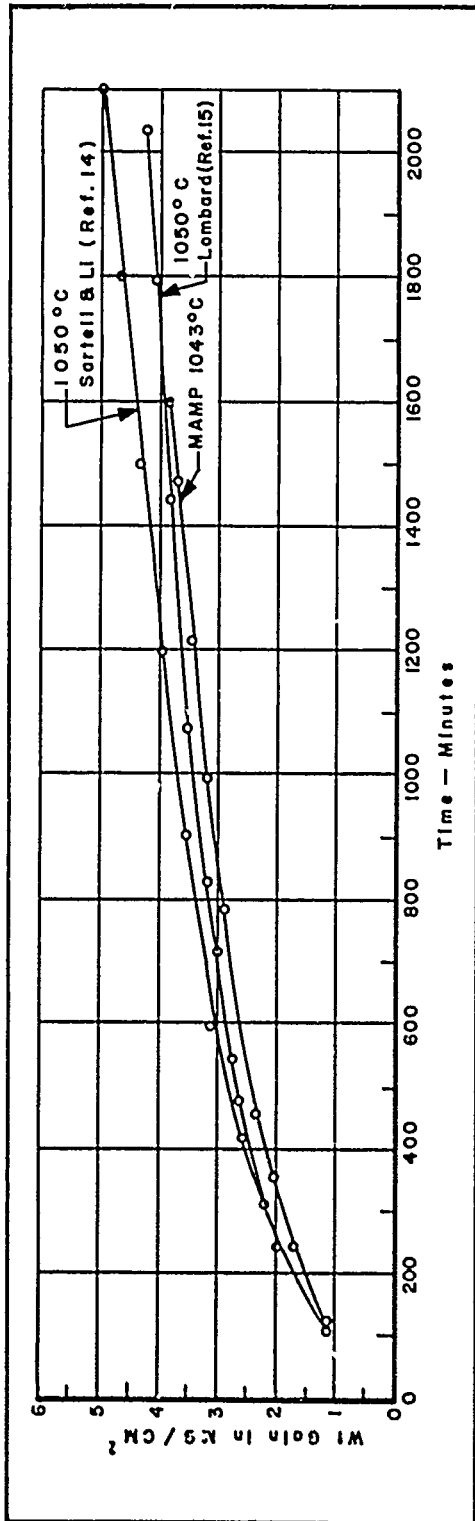


Figure 14. Comparison of Nickel Data at Approximately 1050°C

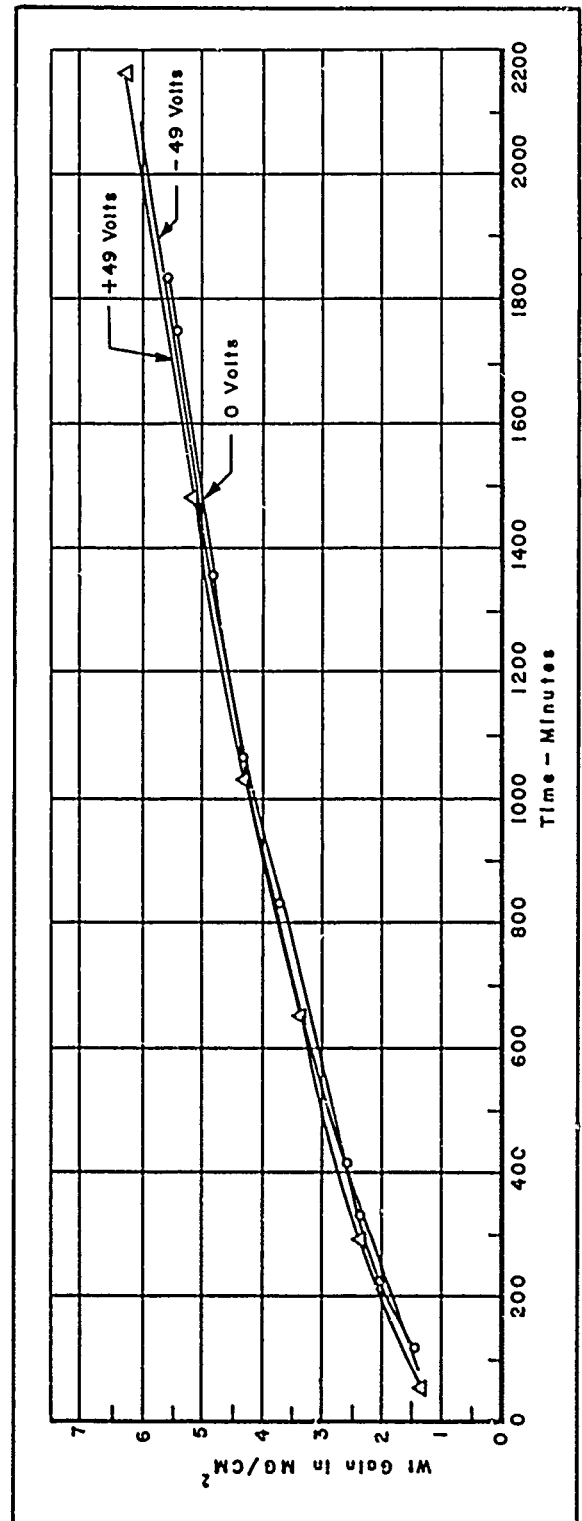


Figure 15. Nickel Oxidized in an Electric Field at 1095°C (0 Volts, ±49 Volts)

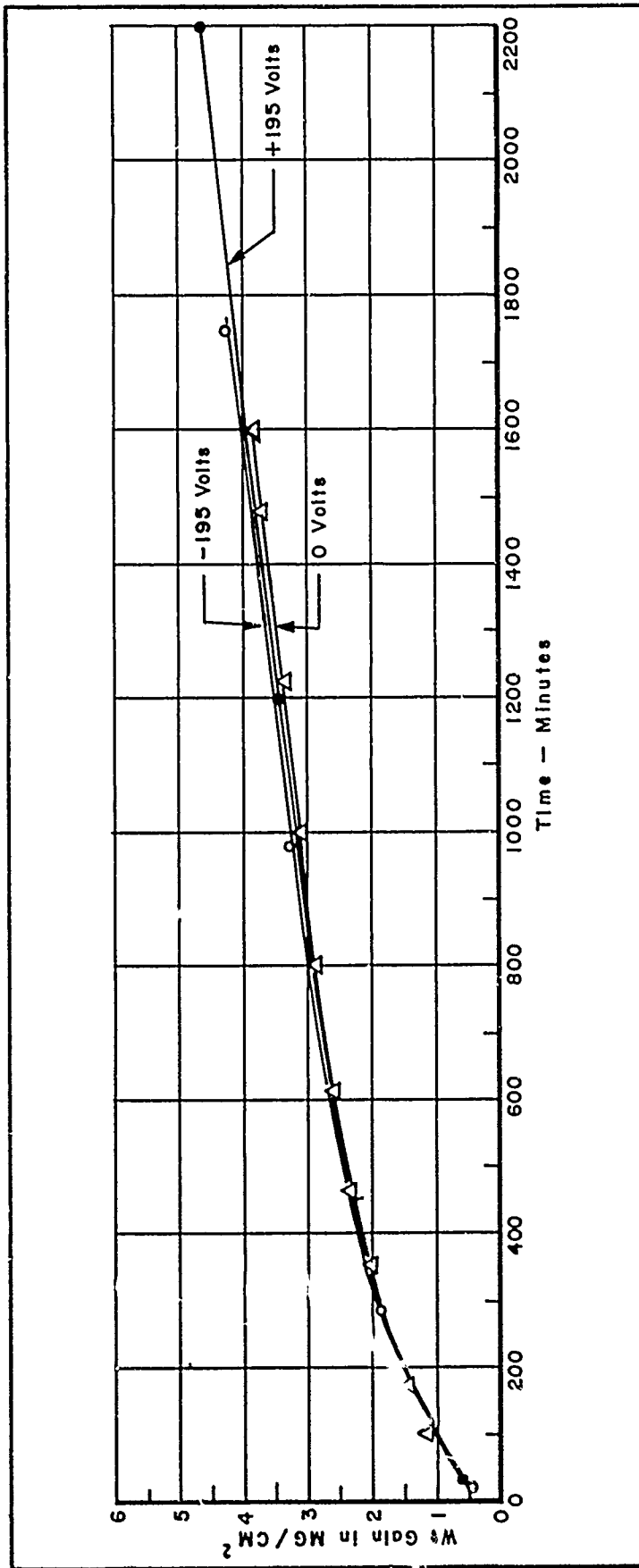


Figure 16. Nickel Oxidized in an Electric Field at 1043°C (0 Volts, ±195 Volts)

SECTION VI

DISCUSSION AND CONCLUSIONS

The fact that the electric field could be removed once oxidation had begun and the rate remained constant in the case of the stainless steel leads to the conclusion that a passivation layer was formed. The authors believe that in the case of stainless steel the experimental results fit the theory of Hauffe (Reference 19). Hauffe noted that "rate-determining field transport through the tarnishing layer... occurs whenever that rate-determining activation energy U_1 for the transfer of a metal ion from the metal phase into the tarnishing layer is smaller than the activation energy U_2 for migration within the layer. In Figure 17, $U_1 > U_2$, so that the transfer is rate-determining.

"Figure 17 is a schematic representation of the saddle jump of the metal ions for vacancy migration in the presence of an electric field in the tarnishing layer ($a \neq a^*$; $U_1 > U_2$) (Reference 19).

"If we assume a sufficiently rapid ion and electron transport through the tarnishing layer, the field influences the rate-determining transfer of ionic defects in the lattice in such a way that the determinative activation energy U_1 is reduced to about U_E (Figure 17)... the number of defects passing per unit time and surface is

$$\dot{n}_1 = A_1 \exp(-U_1/kT). "$$

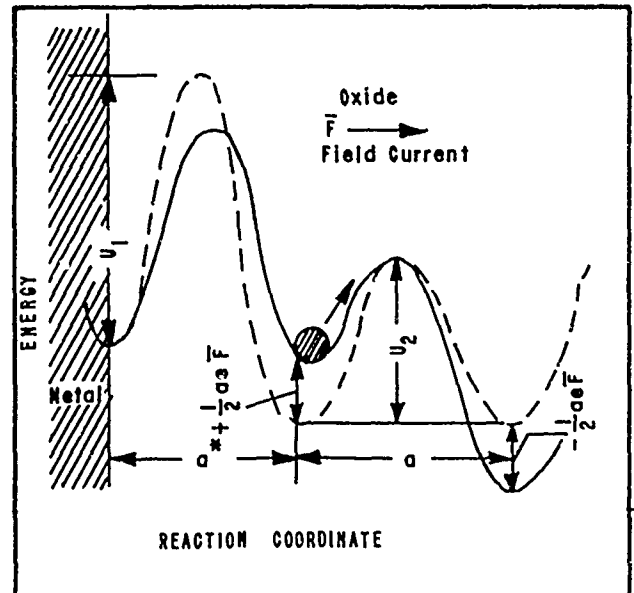


Figure 17. Schematic Representation of a Saddle Jump of Metal Ions

Of the three systems investigated, type 304 stainless steel and molybdenum exhibited a dipolar effect when oxidized under the influence of an electric field. Thus we can conclude that in these two cases the value of U_1 was increased as shown in Figure 17, thereby slowing down the rate of oxidation. No effect was discernible in the case of nickel, probably because the temperatures investigated were too high.

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