

NATIONAL BUREAU OF STANDARDS REPORT

9834

SURFACE PROTECTION OF MARAGING STEEL

QUARTERLY PROGRESS REPORT No. 9

January 1 to March 31, 1968

For

Picatinny Arsenal Project

Order No. A1-8-RF034-01-D1-GG

To

Commanding Officer
Picatinny Arsenal, Dover, N. J. 07801
Attn: Code SMUPA DL-2
Liquid Rocket Propulsion Laboratory



U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards¹ provides measurement and technical information services essential to the efficiency and effectiveness of the work of the Nation's scientists and engineers. The Bureau serves also as a focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. To accomplish this mission, the Bureau is organized into three institutes covering broad program areas of research and services:

THE INSTITUTE FOR BASIC STANDARDS . . . provides the central basis within the United States for a complete and consistent system of physical measurements, coordinates that system with the measurement systems of other nations, and furnishes essential services leading to accurate and uniform physical measurements throughout the Nation's scientific community, industry, and commerce. This Institute comprises a series of divisions, each serving a classical subject matter area:

—Applied Mathematics—Electricity—Metrology—Mechanics—Heat—Atomic Physics—Physical Chemistry—Radiation Physics—Laboratory Astrophysics²—Radio Standards Laboratory,² which includes Radio Standards Physics and Radio Standards Engineering—Office of Standard Reference Data.

THE INSTITUTE FOR MATERIALS RESEARCH . . . conducts materials research and provides associated materials services including mainly reference materials and data on the properties of materials. Beyond its direct interest to the Nation's scientists and engineers, this Institute yields services which are essential to the advancement of technology in industry and commerce. This Institute is organized primarily by technical fields:

—Analytical Chemistry—Metallurgy—Reactor Radiations—Polymers—Inorganic Materials—Cryogenics²—Office of Standard Reference Materials.

THE INSTITUTE FOR APPLIED TECHNOLOGY . . . provides technical services to promote the use of available technology and to facilitate technological innovation in industry and government. The principal elements of this Institute are:

—Building Research—Electronic Instrumentation—Technical Analysis—Center for Computer Sciences and Technology—Textile and Apparel Technology Center—Office of Weights and Measures—Office of Engineering Standards Services—Office of Invention and Innovation—Office of Vehicle Systems Research—Clearinghouse for Federal Scientific and Technical Information³—Materials Evaluation Laboratory—NBS/GSA Testing Laboratory.

¹ Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D. C., 20234.

² Located at Boulder, Colorado, 80302.

³ Located at 5285 Port Royal Road, Springfield, Virginia 22151.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

3120465

April 1, 1968

NBS REPORT

9834

SURFACE PROTECTION OF MARAGING STEEL

BY

V. A. Lamb, J. P. Young, and G. I. Reid

QUARTERLY PROGRESS REPORT No. 9

January 1 to March 31, 1968

for

Picatinny Arsenal Project

Order No. A1-8-RF034-01-D1-GG

to

Commanding Officer

Picatinny Arsenal, Dover, N. J. 07801

Attn: Code SMUPA DL-2

Liquid Rocket Propulsion Laboratory

IMPORTANT NOTICE

NATIONAL BUREAU OF STANDARDS
for use within the Government.
and review. For this reason, the
whole or in part, is not authorized
Bureau of Standards, Washington, D.C.
the Report has been specifically

Approved for public release by the
Director of the National Institute of
Standards and Technology (NIST)
on October 9, 2015

These accounting documents intended
subjected to additional evaluation
listing of this Report, either in
the Office of the Director, National
by the Government agency for which
copies for its own use.



U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

SURFACE PROTECTION OF MARAGING STEEL

A. Objectives of Project

1. To investigate the effect of various metallic coatings on Maraging steel for their ability to protect the steel from contact with mixed hydrazine fuel, for their ability to resist corrosion by mixed hydrazine fuel, and for freedom from catalytic effect on the decomposition of mixed hydrazine fuel.

2. To develop procedures for applying coatings, disclosed as satisfactory under part (1), to the interior surfaces of rocket and missile fuel tanks of complex shape.

B. Summary of Progress in Preceding Quarter

1. Decomposition of MHF-3 fuel in contact with various materials

a. "Background" measurement. The background rate obtained from the mercury manometer units decreased slightly from 0.0058 to 0.0050 cm³/day.

b. Decomposition rates due to various metals at 160°F. Data for units previously on test show little change in trend for the most part. However, there was a small increase in rate for Hg-22, containing a specimen coated with a thin silver deposit. The increase may have been due to porosity in the silver coating. Also, a nearly doubled rate for PA-26 (nickel) was believed due to stopping of a previous leak. A number of new units had been put under test but data from them were not presented because of the shortness of the test period.

2. Results of experiments on kinetics of fuel decompositions.

a. Effect of porosity of coatings. The rate coefficients, based on the areas of Maraging steel exposed through pores in silver coatings, increased a small amount but maintained nearly constant ratios.

b. Effect of area of glass and fuel volume. The continuing results confirm the earlier conclusion that the rate of decomposition of fuel depends primarily on the volume of the liquid phase. Decomposition in the vapor phase and/or at the glass-vapor interface apparently does occur but at a much smaller rate.

c. Effect of contact between metal and fuel vapor. Rate of decomposition of fuel due to contact of vapor with Maraging steel (Unit Hg-38) increased gradually from a very low initial value to a rate approaching one-half that for Maraging steel in contact with liquid fuel. Condensation of liquid on the specimen was suggested as an explanation of the effect.

3. Plating of cylindrical Maraging steel tanks

Two tanks, 26 inches long by 3.46 inches ID, were plated for testing at Picatinny Arsenal. One was plated with electroless nickel and one with cadmium.

C. Progress During Current Report Period

1. Decomposition of MHF-3 fuel in contact with various materials at 160°F.

a. "Background" rate. The rate of evolution of gas from MHF-3 fuel in "blank" mercury manometer test units has continued a slow

decrease. The average is now $0.0044 \text{ cm}^3/\text{day}$ for units Hg-2 and Hg-15, down from $0.0050 \text{ cm}^3/\text{day}$ at the end of the preceding quarterly period.

b. Effects of various metals. Tables 1-3 show the cumulative results to the end of March 1968. The relative reactivity of the various metals is as follows:

Low activity: silver, cadmium, electroless nickel, zinc, tin-nickel alloy, tin, 347 stainless steel, 50-50 lead-tin solder, tungsten, titanium-6 Al-4V, aluminum foil, titanium-3Al-11Cr-13V.

Intermediate activity: gold, lead, nickel, chromium, stainless Maraging steel.

High activity: cobalt, 18% Maraging steel, molybdenum, iron, AM 355 stainless steel, electroless nickel (borohydride), electroless nickel (alkaline bath), Inco-718.

The relative activity of a metal or coating in contact with fuel is not an indication of its corrosion resistance to fuel. Some of the metals, such as molybdenum, induce a high rate of decomposition of fuel but are not corroded. Others, particularly zinc, cause fuel to decompose at a low rate but are severely corroded. Details on corrosion are given in the following section on the examination of exposed specimens (par. C-5).

The behavior of the Type AM 355 stainless steel, and of the electroless nickels prepared from the borohydride and alkaline types of plating baths require special comment.

(1) Type AM 355 stainless steel. The decomposition rate coefficient reported last quarter (Test Unit Hg-44, Table 1), based on only 22 days exposure, was $0.0084 \text{ cm}^3/\text{day}/\text{cm}^2$, higher than found earlier for type 347, but not excessive. However, during the past quarterly

period the rate has accelerated markedly, with an average coefficient to March 31, 1968 of $0.066 \text{ cm}^3/\text{day}/\text{cm}^2$. We do not know the cause. One may hypothesize the following: (1) The passive film has been lost; (2) Some constituent, perhaps by exposure due to loss of the passive film, has a specific accelerating action (molybdenum?). A duplicate of Hg-44, No. Hg-54, has been set up and is yielding essentially the same initial rate as did Hg-44; namely, a coefficient of $0.0087 \text{ cm}^3/\text{day}/\text{cm}^2$ for the first 27 days.

(2) Electroless nickel deposited from the borohydride type bath. The first unit prepared with borohydride electroless nickel contained a specimen plated by a process that is reported to result in incorporation of several percent of boron in the deposit [Narcus, Plating 54, 380 (1967)]. It was extremely active, with a coefficient of gas evolution at room temperature of $0.31 \text{ cm}^3/\text{day}/\text{cm}^2$; at 160°F gas was evolved so rapidly that measurements could be made only over a period of a few minutes. The 15 cm^2 specimen caused evolution of 8.7 cm^3 of gas in 12 minutes, leading to a coefficient of $71 \text{ cm}^3/\text{day}/\text{cm}^2$, or a calculated tank pressure (per Table 1) of 752,000 psi in one year.

In view of this unexpected result we decided to try a proprietary borohydride type electroless nickel that is reputed to give a nearly pure nickel deposit, i.e. nearly boron-free. The results, based on only 27 days exposure, are shown in Table 1, Unit No. Hg-58. The coefficient of $0.018 \text{ cm}^3/\text{day}/\text{cm}^2$ is reasonably comparable to our results for pure nickel.

(3) Electroless nickel deposited from the alkaline type plating bath. This type of electroless nickel is also highly active, based on 29 days exposure.

We do not consider the results described above for the boron-containing electroless nickel or for the deposit from the alkaline bath to be final without further checking. However, they do indicate that some factors not now known may cause high decomposition rates, and serve as a warning that processes or procedures that are superficially similar to acceptable ones may not be trusted without investigation.

Other units in Table 1 placed under test this period, but not already referred to, are Hg-55 (stainless Maraging steel) and Hg-57 (nickel electroplated on Maraging steel).

2. Results of experiments on the kinetics of fuel decomposition.

a. Effect of porosity of coatings. Data on the effect of synthetic pores in silver coatings on Maraging steel are given in Table 4. There has been no significant change from the rate coefficients given in our preceding report (No. 8). Hence, the conclusions stated there still hold; namely, that the rate of fuel decomposition is roughly proportional to pore area, and that the silver in the silver-Maraging steel couple appears to have a marked effect on accelerating the rate at the exposed Maraging steel. Nevertheless, the exposed area is so small that the high specific rate is not significant.

An additional unit that has been set up and is now undergoing leak-checking contains a similar synthetic pore specimen with cadmium as the coating metal.

Unit Hg-51, in Table 4, is also related to these porosity tests. Since, as has previously been discussed, it appears that a bi-metal electrolytic effect may be a factor in both gas evolution and basis metal corrosion, the specimen in Unit Hg-51 was designed to test this effect. The specimen, approximately 4 cm long, was prepared by plating one end, approximately one-half the total surface, with cadmium. The most distant point on the exposed Maraging steel is thus about 2 cm from the cadmium. The rate coefficient of $0.017 \text{ cm}^3/\text{day}/\text{cm}^2$, based on exposed Maraging steel, is only one-fourth to one-sixth that for Maraging steel alone (Unit Hg-23, Table 1; 347-4, Table 3). Hence, the cadmium appears to have a marked inhibiting action over a considerable distance.

b. Effects on fuel decomposition of (a) areas of glass or metal exposed to liquid and vapor and (b) fuel volume. The results for experiments designed to show the effects of glass or metal areas and fuel volume are given in Table 5. Rate coefficients for units Hg-35 and Hg-37 have decreased, but the general conclusions reached previously are unchanged. The coefficient for Hg-35 is now very close to that for Hg-2 and Hg-15, with nearly the same volume of fuel. It is decreasing, approaching the latter with increased time, in line with the behavior of Hg-2 and Hg-15. This low rate occurs even though the glass-vapor area in Hg-35 is five times that in Hg-2 and Hg-15, indicating that the decomposition at the glass-vapor interface is negligible.

The rate for Hg-36 is still about five times that for Hg-2 and Hg-15, and the ratio of fuel volumes is also 5, indicating that the homogeneous liquid phase decomposition is predominant. In Hg-36, the six-fold increase in glass-liquid interface (in comparison with Hg-35) could be the important factor in increasing the rate. However, this is ruled out by the comparison between Hg-36 and Hg-37. In the latter set the glass-liquid areas are the same, but the fuel volume is one-half as large in Hg-37 as in Hg-36, while the rate is decreased correspondingly by a factor of 2.7. Thus, while the ratios are not quantitatively comparable, they are close enough to what would be expected on the basis of liquid-phase decomposition to show that the latter is the dominant factor.

c. Effect of contact between metal and fuel vapor. The rate of decomposition of fuel where the Maraging steel is exposed only to the vapor phase (Hg-38, Table 5) has increased again this quarter to a value approximating one-half that of Maraging steel in liquid fuel. This indicates that condensation of liquid on the metal surface becomes an increasingly important factor with increase in time.

d. Effect of ratio of metal area to fuel volume. Four test units with large areas, respectively, of electroless nickel and of pure nickel in contact with fuel and with varying fuel volume were prepared during the preceding quarterly period and partial preliminary data were shown in Table 6, Report No. 8, January 1, 1968. Their performance has been erratic with the result that no conclusions can be drawn. It was found on review that the metal surfaces were prepared with hydrochloric

acid as a cleaning dip rather than nitric acid, normally used for these metals. Nitric acid is normally used because of its passivating effect. Conversely, hydrochloric acid removes the passive film from these metals. This may be a factor in their erratic effect on fuel decomposition. New units will be set up that avoid this uncertainty.

3. Decomposition of Aerozine-50 fuel in contact with various materials at 160°F.

At the request of Mr. Wahling Ng of Picatinny Arsenal, tests have been started on exposure of several materials to Aerozine-50 fuel. Four test units contain, respectively, type 301 stainless steel-cryogenic form,* titanium (6Al, 4V), 18% Maraging steel, and Teflon (TFE). One unit contains only Aerozine-50, for the purpose of determining the inherent decomposition rate of the fuel itself. Tests have been underway for too short a time to generate meaningful results.

4. Exposure of various materials to oxidizers at 160°F.

The program has been expanded, at the request of Mr. Wahling Ng, of Picatinny Arsenal, to include the exposure of various materials to oxidizers used with rocket fuels. The purpose is to determine rates of corrosion of the materials and rates of decomposition of the oxidizers. The oxidizers to be tested are IRFNA (red fuming nitric acid inhibited with $0.7 \pm 0.1\%$ hydrogen fluoride) and NTO (nitrogen tetroxide containing $0.6 \pm 0.2\%$ nitric oxide). Because of the high vapor pressure of the NTO at test temperature (about 100 psi) it is necessary to use bomb-type test units. Four units have been purchased, including accessory equipment such as valves and gages, but delivery of only two is complete. These

*Aged type.

are now being leak-tested at 300 psig. Leaks that occurred initially appear to have been eliminated by gasket modifications. They will be loaded with test materials in the near future. The test materials to be subjected to oxidizer exposure are as follows:

IRFNA: Teflon, bare Maraging steel

NTO: Teflon, bare Maraging steel, and Type 301 cryogenic form stainless steel

5. Examination of specimens and MHF-3 fuel after tests at 160°F.

The results of the examination of specimens and fuel removed from the units dismantled during the present quarterly period are summarized in Table 6. Photomicrographs of cross-sections of the various metals after exposure are shown in Figures 1 to 13.

a. Corrosion of metals. Of all the metals so far examined after exposure at 160°F, only zinc and nickel underwent extensive corrosion. All of the others showed either no loss of weight or, at most, a few milligrams, corresponding to thickness loss of a few microinch (e.g., the thickness corresponding to 4.8 mg loss of silver, Unit No. 34A, is 12 microinch. However, part of this loss was probably mechanical and part due to dissolving of the underlying Maraging steel).

There is no correlation between activity of a metal in accelerating decomposition of fuel and corrosive attack of the metal. Thus, bare molybdenum, one of the most active decomposers, was uncorroded, while zinc, one of the least active with respect to fuel decomposition, was severely corroded.

Zinc was also most severely corroded at room temperature (see preceding report). Cadmium was less corroded at 160°F than at room temperature. Figure 5 shows that it underwent some pitting attack. Weight loss data for cadmium and electroless nickel (Units Hg-3 and Hg-4) are not given because these coatings were partially amalgamated. Accidental amalgamation spoiled a few early tests. This difficulty was eliminated by use of the glass frit shown in Figure 3, Report No. 7, October 1, 1967.

Data on corrosion of nickel is contradictory. The specimen in Unit No. 316-1 showed little attack. This was a welded specimen, and no effect of the weld on the coating was seen. On the other hand, the nickel coating in Unit No. 347-Ni was visibly etched where it had been in contact with liquid fuel. Weight loss could not be determined because the outside of the tube was severely corroded from submersion in the water bath. However, the high concentration of salts in the fuel, together with the dark green color of the fuel, are evidence that considerable corrosion of the nickel occurred. This unit (similar to Figure 3, Report No. 1, April 1, 1966) was sealed with end plugs made of aluminum. It is possible that the aluminum in the aluminum-nickel couple had an accelerating effect on the corrosion of the nickel. In Unit No. 316-1 the nickel was in the form of a strip specimen, electrically insulated from the stainless steel bomb by placement in a glass cup liner.

The silver plates in Units 34A-D (Table 4 and Table 6) were coated with a hard yellow film which was difficult to remove. Cross-sections of the "pores" on these specimens showed some under-cutting of the silver deposit due to dissolving of the underlying Maraging steel close to the interface (Figure 13), but very little attack of the exposed Maraging steel at greater distance from the silver.

b. Effects on MHF-3 fuel caused by exposure to the metals in the test units. The concentrations of salts dissolved in the fuel were determined by evaporating a known volume of fuel and weighing the residue. Because of the small weight-loss of most of the metals or coatings one would expect to find only small amounts of salts in solution in the fuel. This was the case as seen in Table 6. A determination of the dissolved solids in the original fuel yielded a value of 0.04 g/l. Hence, the amounts shown are in no case worth special comment except in the case of Unit No. 347-Ni. The latter has already been discussed in the preceding paragraph C.5a (p. 10).

Salts that were obtained from the fuel, combined with what could be scraped from specimens, were tested for detonability, with negative results.

D. New Work

1. Tests of special metals.

Duplicates and modifications of a number of the special-interest metals are now being put under test. These tests include nickel and modifications of electroless nickel, such as heat-treatment and special cleaning before exposure.

2. Tests with oxidizers

In addition to the units described above in paragraph 4, several stainless steel bomb-type units, similar to No. 316-1, are being made in the NBS shops for use with IRFNA. A small tank (8" x 2" diam.) made with cryogenic form* type 301 stainless steel was supplied by Picatinny Arsenal. It is to be assembled with fittings and pressure gage and tested with NTO at 90°F. A specimen of 6061-T6 aluminum alloy coated with Teflon was also supplied for exposure to either IRFNA or NTO at 160°F.

3. Other specimens supplied by Picatinny Arsenal

a. One specimen of Maraging steel coated with Teflon, to be tested in MHF-3 at 160°F.

b. One specimen of 7075 aluminum alloy coated with Teflon, to be tested in Aerozine-50 at 160°F.

4. Examination of terminated units

Units for which there is no need for longer testing will be dismantled and examined as time permits. However, it has been requested by Mr. Ng, of Picatinny Arsenal, that materials which show promise
*Unaged type.

remain under test for up to three years. This will alter our planned termination schedule and will result in a larger number of units under test.

TABLE 1

Summary of Test Data for Materials Exposed to MHF-3 at 160°F

Test unit number	Coating or metal	Area of speci- men cm ²	Time under test days	Gas* evolved cm ³	Rate* coeffi- cient cm ³ /day/cm ²		Calculated** tank pressure after 1 year psig ^{xxx}
					present	previous	
					Hg-1 ^x	Silver	
Hg-3 ^x	Cadmium	21.7	473	8.6	0.0014	0.0016	15
Hg-4 ^x	Electroless nickel	19.6	458	14.5	0.0016	0.0016	17
Hg-5 ^x	Tungsten	24.8	358 ^{xx}	46.0	0.0051	0.0052	54
Hg-6 ^x	Gold	22.9	222	49.0	0.0095	--	94
Hg-7 ^x	Zinc	15.6	341 ^{xx}	15.4	0.0029	0.0030	31
Hg-8 ^x	Tin-nickel alloy (65-35)	24.0	358 ^{xx}	51.0	0.0060	0.0057	63
Hg-9 ^x	Cobalt	16.1	199 ^{xx}	--	0.94	--	9,900
Hg-11 ^x	Tin	15.2	432	Zero	Zero	Zero	Zero
Hg-12	Electroless nickel	22.2	432 ^{xx}	10.4	0.0011	0.0012	12
Hg-13 ^x	Cadmium	15.5	85	1.0	0.0019	--	20
Hg-14	347 Stainless steel ^a	13.2	429	Zero	Zero	0.00006	Zero
Hg-16	Cadmium (thin)	9.4	311	0.2	0.0001	0.00007	1
Hg-22	Silver (thin)	16.0	367	39.0	0.0066	0.0058	70
Hg-23	18% Maraging steel ^a	17.0	371 ^{xx}	485	0.097	0.098	1,030
Hg-24	Molybdenum ^a	4.8	368 ^{xx}	217	0.123	0.135	1,300
Hg-25	Iron ^a	4.4	365 ^{xx}	60	0.037	0.045	390
Hg-27	Cadmium (thin)	10.6	263	1.9	0.0007	0.00066	7
Hg-28	347 Stainless steel ^a	6.5	261	5.1	0.0030	0.0013	32

Continued

TABLE 1 (cont'd.)

Summary of Test Data for Materials Exposed to MHF-3 at 160°F

Test unit number	Coating or metal	Area of specimen cm ²	Time under test days	Gas* evolved cm ³	Rate* coefficient		Calculated** tank pressure after 1 year psig
					present	previous	
Hg-29	Silver	14.5	263	Zero	Zero	Zero	Zero
Hg-30 ^b	Titanium ^a (13V, 11Cr, 3Al)	15.0	93	0.3	0.0002	--	. 2
Hg-31	Cadmium	14.7	245	Zero	Zero	Zero	Zero
Hg-32	Tin	13.0	263 ^{xx}	14.7	0.0045	0.0049	48
Hg-33	Solder 50/50 lead-tin ^a	15.3	245	Zero	Zero	Zero	Zero
Hg-39	Inco 718 ^a	12.0	109	120	0.092	0.067	970
Hg-43	Stainless Maraging steel ^a	15.0	161	17.4	0.0072	0.0097	76
Hg-44	Stainless steel type AM 355 ^a	11.4	114 ^{xx}	74.0	0.066	0.0084	670
Hg-45	Titanium ^a alloy, 6Al, 4V	13.3	115	Zero	Zero	Zero	Zero
Hg-48	Aluminum ^c	12.5	107	0.8	0.0006	--	6
Hg-49	Electroless Nickel (Alkaline bath)	12.6	29	84.0	0.23	--	2,400
Hg-50	Chromium	17.0	115	24.0	0.012	0.013	130
Hg-54	Stainless ^a steel AM 355	11.5	27	2.7	0.0087	--	92
Hg-55	Stainless Maraging steel ^a	16.6	27	10.3	0.0230	--	243
Hg-57	Nickel	14.0	27	3.8	0.0101	--	106
Hg-58	Electroless nickel (Proprietary borohydride)	14.2	27	6.8	0.0177	--	187

*Cumulative total, corrected for background rate and to 1 atm. pressure.

**Based on a tank in the form of a cube, 1 cu.ft. volume, 10% ullage.

^xDiscontinued.^{xx}Total days under test are not equal to number of days during which gas was collected, due to temporary leaks, or to interrupted collection due to high rate.^aThese specimens are solid metal. All others are coatings on 18% Maraging steel.^bTitanium alloy, 13% V, 11% Cr, 3% Al. Similar to alloy B120VCA.^cVapor deposited foil from Commonwealth Scientific Corp.

TABLE 2

Results of Tests in Units Equipped with 4 mm Teflon Valves,
Supplied by Picatinny Arsenal.

Temperature, 160°F.

Test unit number	Coating or metal	Area of speci- men cm ²	Time under test days	Gas* evolved cm ³	Rate*		Calculated** tank pressure after 1 year psig		
					coeffi- cient cm ³ /day/cm ²	<u>present</u> <u>previous</u>			
PA-17 ^x	Silver	17.3	55	Unable to obtain tight valve closure					
PA-18	Lead	19.1	55	"	"	"	"	"	"
PA-19 ^{x,xx}	Zinc	15.8	279	56	0.017	--		17	
PA-20 ^{x,xx}	Iron	5.6	193	--	0.23	--		2,400	
PA-21 ^{x,xx}	Molybdenum	3.6	308	--	0.083	--		880	
PA-26 ^{xx}	Nickel	17.0	283	343	0.054	0.043		570	

* Cumulative total, corrected for background rate and to 1 atmosphere pressure.

** Based on a tank in the form of a cube, 1 cu.ft. volume, 10% ullage.

^x Discontinued.

^{xx} Total days under test are not equal to number of days during which gas was collected, due to temporary leaks, or to interrupted collection due to high rate.

TABLE 3

Results of Tests of Materials Exposed to MHF-3 at 160°F
in Stainless Steel Bomb-Type Test Units

Test unit number	Coating or metal	Area of speci- men cm ²	Time under test days	Gas* evolved cm ³	Rate* coeffi- cient cm ³ /day/cm ²		Calculated** tank pressure after 1 year psig
					present	previous	
316-1 ^x	Nickel	16.0	463	169	0.023	0.023	240
347-1 ^x	Lead	20.5	454	40	0.0043	0.0041	45
347-4	18% Maraging steel	14.0	295	308	0.075	0.05	750
347-Ni ^x	Nickel	82	456	1193	0.033	0.033	310

* Cumulative total, corrected for background rate and to 1 atm. pressure.

** Based on a tank in the form of a cube, 1 cu.ft. volume, 10% ullage.

^x Discontinued.

TABLE 4

Data on Decomposition of MHF-3 Fuel at 160°F in Special Test Units

Effect of Porosity in Coatings of Silver on Maraging Steel

Test unit number	Description	Time under test days	Gas evolved cm ³	Rate coefficient of exposed MS	
				present	previous
Hg-34a ^x	Silver on Maraging steel, 20 pores, 0.013" diam., MS area 0.049 cm ²	201	38.8	3.9	4.0
Hg-34b ^x	Silver on Maraging steel, 200 pores, 0.013" diam., MS area 0.42 cm ²	205	85.1	1.02	1.0
Hg-34c ^x	Silver on Maraging steel, 200 pores, 0.026" diam., MS area 1.29 cm ²	189	127.0	0.55	0.58
Hg-34d ^x	Silver on Maraging steel, 1 pore, 0.22" diam., MS area 0.39 cm ²	194	97.0	1.40	1.38
Hg-51	Cadmium on Maraging steel with 1/2 of MS surface exposed	62	8.4	0.017*	--

* Rate coefficient is calculated on basis of exposed Maraging steel. (Total specimen area: 15.4 cm²; exposed Maraging steel: 8 cm².)

^xDiscontinued.

TABLE 5

Effect of Fuel and Vapor Volume and Interfacial Variables
on Rate of Decomposition of MHF-3

Test unit number	Bulb dimensions		Description				Time** under test days	Gas evolved cm ³	Rate cm ³ /day		
	diam. cm	length cm	Liquid Volume cm ³	Vapor Volume cm ³	Glass- liquid cm ²	Glass- vapor cm ²				Metal- vapor cm ²	Interface Area
Hg-2))Av. Hg-15)	2.2	10	31	8	25	35	--	460	2.0	0.0044	0.0050
Hg-35	5.0	10	25	137	25	171	--	207	4.5	0.00061	0.0012
Hg-36	5.0	10	155	7	152	44	--	209	4.7	0.0230	0.021
Hg-37	2.2	20	78	6	158	26	--	213	1.3	0.0084	0.021
Hg-38	2.2	16	4	59	5	141	9.7*	212	82.7	0.048*	0.036*

*The metal is 18% Maraging steel. The rate is cm³/day/cm² of Maraging steel area.

**Total days under test are not equal to number of days during which gas was collected.

TABLE 6

Results of Examination of Fuel and Specimens
After Prolonged Mutual Exposure at 160°F

Unit number	Coating or metal	Gassing rate psi/yr	Period of exposure days	Initial Coating av. thickness mil	Weight of Specimen (g)		Coating Loss as % of weight of coating	Concentration of salts dissolved in fuel g/l**				
					initial	final						
316-1	Ni	237	463	0.9020	1.9	12.290	12.280	10	1.1	0.002	0.21	
Note: Fuel clear-light yellow tint. Residue off-white, very small volume.												
347-1	Pb	43	454	0.9522	2.3	11.6889	11.690	0	0	0	0.43	
Note: Fuel clear-white. Residue white. Small volume. Specimen surface darkened.												
347-Ni	Ni	348	456	--	3	(Outside of cylinder corroded)-						16.7
Note: Fuel dark green. (Ni exposed in contact with Al end plugs). Black, tarry residue. Specimen roughened below fuel line.												
Hg-3	Cd	15	473	1.2815	2.7	11.6882	11.7431	--	--	--	0.23	
Note: Fuel clear-white. Residue brown and sticky. No salts on specimen. Surface roughened. (Partially amalgamated)												
Hg-4	El Ni	17	458	0.5714	2.0	10.1478	10.1517	--	--	--	0.20	
Note: Fuel clear yellow. Residue tan powder. Salts on specimen and bulb-6.5 mg. Specimen unpitted. (Partially amalgamated)												
Hg-5	W	54	358	1.2	1.2	26.1126	26.1132	0	0	0	0.07	
Note: Fuel clear white. No residue. Specimen clean, unpitted.												
Hg-7	Zn	31	341	0.0703	0.25	7.5815	7.5097	71.8	100	0.25	0.31	
Note: Fuel clear light yellow. Residue pink powder. Specimen coated green hard scale. No Zn visible.												

TABLE 6 (cont'd.)

Unit number	Coating or metal	Gassing rate psi/yr	Period of exposure days	Initial Coating		Weight of Specimen (g)		Coating Loss		Concentration of salts dissolved in fuel g/l**
				Initial weight g	av. thickness mil	initial g	final g	loss mg	as % of weight of coating	
Hg-8	Sn-Ni	63	358	--	2.0	25.2724	25.2735	0	0	0.06
Note: Fuel clear white. No residue. Specimen clean, unpitted.										
Hg-11	Sn	0	432	0.6584	2.3	8.3216	8.3145	7.1	1.1	0.025
Note: Fuel clear white. No residue. Specimen surface has blue-black crystal whiskers and is lightly corroded.										
PA-20	Fe	207	193	--	--	0.1465	0.1432	3.3	--	0.18
Note: Fuel clear white. No residue. Specimen has a few pits and is clean and bright.										
PA-21	Mo	99	308	--	--	0.4863	0.4867	0	--	0.07
Note: Fuel clear white. No residue. Specimen and bulb coated with pink salts, 5.3 mg.										
34A	Ag	136	201	1.02	2.6	9.7208	9.7183	4.8*	0.5	0.012
Note: Fuel clear white. No residue. Specimen coated with yellow film (uncollectable).										
34B	Ag	302	205	0.95	2.3	9.8092	9.8112	0	0	0.07
Note: Same as 34A.										
34C	Ag	546	189	0.86	2.2	10.3017	10.3045	0	0	0.06
Note: Same as 34A.										
34D	Ag	417	194	0.93	2.4	8.6265	8.6262	0.3	0.001	0.06
Note: Same as 34A.										

* Removal of stubborn surface film probably removed some silver.

** Original fuel contained 0.04 g/l of dissolved solids. Tabulated values are totals, not corrected for this factor.

Figure 1. (316-1) Cross-section of nickel on welded Maraging steel after 463 days in contact with MHF-3 fuel at 160°F. Welded area is in center of picture. The nickel coating (horizontal white lines) covered the welded Maraging steel uniformly. No attack of the nickel coating is evident. Copper (darker bands) was plated over the nickel coating for protection during polishing. 30X. Modified Fry's etchant.

Figure 2. (316-1) Cross-section of nickel-plated Maraging steel after 463 days exposure to MHF-3 fuel at 160°F. Good coverage of rather rough surface of Maraging steel (bottom) is shown. No attack on nickel (white band) surface is evident. Darker band above nickel is polish-protective copper overlay. 250X. Modified Fry's etchant.

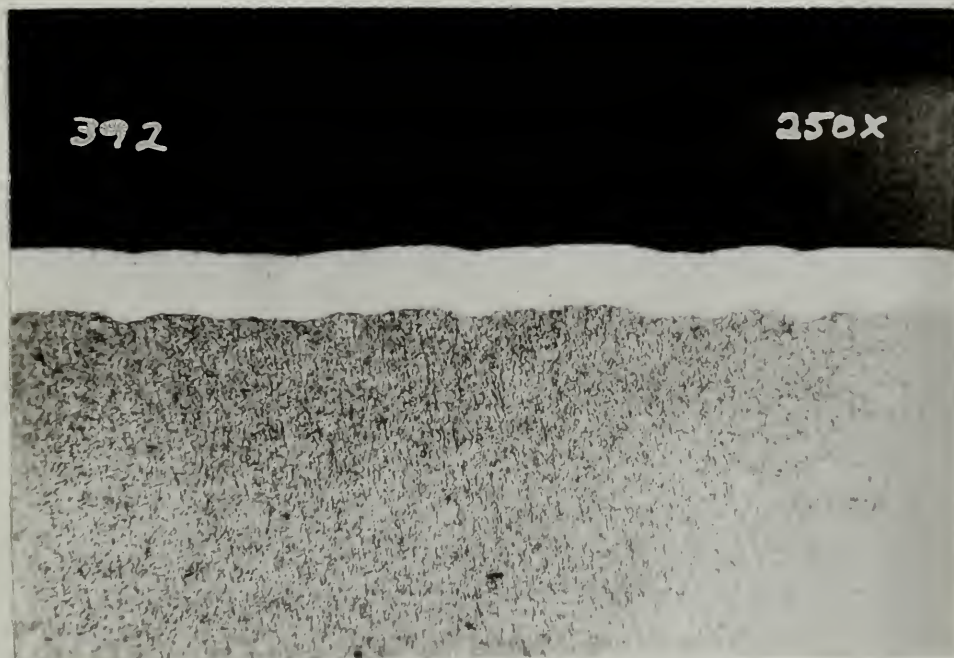
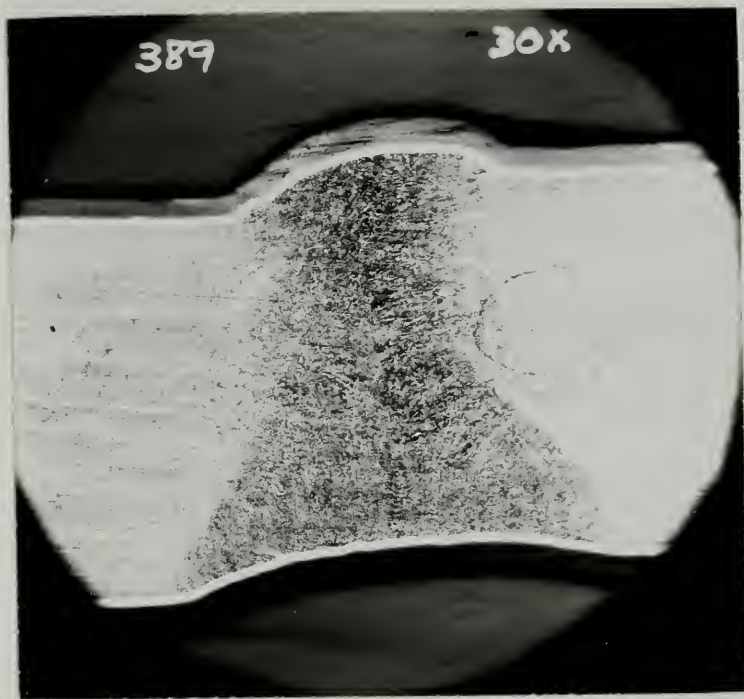


Figure 3. (347-Ni) Cross-section of nickel-plated Maraging steel tube after 456 days in contact with MHF-3 fuel at 160°F. The shallow pit in the nickel coating (light band at top) is of uncertain origin. Slight roughness of surface of nickel believed due to attack of fuel. 250X. Modified Fry's etchant.

Figure 4. (347-1) Cross-section of lead-plated Maraging steel after 454 days in contact with MHF-3 at 160°F. Identification of bands: Bottom (mostly white zone), Maraging steel; center, lead plate (darker band); top (thick, black-edged band), copper coating applied after sectioning as polishing support. As-plated unevenness of the lead deposit is reflected in the copper coating. Little, if any, attack on the lead is evident. 250X. Modified Fry's etchant.

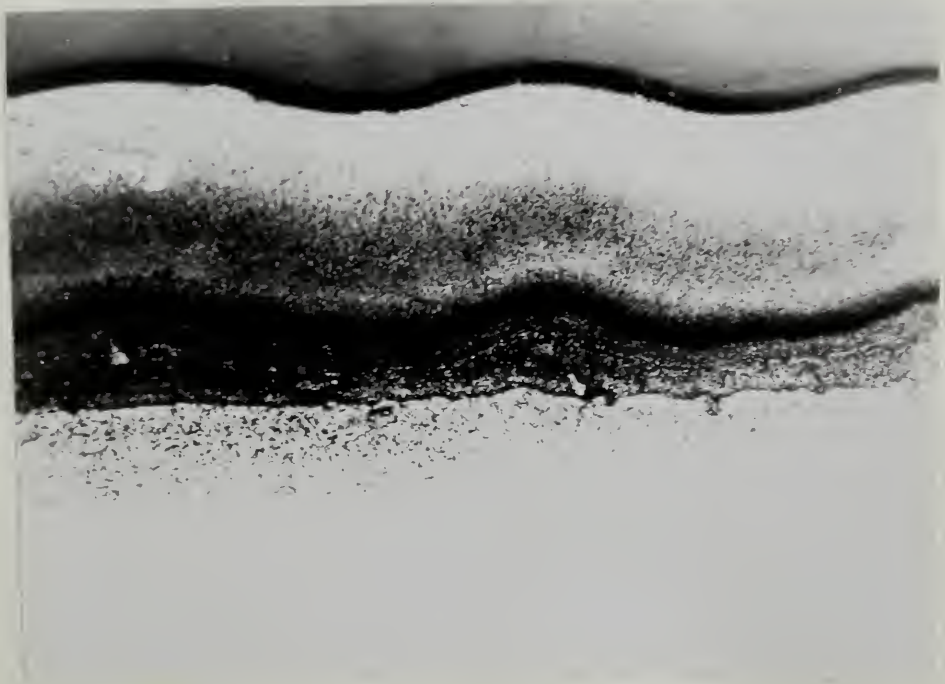
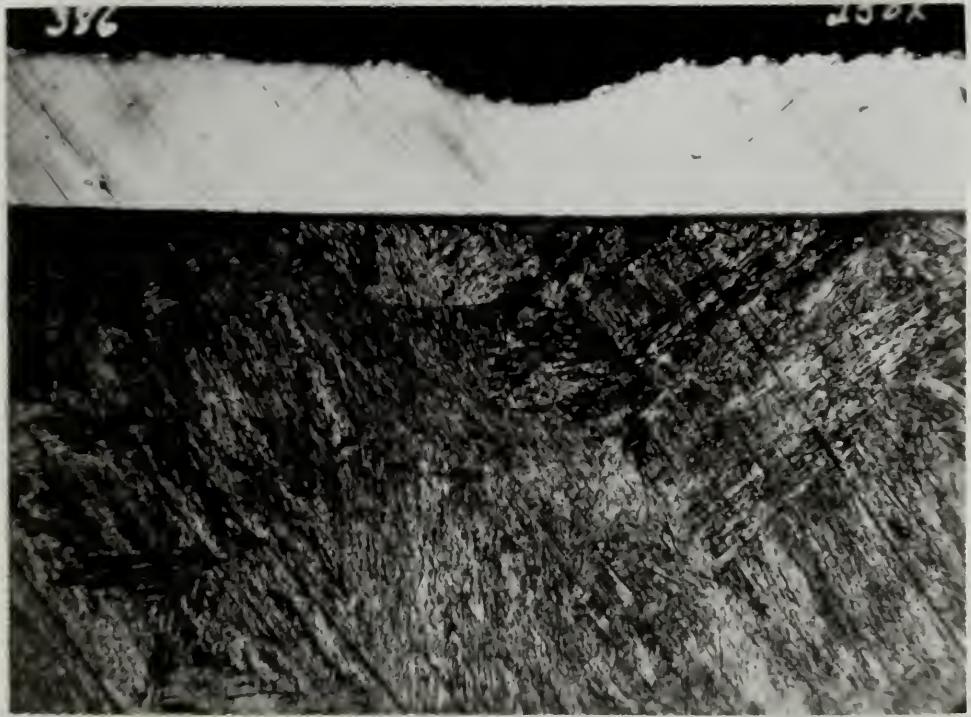


Figure 5. (Hg-3) Cross-section of cadmium plate (band through center of photo) on Maraging steel after 473 days exposure to MHF-3 fuel at 160°F. Irregular etch-corrosion of the surface of the cadmium is evident. The Maraging steel is below and a polish-protective copper deposit above the band of cadmium plate. 150X. Chromic acid etchant.

Figure 6. (Hg-4) Cross-section of electroless nickel on Maraging steel after 458 days exposure to MHF-3 fuel at 160°F. Shown are one edge and part of the sides of the specimen with the Maraging steel at the center surrounded by the white band of nickel, and the thick protective copper overlay applied after sectioning. 30X. Modified Fry's etchant.

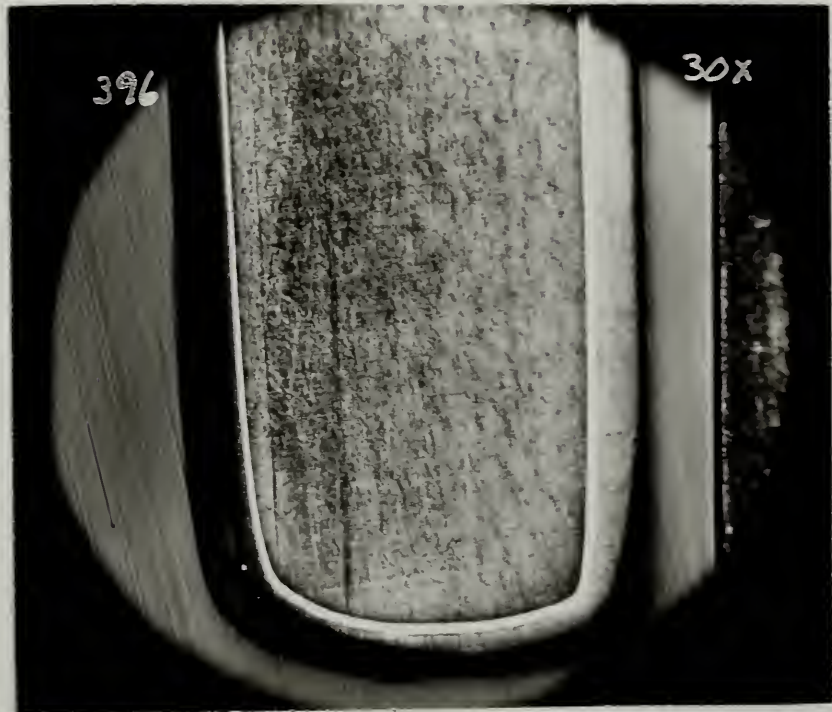
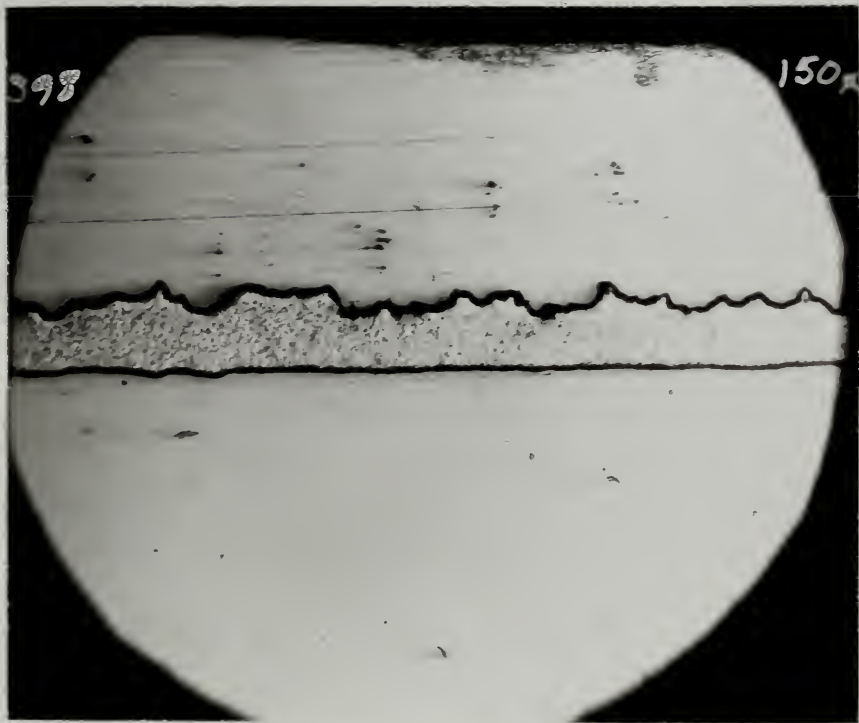


Figure 7. (Hg-4) Cross-section of center of specimen of Maraging steel plated with electroless nickel after 458 days of contact with MHF-3 fuel at 160°F. The nickel (white band at center) shows little evidence of attack. Very slight unevenness at surface of deposit (top) is likely the as-plated condition. 250X. Modified Fry's etchant.

Figure 8. (Hg-5) Cross-section of tungsten deposit on Maraging steel after 358 days in contact with MHF-3 fuel at 160°F. The tungsten is the light band in the center with the Maraging steel below and the copper protective plate above. There was practically no attack of the tungsten. 250X. Modified Fry's etchant.

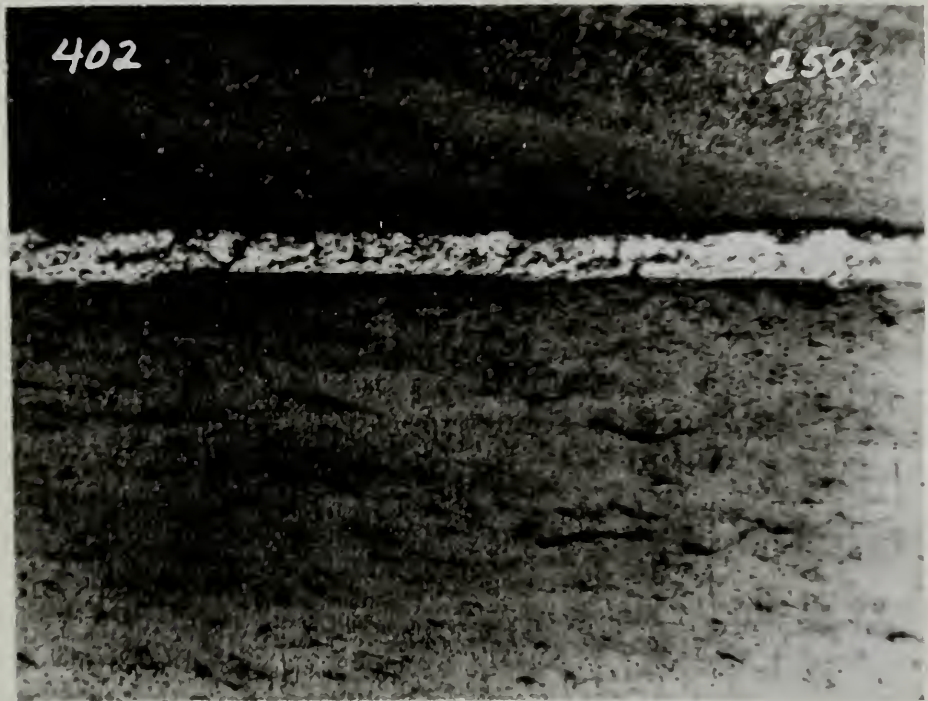
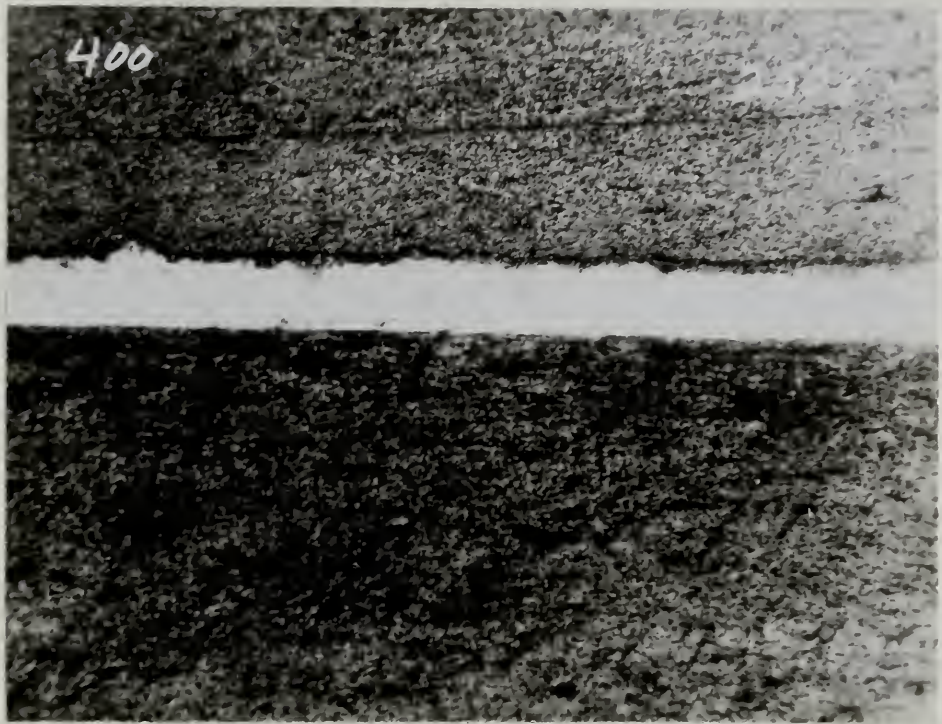


Figure 9. (Hg-7) Surface of zinc-plated Maraging steel after 341 days exposure to MHF-3 fuel at 160°F. This photo shows tightly adherent, dense scale (darker area) partially removed from specimen. All the zinc deposit was affected. 30X. No etch.

Figure 10. (Hg-8) Cross-section of nickel-tin alloy coating on Maraging steel after 358 days exposure to MHF-3 fuel at 160°F. Banded structure of deposit (lighter diagonal strip) due either to interruption during plating, or to a spontaneous cyclic phenomenon during deposition. Unevenness of surface may be due to chipping of the brittle coating during polishing, rather than to attack by fuel. 250X. Modified Fry's etchant.

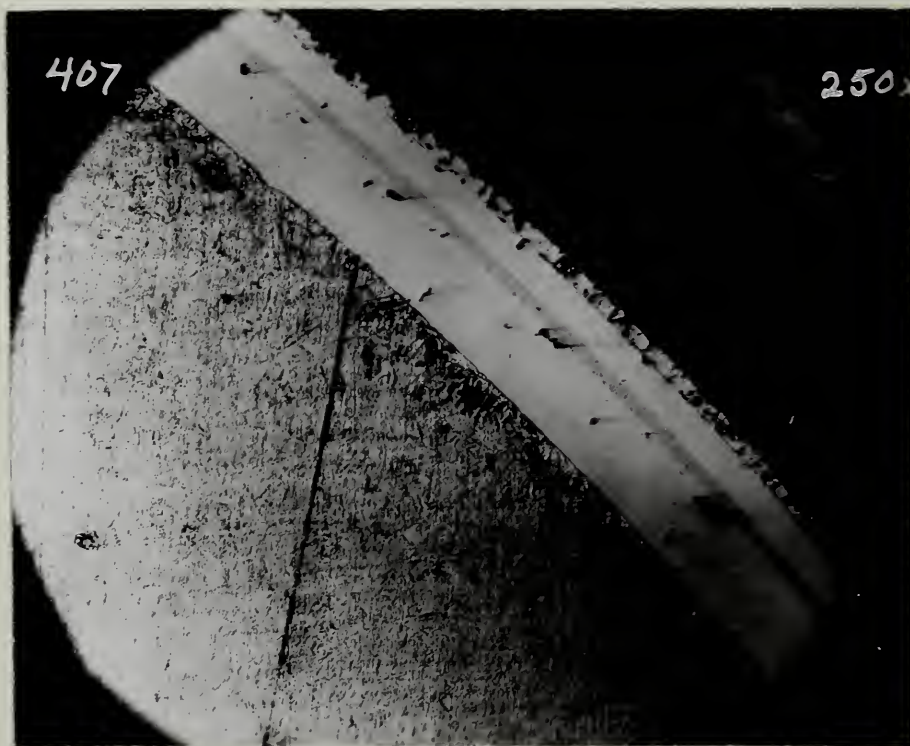
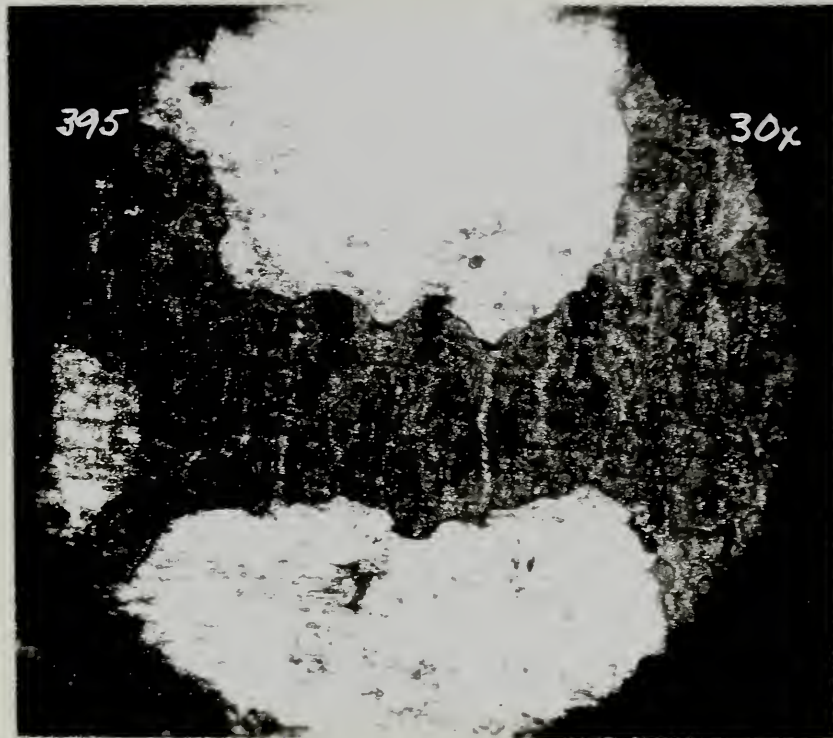


Figure 11. (Hg-11) Cross-section of tin-plated Maraging steel after 432 days exposure to MHF-3 fuel at 160°F. Maraging steel is at bottom of photo with the tin immediately above. Apparent grain structure in deposit appeared without an etch. Roughness of surface of tin coating thought to be as-plated condition. 250X No etch.

Figure 12. (PA 20) Cross-section of iron foil exposed to MHF-3 fuel for 193 days at 160°F. Iron is dark band at center with polish-protective nickel layers above and below. Evidence of some pitting attack may be seen in upper surface of the iron. 250X. Modified Fry's etchant.

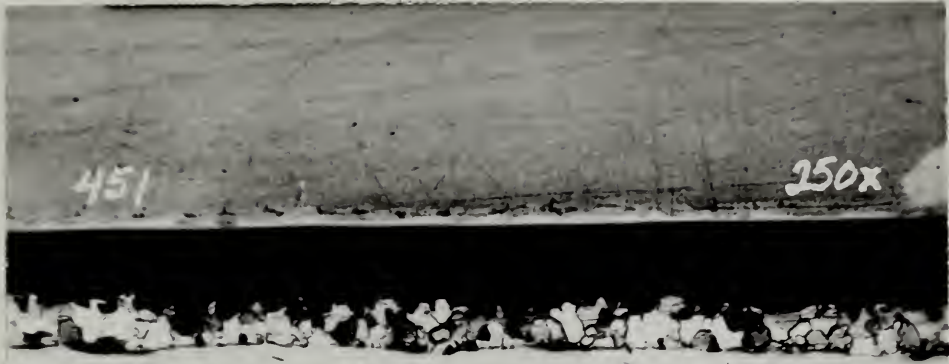


Figure 13. (34-A) Cross-section of silver-plated Maraging steel with artificial pore after 205 days exposure to MHF-3 fuel at 160°F. The layers, top to bottom, are: white nickel polish-protective coating, silver deposit, and Maraging steel. Some undercutting of the silver, which has been filled in with nickel, is evident; otherwise, there is little effect on the exposed Maraging steel. 250X. Modified Fry's etchant.

