

NATIONAL BUREAU OF STANDARDS REPORT

9893

COMPATABILITY OF PROTECTIVE COATINGS, MATERIALS, AND PROPELLANTS

QUARTERLY PROGRESS REPORT No. 10

April 1 to June 30, 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

NATIONAL BUREAU OF STANDARDS

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NBS PROJECT

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By

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U.S. DEPARTMENT OF COMMERCE

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COMPATABILITY OF PROTECTIVE COATINGS, MATERIALS, AND PROPELLANTS

A. Objectives of Project

1. To investigate the effect of various coatings and materials for their ability to withstand the corrosive effects of hydrazine fuels and oxidizers, and for freedom from catalytic effect on the decomposition of fuels and oxidizers.

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. Decomposition rates due to various metals at 160°F.

Metals in the activity ranges of low, intermediate and high remained essentially the same with 347 stainless and the titanium alloys in the low activity classification. Stainless Maraging steel had intermediate activity while AM 355 stainless and Inco-718 were in the high activity range. Of the latter two types it was thought that either the passive film had been lost or some component had an accelerating action. A duplicate of the AM 355 stainless was set up.

Electroless nickel from a proprietary borohydride bath was less active than that from the bath prepared from the formula in the literature.

Electroless nickel from the alkaline bath caused rapid decomposition of fuel. These results were to be checked with duplicate tests.

2. Results of experiments on kinetics of fuel decomposition

a. Effect of porosity of coatings

The results of the continuing experiments confirmed the preliminary conclusion that Maraging steel exposed to fuel through pores in silver coatings caused decomposition of fuel at a rate qualitatively proportional to the pore area. A cadmium coating that partially covered a Maraging steel base had a marked inhibiting action on decomposition of fuel by the Maraging steel.

b. Effect of area of glass and fuel volume

The continued test results confirmed the general conclusions reached previously that decomposition at the glass-vapor interface is negligible and that the homogeneous liquid phase decomposition is predominant.

c. Effect of contact between metal and fuel vapor

The increase in the rate of decomposition indicates that condensation of liquid on the metal surface results in an increasing rate of fuel decomposition with increase in time.

d. Effect of ratio of metal area to fuel volume

No conclusions can be drawn from tests of large areas of pure nickel and electroless nickel because of erratic performance. New units were to be set up.

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

Units were set up with specimens of 301 stainless steel-aged cryogenic form, titanium (6Al, 4V), and 18% Maraging steel and Teflon (TFE) in contact with aeroxine-50, but data were not obtained due to the short period of exposure.

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

Tests were started on rates of corrosion of various materials in contact with two oxidizers, IRFNA and NTO and on rates of decomposition of the oxidizers.

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

a. Corrosion of metals

Out of a large number of metals examined after prolonged exposure to the fuel, only zinc and nickel underwent extensive corrosion. All others at most lost a few milligrams, corresponding to a few microinch in thickness.

b. Effects of exposure to metals on MHF-3 fuel

Except in one experiment with nickel, the concentrations of metallic salts dissolved in the fuel were very small. The composition of the dissolved salts were not determined, but they were separated by evaporation and dried. The dried salts did not detonate under a hammer blow (as might be possible if they were azides).

C. Progress During Current Report Period

1. Decomposition of MHF-3 fuel in contact with various materials at 71°C (160°F)

a. "Background" rate

The rate of evolution of gas from MHF-3 fuel in "blank" (no specimen) mercury manometer test units has continued a slow decrease. The average is now 0.0043 cm³/day for units Hg-2 and Hg-15 with little change from 0.0044 cm³/day at the end of the preceding quarterly period.

b. Effects of various metals

Table 1 contains the cumulative results to the end of June 1968. The relative reactivity of the various specimens is as follows:

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

Intermediate activity: gold, nickel, stainless Maraging steel, electroless nickel (prop. borohydride), Teflon coated Maraging steel.

High activity: Cobalt, 18% Maraging steel, Molybdenum, iron, AM 355 stainless steel, electroless nickel (alkaline bath), Inco-718, chromium.

The behavior of some of the metals and coatings require special comment.

(1) Type AM 355 stainless steel. The two units containing specimens of this steel, Hg-44 and Hg-54, have been set up for 204 and 117 days, respectively. Although the decomposition rate for Hg-54 has increased slightly from 0.0087 cm³/day/cm² at the end

of the last report period to $0.010 \text{ cm}^3/\text{day}/\text{cm}^2$ at the present time, it is much less than the rate attained by Hg-44 of $0.066 \text{ cm}^3/\text{day}/\text{cm}^2$ in about the same period of time. During this reporting period the rate of gas evolution in Hg-44 has further increased to $0.072 \text{ cm}^3/\text{day}/\text{cm}^2$. As noted in the preceding report, the rate for unit Hg-44 was in agreement with that for Hg-54 during the first several weeks of its life, after which the anomolous acceleration started. No explanation for the discrepancy has been found. A third unit is planned for rate confirmation.

(2) Stainless Maraging steel. A discrepancy also exists between the two units containing stainless Maraging steel. The decomposition rate of Hg-43 is nearly three times that of Hg-55 set up some five months later. A third test is therefore planned.

(3) Electroless nickel from borohydride type baths. A duplicate specimen of the non-proprietary borohydride electroless nickel (Report No. 9, p. 4) was tested and an extremely high rate was also obtained. The average coefficient for the two units was $51 \text{ cm}^3/\text{day}/\text{cm}^2$, corresponding to a tank pressure after one year, calculated as in Table 1, of 532,000 psi. Confirmation of the high catalytic effect with this coating indicates that it is a characteristic of the material and is not due to accidental causes. In contrast, the rate of decomposition of fuel in contact with the proprietary borohydride electroless nickel is much lower (Table 1, Unit Hg-58). The non-proprietary deposit is reported to contain several percent of boron, whereas it is claimed that the proprietary

deposit is nearly pure nickel. Such a difference might account for the differing catalytic effects.

(4) Electroless nickel from the alkaline type bath. A second specimen from the alkaline bath has been under test (Table 1, Unit Hg-52). It shows a considerably reduced rate of decomposition compared to the first specimen tested from this bath. Before placing this specimen in a test unit it was placed in boiling water for 60 min., dipped in 10% HCl, rinsed and soaked in fuel for two weeks to remove trace residues that might contaminate the surface. The rate of decomposition of fuel is about one-eighth that obtained with the first specimen plated in the alkaline electroless nickel bath in unit Hg-49, but still much higher than that for electroless nickel plated from the acid-type bath (Table 1, Hg-12).

Other specimens placed under test this period with initial data reported in Table 1 are electroless nickel from the standard acid bath but heated to 800°C before testing to simulate the effect of a welding operation after plating (Hg-61), 301 cryogenic form stainless steel (Hg-53), and Teflon coated Maraging steel (Hg-68).

2. Results of experiments on the kinetics of fuel decomposition

a. Effects of the porosity of coatings

The tests of silver plated specimens with synthetic pores have been terminated. However, two cadmium plated Maraging

steel specimens are under test. One was started during this report period to compare results with those of the silver plated specimens. The data are given in Table 2. The results with the cadmium plated specimens thus far are different than those obtained with the silver where the gas accumulation is roughly proportional to the area of Maraging steel exposed and is greater than for Maraging steel alone. The contrary effect is evidenced by the cadmium plated specimens which have a qualitatively lower accumulation for the area of Maraging steel exposed than for Maraging steel alone. Thus, it appears that in these couples, there is a bi-metallic electrolytic effect, with silver, which is cathodic to Maraging steel, exerting an accelerating effect on both corrosion and fuel decomposition, and cadmium, which is anodic to Maraging steel, having the opposite effect.

b. Effect of ratio of metal area to fuel volume

Three new units have been set up to check the effect of the ratio of metal area to fuel volume (Table 3). Units Hg-65 and Hg-66 contain electroformed nickel sheet 100 cm^2 as the metal specimen. The data show that, for a range of A/V from 0.5 to 6.8, there is no significant difference in the rate coefficient.

3. Decomposition of Aerozine-50 fuel in contact with various materials at 71°C (160°F).

Initial results for these exposure tests are given in Table 4. The background rate is $0.0147 \text{ cm}^3/\text{day}$ for the glass manometer unit. As will be noted in the table, the rates for titanium (Hg-60) and for solid Teflon (Hg-62) are zero after deduction

of the background value. The rates for Maraging steel (Hg-59) and the Teflon coated aluminum (Hg-79) are quite high. The color of the latter specimen has changed from a reddish brown to a light grey which may indicate some reaction with the fuel or leaching of some constituent from the sprayed Teflon. Such effects might cause the difference between the rates for Hg-59 and Hg-62.

4. Exposure of various materials to oxidizers
at 71°C (160°F)

Nine bomb type units have been loaded with oxidizers and specimens of various materials as shown in Table 5. The oxidizers used are NTO (nitrogen tetroxide inhibited with 0.6±0.2% nitric oxide^{*}) and IRFNA (Inhibited red fuming nitric acid. The inhibitor is 0.7±0.1% hydrogen fluoride). Also two background units have been set up, one with each oxidizer. The bombs are similar in design to that shown in Report No. 1, Figure 1. Bodies are type 316 stainless steel, fittings type 304L, and gage tubes type 316 and 347.

The background unit containing IRFNA was erratic and will be disassembled and restarted. Thus there is no background deduction in the data for RFN-2. The other units containing IRFNA showed no increase in pressure so are indicated in Table 2 as having zero reaction.

* 0.6% nitric oxide was added by the supplier, Matheson Co., Inc. It was requested by Picatinny Arsenal that we verify the composition by chemical analysis. Consultation with analytical staff at NBS disclosed no suitable method. The literature on the chemistry of the nitrogen oxides shows that complex labile equilibria exist between NO₂, N₂O₄, NO, N₂O₅, N₂O₃, O₂, and N₂, dependent on temperature and pressure. A meaningful determination of NO therefore appears to be impractical.

A thin walled tank of 301 stainless steel-cryogenic form, unaged, furnished by Picatinny Arsenal, has been loaded with NTO and is being tested at 32°C (90°F). The tank volume is 385 ml; it was filled with oxidizer to 90% of capacity. This tank is designated as 301-NTO in Table 5. There is no appreciable decomposition of NTO thus far.

The background rate for NTO is 0.0126 cm³/day. This figure is used in computing the data for this oxidizer given in Table 2. It is interesting to note that the rate for Teflon is greater than that for Maraging steel.

5. Examination of specimens and MHF-3 fuel after tests at 71°C (160°F).

Four more units were disassembled during this period. Results of examination of specimens and fuel are summarized in Table 6. The fuel from Hg-38 was lost so no salt concentration test could be made.

a. Corrosion of metals

Only the nickel plated Maraging steel from Unit PA-26 showed evidence of attack by the fuel. The nickel was slightly pitted, but since pitted areas did not appear to be large enough to account for the total weight loss, general corrosion of the nickel surface must have occurred. A cross-section of a pit is shown in Figure 1 and of an unpitted area in Figure 2. The surface roughness in the latter indicates general corrosion.

The insignificant weight loss from molybdenum, iron, and Maraging steel would imply no visible corrosion, and none was observed.

b. Effects on MHF-3 fuel caused by exposure to the metals in the test units

As might be expected, the fuel exposed to the nickel specimen contains a significant amount of dissolved salts. The amount in the fuel exposed to Mo is in agreement with the blank. The very small amount in the fuel exposed to iron is consistent with the very minor corrosion loss of the iron specimen.

Salts obtained from the fuel and scraped from the specimens could not be detonated by impact.

D. New Work

1. Tests of special metals

Duplicates of several of the special interest metals and coatings will be set up for tests as indicated in other sections of this report.

2. Tests with oxidizers

The background unit for the test with IRFNA is being dismantled, cleaned and reassembled.

3. Examination of terminated units

Several more units for which there is no need for further testing will be dismantled and examined during the next quarter.

TABLE 1

Summary of Test Data for Materials
Exposed to MHF-3 at 71°C (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		Calculated ^{**} tank pressure after 1 year psig ^{xxx}
					cm ³ /day/cm ²		
					present	previous	
Hg-12	Electroless nickel	22.2	522	12.1	0.0011	0.0011	11
Hg-14	347 Stainless steel ^a	13.2	519	Zero	Zero	Zero	Zero
Hg-16	Cadmium (thin)	9.4	492	2.5	0.0005	0.0001	6
Hg-22	Silver (thin)	16.0	457	53.3	0.0073	0.0066	77
Hg-23 ^x	18° Maraging steel ^a	17.0	461	504	0.081	0.097	854
Hg-24 ^x	Molybdenum ^a	4.8	458	235	0.111	0.123	1,174
Hg-25 ^x	Iron ^a	4.4	455	63.7	0.033	0.037	349
Hg-27	Cadmium (thin)	10.6	353	2.9	0.0008	0.0007	8
Hg-28	347 Stainless steel ^a	6.5	351	6.1	0.0027	0.0030	28
Hg-29	Silver	14.5	353	0.6	0.0001	Zero	1
Hg-30	Titanium ^{a,b} (13V,11Cr,3Al)	15.0	183	0.2	0.00007	0.0002	1
Hg-31	Cadmium	14.7	335	Zero	Zero	Zero	Zero
Hg-32	Tin	13.0	353 ^{xx}	18.3	0.0041	0.0045	43
Hg-33	Solder 50/50 lead-tin ^a	15.3	335	Zero	Zero	Zero	Zero
Hg-39	Inco 718 ^a	12.0	199	131	0.095	0.092	996
Hg-43	Stainless Mar- aging steel ^a	15.0	251	23.5	0.0062	0.0072	66
Hg-44	Stainless steel type AM 355 ^a	11.4	204 ^{xx}	87.3	0.072	0.066	763

Continued

TABLE 1 (Cont'd.)

Summary of Test Data for Materials
Exposed to MHF-3 at 71°C (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 ^{xxx}
					present	previous	
Hg-45	Titanium ^a alloy, 6Al,4V	13.3	200	Zero	Zero	Zero	Zero
Hg-48	Aluminum ^{a,c}	12.5	197	0.9	0.0004	0.0006	4
Hg-49	Electroless nickel (Alkaline bath)	12.6	30	94.2	0.249	0.23	2,630
Hg-50	Chromium	17.0	205 ^{xx}	44.9	0.0145	0.012	253
Hg-52	Electroless nickel (Alkaline bath)	13.8	25	11.3	0.033	--	346
Hg-53	Stainless steel-301 (Cryogenic form)	14.7	56	19.5	0.024	--	250
HG-54	Stainless ^a steel AM 355	11.5	82	8.4	0.010	0.0087	103
Hg-55	Stainless Maraging steel ^a	16.6	87	33.1	0.0229	0.0230	242
Hg-57	Nickel	14.0	117	17.4	0.0106	0.0101	112
Hg-58	Electroless nickel (Proprietary borohydride)	14.2	117	29	0.0176	0.0177	185
Hg-61	Electroless nickel (Heated to 800°C)	12.2	43	0.5	0.001	--	10
Hg-68	Teflon (on Maraging steel)	14.2	14	2.5	0.0126	--	133
347-4 (bomb unit)	18% Maraging steel ^a	14.0	385	431	0.084	0.075	882

*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

Data on Decomposition of MHF-3 Fuel at 71°C (160°F)
 in Special Test Units
 Effect of Porosity in Coatings of Cadmium on Maraging Steel

Test unit number	Description	Time under test days	Gas evolved cm ³	Rate coeffi- cient cm ³ /day/cm ² of exposed MS	
				<u>present</u>	<u>previous</u>
Hg-51	Cadmium on Maraging steel with 1/2 of MS surface exposed - 8 cm ²	155	15.3	0.017*	0.017
Hg-63	Cadmium on Maraging steel with 200 0.026" diam. pores. MS area 1.04 cm ²	36	3.4	0.09**	--

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

** Rate coefficient based on pore area: 1.04 cm².

TABLE 3

Effect of Variation in Ratio of Metal Area to Fuel Volume

Test unit number	Coating or metal	Description			Metal Area (A) cm ²	$\frac{A}{V}$ cm ⁻¹	Time under test days	Gas evolved cm ³	Rate coefficient cm ³ /day/cm ²
		Liquid cm ³	Volume (V) Vapor cm ³						
Hg-46	Nickel ^a	27	15		100	3.7	28	192.6	0.069
Hg-65	ditto	16	18.7		100	6.8	17	68.4	0.042
Hg-66	ditto	28	6.5		100	3.6	14	55.7	0.040
PA-26	Nickel	32	3.2		16	0.5	283	343	0.054
347-Ni	ditto	45	42		82	1.8	456	1193	0.033

^aElectroformed sheet.

TABLE 4.

Test Data for Materials Exposed to Aerozine-50
at 71°C (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
Hg-56	None (background)		94	1.38	0.147	--
Hg-59	Maraging ^a steel	15.2	0.8	41.2	3.39	35,760
Hg-60	Titanium ^a	14.5	94	zero	zero	zero
Hg-62	Teflon ^a (TFE)	17.5	94	zero	zero	zero
Hg-69	Teflon coated 7075-T6 Al	14.2	0.33	9.4	2.01	21,170

* 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.

^a These specimens are uncoated, solid material.

TABLE 5

Results of Tests of Materials Exposed to Oxydizers
at 71°C (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
Inhibited red fuming nitric acid						
RFN-1	Teflon (TFE)	14.5	14	zero	zero	zero
RFN-2	Stainless steel (301 Cryogenic form-aged)	14.7	14	1.48	0.0072	76
RFN-3	Maraging steel	13.6	14	zero	zero	zero
RFN-4	Teflon on 6061-T6 Al	15.1	17	zero	zero	zero
Nitrogen tetroxide						
301-NTO ^x	Stainless steel (301 Cryogenic form-unaged)	338	24	zero	zero	zero
NTO-1	None (background)	--	53	0.67	0.0126	--
NTO-2	Teflon (TFE)	19.9	30	2.7	0.0045	48
NTO-3	Stainless steel (301 Cryogenic form-aged)	14.7	53	zero	zero	zero
NTO-4	Maraging steel	13.6	39	0.2	0.0004	4

* 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 Tested at 32°C (90°F).

TABLE 6

Results of Examination of Fuel and Specimens
After Prolonged Mutual Exposure at 71°C (160°F)

Unit number	Coating or metal	Gassing rate psi/yr	Period of expo- sure days	Initial weight g	Initial Coating		Weight of Specimen (g)		Coating Loss		Concentration of salts dissolved in fuel g/l**
					av. thick- ness mil	initial	final	loss	as % of weight of coating	as av. re- duction of thickness mil	
PA-26	Nickel	570	283	0.730	0.0019	17.2196	17.2006	19	2.6	0.05	1.98
Note: Fuel dark green-brown. Dark residue. Salts on specimen and bulb.											
Hg-24	Molybdenum	1300	368	None	None	0.3636	0.3634	0.2	--	--	0.03
Note: Fuel clear-white. No residue. No salts in bulb.											
Hg-25	Iron	390	365	None	None	0.1634	0.1609	2.5	--	--	0.18
Note: Fuel clear-white. Little light colored residue. No salts in bulb.											
Hg-38	Maraging Steel	424	212	None	None	4.6347	4.6311	3.6	--	--	
Note: Fuel lost.											

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

Figure 1. Cross-section of pit in nickel coating on
Maraging steel (bottom). Modified Fry's
etchant. 250 X

Figure 2. Cross-section of nickel on Maraging Steel
(bottom) showing some attack on the nickel
surface. Modified Fry's etchant. 250 X



