Mines Branch Technical Bulletin TB 74

A COMPARISON OF THE EFFECTS OF URANIUM AND MOLYBDENUM
ALLOYING ADDITIONS ON THE CORROSION RESISTANCE

OF AISI TYPE 430 STAINLESS STEEL

by

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ABSTRACT

To compare the effects of uranium and molybdenum alloying additions, laboratory corrosion tests were performed on AISI Type 430 stainless steels containing 0.24% and 0.55% uranium, and 1.02% and 2.03% molybdenum. In comparison with a similar unalloyed steel, both additions brought about qualitativelysimilar improvements of corrosion resistance both in non-oxidizing sulphuric acid and hydrochloric acid solutions and oxidizing ferric chloride solution. In these solutions, the corrosion resistance of the molybdenum-bearing steels was definitely superior to that of the uranium-bearing steels. In tests in boiling 65% nitric acid, the uranium-bearing steels and the steel containing 1.02% molybdenum showed excellent corrosion resistance, resembling that of the unalloyed steel. However, the steel containing 2.03% molybdenum showed extremely poor corrosion resistance in this medium. Anodic polarization measurements on the steels yielded results which appeared to correlate with the behaviour of the steels in the corrosion tests in oxidizing solutions containing the chloride ion.

The uranium content for maximum corrosion resistance appears to lie at 0.5% or less. It is therefore clear that molybdenum is capable of imparting greater improvements in corrosion resistance to AISI Type 430 stainless steel than uranium, although higher levels of molybdenum are necessary to achieve this.

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Direction des mines

Bulletin technique TB 74

COMPARAISON DES EFFETS DES ADDITIONS D'URANIUM ET DE MOLYBDÈNE SUR LA RÉSISTANCE À LA CORROSION DE L'ACIER INOXYDABLE DE TYPE AISI 430

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RÉSUMÉ

Pour comparer les effets des additions d'uranium et de molybdène, les auteurs ont procédé en laboratoire à des épreuves de corrosion sur des aciers inoxydables de type AISI 430 à teneurs en uranium de 0.24 p.100 et 0.55 p.100 et en molybdene de 1.02 p.100 et 2.03 p.100. Comparativement à un acier semblable non allié, les deux additions ont amélioré la résistance à la corrosion dans des solutions non oxydantes d'acide sulfurique et d'acide chlorhydrique et dans une solution oxydante de chlorure ferrique. Dans ces solutions, la résistance à la corrosion des aciers au molybdène est nettement supérieure à celle des aciers à l'uranium. Lors d'épreuves dans l'acide nitrique bouillant à 65 p. 100, les aciers à l'uranium et l'acier à teneur de 1.02 p.100 en molybdène montrent une excellente résistance à la corrosion qui ressemble à celle de l'acier non allié. Cependant, l'acier à teneur de 2.03 p. 100 en molybdène offre une pauvre résistance à la corrosion dans ce milieu. Des mesures de polarisation anodique effectuées sur les aciers ont donné des résultats qui semblent concorder avec le comportement des aciers dans les épreuves de corrosion dans les solutions oxydantes qui renferment l'ion chlorure.

La teneur en uranium qui donne la résistance maximum à la corrosion paraît être 0.5 p.100 ou moins. Il est évident par conséquent que le molybdène peut, plus que l'uranium, améliorer la résistance à la corrosion des aciers inoxydables de type AISI 430 quoique, pour l'obtenir, il faille utiliser de plus fortes proportions de molybdène.

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INTRODUCTION

Because of a decreased demand for uranium towards the close of the nineteen-fifties, the Canadian Government and the uranium industry undertook large-scale research which had as its objective the development of new non-nuclear uses for the metal. The initial results obtained in research on uranium-bearing steels have been comprehensively reviewed There were indications from this work that uranium alloying additions brought about minor improvements in the corrosion resistance of some non-austenitic steels in acid solutions.

Some of the most definite effects were observed when uranium was alloyed with AISI Type 430 chromium stainless steel. Subsequent results obtained with this steel (3) indicated that an optimum level of 0.1-0.5% uranium brought about improved corrosion resistance in non-oxidizing sulphuric acid and hydrochloric acid solutions and in oxidizing ferric chloride solution, and also improved resistance to crevice corrosion in 3% sodium chloride solution.

Similar corrosion tests on two commercial AISI Type 430 steels, one molybdenum-free and the other containing 0.76% molybdenum, indicated that the effects of molybdenum and uranium additions were qualitatively similar. Since molybdenum additions to ferritic stainless steels such as AISI Type 430 are known to confer improved corrosion resistance in service applications (4,5,6), it was decided to carry out additional measurements with the object of defining more clearly the relative effects of uranium and molybdenum. Such a comparison would necessitate the use of molybdenumbearing steels having higher molybdenum contents than the one examined previously, as it appeared probable, from the scanty amount of data available, that the molybdenum content for optimum corrosion resistance was considerably higher than 0.76%.

It was decided that anodic polarization measurements, of a type designed to predict aqueous corrosion behaviour (7), would be carried out concurrently with the corrosion tests. This was done in order to confirm the results of the corrosion tests and also to obtain experience in the use of such methods for the initial screening of alloys of novel composition and/or non-standard heat treatment and thus of unknown corrosion resistance.

EXPERIMENTAL

1. Materials

The steels were prepared in the Physical Metallurgy Division as 50-lb aluminum-deoxidized melts in an induction furnace. Target compositions for the two uranium-bearing steels were 0.25% and 0.5% uranium, and target compositions for the molybdenum-bearing steels were 1.0% and 2.0% molybdenum. Chemical analyses of the steels are presented in Table 1.

Forging and rolling were carried out at 1010-1180°C (1850-2160°F) after which the 1/4-in, -thick rolled steels were heat-treated for 4 hours at 790°C (1450°F) and furnace cooled. Small flat specimens - as described further below - were then machined so that their major faces lay in the plane of rolling.

2. Corrosion Tests

The specimens used in the corrosion tests were rectangular and were provided with small holes for suspension during tests. Prior to tests, all specimen surfaces were ground on water-cooled 120 grit silicon carbide papers, then degreased in ultrasonically agitated carbon tetrachloride and rinsed in alcohol to facilitate hot air drying. Specimen areas were calculated from dimensional measurements made using a micrometer and were usually 6-8 cm². Specimens were weighed, before and after testing, on an analytical balance accurate to ±0.1 mg.

Most of the tests were carried out in a large Pyrex jar containing 15 liters of test solution. The jar was provided with a tightly fitting Lucite lid, and specimens were suspended from a Lucite turntable reolving at a rate of $8\frac{1}{2}$ revolutions per hour (Figure 1). In each of the tests in this apparatus, 15 corrosion coupons were exposed simultaneously, i. e. three replicates of each of the five steels under investigation. Each specimen was suspended from the rotating turntable by means of a Teflon fluorocarbon strip. The tests in normal solutions of sulphuric acid, hydrochloric acid and ferric chloride were carried out in stagnant solutions at the room temperature of $24^{\circ}\text{C} \pm 1^{\circ}$ ($75^{\circ}\text{F} \pm 2^{\circ}$). The crevice corrosion test in 3% sodium chloride solution was carried out at a temperature of $50^{\circ}\text{C} \pm 1^{\circ}$ ($122^{\circ}\text{F} \pm 2^{\circ}$), rather than room temperature, in order to hasten the rate of attack (3), and oxygen was bubbled into the jar during the test at a rate of about 40 ml/min. In this test, each specimen was held between two sheets of glass by means of rubber bands (see Figure 1).

The Huey test was carried out in 65% nitric acid on single specimens suspended from Teflon fluorocarbon resin strips into 400 ml of the boiling solution for as many as five successive 48-hour periods. Fresh solution was used for each 48-hr exposure.

3. Polarization Measurements

Specimens used in the polarization measurements were rectangles with a thin shaft extending along one edge, to permit the passage of electrical current into the specimen while it is immersed in a test solution. The polarization specimens, which had a total immersed area of about 8 cm², were given the same surface pretreatment as were the rectangular corrosion test specimens described above.

The polarization test vessel held 1.6 litres of test solution (Figure 2). It was provided with three vertical arms, one of which held the platinum auxiliary electrode, which was separated from the remainder of the

vessel by a sintered glass diaphragm. The steel specimens were positioned in the centre arm of the vessel, and a Luggin probe and a salt bridge were arranged so that the potential of the specimen could be measured with respect to a saturated calomel electrode. The third vertical arm was connected directly to the centre arm by two horizontal tubes and contained a gas dispersion tube through which purified nitrogen was bubbled into the vessel during tests. This caused the liquid in the main vessel to circulate, but avoided impingement of gas bubbles on the steel specimens. The measurements were all carried out at the room temperature of 24°C ±1°C (75°F ±2°F).

A vacuum tube voltmeter (Simpson, Model 311) was used for trial potential measurements, and a potentiostat (Anotrol, Model 4100) was used to hold the specimens at desired potentials by means of an electronically adjusted electrical current, which flowed from the steel specimens to the platinum auxiliary electrode or vice versa. When desired, the electrical current was recorded continuously on a potentiometric chart recorder (Westronics, Model 511A/S).

The potentiostat was provided with a linear scanning drive so that "potentiodynamic" experiments could be performed, i.e., the potential could be varied at a constant rate over a selected range, and the current measured continuously.

In the anodic polarization measurement the experimental procedure was as follows:

- (a) Purified nitrogen was bubbled through the normal sulphuric acid test solution (which sometimes contained 0, 5 N sodium chloride solution as well) for 1 hour at a rate of 8 ml/min. This process was continued for the remainder of the experiment.
- (b) The specimen to be tested was then immersed for 1 hour in an unpolarized state, after which the corrosion potential E was measured, using the vacuum tube voltmeter.

- (c) The potentiostat was adjusted so that, in a linear scan of 15 min duration, the initial potential was E corr, and the final potential some value 1.5 volts more noble than E corr. The scan rate was therefore 6 volts/hr.
- (d) The density of the electrical current passing through the metal specimen during the scan, and the potentials and current densities defined below and indicated in Figure 3, were calculated from the curves traced out on the chart paper.

E cpp : the potential at the critical current density for primary passivation

E, E, in the potentials at the active and the noble limits of the passive region, which is arbitrarily defined as lying below a current density of 0.1 mA/cm

i the critical current density for primary passivation

i the minimum current density observed in the passive region

The above symbols and quantities were based on those used in Réferences 7 and 10.

RESULTS

1. Corrosion Tests

The weight losses in 4-hour corrosion tests in normal room-temperature solutions of hydrochloric acid, sulphuric acid, and ferric chloride are listed in Table 2. An examination of specimen surfaces, after the tests, showed that the addition-free steel 5555 was considerably more roughened by sulphuric acid and hydrochloric acid than the other four steels. After the test in ferric chloride, steel 5555 showed the most pits and the

molybdenum-bearing steels the fewest,

The weight changes observed in the 7-day crevice corrosion test in oxygenated 3% sodium chloride solution at 50°C (122°F) are listed in Table 3, which also presents some details concerning the appearance of the specimens after the test.

Table 4 presents the weight losses observed in as many as five successive 48-hr periods of the Huey test, which is carried out in boiling 65% nitric acid. Metallographic sectioning showed that the steel with 2.03% molybdenum, which survived only one 48-hr exposure, had experienced strong intergranular corrosion attack.

2. Polarization Measurements

Single polarization scans, as described previously, were performed on each of the steels in nitrogenated normal sulphuric acid at room temperature. A similar series of single scans was carried out in normal sulphuric acid containing 0.5 N sodium chloride. The results, in terms of the quantities indicated in Figure 3, are presented in Table 5.

DISCUSSION

1. Effect of Uranium

The results of the corrosion tests confirm and extend previous observations (2,3) on the effect of uranium alloying additions to AISI Type 430 stainless steels. The improvements in corrosion resistance brought about by 0.24% and 0.55% uranium in ferric chloride solution and in crevice corrosion in 3% sodium chloride solution are of practical significance because this steel type is generally employed in oxidizing environments. The results of the corrosion tests in boiling 65% nitric acid indicated that as much as 0.55% uranium could be tolerated in nitric acid service, which is a common

application for AISI Type 430 stainless steel. Significant improvements in corrosion resistance in non-oxidizing solutions were also brought about by the uranium additions, in agreement with previous findings (2,3).

Considered in conjunction with previous results, the uranium contents of the two steels investigated lay in the optimum range, and thus could be assumed to show the maximum improvement in corrosion resistance obtainable in AISI Type 430 stainless steel by alloying with uranium.

2. Effect of Molybdenum

The results of the corrosion tests with the steels containing 1.02% and 2.03% molybdenum confirmed previous observations of a qualitative similarity between the effects of uranium and molybdenum alloying additions in AISI Type 430 stainless steels (3). However, molybdenum additions conferred significantly greater resistance to corrosion than uranium in all four solutions in which uranium additions were beneficial, i.e. in non-oxidizing hydrochloric and sulphuric acid solutions, in oxidizing ferric chloride solution, and in crevice attack in 3% sodium chloride solution. The corrosion tests in boiling 65% nitric acid indicated that alloying additions of 1% molybdenum, but not 2%, could be tolerated in nitric acid service.

The literature contains very little quantitative information on the effect of molybdenum additions on the aqueous corrosion of plain chromium stainless steels, though a beneficial effect on corrosion in hydrochloric acid, sulphuric acid and acetic acid has been reported 18. The effect of molybdenum as an alloying element in chromium-nickel stainless steels is, of course, well known 19.

3. Polarization Measurements

In the past decade, polarization methods have been used extensively to predict corrosion behaviour, and recently a number of reviews on the subject have appeared -- for example, those in References 7, 10, 11 and

12. A detailed consideration of these methods is beyond the scope of this report, but a few general remarks regarding the interpretation of the anodic polarization curves follow.

Polarization measurements on ferrous alloys are often carried out in deoxygenated normal sulphuric acid, as in the present work. If a steel specimen immersed in this solution is polarized in the noble direction, starting from the corrosion potential $E_{\rm corr}$, an S-shaped anodic polarization curve similar to that shown in Figure 3 is obtained. It must be kept in mind that the shape of a potentiodynamic anodic polarization curve can be highly dependent upon the rate of change of the voltage with time (13). However, when this factor is held constant, as in the present work, comparisons can be made between different alloys.

AISI Type 430 stainless steels are generally used in oxidizing environments in which passivity - and consequently a low corrosion rate - is maintained. Hence, the ease of passivation of these steels is a crucial factor. It is known that an increased ease of passivation is correlated with decreased values of i and/or shifts in E to more active potentials and that the most important effect of a sufficient chloride addition is to shift E_t to more active potentials, thus contracting the passive zone (14). Pitting corrosion would be expected at potentials more noble than E_t , and increases in i might also be brought about.

In terms of the foregoing, the results in the normal sulphuric acid solutions indicated that both uranium and molybdenum additions had conferred an increased ease of passivation. This showed up principally in reductions of the critical current density for primary passivation i cpp. The lowering of i as a result of the molybdenum additions was distinctly greater than that brought about by the uranium additions.

In the solutions containing 0.5 N sodium chloride, the i cpp values of the steels were arranged in about the same order as that observed in the chloride-free solutions, and had the same significance as that noted above.

In addition, the effect of the chloride ions in raising i min, the minimum current density observed in the passive region, was much greater for addition-free steel 5555 than for the other steels. The molybdenum additions (though not the uranium additions) were observed to have brought about broader passive regions, as defined by the potential difference between E_{p} and E_{t} .

The results could be taken, therefore, to indicate that the relative ease of passivation of the steels in suitable chloride-containing solutions is in the order molybdenum-bearing > uranium-bearing > addition-free. This parallels the relative corrosion resistances of the steels which were observed in the corrosion tests in the oxidizing environments containing chloride, i.e. the ferric chloride solution and the crevice corrosion tests in 3% sodium chloride solution. Although the relative corrosion resistances of the steels in the active state - i.e. in the non-oxidizing solutions of hydrochloric acid and sulphuric acid - showed the same ranking as that noted above, deduction of this from polarization measurements would have required a different procedure, which was not attempted.

CONCLUSIONS AND FUTURE PROGRAM

This work, like parallel research on resulphurized chromium stainless steels (15), showed that uranium alloying additions are capable of conferring increased corrosion resistance in some aqueous solutions. The present work indicates that, in AISI Type 430 chromium stainless steel, optimum additions of 0.25-0.5% uranium have a beneficial effect upon corrosion resistance in non-oxidizing sulphuric acid and hydrochloric acid solutions, in oxidizing ferric chloride solution, and in crevice corrosion in oxygenated 3% sodium chloride solution.

The effects of molybdenum additions were shown to be qualitatively similar to those of uranium, but it was found that steels containing 1% and 2% molybdenum showed, on the whole, distinctly improved corrosion resistance as compared with the uranium-bearing steels. It was concluded that molybdenum is capable of conferring greater improvements in the aqueous corrosion resistance of AISI Type 430 steel than uranium additions, though higher levels of molybdenum are necessary to achieve this.

With the completion of these measurements, laboratory corrosion-testing of uranium-bearing steels has been terminated. Small-scale field tests of some of these steels in various corrosive environments have been in progress since the summer of 1964⁽³⁾. It is expected that the results of these tests will show whether significant improvements in corrosion resistance under service conditions can be expected by alloying with uranium.

ACKNOWLEDGEMENTS

The chemical analyses of the steels were carried out by personnel of the Mineral Sciences Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa.

Discussions with Professor N. D. Greene and other personnel of the School of Engineering, Rensselaer Polytechnic Institute, Troy, N. Y., were helpful.

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TABLE 1
Chemical Analyses of Steels

Per Cent								
Steel No.	С	Mn	Si	S	P	Cr	υ	Мо
5555	0, 13	1,14	0, 96	0,019	0,020	17. 29		-
5556	0.14	1,10	0, 93	0,017	0,018	17.29	0, 24	-
5557	0.12	1.10	0.91	0.021	0.021	16.95	0,55	-
5801	0.11	1.14	1.02	0.020	0.017	17.64		1.02
5802	0.10	1,12	1.03	0,019	0,018	17.55	~	2, 03

TABLE 2

Corrosion in Normal Solutions at Room Temperature

			Weight Losses in 4 hr, mg/cm ²						
Steel No.	% U	% Mo	1.0 N H ₂ SO ₄	1.0 N HC1	1.0 N FeC1 ₃				
555 5		_	12.6, 12.9, 13.4	6.6, 5.2, 6.5	11.7, 12.0, 12.4				
5556	0,24	-	9.2, 9.4, 9.3	2.9, 2.8, 2.9	4.2, 4.3, 4.6				
555 7	0.55	-	9.1, 8.9, 9.0	2.9, 3.0, 3.2	5, 2, 5, 8, 5, 3				
5801	***	1.02	5.4, 5.4, 5.4	1.6, 1.7, 1.7	3, 5, 2, 3, 3, 8				
5802	-	2,03	3.7, 3.8, 3.7	1.2, 1.3, 1.3	1.4, 0.9, 1.5				

TABLE 3

Crevice Corrosion in Oxygenated 3% Sodium Chloride

Solution at 50°C (122°F)

Steel No.	% U	% Mo	Weight Changes after 7 days, mg/cm	Appearance of Specimen Surfaces after Test
5555	-	•	-2,4, -2.5, -2.4	Dark staining and many pits on all crevice surfaces.
5556	0,24		=0.3, =0.3, =0.3	Slight staining and slight pitting on all crevice surfaces.
5557	0,55	-	-0.2, +0.1, -0.4	Slight staining and slight pitting on all crevice surfaces.
5801		1.02	+0.1, - 0.1, 0	3 out of 6 crevice surfaces essentially unattacked, slight attack on remainder.
580 2	a	2.03	+0.1, +0.1, -0.1	5 out of 6 crevice surfaces essentially unattacked, slight attack on one.

TABLE 4

Corrosion in Successive 48-hr Periods in Boiling 65% Nitric Acid (Huey Test)

				Weight Losses, mg/cm ²							
Steel No.	% U	% Mo	lst period	2nd period	3rd period	4th period	5th period	Total, 5 periods			
5555	_	-	5.9, 5.8	6.4, 6.2	6.7, 6.6	7.6, 7.0	7.8, 7.6	34.4, 33.2			
5556	0.24	- ·	6.3, 6.2	7.2, 7.4	7.3, 7.0	8.3, 7.6	8.7, 7.7	37.8, 35.9			
5557	0, 55	-	6.1, 6.0	6 .7, 7. 1	7.7, 8.2	9.1, 9.8	11.2, 11.5	40.8, 42.6			
5801	-	1.02	5.1, 5.2	7.1, 7.2	7.9, 8.9	9.0, 10.2	8.9, 10.5	38.0, 42.0			
5802	· -	2, 03	112.9, 125.2								

TABLE 5

Results Obtained in Anodic Polarization Measurements

			·	Potentials, mV Current DemA/cm				ensities,	
Steel No.	% Т	% Mo	Solution	E	E cpp	E P	E t	i	ⁱ min
5555	-	es	1.0 N H ₂ SO ₄	- 510	- 430	-200	+960	21.0	0.012
5556	0.24	-	11 ,	- 500	- 460	-2 90	+960	7.6	0.018
5557	0.55	-	11	- 490	- 450	-290	+8 2 0	7.1	0.020
5801	-	1.02	11	- 490	- 450	-320	+980	3.7	0.011
5802	-	2.03	11	- 490	-440	-2 30	+970	2, 2	0.009
5555	-		1.0 N H ₂ SO ₄ plus 0.5 N NaCl	- 500	- 390	-130	+290	21.2	0.045
5556	0.24	-	11	- 490	- 390	- 190	+310	11.1	0.019
5557	0.55	-	11	- 490	-420	-200	+240	11.6	0.021
5801	-	1.02	. 11	- 480	-410	-240	+390	7.0	0.019
5802	_	2.03	. 11	- 480	-410	-240	+600	4.4	0.015

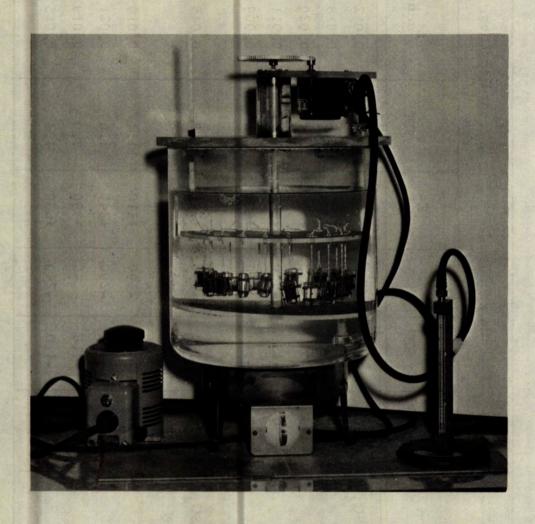


Figure 1 - Apparatus used in corrosion tests. The specimens shown are being tested for crevice corrosion.

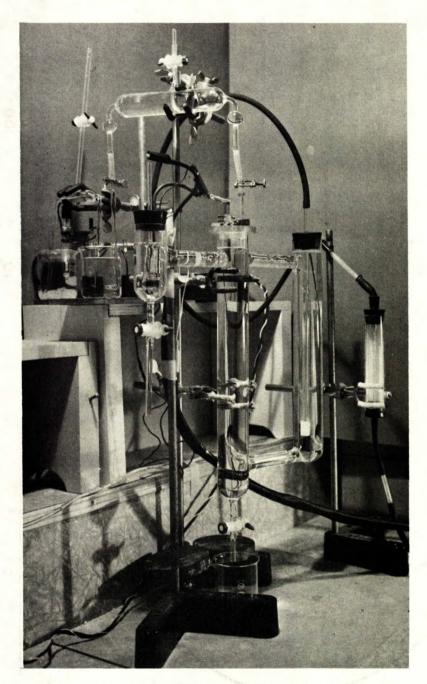


Figure 2 - Test vessel used in the anodic polarization measurements.

The platinum auxiliary electrode is in the left-hand arm of the vessel, and the sintered glass diaphragm used to bubble nitrogen into the vessel is in the right-hand arm. The specimen and probe are in the centre arm. The probe is connected to the saturated calomel electrode through the salt bridges shown at the upper left.

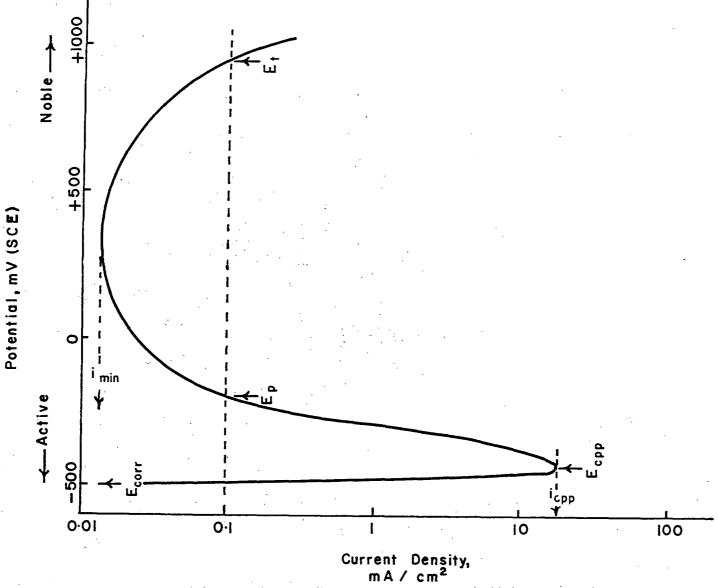


Figure 3 - Model anodic polarization curve, showing the potentials and the current densities which were measured.

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