

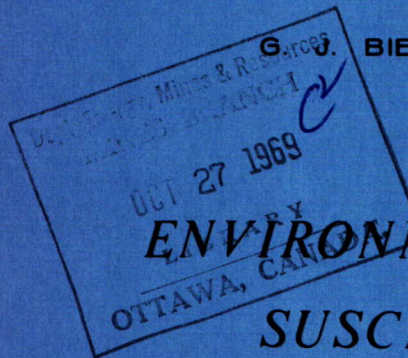
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DEPARTMENT OF
ENERGY, MINES AND RESOURCES
MINES BRANCH
OTTAWA

*COMPARISON OF U AND MO IN IMPROVING
THE CORROSION RESISTANCE OF
AISI 430 STAINLESS STEEL*

G. J. BIEFER AND J. G. GARRISON



*ENVIRONMENTAL CRACKING
SUSCEPTIBILITY OF
HIGH STRENGTH STEELS*

G. J. BIEFER

PHYSICAL METALLURGY DIVISION

Reprinted from *Materials Protection* 7, 39-40 and 23-26, 1968

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Queen's Printer and Controller of Stationery
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1969

Environmental Cracking Susceptibility of High Strength Steels

SUMMARY

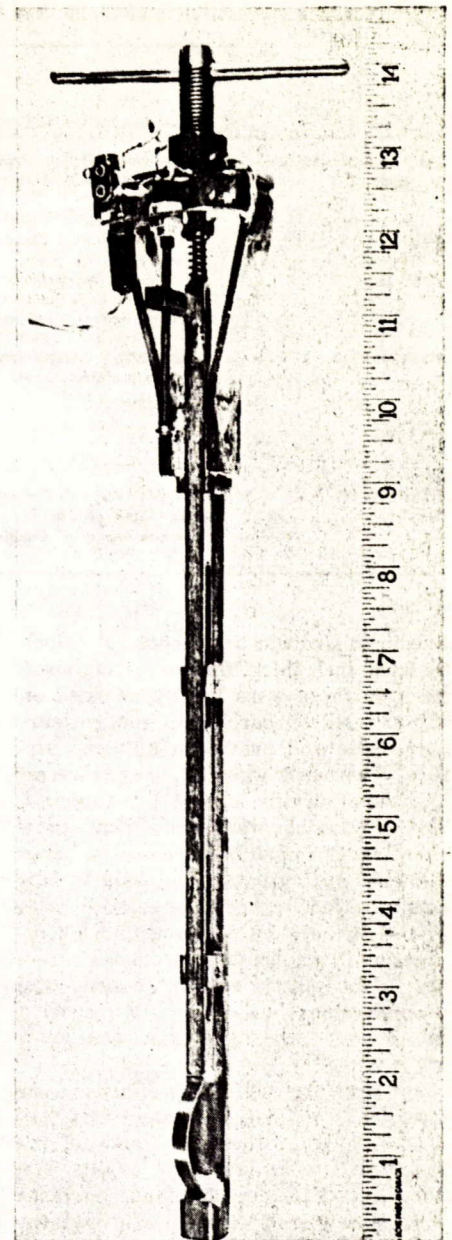
Exploratory stress corrosion cracking (SCC) and H₂ embrittlement cracking (HEC) test were carried out on high strength steels using an apparatus in which the test specimens are small bent strips held in this configuration under spring loading. Effects of alloy, electrochemical potential, test solution, pre-cracking, and coating are examined.

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AT THE CANADIAN DEPARTMENT of Energy, Mines and Resources (DEMRR) it recently became important to gain preliminary information on the probable effect of certain factors upon the stress corrosion cracking (SCC) and the H₂ embrittlement cracking (HEC)⁽¹⁾ of 18% Ni maraging steel, and the cracking susceptibility of other steels. Because the steels were in short supply, an Inconel⁽²⁾ SCC testing rig was used, modeled after one used by

⁽¹⁾In this paper the general term "environmental cracking" is used; it includes both SCC and HEC, which are used only when the cracking mechanism is known.

⁽²⁾Trademark of the International Nickel Co., New York, N.Y.



APPARATUS USED in the spring loaded bent strip stress corrosion cracking tests. The spring loading feature of the rig maintains tensile stress and ensures that cracking will proceed to an easily identifiable failure.

Uhlig and Sava,¹ in which a small strip of metal is held in a bent configuration under spring loading. This rig is small, relatively inexpensive, and can be used to test specimens in boiling solutions under reflux. The spring loading feature of the rig maintains tensile stress and ensures that cracking will proceed to an easily identifiable failure, thus avoiding a disadvantage of the widely-used fixed bend beam SCC test.

Experimental Procedure

Apparatus Used

The SCC test apparatus is shown above with a specimen in position.

TABLE 1—Description of Steels Used in the Environmental Cracking Tests

Steel	Source and As-Received Condition	Heat Treatment	Nominal Yield Strength After Heat Treatment, kpsi
18% Nickel (250 grade) Maraging	Vacuum melt 3960969, Republic Steel Co. 9/16 inch solution-annealed plate.	Aged 3 hrs at 482 C (900 F)	250
Carpenter Custom 455 Stainless	Heat 81643, Carpenter Steel Co. 0.050 inch thick annealed sheet	Aged 4 hrs at 510 C (950 F)	250
HP 9-4-25	Vacuum-melt from Republic Steel Co. 3/4 inch thick quenched and tempered plate. Tempering temperature, 538 C (1000 F)	None	190
AISI Type 4340	Heat 3321713, Republic Steel Co. 5/8 inch annealed rod, "Aircraft Quality."	Austenitized 15 min. at 843 C (1550 F), oil quenched, then tempered for one hr at 454 C (850 F)	180
Armco 17/4 PH Stainless	Heat 64190-2C, G.O. Carlson Inc. t/2 inch plate in the solution-treated "A" condition.	Aged 4 hrs at 538 C (1000 F) to produce the "H1000" condition.	150

Specimen size was 1 1/4 inches by 1/4 inch by 0.040 inch thick. Before testing specimen surfaces were lightly abraded on 120-grit silicon carbide to remove surface oxide and provide a uniform surface. Thorough washing, degreasing and rapid drying in hot air followed. The specimens were then bent to a chord length of 17/16 inches (without allowing spring-back) and held in this configuration between the ceramic jaws of the SCC rig by a spring under compression. This bending produced stresses in the plastic region. In tests, the commencement of cracking caused a movement of the upper jaw, deactivating a timer.

In tests in which specimens were electrically polarized, a steel wire was spot welded to the lower end of the specimen, using low heat input. The wire and adjacent specimen surfaces were then coated with paraffin wax before immersion in the test solution. The auxiliary electrode in the polarization tests was platinum.

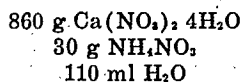
Test Solutions

The following test solutions were employed:

1. Normal sulfuric acid at room temperature.
2. As in (1), but containing 5 drops per

liter of cathodic poison composed of 2 g phosphorus dissolved in 40 ml CS₂ (see reference 2).

3. Boiling calcium ammonium nitrate solution. The solution had the composition,



and was similar to that used by Uhlig and Sava.

4. 3.5% sodium chloride solution at room temperature.

Materials

Information regarding types of steel used, including heat treatments employed, nominal yield strengths, and chemistry are in Tables 1 and 2. Small strip specimens were always cut so their lengths lay in the rolling direction. In all cases, the heat treatments of Table 1 were carried out on the metals in final specimen form.

Test Results

In 3.5% Sodium Chloride

The behavior of the steels in short-term tests in 3.5% sodium chloride solution is shown in Table 3, which provides data for the unpolarized steels

and also for the steels with impressed cathodic currents. In the unpolarized condition [for 18% Ni maraging steel, at a potential of -430 to -610 mv with respect to the saturated calomel electrode (SCE)] all the steels survived for a week without cracking. However, with a cathodic current of 1 ma/cm², which yielded a potential of about -1200 mv for maraging steel, the two steels with the highest strengths broke quite readily by HEC; of the three lower-strength steels, only the AISI 4340 showed a single break.

For the maraging steel, two additional lower cathodic current densities were investigated. At 0.05 ma/cm² potentials showed a tendency to slow drift within the range -920 to -1010 mv, whereas the lowest cathodic current density of 0.003 ma/cm² did not provide cathodic protection, and potential measurements were not made. In both cases, breaks were not produced within one week.

For the maraging steel, a series of longer-term tests was also carried out on unpolarized specimens and on specimens coupled galvanically to each of Inconel 718 (-400 to -580 mv), 5083 aluminum alloy (-770 to -810 mv) and zinc (-1020 to -1030 mv). In these tests, none of the specimens had broken after 1000 hours, and the SCE potentials lay within the ranges indicated. These results, and those of Table 3, are shown in Figure 1 (page 26).

Tests in Aggressive Solutions

In addition to these tests, specimens of the five steels were exposed to three highly aggressive test solutions. Results are shown in Table 4. In sulfuric acid, two of the steels were resistant; however, in "poisoned" sulfuric acid, all the steels broke. Without exception, no specimen survived more than 5.4 hours. The two stainless steels did not crack in the boiling nitrate solution, (as has been shown previously³), but the other three steels did.

Pre-cracked Specimens

Tests on smooth specimens in 3.5% NaCl solution had shown that maraging steel would crack readily by HEC if polarized to a potential of about

TABLE 2—Chemical Analysis of the Steels

Steel	C	Mn	Si	S	P	Cu	Co	Cr	Mo	Ni	Others
18% Maraging ⁽¹⁾	0.019	0.10	0.10	0.004	0.008	—	7.25	—	4.86	17.83	Ti, 0.38; Al, 0.09
Carpenter Custom 455 Stainless ⁽²⁾	<0.03	<0.50	<0.50	—	—	1-3	—	11-13	—	7-10	Ti, 0.9-1.4; Cb+Ta, 0.25-0.50; B, <0.005
HP 9-4-25 ⁽³⁾	0.27	0.21	0.02	0.014	0.005	—	3.86	0.39	0.51	9.48	V, 0.08
AISI Type 4340 ⁽¹⁾	0.415	0.75	0.30	0.010	0.011	—	—	0.70	0.25	1.80	—
17/4 PH Stainless ⁽⁴⁾	0.039	0.20	0.58	0.008	0.017	3.49	—	15.91	—	4.28	Cb, 0.20; Ta, 0.02

⁽¹⁾Supplied by Republic Steel Co.

⁽²⁾Nominal Analysis supplied by Carpenter Steel Co.

⁽³⁾Analysis at the Department of Energy, Mines & Resources.

⁽⁴⁾Supplied by G. O. Carlens, Inc.

—1200 mv, SCE. However, because there had been no breaks, resistance to environmental cracking appeared independent of potential from —400 to —1030 mv. It was decided, therefore, to carry out additional tests in this potential range on maraging steel specimens which had been pre-cracked; this was expected to reduce the time-to-failure markedly and thus permit some assessment of the effect of potential.

Specimens were to be pre-cracked chemically. Of the aggressive solutions investigated, the “poisoned” sulfuric acid appeared most suitable. The following procedure was adopted: a standard bent specimen was immersed in the “poisoned” sulfuric acid solution. Upon initial cracking (signalled by an alarm) the specimen was immediately removed from solution, rinsed in normal sulfuric acid, then salt solution, and finally immersed in 3.5% NaCl solution, with or without galvanic coupling. The time to recommencement of cracking was then measured. Results are shown in Figure 1. While it was not certain that all the “poison” had been washed from the pre-cracked, results of Figure 1, (controlling effect of potential, extended times-to-failure in some cases), indicated that the effect of such residuals had been secondary.

Pre-cracking caused a large decrease in the time-to-failure and also revealed a distinct effect of potential. About —800 mv was apparently optimum for resistance to environmental cracking (though scatter was evident). In contrast to results obtained on smooth specimens, coupling to zinc, at a potential of about —1020 mv SCE, was extremely detrimental for pre-cracked specimens, resulting in very low times-to-failure by HEC. It is of interest that these trends are in line with those obtained at the US Naval Research Laboratory, using the much more sophisticated and quantitative cantilever stress corrosion test, in which the specimens are notched bars, pre-cracked in the notch by fatiguing.⁴ Previous to this, Dean and Copson had reported that maraging steel was susceptible to HEC

TABLE 4—Environmental Cracking of High-Strength Steels in Various Aggressive Solutions

Steel	Nominal Yield Strength, kpsi	In Normal Sulphuric Acid at R.T.	In Normal Sulphuric Acid plus poison, at R.T.	In Calcium Ammonium Nitrate Solution at the Boil
18% Nickel (250) Maraging	250	0.8	0.8	9.8
Carpenter Custom 455	250	0.3	0.1	10.2
HP 9-4-25	190	Unbroken after 4.0 hr	0.2	Unbroken after 199 hr
AISI Type 4340	180	0.1	0.8	Unbroken after 93 hr
17/4 PH Stainless	150	Unbroken after 24 hr	0.7	2.7
			0.1	3.0
			0.1	0.9
			0.1	0.2
			3.1	Unbroken after 90 hr
			0.6	Unbroken after 93 hr
			5.4	

when polarized to too negative a potential;⁵ this, too, corresponded with results of Figure 1.

Not all high-strength steels could have been suitably pre-cracked by the solution used. Of the five steels tested, the AISI 4340 and the HP 9-4-25 steels would have been unsuitable since once the cracking started, it proceeded almost immediately to complete failure (the specimen breaking in two) in the “poisoned” sulfuric acid. The Carpenter Custom 455 and the 17/4 PH steels cracked in stages, so that chemically pre-cracked specimens could have been prepared and tested.

Gold Plated Specimens

Other work at the Canadian DEMR⁶ had indicated that a very thin gold plating can serve as a barrier to the passage of embrittling hydrogen through steel. To obtain data on the probable behavior of such plating in the presence of an aqueous corrodent, it was decided to carry out environmental cracking tests on some gold plated maraging steel specimens in 3.5% NaCl solution.

It appeared that gold can polarize the steel (at defects in the plating) in the positive direction, leading to increased susceptibility to SCC. This is indicated by the results of Table 5, (page 26) which showed rapid failures in 3 out of 7 cases. Clearly, it could be dangerous to allow a corrosive liquid to come in contact with an imperfectly

gold plated crack-susceptible steel under tensile stress.

Neoprene Coated Specimens

In an attempt to verify statements that neoprene coatings were permeable to water and/or ions^{7,8}, environmental cracking tests were carried out on bent strips of neoprene coated (0.020 inch thick) maraging steel. Specimens with both damaged and undamaged coatings were immersed in 3.5% NaCl solution. After times ranging from 1574 to 1878 hours the specimens began to crack, performance for specimens with initially damaged and undamaged coatings being about equivalent (Table 5).

Post test examinations indicated some loss of coating adhesion and cracking. Specimens with coatings which had been deliberately slashed did not crack at the point of damage, but did crack under whole neoprene at some other location. Results, therefore, provided additional evidence that neoprene coatings may not provide reliable long-term protection for steels immersed in neutral salt solutions (e.g. sea water).

Conclusions

In terms of original objectives and expectations, results obtained with the spring-loaded bent strip apparatus were highly satisfactory. This simple and inexpensive method which makes minimum demands on laboratory space and test material, provided excellent preliminary information on the environmental cracking susceptibility of high-strength steels, including the effects of such factors as alloy composition, potential, test solution, pre-crack-

TABLE 3—Environmental Cracking Tests in 3.5% NaCl Solution at Room Temperature

Steel (plus Nominal Yield Strength)	Impressed Cathodic Current Density, mA/cm ²	Approximate Potential, (S.C.E.), mV	Total No. of Specimens	No. of Specimens Breaking within one week	Time-to-Break, hr
18% Ni Maraging (250 kpsi)	None	-430 to -610	6	0	0.2, 0.3, 0.5, 5-68, 6.5-22, 23.7, 107
	0.003	—	3	0	
	0.05	-920 to -1010	6	0	
	1.0	-1190 to -1210	8	7	
Carpenter Custom 455 Stainless (250 kpsi)	None	—	3	0	0.1, 0.1, 0.5
	1.0	—	3	3	
	1.0	—	3	0	
HP 9-4-25 (190 kpsi)	None	—	3	0	59.2
	1.0	—	3	0	
	1.0	—	3	1	
AISI Type 4340 (180 kpsi)	None	—	3	0	59.2
	1.0	—	3	1	
17/4 PH Stainless (150 kpsi)	None	—	3	0	59.2
	1.0	—	3	0	

ing, and coating. It is surprising that this type of SCC test apparatus is not more widely used and mention of it is often omitted in reviews which are otherwise comprehensive.⁹

Acknowledgments

Some of the chemical analyses were provided by the Mineral Sciences Division of DEMR. B. G. Olivier and J. G. Garrison of the Corrosion Section, Physical Metallurgy Division, DEMR, performed the tests.

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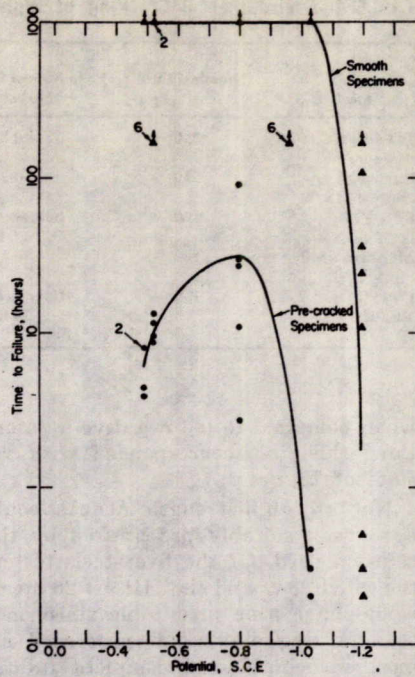


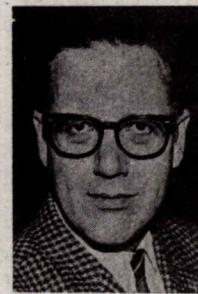
Figure 1—Environmental cracking behavior of 18% maraging steel in 3.5% NaCl solution. The circular points were obtained on pre-cracked specimens; the triangular points on smooth specimens.

TABLE 5—Environmental Cracking Tests on Coated Specimens of 18% Nickel Maraging Steel in 3.5% NaCl Solution.

Coating Material	Time-to-Break, hr
Gold	
1	4.0
2(2)	2.4
3(2)	Unbroken after 336 hr
4(2)	Unbroken after 336 hr
5(1)	Unbroken after 336 hr
6	63.1
7	Unbroken after 336 hr
Neoprene	
1	1574
2(1)	1794
3	1878
4(1)	1649

(1) Coating deliberately damaged prior to testing.

(2) Nickel substrate was under the gold plating.



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Comparison of U and Mo in Improving the Corrosion Resistance of AISI 430 Stainless Steel

IS URANIUM EQUAL to or better than molybdenum as a means of improving corrosion resistance of AISI Type 430 stainless steel? Laboratory tests in several different corrosive solutions indicate that in ferritic AISI Type 430 stainless steel, alloying additions of 0.24% and 0.55% U resulted in improvements in corrosion resistance. However, somewhat greater improvements generally occurred with additions of 1.02% and 2.03% Mo.

Unlike the situation with the well-known austenitic and martensitic stainless steel types, there has been comparatively little published on ferritic stainless steels (AISI Types 405, 430 and 446) with respect to their corrosion resistance, particularly in controlled laboratory tests. Reports show that 1% Mo in Type 430 (termed Type 434) has better corrosion resistance than straight Type 430 in car trim and that Mo additions have also brought about improved corrosion resistance for ferritic

SUMMARY

A comparison of molybdenum and uranium as a means of improving corrosion resistance of ferritic AISI Type 430 stainless steel is shown by laboratory tests.

stainless steels in other service applications.^{1,2} Conversely, it has been stated that the beneficial effects of molybdenum observed in austenitic stainless steels do not extend to the nickel-free ferritic stainless steels.⁴

The laboratory tests to determine whether uranium additions to AISI Type 430 ferritic stainless steels in the optimum range of about 0.25-0.5% were competitive with additions of molybdenum to the same steel type are described as follows.^{5,6}

Testing

Procedure

Five steels (compositions shown in

Table 1) were prepared as 50-lb aluminum-deoxidized melts in an induction furnace. After forging and rolling to ¼-inch plate, the steels were heat-treated for four hours at 1450 F (790 C) then furnace cooled. The corrosion tests were conducted with flat specimens which had been given standardized surface pretreatment. (Conventional experimental techniques used are described elsewhere.)¹

Results

The tests were conducted in normal solutions of sulfuric acid, hydrochloric acid, and ferric chloride at 75 F (24 C), 122 F (50 C), and 158 F (70 C). Figures 1-3 show the weight losses in the specimens; every point of these figures is an average of two or three replicates.

The Huey test, conducted in 65% boiling nitric acid, resulted in specimen weight losses in five successive 48-hr exposures. The results are presented in Table 2. Weight losses and surface appearances observed after creviced specimens of the five steels had been exposed to a 3% sodium chloride solution at 122 F (50 C) for one week (previous work had shown that crevice corrosion attack is slow at room temperature) are given in Table 3.

The laboratory tests demonstrated that in AISI Type 430 ferritic stainless steels uranium and molybdenum additions are both capable of producing improved corrosion resistance in aqueous media. Improvements imparted by the Mo additions were somewhat greater than those resulting from the U additions though higher levels of Mo were necessary. An interesting exception to this was observed in tests in ferric chloride at the two higher temperatures where uranium was of some benefit and molybdenum was ineffective.

In conclusion, molybdenum is generally more effective in improving corrosion resistance of AISI Type 430 stainless steel than uranium.

Acknowledgment

The chemical analyses were carried out by the Mineral Sciences Division of the Dept. of Energy, Mines and Resources. Some of the corrosion tests

TABLE 1—Chemical Analyses of Steels

STEEL NO.	PERCENTAGES							
	C	Mn	Si	S	P	Cr	U	Mo
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	-	1.03
5802	0.10	1.12	1.03	0.019	0.018	17.55	-	2.03

TABLE 2—Corrosion in Successive 48-Hr Periods in Boiling 65% Nitric Acid (Huey Test)

STEEL NO.	% U	% Mo	WEIGHT LOSSES, MG/CM ²					TOTAL, 5 PERIODS
			1ST PERIOD	2ND PERIOD	3RD PERIOD	4TH PERIOD	5TH PERIOD	
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	-	-	-	-	-

were conducted by J. Mar of the Physical Metallurgy Div.

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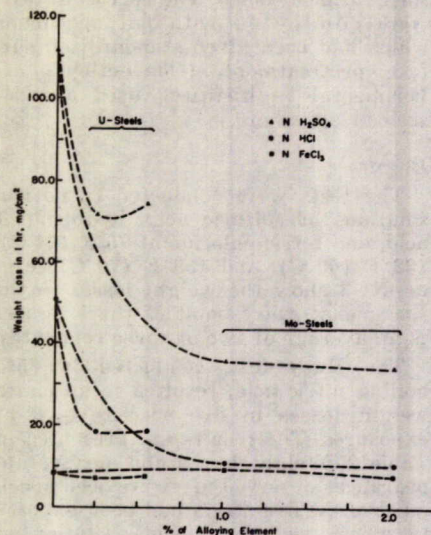


Figure 3—Corrosion of uranium and molybdenum bearing AISI Type 430 stainless steels in normal solutions at 158 F (70 C)



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TABLE 3—Crevice Corrosion in Oxygenated 3% Sodium Chloride Solution at 122 F (50 C)

STEEL NO.	% U	% Mo	WEIGHT CHANGES AFTER 7 DAYS, mg/cm ²	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.
5802	-	2.03	+0.1, +0.1, -0.1	5 out of 6 crevice surfaces essentially unattacked, slight attack on one.

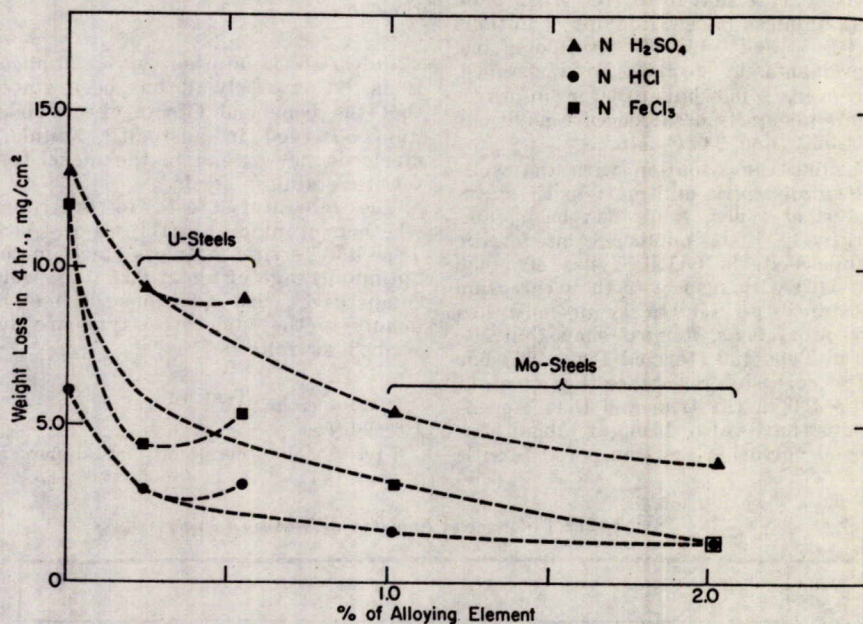


Figure 1—Corrosion of uranium and molybdenum bearing AISI Type 430 stainless steels in normal solutions at room temperature.

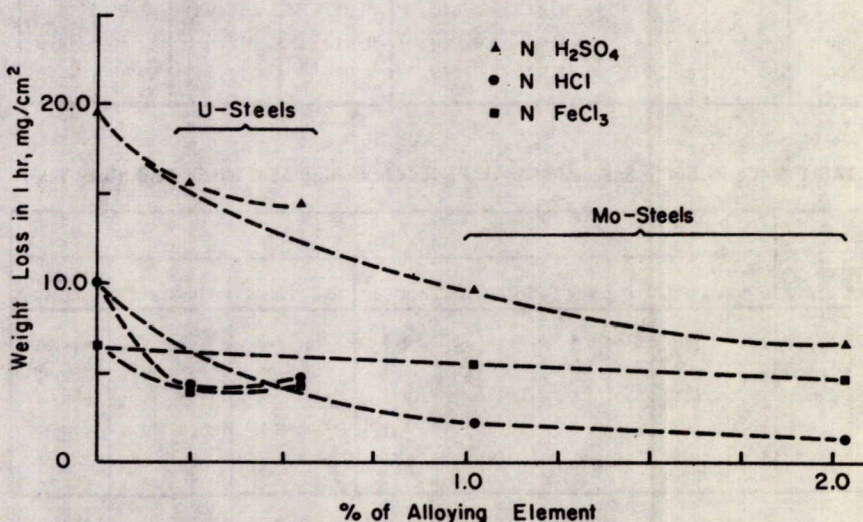


Figure 2—Corrosion of uranium and molybdenum bearing AISI Type 430 stainless steels in normal solutions at 122 F (50 C)

