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NITRIDED STAINLESS STEELS FOR HIGH-TEMPERATURE
WATER SERVICE

by

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Reactor Engineering Division

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NITRIDED STAINLESS STEELS FOR HIGH-TEMPERATURE WATER SERVICE

by

Kermit Anderson

ABSTRACT

The report includes a discussion of the nitriding process, the variables affecting it, and an evaluation of the corrosion and wear resistance of nitrided materials for use in high-temperature, oxygenated water.

The results of concurrent investigations by Argonne, Battelle, Babcock & Wilcox, and Westinghouse are correlated and analyzed; data are presented in both tabular and graphical form.

I. INTRODUCTION

During early stages of work at Argonne on systems requiring operation of machine elements immersed in high-temperature oxygenated water, it was difficult to find structural materials with adequate wear and corrosion resistance, machinability, and suitable mechanical properties. Since high-temperature water is a very poor lubricant, elements such as gears, bearings, sleeves, cylinders, and pistons tend to wear rapidly when operated in water. In addition, galling of the parts occurs frequently.

Preliminary tests verified the expectation that case hardening extended the applicability of many steels by lengthening their useful life at heavy loads and in many cases by preventing galling where its occurrence would have rendered the metal useless. Various case hardening methods were investigated; of these, nitrided surfaces appeared to offer many advantages. Therefore, a more extensive investigation of the applicability of nitrided stainless steels for use as structural materials in high-temperature, oxygenated water was conducted by staff members of the Reactor Engineering Division at Argonne and by the Battelle Memorial Institute and the Babcock and Wilcox Company under subcontract to Argonne. The Westinghouse Atomic Power Division simultaneously carried out a complementary program. The purpose of this report is to correlate and analyze the data resulting from these investigations.

II. SUMMARY

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Nitrided surfaces appear to have definite applicability in the design of machine elements operating in high-temperature oxygenated water. The major drawback appears to be couple corrosion, but with careful design this difficulty can probably be obviated.

Gas-phase nitriding, using ammonia which is cracked on the metal surface, produces a very satisfactory nitrided case. The Malcomizing process is a commercial example of this operation. Salt bath cases are usually too thin and brittle to be of value for machine elements lubricated only by hot water.

Precipitation-hardening stainless steels appear to be the best class of stainless steels for this application; of these, Armco 17-4 PH is preferred. USS Stainless W is not too satisfactory because of the possible formation of titanium stringers which might serve as active centers for corrosion attack.

Experiments both on small samples and on actual machine element components indicate that properly nitrided surfaces improve the wear resistance of suitable stainless steels.

It would be expected that pre-nitriding surface treatment, nitriding conditions, post-nitriding surface treatment, and variables during corrosion testing affect corrosion resistance. It was found that a simple sand blasting or grit blasting is the most effective pre-nitriding treatment. Neither temperature nor percent ammonia dissociation within the range 975 - 1075F and 32 - 82% respectively, during nitriding, noticeably affected corrosion resistance. Of the two industrial recommended post-nitriding treatments, dipping the sample in dilute sodium dichromate apparently has little effect, while lapping of all nitriding surfaces to remove the white layer reduces corrosion rate, although for low oxygen concentrations the pitting of lapped specimens may be as objectionable as the higher corrosion rate of the unlapped specimen.

Low initial temperatures with low oxygen content of the water cause more rapid corrosion. Apparently, temperatures of about 500F with oxygen concentrations above 1 cc/liter tend to produce a self-healing film which protects the nitrided surface. If such a film is produced when the surface is fresh, it probably protects the metal even during subsequent periods of operation in cold water of low oxygen content. It also appears that removal of carbon dioxide, increase in pH, and improved water resistivity improve corrosion resistance. Corrosion rate is lower at low water velocities, but pitting is increased.

Couple corrosion appears to be more serious at the lower temperatures (about 200F). To reduce such corrosion it is recommended that mechanisms be so designed that a constant flow of fresh, neutral, high resistivity water could be forced between mating surfaces during operation.

III. THE NITRIDING PROCESS

A. Description

A nitrided case can be produced on stainless steel by two methods: the salt bath method and the gas-phase method.

1. Salt Bath Method

In the salt bath method, some form of a cyanide bath is employed. Both carbides and nitrides may be formed in the case, but if the conditions are properly controlled, a shallow, very hard nitride case is normally produced. A commercial process known as "Scottsonizing" is probably of this general class.

2. Gas-phase Nitriding

For gas-phase nitriding, a chemically clean metal surface is required. A clean surface is usually obtained by employing hydrogen to reduce the oxide layer on the metal at a temperature slightly above the nitriding temperature. This operation is followed by immersion of the metal for a considerable period of time in cracked ammonia to produce a nitride case. On a laboratory scale, this procedure has been used successfully at Argonne, Battelle, and Westinghouse to nitride a wide variety of stainless steels.

The Malcomizing process is used commercially by the Chapman Valve Co., Indian Orchard, Massachusetts, or by several of its licensees, including the Lindberg Steel Treating Company, Chicago, Illinois. The process is a straightforward application of the principles of gas-phase nitriding. The hydrogen used for surface cleansing is obtained by cracking ammonia in an electric arc system located upstream from the nitriding retort. The gaseous dissociation products of ammonia, consisting of H_2 and N_2 in a 3:1 proportion, are led into the nitriding retort and contacted with the surfaces to be nitrided. These gases, acting as slightly diluted hydrogen, clean the surface from oxide before the nitriding process is started. The nitriding process is initiated by turning off the external ammonia dissociation apparatus and allowing the ammonia to crack on the surface of the metal. Ionized ammonia is not involved in these processes: the cleaning action is produced by molecular hydrogen, and the nitriding action by atomic nitrogen liberated by the decomposition of ammonia on the surface to be nitrided.

B. Variables

Factors which might be expected to have the most effect on the quality of the nitrided steels are the method of surface preparation before nitriding and the variation of conditions during nitriding.

1. Surface Preparation

From the general physical chemistry of gas-metal reactions it seems apparent that the steel should present a clean surface to the nitriding atmosphere. Several proposed methods of preliminary surface preparations were considered:

- a. Sandblasting thoroughly followed by immediate nitriding. This is the standard practice of the Chapman Valve Company and is incorporated in their Malcomizing process.
- b. Superfinishing by the Chrysler Motor Corporation process.
- c. The following steps were used by Westinghouse:
 - (1) Degrease in acetone or trichloroethylene
 - (2) Wash inalconox or similar detergent
 - (3) Rinse repeatedly in distilled water
 - (4) Dry on Kleenex or similar tissue
 - (5) Apply metallographic polish finish on 0000 paper. To prevent contamination of the surface by chloride ions, the specimen should not be touched with the fingers after being washed.
- d. Various modifications of the Westinghouse treatment, such as chemical cleaning, electropolishing and vaporblasting in addition to the detergent wash.
- e. Dr. Otto Zmeskal of Illinois Institute of Technology proposed that specimens be pickled in a solution containing up to 30 % HCl and some H_2SO_4 prior to nitriding.

Leeds and Northrup Company have incorporated this procedure in their nitriding operation with the following results:

EFFECT OF PICKLING PRIOR TO NITRIDING

Pickle Bath: 10 % HCl + 10 % H_2SO_4

Nitriding: 2 hr at 1150F; ammonia dissociation: 45-48 %, plus 43 hr at 1050F with an unspecified degree of ammonia dissociation.

Condition of Specimen	18-8 Steel		14 Cr Steel	
	Case Thickness, in.	Hardness, R _{15N}	Case Thickness, in.	Hardness, R _{15N}
Pickled	0.005	93-94	0.011	93-94
Not Pickled	No Case	70-71	0.006-0.010	92-93

Armco 17-4 PH was selected as the material with which to test the various pre-nitriding treatments at Argonne. Specimens were heat treated by heating to 1950F for 1 hr, followed by a water quench and re-heating to 950F for 1/2 hr. The samples were then pickled using 10% H_2SO_4 containing about 0.1% of a pyridine inhibitor.

The pretreatments before nitriding were:

- (1) Manual alkaline clean plus dip in 10% HCl; dry
- (2) Cathodic alkaline clean plus dip in 10% HCl; dry
- (3) Anodic alkaline clean plus dip in 10% HCl; dry
- (4) Cathodic alkaline clean plus cathodic 10% HCl treatment; dry
- (5) Machined surface plus treatments 1, 2, 3, and 4
- (6) Ground surface plus treatments 1, 2, 3, and 4
- (7) Vaporblast
- (8) Vaporblast plus treatments 1, 2, 3, and 4.

Of all the above treatments, the vaporblast gave the most uniform nitrided layer both as to surface hardness and depth of penetration. The various cleaning methods tested did not show any significant improvement over the straight sandblasted samples produced and tested at the Lindberg Steel Treating Company.

Since sandblasting or vaporblasting is both simple and inexpensive and since the results are satisfactory, there seems to be little need to specify more elaborate methods of precleaning. It seems advisable, however, to specify that, after sandblasting, diligent care should then be exercised to minimize contamination of surfaces with foreign materials, particularly those of ionic nature.

It must be emphasized that the proposed surface preparation methods have as their objective the initial removal of organic matter and of heavier oxide coats together with an increase in surface area because of roughening. The final cleaning operation is believed to occur when hydrogen contacts the adsorbed oxygen film on the metal specimens immediately before the nitriding operation. The hydrogen is introduced either as gaseous hydrogen or by thermal or electrical dissociation of the ammonia used in the nitriding process.

2. Variations of Conditions during Nitriding

Case depth and case hardness will be affected by temperature and duration of the nitriding cycle, and also by the rate of ammonia throughput and the consequent degree of dissociation of the nitriding gas.

Commercial practice is fairly well established as to temperature and cycle times. The Lindberg Steel Treating Company, in the Malcomizing process, uses temperatures near 1050F with an ammonia dissociation of about 20-25 %, and a cycle time of 48 hr.

The effects of temperature, rate of gas flow, and the degree of ammonia dissociation in the nitriding process were investigated at Argonne. In these tests all specimens were Armco 17-4 PH stainless steel to permit direct comparison of the different nitride cases resulting from variation in operational conditions.

a. Description of the Nitriding Furnace

A 2 in. diameter stainless steel pipe vacuum furnace was used for the nitriding operations. The electric heating elements were divided into 3 individually controlled sections to maintain an even temperature in the furnace throughout the introduction of the cool ammonia gas. During operation there was less than a 10° temperature differential over the central 15-in. zone of the furnace. The temperature in each of the 3 sections was controlled automatically to within $\pm 5^\circ$. The gas entered one end of the furnace through a system of baffles to insure turbulent flow about the samples. The samples were cylindrical in shape, 1/2 in. diameter by 3/4 in. long, with a short, blind hole drilled in one end. They were mounted vertically in the furnace on stainless steel pegs in a stainless steel plate. Twelve samples were placed in the furnace at one time. The nitriding system and furnace are shown schematically in Figures 1 and 2, and pictorially in Figure 3.

b. Nitriding Procedure

After inserting the samples, the furnace was evacuated and heating was started. The furnace was then flushed with hydrogen for 5 min and re-evacuated. Following this, the ammonia gas was introduced at a rate of 5ℓ./min, equivalent to a velocity of 9 ft/min.

When the furnace had reached operating temperature, the flow rate was decreased until the desired degree of ammonia dissociation was attained.

It was later found that the furnace did not need to be exhausted, provided it was adequately flushed with hydrogen before beginning the heating cycling. At the end of each run ammonia flow was continued while the furnace cooled. The samples were then removed and visually examined.

FIG.1
SCHEMATIC DIAGRAM OF
NITRIDING SYSTEM

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FIG. 2
SCHEMATIC DIAGRAM
OF NITRIDING FURNACE

RE-6-11337-A

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FIGURE 3
NITRIDING APPARATUS

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c. Discussion of Operation

Two flow rates and two temperatures were investigated: 1-1/2 l./min and 4-1/2 l./min at 975F and 1075F.

The per cent dissociation, analyzed in the regular commercial manner under each of these separate conditions, is given in the following tabulation.

Flow Rate, l./min	<u>Dissociation of Ammonia, %</u>	
	<u>975F</u>	<u>1075F</u>
1-1/2	56	82
4-1/2	32	59

d. Results

Following visual examination, each sample was cut perpendicular to its axis, and the 2 pieces were mounted for microscopic examination. The nitrided case was of uniform thickness on all samples; even the blind hole sitting on the peg support showed a thin uniform case. One sample from each end of the furnace was etched and examined under the microscope. A second sample from each end was tested for hardness using a Vickers microhardness tester. Three series of indentations from the center of the samples to the outside of the case were made on each sample. Typical hardness traverses of the cases and cores are shown in Figure 4.

An examination of the micrographs and of the Vickers hardness numbers of the case and cores shows the following:

- (1) All samples exhibited a uniform nitrided coating.
- (2) The nitrided layers were 100 DPH harder when produced at a temperature of 975F than when produced at 1075F.
- (3) The hardness of the nitrided cases made at a given temperature was independent of the rate of ammonia flow.
- (4) At 1075F the depth of the nitrided case was 0.0105 in. regardless of ammonia flow.
- (5) At 975F the higher rate of ammonia flow produced a case thickness of 0.07 in., while the lower rate of ammonia flow produced a case thickness of 0.0045 in.
- (6) No white layer was observed on any of the samples. The crystals were smaller and more jagged near the outside surface in all cases; these could not be definitely identified.

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IV. WEAR RESISTANCE

Measurement of the wear resistance of metal surfaces is difficult. The most satisfactory method for evaluating a given material combination for use in a given mechanical element is to fabricate the desired element of the material combination proposed and to test the fabricated element as a component of a unit assembly. There are, however, certain screening tests which give a good qualitative indication of the relative wear resistance of metals. These tests were used in addition to simulated component and component tests for the evaluation of the mechanical applicability of nitrided steels.

A. Screening Tests

1. Falex Tests

The Falex machine, manufactured by the Faville-Levally Corporation, Chicago, Illinois, was originally developed to test the lubricity of oils and is used for this purpose by petroleum technologists. In a Falex test, standard metal tests specimens under a controlled load are used to compare the lubricating properties of oils. For the present application, the normal test procedure was reversed; the machine was employed with water as a standard lubricant to test the wear resistance of metal specimens.

a. Equipment and Test Procedure

The Falex machine is shown in Figure 5: (a) in the operating position, and (b) open for insertion of the test pieces. The test pieces, shown in Figure 6, consist of 2 anvils, or V-blocks, and a pin. In operation, the V-blocks are clamped against the pin by the 2 arms of the machine visible in Figure 5(a). The pin is rotated at 200 rpm under a predetermined load, while the test pieces are immersed in a glass dish filled with distilled water initially at room temperature. The load is observed on the front gauge, while the torque applied by the revolving pin is indicated on the gauge at the rear of the machine. Galling of the test pieces is indicated by a rapid and large change in torque and can usually be detected also by observation of the test pieces through the wall of the glass dish.

b. Experimental Data

Table I summarizes data obtained from Falex tests of several nitrided steels. The results show that the apparent coefficient of friction at low loadings was somewhat high, accounting perhaps for the abrasive loss. The wear or abrasion was greater for the specimens with a softer core material.

(a)
OPERATING POSITION

(b)
OPEN POSITION

FIG. 5
FALEX MACHINE

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FIGURE 6
TYPICAL PINS AND V-BLOCKS
FOR FALEX MACHINE TESTS

Table I

FALEX DATA ON NITRIDED MATERIALS

Test No.	Material Combinations, Pin Material V-Block Material	Time, Min	Load, lb	Sample Press, psi	Torque, in.-lb	Apparent Coeff. of Friction	Wear, in.	Appearance After Test
12	Stellite 6 USS Stainless W, nitrided, (R ₁₅ N ₉₆)	70	10	---	0-5	0.71	0.0010 ---	Burnished. Chipped at edges (water milky).
12a	Retest of samples from test 12	60	25	---	4-8-1/2	0.48	0.0001 ---	Burnished. More edge chipping (water milky).
12b	Retest of samples from test 12a	60	100	---	2-17	0.24	0.0020	Heavily scored. Galled (metal chips in water).
13	USS Stainless W, nitrided, unground Type 446 (R _C 12)	31	10	---	0-4	0.56	---	No change. Galled.
14	USS Stainless W, nitrided, unground USS Stainless W, nitrided, unground	60	10	---	0-5	0.71	0.0005 0.0009	Burnished (brown). Burnished (brown).
14a	Retest of samples from test 14	60	25	---	1-16	0.90	None 0.0005	No change. No change (water opalescent).
14b	Retest of samples from test 14a	60	100	175	2-19-1/2	0.25	0.0002 0.0028	Lightly scratched. Scratched (water murky).
14c	Retest of samples from test 14b	60	500	690	40-90	0.26	0.0021 ---	Deeply flaked. Chipped.
18	Armco 17-7 PH, heat treated, nitrided, (R ₁₅ N ₉₅)	60	10	750	0-3-1/2	0.50	None 0.0040	Smooth wear. Smooth wear.
18a	Retest of samples from test 18	60	25	1300	0-3-1/2	0.31	0.0001 None	Heat tinted, smooth wear. Heat tinted, smooth wear.
18b	Retest of samples from test 18a	60	100	5350	6-1/2-35	0.50	None None	Heat tinted, smooth wear. Smooth wear, some chipping.
18c	Armco 17-7 PH, heat treated, nitrided (R ₁₅ N ₉₅) Armco 17-7 PH, heat treated, nitrided (R ₁₅ N ₉₅)	12	500	---	8-45	0.13	---	Nitride case chipped off. Galled badly.
20	Graphitic Stellite (R _C 48) USS Stainless W, nitrided, (R ₁₅ N ₉₅)	60	10	633	0-3-1/2	0.20	0.0001 0.0007	Burnished. Smooth wear.
20a	Retest of samples from test 20	60	25	1400	0-5-1/2	0.31	0.0002 0.0003	Burnished. Smooth wear.
20b	Retest of samples from test 20a	60	100	5050	3-14	0.24	---	Burnished. Smooth wear (water murky).
21	Type 431, heat treated, (R _C 31) USS Stainless W, nitrided, (R ₁₅ N ₉₅)	60	10	670	0-4	0.57	0.0014 0.0013	Badly scored. Chipped and galled.
22	Type 420, heat treated, (R _C 47) USS Stainless W, nitrided, (R ₁₅ N ₉₅)	60	10	815	0-3-1/2	0.50	0.0003 0.0017	Scored. Smooth wear (water murky).
22a	Retest of samples from test 22	60	25	1400	2-3	0.17	0.0020 0.0008	Badly scored. Chipped and galled (water murky).
28a	USS Stainless W, Malcomized, (R ₁₅ N ₉₁) USS Stainless W, Malcomized, (R ₁₅ N ₉₄)	60	10	1070	1-9	1.27	None 0.0006	Smooth wear. Smooth wear.
28b	Retest of samples from test 28a	60	25	1990	6-7	0.40	None None	Finely scratched. Finely scratched.
28c	Retest of samples from test 28b	60	100	5660	15-17	0.24	0.0010 None	Finely scratched. Finely scratched, scored.
28d	Retest of samples from test 28c	17	500	---	10-20	0.06	---	Galled. Galled.
123a	Stellite 3 (R _C 58) Type 347, nitrided	60	10	490	2-4-1/2	0.64	None 0.0018	Smooth wear. Smooth wear.
123b	Retest of samples from test 123a	60	25	1120	4-5	0.28	0.0030 0.0001	Very smooth wear. Abraded.
103a	Armco 17-4 PH, Chrome plated, (R ₁₅ N ₈₆) Stellite 3, (R _C 47)	60	10	280	1-1/2-4-1/2	0.63	0.0005 0.0004	Smooth wear. Finely scratched.
103b	Retest of samples from test 103a	60	25	615	4-1/2-12-1/2	0.71	0.0006 0.0001	Smooth wear. Lightly scratched.
103c	Retest of samples from test 103b	60	100	2100	10-1/2-17	0.24	0.0006 0.0001	Polished. Lightly scratched.
48	Armco 17-7 PH, (R _C 36) Type 446, (R _C 13)	1	10	---	1-1/2	---	---	Galled. Galled.

It was noted in every Falex test of a nitrided material that, after the test had been in progress for a short time, cloudiness developed in the water; apparently very small particles of the case were rubbed off. Furthermore, after short test periods with loads as light as 10 lb, the corners of the specimens were usually chipped off and became progressively worse with increased time or pressure. These phenomena indicate extreme sensitivity of performance to design. The Falex machine operates with somewhat irregular loading as the free end of the test pin does not operate in a bearing. This is considered to be the cause of chipping, and such chipping would certainly be the result of inherent brittleness of nitrided surfaces.

2. Low-Temperature Materials Wear Test

Tests were made in low-temperature (185F) water exposed to the atmosphere in a modified drill press operated at a speed of 0.03 fps at the periphery of the test specimen. These tests represented the first efforts to screen out unpromising materials. Two types of sample, shown in Figure 7, were used in the test.

a. Solid Ring Samples

A raised solid ring, 1/2 in. OD by 7/16 in. ID, cut on the face of a 3/4-inch disc, rubs against the flat surface of a second 3/4 in. OD disc. The surface contact area is 0.0442 sq in.

b. Interrupted Surface Samples

A raised ring, 3/4 in. OD and 1/2 in. ID, with 4 cuts, 1/8 in. wide at 90° to provide the interrupted surface, rubs against the flat surface of a second 3/4 in. OD disc. The surface contact area is 0.183 sq in.

The 2 types of specimens were interchangeable in the apparatus. Specimen loads and pressure may be varied up to 750 psi. Specimens were checked periodically, approximately every hour, for surface condition and evidence of wear or corrosion.

Wear Test Results on Nitrided Materials

Test Type	Material		Evaluation
	Rotor	Stator	
1-A	Type 410	Type 410, Malc.	Doubtful
1-A	USS Stainless W	USS Stainless W	Acceptable
1-A	USS Stainless W	Haynes Alloy 25	Doubtful
2-B*	USS Stainless W, Malc.	USS Stainless W, Malc.	Acceptable

*Designation indicates use of a Type B specimen, Figure 7, in a high-temperature wear test autoclave.

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FIG. 7
SPECIMENS FOR MATERIALS WEAR TESTS

RE-6-11325-A

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Specimens used in these tests are shown under magnification in Figures 8 and 9. Figure 8 shows a photograph and a profilometer tracing of the surface of a flat sample of Malcomized USS Stainless W after test. In Figure 9 the same information is presented for Malcomized Type 410. The superiority of the precipitation hardening steel is attested both by the photograph and by the profilometer traces; confirming results were obtained in the Falex tests.

B. Simulated Component Tests

Simulated component tests performed at Argonne were qualitative only, but did tend to indicate that under light loadings nitrided specimens showed comparatively high wear resistance. An example of the test specimen, used with the high-temperature wear test apparatus, shown in Figure 10, is the ball and race specimen shown in Figure 7 (C).

Piston and cylinder wear tests conducted at Westinghouse showed that opposing samples of nitrided Type 347 stainless steel exhibited the lowest weight loss due to abrasion. The weight loss was 0.64 mg/lb load per 10^6 cycles as compared to opposing samples of a very similar steel, Type 304, in the unnitrided condition which had a corresponding loss of 3200 mg/lb load.

In the Westinghouse Sleeve Bearing Test, chrome-plated Armco 17-4 PH versus nitrided Resistal #3311 showed a loss of 8.3 mg/lb load per 10^6 cycles, while chrome plating against Armco 17-4 PH had a corresponding loss of 61 mg/lb load per 10^6 cycles. This indicates a somewhat superior wear resistance for nitrided surfaces as compared to chrome-plated surfaces.

C. Component Tests

The component tests conducted at Argonne confirm the wear resistance of nitrided steels; for example:

Two opposing USS Stainless W gears completed in excess of 2,500,000 revolutions without failure of the gear cases. Tooth load was 72 lb/in. at 1.65 to 2.65 cycles/sec.

Two Malcomized Type 410 gears completed 3,853,000 cycles with no undue wear plus an additional 3,500,000 cycles without failure and with only slight additional damage. Tooth load was 260 lb/in. at 5 cycles/sec.

A Malcomized Type 410 lead screw and ball nut assembly with Haynes Alloy 25 balls completed 154,980 revolutions without failure, under 50-lb load, at a speed of 14 rpm over a 10.25-in. stroke.

A - SURFACE OF FLAT SAMPLE SHOWING WEAR GROOVE - 10X

FIGURE 8

MALCOMIZED USS STAINLESS W FLAT SAMPLE AFTER
WEARING BY MALCOMIZED USS STAINLESS W SOLID
RING SAMPLE. TESTED FOR 6 HR AT 680 psi
SAMPLE PRESSURE IN 185F WATER.

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A - SURFACE OF FLAT SAMPLE SHOWING WEAR GROOVE -10X

TYPICAL WEAR GROOVE
0.054 IN.

B - PROFILOMETER TRACES SHOWING DEPTH OF WEAR
ON FLAT SAMPLE

FIGURE 9

MALCOMIZED TYPE 410 FLAT SAMPLE AFTER WEARING
BY MALCOMIZED TYPE 410 SOLID RING SAMPLE.
TESTED FOR 6 HR AT 680 psi SAMPLE PRESSURE
IN 185F WATER.

FIGURE 10
HIGH-TEMPERATURE WEAR TESTER.

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V. EFFECT OF VARIABLES ON CORROSION RESISTANCE OF NITRIDED MATERIALS

A. Pretreatment of Samples

Samples of Armco 17-4 PH and USS Stainless W, pretreated as described on page 10, were static corrosion tested in autoclaves containing 500F oxygenated water (30 cc $O_2/2.$). Representative data are shown in Table II and may be summarized as follows:

1. With pretreatment including electrocleaning, alkaline soaking and degreasing, the rate of corrosion averaged about $+0.35 \text{ mg}/(\text{cm}^2)(\text{mo})$. Some samples exhibited a black lustrous film, while on others the black film was overlaid with red rust spots.

2. With pretreatments including sandblasting, the corrosion rate varied from -2.42 to $+0.96 \text{ mg}/(\text{cm}^2)(\text{mo})$. Those samples showing a loss in weight had a reddish rusty appearance; others showed black corrosion products mixed with rust.

B. Nitriding Conditions

The data in Table II further indicate that neither the per cent dissociation of the ammonia gas nor the nitriding temperature affected significantly the corrosion rate of the samples tested.

C. Post-Nitriding Treatment

1. Lapping of Specimens

Most authorities agree that, for normal applications where nitrided cases are used, the best practice is to grind off about 0.002 in. of the outer case, prior to placing the material into service. This outer, or white layer, is a hard and quite brittle skin on the nitrided case in which conversion of the original metals to nitrides is relatively complete. Such an outer case corresponds to the so-called "white-layer" present when ordinary nitriding steels have been case-hardened.

Data from Table II show that grinding 0.002 in. from the surface of the nitrided sample produced mixed results. There was no difference in the corrosion rate of two portions of the sample where one-half of the sample had 0.002 in. ground from the surface while the other half was in the as-nitrided condition.

The results of loop corrosion tests made by the Babcock and Wilcox Company on lapped and unlapped samples of Armco 17-4 PH are given in Table III.

Table II

EFFECT OF PRETREATMENT AND NITRIDING CONDITIONS ON CORROSION RESISTANCE IN 500F WATER

Material	Sample No.	Pretreatment	Time, hr	pH	Weight Change, mg/(cm ²)(mo)	Appearance After Test
17-7 PH	2587	Rough machined, alkaline soak and degrease	324	7.2-6.7	+0.360	Adherent dull black coating.
17-4 PH	2601	Alkaline soak, degrease, electrolytically clean at 200F, rinse, acid dip and rinse.	324	7.1-6.7	+0.37	Loose, streaked rust over black coating.
17-4 PH	2601 B-4	Alkaline soak, degrease, electrolytically clean at 200F, rinse, acid dip and rinse.	324	7.1-6.7	+0.37	Loose, streaked rust over black coating.
17-4 PH	2711	Clean same as 2601. 1/2 of sample ground to a depth of 0.002 in. nitrided. 1075F, 50 hr; dissociation 85%.	323	6.7-7.7	+0.268	No difference between ground and normal surfaces.
17-4 PH	2711 C & D-1	Clean same as 2601 1/2 of sample ground to a depth of 0.002 in. nitrided 1075F, 50 hr; dissociation 85%.	323	6.8-7.8	C:+0.434 D-1:+0.96	---
17-4 PH	2711 C-1, H, E, & F	Grit blast. 1/2 of sample ground to a depth of 0.002 in. nitrided 1075F, 50 hr; dissociation 85%.	---	---	C-1:-1.15 E:-0.782 F:-2.42 H:-2.16	---
17-4 PH	2712 F & D	Grit blast. Nitrided 1075F, 50 hr; dissociation 58%.	335	6.9-7.5	F:+0.895 D:+0.915	Dull streaked black and gray coating.
17-4 PH	2713 T	Grit blast. Nitrided 975F, 50 hr; dissociation 32%. 1/2 of sample ground after nitriding.	323	6.9-7.3	T:+0.162	Ground surface has lustrous black tarnish, unground surface dull black.
17-4 PH	2713 J & S	Same as 2713-T except samples not ground.	323	6.7-7.9	J:+0.561 S:+0.227	Dull black adherent coating.
17-4 PH	2714	Same as 2713-T except dissociation is 56%.	323	6.6-7.7	R:+0.100	Dull black adherent coating except loose rust at point of contact between the samples.
17-4 PH	2711 C & D	Same as 2714-R except not ground.	323	6.8-7.8	C:+0.434 D:+0.96	Loose rust over black coating.
USS Stainless W	2594	Wash and degrease; Malcomize, machine.	324	6.4-7.1	-1.91	Loose rust coating.
17-7 PH	2594	Wash and degrease; Malcomize. Sample ground to a depth of 0.002 in. after malcomizing.	736	7.1-7.1	-0.029	Lustrous black tarnish.
17-7 PH	2598	Same as 2594.	736	7.1-7.1	-0.029	Lustrous black tarnish.

Table III

EFFECT OF REMOVAL OF THE WHITE LAYER ON CORROSION OF
MALCOMIZED ARMCO 17-4 PH STAINLESS STEEL

Gas Content of Water	Sample Number	Condition	Time, hr	Weight Change, mg/(cm ²)(mo)
2cc O ₂ /ℓ.	C-1	Lapped	0-250	+0.68
	C-2	Lapped	0-250	+0.44
	ANL-1	Unlapped	0-500	+0.70
	ANL-2	Unlapped	0-500	+0.68
Degassed Water (0.1-0.2cc O ₂ /ℓ.)	C-1	Lapped	0-500	-2.92
	C-2	Lapped	0-500	-2.84
	ANL-3	Unlapped	0-500	+0.04
500cc H ₂ /ℓ.	C-1	Lapped	0-250	+0.20
	C-2	Lapped	0-250	+0.34
	ANL-4	Unlapped	0-250	+0.44
0-5cc O ₂ /ℓ. (Variable)	C-1	Lapped	0-250	-0.06
	C-2	Lapped	0-250	-0.23
	ANL-5	Unlapped	0-250	+1.74

In discussing the data given in Table III, Green and Price¹ of Babcock and Wilcox Company stated:

"The external surface produced by nitriding ("white layer") displayed inadequate resistance to general corrosion in all test conditions. This general corrosion, however, appeared to be less serious than the progressive pitting noted in all lapped specimens tested in water containing 0.1 to 0.2 cc O₂/ℓ."

2. Sodium Dichromate Treatment of the Surface

Several authorities on nitrided materials insist that a dip in 0.02N sodium dichromate is essential in order to develop the maximum corrosion resistance of a nitrided case to water. They admit that such weak oxidizing media would not truly passivate the nitrided surface, but believe that such treatment would "dissolve" any accidentally acquired iron particles nestling in the nitrided surface matrix which, if left undisturbed, might

¹H. T. Green, D. E. Price, "A Study of the Corrosion Behavior of Some Nitrided and Unnitrided Stainless Steels in High-Temperature Distilled Water," Babcock and Wilcox Company Report No. 5309, March 26, 1952, p. 3.

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become active centers for initiating corrosion reactions. As a check, a simple test was carried out at Argonne. Carbonyl iron powder was treated with a 5% solution of sodium dichromate for several hours at temperatures below the boiling point; the supernate was filtered off and tested spectrographically for iron. None was found. These results would indicate that no real good is to be obtained from such a dichromate dip, although the practice probably would be harmless.

D. Variables Significant During the Corrosion Test

Some general factors which have been found to affect the rate of corrosion of stainless steels in water are:

1. Oxygen Concentration

Tests were conducted at Argonne in room temperature distilled water under 3 conditions of oxygenation with Malcomized USS Stainless W and Armco 17-4 PH as the test materials:

- a. Distilled water saturated with a continuous stream of argon. Analysis for dissolved oxygen showed approximately 1.5 cc O_2/l .
- b. Distilled water saturated with a continuous stream of air. Analysis for dissolved oxygen showed approximately 5.2cc O_2/l .
- c. Distilled water saturated with a continuous stream of oxygen. Analysis for dissolved oxygen showed approximately 17.6cc O_2/l .

Three covered 2- $\frac{1}{2}$ beakers were used as test vessels, each containing approximately 1500 ml of water. In order to displace soluble corrosion products which might be formed, deionized, distilled water was run through each beaker at the rate of 5 ml/min. The constant addition of fresh, deionized, distilled water maintained the pH of the test water at about 7 and kept its resistivity high. The gases were added through fritted glass discs to get uniform dispersion in the water. The metal specimens were suspended in the water by a quartz fiber cord.

A fourth test beaker was added later in order to test samples in static nonchanging water, saturated with a continuous stream of air. Just enough water was added to this beaker to make up for evaporation losses.

The specimens were heat treated at Argonne before Malcomizing. The USS Stainless W samples were heat treated at 1925F for 10 min, air cooled, then heated to 950F for 30 min and water cooled. This produced a hardness of R_C49 . The Armco 17-4 PH specimens were heated to 1900F for 1/2 hr, water cooled, then heated to 950F for 30 min and water cooled. This treatment produced a hardness of R_C43 to 44.

The specimens were then Malcomized by Lindberg Heat Treating Company and were tested in the as-Malcomized condition. The USS Stainless W specimens were spalled to some extent. (Except for the band around the center, these specimens have the same appearance as before test.)

The test conditions and corrosion data are given in Figures 11 and 12.

From these results it is seen that:

(1) The presence of dissolved oxygen decreases rather than increases corrosion at room temperature.

(2) Specimens e and f, in beaker 3, and g and h, in beaker 4, were tested under identical conditions except that the water in beaker 3 was constantly changed. Although the corrosion rates of these specimens were not greatly different, appearance indicates that specimens e and f in beaker 3 had suffered greater damage. The explanation for this difference probably lies in the fact that the pH in beaker 4 (static) was higher than that in beaker 3.

(3) The USS Stainless W samples stood up slightly better than the Armco 17-4 PH.

(4) The points of contact of the quartz fiber thread with the samples were the points of first and worst corrosion.

2. Presence of CO₂

Early work by Westinghouse showed that Graphitar 14 placed in contact with nitrified stainless steel produced an accelerated corrosion effect in room temperature water. It was thought that this effect might be due, at least in part, to oxidation of the carbon to CO₂, and the resultant corrosive effect of this gas.

To test the effect of CO₂ on the corrosion of nitrified stainless steels, samples were suspended in room temperature distilled water through which CO₂ was passed. Within 2 hr a flaky black coating of corrosion product formed on the surface of the nitrified steel; this could be removed by scraping with a steel blade.

Subsequent confirmatory work was done at Argonne. Two nitrified samples of USS Stainless W were suspended in carbonated water for 4 hr. Carbonation was obtained by bubbling CO₂, made by sublimation of dry-ice, through the water. A fritted glass disc was used for dispersing the CO₂ into fine bubbles. Figure 13(A) shows the specimen before treatment, while Figure 13(B) shows the corroded specimen.

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SAMPLE	A	B	C	D	E	F	G	H
BEAKER NO.	1	1	2	2	3	3	4	4
GASIFICATION	ARGON SATURATED		OXYGEN SATURATED		AIR SATURATED		AIR SATURATED	
WATER COND.	CHANGED		CHANGED		CHANGED		STATIC	
AVG. DISSOLVED O ₂ cc/liter	1	1	17	17	5	5	5	5
TIME, hr	336	336	314	314	314	314	216	216
WT. CHANGE, mg/cm ² (mo)	0.43	0.33	0.20	0.55	0.50	0.30	0.22	0.65

FIG. 11
CORROSION OF MALCOMIZED ARMCO 17-4 PH IN
WATER AT ROOM TEMPERATURE. (SAMPLE LOT NO. A-2522;
HEAT NO. 30273)

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SAMPLE	A	B	C	D	E	F	G	H
BEAKER NO.	1	1	2	2	3	3	4	4
GASIFICATION	ARGON SATURATED		OXYGEN SATURATED		AIR SATURATED		AIR SATURATED	
WATER COND.	CHANGED		CHANGED		CHANGED		STATIC	
AVG. DISSOLVED O ₂ cc/liter	1	1	17	17	5	5	5	5
TIME, hr	336	148	314	120	120	314	216	216
WT. CHANGE, mg/(cm ²)(mo)	0.37	0.62	0.095	0.60	0.35	0.35	0.60	0.79

FIG. 12
CORROSION OF MALCOMIZED USS STAINLESS W IN
WATER AT ROOM TEMPERATURE. (SAMPLE LOT NO. A-2521;
U.S.S. HEAT NO. 8-X-6357)

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A

B

FIGURE 13
EFFECT OF CARBON DIOXIDE ON CORROSION OF
NITRIDED USS STAINLESS W

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The higher concentrations of CO_2 definitely corrode nitrided steels quite rapidly; hence, it is assumed that lower concentrations of CO_2 would act as a corrodant at some functionally connected rate.

3. Temperature

Little corrosion data is available from tests in which temperature alone was varied while such variables as metal environment, oxygen concentration, pH, and conductivity of water were held as nearly constant as possible. Hence, it is difficult to deduce the effect of temperature on the corrosion of nitrided steels.

The only data available in which temperature was varied under controlled conditions are those resulting from work at Battelle Memorial Institute;² pertinent data are given in Table IV.

Table IV

EFFECT OF TEMPERATURE ON CORROSION OF NITRIDED STAINLESS STEELS

Media: Degassed Water

Material	Temp., F	Time, days	Appearance After Test
USS Stainless W	Room	42	Dark tarnish patches, scattered light rust spots
Armco 17-4 PH	200	42	Dark coating, some rust spots
USS Stainless W	200	42	Dark film and some rusting
Armco 17-4 PH	200	42	Dark film and some rusting
USS Stainless W	500	31	Black coating
Armco 17-4 PH	500	31	Black coating

These observations indicate that higher temperatures give less corrosion. However, even the "degassed" water used in these experiments contains some dissolved oxygen. This oxygen will react at the higher temperatures to form a black, adherent oxide coating, tending to be impervious to further penetration by oxygen. Such an action might be a true passivation, and as long as the nitrided body is subject to extensive wear and corrosion only under such high temperature conditions, the nitrided case should be satisfactory for any reasonable use. After such a film is formed, it should then be reasonable to permit significantly long residences in colder water if reasonable purity of such water could be maintained.

²H. A. Pray, et al., "The Corrosion of Nitrided Stainless Steels in Degassed and Oxygenated Water," BMI-742, May 1, 1952

4. pH of Water

Experimental data as to the effect of pH of the water on the corrosion behavior of nitrided materials are very limited, but some indirect evidence is given on page 30. Samples of Malcomized USS Stainless W and Malcomized Armco 17-4 PH were tested at room temperature in two beakers containing about 5 cc O_2 /l. In one beaker, fresh water was continuously added, with the overflow going to the drain; in the other beaker the water was stagnant. The average pH's in the 2 vessels were about 7.0 and 7.4, respectively. The samples showed slightly less corrosion at 7.4 than at 7.0.

5. Conductivity of the Water

No direct data is available to show the effect of water conductivity on the corrosion of nitrided materials in the presence of small amounts of oxygen, i.e. 2 cc O_2 /l. Available data on stainless steels (Type 304 and Type 316) that have not been nitrided show the weight changes in a 500-hr test at 500F to be about 4 times as great in system water not circulated through an ion exchanger as that in water which was circulated through an iron exchanger. The resistivity of the former was about 20,000 ohm-cm, whereas the resistivity of the latter was 500,000 ohm-cm higher. It is believed that nitrided stainless steel would also show increased corrosion in water of high conductivity as compared to that of low conductivity in the presence of this amount of oxygen.

6. Velocity of Water Relative to the Corroding Surface

As part of the general Argonne corrosion program, tests on miscellaneous structural materials were run at Babcock and Wilcox at 500F under several oxygen concentrations at 2 velocities, namely, 30 fps and 1 fpm. The results on nitrided materials, i.e., Malcomized Type 410 stainless steel, were somewhat doubtful due to the hardness of the case which resulted in chipping, particularly at the clamping areas. The trend, however, indicated that the over-all corrosion was greater at the high velocity, but the low velocity favored localized attack such as pitting and contact corrosion.

7. Contact Corrosion

Work at Westinghouse first pointed out the significance of contact corrosion as applied to the usefulness of nitrided bodies on contact with water media. It was found that a contact cell, in which Graphitar 14 or graphite was coupled with the nitrided material, furnished an excellent accelerated corrosion test for the nitrided material.

The coupling of graphitic materials with stainless steel and a resultant rapid corrosion process, even in high-humidity air, had long been known to the valve industry. The Chapman Valve Company has stated its experience in storing nitrided valves in a humid atmosphere in which graphite packings were employed. Such valves were observed to have completely rusted-out stems after brief periods.

The possibility that such types of corrosion might make nitrided machine components useless for service in water media led to a contact corrosion study by the Battelle Memorial Institute in which nitrided USS Stainless W and Armco 17-4 PH were tested uncoupled with Stellite 3, Stellite 6, Stellite 21, graphite, Graphitar 14, Type 410 and Haynes Alloy 25. A description of this work, together with a complete record of the observations, is given in Appendix B.

Based on the results, Pray and Peoples of Batelle Memorial Institute concluded that:

- "1. The nitrided samples of the Armco 17-4 PH and USS Stainless W alloys (both coupled and uncoupled) rusted when exposed in degassed or oxygenated water at room temperature and 200F, while no rust was observed on the samples exposed at 500F.
- "2. Uncoupled samples of the stellites 3, 6, and 21, Haynes Alloy 25, graphite, Graphitar 14, and chromium-plated Armco 17-4 PH exhibited the same corrosion resistance regardless of the test temperature.
- "3. At room temperature and 200F, the majority of the couples showed a buildup of corrosion product along the periphery of the contact area.
- "4. At 500F, little or no buildup was observed. However, pitting was detected in the contact area on the samples of Stellites coupled with the nitrided alloys. Although the pitting was not extensive, it might become serious after long exposures.
- "5. Of the couples tested, only the Haynes 25 versus chromium plated 17-4 PH exhibited satisfactory corrosion properties under all test conditions.
- "6. Significant differences in the pH and specific resistance between the oxygenated and degassed water were observed only for the 200F tests."

One of the most complete tests of a nitrided component was carried out at Westinghouse. A 7-in. valve, which had been nitrided at Chapman Valve Company, was given a thorough test under a wide range of operating conditions. These conditions included 2,500 hr in cold water and 2,500 hr in oxygenated water at 500F. Although the valve was idle during both periods of operation for as long as 2 months at a time, its characteristics were acceptable during the entire test run. During either test period

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no significant contact corrosion occurred at points where the nitrided surface touched Stellite or other unnitrided alloys. Some rust was found on the as-nitrided surfaces, but only a few minor pits were observed on the surfaces from which the outer 2 mils of case had been removed. It appears from this test that under any reasonable operation sequence, contact-type corrosion is not too significant. In fact, this test supports the contention that contact corrosion with nitrided surfaces can be materially reduced by pretreatment in 500F water to form a protective oxide layer prior to use in cooler water.

VI. SELECTION OF PREFERRED STEEL

Various tests were carried out which, together with known mechanical and physical properties of the stainless steels, led to the selection of Armco 17-4 PH as the preferred material for nitriding purposes. A general discussion of the results with typical stainless steels is given below.

A. Austenitic Steels

These materials, typified by Type 347 and Type 304 stainless steels, show no worse irregularities after nitriding than other acceptable types. However, the general feeling in industry is that a nitrided case should be supported by a reasonably hard and elastic core. This belief is substantiated by the high abrasion losses for softer materials, as discussed in Section IV-A and listed in Table I.

B. Martensitic Stainless Steels

These materials, typified by Type 410 and Type 440C stainless steels, are hardenable steels. However, in the unnitrided condition they are not acceptable from the standpoint of corrosion resistance; as shown in Tables V and VI the same is true in the nitrided condition, at least in the presence of high (30 cc/l.) oxygen concentrations.

C. Precipitation Hardening Steels

Three members of this class of stainless steels received a great deal of attention because of their apparently more desirable physical properties. These were USS Stainless W, Armco 17-7 PH, and Armco 17-4 PH.

1. USS Stainless W

This steel develops good physical properties after proper hardening treatment and shows excellent resistance to wear in the nitrided condition. However, the element used to promote precipitation of grain boundary carbides is titanium, which tends to deposit irregularly and forms slag inclusions and titanium stringers. As a result, USS Stainless W shows bad spalling of the nitrided case in the vicinity of the stringers. Figure 14

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Table V

COMPARISON OF AUTOCLAVE CORROSION OF MALCOMIZED STAINLESS STEELS IN WATER
AT 500F WITH AN INITIAL OXYGEN CONCENTRATION OF 30 CC/L.

Material	Total Exposure Time, mo	Weight Change, mg/(cm ²)(mo)	Comments
Type 440C	6	-2.24	In early stages dull gray to black non-adherent coating. In later stages rust and pitting began to appear.
Type 410	11	+0.023	Rust streaks began to appear on faces of sample at end of second month. By the third month loose rust had increased. Toward the latter part of the test period black corrosion products predominated. Maximum corrosion rate: $\sim 0.5 \text{ mg}/(\text{cm}^2)(\text{mo})$.
Type 347	2	-0.38	Loose rust over blue and yellow tarnish.
USS Stainless W	10	-0.00	Adherent black coating which is slightly lustrous on ground face. Some rusting near edge. Maximum corrosion rate: $\sim 0.108 \text{ mg}/(\text{cm}^2)(\text{mo})$.
Armco 17-7 PH	11	-0.086	Lustrous straw-colored tarnish began to appear after second month. After 6 months dull black powder was observed.
Armco 17-4 PH	14	+0.530	Early appearance of a lustrous black coating. No change occurred for the first 11 months and the corrosion rate remained below $0.05 \text{ mg}/(\text{cm}^2)(\text{mo})$ except for the first, second, seventh, and tenth periods.
Type 446*	0.5	-4.0 to -12.1	Completely and heavily rusted in two weeks.

*Tested at 200F in open air.

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Table VI

LOOP CORROSION OF MALCOMIZED STAINLESS STEELS
IN 500F WATER

Gas Content: 2 cc O₂/l. (Avg)

<u>Material</u>	<u>Sample No.</u>	<u>Weight Change, mg/(cm²)(mo)</u>	
		<u>0-250 hr</u>	<u>250-750 hr</u>
USS Stainless W	A-1	+ 0.46	+ 0.05
USS Stainless W	A-2	+ 0.28	- 0.05
USS Stainless W	A-3	+ 0.18	+ 0.03
Armco 17-7 PH	B-1	+ 0.50	+ 0.04
Armco 17-7 PH	B-2	+ 0.44	- 0.01
Armco 17-7 PH	B-3	+ 0.50	- 0.03
Armco 17-4 PH	C-1	+ 0.68	- 0.03
Armco 17-4 PH	C-2	+ 0.44	+ 0.03
Type 329	D-1	+ 0.48	+ 0.03
Type 329	D-2	+ 0.90	- 0.03
Type 431	E-1	+ 0.86	+ 0.01
Type 431	E-2	+ 1.40	- 0.08
Type 440A	F-1	+ 0.96	- 0.04
Type 440A	F-2	+ 1.46	- 0.08
Type 446	G-1	+ 0.36	- 0.03
Type 446	G-2	+ 0.16	+ 0.04
Type 446	G-3	+ 0.18	- 0.02

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A. PARALLEL TO ROLLING DIRECTION

B. NORMAL TO ROLLING DIRECTION

FIGURE 14
STRINGERS IN USS STAINLESS W

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illustrates pitting due to inclusions in the metal which are attributed to titanium stringers. No such inclusions appear in photographs of any of the other precipitation hardening stainless steels.

Tests were carried out at Argonne and Westinghouse to determine the effect of titanium content on the hardness, corrosion resistance, and condition of the nitrided case. Actually, mill shipments of USS Stainless W to Westinghouse varied from 0.57 to 1.14 % titanium, while the published analysis showed a nominal 0.70 % titanium content.

Figure 15 shows the effect of titanium content on hardness of USS Stainless W precipitation hardened for 1/2 hr at 950F. The hardness is not increased by titanium content above 0.82 %. Apparently, this represents the limit of solid solution at the solution annealing temperature of 1900F.

Experimental nitriding showed that the higher titanium contents resulted in inferior nitrided cases. In general, specimens having a titanium content greater than 0.70 % showed inferior cases in terms of corrosion resistance, to those of lower titanium content. Table VII shows results on surfaces ground parallel to the rolling direction.

Table VII

INFLUENCE OF TITANIUM CONTENT ON PITTING CORROSION
OF NITRIDED USS STAINLESS W

<u>Heat No.</u>	<u>Ti Content, %</u>	<u>Surface Appearance</u>
X14426	0.57	Several small pits
5X9573	0.64	Good
X15082	0.68	Good
X13482	0.70	Good
X18765	0.71	Good
4X7854	0.73	Streak of pits
X15110	0.83	Streak of pits
X15086	0.88	Pitted
X18527	0.89	Pitted
X15540	0.95	Good (forging billet)
X15521	0.98	Pitted
X19271	1.10	Pitted
X26055	1.14	Streak of pits

W.K. ANDERSON:MC,6-30-52

FIG. 15
HARDNESS VS TITANIUM
CONTENT OF USS STAINLESS W
AGED 1/2 HR AT 950F

NR-G-3055-A

2. Armco 17-7 PH

Armco 17-7 PH shows satisfactory corrosion resistance and excellent resistance to wear in the nitrided condition but is difficult to obtain in the desired shapes for machine elements.

3. Armco 17-4 PH

Armco 17-4 PH shows satisfactory corrosion resistance and excellent resistance to wear in the nitrided condition; therefore it was selected as the preferred material for nitriding.

VII. CONCLUSIONS

A. Nitriding Process

Practically all stainless steels can be nitrided.

Of the various pretreatments studied, the best found was some form of grit blasting, the operation widely preferred in industry.

Temperatures of 975F and 1075F produce satisfactory nitrided cases at ammonia velocities of 2.4 and 7.2 fpm. The case depth is a function of ammonia flow rate at the lower temperature, but not at the higher temperature.

The case soundness attainable for USS Stainless W is related to the titanium content.

B. Wear Resistance

Nitrided cases improve the wear resistance of stainless steels.

The best of the nitrided stainless steels, with respect to wear resistance, appear to be the precipitation hardening steels, probably because of the better support for the case provided by the higher hardness of the core.

C. Corrosion Resistance

Temperature and per cent ammonia dissociation during nitriding do not noticeably affect corrosion resistance.

The corrosion resistance of nitrided stainless steel is apparently unaffected by treating the sample in a 0.02N solution of sodium dichromate before the steel is used in water at 500F.

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Lapping of all nitrided surfaces to remove the "white layer" is generally recommended, though for low oxygen concentrations the pitting of lapped specimens may be as objectionable as the higher corrosion rate of the unlapped specimens.

Corrosion resistance seems to improve as a function of the following variables of corrosion testing:

1. Increase in oxygen concentration (0 to 30 cc/l. of water).
2. Increase in pH (7 to 7.5).
3. Increase in water resistivity (20,000 to 500,000 ohm-cm).
4. Increase in temperature (70 to 500F).
5. Removal of carbon dioxide.
6. Lower water velocity (although pitting increases at lower velocity).

The most serious drawback to the use of nitrided stainless steels in high-temperature water appears to be couple corrosion and extreme caution in design using such components should therefore be used.

D. Preferred Steel

Armco 17-4 PH appears to be the most satisfactory of the stainless steels for the purpose of nitriding.

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APPENDIX ACOMPILATION OF CORROSION TESTS OF NITRIDED STAINLESS
STEELS AT ARGONNE

The following data, complete up to March 7, 1952, have been compiled from general Argonne corrosion reports.

In the interests of uniformity and brevity, the water conditions of the various tests have been coded and are tabulated below. Distilled water was used in preparation of all solutions. Except where otherwise indicated, tests were run under static conditions in individual autoclaves.

All samples were Malcomized by the Lindberg Steel Treating Company, Chicago, Illinois, unless otherwise noted.

<u>Code</u>	<u>Condition</u>
Degas	Degassed water.
Air	Air saturated water (glass container unless otherwise specified if at room temperature). Calculated initial oxygen concentration = 5 cc/l.
Air-Open	Air saturated water open to atmosphere during test. Air stream through solution was maintained throughout test. Calculated oxygen concentration; 5 cc/l. at room temperature, 0.9 cc/l. at 200F.
Argon-Open	Argon saturated water open to atmosphere. Argon stream through test solution was maintained throughout test. Analyzed oxygen concentration was approximately 2 cc/l. at room temperature.
O ₂ -Open	Oxygen saturated water open to atmosphere during test. Oxygen stream through test solution was maintained throughout test. Analyzed oxygen concentration at room temperature was approximately 20 cc/l.

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Code	Condition
O ₂ -30	Oxygen dissolved in degassed water at room temperature under partial pressure of 1 atm at start of test. (Calculated oxygen concentration = 30 cc/l.).
O ₂ -1500/2300	Autoclave connected to oxygen bottle. Partial pressure of oxygen above water in autoclave maintained between 335 and 510 psia during test. Calculated oxygen concentration = 2300 cc/l.
O ₂ -2300/3300	One autoclave containing 6 samples in individual baskets connected to oxygen bottle. Partial pressure of oxygen above water in autoclave maintained between 510 and 730 psia during test. Calculated oxygen concentration = 2300/3300 cc/l.
O ₂ -1500/2300-pH 11	Autoclave connected to oxygen bottle. Partial pressure of oxygen above sodium hydroxide solution in autoclave maintained at between 335 and 510 psia during test. Initial pH = 11. Calculated oxygen concentration = 1500/2300 cc/l.
O ₂ -1500-pH 9	Autoclave connected to oxygen bottle. Partial pressure of oxygen above sodium hydroxide solution in autoclave maintained at 335 psia during test. Initial pH = 9. Calculated oxygen concentration = 1500 cc/l.
O ₂ -1500 pH 10	Autoclave connected to oxygen bottle. Partial pressure of oxygen above sodium hydroxide solution in autoclave maintained at 335 psia during test. Initial pH = 10. Calculated oxygen concentration = 1500 cc/l.
O ₂ -1500-pH 11	Autoclave connected to oxygen bottle. Partial pressure of oxygen above sodium hydroxide solution in autoclave maintained at 335 psia during test. Initial pH = 11. Calculated oxygen concentration = 1500 cc/l.

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Test No.	Material	Temp, F	Time, hr	Test Condition	Weight Change, mg/(cm ²)(mo)	Appearance and Evaluation
1	304, Malcomized and ground by Chapman Valve Co. Malcomized surface ground off to base metal on one end.	200	190	Air	-2.53	Loose rust and black coating on malcomized surface. Can be rubbed to straw tarnish over about half of area. Poor.
2	304, Malcomized and ground.	500	337	Degas	+0.915	Sample covered with black powdery film over semi-adherent black film which can be rubbed off with rubber eraser. Poor.
3	304, Malcomized by Chapman Valve Co. Ground. Passivated in dichromate.	500	334	Degas	+0.0484	Dull black-brown coating on malcomized surfaces. Unmalcomized face has iridescent tarnish. Good.
4	304, Malcomized and ground at Chapman Valve Co.	500	375	O ₂ -30	-0.410	Loose rust over blue and yellow tarnish. Doubtful.
5	304, Malcomized by Chapman Valve Co. Passivated in dichromate solution before test.	500	4849	O ₂ -30	See Page 79	Dull black brown-gray coating on malcomized surfaces with some loose powder. Unmalcomized face dark straw color. Doubtful.
6	304, Malcomized. Same sample as Test 5. Polished on new alundum paper after above test.	500	334 1116	Degas O ₂ -30	See Page 80	Loose heavy streaks. Powdery black deposit on malcomized surface. Unmalcomized surface faintly lustrous black. Poor.
7	304, Malcomized and ground.	500	340	O ₂ -30	-0.141	Sample covered with powdery black coating. Many rust streaks. Poor.
8	304, Malcomized, ground and polished.	500	340	O ₂ -30	+0.385	Sample has streaky lustrous black appearance. Some gray powder can be rubbed off. Streaky appearance probably due to faulty surface preparation. Poor.
9	304, Malcomized and ground.	600	354	Degas	+0.406	Sample covered with black coating which can be scraped off with steel implement. Rockwell C Hardness - Before: 98; After: 95. Poor.
10	304, Malcomized and ground.	600	337	Degas	+1.27	Sample covered with black powdery film over semi-adherent black film which can be removed with rubber eraser. Poor.
11	304, Malcomized, ground and polished.	600	337	Degas	+0.420	Sample covered with lustrous black tarnish. Good.
12	304, Malcomized, ground and polished.	600	337	Degas	+0.496	Sample covered with faintly lustrous black film under loose black powder. Poor.
13	309, Malcomized and ground by Chapman Valve Co. Malcomized surface ground off to base metal on one end.	200	190	Air-open	-1.18	Loose rust and black coating on malcomized surface. Can be rubbed to straw tarnish over one-half of area. Poor.
14	309, Malcomized.	500	343	Degas	+0.330	Sample covered with black coating which can be removed with rubber eraser. Doubtful.
15	309, Malcomized, ground and polished.	500	343	Degas	+0.137	Sample covered with adherent shiny black tarnish. Good.
16	309, Malcomized.	500	340	O ₂ -30	+0.330	Sample covered with loose powdery black oxide. Poor.
17	309, Malcomized, ground and polished.	500	361	O ₂ -30	-0.110	Loose black powder over lustrous black film. Poor.
18	309, Malcomized and ground by Chapman Valve Co.	500	375	O ₂ -30	-1.05	Loose rust on about 50% of area. Straw tarnish on remainder. Poor.
19	309, Malcomized.	600	343	Degas	+0.498	Loose black powder over adherent shiny black tarnish. Poor.
20	309, Malcomized, ground and polished.	600	343	Degas	+0.159	Sample covered with lustrous black tarnish which can be rubbed off to lustrous gray tarnish. Doubtful.
21	316, Malcomized.	500	343	Degas	+1.48	Sample covered with black film. Very loose in one spot. Poor.

Test No.	Material	Temp, F	Time, hr	Test Condition	Weight Change, mg/(cm ²)(mo)	Appearance and Evaluation
22	316, Malcomized, ground and polished.	500	343	Degas	+0.487	Sample covered with lustrous black tarnish. Uneven because of imperfections in surface preparation. Good.
23	316, Malcomized.	500	361	O ₂ -30	+1.98	Sample covered with dull black film. Some can be rubbed off. Poor.
24	316, Malcomized, ground and polished.	500	361	O ₂ -30	+0.13	Sample covered with faintly lustrous streaky black film. Some can be rubbed off. Poor.
25	316, Malcomized.	600	343	Degas	+0.93	Sample with streaky black film. One face has concentric streaks. Doubtful.
26	316, Malcomized and ground.	600	354	Degas	+0.263	Sample covered with black coating which can be scraped off with steel implement. Rockwell C Hardness - Before: 96; After: 95. Good.
27	316, Malcomized, ground and polished.	600	343	Degas	+0.53	Sample covered with lustrous blue-black and black. Area of loose black film on one face. Rust colored pits on one face. Poor.
28	321, Malcomized and ground by Chapman Valve Co. Malcomized surface ground off to base metal on one end.	200	190	Air-open	-1.80	Loose rust and black coating on malcomized surface. Poor.
29	321, Malcomized and ground by Chapman Valve Co. Malcomized surface ground off to base metal at one end.	200	190	Air-open	-1.80	Loose rust and black coating on malcomized surface. Can be rubbed to a straw colored tarnish over about half of area. Poor.
30	321, Malcomized and ground by Chapman Valve Co.	500	375	O ₂ -30	-0.30	Loose rust over straw colored tarnish. Poor.
31	329, Malcomized and ground.	room	1008	Argon-open	+0.25 +0.15*	Mottled gray-brown after test. Small amount of red rust after test. Good.
32	329, Malcomized and ground.	room	110	Air-open	±0.0 -0.01*	Unattched except for one end which was only slightly rusted. *Corrosion rate calculated after brushing. Good.
33	329, Malcomized and ground.	room	1008	Oxygen-open	+0.19 +0.19*	No attack. Good.
34	329, Malcomized and ground.	200	110	Air-open	+2.9 -1.5*	Approximately half of specimen rusted. Black coat underneath loose rust spotted with salt and pepper type attack. Poor.
35	329, Malcomized by Lindberg Steel Treating Co. Ground.	200	110	Air-open	+1.4 -3.5*	Approximately half of specimen attacked. Salt and pepper attack on black coat underneath loose rust. Poor.
36	347, Malcomized and ground by Chapman Valve Co. Malcomized surface ground off to base metal on one end.	200	190	Air-open	-1.12	Loose rust and black coating over malcomized surface. Poor.
37	347, Malcomized, ground and polished.	500	328	O ₂ -30	+0.648	Sample covered with streaky black and rust colored film. Can be rubbed off. Some uncorroded metal visible on polished faces. Poor.
38	347, Malcomized and ground by Chapman Valve Co.	500	375	O ₂ -30	-0.38	Loose rust over blue and yellow tarnish. Poor.
39	347, Malcomized and ground.	600	354	Degas	+0.347	Sample covered with black coating, which can be rubbed off with steel implement. Good.
40	410, Malcomized and ground by Chapman Valve Co.	200	190	Air-open	-12.1	Loose rust and black powdery coating on all surfaces. Lustrous straw tarnish underneath. Poor.
41	410, Malcomized, ground and polished. (2 samples.)	200	762	Air-open	1) +0.398 2) -0.920	Faintly lustrous light black-brown tarnish with loose streaks black powder and rust deposits. Poor.

*Weight change calculated after brushing.

Test No.	Material	Temp, F	Time, hr	Test Condition	Weight Change, mg/(cm ²)(mo)	Appearance and Evaluation
42	410, Malcomized, ground and polished.	500	9458	O ₂ -30	See Page 81	Faintly lustrous dark gray and black coating after previous loose rust coatings had been removed with emery paper and then stainless wire brushed. Ends are pitting. Doubtful.
43	410, Malcomized, ground and polished.	500	328	O ₂ -30	+0.561	Sample covered with adherent black oxide. Some uncorroded metal visible on polished faces. Poor.
44	410, Malcomized, and ground by Chapman Valve Co.	500	375	O ₂ -30	-1.81	Loose rust over dull blue black coating. Poor.
45	410, Malcomized, ground and polished.	500	328	O ₂ -30	+2.93	Polished faces are lustrous black. Cylindrical surfaces have dull black and rust colored coating; some can be rubbed off. Poor.
46	410, Malcomized, ground and polished.	500	101	O ₂ -1500/2300	+1.37	Sample has dull silver and iridescent tarnish. Stamped numbers not rusty. Good.
47	420, Malcomized, ground and polished.	500	101	O ₂ -1500/2300	+2.23	Sample has streaked blue-gray luster. Stamped numbers rusty. Good.
48	420, Malcomized, ground and polished.	500	101	O ₂ -1500/2300 pH 11	+2.42	Sample covered with lustrous blue-gray tarnish. Good.
49	430, Malcomized and ground by Chapman Valve Co.	200	190	Air-open	-1.12	Loose rust and black coating over Malcomized surface. Poor.
50	430, Malcomized and ground by Chapman Valve Co.	500	375	O ₂ -30	-1.21	Loose rust over blacken surface. Poor.
51	431, Malcomized and ground.	room	1104	Argon-open	+0.06 -3.24*	Specimen was blackened and developed red rust on all but one face. Poor.
52	431, Malcomized and ground.	room	110	Air-open	+0.05 +0.05*	No corrosion attack. Good.
53	431, Malcomized by Lindberg Steel Treating Co. Ground.	room	1008	Oxygen-open	+0.16 +0.16*	Little attack except local pitting. Poor.
54	431, Malcomized and ground.	200	14	Air-open	+2.9 -1.5*	One-half of specimen badly rusted. Poor.
55	431, Malcomized and ground.	200	14	Air-open	+1.4 -3.5*	About one-half of specimen rusted. Poor.
56	440A, Malcomized and ground.	room	1104	Argon-open	-0.93 -4.33*	Blackened, with signs of red rust on all sides but one. Poor.
57	440A, Malcomized and ground.	room	110	Air-open	+1.2 -2.5*	Two edges and 1/3 of one flat face badly rusted. Otherwise specimen unattacked. Poor.
58	440A, Malcomized and ground.	room	1008	Oxygen-open	+0.20 ±0.00*	Slight rust on one side. Good.
59	440A, Malcomized and ground.	200	14	Air-open	+3.2 +3.2*	Approximately 3/4 of specimen rusted. Salt and pepper attack on black undercoat. Poor.
60	440A, Malcomized and ground.	200	14	Air-open	+5.8 -0.6*	Specimen badly corroded. Loose red rust over black undercoat with salt and pepper attack. Poor.
61	440A, Malcomized and ground by Chapman Valve Co.	200	190	Air-open	-11.0	Loose rust and black powdery coating on all surfaces. Lustrous straw tarnish underneath. Poor.
62	440A, Malcomized and ground by Chapman Valve Co.	500	375	Air-open	-0.40	Loose rust over faintly lustrous blue and straw-colored tarnish. Poor.
63	440C, Malcomized, ground and polished.	500	328	O ₂ -30	+4.67	Polished faces have dull, adherent black and gray coating. Some black powder can be rubbed off cylindrical surface. Poor.

*Weight change calculated after brushing.

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Test No.	Material	Temp, F	Time, hr	Test Condition	Weight Change, mg/(cm ²)(mo)	Appearance and Evaluation
64	440C, Malcomized, ground and polished.	500	5188	O ₂ -30	See Page 82	Streaked powdery black and rust colored coating. Pitting. Loose rust on unpolished surface. Poor.
65	440C, Malcomized, ground and polished.	500	280	O ₂ -2300/3300	+0.027	Polished faces have iridescent tarnish (includes some brownish streaks). Un-ground edges have rust deposits. Some pitting in polished faces. Doubtful.
66	440C, Malcomized, ground and polished.	500	101	O ₂ -1500/2300-pH 11	+1.10	Sample covered with dull blue-gray tarnish. Stamped numbers rusty. Good.
67	446, Malcomized and ground.	room	1104	Argon-open	+0.80 -2.55*	Very badly rusted on all surfaces. Thick loose red rust over heavy black layer. Poor.
68	446, Malcomized and ground.	room	110	Air-open	+0.86 -6.2 *	Two edges and 1/2 of one face badly rusted. Remainder unattacked. Poor.
69	446, Malcomized and ground.	room	1008	Oxygen-open	+0.76 +0.28*	Some corrosion on one face. Doubtful.
70	446, Malcomized and ground.	200	14	Air-open	-4.0 -10.6*	Completely and heavily rusted over thick loose black coat. Poor.
71	446, Malcomized and ground.	200	14	Air-open	-2.7 -12.1*	Same as above.
72	446, Malcomized and ground by Chapman Valve Co.	200	190	Air-open	-9.55	Rusted heavily over a thick loose black coat. Poor.
73	446, Malcomized and ground by Chapman Valve Co.	500	375	O ₂ -30	-0.40	Loose rust over rough dull blue coating and yellow tarnish. Poor.
74	17-4 PH, Malcomized, not ground	room	336	Argon-open	+0.43	Specimen entirely blackened and starting to form red rust. Poor.
75	17-4 PH, Malcomized, not ground	room	336	Argon-open	+0.33	Specimen all black. Some patches of red rust overlay forming. Poor.
76	17-4 PH, Malcomized, not ground	room	314	Air-open	+0.50	About 1/2 of specimen blackened. Remainder unattacked. Doubtful.
77	17-4 PH, Malcomized, not ground	room	314	Air-open	+0.30	Slightly more than 1/2 of specimen blackened. Remainder unattacked. Doubtful.
78	17-4 PH, Malcomized, not ground	room	216	Air-open	+0.20	Same appearance as preceding sample. Doubtful.
79	17-4 PH, Malcomized, not ground	room	216	Air-open	+0.65	Specimen only slightly attacked. Doubtful.
80	17-4 PH, Malcomized, not ground	room	314	Oxygen-open	+0.20	Specimen very slightly attacked. Black ring where held by quartz fiber thread. Good.
81	17-4 PH, Malcomized, not ground	room	314	Oxygen-open	+0.55	Specimen very slightly attacked. Black ring where held by quartz fiber thread, and one corner starting to turn black. Good.
82	17-4 PH, Malcomized and ground by Chapman Valve Co.	200	190	Air-open	-8.82	Loose rust and black powdery coating on all surfaces. Lustrous straw tarnish underneath. Poor.
83	17-4 PH, Malcomized and ground by Chapman Valve Co.	500	375	O ₂ -30	-0.64	Heavy loose rust deposit over blue tarnish. Poor.
84	17-4 PH, Malcomized and ground.	500	9976	O ₂ -30	See Page 83	Dull gray-black coating. Some rust on sawed unmalcomized surface, and on one corner. Good.
85	17-4 PH, Malcomized and ground.	500	101	O ₂ -1500/2300	+2.36	Lustrous silver-blue tarnish on Malcomized faces. Unmalcomized face dull black-brown. Good.
86	17-4 PH, Malcomized, ground and polished.	500	101	O ₂ -1500/2300	+5.48	Sample has dull adherent blue-gray coating. No difference between Malcomized and unmalcomized surfaces. Good.

*Weight change calculated after brushing.

Test No.	Material	Temp, F	Time, hr	Test Condition	Weight Change, mg/(cm ²)(mo)	Appearance and Evaluation
87	17-7 PH, Malcomized and ground.	room	1104	Argon-open	+0.24 -0.41*	Badly rusted. Surface coat black under loose outer rust. Poor.
88	17-7 PH, Malcomized and ground.	room	110	Air-open	+1.5 -1.6*	Unattacked except for one face. This face badly rusted. Doubtful.
89	17-7 PH, Malcomized and ground.	room	1008	Oxygen-open	+0.17 +0.17*	Unattacked. Good.
90	17-7 PH, Malcomized and ground.	room	14	Air-open	+5.0 -6.5*	Covered with loose rust. Black layer underneath pitted. Two areas unattacked. Poor.
91	17-7 PH, Malcomized and ground.	room	14	Air-open	+12.2 ±0.0*	Completely covered with loose red rust. Poor.
92	17-7 PH, Malcomized and ground. by Chapman Valve Co.	200	190	Air-open	-2.07	Loose rust and black powdery coating on all surfaces. Lustrous straw tarnish underneath. Poor.
93	17-7 PH, Malcomized by Chapman Valve Co. (2 samples).	500	358	O ₂ -30	1) +0.312 2) +0.182	Samples have faintly lustrous adherent black coating. Area of contact between samples visible but no marked or excessive galvanic action. Good.
94	17-7 PH, Malcomized by Chapman Valve Co. 0.002 in. ground off surface for 1/2 length. (3 samples).	500	358	O ₂ -30	1) -0.113 2) -0.158 3) -0.069	Samples previously tested for 264 hr at 600F in degassed, distilled water. Covered with adherent black tarnish. Slightly more lustrous on ground area. This was the same appearance as after the 600F test. Only treatment between tests was washing with distilled water. Good.
95	17-7 PH, Malcomized by Chapman Valve Co. 0.002 in. ground off surface for 1/2 length. Treated with 0.2 N potassium dichromate for 1 hr before test.	500	358	O ₂ -30	±0.00	Sample previously tested for 264 hr at 600F in degassed, distilled water. Covered with adherent black tarnish. Slightly more lustrous on ground area. This was the same appearance as after the 600F test. Only treatment between tests was washing with distilled water. Good.
96	17-7 PH, Malcomized and ground by Chapman Valve Co.	500	375	O ₂ -30	-0.23	Loose rust coating over blue and yellow tarnish. Doubtful.
97	17-7 PH, Malcomized by Chapman Valve Co. Ground down to base metal for 1/2 of length.	500	5557	O ₂ -30	See Page 84	Unground Malcomized surface has dull adherent black coating. Base metal has lustrous dark straw colored tarnish. Note: Sample had previously been tested for 264 hr at 600F in degassed, distilled water. Washed in distilled water before this part of test. Good.
98	17-7 PH, Malcomized by Chapman Valve Co. One sample cut in half exposing base metal.	500	5557	O ₂ -30	See Page 85	Malcomized surfaces have dull black coating with some powder. Base metal has lustrous violet tarnish. Good.
99	17-7 PH, Malcomized by Chapman Valve Co. 0.002 in. ground off for 1/2 length.	500	5557	O ₂ -30	See Page 86	Dull blue-gray tarnish with slight amount of loose powder. Good.
100	USS Stainless W, Malcomized, not ground.	room	336	Argon-open	+0.37	Specimen heavily blackened with red rust starting to form. Poor.
101	USS Stainless W, Malcomized, not ground.	room	148	Argon-open	+0.62	Specimen heavily blackened with red rust starting to form. Poor.
102	USS Stainless W, Malcomized, and ground.	room	1104	Argon-open	+0.20 +0.20*	Specimen metallic mottled bluish color after test. No badly rusted areas. Good.
103	USS Stainless W, Malcomized, not ground.	room	120	Air-open	+0.35	Specimen darkened over about one-half of area. Also blackened at line of contact with quartz fiber thread. Doubtful.

*Weight change calculated after brushing.

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Test No.	Material	Temp, F	Time, hr	Test Condition	Weight Change, mg/(cm ²)(mo)	Appearance and evaluation
104	USS Stainless W, Malcomized, not ground.	room	314	Air-open	+0.35	Specimen darkened over about one-half of its area. Also blackened at line of contact with quartz fiber thread. Doubtful.
105	USS Stainless W, Malcomized, not ground.	room	216	Air-open	+0.60	Same as above.
106	USS Stainless W, Malcomized, not ground.	room	216	Air-open	+0.79	Same as above.
107	USS Stainless W, Malcomized and ground. (2 samples.)	room	110	Air-open	+0.37 +0.37*	No attack. Good.
108	USS Stainless W, Malcomized and ground. (2 samples.)	room	1008	Oxygen-open	+0.09 +0.09*	No attack. Good.
109	USS Stainless W, Malcomized, not ground.	room	314	Oxygen-open	+0.10	No attack except where contacted with quartz fiber thread. Good.
110	USS Stainless W, Malcomized, not ground.	room	120	Oxygen-open	+0.60	No attack except where contacted with quartz fiber thread. Good.
111	USS Stainless W, Malcomized and ground. (2 samples.)	200	14	Air-open	+1.5 -3.7*	Uniformly rusted and blackened. Salt and pepper type attack under loose red rust on blackened surface. Poor.
112	USS Stainless W, Malcomized and ground. (2 samples.)	200	14	Air-open	+2.2 -0.7*	Uniformly rusted and blackened. Salt and pepper type attack under loose red rust on blackened surface. Poor.
113	USS Stainless W, Malcomized and ground by Chapman Valve Co.	200	190	Air-open	-2.24	Loose rust and powdery black coating over silver-yellow tarnish. Poor.
114	USS Stainless W, Malcomized and ground by Chapman Valve Co.	200	263	Air-open	+0.08	No change except for one large rust spot on polished face and edge. Doubtful.
115	USS Stainless W, Malcomized and ground by Chapman Valve Co. One face and end polished at Argonne.	250	6129	Degas	See Page 87	Rust at one corner of Malcomized surface. Gray area at one edge. Good.
116	USS Stainless W, Heat #4x 7854. Malcomized and ground by Chapman Valve Co. One face and edge ground at Argonne.	250	353	Degas	+0.13	Black coating on all surfaces. Polished surfaces still have some luster. Good.
117	USS Stainless W, Heat #4x 7854, Malcomized and ground by Chapman Valve Co. One face and edge ground at Argonne. Coupled to Graphitar.	250	353	Degas	USS W)+0.27 Graph)+12.2	No change in appearance of Graphitar. USS W had black coating on all surfaces. Polished surface still has some luster. Streaky appearance. Good.
118	USS Stainless W, Heat #4x 7854, Malcomized and ground by Chapman Valve Co. One face and edge ground at Argonne.	250	401	Air	+0.18	Black coating on all surfaces. Polished surfaces still have some luster. Doubtful.
119	USS Stainless W, Heat #4x 7854. Malcomized and ground by Chapman Valve Co. One face and edge ground at Argonne. Coupled to Graphitar.	250	353	Air	USS W)-3.41 Graph)+14.4	No change in appearance of Graphitar. USS W has black coating with rust around contact area. Poor.
120	USS Stainless W, Heat #9x 7854. Malcomized and ground by Chapman Valve Co. One face polished by Argonne and this face coupled to Graphitar 14.	500	3262	Degas	See Page 88	Comments on referenced page. Good
121	USS Stainless W, Heat #9x 7854. Malcomized and ground by Chapman Valve Co. One face polished by Argonne and this face coupled to Graphitar 14.	500	3262	Air	See Page 89	Comments on referenced page. Good

*Weight change calculated after brushing.

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Test No.	Material	Temp, F	Time, hr	Test Condition	Weight Change, mg/(cm ²)(mo)	Appearance and Evaluation
122	USS Stainless W, Malcomized and ground by Chapman Valve Co.	500	375	O ₂ -30	-0.06	Light rust coating over blue-yellow tarnish. Good.
123	USS Stainless W, Malcomized and ground by Chapman Valve Co.	500	3269	O ₂ -30	See Page 90	Adherent black coating which is slightly lustrous on ground face. Some rusting near broken edge. Good.
125	USS Stainless W, Malcomized and ground.	500	332	O ₂ -30	+0.108	Sample covered with adherent black coating. Slightly lustrous on ground faces. Some rust streaks on rough unground unmalcomized areas. Good.
126	USS Stainless W, Malcomized. Half of cylindrical surface ground 0.003 in. and other half 0.006 in.	500	376	O ₂ -30	-7.24	Sample covered with loose black powder. Small areas of lustrous tarnish visible. Photomicrograph reveals that case was defective for entire depth. Poor.
127	USS Stainless W, age hardened, ground, washed, nitrided at Argonne for 72 hr at 953/1039F (18% dissociation) and centerless ground to remove 0.002 in.	500	314	O ₂ -30	-1.87	Loose black and rust colored coating over silvery tarnish.
128	USS Stainless W, age hardened, ground, washed, nitrided at Argonne for 72 hr at 953/1039F (18% dissociation) and centerless ground to remove 0.002 in.	500	314	O ₂ -30	+0.94	Loose rust over dull black coating.
129	USS Stainless W, age hardened, ground, washed, nitrided at Argonne for 72 hr at 953/1039F (18% dissociation) and centerless ground to remove 0.002 in. Sample not touched by hand during pre-nitriding cleaning.	500	314	O ₂ -30	+1.17	Loose rust over dull black coating.
130	USS Stainless W, age hardened, ground, washed, nitrided at Argonne for 72 hr at 953/1039F (18% dissociation) and centerless ground to remove 0.002 in.	500	314	O ₂ -30	-3.66	Loose rust over dull black coating. Some luster remains.
131	USS Stainless W, Malcomized, ground and polished.	500	353	O ₂ -1500-pH 9	+0.266	Sample covered with faintly lustrous black film. Many rust streaks on one face, possibly from drilled hole which was not polished. Good.
132	USS Stainless W, Malcomized, ground and polished.	500	353	O ₂ -1500-pH 10	+0.244	Sample has dull black coating except where in contact with autoclave. Lustrous blue-violet black on this surface. Good.
133	USS Stainless W, Malcomized, ground and polished.	500	353	O ₂ -1500-pH 11	+0.155	Sample has lustrous black tarnish on polished faces. Slight trace of rust on unpolished faces. Good.
134	USS Stainless W, Malcomized, ground and polished.	500	280	O ₂ -2300/3300	+0.463	Polished Malcomized faces have adherent faintly lustrous black tarnish. Cut faces (heat treated only) have adherent dull black coating. Good.
135	USS Stainless W, Malcomized and ground.	500	101	O ₂ -2500/3300	+1.68	Sample has dull adherent blue-gray coating. No difference between malcomized and unmalcomized surfaces. Good.
136	Haynes Alloy 25, sand blasted and Malcomized. No significant hardness change.	500	327	O ₂ -30	-0.278	Loose powdery black coating. Poor.
137	Haynes Alloy 25, sand blasted and Malcomized. No significant hardness change.	500	327	O ₂ -30	-0.52	Loose powdery black coating. Poor.
138	Haynes Alloy 25, Degreased before Malcomizing. No significant hardness change.	500	327	O ₂ -30	+0.088	Silvery tarnish with streaked loose black powder.
139	Haynes Alloy 25, Degreased before Malcomizing. No significant hardness change.	500	327	O ₂ -30	-0.113	Silvery blue tarnish with black pits. Much loose powder on each. Less on cylindrical surface. Poor.
258	17-7 PH, water quenched from 1950 F, rough machined and stress relieved at 1300-1450F by Chapman Valve Co. Rough, pickled finish.	500	358	O ₂ -30	-0.715	Faint lustrous dark brown tarnish. Some loose powder. Evidence of contact corrosion. Initial pH and resist: 6.9 and 132,000; Final pH and resist: 6.9 and 48,000. Poor.
261	17-7 PH, water quenched from 1950F by Chapman Valve Co. (2 samples.)	500	358	O ₂ -30	1) -0.752 2) -0.815	Faint lustrous dark brown tarnish. Heavy powdery coating. Evidence of contact corrosion. Initial pH and resist: 7.1 and 73,000; Final pH and resist: 6.2 and 37,000. Poor.

56

Material: Type 304, Malcomized.

Malcomized by Chapman Valve Co.
 Passivated in 0.02 N $\text{Na}_2\text{Cr}_2\text{O}_7$ and
 tested in degassed, distilled water
 for 196 hours at 450F.

Temperature: 500F

Test Condition: O_2 -30

Period	Time, hr	Weight Change, $\text{mg}/(\text{cm}^2)(\text{mo})$	Comments
1	334	-0.2440	Dull black-brown-gray coating on Malcomized surfaces. Unmalcomized face has iridescent tarnish.
2	738	-0.0420	No change except unmalcomized surface has faintly lustrous dark straw color.
3	785	+0.0641	No change except for small amount of loose powder on malcomized surface.
4	714	-0.00542	Moderately heavy deposit of brown powder on Malcomized surface. Cleaned and test continued.
5	761	-0.061	Malcomized surface dull brown. Unmalcomized surface faintly lustrous dark straw and violet tarnish. Some loose powder. Malcomized surfaces have least amount of loose powder. Starting with next period, fresh water used each period.
6	732	-0.0211	No change. Initial pH and resist: 7.0 and 145,000. Final pH and resist: 7.3 and 50,000.
7	785	+0.0407	No change. Initial pH and resist: 7.0 and 190,000. Final pH and resist: 6.8 and 32,000.
4849			

Material: Type 304, Malcomized.

Malcomized by Chapman Valve Co.
 Passivated in 0.02 N $\text{Na}_2\text{Cr}_2\text{O}_7$.

Temperature: 500F

Test Condition: Degas O_2 -30

Period	Time, hr	Weight Change, $\text{mg}/(\text{cm}^2)(\text{mo})$	Comments
1	334	+0.0484	Dull black-brown coating on Malcomized surfaces. Unmalcomized surface has iridescent tarnish. Degassed, distilled water.
2	354	-0.117	Oxide film removed from Malcomized surface with clean metallographic (alundum) paper. Starting with this period, 30 cc/liter oxygen added to water at start of each period. Autoclave upside down. Malcomized surface has lustrous gray coating. Small amount of brown powder - probably from cylindrical edge. Edges stainless steel wire brushed and test continued.
3	762	-0.412	Malcomized surfaces have loose heavy streaky black powdery deposit. Unmalcomized surface has faintly lustrous black tarnish.
1450			

57

Material: Type 410, Malcomized.

Flat surfaces ground and polished.

Hardness: R_{15N}^{90} .

Temperature: 500F

Test Conditions: O_2 -30

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	328	+0.561	Sample covered with adherent black oxide. Some uncorroded metal visible on polished faces.
2	860	-0.0752	Rust colored streaks on polished faces and cylindrical surface.
3	400	-0.300	Loose coating removed with emery paper before this period. Starting with this period autoclave placed in furnace upside down. Dull black coating on cylindrical surface after test. Rust streaks on ground faces.
4	687	-0.444	Loose rust on ground faces has increased.
5	591	-0.550	Same appearance as above.
6	713	-0.026	Same appearance as above. Loose rust brushed off with stainless steel wire brush and test continued. Hardness: R_{15N}^{93} .
7	712	+0.323	Cylindrical surfaces have dark gray coating. Polished ends black with some rust pits.
8	713	+0.0388	Sample covered with faintly lustrous black coating on polished surfaces. Some loose rust over black coating on cylindrical surface. Autoclave dry at end of this period.
9	693	-0.01334	Faintly lustrous dark gray coating on cylindrical surface. Ends streaky black with few traces of rust.
10	742	-0.075	No change in appearance. Hardness: Ends - R_{15N}^{92} ; Cyl. Surface - R_{15N}^{90} .
11	790	+0.023	No change in appearance.
12	714	+0.0904	No change except for small rust and black streaks on end.
13	760	-0.0486	No change. Hardness.
14	755	+0.085	Dull, dark gray coating. Ends are pitting.
	9458		

58

Material: Type 440C, Malcomized.Flat faces ground and polished.
Hardness: $R_{15N}84$. Malcomized by
Lindberg Steel Treating Co.

Temperature: 500F

Test Condition: O_2 -30

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	328	+4.67	Polished faces have dull, adherent black and gray coating. Some black powder can be rubbed off cylindrical surfaces.
2	860	-0.0643	Autoclave dry at end of this period. Rust coating on cylindrical surface and flat faces.
3	400	+0.324	Rust coating removed with emery paper before start of this period. Autoclave placed upside down starting with this period. Cylindrical surface and one flat face covered with rust over dull black luster. Other face has dull black luster.
4	867	-0.167	No change in appearance.
5	594	-0.203	No change in appearance.
6	713	+0.064	No change in appearance. Case Hardness: $R_{15N}87$.
7	713	+0.310	No change in appearance except rust deposit is heavier.
8	713	-2.24	Polished faces covered with streaked powdery black coating. Some rust and pitting. Loose rust on unpolished cylindrical surface. Test discontinued.

5188

59

Material: Armco 17-4 PH, Malcomized. Two faces ground. Malcomized by Lindberg Steel Treating Co. Ground.

Temperature: 500F

Test Condition: O₂-30

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	332	+0.790	Ground surfaces have faintly lustrous black coating, sawed surfaces have dull black coating. Some rust streaks on sawed surface. (Unmalcomized.)
2	738	+0.182	No change except luster has decreased.
3	763	+0.024	No change in appearance.
4	691	+0.031	No change.
5	714	+0.0171	No change.
6	736	-0.0410	No change.
7	810	-0.0753	No change except for few rust spots on Malcomized surface near edge.
8	749	-0.065	No change. Hardness: R _{15N} ⁹² .
9	712	-0.051	No change in appearance.
10	738	+0.116	No change in appearance.
11	734	-0.041	No change except for very slight amount loose powder. Hardness: R _{15N} ⁹² . Sample cleaned with bristle brush (0.5 mg removed). Starting with next period, fresh water used each test period. Final pH and resist: 6.4 and 22,000.
12	766	-0.079	Dull gray coating with small amount loose powder. Some rust, particularly heavy on one corner of cut surface. Initial pH and resist: 6.8 and 60,000. Final pH and resist: 6.3 and 33,000.
13	708	-0.19	No change. Initial pH and resist: 7.0 and 250,000. Final pH and resist: 6.2 and 36,000.
14	785	-0.530	No change in appearance. No loose powder on Malcomized surfaces except at large corner rust spot. Stainless steel gasket used this period. Initial pH and resist: 6.8 and 155,000. Final pH and resist: 6.4 and 30,000. Test concluded.

60

Material: Armco 17-7 PH, Malcomized.Malcomized by Chapman Valve Co.
Ground down to bare metal for
1/2 length.

Temperature: 500F

Test Condition: O₂-30

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	358	-0.047	Unground Malcomized surface has dull adherent black coating. Base metal has lustrous dark straw colored tarnish. Sample previously tested 264 hr at 600F in degassed water.
2	716	±0.00	Malcomized surface dull brown. Base metal dull black.
3	738	+0.056	No change in appearance.
4	762	±0.00	No change in appearance.
5	712	-0.048	No change.
6	781	-0.032	Dull black coating with small amount of loose black powder (more on base metal). Rust spots (where in contact with autoclave) at both ends. Starting with next period, fresh water used each period.
7	708	+0.083	No change in appearance except rust spot on Malcomized surface has disappeared. Initial pH and resist: 6.9 and 130,000. Final pH and resist: 7.1 and 32,000.
8	782	+0.086	No change. Initial pH and resist: 7.0 and 170,000. Final pH and resist: 7.5 and 28,000.
5557			

61

Material: Armco 17-7 PH, Malcomized.

Malcomized by Chapman Valve Co. One sample (3) cut in half, exposing base metal.

Temperature: 500F

Test Condition: O₂-30

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	358	1) -0.136 2) -0.119 3) -0.156	Malcomized surfaces have dull black adherent coating. Base metal lustrous violet.
2	716	1) -0.128 2) -0.191 3) -0.142	No change except some rust at edges of cut edges of cut surface. Removed before next period.
3	738	1) +0.121 2) +0.162 3) +0.060	Same as above except for slight rust streak on side of Sample No. 1. No water.
4	762	1) -0.021 2) ±0.00 3) ±0.00	Dull black-brown coating with rust streak on side of Sample No. 1.
5	712	1) -0.023 2) ±0.00 3) ±0.00	No change.
6	781	1) -0.010 2) -0.022 3) -0.072	Dull black coating. Very slight amount loose black powder. Starting with next period, fresh water used each period.
7	708	1) -0.046 2) +0.061 3) +0.032	No change. Initial pH and resist: 7.1 and 120,000. Final pH and resist: 7.2 and 30,000.
8	782	1) +0.094 2) -0.033 3) -0.043	No change. Initial pH and resist: 7.2 and 145,000. Final pH and resist: 7.3 and 37,000.

5557

62

Material: Armco 17-7 PH, Malcomized. Malcomized by Chapman Valve Co.
0.002 in. ground off surface
for 1/2 length.

Temperature: 500F

Test Condition: O₂-30

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	358	1) -0.113 2) -0.158 3) -0.069	Sample covered with adherent black tarnish. Slightly more lustrous on ground area. Sample previously tested 264 hr in 600F de- gassed water.
2	716	1) -0.022 3) -0.034	No change. Sample No. 2 removed.
3	738	1) +0.021 3) ±0.00	No change in appearance.
4	762	1) ±0.00 3) +0.021	No change in appearance.
5	712	1) -0.034 3) -0.023	Dull blue-gray tarnish. One rust streak on cylindrical surface of Sample No. 3.
6	781	1) +0.062	No change in appearance. Very slight amount loose powder. Starting with next period, fresh water used each period.
7	708	1) ±0.00 3) +0.023	No change. Initial pH and resist: 7.3 and 150,000. Final pH and resist: 7.2 and 40,000.
8	782	1) -0.050 3) -0.052	No change. Initial pH and resist: 7.1 and 160,000. Final pH and resist: 7.3 and 32,000.
5557			

63

Material: USS Stainless W (Heat #4x7854). Malcomized and ground at Chapman Valve Co. One face and edge polished at Argonne.

Temperature: 500F

Test Condition: Degas

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	353	+0.176	Black coating on all surfaces. Polished surface lustrous.
2	737	+0.0682	Dull brown coating. Some loose powder on all surfaces.
3	1240	-0.0361	No change.
4	834	-0.040	No change. Polished surface still lustrous but spotted.
5	712	+0.023	No change. Starting with next period, fresh water will be used each period.
6	782	+0.057	No change. Autoclave dry at end of this period. Initial pH and resist: 7.0 and 95,000.
7	709	±0.00	No change Initial pH and resist: 7.4 and 135,000. Final pH and resist: 6.7 and 35,000.
8	762	-0.014	Rust at one corner of Malcomized surface. Gray area at one edge of Malcomized surface. Initial pH and resist: 7.2 and 110,000. Final pH and resist: 7.2 and 110,000. Final pH and resist: 6.8 and 30,000.
	6129		

Material: USS Heat #9x7854, Malcomized. Malcomized and ground at Chapman Valve Co. One face polished at Argonne, coupled to Graphitar 14. (Polished/face is contact area.)
USS Stainless W

Temperature: 500F

Test Condition: Degas

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	401	SS W +0.139 G 14 -3.02	Dull black coating. Same appearance except some of sample washed away.
2	401	SS W +0.138 G 14 -6.20	Dull black coating on all surfaces. Same appearance except some of sample washed away.
3	357	SS W -0.312 G 14 -5.02	Dull brown and black coating on all surfaces. Dull black appearance with brown around contact area.
4	736	SS W -0.682 G 14 -0.254	Same as above with dark black coating at contact area. Black coating with reddish-brown coating on edges.
5	1367	SS W -0.0204	USS Stainless W has dull black adherent coating on all but contact face. Contact area has faint lustrous black coating. Area surrounding this loose reddish brown deposit. Graphitar 14 has loose black deposit with rust colored ring around edge at non-contact face. Test discontinued.
	3262		

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Material: USS Stainless W, Heat #9x7854, Malcomized. Malcomized and ground at Chapman Valve Co. One face polished at Argonne coupled to Graphitar 14.

Temperature: 500F

Test Condition: Air

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	401	SS W -0.149 G 14 -1.74	Black-brown coating. Same appearance; some of sample washed away.
2	401	SS W -0.014 G 14 -6.450	Loose brown, black coating on all surfaces. Same appearance; some of sample washed away.
3	357	SS W -3.08 G 14 +0.201	Brown and black coating on all surfaces, except contact area which has a loose brown coating around contact area. Dull black appearance with loose brown coating around contact area.
4	736	SS W -0.113 G 14	No change in appearance except that brown coating has extended to edges.
5	1367	SS W -0.0286	USS Stainless W has dull black and brown coating with small amount loose black powder on all surfaces except contact face. This is covered with heavy deposit of loose black powder with deposit of loose rust at periphery of contact area. Some luster remaining at contact area. Graphitar has loose black powdery coat. Some brown powder on contact face. Test discontinued.
	3262		

Material: USS Stainless W, Malcomized. Malcomized and ground by Chapman Valve Co.

Temperature: 500F

Test Condition: O₂-30

Period	Time, hr	Weight Change, mg/(cm ²)(mo)	Comments
1	332	+0.108	Adherent black coating which is slightly lustrous on ground faces. Rust streak on unground area.
2	380	-0.0154	Sample cut in half (only one ground face remains) and drillings taken from unmalcomized face before this period. Lustrous black tarnish.
3	354	-0.033	No change except some rust broken edge.
4	732	-0.008	No change. Starting with next period, fresh water used each period.
5	708	+0.016	No change. Initial pH and resist: 7.3 and 105,000. Final pH and resist: 6.7 and 20,000.
6	763	±0.00	No change. Autoclave dry at end of this period. Initial pH and resist: 7.3 and 155,000.
	3269		

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APPENDIX B

COUPLE CORROSION TESTS AT BATTELLE

The nitriding operation was performed at Battelle using a laboratory scale conventional cracked ammonia apparatus. This process consisted of subjecting pickled samples to the action of ammonia gas at 1000F for 50 hr in a tube furnace fitted with a pyrex tube. The samples were supported in the tube on alundum boats. Final traces of water in the commercial anhydrous ammonia were removed using calcium sulfate as a desiccant. Ammonia was introduced at a constant rate of 100 cc/min during the 50-hr nitriding period and was continued until the furnace had cooled to room temperature.

One sample from each run was sectioned and its case examined under the microscope. The average case hardness was 999 Knoop, while the cases ranged near 411 Knoop for Armco 17-4 PH and 450 Knoop for USS Stainless W. Typical microstructures for both USS Stainless W and Armco 17-4 PH are shown in Figure 16.

The corrosion studies are described in detail in BMI-742; pertinent data are quoted in part.

"Samples of nitrided 17-4 PH and nitrided 322W in the as-nitrided and in the ground condition (0.002 to 0.004 in. removed from nitrided case by surface grinding) were coupled with the following materials; (1) Stellite 3, (2) Stellite 6, (3) Stellite 21, (4) graphite, (5) Graphitar 14, (6) Type 410 stainless, and (7) Haynes 25. Chromium-plated 17-4 PH coupled to Haynes 25 and a sample of each of the materials uncoupled were also exposed under each test condition.

"The couples were formed by placing several specimens representing various material combinations between 1-1/2 inch-diameter stainless steel washers which were fastened together with a stainless bolt and nut. The couples were insulated from the stainless washer by stove mica. Care was exercised so that the couples did not touch each other or the stainless bolt. Good contact between materials was insured by tightening the fixture with a wrench. Prior to coupling, each material was carefully degreased in acetone, dried, and weighed.

"Static corrosion tests were conducted at room temperature, 200F and 500F in degassed and oxygenated (30 ml dissolved oxygen per liter of water) distilled water. The specific resistance and pH of the water was obtained at the beginning and end of each exposure period. After each two-week exposure period, fresh water was added to the test cells.

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ARMCO 17-4 PH

S.S. USS STAINLESS W

FIGURE 16
TYPICAL MICROSTRUCTURES OF ARMCO 17-4 PH AND
USS STAINLESS W

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 "The oxygenated-water, room temperature, corrosion tests were conducted in covered one-liter electrolytic beakers containing approximately 500 ml of water. To insure that the water was saturated with oxygen (30 ml per liter), continuous stream was bubbled through each test cell. Fritted glass discs were used to disperse the oxygen in the water.

"The test vessel employed in the degassed-water studies consisted of a large vacuum desiccator fitted with six 600-ml beakers. Water for this test was degassed by boiling for two hours."

The results of the corrosion tests are given in the following tabulation.

CORROSION OF NITRIDED STAINLESS STEELS COUPLED WITH
VARIOUS MATERIALS

(N) = Nitrided; (G) = Ground; (CP) = Chrome Plated

<u>Couple No.</u>	<u>Coupled Materials</u>	<u>Time, Days</u>	<u>Appearance After Test</u>
<u>Room Temperature - Oxygenated Water</u>			
1	USS Stainless W (N)	40	Rust on surface; stained at contact area.
	Stellite 3		Rust build up at periphery of contact area.
2	USS Stainless W (N)	40	Rust on surface; stained at contact area
	Stellite 6		Rust build up at periphery of contact area
3	USS Stainless W (N)	40	Rust on surface; stained at contact area.
	Stellite 21		Rust build up at periphery of contact area

Couple No.	Coupled Materials	Time, Days	Appearance After Test
4	USS Stainless W (N)	40	Rust on surface; contact area bright.
	Graphite		No apparent change
5	USS Stainless W (N)	40	Rust on surface; moderate build up at periphery of contact area.
	Type 410		Slight build up; mostly stain.
7	USS Stainless W (N)	40	Rust on surface; some rust in contact area.
	USS Stainless W (N)		Rust on surface; some rust in contact area.
8	USS Stainless W (N)	40	Rust on surface and contact area; build up at periphery of contact.
	Haynes Alloy 25		Staining in contact area
9	Armco 17-4 PH (N)	40	Slight rusting of surface; contact area, light stain.
	Stellite 3		Light stain in contact area
10	Armco 17-4 PH (N)	40	Rust on surface; build up at periphery of contact area.
	Stellite 6		Slight build up at periphery of contact area
11	Armco 17-4 PH (N)	40	Rust on surface; build up at periphery of contact area.
	Stellite 21		Moderate build up at periphery of contact area
12	Armco 17-4 PH (N)	40	Some rust on surface; contact area bright.
	Graphite		No apparent change

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
13	Armco 17-4 PH (N)	40	Rust on surface and at contact area build up.
	Graphitar 14		Some build up at periphery of contact area.
14	Armco 17-4 PH (N)	40	Rust on surface; slight build up at periphery of contact area.
	Type 410		Stained on surface; some pitting; stain at contact area.
15	Armco 17-4 PH (N)	40	Rust on surface; rust in contact area; slight build up.
	Armco 17-4 PH (N)		Rust on surface; rust in contact area; slight build up.
16	Armco 17-4 PH (N)	40	Rust on surface; build up at periphery of contact area.
	Haynes Alloy 25		Slight build up; mostly stain at periphery of contact area.
17	Armco 17-4 PH (CP)	40	No apparent change
	Haynes Alloy 25		No apparent change
103	USS Stainless W (N & G)	12	Stain on surface; contact area bright.
	Graphite		No apparent change
104	USS Stainless W (N)	12	Stain on surface; stain at contact area.
	Graphitar 14		Stain at contact area
105	USS Stainless W (N)	12	Stain on surface; contact area bright.
	Type 410		Stain and some pitting on surface; contact area bright.

Couple No.	Coupled Materials	⁷⁰ Time, Days	Appearance After Test
106	USS Stainless W (N)	12	Stain on surface; stain and slight build up in contact area.
	Type 410		Stain and some pitting on surface; contact area bright.
107	Armco 17-4 PH (N & G)	12	Stain on surface; stain and slight build up at contact area.
	Stellite 3		Slight build up at periphery of contact area
108	Armco 17-4 PH (N & G)	12	Stain on surface; slight staining of contact area.
	Stellite 6		Slight staining of contact area
109	Armco 17-4 PH (N & G)	12	Stain on surface; moderate build up at periphery of contact area.
	Stellite 21		Moderate build up at periphery of contact area
110	Armco 17-4 PH (N & G)	12	Stain on surface; patch of rust in contact area.
	Graphite		No apparent change
111	Armco 17-4 PH (N & G)	12	Stain on surface; rusting and build up in contact area.
	Graphitar 14		Build up at periphery of contact area
112	Armco 17-4 PH (N & G)	12	Stain on surface; contact area bright.
	Type 410		No apparent change

Couple No.	Coupled Materials	Time, Days	Appearance After Test
113	Armco 17-4 PH (N & G)	12	Stain on surface; light build up in contact area.
	Armco 17-4 PH (N & G)		Stain on surface; light build up in contact area.
<u>Room Temperature - Degassed Water</u>			
18	USS Stainless W (N)	40	Rust on surface; rust and build up in contact area.
	Stellite 3		Rust and build up in contact area
19	USS Stainless W (N)	40	Rust on surface; rust and build up in contact area.
	Stellite 6		Rust and build up in contact area
20	USS Stainless W (N)	40	Rust on surface; rust and build up in contact area.
	Stellite 21		Rust and build up in contact area
21	USS Stainless W (N)	40	Rust on surface; light build up at periphery of contact area.
	Graphite		Stain in contact area
22	USS Stainless W (N)	40	Rust on surface; light build up at periphery of contact area.
	Graphitar 14		Stain in contact area
23	USS Stainless W (N)	40	Rust on surface; rust in contact area.
	Type 410		Light build up at periphery of contact area

Couple No.	Coupled Materials	Time, Days	Appearance After Test
24	USS Stainless W (N)	40	Rust on surface; rust in contact area.
	USS Stainless W (N)		Rust on surface; rust in contact area.
25	USS Stainless W (N)	40	Rust on surface; build up and rust in contact area.
	Haynes Alloy 25		Stain in contact area
26	Armco 17-4 PH (N)	40	Rust on surface; build up at periphery of contact area.
	Stellite 3		Build up at periphery of contact area
27	Armco 17-4 PH (N)	40	Rust on surface; build up at periphery of contact area.
	Stellite 6		Build up at periphery of contact area
28	Armco 17-4 PH (N)	40	Rust on surface; build up at periphery of contact area.
	Stellite 21		Build up at periphery of contact area
29	Armco 17-4 PH (N)	40	Rust on surface; rust and slight build up in contact area.
	Graphite		Stain in contact area
30	Armco 17-4 PH (N)	40	Rust on surface; rust and slight build up in contact area.
	Graphitar 14		Stain in contact area
31	Armco 17-4 PH (N)	40	Rust on surface; slight build up at periphery of contact area.
	Type 410		Stain and slight build up in contact area

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
32	Armco 17-4 PH (N)	40	Rust on surface; rust and light build up in contact area.
	Armco 17-4 PH (N)		Rust on surface; rust and light build up in contact area.
33	Armco 17-4 PH (N)	40	Rust on surface; rust and build up in contact area.
	Haynes Alloy 25		Stain in contact area
34	Armco 17-4 PH (CP)	40	No apparent change
	Haynes Alloy 25		No apparent change
114	USS Stainless W (N &G)	12	Stain on surface; stain in contact area.
	Graphite		No apparent change
115	USS Stainless W (N &G)	12	Stain on surface; some rust and build up in contact area.
	Graphitar 14		Stain in contact area
116	USS Stainless W (N &G)	12	Stain on surface; stain in contact area.
	Type 410		Light build up at periphery of contact area
117	USS Stainless W (N &G)	12	Stain on surface; patches of build up along periphery of contact.
	USS Stainless W (N &G)		Stain on surface; patches of build up along periphery of contact.
118	Armco 17-4 PH (N &G)	12	Stain on surface; light build up at periphery of contact area.
	Graphite		No apparent change.

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
119	Armco 17-4 PH (N &G)	12	Stain on surface; light build up at periphery of contact area.
	Graphitar 14		Stain in contact area
120	Armco 17-4 PH (N &G)	12	Stain on surface; contact area bright.
	Type 410		No apparent change
121	Armco 17-4 PH (N &G)	12	Stain on surface; stain and light build up in contact area.
	Armco 17-4 PH (N &G)		Stain on surface; stain and light build up in contact area.

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35	USS Stainless W (N)	28	Black coating and rust on surface; stain in contact area.
	Stellite 3		Heavy build up at periphery of contact area
36	USS Stainless W (N)	28	Black coating and rust on surface; stain in contact area.
	Stellite 6		Heavy build up at periphery of contact area
37	USS Stainless W (N)	28	Black coating and rust on surface; stain in contact area.
	Stellite 21		Heavy build up at periphery of contact area
38	USS Stainless W (N)	28	Black coating and rust on surface; stained in contact area.
	Graphite		Stained in contact area

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
39	USS Stainless W (N)	28	Black coating; rust on surface; moderate build up at periphery of contact.
	Graphitar 14		Light build up at periphery of contact area
40	USS Stainless W (N)	28	Black coating and rust on surface; light build up and rust in contact area.
	Type 410		Rust on surface; build up at periphery of contact area.
41	USS Stainless W (N)	28	Black coating and rust on surface; light build up at periphery of contact area.
	USS Stainless W (N)		Black coating and rust on surface; light build up at periphery of contact area.
42	USS Stainless W (N)	28	Severe rusting of surface; moderate build up at periphery of contact area.
	Haynes Alloy 25		Build up at periphery of contact area
43	Armco 17-4 PH (N)	28	Black coating and rust on surface; build up at periphery of contact area.
	Stellite 3		Build up at periphery of contact area
44	Armco 17-4 PH (N)	28	Black coating and rust on surface; build up at periphery of contact area.
	Stellite 6		Build up at periphery of contact area

Couple No.	Coupled Materials	Time, Days	Appearance After Test
45	Armco 17-4 PH (N)	28	Black coating and rust on surface; build up at periphery of contact area.
	Stellite 21		Build up at periphery of contact area
46	Armco 17-4 PH (N)	28	Black coating and rust on surface; moderate build up at periphery of contact area.
	Graphite		Stain and build up in contact area
47	Armco 17-4 PH (N)	28	Black coating and rust on surface; moderate build up at periphery of contact area.
	Graphitar 14		Light build up at periphery of contact area
48	Armco 17-4 PH (N)	28	Black coating; rust on surface; light build up at periphery of contact area.
	Type 410		Rust on surface; light build up at periphery of contact area.
49	Armco 17-4 PH (N)	28	Black coating; rust on surface; light build up at periphery of contact area
	Armco 17-4 PH (N)		Rust on surface; light build up at periphery of contact area.
50	Armco 17-4 PH (N)	28	Black coating and rust on surface; heavy build up at periphery of contact area.
	Haynes Alloy 25		Heavy build up at periphery of contact area

Couple No.	Coupled Materials	Time, Days	Appearance After Test
51	Armco 17-4 PH (CP)	28	Very light stain at periphery of contact area
	Haynes Alloy 25		Very light stain at contact area
122	USS Stainless W (N & G)	14	Rust on surface; stain and slight build up in contact area
	Stellite 3		Light build up at periphery of contact area
123	USS Stainless W (N & G)	14	Rust on surface; stain and slight build up in contact area
	Stellite 6		Light build up at periphery of contact area
124	USS Stainless W (N & G)	14	Rust on surface; stain and slight build up in contact area
	Stellite 21		Light build up at periphery of contact area
125	USS Stainless W (N & G)	14	Rust on surface; build up at periphery of contact area
	Graphite		Stain in contact area
126	USS Stainless W (N & G)	14	Rust on surface; build up at periphery of contact area
	Graphitar 14		Stain in contact area
127	USS Stainless W (N & G)	14	Stain on surface; build up at periphery of contact area
	Type 410		Some rust on surface; moderate build up at periphery of contact area
128	USS Stainless W (N & G)	14	Rust on surface; light build up at periphery of contact area
	USS Stainless W (N & G)		Rust on surface; light build up at periphery of contact area

Couple No.	Coupled Materials	Time, Days	Appearance After Test
129	USS Stainless W (N & G)	14	Rust on surface; heavy build up at periphery of contact area.
	Haynes Alloy 25		Heavy build up at periphery of contact area
130	Armco 17-4 PH (N & G)	14	Rust on surface; stain and light build up in contact area.
	Stellite 3		Light build up at periphery of contact area
131	Armco 17-4 PH (N & G)	14	Rust on surface; stain and light build up in contact area.
	Stellite 6		Light build up at periphery of contact area
132	Armco 17-4 PH (N & G)	14	Rust on surface; stain and light build up in contact area.
	Stellite 21		Light build up at periphery of contact area
133	Armco 17-4 PH (N & G)	14	Severe rust on surface; moderate build up at periphery of contact area.
	Graphite		Light build up at periphery of contact area
134	Armco 17-4 PH (N & G)	14	Severe rust on surface; moderate build up at periphery of contact area.
	Graphitar 14		Light build up at periphery of contact area
135	Armco 17-4 PH (N & G)	14	Rust on surface; build up at periphery of contact area
	Type 410		Rust on surface; build up at periphery of contact area.

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
136	Armco 17-4 PH (N & G)	14	Rust on surface; build up at periphery of contact area.
	Armco 17-4 PH (N & G)		Rust on surface; build up at periphery of contact area.
137	Armco 17-4 PH (N & G)	14	Rust on surface; build up at periphery of contact area.
	Haynes Alloy 25		Rust on surface; build up at periphery of contact area.

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52	USS Stainless W (N)	28	Black coating and rust on surface; slight build up at periphery of contact area.
	Stellite 3		Slight build up at periphery of contact area
53	USS Stainless W (N)	28	Black coating and rust on surface; slight build up at periphery of contact area.
	Stellite 6		Slight build up at periphery of contact area
54	USS Stainless W (N)	28	Black coating and rust on surface; slight build at periphery of contact area.
	Stellite 21		Slight build up at periphery of contact area
55	USS Stainless W (N)	28	Black coating and rust on surface; slight build up at periphery of contact area.
	Graphite		Stain in contact area

Couple No.	Coupled Materials	Time, Days	Appearance After Test
56	USS Stainless W (N)	28	Black coating and rust on surface; build up at periphery of contact area.
	Graphitar 14		Build up at periphery of contact area
57	USS Stainless W (N)	28	Black coating and rust on surface; slight build up at periphery of contact area.
	Type 410		Several rust spots on surface; build up at periphery of contact area.
58	USS Stainless W (N)	28	Black coating and light rust on surface; slight build up at periphery of contact area.
	USS Stainless W (N)		Black coating and light rust on surface; slight build up at periphery of contact area.
59	USS Stainless W (N)	28	Black coating and rust on surface; rust and build up in contact area.
	Haynes Alloy 25		Build up at periphery of contact area
60	Armco 17-4 PH (N)	28	Black coating and rust on surface; rust and build up in contact area.
	Stellite 3		Build up at periphery of contact area
61	Armco 17-4 PH (N)	28	Black coating and rust on surface; rust and build up in contact area
	Stellite 6		Build up at periphery of contact area

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
62	Armco 17-4 PH (N)	28	Black coating and rust on surface; rust and build up in contact area.
	Stellite 21		Build up at periphery of contact area
63	Armco 17-4 PH (N)	28	Black coating and rust on surface; rust and build up in contact area.
	Graphite		Build up at periphery of contact area
64	Armco 17-4 PH (N)	28	Black coating and rust on surface; rust and build up in contact area.
	Graphitar 14		Build up at periphery of contact area
65	Armco 17-4 PH (N)	28	Build up at periphery of contact area.
	Type 410		Build up at periphery of contact area
66	Armco 17-4 PH (N)	28	Black coating and rust on surface; rust and build up in contact area.
	Armco 17-4 PH (N)		Build up at periphery of contact area
67	Armco 17-4 PH (N)	28	Black coating and rust on surface; rust and build up in contact area.
	Haynes Alloy 25		Build up at periphery of contact area
68	Armco 17-4 PH (CP)	28	Light stain at periphery of contact area
	Haynes Alloy 25		Light stain at periphery of contact area

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
138	USS Stainless W (N &G) Stellite 3	14	Stain on surface; slight build up at periphery of contact area. Slight build up at periphery of contact area.
139	USS Stainless W (N &G) Stellite 6	14	Stain on surface; slight build up at periphery of contact area. Slight build up at periphery of contact area.
140	USS Stainless W (N &G) Stellite 21	14	Stain on surface; slight build up at periphery of contact area. Slight build up at periphery of contact area
141	USS Stainless W (N &G) Graphite	14	Rust on surface; stain in contact area. Stain in contact area
142	USS Stainless W (N &G) Graphitar 14	14	Rust on surface; build up at periphery of contact area. Stain in contact area
143	USS Stainless W (N &G) Type 410	14	Rust on surface; light build up at periphery of contact area Stain and build up in contact area
144	USS Stainless W (N &G) USS Stainless W (N &G)	14	Rust on surface; light build up at periphery of contact area. Rust on surface; light build up at periphery of contact area.

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
145	USS Stainless W (N & G) Haynes Alloy 25	14	Rust on surface; build up at periphery of contact area. Build up at periphery of contact area
146	Armco 17-4 PH (N & G) Stellite 3	14	Rust on surface; build up at periphery of contact area Build up at periphery of contact area
147	Armco 17-4 PH (N & G) Stellite 6	14	Rust on surface; build up at periphery of contact area Build up at periphery of contact area
148	Armco 17-4 PH (N & G) Stellite 21	14	Build up at periphery of contact area Build up at periphery of contact area
149	Armco 17-4 PH (N & G) Graphite	14	Build up at periphery of contact area Build up at periphery of contact area
150	Armco 17-4 PH (N & G) Graphitar 14	14	Rust on surface; build up at periphery of contact area Build up at periphery of contact area
151	Armco 17-4 PH (N & G) Type 410	14	Rust on surface; build up at periphery of contact area. Build up at periphery of contact area
152	Armco 17-4 PH (N & G) Armco 17-4 PH (N & G)	14	Rust on surface; build up at periphery of contact area Build up at periphery of contact area

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<u>Couple No.</u>	<u>Coupled Materials</u>	<u>Time, Days</u>	<u>Appearance After Test</u>
153	Armco 17-4 PH (N &G)	14	Rust on surface; build up at periphery of contact area.
	Haynes Alloy 25		Build up at periphery of contact area

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69	USS Stainless W (N)	14	Black coating
	Stellite 3		Tarnish film; some pitting and very slight build up at periphery of contact area.
70	USS Stainless W (N)	14	Black Coating
	Stellite 6		Tarnish film; some pitting and very slight build up at periphery of contact area.
71	USS Stainless W (N)	14	Black coating
	Stellite 21		Tarnish film; some pitting and very slight build up at periphery of contact area.
72	USS Stainless W (N)	14	Black coating
	Graphite		No apparent change
73	USS Stainless W (N)	14	Black coating; light stain in contact area.
	Graphitar 14		Light stain in contact area
74	USS Stainless W (N)	14	Black coating
	Type 410		Black coating
75	USS Stainless W (N)	14	Black coating
	USS Stainless W (N)		Black coating

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
76	USS Stainless W (N)	14	Black coating
	Haynes Alloy 25		Tarnish film
77	Armco 17-4 PH (N)	14	Black coating
	Stellite 3		Tarnish film; some pitting and very slight build up at periphery of contact area
78	Armco 17-4 PH (N)	14	Black coating
	Stellite 6		Tarnish film; some pitting and very slight build up at periphery of contact area
79	Armco 17-4 PH (N)	14	Black coating
	Stellite 21		Tarnish film; some pitting and very slight build up at periphery of contact area.
80	Armco 17-4 PH (N)	14	Black coating
	Graphite		No apparent change
81	Armco 17-4 PH (N)	14	Black coating; light stain in contact area.
	Graphitar 14		Light stain in contact area
82	Armco 17-4 PH (N)	14	Black coating
	Type 410		Black coating
83	Armco 17-4 PH (N)	14	Black coating
	Armco 17-4 PH (N)		Black coating
84	Armco 17-4 PH (N)	14	Black coating
	Haynes Alloy 25		Tarnish film

Couple No.	Coupled Materials	Time, Days	Appearance After Test
85	Armco 17-4 PH (CP)	14	Tarnish film
	Haynes Alloy 25		Tarnish film
154	USS Stainless W (N)	12	Black coating
	Graphite		No apparent change
155	USS Stainless W (N &G)	12	Black coating; light stain in contact area.
	Graphitar 14		Light stain in contact area
156	USS Stainless W (N &G)	12	Black coating
	Type 410		Black coating
157	USS Stainless W (N &G)	12	Black coating
	USS Stainless W (N &G)		Black coating
158	Armco 17-4 PH (N &G)	12	Black coating
	Graphite		No apparent change
159	Armco 17-4 PH (N &G)	12	Black coating; light stain in contact area.
	Graphitar 14		Light stain in contact area
160	Armco 17-4 PH (N &G)	12	Black coating
	Type 410		Black coating
161	Armco 17-4 PH (N & G)	12	Black coating
	Armco 17-4 PH (N & G)		Black coating
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86	USS Stainless W (N)	31	Black coating
	Stellite 3		Tarnish film; some pitting and very slight build up at periphery of contact area.

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
87	USS Stainless W (N)	31	Black coating
	Stellite 6		Tarnish film; some pitting and very slight build up at periphery of contact area.
88	USS Stainless W (N)	31	Black coating
	Stellite 21		Tarnish film; some pitting and very slight build up at periphery of contact area.
89	USS Stainless W (N)	31	Black coating
	Graphite		No apparent change
90	USS Stainless W (N)	31	Black coating
	Graphitar 14		No apparent change
91	USS Stainless W (N)	31	Black coating; slight build up at periphery of contact area.
	Type 410		Tarnish film; slight build up at periphery of contact area.
92	USS Stainless W (N)	31	Black coating on both samples; couple had to be pried apart.
	USS Stainless W (N)		
93	USS Stainless W (N)	31	Black coating
	Haynes Alloy 25		Tarnish film
94	Armco 17-4 PH (N)	31	Black coating
	Stellite 3		Tarnish film; some pitting and very slight build up at periphery of contact area.

Couple No.	Coupled Materials	Time, Days	Appearance After Test
95	Armco 17-4 PH (N) Stellite 6	31	Black coating Tarnish film; some pitting and very slight build up at periphery of contact area.
96	Armco 17-4 PH (N) Stellite 21	31	Black coating Tarnish film; some pitting and very slight build up at periphery of contact area.
97	Armco 17-4 PH (N) Graphite	31	Black coating No apparent change
98	Armco 17-4 PH (N) Graphitar 14	31	Black coating No apparent change
99	Armco 17-4 PH (N) Type 410	31	Black coating; slight build up at periphery of contact area. Tarnish film; slight build up at periphery of contact area.
100	Armco 17-4 PH (N)	31	Black coating on both samples; couple had to be pried apart.
101	Armco 17-4 PH (N) Haynes Alloy 25	31	Black coating Tarnish film
102	Armco 17-4 PH (CP) Haynes Alloy 25	31	Light tarnish film Light tarnish film
162	USS Stainless W (N & G) Graphite	12	Black coating No apparent change

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Couple No.	Coupled Materials	Time, Days	Appearance After Test
163	USS Stainless W (N &G) Graphitar 14	12	Black coating; light stain on contact area. Light stain in contact area
164	USS Stainless W (N) Type 410	12	Black coating Black coating
165	USS Stainless W (N &G) USS Stainless W (N &G)	12	Black coating Black coating
166	Armco 17-4 PH (N &G) Graphite	12	Black coating No apparent change
167	Armco 17-4 PH (N &G) Graphitar 14	12	Black coating; light stain in contact area. Light stain in contact area
168	Armco 17-4 PH (N &G) Type 410	12	Black coating Black coating
169	Armco 17-4 PH (N &G) Armco 17-4 PH (N &G)	12	Black coating Black coating