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*THE EFFECTS OF ALLOYING ADDITIONS
UPON THE POLARIZATION BEHAVIOUR
OF AISI TYPE 430 STAINLESS STEEL*

G.J. BIEFER AND J.G. GARRISON

PHYSICAL METALLURGY DIVISION

DECEMBER 1967

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THE EFFECTS OF ALLOYING ADDITIONS UPON THE POLARIZATION
BEHAVIOUR OF AISI TYPE 430 STAINLESS STEEL

by

G. J. Biefer* and J. G. Garrison**

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ABSTRACT

Anodic and cathodic polarization measurements were performed in normal H_2SO_4 , with and without an addition of 0.5 normal NaCl, on AISI Type 430 stainless steels alloyed with each of Mo, V, W, Ta, Si, Re, Pd, and Ge. For each addition, the changes brought about in anodic and/or cathodic polarization behaviour were related to changes in corrosion resistance in sulphuric acid and in other aqueous corrodants.

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Direction des Mines

LES EFFETS D'ADDITIONS D'ALLIAGE SUR
LA POLARISATION DES ACIERS INOXYDABLES
DE TYPE AISI 430

par

G. J. Biefer* et J. G. Garrison**

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RÉSUMÉ

Des mesures de polarisation anodique et cathodique ont été effectuées dans une solution de H_2SO_4 N et une autre de H_2SO_4 N et NaCl 0.5 N, sur les aciers inoxydables de type AISI 430 alliés tour à tour avec Mo, V, W, Ta, Si, Re, Pd, et Ge. A chaque addition, les changements notés dans la polarisation anodique ou cathodique étaient reliés aux changements de la résistance à la corrosion dans l'acide sulfurique et autres corrodants aqueux.

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INTRODUCTION

The 400-series chromium stainless steels are, in general, more susceptible to corrosion than the more expensive 300-series chromium-nickel stainless steels. It was decided, therefore, to attempt to improve the corrosion resistance of one of the 400-series steels - Type 430 (14-18% Cr) stainless - by means of alloying additions. The eventual goal of the work was to produce, if possible, a modified Type 430 stainless steel with corrosion resistance equal to or greater than that of a typical 300-series steel - austenitic Type 304 (18-20% Cr, 8-10% Ni) stainless.

The alloying additions selected for initial study were Mo, V, W, Ta, Si, Re, Pd, and Ge. All are soluble to at least some extent in ferritic iron, and all demonstrate good to excellent corrosion resistance in sulphuric and/or hydrochloric acid. Although some of the additions appeared to be too expensive to be used widely, they were nonetheless included in this work because of their theoretical interest.

It is well known that Mo improves the aqueous corrosion resistance of ferritic stainless steels such as AISI Type 430. In practice, Mo additions have been used for this purpose⁽¹⁾. Laboratory tests have also supplied evidence that additions of Mo to plain chromium steels bring about increased corrosion resistance in sulphuric, hydrochloric and acetic acids⁽²⁾ and in ferric chloride and also confer increased resistance to crevice corrosion in a neutral salt solution⁽³⁾. In addition, polarization measurements have been carried out on 17% chromium steels, with and without alloying additions, in nitrogenated 5% sulphuric acid⁽⁴⁾. This work demonstrated that Mo additions brought about decreased corrosion rates and increased stability of the passive film in sulphuric acid.

For the elements V, W, Ta, Si and Ge, a study of the "Corrosion Abstracts" published from 1962 to the present did not reveal any quantitative information regarding their effects upon the aqueous-corrosion behaviour of

17% chromium ferritic steels. However, the polarization measurements referred to above⁽⁴⁾ indicated that 1% Si, as a supplement to Mo in 17% chromium steels, increased the stability of the passive films in sulphuric acid; however, such additions did not bring about decreased active-corrosion rates in nitrogenated 5% sulphuric acid. It might be noted that the effects of V, W, Ta and Si additions upon the polarization behaviour and corrosion resistance of austenitic stainless steels have been investigated (for example, references 5 and 6).

With respect to additions of Pd to ferritic and austenitic stainless steels, Tomashov was the first to demonstrate their pronounced beneficial effect upon corrosion resistance in certain aqueous media⁽⁷⁾. Recently, he has supplied further information on the effects of Pd, and also Re, additions upon the corrosion of ferritic stainless steels⁽⁸⁾.

In recent years it has been demonstrated that aqueous-corrosion rates may be derived from anodic and/or cathodic polarization measurements⁽⁹⁾. Further, it has been shown that such measurements can be an extremely useful means of predicting the effects of alloying additions upon corrosion behaviour^(10, 11). It was decided, therefore, to determine the effects of the alloying additions primarily by means of anodic and cathodic polarization measurements in a medium widely used for this purpose - normal sulphuric acid. Since chloride ions are commonly encountered in service environments, and are known to be deleterious to the corrosion resistance of stainless steels, it was decided that the measurements would also be carried out in chloride-containing acid solutions.

In order to verify and extend the initial findings in the polarization measurements, conventional weight-loss corrosion tests were carried out on the steels in normal solutions of sulphuric acid, hydrochloric acid and ferric chloride and in boiling 65% nitric acid. The results of these tests are reported in detail elsewhere⁽¹²⁾ and the principal trends are summarized in Table 1 of the present report.

TABLE 1
Principal Effects of the Alloying Additions in Corrosion Tests*

Steel No.	Per Cent of Alloying Addition	Test Solution				Steel No.	Per Cent of Alloying Addition	Test Solution			
		H ₂ SO ₄	HCl	FeCl ₃	HNO ₃			H ₂ SO ₄	HCl	FeCl ₃	HNO ₃
A	-					7A	-				
B	0.70 Mo	+	+	++	+	7B	0.59 Re		--	+	+
C	1.48	+	+	++	++	8C	1.15		--	+	
D	3.11	+	+	++	++	8D	1.84	+	+	+	+
I	-					9A	-				
J	0.67 V			++		9B	0.06 Pd			--	--
K	1.13	+	+	++		10C	0.10	--	--	--	--
L	2.18	+	+	++		10D	0.26	--	--	--	--
M	-					3B	0.46	+	+	--	--
N	0.47 W	+	+	++		4C	0.99	+	+	--	--
O	0.94	+	+	++		4D	1.91	+	+	--	--
P	2.30	+	+	++						--	--
Q	-					5A	-				
R	0.41 Ta					5B	0.41 Ge	-	--	--	--
S	0.72					6C	1.03	-	--	--	--
T	1.37					6D	2.01		--	--	--
U	0.39** Si										
V	0.84										
W	1.33				--						
X	1.78				--						

* + signifies 0.5 or less of the corrosion rate of the control steel.
- signifies 2.0 or more than corrosion rate of the control steel.

** Base Si content.

EXPERIMENTAL

Materials

In all, the polarization measurements were carried out on 35 different heats of Type 430 stainless steel and one heat of Type 304 stainless steel. Complete chemical analyses of these steels⁽¹²⁾ appear in Appendix I; the present report lists the levels of each of the alloying elements (see Tables 2-5). The steels were produced at the Physical Metallurgy Division as follows:

- I. Steels A to X (omitting E, F, G and H) included alloys with each of Mo, V, W, Ta, and Si. For each alloying element, a 50-lb, aluminum-deoxidized induction-furnace heat was prepared which was split into four 12-lb ingots. The first ingot was the base material. Additions of the alloying element were then made to the furnace, to produce ingots containing the alloying element at three different levels.

After being forged to 1/4-in. plate, the steels were annealed for 1 hr at 840°C (1550°F), and then were furnace-cooled.

- II. Steels 3B to 10D (15 in all) included alloys with each of Re, Pd and Ge. Because of the high cost of these additions, smaller ingots were made. Initially, 4-lb heats of the base Type 430 steel were melted in a small induction furnace. Each heat was split into two 1-in. -diameter ingots, the first being the base material and the second containing the alloying addition at a selected level. A second 4-lb heat was then split in the same way to yield two more ingots containing different and higher levels of the alloying addition. In all cases, the additions were made to the furnace.

The 1-in. - diameter ingots were machined to 3/4-in. diameter, then swaged to a diameter of 3/8 in. After this they were annealed for 1 hr at 840°C (1550°F) and furnace-cooled.

TABLE 2
Results of Anodic Polarization Measurements in Nitrogenated Normal
Sulphuric Acid (Scan Speed, 8 Volts/hr)

Melt No.	Alloying Addition	E _{corr} (mV)	E _{cpp} (mV)	E _p (mV)	E _t (mV)	E _{csp} (mV)	i _{cpp} ² mA/cm ²	i _{min1} ² mA/cm ²	i _{csp} ² mA/cm ²	i _{min2} ² mA/cm ²
A	-	-510	-454	-232	+980	+1150	15.6	0.015	2.0	1.3
B	0.70% Mo	-450	-397	-332	+1010	+1100	3.7	0.018	1.7	1.4
C	1.48% Mo	-470	-424	-390	+1017	+1180	3.1	0.015	2.0	1.9
D	3.11% Mo	-460	-406	-352	+1039	+1230	1.6	0.018	3.1	2.4
I	-	-520	-479	-232	+955	+1130	18.8	0.029	5.3	2.3
J	0.67% V	-510	-422	-248	+950	+1130	10.0	0.030	5.0	2.2
K	1.13% V	-500	-421	-266	+975	+1220	8.8	0.027	4.4	2.5
L	2.18% V	-490	-456	-328	+915	+1230	5.8	0.028	3.9	2.7
M	-	-520	-405	-241	+985	+1110	17.3	0.016	2.5	1.8
N	0.47% W	-500	-437	-312	+998	+1140	6.4	0.015	1.8	1.5
O	0.94% W	-500	-437	-320	+1003	+1140	6.0	0.016	2.3	1.4
P	2.30% W	-480	-408	-323	+1002	+1150	4.4	0.018	1.8	1.8
Q	-	-510	-438	-262	+985	+1140	30.8	0.014	2.6	1.7
R	0.41% Ta	-510	-463	-291	+980	+1210	16.4	0.019	1.6	1.6
S	0.72% Ta	-500	-466	-246	+980	+1130	13.8	0.017	1.7	1.5
T	1.37% Ta	-510	-472	-229	+940	+1090	16.0	0.019	2.2	1.6
U	0.39% Si	-520	-476	-212	+985	+1130	19.3	0.017	2.8	1.7
V	0.84% Si	-510	-478	-260	+1000	+1170	13.4	0.016	3.6	2.2
W	1.33% Si	-510	-473	-241	+982	+1150	12.7	0.015	5.4	3.0
X	1.78% Si	-500	-457	-272	+995	+1180	18.4	0.015	8.1	2.3
7A	None	-520	-467	-291	+990	+1150	15.9	0.015	2.6	1.2
7B	0.59% Re	-400	-378	-346	+980	+1130	2.5	0.024	2.8	1.5
8C	1.15% Re	-390	-368	-368	+944	+1100	1.1	0.071	2.7	1.8
8D	1.84% Re	-380	-358	-345	+1030	+1120	0.61	0.060	2.9	1.7
9A	None	-490	-393	-238	+1042	+1220	17.4	0.024	3.5	1.7
9B	0.06% Pd	-485	-441	-310	+1025	+1180	13.6	0.071	2.9	1.6
10C	0.10% Pd	-500	-393	-228	+945	+1140	16.1	0.028	2.7	1.6
10D	0.26% Pd	-420	-383	-257	+663	+1180	17.0	0.093	3.3	1.4
3B	0.46% Pd	+270**	-	-	-	+1250	-	-	2.9	2.2
4C	0.99% Pd	+240**	-	-	-	+1170	-	-	3.2	2.5
4D	1.91% Pd	+270**	-	-	-	+1160	-	-	5.8	3.1
5A	None	-510	-441	-314	+1010	+1260	12.9	0.026	2.7	2.2
5B	0.41% Ge	-450	-419	-146	+890	+1150	4.8	0.027	2.6	2.0
6C	1.03% Ge	-420	-213	-140	+960	+1060	3.1	0.025	3.1	1.6
6D	2.01% Ge	-400	-216	-119	+815	+970	5.0	0.033	5.3	1.6
6266***	-	-390	-310	-241	+890	+1300	3.0	0.014	19.1	8.5

*Base composition.

**Passivated spontaneously.

***Type 304 stainless steel.

TABLE 3
Results of Anodic Polarization Measurements in Nitrogenated Normal
Sulphuric Acid Containing 0.5 Normal NaCl
(Scan Speed, 8 Volts/hr)

Melt No.	Alloying Addition	E_{corr} (mV)	E_{cpp} (mV)	E_p (mV)	E_t (mV)	i_{cpp} (mA/cm ²)	i_{min_1} (mA/cm ²)
A	None	+517	-402	-174	+351	19.4	0.124
B	0.70% Mo	-488	-355	-260	+406	8.0	0.029
C	1.48% Mo	-473	-376	-272	+526	5.5	0.022
D	3.11% Mo	-458	-379	-276	+647	3.7	0.024
I	None	-528	-464	-178	+372	14.8	0.255
J	0.67% V	-520	-381	-137	+377	22.9	0.190
K	1.13% V	-507	-381	-230	+316	18.2	0.079
L	2.18% V	-503	-391	-261	+488	8.8	0.040
M	None	-515	-442	-196	+388	16.3	0.032
N	0.47% W	-500	-386	-230	+392	17.0	0.023
O	0.94% W	-495	-365	-237	+414	11.7	0.023
P	2.30% W	-484	-362	-244	+417	7.4	0.031
Q	None	-510	-373	-172	+371	16.9	0.046
R	0.41% Ta	+505	-393	-233	+419	16.9	0.038
S	0.72% Ta	-514	-393	-218	+385	31.2	0.046
T	1.37% Ta	-517	-385	-236	+398	15.5	0.040
U	0.39% Si*	+515	-430	-228	+368	20.2	0.043
V	0.84% Si	-512	-439	-139	+402	16.4	0.083
W	1.33% Si	-303	-363	-187	+509	19.4	0.038
X	1.78% Si	-500	-385	-202	+531	42.4	0.024
7A	None	-540	-459	-218	+313	20.6	0.061
7B	0.59% Re	-420	-366	-277	+392	9.0	0.093
8C	1.15% Re	-380	-355	-276	+450	5.6	0.094
8D	1.84% Re	-370	-338	-288	+539	3.0	0.093
9A	None	-505	-319	-230	+270	24.6	0.13
9B	0.06% Pd	-497	-391	-217	+275	12.5	0.10
10C	0.10% Pd	-501	-364	-195	+298	18.2	0.13
10D	0.26% Pd	-438	-359	-52	+208	25.2	0.18
3B	0.46% Pd	-405	-374	-214	+412	8.2	0.127
4C	0.99% Pd	-360	-350	-193	+335	12.6	0.220
4D	1.91% Pd	-305	-82	+116	+260	6.9	0.380
5A	None	-500	-381	-236	+322	17.5	0.053
5B	0.41% Ge	-440	-373	-156	+398	12.7	0.040
6C	1.03% Ge	-400	-365	-162	+425	8.9	0.035
6D	2.01% Ge	-380	-283	-64	+495	7.4	0.054
6266**	-	-427	-280	-184	+518	9.4	0.049

* Base composition.

**Type 304 stainless steel.

TABLE 4
Results of Cathodic Polarization Measurements in Nitrogenated
Sulphuric Acid (Scan Speed, 4 Volts/hr)

Melt No.	Alloying Addition	E_{corr} (mV)	B_c mV/decade	i_o μ A/cm ²	i_{corr} μ A/cm ²	Melt No.	Alloying Addition	E_{corr} (mV)	B_c mV/decade	i_o μ A/cm	i_{corr} μ A/cm
A	None	-518	99	15	3400	7A	None	-512	88	12	4000
B	0.70% Mo	-490	99	20	2150	7B	0.59% Re	-402	48	50	5700
C	1.48% Mo	-479	99	14	1200	8C	1.15% Re	-383	37	10	2100
D	3.11% Mo	-466	89	6.8	620	8D	1.84% Re	-372	45	71	2250
I	None	-522	74	4.5	5900	9A	None	-504	90	11	2500
J	0.67% V	-518	74	5.7	3600	9B	0.06% Pd	-480	102	36	2500
K	1.13% V	-510	84	3.7	1630	10C	0.10% Pd	-496	42	0.12	6300
L	2.18% V	-505	89	4.6	1200	10D	0.26% Pd	-427	43	7.2	11,000
M	None	-513	104	29	3900	3B	0.46% Pd	+275	52	82	<1
N	0.47% W	-498	103	13	1330	4C	0.99% Pd	+265	53	210	<1
O	0.94% W	-496	90	6.9	1240	4D ⁱ	1.91% Pd	+235	58	1000	~2
P	2.30% W	-488	92	10	1340						
Q	None	-513	92	15	3900	5A	None	-480	104	17	1400
R	0.41% Ta	-505	91	16	3500	5B	0.41% Ge	-444	62	13	3600
S	0.72% Ta	-510	90	10	2700	6C	1.03% Ge	-412	50	9	2150
T	1.37% Ta	-513	93	15	3500	6D	2.01% Ge	-385	55	42	2000
U	0.39% Si*	-520	85	7.3	4000	6266**	-	-395	104	17	150
V	0.84% Si	-513	92	14	3600						
W	1.33% Si	-507	93	15	3070						
X	1.78% Si	-498	87	13	3100						

* Base composition.

**Type 304 stainless steel.

TABLE 5

Results of Cathodic Polarization Measurements in Nitrogenated Sulphuric

Acid Containing 0.5 Normal NaCl (Scan Speed, 4 Volts/hr)

Melt No.	Alloying Addition	E _{corr} (mV)	B _c mV/decade	i _o μA/cm ²	i _{corr} μA/cm ²	Melt No.	Alloying Addition	E _{corr} (mV)	B _c mV/decade	i _o μA/cm ²	i _{corr} μA/cm ²
A	None	-517	83	3.8	1180	7A	None	-520	83	3.2	1750
B	0.70% Mo	-492	83	1.9	510	7B	0.59% Re	-417	60	43	5000
C	1.48% Mo	-475	79	1.8	400	8C	1.15% Re	-388	61	185	6000
D	3.11% Mo	-458	101	7.2	350	8D	1.84% Re	-372	60	245	4600
I	None	-525	77	2.6	2950	9A	None	-534	86	1.4	1000
J	0.67% V	-523	73	0.84	1100	9B	0.06% Pd	-525	76	1.0	1200
K	1.13% V	-520	73	0.39	520	10C	0.10% Pd	-519	115	29	3000
L	2.18% V	-511	76	0.36	380	10D	0.26% Pd	-463	129	300	6400
M	None	-528	86	2.9	1600	3B	0.46% Pd	-420	144	1630	12,600
N	0.47% W	-507	78	0.76	460	4C	0.99% Pd	-369	131	3700	14,100
O	0.94% W	-505	78	0.76	430	4D	1.91% Pd	-315	138	2600	3700
P	2.30% W	-493	65	0.47	600						
Q	None	-523	75	0.74	900	5A	None	-502	87	1.7	460
R	0.41% Ta	-525	71	0.26	540	5B	0.41% Ge	-445	58	6.2	2800
S	0.72% Ta	-524	73	0.40	600	6C	1.03% Ge	-415	69	81	5000
T	1.37% Ta	-519	82	1.5	860	6D	2.01% Ge	-392	60	94	4250
U	0.39% Si*	-522	75	1.3	1500	6266**	-	-430	96	6.2	180
V	0.84% Si	-520	72	0.96	1400						
W	1.33% Si	-511	61	0.35	1340						
X	1.78% Si	-508	56	0.16	1120						

* Base composition.

**Type 304 stainless steel.

III. A heat of Type 304 chromium-nickel stainless steel was produced as a 50-lb melt in an induction furnace. The ingot was forged to 1/4-in. thickness, annealed at 1070°C (1950°F) for 1 hr, then water-quenched.

Specimen Preparation

The steels made under Parts I and III above were machined into small rectangular flat specimens, each containing an arm with a threaded socket, for use in the polarization measurements. The specimens were ground manually on 120-grit silicon-carbide papers to provide a standard surface. The steels made under Part II were machined into small cylindrical specimens and surface-finished as above, using a small lathe.

Before testing, the specimens were degreased in ultrasonically agitated carbon tetrachloride, rinsed in alcohol, and dried in a hot air blast. Prior to the suspension of a specimen in the test solution, a steel support rod was threaded into the socket and paraffin wax was used to cover the specimen arm and support rod. However, the paraffin was not extended to the solution line, in order to avoid the possibility of crevice corrosion under the paraffin.

Apparatus

The polarization measurements were carried out in a constant-temperature room at an average temperature of 24°C (75°F). The test solution was normal (about 5 weight %) sulphuric acid as used by Prazak⁽¹⁰⁾, Greene⁽¹¹⁾ and others as a standard solution in polarization measurements on steels. Tests were also carried out in normal sulphuric acid containing 0.5 normal sodium chloride. This concentration of sodium chloride was decided upon after exploratory tests, using both higher and lower levels, performed on unalloyed AISI Type 430 steel.

The glass test vessel is shown in Figure 1. It contains 1.6 litres of solution in all and is modelled after one used by Engell and Stolica⁽¹³⁾. The smaller compartment, containing a platinum auxiliary electrode, is separated from the remainder of the test vessel by a sintered-glass diaphragm. The centre compartment contains the steel specimen being tested. This compartment is connected directly to a third compartment (containing a nitrogen bubbler) by means of two horizontal arms. For an hour prior to tests, and thereafter, purified nitrogen was bubbled into the solution such that it was deoxygenated and circulated but nitrogen bubbles did not impinge on the test specimens.

A Luggin-Haber probe and two salt bridges were arranged to permit measurement of the potential of the steel specimens with respect to a saturated calomel electrode (Figure 1). A potentiostat (Anotrol, Model 4100) was used to hold the steel specimens at selected potentials. The potentiostat was also equipped so that the specimen potential could be altered, at a linear rate, between two pre-selected voltages. A potentiometric chart recorder (Westronics, Model 511A/U) was used to measure continuously the impressed currents necessary to produce the required voltages.

Test Procedures

The test specimen was placed in the nitrogenated sulphuric acid solution and allowed to corrode freely for one hour. The corrosion potential E_{corr} was then measured, a Rubicon portable potentiometer being used for this purpose. The following procedures were then employed:

- I. For anodic polarization tests, the potentiostat was set for linear scan. The starting voltage was set at E_{corr} , and the final voltage adjusted to a value 2000 mV more positive (more noble) than this. The scan was completed in 15 min; i.e., the scan rate was 8 volts/hr.

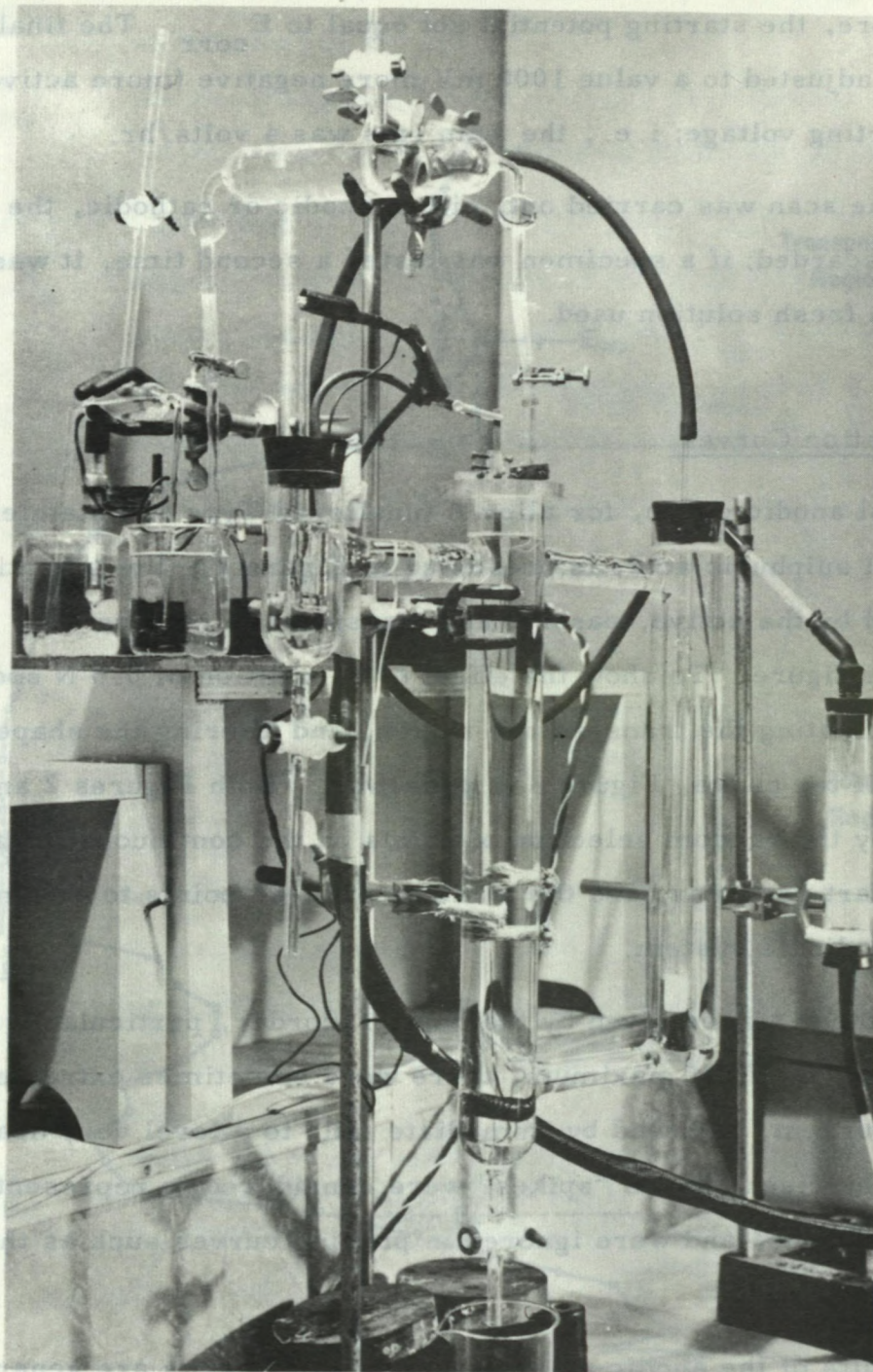


Figure 1. Test vessel used in the polarization measurements. The platinum auxiliary electrode is in the left-hand arm, and the specimen and the probe are in the central arm. The sintered-glass diaphragm used to bubble purified nitrogen into the solutions is in the right-hand arm. The probe is connected, through two salt bridges, to a calomel cell (in left-hand beaker).

II. For cathodic polarization tests, the potentiostat was set for linear scan, and, as before, the starting potential set equal to E_{corr} . The final voltage was adjusted to a value 1000 mV more negative (more active) than the starting voltage; i. e., the scan rate was 4 volts/hr.

After one scan was carried out, either anodic or cathodic, the solution was discarded; if a specimen was tested a second time, it was re-surfaced, and a fresh solution used.

Anodic Polarization Curves

A typical anodic curve, for alloy A (unalloyed Type 430 stainless steel) in normal sulphuric acid, is presented in Figure 2. The potential ranges occupied by the active, passive and transpassive regions are indicated on this figure. To show the effect of an addition of 0.5 N sodium chloride in eliminating the transpassive region, and altering the shape of the remainder of the curve, Figure 3 is presented. Both Figures 2 and 3 were obtained by the random selection of points on the continuous linear traces of the chart recorder, and the transfer of these points to a semi-logarithmic coordinate system.

On the linear traces given by the chart recorder, particularly in the active region near the first maximum, there were sometimes extremely rapid rises of current, followed by immediate falls to a level very nearly that preceding the rise. These "spikes" were considered to represent non-equilibrium conditions, and were ignored in plotting curves such as those shown in Figures 2 and 3.

The results of the anodic polarization measurements are generally presented in tabular form (see Tables 2 and 3), using the parameters indicated on Figures 2 and 3 and described briefly below:

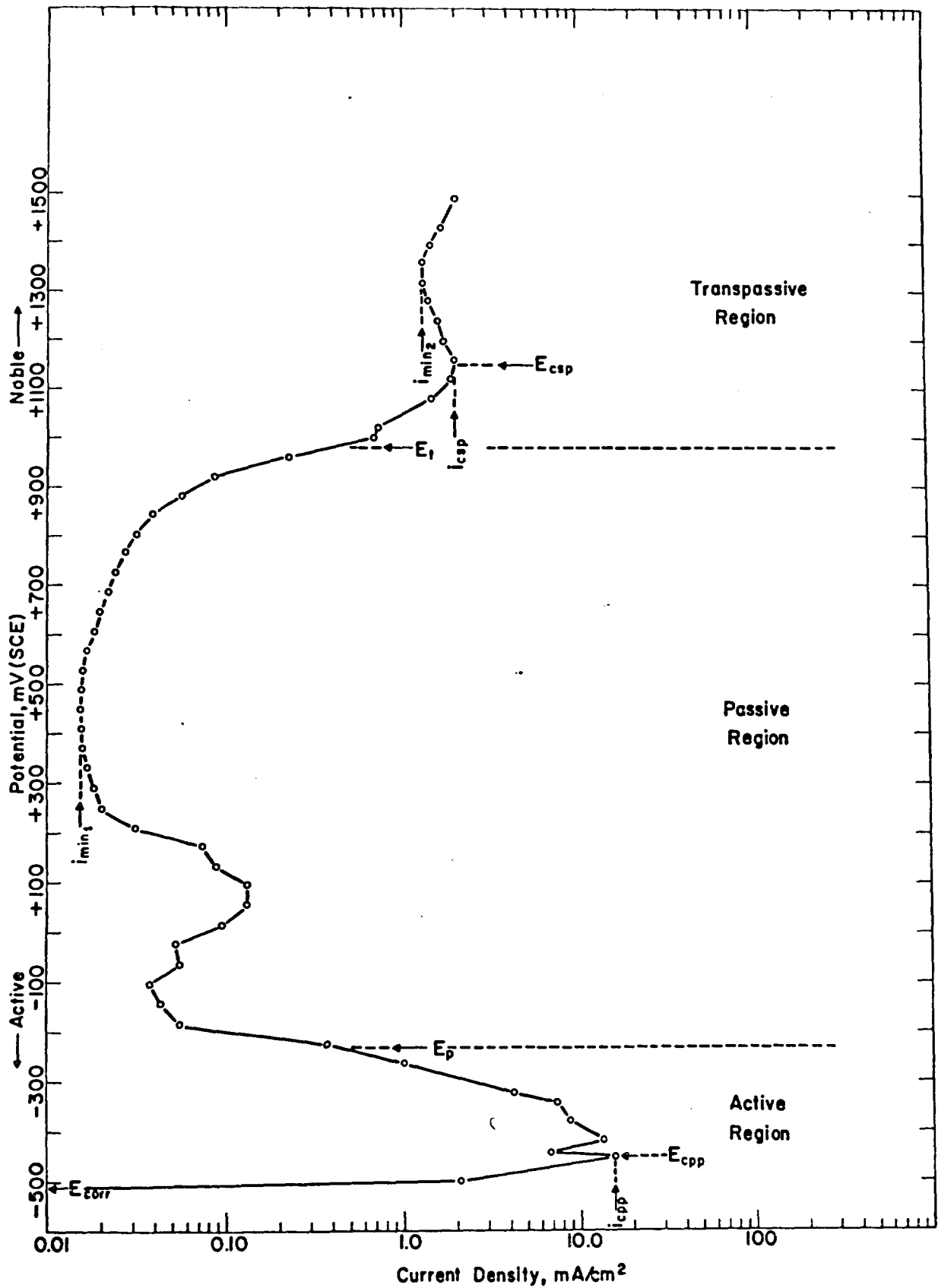


Figure 2. Anodic polarization curve for Alloy A (unalloyed AISI Type 430 stainless steel) in nitrogenated normal sulphuric acid.

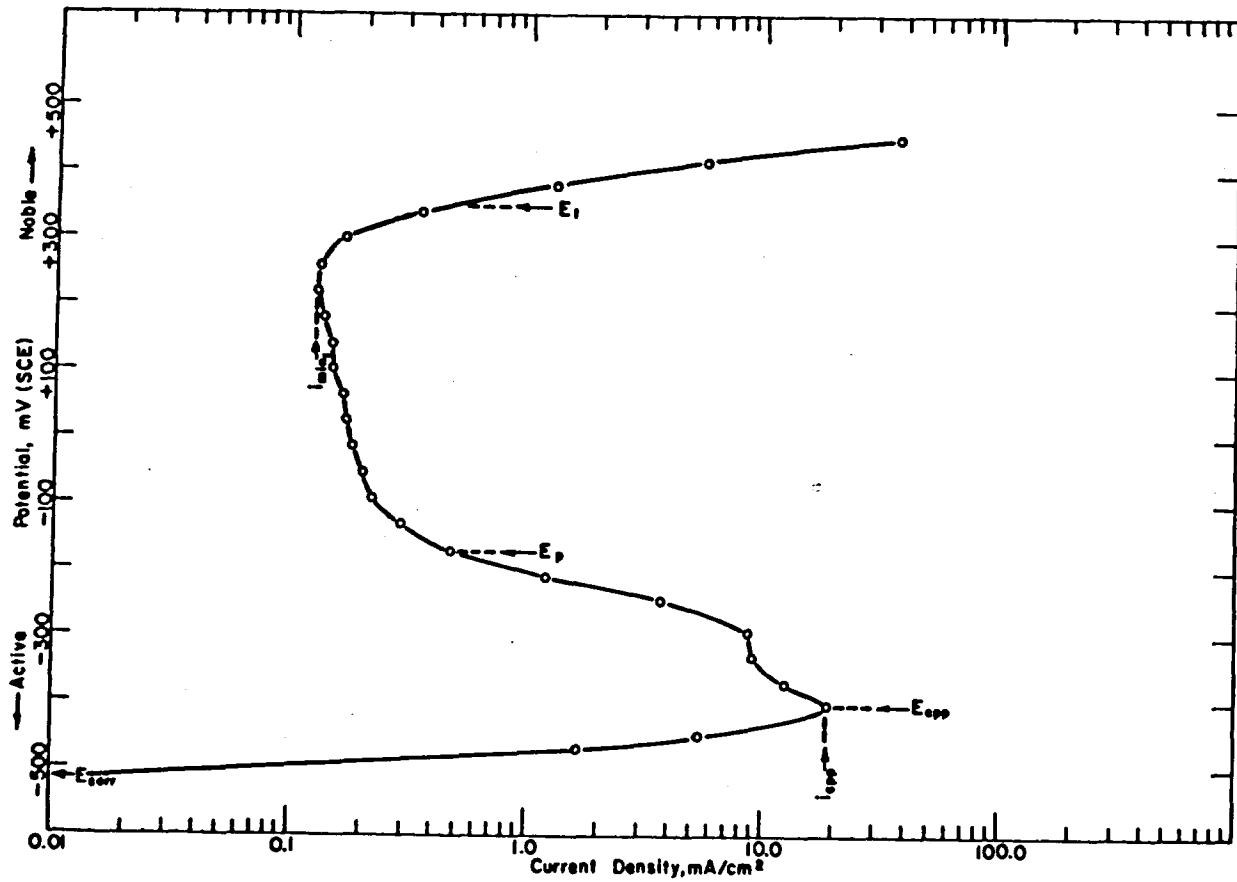


Figure 3. Anodic polarization curve for Alloy A (unalloyed AISI Type 430 stainless steel) in nitrogenated normal sulphuric acid containing 0.5 N sodium chloride.

- E_{corr} - the corrosion potential of the freely corroding specimen at the start of the test.
- E_{cpp} - the critical potential for primary passivation; i. e. the first potential maximum in the anodic polarization curve, at which i_{cpp} occurs.
- E_p, E_t - respectively the potentials at the beginning of passivation (at some potential more noble than E_{cpp}) and the termination of passivation. Both potentials are taken at the arbitrarily-selected current-density level of 0.5 mA/cm^2 .
- E_{csp} - the critical potential for secondary passivation, i. e. the potential at the second maximum in the curve, at which i_{csp} occurs.
- i_{cpp} - the critical anodic current density for primary passivation, i. e. the current density at the first maximum on the curve, at E_{cpp} .
- i_{min_1} - the minimum current density in the passive region, i. e. in the region bounded by E_p and E_t .
- i_{csp} - the critical anodic current density for secondary passivation, i. e. the current density at the second maximum on the curve, at E_{csp} .
- i_{min_2} - the minimum current density in the region of secondary passivity, occurring at some potential more noble than E_{csp} .

Cathodic Polarization Curves

A typical cathodic curve, obtained on a specimen of alloy A (unalloyed Type 430 stainless steel) in normal sulphuric acid solution, is presented in Figure 4. As an illustration of the large effects of some of the additions, Figure 5 shows cathodic polarization curves obtained on

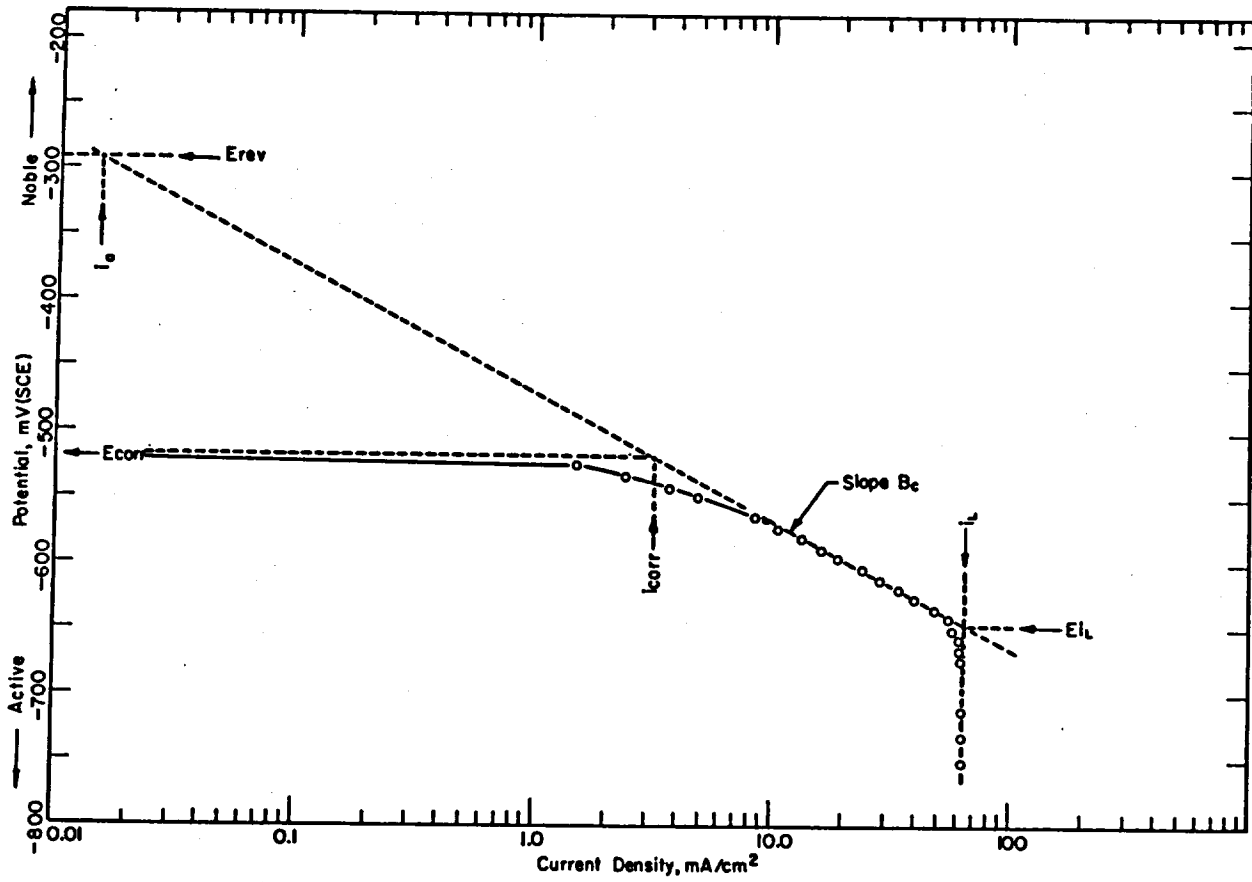


Figure 4. Cathodic polarization curve for Alloy A (unalloyed AISI Type 430 stainless steel) in nitro-nitrogenated normal sulphuric acid.

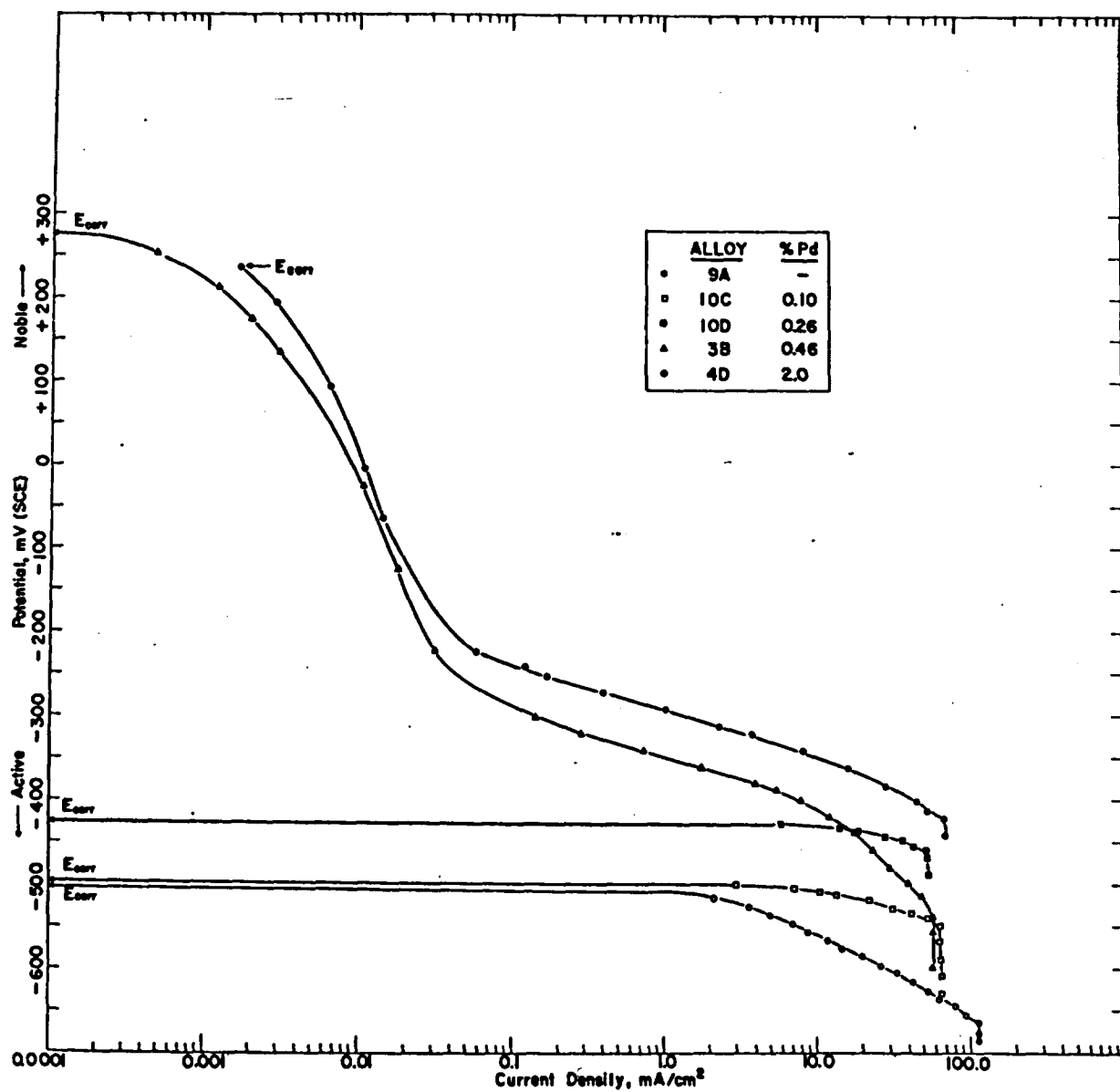


Figure 5. Cathodic polarization curves for Pd-free unalloyed AISI Type 430 stainless steel, and for some Pd-containing steels, in nitrogenated normal sulphuric acid.

Pd-containing alloys (omitting alloys 9B and 4C for simplicity) and a Pd-free control steel. As in the case of Figures 2 and 3, the points of Figures 4 and 5 were obtained by the transfer of points from the continuous linear curves obtained on the chart recorder to a semi-logarithmic coordinate system.

The results of the cathodic measurements are generally reported in tabular form (Tables 4 and 5) in terms of the following parameters (which are also indicated on Figure 4):

- E_{corr} - the potential of the freely corroding specimen, at the start of the test.
- B_c - The Tafel slope of the linear part of the cathodic semi-logarithmic curves, in the units mV/decade.
- i_o - The exchange current, i. e. the current density at the reversible potential for hydrogen reduction in normal H_2SO_4 . From measurements of pH, the reversible potential E_{rev} was taken as -292 mV (with respect to the saturated calomel electrode). Extrapolation of the linear part of the semi-logarithmic cathodic curve to this reversible potential yields i_o . Such an extrapolation is shown in Figure 4.
- i_{corr} - The corrosion current, i. e. the loss of metal by corrosion, represented as a current of dissolving ions. As shown in Figure 4, i_{corr} is obtained by extrapolating the linear part of the semi-logarithmic cathodic curve to the corrosion potential E_{corr} .
- E_{rev} - As seen above, the reversible potential for the hydrogen reduction reaction in normal sulphuric acid, determined to be -292 mV (saturated calomel electrode).

Two other parameters, defined below, are shown on Figure 4. However, it was not clear in all cases whether the current maxima in the cathodic tests were truly limiting diffusion currents, or merely the maximum currents which the potentiostat could supply. Values of i_L and E_{i_L} were, therefore, not tabulated.

- i_L - The limiting diffusion-current density, obtained at the more active potentials on the cathodic polarization curves.
- E_{i_L} - The voltage at the intersection between a vertical line representing i_L and the extrapolated linear part of the semi-logarithmic curve.

It should be noted that in many cases it was difficult to determine where to draw the straight line of slope B_c , because some of the slopes appeared to be curved or to show two or more straight portions. It was decided, therefore, to use the following guiding principles:

1. Within each group consisting of a control steel and steels containing one particular alloying element, the lines were drawn as nearly as possible to give values of i_{corr} ranking the metals in the same order as given by the relevant corrosion-test results of reference 12.
2. Within each group, the straight lines were drawn as nearly as possible (bearing in mind paragraph 1, above) over the same current-density ranges.

DISCUSSION OF RESULTS

General

The significance of the polarization curves is best considered in conjunction with the results of the parallel corrosion tests, which were carried out in normal solutions of each of H_2SO_4 , HCl and $FeCl_3$ at room

temperature, and in 65% nitric acid at the boil⁽¹²⁾. The principal trends observed in these tests are summarized in Table 1, which is reproduced from reference 12.

Some of the results are best explained in terms of "actual" polarization curves, rather than "measured" curves such as those shown in Figures 2-5. Actual curves represent the anodic and cathodic polarization behaviours taken separately, by elimination of the interaction between the two that occurs in the measured curves, especially in the vicinity of E_{corr} . Greene⁽¹¹⁾ has provided some illustrative examples of the differences between actual and measured curves. An example of hypothetical actual polarization curves for Type 430 steel in normal sulphuric acid (which presents the curves only in the vicinity of the corrosion potential) appears in Figure 6. Some of the anodic parameters are indicated on this figure, which may be contrasted with the measured curves of Figures 2 and 4. There are further examples of hypothetical actual polarization curves in Figures 7 and 8.

A study of the data obtained on the "control" unalloyed steels of Tables 2-5 (which would be expected to exhibit similar behaviour) showed considerable variation in some of the electrochemical parameters. The following guiding principles were therefore accepted:

1. Only the largest effects could be taken as a valid consequence of the presence of an alloying addition.
2. The data should be assessed primarily in terms of trends shown within each group consisting of a "control" steel and three or more steels containing one particular alloying addition at different levels.
3. Confirmation of indicated trends by the results of the corrosion tests⁽¹²⁾ would be of considerable value.

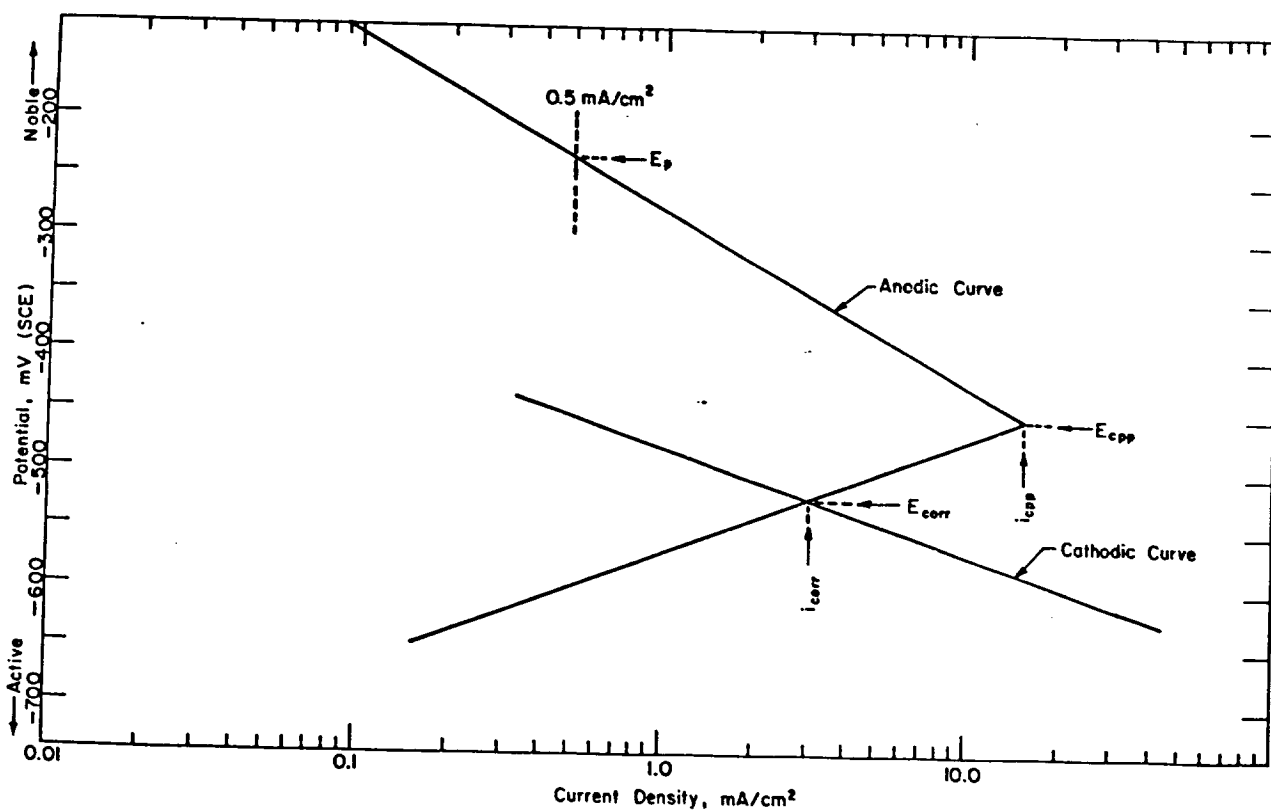


Figure 6. "Actual" anodic and cathodic polarization curves in the vicinity of E_{corr} for Alloy A (unalloyed AISI Type 430 stainless steel) in nitrogenated normal sulphuric acid.

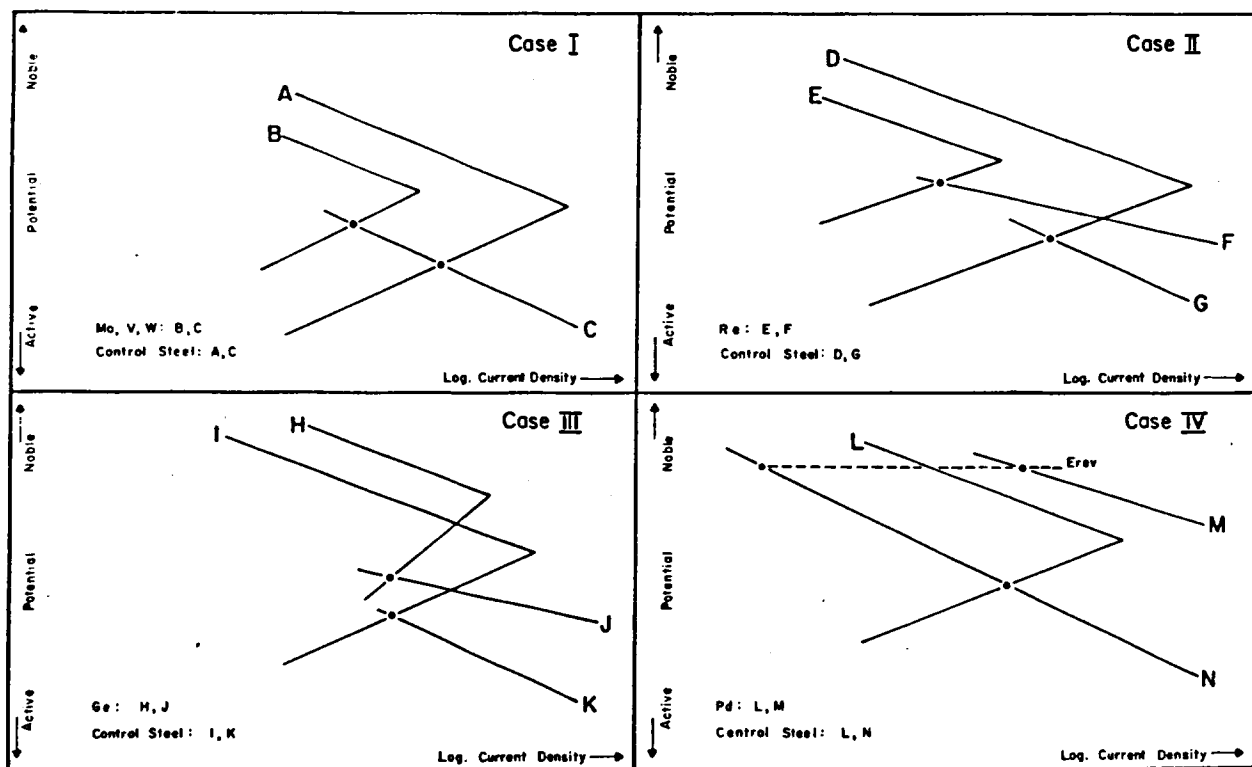


Figure 7. "Actual" anodic and cathodic polarization curves showing the effects of some of the alloying additions in tests in nitrogenated normal sulphuric acid.

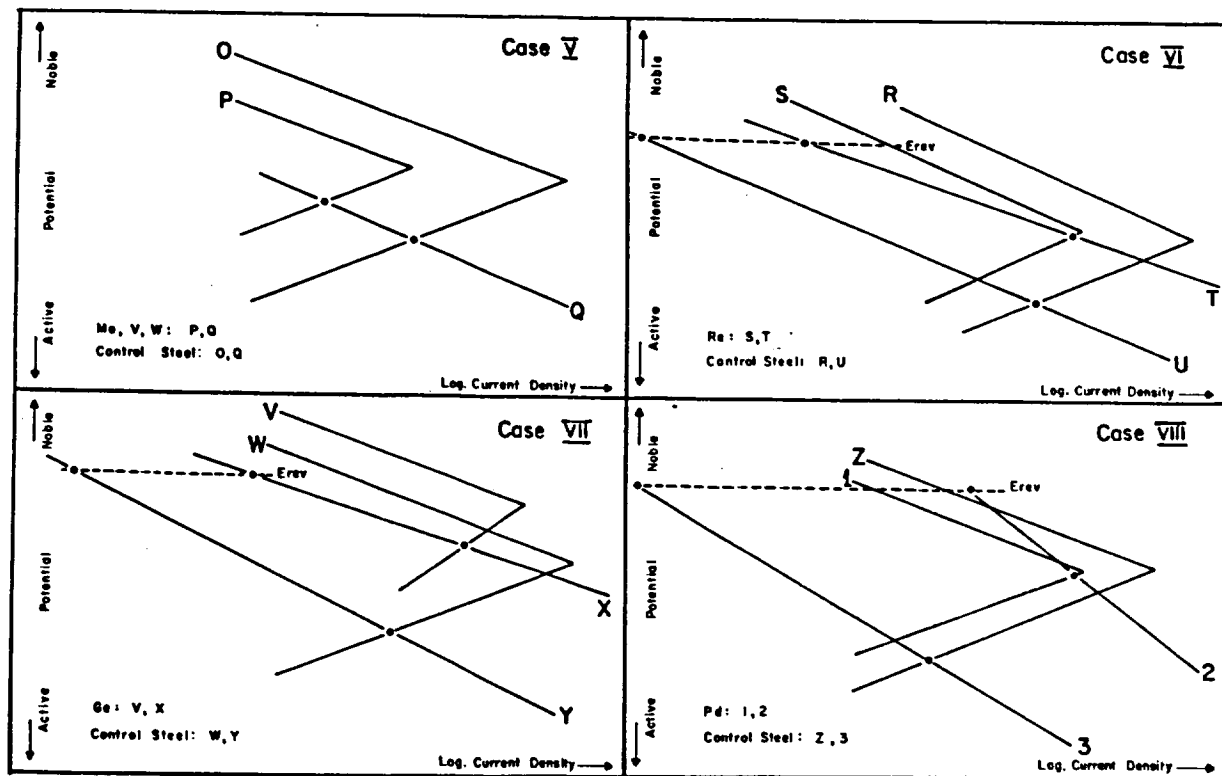


Figure 8. "Actual" anodic and cathodic polarization curves showing the effects of some of the alloying elements in tests in nitrogenated sulphuric acid containing 0.5 N sodium chloride.

In the following paragraphs an attempt is made to describe qualitatively the effects of each of the alloying elements. Thus, the effect reported is in some cases an average of the effects at several levels of the addition. In adopting this approach, it was recognized that a few individual alloys would not fit into the general pattern. However, the most important trends attributable to each addition are thus represented.

Corrosion in Normal Sulphuric Acid

In their effects on anodic and cathodic polarization behaviour as it relates to corrosion behaviour in the active and passive regions, the additions can be divided into four classes, as follows:

1. Mo, V and W. The major effect of these three additions was upon anodic behaviour, while cathodic behaviour was affected to only a minor extent. Specifically, additions of this group brought about the following:

- (a) E_{corr} shifted to a more noble potential.
- (b) E_p shifted to a more active potential.
- (c) i_{cpp} decreased.
- (d) i_{corr} , the active corrosion rate (derived from cathodic data such as shown in Figure 4), decreased.

The effect of each of Mo, V and W therefore resembles qualitatively Case I of the hypothetical actual polarization curves of Figure 7. In this figure, only one cathodic curve appears, representing the fact that these additions did not cause a significant change in cathodic polarization behaviour. The qualitative effect upon anodic behaviour is indicated by the shift from anodic curve A to anodic curve B. It will be noted that this change in anodic behaviour also brings about the shifts in the values of E_{corr} , E_p , i_{cpp} and i_{corr} which were noted above (see Figure 6 for assistance in locating the points on the curves corresponding to these parameters). Agreement

with the trends of the corrosion tests⁽¹²⁾ was also shown.

In Case I of Figure 7, it is also indicated that the addition caused E_{cpp} to shift to a slightly more noble value. Such a shift was observed experimentally for Mo and V, but not for W.

2. Re and Ge. These two additions caused significant changes in both anodic and cathodic polarization behaviour. However, the effects of the two additions were not qualitatively similar in all respects. Considering the anodic behaviour, Re and Ge brought about the following:

- (a) E_{corr} shifted to a more noble potential.
- (b) E_{cpp} shifted to a more noble potential.
- (c) E_p shifted to a more active potential for Re, but shifted to a more noble potential for Ge.
- (d) i_{cpp} decreased.

Considering the cathodic polarization behaviour, Re and Ge brought about:

- (e) Decreased values of B_c .

Considering its behaviour in the weight-loss corrosion tests⁽¹²⁾, Re, at its higher levels, brought about improved resistance to corrosion in normal sulphuric acid, while Ge did not. Therefore:

- (f) Decreased active corrosion rate i_{corr} for Re but not for Ge (see also Table 4).

The effects of Re upon polarization behaviour are indicated qualitatively in the hypothetical actual curves of Figure 7, Case II. It will be seen that these curves are drawn so that the Re additions yield a lower corrosion rate and also cause the shifts in the polarization parameters which were noted above (refer to Figure 6 for aid in locating the points on the graphs corresponding to the parameters).

The effect of Ge is indicated qualitatively as Case III of Figure 7. It will be noted that Ge is shown to cause profound changes of both cathodic and anodic polarization behaviour. However, as the curves are drawn in Figure 7, there is no change in active corrosion rate. All the other parameters are seen to shift, as noted under headings (a) to (e).

It might be further remarked that, at the highest addition level, both Re and Ge brought about increased values of i_o (see Table 4). This is not indicated in Figure 7, Cases II and III, which represent the average rather than the extreme behaviour of the two additions.

It is perhaps worth mentioning that, for the Ge-bearing steels, in repeat scans (i. e. immediately after the specimen had gone through the transpassive range in a normal scan) the polarization behaviour of the specimens was quite different from that obtained initially. Specifically, the specimen remained shiny; i_{cpp} dropped to 0.14 mA/cm^2 and i_{min_1} to 0.007 mA/cm^2 . It is not known whether specimens of other alloys would have reacted comparably to repeat scans.

3. Pd. Because spontaneous passivity was achieved in normal sulphuric acid at levels of 0.46% Pd or more, it was not possible to determine the average effect of Pd upon anodic polarization behaviour from the simple scans carried out. There were, however, definite effects of Pd upon cathodic polarization behaviour which are cited below.

Considering the cathodic behaviour of the Pd alloys at the three highest levels of Pd, the following were observed to be caused by Pd additions:

- (a) E_{corr} rose to a noble potential in the range +235 to +275 mV, SCE.
- (b) Decreased values of B_c .
- (c) Increased values of i_o .
- (d) i_{corr} decreased to insignificantly low values in the passive potential region.

Case IV of Figure 7 indicates the average effects of the higher levels of Pd on the actual polarization curves. It is seen that, in order for passivity to be achieved, the Pd additions must shift the cathodic curve to the right and above the anodic curve (tentatively assumed to be unchanged), as is shown in this diagram for curve M. Only such a relationship can bring about passivity, with its attendant extremely low values of i_{corr} at noble values of E_{corr} . This behaviour is shown in the measured curves of Figure 5, whereas Figure 7, Case IV, also indicates the other parameter shifts referred to under headings (a) to (d) above.

4. Ta and Si. These two additions had very little effect on either the cathodic or the anodic polarization behaviour in the active region. In agreement with this, the relevant corrosion tests⁽¹²⁾ showed no significant beneficial effect upon corrosion resistance because of additions of either of these two elements.

Corrosion in Normal Sulphuric Acid Plus 0.5 N NaCl

In the following paragraphs, the effects of the alloying additions upon polarization behaviour as it affects corrosion in the active region are discussed. For the chloride-containing solutions there was assumed to be a parallel between the polarization results and the results obtained in the weight-loss corrosion tests in normal hydrochloric acid⁽¹²⁾. For purposes of discussion, the additions can be grouped (as before, in dealing with the results in the chloride-free solution) as follows:

1. Mo, V and W. These three additions, on the whole, showed the same behaviour as that observed in chloride-free H_2SO_4 ; that is, they appeared to affect anodic but not cathodic polarization behaviour, as shown qualitatively in Case V of Figure 8 (which is equivalent to Case I of Figure 7). It should be noted that, for V and W, the decreases in i_{cpp} were less marked than in the case of Mo. However, all three additions caused E_{cpp} to shift to more noble values. The net results, in terms of both i_{corr} values and the

data from the corrosion tests⁽¹²⁾, indicated that Mo, V and W were beneficial. It therefore appears that, on the whole, their effect corresponds to that brought about by a change from anodic curve O to anodic curve P, cathodic curve Q remaining unaltered.

2. Re and Ge. These two elements can be grouped together, because they produced somewhat similar (though not identical) effects on both anodic and cathodic polarization behaviour. Changes in the anodic and cathodic parameters resembled those listed previously, under headings (a) to (e), in discussing the results obtained in the tests in chloride-free H_2SO_4 . It must be noted, however, that in the chloride-containing solution neither Re nor Ge brought about decreased corrosion rates. Rather, from the results of the weight-loss corrosion tests in normal hydrochloric acid⁽¹²⁾ and from the i_{corr} values of Table 5, both additions were deleterious; i. e., i_{corr} was generally increased. At the same time, the higher levels of both additions caused an effect not observed to such an extent in chloride-free H_2SO_4 , namely, increased values of the exchange current i_o .

Case VI of Figure 8 represents qualitatively the behaviour of the Re additions in bringing about the shifts noted in the polarization parameters, while Case VII represents qualitatively the effect of the Ge additions.

3. Pd. Although discussed separately, this element could be considered as falling broadly into the same group as Re and Ge; that is, both anodic and cathodic polarization curves were affected by Pd. Passivity was not brought about in the chloride-containing solutions (unlike behaviour in the chloride-free solutions), and corrosion rates were (as with Re and Ge) increased. However, the effect of Pd was somewhat more erratic than the effects of Re and Ge and can be summarized as follows:

- (a) E_{corr} shifted to more noble potentials.
- (b) E_{cpp} shifted to more active potentials, except for the steel with 1.91% Pd, which showed a strong shift in the noble direction.
- (c) E_{p} was either unaffected or shifted unpredictably in the noble direction.
- (d) i_{cpp} values were erratic at the lower Pd levels, and were somewhat decreased at the higher Pd levels.
- (e) Values of B_{c} were increased (compare decreases caused by Re and Ge).
- (f) Values of i_{o} were strongly increased.
- (g) Values of i_{corr} were strongly increased.

Some (not all) of the above effects of Pd additions on the polarization behaviour are shown or can be deduced from the hypothetical actual polarization curves of Case VIII, Figure 8. It is seen that passivity is almost, but not quite, brought about by the Pd, resulting in considerably higher corrosion rates.

4. Ta and Si. As noted in the tests in chloride-free H_2SO_4 , these two additions failed to produce significant changes in the cathodic polarization behaviour, and also in the anodic behaviour in the active region. In agreement with this, the weight-loss corrosion tests in normal hydrochloric acid⁽¹²⁾ failed to show significant beneficial effects from additions of Ta and Si.

Corrosion in Normal FeCl₃—

In the corrosion tests in FeCl₃, only Mo and, to a lesser extent, Re additions brought about significant improvements in corrosion resistance.

From a determination carried out on specimen A (unalloyed Type 430 steel), the corrosion potential in normal FeCl₃ at room temperature was approximately -75 mV. It appears, therefore, that in terms of the anodic data of Table 3, good resistance to FeCl₃ should be correlated with a low anodic current density in the vicinity of this potential. Without making that particular measurement, it appears that the alloys showing the most active E_p values and also the greatest effect in lowering i_{c_{pp}} and i_{min₁} in the presence of chlorides (see Figure 3) might be expected to show the best resistance to corrosion in FeCl₃.

It appears significant, therefore, that Mo and Re brought about the most active values of E_{c_{pp}} and also the lowest values of i_{c_{pp}}, both in the chloride-containing solutions (Table 3). As to the third parameter mentioned above, i_{min₁}, Mo brought about low values, while Re did not; as noted above, the action of Mo in conferring increased corrosion resistance in FeCl₃ solution was more marked than that of Re⁽¹²⁾.

Conversely, the Pd-containing alloys showed the poorest corrosion resistance in FeCl₃ solution. In keeping with this, Pd additions showed a tendency to produce more noble (rather than more active) values of E_p, an erratic effect upon i_{c_{pp}}, and the highest values of i_{min₁} attributable to any of the alloying additions.

It is also of interest that, in earlier work⁽¹⁴⁾, polarization measurements on uranium-bearing Type 430 stainless steels showed that each of 0.24% and 0.55% uranium brought about the same shifts in electrochemical parameters as did those observed for Mo and Re; that is, E_p was made more active, and i_{c_{pp}} and i_{min₁} showed lower values in nitrogenated normal sulphuric acid containing 0.5 normal sodium chloride. In keeping with this, the uranium additions were observed to bring about increased corrosion

resistance in normal ferric chloride solution. Alloys containing 1% and 2% Mo, in this work, brought about more active values of E_p and lower values of i_{cpp} as compared with those shown by the uranium-bearing steels. Corresponding to this, the corrosion resistance of these alloys in normal ferric chloride solution at room temperature was greater than that shown by the uranium-bearing steels.

It appears, therefore, that the results of the polarization measurements in chloride-containing acid, both in the present investigation and in the previous work⁽¹⁴⁾, can be correlated satisfactorily to corrosion resistance in normal ferric chloride solution.

Corrosion in Nitric Acid

As indicated in the corrosion measurements⁽¹²⁾, none of the additions produced improved resistance to corrosion in nitric acid; some were detrimental. Corrosion in boiling 65% nitric acid proceeds at E_{corr} values in the vicinity of +1000 mV SCE⁽¹⁵⁾. Any relationship between corrosion in this solution and the room-temperature anodic polarization behaviour in sulphuric acid might, therefore, be expected to be shown by the parameters determined in the transpassive region; namely, E_t , E_{csp} , i_{min_2} and i_{csp} (see Table 2 and Figure 2).

However, there did not appear to be any correlation between the effects of the additions upon E_t and E_{csp} , and behaviour in the boiling 65% nitric acid. Nor was there a consistent correlation with the effect of the additions upon i_{csp} , though it was noteworthy that, when the additions produced large increases in i_{csp} , higher corrosion rates in the nitric acid also resulted (for Si, Pd and Ge). A fairly consistent increase in i_{min_2} was exhibited in cases where the additions brought about high corrosion rates in nitric acid. However, this parameter is measured at a higher potential than that attributed to corrosion in nitric acid⁽¹⁵⁾. The apparent correlation, therefore, may have been fortuitous.

The general lack of correlation between the polarization measurements and corrosion behaviour in boiling 65% nitric acid is probably due, for the most part, to differences in the kinetics of the two processes. In the polarization measurements, the specimens are brought through the relevant potential range in approximately 2 min. The corrosion tests are carried out in successive 48-hr periods.

SUMMARY AND CONCLUSIONS

In research on the effect of various alloying additions upon the polarization behaviour of AISI Type 430 stainless steel in normal sulphuric acid, with and without additions of sodium chloride, the following main trends were noted:

1. Each of Mo, V, W, Re and Ge produced marked changes in anodic polarization behaviour in the chloride-free sulphuric acid. In the chloride-containing acid, the additions producing the greatest effect upon anodic polarization behaviour were Mo and Re.
2. Each of Pd, Re and Ge produced marked changes in cathodic polarization behaviour in both the chloride-free and chloride-containing solutions.
3. It was found possible to relate the changes in electrochemical parameters brought about by the additions to the behaviour of the same steels in weight-loss corrosion tests in normal solutions of sulphuric acid, hydrochloric acid, and ferric chloride⁽¹²⁾.

ACKNOWLEDGEMENT

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TABLE 1

Chemical Analyses of the AISI Type 430 Stainless Steels

Steel No.	Basic Composition						Alloying Addition	Steel No.	Basic Composition						Alloying Addition
	% C	% Cr	% Mn	% P	% S	% Si			% C	% Cr	% Mn	% P	% S	% Si	
A B C D	0.06	17.38	0.80	0.013	0.018	0.42	- 0.70% Mo 1.48% Mo 3.11% Mo	7A 7B 8C 8D	0.09	17.02	0.53	0.003	0.024	0.37	- 0.59% Re* 1.15% Re* 1.84% Re*
I J K L	0.07	17.39	0.84	0.011	0.028	0.62	- 0.67% V 1.13% V 2.18%	9A 9B 10C 10D							- 0.06% Pd 0.10% Pd 0.26% Pd
M N O P	0.06	17.31	0.76	0.012	0.023	0.47	- 0.47% W 0.94% W 2.30% W	3B 4C 4D	0.06	16.94	0.76	0.014	0.019	0.44	0.46% Pd 0.99% Pd 1.91% Pd
Q R S T	0.06	17.40	0.82	0.015	0.022	0.48	- 0.41% Ta 0.72% Ta 1.37% Ta	5A 5B 6C 6D							- 0.41% Ce 1.03% Ce 2.01% Ce
U V W X	0.06	17.52	0.64	0.018	0.024	0.39 0.84 1.33 1.78	- Si Si Si	6266**	0.06	19.47	1.77	0.017	0.022	0.57	10.19% Ni

* X-ray analysis.

** AISI Type 304 steel.

(Reproduced from prior Mines Branch Technical Bulletin,
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APPENDIX I