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A COMPARISON OF FIELD AND LABORATORY CORROSION TESTS OF AISI TYPE 430 STAINLESS STEELS

J.G. GARRISON AND G. J. BIEFER

PHYSICAL METALLURGY DIVISION

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A COMPARISON OF FIELD AND LABORATORY CORROSION TESTS OF AISI TYPE 430 STAINLESS STEELS

by

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

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ABSTRACT

Field tests and accelerated dip-and-dry laboratory tests were carried out to determine the effects of additions of uranium and molybdenum on the corrosion properties of AISI Type 430 stainless steel used as automotive trim.

A slight increase in corrosion resistance was obtained with an alloy containing 0.24% uranium, while no deleterious effect was experienced with an alloy containing 0.55% uranium. Increased corrosion resistance was observed with increased molybdenum for two alloys, one of which contained 1.02% and the other 2.03% molybdenum.

Correlation of results between the accelerated dip-and-dry laboratory tests and the field tests was not entirely satisfactory. There was, however, some area of agreement between these tests, and further use of the accelerated laboratory test appears warranted.

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Direction des mines

COMPARAISON DES ESSAIS DE CORROSION, EN SERVICE ET EN LABORATOIRE, DES ACIERS INOXYDABLES DE TYPE AISI 430

par

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

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RÉSUMÉ

Les auteurs ont fait des essais accélérés d'immersion suivie du séchage en laboratoire, ainsi que des essais en service, afin de déterminer les effets des additions d'uranium et de molybdène sur les caractéristiques de corrosion de l'acier inoxydable de type AISI 430 employé comme garniture d'automobile.

Une légère augmentation de résistance à la corrosion a été obtenue pour un alliage contenant 0.24 p. 100 d'uranium, et il n'y eut aucun effet nuisible pour un alliage contenant 0.55 p. 100 d'uranium. Une amélioration de la résistance à la corrosion a été observée à la suite d'une augmentation à 1.02 p. 100 de la teneur en molybdène dans un des deux alliages, et à 2.03 p. 100 dans l'autre.

La corrélation des résultats des essais accélérés d'immersion suivie du séchage en laboratoire et des essais en service n'a pas été entièrement satisfaisante. Certains résultats ont concordé cependant dans les deux essais et il semble que d'autres essais accélérés en laboratoire soient justifiés.

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INTRODUCTION

A decrease in the demand for uranium in the late nineteen-fifties prompted the Canadian Government and the uranium industry to launch an intensive research program aimed at the development of new non-nuclear uses for the metal. A comprehensive review of the initial results obtained in research on uranium-bearing steels has been published by the Mines Branch[(1)]. In this work, there were indications of minor improvements in the corrosion resistance of some non-austenitic steels which had been alloyed with uranium[(2)].

The most definite effects were observed when uranium was alloyed with AISI Type 430 stainless steel. Results obtained with this steel[(3)] in the Physical Metallurgy Division of the Mines Branch indicated that an optimum level of 0.1-0.5 per cent uranium brought about improved corrosion resistance in non-oxidizing sulphuric acid and hydrochloric acid solutions and in oxidizing ferric chloride solutions. Improved resistance to crevice corrosion in 3 per cent sodium chloride solution was also indicated.

Similar corrosion tests on two commercial AISI Type 430 stainless steels, one molybdenum-free and the other containing 0.76 per cent molybdenum, indicated that the effects of molybdenum and uranium additions were qualitatively similar. Molybdenum additions to ferritic stainless steels such as AISI Type 430 are known to confer improved corrosion resistance in service applications[(4,5,6)]. Additional results obtained with uranium-bearing and molybdenum-bearing AISI Type 430 stainless steels[(7)] confirmed that uranium alloying additions of 0.25-0.50 per cent to this steel are capable of conferring increased corrosion resistance in some aqueous solutions. Molybdenum alloying additions of approximately 1 and 2 per cent were shown to bring about effects qualitatively similar to those brought about by uranium. However, on the whole, the molybdenum-bearing steels showed distinctly improved corrosion resistance as compared with the uranium-bearing steels.

To expand upon the results obtained in the laboratory tests, it was decided to carry out field tests on uranium-bearing AISI Type 430 stainless steel. These field tests were to include exposure to automotive-trim environmental conditions, atmospheric conditions, and sea-water immersion. It was also decided that laboratory tests using the dip-and-dry method of determining pitting susceptibility, as developed by General Motors Corporation[8], would be carried out concurrently with the field tests, to determine the feasibility of this method of testing as a substitute for long-term field tests (see Figure 1 for apparatus used).

Molybdenum-bearing AISI Type 430 stainless steels were also subjected to the automotive-trim field test and the accelerated dip-and-dry laboratory test.

Because the atmospheric tests are not complete as yet, only the automotive-trim test and the accelerated dip-and-dry laboratory tests will be discussed herein. The results of the sea-water immersion tests will be made available at a later date.

EXPERIMENTAL

Materials

The uranium- and molybdenum-bearing Type 430 stainless steels were produced as 50-lb aluminum deoxidized melts in an induction furnace at the Physical Metallurgy Division. These included one heat with no alloying additions, which was used as a "control" standard; two heats representing two levels of uranium; and two heats representing two levels of molybdenum. Chemical analyses of these heats appear in Table 1.

The reference ingot (No. 5555) and the uranium-bearing steel ingots (Nos. 5556 and 5557) were forged and rolled at a starting temperature of 1008°C (1850°F)* to 3/16-in.-thick plates. The plates were annealed at 790°C (1450°F) for four hours and furnace-cooled. The

*i.e. below the melting temperature of the U-UFe_2 eutectic 1080°C (1976°F) to avoid hot shortness.

molybdenum-bearing steel ingots (Nos. 5801 and 5802) were forged and rolled at 1176°C (2150°F) to 1/8-in.-thick plates and these plates were also annealed at 790°C (1450°F) for four hours and furnace-cooled.

Corrosion specimens, 1 in. square, having a 1/4-in. centre hole, were obtained from the plates. When received from the machine shop, the corrosion coupons had a fine-ground surface finish. To ensure uniformity of the surface finish, the coupons were manually roughened to a uniform 120-grit-silicon-carbide finish.

The uranium-bearing alloy coupons were autoradiographed and a sufficient number of segregate-free specimens were chosen to carry out the required tests.

Automotive Trim Test

Twelve sets of the uranium-bearing steel specimens were selected for testing in Ontario, with five additional sets to be tested in the Maritime Provinces. Five sets of the molybdenum-bearing steel specimens were also chosen for testing in Ontario. Each set consisted of one "control" alloy specimen and one specimen each of the two levels of uranium or molybdenum.

The coupons were cleaned in an ultrasonic cleaner and a vapour degreaser, using carbon tetrachloride, and their weights were recorded. Each set of specimens was then mounted on a 6-in. x 2-in. x 3/8-in. Plexiglas panel (Figures 2 and 3). The specimens were mounted so that the rubber washers created a crevice on both the exposed and protected (Plexiglas facing) surfaces of the specimens. Care was taken to avoid contact between the mounting bolts and the specimens (the bolts were wrapped with Teflon tape). The panels were mounted on the front grill (near centre) of the test vehicles. The uranium-bearing alloys were tested for a 12-month period, commencing in May, while the molybdenum-bearing alloys were tested for 7 months, commencing in October.

Accelerated Dip-and-Dry Laboratory Test

The specimens used in this test were degreased in an ultrasonic cleaner, using carbon tetrachloride. After ultrasonic degreasing, they were soaked in a detergent solution (8 grams of detergent per litre of water) for 20 to 30 minutes at 80°C (176°F), rinsed in distilled water, and degreased again in boiling carbon tetrachloride vapour. The specimens were then dried and weighed.

The experimental procedures for the accelerated dip-and-dry laboratory tests were based on specifications laid down by General Motors Corporation[8], with some modifications. The test solution was made up according to General Motors Corporation specifications (Appendix I). It was found that this solution could be kept effectively for a period of five days (120 hours) but was not reliable if kept longer. Separate batches of solutions were made up, one for the uranium-bearing and one for the molybdenum-bearing steels.

The specimens were tested in groups of three. Each group consisted of one "control" coupon and one each of the two levels of uranium or molybdenum. Because the dip-and-dry apparatus (Figure 1) was made of Plexiglas, the use of heat lamps as suggested by General Motors Corporation was not possible. It was found that an 11-minute drying time was sufficient to allow adequate drying of the specimens at room temperature. The apparatus was therefore set to give a 2-second dip and an 11-minute drying cycle.

Each group of specimens was tested for a period of 8 hours. After testing, the specimens were rinsed thoroughly in running tap water, rinsed with ethyl alcohol, and dried rapidly in hot air. The specimens were then rated as outlined below.

Superficial Ranking of Appearance

The automotive-trim test specimens (but not the specimens from the dip-and-dry tests) were ranked as to appearance by means of a visual examination by five different observers. Ranking consisted of assigning numbers or points to the individual specimens in a group according to their appearance in relation to the other two specimens in the group.

Points were given in such a way that no specimen received more than 3 points and the total number of points for the three specimens in a group did not exceed 6. The specimen with the worst appearance (rust, pits) was given the highest number of points, while the one with the best appearance was given the lowest number of points.

Evaluation of Degree of Attack

A method of evaluation developed by researchers at the Ford Motor Company[9], which was based on the ASTM "Area Rating" method, was used to determine the degree of attack on the individual test specimens.

By this method, the specimens were assigned "protection" numbers. This number indicated the amount of corrosion by pitting and/or etching, by recording the actual area defective for each type of defect. This was done by measuring the areas with a transparent grid (1-mm squares). The numerical value of each area was then multiplied by the factor for that defect, and the total weighted area defective was then obtained by addition. The weighted area defective was then converted to a "protection" number by the use of a chart. Protection numbers ranged from 0-10, with 10 representing complete corrosion resistance.

In some cases, affected areas were of both an etched and a pitted nature. Accurate measurement of individual pits and etched areas was not practicable, so such areas were measured as a whole and then multiplied by the average of the factors assigned to both types of defect.

The specimens were derusted by immersion for 5-10 seconds in a solution of 0.5 gram of thiourea in 150 ml of concentrated H_2SO_4 made up to 1 litre with distilled water. This solution was used at 80°C (176°F).

RESULTS

Accelerated Dip-and-Dry Tests

After testing, the specimens were photographed on both sides (Figures 4 and 5) and were then derusted.

No crevice was provided on the dip-and-dry test specimens, and protection numbers were therefore based on the entire surface area. The protection numbers obtained for the individual uranium-bearing specimens are given in Table 2. Those for the molybdenum-bearing specimens are given in Table 3. Average figures for these protection numbers may be found in Tables 4 and 5.

Automotive-Trim Tests

Subsequent to testing and prior to derusting, the automotive-trim test specimens were visually ranked, as to appearance, by five individuals of the Mines Branch. The numerical rankings were obtained as described previously. The averages of the results are given in Table 6.

Measured evaluation of the automotive-trim test specimens and the calculation of protection numbers was accomplished as outlined previously. The specimens were considered in three ways:

1. "Crevice" area only.
2. Exposed area only.
3. Entire surface area.

Average figures for the protection numbers obtained are given in Table 7.

DISCUSSION OF RESULTS

Accelerated Dip-and-Dry Laboratory Tests

It was noted that, in general, the protection numbers for the "control" specimens tested with the molybdenum-bearing steels were higher than those obtained for the "control" steels tested with the uranium-bearing steels (Tables 4 and 5). It appeared possible that this difference was due to the fact that the two types of steels were tested in different batches of the test solution. However, since there was reasonably good agreement of the controls within each batch (Tables 2 and 3), it appeared that this difference in the controls in no way altered the trends in corrosion resistance indicated by these tests.

For the uranium-bearing specimens and their control steels, the protection numbers suggested a trend towards improved corrosion resistance with increased uranium. However, the effect was slight and may not have been statistically significant.

Improvement in the corrosion resistance of the alloy containing 1.02 per cent molybdenum, and a marked improvement in the corrosion resistance of the alloy containing 2.03 per cent molybdenum, in relation to the "control" steel, was indicated.

It is noteworthy that this trend, however, was not obvious from superficial examination of the test specimens (Figure 5). This latter indicated that the steel containing 1.02 per cent molybdenum has a definitely inferior surface appearance.

The protection numbers were, in fact, valid for these alloys in that the "control" steel specimens had been deeply pitted. These pitted areas required a high "weighting factor". On the other hand, the 1.02 per cent molybdenum-bearing alloy specimens were attacked over relatively large areas by light etching. Since these etched areas required only a low "weighting factor" in the determination of the protection numbers, these protection numbers were necessarily better than those for the "control" specimens.

Automotive-Trim Tests

The evaluation numbers, both visual and measured, obtained for the uranium-bearing automotive-trim test specimens (Tables 6 and 7) indicated slightly improved corrosion resistance for the 0.24 per cent uranium-bearing steel, while the 0.55 per cent uranium-bearing steel was found to be somewhat similar in behaviour to the "control".

Protection numbers obtained for the molybdenum-bearing alloys showed increased corrosion resistance with increased molybdenum content, the 1.02 per cent molybdenum-bearing alloy being similar in corrosion resistance to the 0.24 per cent uranium-bearing alloy and the alloy with 2.03 per cent molybdenum showing the best corrosion resistance of all.

From the average protection numbers obtained (Table 7), it was obvious that most of the corrosion occurred in the "crevice" area, and that the "control" specimens for the molybdenum-bearing group and the 1.02 per cent molybdenum steel specimens were the most severely attacked in this area. It appeared that the commencement dates of the tests may have been partly responsible for this difference in corrosion behaviour shown by the "control" steels.

The uranium-bearing steel specimens were exposed to atmospheric conditions for several months before being exposed to road-salt conditions; thus there was ample opportunity for a protective film to be formed on their surfaces. The molybdenum-bearing steels, however, were almost immediately exposed to road-salt conditions.

It is noteworthy that, even under the above-mentioned adverse conditions, the 2.03 per cent molybdenum-bearing alloy exhibited corrosion resistance superior to all of the other alloys tested.

The visual ranking tests for the molybdenum-bearing and control steels showed a very definite improvement in appearance of the exposed specimens with increasing molybdenum content.

The results obtained from these automotive-trim tests conducted in the field did not agree entirely with the results obtained by the accelerated dip-and-dry laboratory tests, in that the accelerated tests showed increased improvement in corrosion resistance with increased uranium content, whereas the field automotive-trim tests indicated an optimum effect with 0.24 per cent uranium. Both tests, however, did indicate improved corrosion resistance with increased molybdenum content.

It has been stated[10] that accelerated laboratory tests, such as the dip-and-dry test, do not correlate well with actual service tests. On the basis of the above results, however, there was a reasonable measure of correlation, and further use of this dip-and-dry method would appear to be warranted.

CONCLUSIONS

The results of accelerated laboratory dip-and-dry tests, and of field tests on automobiles, showed that:

1. There is a reasonable, though not entirely satisfactory, degree of correlation between the laboratory and the field tests. Further work needs to be done in this area.

2. For uranium-bearing AISI Type 430 stainless steels, an addition of 0.24 per cent uranium produced a slight increase in corrosion resistance. The effects of the 0.55 per-cent uranium addition were inconclusive.

3. Additions of molybdenum to AISI Type 430 stainless steels produced greater improvements in corrosion resistance than were produced by uranium. Of the steels tested, that with 2.03 per cent molybdenum showed the best corrosion resistance.

ACKNOWLEDGEMENTS

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

The photographs were taken by the Photographic Services Section of the Physical Metallurgy Division.

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TABLE 1

Chemical Analysis of Uranium- and Molybdenum-Bearing AISI Type 430 Stainless Steels

Heat No.	Composition, %							
	C	Mn	Si	S	P	Cr	Mo	U
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	0.11	1.12	1.03	0.019	0.018	17.55	2.03	-

TABLE 2

Protection Numbers for the Uranium-Bearing Specimens

(Accelerated Dip-and-Dry Test)

Group No.	Sample No.	Uranium Content	Protection No.
1	5555-17	Nil	4.8
1	5556-20	0.24%	5.1
1	5557-15	0.55%	4.6
2	5555-18	Nil	5.2
2	5556-21	0.24%	5.2
2	5557-21	0.55%	5.6
3	5555-19	Nil	5.3
3	5556-23	0.24%	5.1
3	5557-24	0.55%	5.6
4	5555-20	Nil	5.2
4	5556-24	0.24%	5.4
4	5557-25	0.55%	5.5
5	5555-21	Nil	5.2
5	5556-25	0.24%	5.4
5	5557-26	0.55%	5.6

TABLE 3

Protection Numbers for Molybdenum-Bearing Specimens (Accelerated Dip-and-Dry Test)

Group No.	Sample No.	Molybdenum Content	Protection No.
6	5555-22	Nil	6.7
6	5801-12	1.02%	7.5
6	5802-12	2.03%	8.9
7	5555-23	Nil	6.5
7	5801-13	1.02%	6.8
7	5802-13	2.03%	8.9
8	5555-24	Nil	6.7
8	5801-14	1.02%	7.5
8	5802-14	2.03%	9.2
9	5555-25	Nil	6.4
9	5801-15	1.02%	7.7
9	5802-15	2.03%	8.9
10	5555-26	Nil	6.7
10	5801-16	1.02%	7.9
10	5802-16	2.03%	9.2

TABLE 4

Average Protection Numbers for the Uranium-Bearing Dip-and-Dry Test Specimens

Heat No.	Uranium Content	Average Protection No.
5555	Nil	5.1
5556	0.24%	5.2
5557	0.55%	5.4

TABLE 5

Average Protection Numbers for the Molybdenum-Bearing Dip-and-Dry Test Specimens

Heat No.	Molybdenum No.	Average Protection No.
5555	Nil	6.5
5801	1.02%	7.3
5802	2.03%	8.9

TABLE 6

Visual Appearance Ratings for Automotive-Trim Test Specimens (Average Figures)

Group	1	2	3	4	5
"Control"	2.2	2.2	2.2	2.0	2.2
0.24% U	1.7	1.7	1.7	1.9	1.5
0.55% U	2.0	2.0	2.0	2.1	1.8
"Control"	3.0	3.0	3.0	3.0	3.0
1.02% Mo	1.9	1.9	2.0	2.0	2.0
2.03% Mo	1.1	1.1	1.0	1.0	1.0

TABLE 7

Average Figures for Protection Numbers Obtained from the Automotive-Trim Test Specimens

	"Crevice" Area	Exposed Area	Entire Surface
"Control"	4.1	6.3	6.5
0.24% U	4.9	7.4	6.9
0.55% U	4.4	6.9	6.5
"Control"	3.2	6.2	6.1
1.02% Mo	3.8	7.5	6.8
2.03% Mo	5.6	8.8	7.9

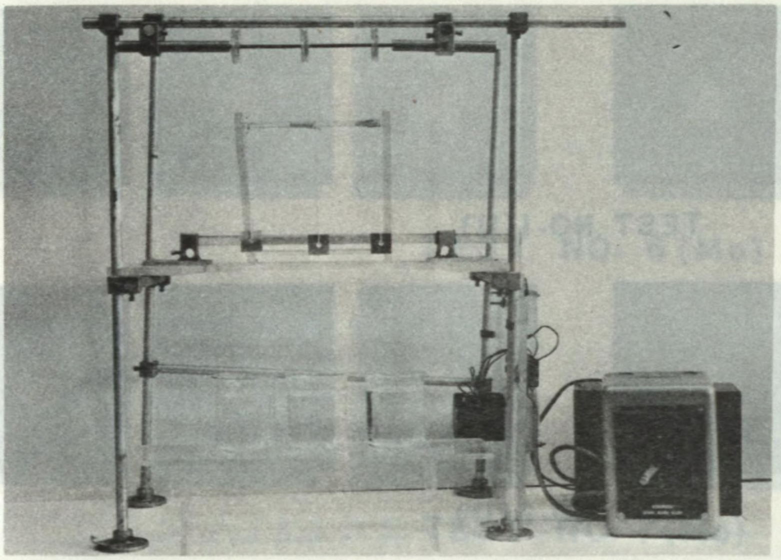

Figure 1

Accelerated Dip-and-Dry Laboratory Test Apparatus

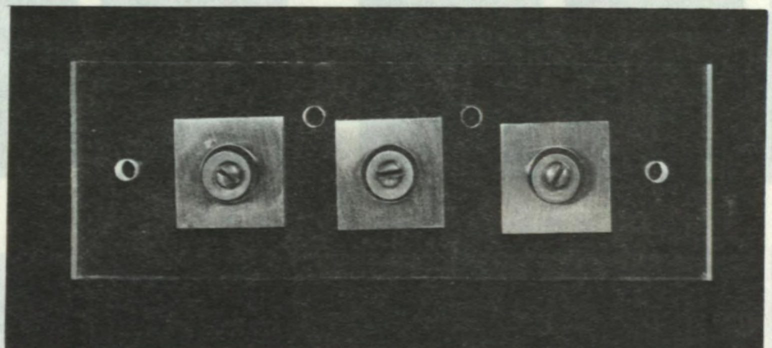

Figure 2. Front View of a Test Panel used in an Automotive-Trim Test.

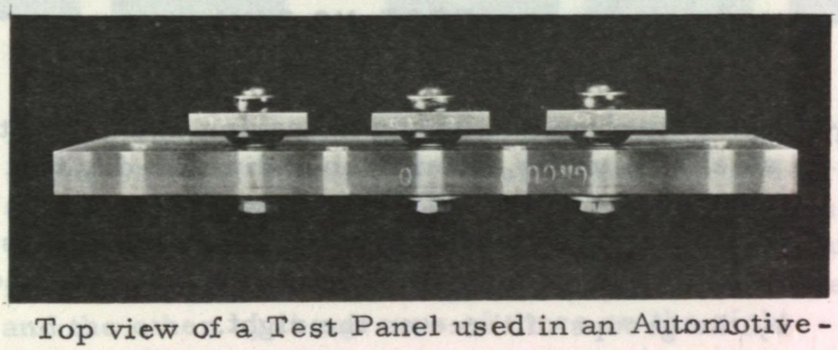

Figure 3. Top view of a Test Panel used in an Automotive-Trim Test.

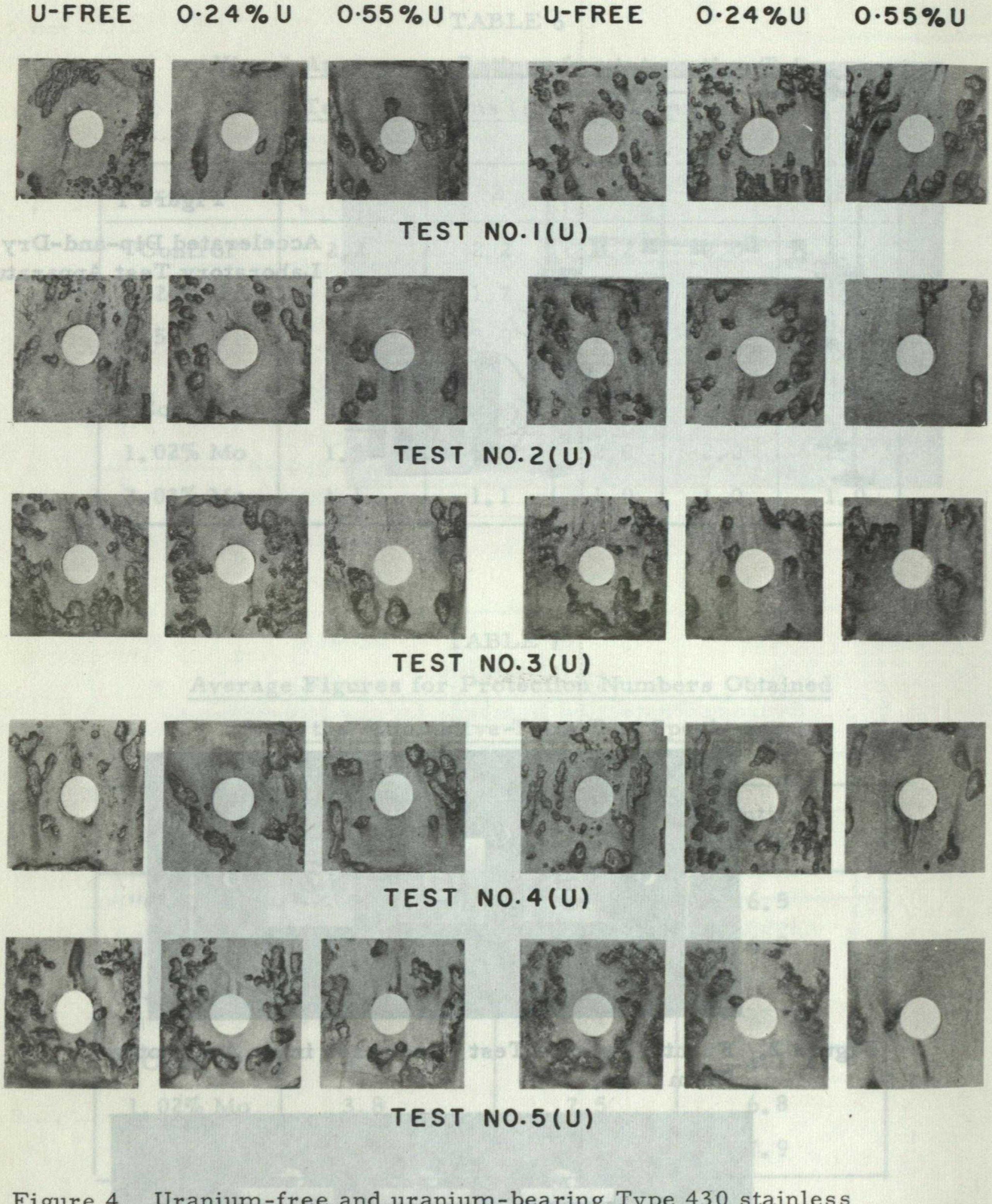

Figure 4. Uranium-free and uranium-bearing Type 430 stainless steel specimens, after 8 hour dip-and-dry test and prior to derusting. The photograph shows both major surfaces of the test specimens. One surface is represented by the groups of three on the left, and the other by the groups of three on the right.

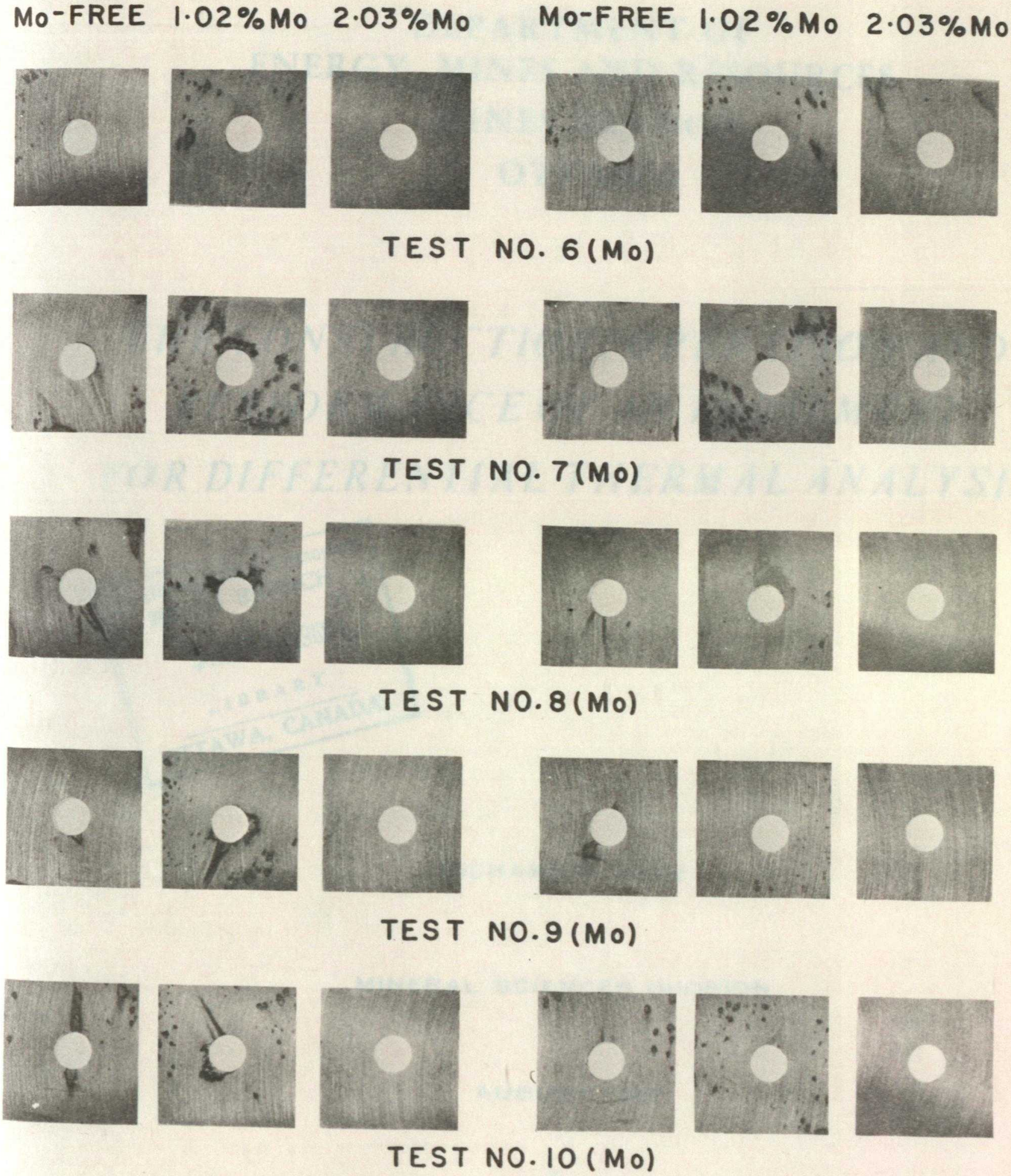

Figure 5. Molybdenum-free and molybdenum-bearing Type 430 stainless steel specimens, after 8 hour dip-and-dry test and prior to derusting. The photograph shows both major surfaces of the test specimens. One surface is represented by the groups of three on the left, and the other by the groups of three on the right.