NATIONAL BUREAU OF STANDARDS REPORT

10 069

COMPATIBILITY OF PROTECTIVE COATINGS, MATERIALS, AND LIQUID PROPELLANTS

QUARTERLY PROGRESS REPORT NO. 14
April 1 to June 30, 1969

For
Picatinny Arsenal Project
Order No. A1-8-RFO34G01-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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[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		NBS REPORT
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COMPATIBILITY OF PROTECTIVE COATINGS, MATERIALS, AND LIQUID PROPELLANTS

By
J. P. Young and G. I. Reid

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COMPATIBILITY OF PROTECTIVE COATINGS, MATERIALS, AND LIQUID 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

There was little change in the activity of any of the metals in contact with MHF-3 at 160°F. Tests of some materials, including some stainless steels, were discontinued because of a persistent high rate of fuel decomposition. Sixteen new specimens were assembled in test units and testing started.

2. Experiments on kinetics of fuel decomposition

A unit containing uncoated Maraging steel with end caps of cadmium was added to the group to test the effect of exposed steel. The activity was lower than for Maraging steel alone and was decreasing.

Results of the effect of metal area to fuel volume indicated that the volume of gas produced per unit area per day by a metal is independent of the A/V ratio for the two coatings tested, electrolytic nickel and electroless nickel.

3. Decomposition of Aerozine-50 fuel

Tests of Maraging steel and Teflon coated aluminum with this fuel were discontinued because of excessive activity. Other materials, including 301 stainless steel, titanium, Teflon and MP35N steel, were still under test at 160°F.

4. Exposure of materials to UDMH fuel

Two units containing uncoated Maraging steel specimens show little activity at 160°F.

5. Exposure of materials to oxidizers

There was a slight decrease in the activity of specimens in NTO (Nitrogen Tetroxide) at 160°F. Activity of specimens in IRFNA (red fuming nitric) at 90°F was practically nil.

C. Summary of Progress During Current Period

1. The rates of fuel decomposition caused by specimens exposed to MHF-3 at 160°F remained substantially the same as in the previous quarter. Several units with higher rates were discontinued at the end of this quarter. Fourteen new units were started, including MS coated aluminum by three different companies, two types of steel, and a vaporized-aluminum coated cylinder of Maraging steel. Data on the units containing MHF-3 are given in Table 1 and Figure 1.

2. Effect of porosity of coatings on MS

Tests of the effects of pores in coatings and exposed areas of Maraging steel adjacent to coating metals were continued. Rates for specimens plated with cadmium are low and decreasing while the rate for one speicmen partially coated with electroless nickel is high, as shown in Table 2.

3. Decomposition of Aerozine-50 fuel

The unit containing 301 stainless was terminated because of a consistent high rate of fuel decomposition. Rates for titanium and Teflon did not change significantly. The MP35N alloy steel has a high rate of fuel decomposition (Table 3).

4. Exposure to UDMH fuel

A background unit containing no specimen has been added to the two units with UDMH and Maraging steel which have a low rate of fuel decomposition (Table 4).

5. Exposure of materials to oxidizers

Table 5 shows that the rate of gas evolution for 301 stainless steel, Teflon, Maraging steel, and titanium in contact with NTO remained very low for the current period.

Specimens of Teflon and 301 stainless steel have very little reaction with IRFNA at 90°F. Maraging steel shows some activity.

D. Details of Progress During the Current Report Period

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

a. "Background" rate.

The average rate of evolution of gas in blank (no specimen) mercury manometer test units Hg-2 and Hg-15 has remained the same at 0.0037 cm^3/day during the present period. The rate of the large size background unit has also remained about the same and is close to the value for the standard sized units above. Therefore, it was not considered necessary to use a separate value in calculating results in the

larger units. This result is consistent with previously obtained fuel-volume/surface-area relationships in the decomposition of fuel. It was determined that decomposition occurred mainly in the fuel itself and very little at the fuel-glass interface.

b. Effects of various metals

Table 1 and Figure 1 contain the cumulative results to the end of June 1969. The relative reactivity of the various specimens is as follows:

Low Activity: solder (50/50-Pb/Sn), titanium, aluminum, silver, 347 stainless cadmium, 355 stainless, electroless nickel (std. acid bath), zinc, tin-nickel alloy, tin, tungsten, lead, dip coated solder (50/50-Pb/Sm), Teflon coated MS, oxidized nickel, 301 stainless, dip coated lead-tin alloy.

Moderate activity: gold, nickel, stainless Maraging steel, chromium, electroless nickel 0.1 mil thick on MS, silver 0.5 mil thick on MS and GE aluminum on MS.

High activity: cobalt, 18% Maraging steel, molybdenum, iron, AM 355 stainless, Inco 718, Commonwealth Scien. vapor aluminum on MS, Dow Chemical aluminum on MS.

(1) Behavior of recent units

The performance of several coatings of aluminum on Maraging steel (MS) specimens was disappointing. The high rate of fuel decomposition (see Table I) must be attributed to porosity or incomplete coverage of the MS as aluminum itself has little reaction with fuel. The types of application and the companies who furnished specimens are as follows:

Hg-94-97 Vapor deposition, Commonwealth Engineering, Inc.

Hg-104 Electrodeposited from organic (ether) bath, General Electric Company.

Hg-105 Electroless plated, Dow Chemical Company.

Of these three coatings, the electrodeposit from the ether bath was by far the least reactive. This is most likely because of its thickness of about 2 mils. The two other deposits were less than 0.2 mil thick. The surface of the vapor deposited coating was quite rough, as shown in Figure 3. The appearance of the electroless coating was dark, dull and uneven. Only the electrodeposited coating had the normal white color of aluminum.

A test was developed to check the continuity of a coating on Maraging steel. It consisted of boiling a specimen in distilled water for one-half to two hours and then letting the specimen stand in the water overnight. Any porosity was indicated by rust spots. Of the three aluminum coatings listed above, only the electrodeposited aluminum showed no rust spots.

A second specimen of electroless nickel from the citrate bath on MS (Hg-99) resulted in a low rate, normal for this type of deposit.

A new high alloy, steel, MP35N (Hg-99), produced by the Latrobe Steel Company, had a very low reaction rate with MHF-3. Another steel, 301 stainless, had been exposed to oxidizers (NTO IRFNA) previously but not to MHF-3. Two specimens in units Hg-100 and 101 indicate very low reaction rates.

A cadmium-tin alloy dip-coated MS specimen was prepared and exposed to fuel in unit Hg-102. It had a very low reaction rate. Results to date show that lead-tin or cadmium-tin alloys have among the lowest rates of fuel decomposition. They are inexpensive and may be

applied to the steel by mechanical methods. For these reasons they show much promise as fuel tank coatings for missile tankage systems produced on a volume basis.

The special aluminum alloy, 2021, furnished by the Aluminum Company of America (Hg-96,98) gave no reaction with the fuel during the first 80 days of testing.

(2) Deposit thickness tests on Maraging steel

Although the fuel decomposition rates for thin cadmium deposits of from 0.1 and 0.2 mil (Hg-16,27,81) are in the low range and are decreasing, the rates for similar thin deposits of electroless nickel (Hg-82,103) are substantially higher than for thicker deposits of 1 to 2 mils (Hg-12,61).

(3) Aluminum coated MS cylinder

A 1" diam. 7" long cylinder of MS furnished by Mr. Ng (Picatinny Arsenal) was vapor coated with aluminum on the inside surface by Commonwealth Scientific Corporation. The deposit was in the order of 0.6 mil average thickness. The cylinder was fitted with aluminum end-plugs and Teflon gaskets. One end-plug was machined to allow attachment of a valve and pressure gage. The cylinder was cleaned and filled to 10% ullage with MHF-3 and tested at 160°F. Data are given in Table 1, No. 110. Initial results indicate a low reaction rate with the fuel in spite of the poor showing of the above mentioned MS specimen with a thinner coating by the same process.

2. Results of experiments on the effects of porosity in coatings on MS

The rate of fuel decomposition of the cadmium plated Maraging steel specimens with one-half of the steel surface exposed (Hg-51), the cadmium plated MS specimen with pores drilled to expose the steel (Hg-63) and the MS specimen with end caps of cadmium (Hg-84) continued to decrease. This considerable reduction from the normal rate of decomposition of MHF-3 by MS is attributed to the cathodic protection afforded by the cadmium. The rates in Table 2 are based on the area of exposed MS.

The specimen consisting of electroless nickel on MS with one-half of the steel surface exposed (Hg-71) continued to have a rate nearly ten times that for MS in contact with cadmium. The rate did decline somewhat from that of the previous quarter, however.

3. Fuel tanks to be plated for Picatinny Arsenal

Two one-half scale fuel tanks have been modified and heat treated by the NBS shops. One tank is to be coated inside with aluminum. General Electric Company, which has large facilities for plating aluminum from the ether bath, is now preparing an estimate for the aluminum coating job. The other tank is to be dip coated with a 50/50-lead/tin solder coat. An initial attempt at tinning the tank by coating with zinc chloride flux, heating and pouring in molten solder did not give a satisfactory coating. Two other methods are now under consideration for covering the inside surface of the tank with a continuous coat of solder.

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

The "background" rate for this fuel in a unit with no specimen has increased further from 0.0074 to 0.0084 cm^3/day. The fuel decomposition rates for titanium and Teflon increased very slightly. The new unit containing MP35N alloy steel showed a rather high rate of decomposition during the first 48 days of testing. The data are shown in Table 3.

5. Decomposition of UDMH in contact with specimens at 160°F

The results of the exposure of two specimens of MS to UDMH (unsymmetrical dimethyl hydrazine) fuel are shown in Table 4. The reaction rate is low.

6. Exposure of various materials to oxidizers

Results of the tests in nine bomb-type units are given in Table 5 and Figure 2. The four units containing NTO (Nitrogen Tetroxide inhibited with 0.6 $\pm$ 0.2% of nitric oxide) at 160°F containing 301 stainless, Teflon, MS and titanium changed very little during the quarter and all had a low reaction rate. Also, the small 301 stainless tank containing NTO at 90°F did not change from its previous zero reaction rate.

During this quarter the units containing IRFN (red fuming nitric acid inhibited with 0.7 $\pm$ 0.1% hydrogen fluoride) were run at 90°F because of the relatively rapid loss of the inhibitor at 160°F and the resultant attack on the stainless steel units. At this temperature the reaction rate is very low except for MS which showed some activity on a new specimen started when a previous specimen failed after eleven months of exposure. A sudden increase in pressure in the unit resulted when the

first MS specimen was attacked by the IRFNA. The 60 mil thick specimen was perforated and lost about one-half its weight. It is shown in Figure 4.

Two fluorocarbon resin dispersions were obtained for use in coating the inside of the metal test units to reduce reaction with the oxidizers and for possible testing as tank coatings. The materials are TFE-30 fluorocarbon resin dispersion manufactured by DuPont, and Kel-F dispersion grade KF-633 produced by the 3M Company. The dispersions may be applied by dipping or spraying and in the case of TFE-30 by electrodeposition. However, initial attempts to obtain a pore-free coating by electrodeposition that did not crack on sintering at 760°F were not successful. Stainless steel tubular specimens were dip coated and sintered and the resulting coating withstood IRFNA for several days at room temperature with no visible effect. The main difficulty was obtaining good adhesion to the stainless steel surface. However, it was felt that these fluorocarbon resin coatings were sufficiently effective to be used for protection of the inner surfaces of test bombs used with IRFNA.

D. Summary

The results of the compatibility tests thus far show that different coatings in the acceptable low reaction range have different benefits as follows:

Ease of application - electroless nickel

Greatest protection of MS in fuel - cadmium

Widest range of protection from both fuels and oxidizers - aluminum

Best possibilities for use as thin-film collapsable fuel tank liners - oxidized nickel

Low cost protection of steel - solder (50/50-Pb/Sn)

Other materials which our tests show might be used alone or in conjunction with others and are acceptable for use with fuels and/or oxidizers are: titanium, stainless steels 301, 347, 355AM (oxidized), lead, tin, silver and Teflon.

E. Further Work

It is recommended that further work be done to complete the investigation of materials for possible use in the tankage for the four liquid propulsion systems under consideration. This would include the following:

Compatibility tests of the most promising coatings with Aerozine-50, UDMH and MHF-5.

Stress corrosion tests of the best coatings on MS and the four fuels.

Tests of aluminum coatings with oxidizers.

Metallurgical examination of exposed specimens.

Experimental plating or coating of one-half scale and full-size fuel and oxidizer tanks.

TABLE 1

Summary of Test Data for Materials

Exposed to MHF-3 at 160°F

Test unit number	Specimen (Deposits are 2 mils min. unless otherwise noted)	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	present	Calculated** tank pressure after 1 year psig
Hg-2	Background	--)					
Hg-15	Background	--) av.	909	3.35	0.0037	0.0037	--
Hg-48	Aluminum[a,c]	12.5	566	1.7	0.00019	0.00024	3
Hg-94	Aluminum on MS[c] (0.2 mil thick)	14	43	53.6	--	0.0890	940
Hg-97	Aluminum on MS[c] (0.2 mil thick)	14.3	26	57.4	--	0.1544	1629
Hg-96	Aluminum[a],Type 2021	11.8	80	0.1	--	0.00011	1
Hg-98	Aluminum[a],Type 2021	11.8	80	Zero	--	Zero	Zero
Hg-104	Aluminum on MS by General Electric Co.	14	10	1.5	--	0.0107	113
Hg-105	Aluminum on MS (0.2 mil thick) by Dow Chemical Co.	14	10	10.8	--	0.0771	814
No.110	Aluminum coated MS cylinder[c] (0.6 mil)	114.4	4	2.2	--	0.0048	51
Hg-31	Cadmium	14.7	705	1.9	0.00012	0.00018	2
Hg-16	Cadmium (0.5 mil)	9.4	861	13.4	0.0015	0.0017	18
Hg-27	Cadmium (0.5 mil)	10.6	720	7	0.0009	0.0009	10
Hg-81	Cadmium (0.1 mil)	14.4	94	5.7	0.0059	0.0042	44
Hg-80	Cadmium[a]	13.4	148	7.9	0.0060	0.0040	42
Hg-102	Cadmium-tin alloy (Dipped coating on MS)	14	49	1.5	--	0.0022	23
Hg-12	Electroless nickel	22.2	879	19.1	0.00097	0.0010	10
Hg-61	Electroless nickel (800°C	12.2	396	5.6	0.0011	0.0012	12
Hg-77	Electroless nickel[a]	15.0	141	9.9	0.0047	0.0047	49
Hg-82	Electroless nickel (0.1 mil)	15.3	151	23.8	0.0078	0.0103	109
Hg-90	Electroless nickel (oxidized)	14.4	126	8	0.0061	0.0044	47
Hg-103	Electroless nickel (0.3 mil thick)	15	47	14.4	--	0.0204	216
Hg-106	Electroless nickel from citrate bath	13.6	17	0.5	--	0.0022	23
Hg-87	Maraging steel (oxidized)	15.0	89	90	0.0835	0.0674	712

TABLE 1 (cont.)

Summary of Test Data for Materials

Exposed to MHF-3 at 160°F

Test unit number	Specimen (Deposits are 2 mils min. unless otherwise noted)	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	present	Calculated** tank pressure after 1 year psig
Hg-57	Nickel	14.0	496	27	0.0042	0.0039	41
Hg-75	Nickel (oxidized)[a]	18.5	179	13.9	0.0041	0.0042	44
Hg-29	Silver	14.5	716	3.5	0.00025	0.00037	4
Hg-22	Silver (0.5 mil)[x]	16.0	774	138.9	0.0112	0.0172	181
Hg-33	Solder (Pb/Sn, 50-50)[a]	15.3	705	Zero	Zero	Zero	Zero
Hg-91	Solder (Pb/Sn, 50-50)(Dip coated)	14.3	110	0.7	0.0015	0.00045	5
Hg-107	Solder (Pb/Sn, 50-50)(Dip coated)	14.1	3	Zero	--	Zero	Zero
Hg-14	Stainless steel (347)[a]	13.2	888	Zero	Zero	Zero	Zero
Hg-28	Stainless steel (347)[a]	6.5	716	9.6	0.0019	0.0021	22
Hg-43	Stainless Maraging steel[a]	15.0	605	48.9	0.0058	0.0054	57
Hg-74	Stainless steel (AM355)[a] (oxidized in air at 500°F)	18.5	196	7.1	0.0021	0.0019	21
Hg-89	Stainless (301, aged)[a]	14.7	131	13	0.0043	0.0069	72
Hg-100	Stainless steel (301)[a]	11.9	80	0.4	--	0.00042	4
Hg-101	Stainless steel (301)[a]	10.3	80	0.8	--	0.00097	10
Hg-99	MP35N steel[a] (by Latrobe Steel Co.)	1.855	80	0.1	--	0.00067	7
Hg-68	Teflon coated MS	14.2	381	15.3	0.0033	0.0028	30
Hg-32	Tin	13.0	707	29.1	0.0033	0.0032	33
Hg-30	Titanium[a,b]	15.0	547	0.1	Zero	0.00001	Zero
Hg-45	Titanium (6Al,4V)[a]	13.3	569	Zero	Zero	Zero	Zero

*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.

[a]These specimens are solid metal. All others are coatings on 18% Maraging steel.

[b]Titanium alloy, 13% V, 11% Cr, 3% Al. Similar to alloy B120VCA.

[c]Vapor-deposited from Commonwealth Scientific Corp.

TABLE 2

Data on Decomposition of MHF-3 Fuel at 160°F

in Special Tests

Effect of Porosity in Coatings of Cadmium on Maraging Steel

Test unit number	Description (Deposits 2 mils thick)	Time under test days	Gas evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	present
Hg-51	Cadmium on Maraging steel with 1/2 of MS surface exposed - 8 cm^2	509	40	0.0100	0.0098
Hg-63	Cadmium on Maraging steel with 200 0.026" diam. pores. MS area 1.04 cm^2	401	9.8	0.0248	0.0235
Hg-71+	Electroless nickel on Maraging steel with 1/2 of MS surface exposed - 14 cm^2	252xx	177.3	0.2020	0.1835x
Hg-84	Maraging steel in electrical contact with Cd end caps	118	39	0.0410	0.0383

*Rate coefficient is calculated on basis of exposed Maraging steel only.

**Rate coefficient based on pore area: 1.04 cm^2.

xHighest rate for Maraging steel alone was 0.098 and for standard electroless nickel 0.0047.

xxTotal days under test are not equal to days during which gas was collected.

+Discontinued.

TABLE 3

Test Data for Materials Exposed to Aerozine-50

at 160°F

Test unit number	Specimen	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	Rate* coefficient $cm^3/day/cm^2$ present	Calculated** tank pressure after 1 year psig
Hg-56A	None (Background)		459	3.84	0.0074	0.0084	--
Hg-60A	Titanium[a]	14.5	455	1.8	0.00006	0.00027	3
Hg-62A	Teflon (TFE)[a]	17.5	461	1.3	0.00011	0.00016	2
Hg-95A	MP35N steel[a,b]	1.869	48	6.9	--	0.0769	812

*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.

[b]Latrobe Steel Co., Latrobe, Pennsylvania.

TABLE 4

Test Data for Materials Exposed to UDMH

at 160°F

Test unit number	Specimen	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	Rate* coefficient $cm^3/day/cm^2$ present	Calculated** tank pressure after 1 year psig
Hg-85U	Maraging steel[a]	14.0	117	0.7	0.0019	0.00043	5
Hg-92U	Maraging steel[a]	14.0	111	1.4	0.0011	0.0011	12
Hg-93U	Background	--	80	0.31	--	0.0039	--

*Cumulative total corrected 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

Stainless Steel Bomb-Type Test Units

Test unit number	Specimen	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	present	Calculated** tank pressure after 1 year psig
		Inhibited red fuming nitric acid at 90°F					
RFN-1	Teflon (TFE)	14.5	381xx	14.4	0.0049	Zero	Zero
RFN-2	Stainless steel (301 Cryogenic form aged)	14.7	381xx	13.4	0.0043	Zero	Zero
RFN-3	Maraging steel (New specimen)	25	25	2.0	--	0.0038	40
RFN-4	Teflon on 6061-T6 Al	15.1	384xx	9.5	0.0034	Zero	Zero
RFN-5	None (Background)	--	369xx	6.2	0.038	Zero	Zero
		Nitrogen tetroxide at 90°F					
301-NTO	Stainless steel (301 Cryogenic form un-aged)	338	393	Zero	Zero	Zero	Zero
		Nitrogen tetroxide at 160°F					
NTO-1	None (Background)x	--	115	5.8	0.0417	--	--
NTO-2	Teflon (TFE)	19.9	397xx	23	0.0047	0.0041	43
NTO-3	Stainless steel (301 Cryogenic form aged)	14.7	420xx	6.6	0.0007	0.0008	8
NTO-4	Maraging steel	13.6	406xx	3.7	0.0009	0.0015	16
NTO-5	Titanium (6Al, 4V)	13.4	273xx	0.8	0.00050	0.00035	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.

xDiscontinued.

xxTotal days under test are not equal to days during which gas was collected.

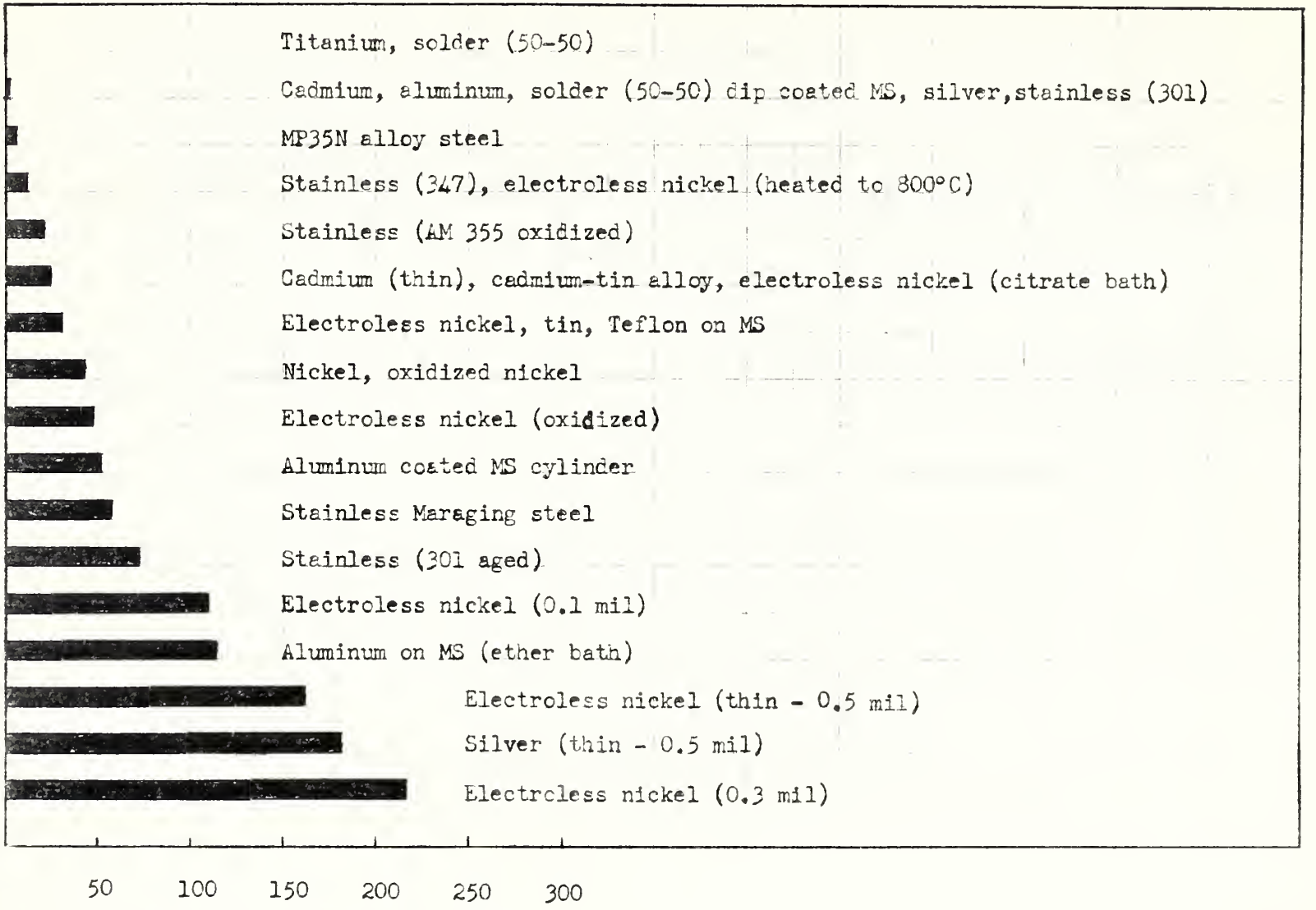

Figure 1

Rate of gas evolution from MHF-3 in contact with indicated metals at 160°F.

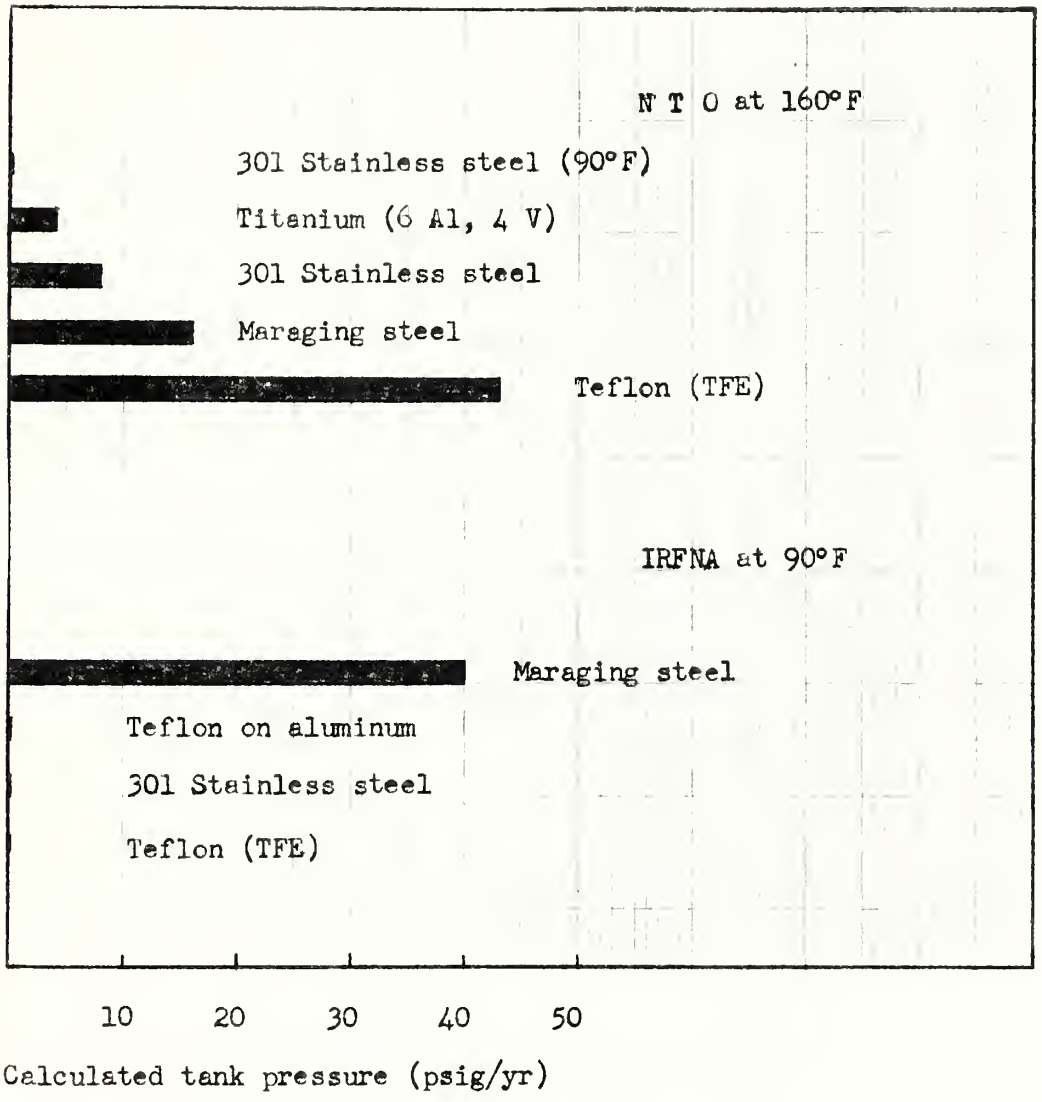

Figure 2

Cumulative gas evolution as indicated by pressure increases resulting from contact of above materials with NTO and IRFNA.

FIGURE 3. As-deposited surface of Maraging steel specimen coated with aluminum by the vapor deposition process of Commonwealth Scientific Corporation. Thickness of deposit about 0.2 mil.

FIGURE 4. Maraging steel specimen (uncoated) exposed to IRFNA for eleven months at 160°F. Note complete perforation near center of 60 mil thick specimen. Magnification 5X.


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