

NRL Report 6353

**Protective Coatings for Magnesium Alloys**  
**Part 2 - Resistance of Flame-Fused Teflon-Coated**  
**Magnesium and Aluminum Alloys to Corrosion by**  
**3% Sodium Chloride Solutions**

D. L. VENEZKY, A. G. SANDS, AND E. B. SIMMONS, JR.

*Organic and Biological Chemistry Branch*  
*Chemistry Division*

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PREVIOUS REPORT IN THIS SERIES

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## ABSTRACT

The corrosion resistance toward 3% sodium chloride solution of flame-fused Teflon-coated magnesium alloy AZ31B-H24, and aluminum alloys 2024-T3, 5052-H32, and 6061-T6 has been studied. The formation of pores in the Teflon coatings applied to magnesium alloy AZ31B-H24 is attributed to hydrogen gas which is evolved from the reaction between the highly acidic Teflon formulations and reactive magnesium alloy. Although a methacrylic acid pretreatment of the magnesium test specimens did not reduce pore formation, the presence of a chrome-pickle film on the magnesium alloy specimens and the use of Teflon one-coat green enamel (du Pont 851-204) alleviated pore formation. Cracks which form when the Teflon is flame fused, especially in thick Teflon coatings on aluminum and magnesium alloys, are attributed in part to the volatile tetrafluorethylene formed when the Teflon is thermally degraded during the flame fusion. Mass spectral data indicate that the major decomposition product, tetrafluoroethylene, is not altered by the magnesium or glass substrate on which it is present during the thermal decomposition. Teflon one-coat green enamel, which has been applied in thin films, 0.0002 to 0.0004 in. thick, and which has been properly flame fused, will not be wet by the 3% sodium chloride solution during the test period; specimens having such Teflon coatings exhibit enhanced corrosion resistance. Three methods were evaluated as accelerated corrosion tests: an immersion test, a salt droplet test, and a spherical-joint contact test which could be modified to measure the relative resistance of the coated specimens. Magnesium alloy AZ31B-H24, chrome pickled, was most resistant to corrosion when coated with flame-fused Teflon one-coat green enamel; magnesium alloy specimens treated otherwise exhibited enhanced corrosion rates. Aluminum alloy 6061-T6, Teflon coated and flame fused, exhibited the greatest corrosion resistance when compared with the other aluminum alloys tested. No correlation was found between the various aluminum alloy tempers studied and the corrosion rate. In general, the corrosion tests indicate that the flame-fused Teflon coating afforded some protection against corrosion as compared to the uncoated specimens.

## PROBLEM STATUS

This is an interim report; work on the problem is continuing.

## AUTHORIZATION

NRL Problem C04-04  
BuWeps Project RRMA 03-073/652-1/F020-03-01

Manuscript submitted September 7, 1965.

## PROTECTIVE COATINGS FOR MAGNESIUM ALLOYS

### PART 2 - RESISTANCE OF FLAME-FUSED TEFLON-COATED MAGNESIUM AND ALUMINUM ALLOYS TO CORROSION BY 3% SODIUM CHLORIDE SOLUTIONS

#### INTRODUCTION

Applied protective coatings and procedures prescribed to maintain the efficiency of the protective coating are all too often found inadequate to prevent extreme corrosion from rendering aircraft unsafe after relatively short marine atmosphere exposures. Greatly improved protective coatings for magnesium and aluminum alloys could extend the use of these light metal alloys, especially the more reactive magnesium alloys, in aircraft construction, as well as reduce the amount of time required to maintain aircraft in proper operational condition. This Laboratory has undertaken the development of improved magnesium protection systems. The study involves three approaches: the chemistry of magnesium and magnesium coordination compounds, the investigation of magnesium protective systems, and development of techniques for application or curing protective coatings.

Since oxides and water between the metal and coating can reduce the effectiveness of protective coatings, a thermal method and properly chosen coating could be used to remove the interfering substances by chemical reaction or evaporation. The application of coatings on alloys by thermal methods was investigated and the effect on mechanical properties of a new technique for fusing Teflon to magnesium and aluminum alloys was recently reported (1). The flame technique which was initially applied to Teflon on rubber substrates (2) caused less alteration of the mechanical properties of the tested alloys than the frequently used 750°F oven method. Since the purpose of the flame technique study was to determine the feasibility of using protective coatings which would require either thermal fusion or thermal cures on magnesium alloys, the initial investigations were limited to commercially available Teflon formulations. Although Teflon was selected because of the thermal fusion process used in its application to various surfaces, and not because of its possible corrosion protection properties (3), initial corrosion results were encouraging enough to warrant further study of thin flame-fused Teflon coatings on magnesium and aluminum alloys.

Where a low coefficient of friction is desired, Teflon coatings have found considerable commercial value on aluminum alloys as well as steel (4). The use of Teflon as a protective coating has been discussed by FitzSimmons and Zisman (5) and more extensively by Thompson and Scott (6) who studied fluorocarbon primer-enamel systems on aluminum alloys and steel. Even though pores have been noted in the coating surface, the metals are reported to be protected against corrosion; a second thin Teflon coating over the fused Teflon coating was recommended for improved protection (3,5). A fluorocarbon-primer surface covered with a fluorocarbon enamel afforded the optimum corrosion protection of steel and aluminum alloys (6). Similar studies of Teflon on magnesium alloys have not yet been reported.

The present study was undertaken to determine the characteristics of flame-fused Teflon surfaces and to determine the resistance of flame-fused Teflon-coated magnesium and aluminum alloys to corrosion in 3% sodium chloride solutions. Several static-test methods were used in the corrosion resistance evaluation of uncoated, Teflon-coated, and methacrylic acid-pretreated Teflon-coated specimens.

## MATERIALS, APPARATUS, AND PROCEDURE

The magnesium alloy used in this work was AZ31B-H24. The aluminum alloys were 2024-T3, 5052-H32, and 6061-T6.

The high-tensile-strength 2024-T3 alloy was selected because the alloy had been solution heat treated, naturally aged, cold worked, and because it exhibits a moderate corrosion rate in 3% sodium chloride solution. Clad and unclad alloys were chosen to illustrate the effects of cladding on the adhesion of the Teflon to the different surfaces, as well as cladding effects on the corrosion properties. For comparison of aging processes on corrosion resistance, two other aluminum alloys were selected: the 6061-T6 alloy, which had been heat treated and artificially aged at elevated temperatures, and the 5052-H32 alloy, which had been cold worked for better ductility than the other two alloys and one-quarter strain hardened. The only magnesium alloy tested was AZ31B-H24, since it is the alloy primarily used in aircraft construction. Chrome-pickled samples were used to estimate effects of chrome pickling on adhesion of the Teflon coating and on corrosion resistance.

The alloy samples, approximately 1/16 in. thick, were cut into test strips measuring 1 in. by 3 in. The test specimens used for controls were washed with xylene before testing; the chrome-pickled magnesium alloy and the clad 2024-T3 aluminum alloy specimens were washed with xylene prior to the application of the Teflon coating. Other specimens used were cleaned by sandblasting with fine grit sand. The process also gave a satin finish, conducive to good adhesion. A xylene wash was used to remove traces of sand or metal particles remaining. All samples were dried in a 50°C oven for 1 hour prior to application of the Teflon coating.

Prior to the Teflon application, a series of magnesium and aluminum alloy specimens were treated with glacial methacrylic acid, which was used as received from Monomer-Polymer, Inc. A thin methacrylate film was applied to the metal surfaces by immersing the specimens in a small quantity of methacrylic acid at room temperature. A 1-minute immersion was optimum; longer immersions caused a gummy formation which was easily removed from the specimen. During the methacrylic acid treatment of magnesium alloys, gassing was observed, indicating chemical reaction; reaction was not evident when the aluminum alloys were treated. The pretreated specimens were drained of excess liquid, air dried, and then dried in a 50°C oven for 1 hour prior to Teflon application.

The procedure fully described in NavWeaps OD 23684 (7) and other reports (3,5,6,8) was followed in the application of the Teflon coatings. The commercially available Teflon spray formulations used were du Pont's Teflon resins: green primer (No. 850-204), clear finish (No. 852-201), and one-coat enamel (No. 851-204). The reported solid contents (% solids by weight) (3) and measured pH's of the formulations were 39%, 1.7; 48%, 4.5; and 48%, 1.5, respectively. The filtered formulations were sprayed on the metals with a DeVilbiss EGA Series spray gun with an F nozzle at 25 to 30 psi air pressure. An approximately 0.0002- to 0.0004-in.-thick coating was applied to both sides and the edges of the test specimen. The sprayed samples were dried in the air overnight and then in an oven at 50°C for 1 hour.

For flame fusion, a natural gas-air flame served as a concentrated source of heat. A National Welding Company Type 3A blowpipe torch fitted with a W-2 nozzle was used to produce a flame of 1-1/2 in. overall length with an inner cone tip of 1 in. measured from the end of the torch (Fig. 1). The flame 1/8 in. in front of the inner cone was directed upon the specimen, passed across the surface at a rate of approximately 1 in./sec, then swept across the panel in the reverse direction, and finally passed over the same path for a third time. The Teflon usually fused during the last sweep; however, the Teflon on the hot

reverse side required fewer sweeps for fusion than unheated surfaces. The Teflon fusion was accompanied by a color or shade change of the applied coating in the area just following behind the flame. The Teflon one-coat green enamel and green primer changed from a yellowish green to a darker green; no color change was observed with the Teflon clear finish. The completely Teflon-covered specimens were more prone to overheating than the specimens coated and fused on one side only as prepared for testing of mechanical properties.

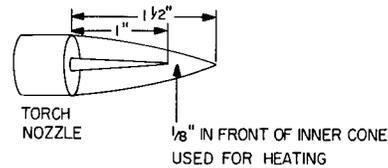


Fig. 1 - Typical flame

Proper fusion of the Teflon was tested by applying a pressure-sensitive tape to the coated surface and rapidly pulling it off. A properly fused coating was not removed by the tape. The surface characteristics of the Teflon coatings were examined under 90X magnification prior to the corrosion tests.

Corrosion resistance of Teflon-coated and uncoated specimens was evaluated by using 3% sodium chloride solutions. The simple immersion tests (Fig. 2) were run in 4-oz wide-mouth jars, 1-1/2 in. in diameter and 3-1/4 in. tall; each test specimen was placed in a separate jar. The 1-in. by 3-in. specimens were inclined at an approximately 45° angle. The jars were one-half filled with 3% sodium chloride solution. During the corrosion test, the tops were loosely fitted so that evaporation would be retarded and any evolved gas could escape. The test solution was agitated daily by swirling the vessel. When the solutions were swirled for mixing, a few drops of solution were allowed to remain on the upper regions of the test panel. In this manner, the effect of salt solution droplets under high humidity conditions could be observed along with the total immersion test.

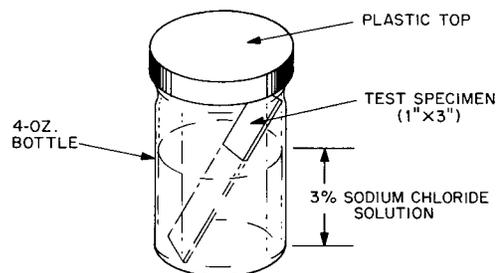


Fig. 2 - Apparatus used for the immersion test

Another test procedure was conducted by clamping an outer 18/9 spherical joint and a neoprene gasket to the test specimen; the standard spherical joint was then filled with 3% sodium chloride (Fig. 3). This test procedure had the advantage of allowing the corrosion tests to be run on several areas of the specimen simultaneously or at a later time as well as providing a test that could be used on a sample coated only on one side of the metal; the reverse side of the same specimen could be used as the control surface. The differences in the resistance of various specimens were measured by filling two spherical joints on either side of Teflon-coated or uncoated specimens (Fig. 4) with a conducting solution and immersing the test assemblage in a constant-temperature bath at  $25^{\circ}\text{C} \pm 0.01^{\circ}\text{C}$ . An Industrial Instruments, Inc., Model RC 1603 conductance bridge was used to measure the resistance of the solutions separated by the alloy specimens.

A third method of testing the specimens was a modified salt droplet test method (9,10). The apparatus consisted of a 4-oz jar fitted with a No. 9 solid rubber stopper having a slot

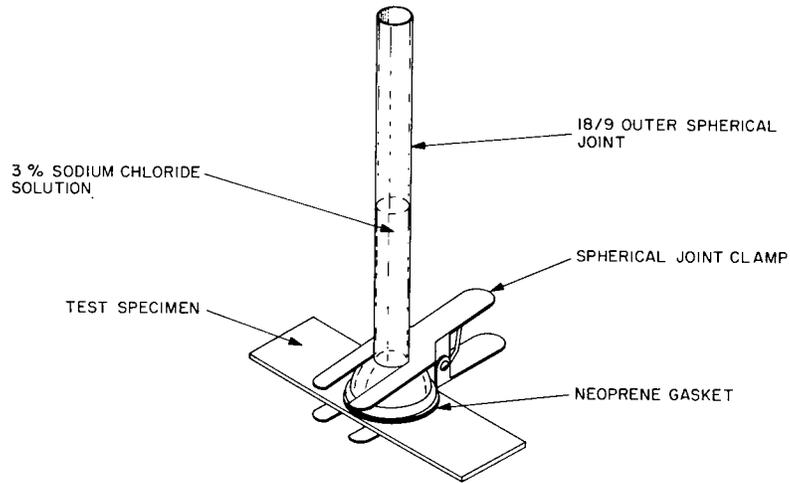


Fig. 3 - Apparatus for static corrosion tests

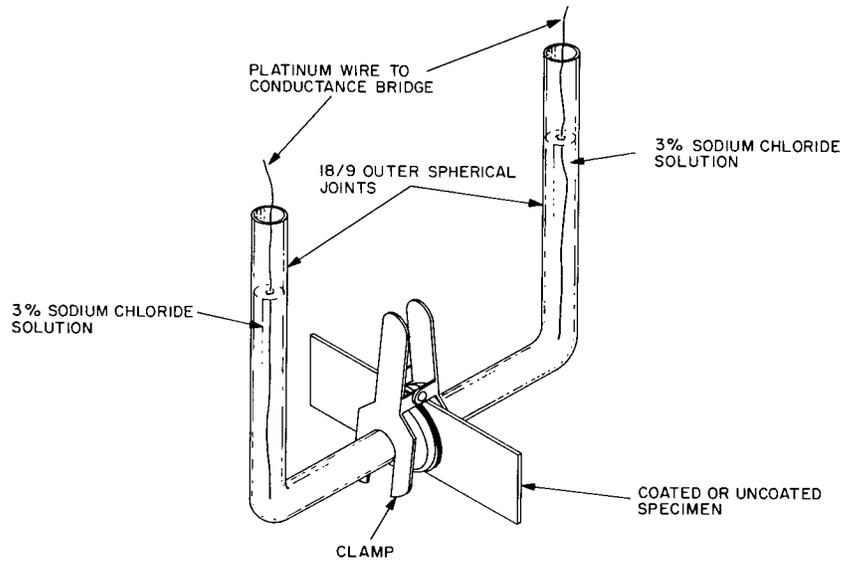


Fig. 4 - Apparatus used to measure the resistance of a coated specimen

on the bottom diameter for holding the test specimen; the slot was made deep enough to allow gases to escape when the jar was stoppered (Fig. 5). The test panels were sprayed every 24 hours with 3% sodium chloride solution using a DeVilbiss No. 251 atomizer. To maintain the environment saturated with water vapor, a small amount of the salt solution was kept on the bottom of the jar, out of contact with the suspended specimen.

No attempts were made to control the temperature of the experiments. All of the corrosion tests were run at room temperatures which varied from 65 to 75°F in the winter and 75 to 90°F in the summer. The test periods varied according to the specimens being

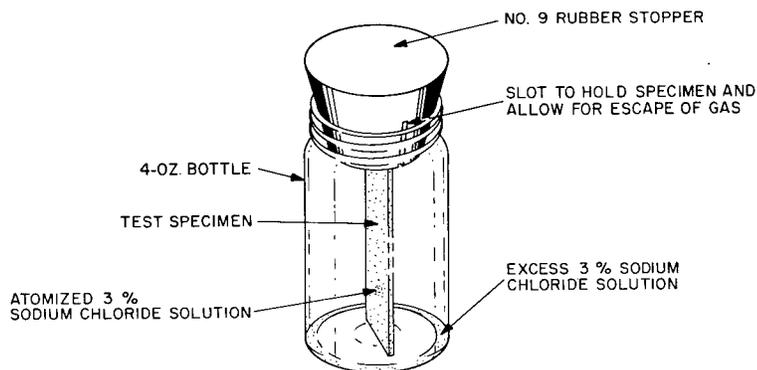


Fig. 5 - Apparatus used in the salt droplet test

tested. Some aluminum samples were immersed for periods of several months, while some magnesium specimens showed complete deterioration in a matter of a few hours or days. After the corrosion test was completed, the fused Teflon coating from several samples which exhibited good corrosion resistance was removed with a razor blade. Care was taken not to scratch the metal surface in the search for pitting under the coating. For specimens tested in this manner, no evidence of pitting was observed under the adherent Teflon coating in the areas tested.

The mass spectral determinations were run on a CEC-620 mass spectrometer. The Teflon-coated specimens and Teflon sheet were 1/2-in. disks stamped out by a metal press. The samples were placed in a 4 in. by 3/4 in. test tube fitted with an outer 19/38 standard tapered joint (Fig. 6). A ring seal was used to allow the tube leading to the mass spectrometer to be close to the decomposing Teflon; the connection from the test tube to the spectrometer was through an inner 12/30 standard joint. The tube containing the specimens was heated by a heating mantle and the temperatures followed by a thermocouple attached to the heating mantle. A thermometer between the heating mantle and test tube was used to check the temperatures read on the thermocouple. The temperature at 500°C (932°F), maximum used, was accurate to  $\pm 50^\circ\text{C}$ . The mass spectrometer was calibrated using n-butane and sulfur hexafluoride. To remove volatile surface contaminants, the specimens were subjected to reduced pressures prior to heating. The relative abundances of the ions were calculated based on the largest ion current observed set at 100%.

Photographs of specimen surfaces were taken by a Polaroid camera attached to a variable-objective microscope. For a record, the extent of the corrosion on a specimen at the conclusion of the testing period was photographed without magnification. The amount and onset of corrosion were made by visual observations and only in a qualitative manner.

## RESULTS AND DISCUSSION

The electron-microscopic study of the poly(tetrafluoroethylene) sintering mechanism by Kargin, Gorina, and Koretskaia (11) indicated that the polymer consists of a packet structure, that is, the macromolecular chains are aggregated in packets which in turn are united into larger aggregates producing secondary structures of a network character. The authors report that in the sintering temperature range (370 to 450°C) no melting which would lead to formation of an integrated mass takes place, nor is there even a partial increase in particle contact. FitzSimmons and Zisman (5) reported that Teflon coatings applied by oven fusion contained many small pores which could not be eliminated because of the high melt viscosity of the Teflon. The pores appeared to offer points of attack for

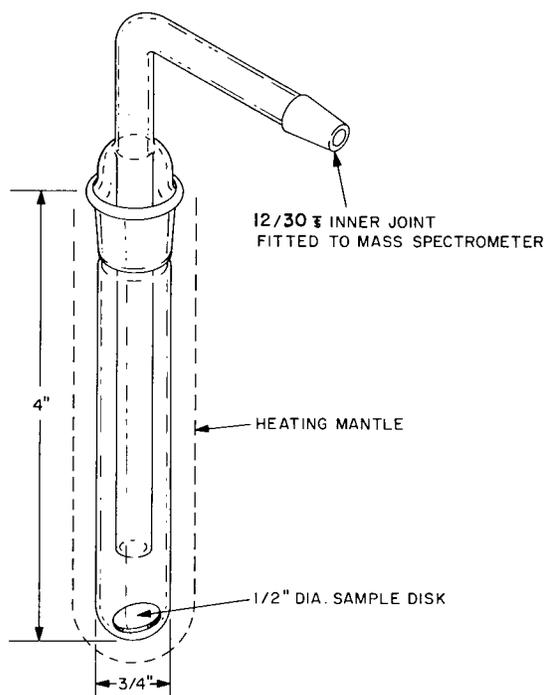


Fig. 6 - Apparatus used for mass spectral analysis of volatile decomposition products

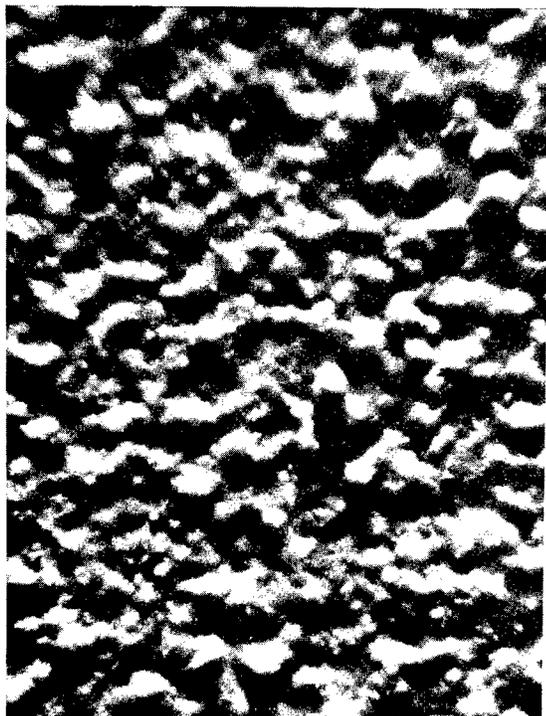
corrosive action, and for the most favorable corrosion protection, additional thin Teflon coatings were recommended. To prevent cracking from developing during the fusion process the applied Teflon coatings should not exceed 0.0003 in. in thickness (5).

A typical applied Teflon coating surface on an aluminum alloy substrate (Fig. 7a) showed an irregular surface with no apparent pores. The same surface after fusion (Fig. 7b) showed little, if any, change; however, if the applied Teflon coating was too thick, the fused surface was characterized by hair-line cracks (Fig. 7c). The Teflon surface on magnesium alloy AZ31B-H24 was often characterized by a perfusion of pores before and after the flame fusion. The Teflon green primer (du Pont No. 850-204) caused the greatest number of pores on cleaned magnesium alloy surfaces (Fig. 8a); a reduced number of pores formed when the Teflon green primer was applied to chrome-pickled magnesium alloy surfaces (Fig. 8b). Teflon one-coat green enamel (du Pont No. 851-204) applied to the magnesium alloy surface showed a reduction in the number of pores (Fig. 8c); however, only when the Teflon one-coat green enamel was applied to the chrome-pickled magnesium alloy was the complete absence

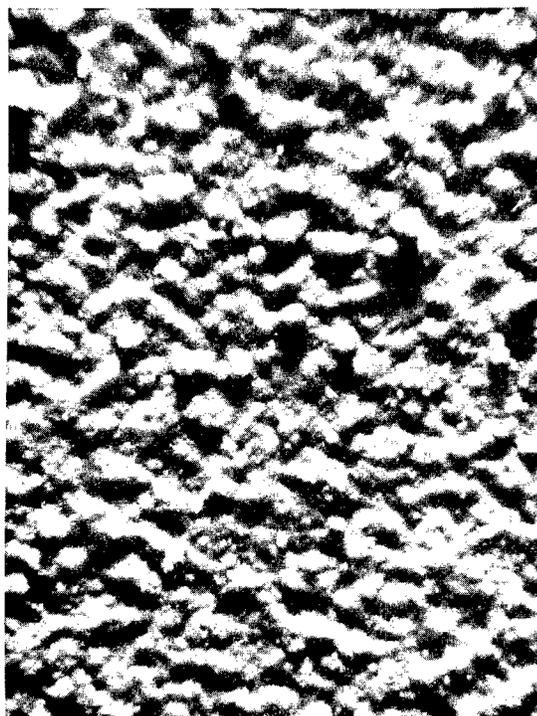
of pores noted (Fig. 8d). The thick Teflon coatings on magnesium alloys (Fig. 9a) cracked more profusely than the thick coatings on the aluminum alloys (Fig. 7c); however, the relative excess thicknesses of the coatings were not ascertained. Where the cracks developed, bare metal could be observed under higher magnification (Fig. 9b).

Pore formation in Teflon coatings applied to the magnesium specimens is attributed to the low pH (~1.5) of the Teflon one-coat enamel and primer and to the high chemical reactivity of the magnesium. During the spraying procedure and during the flame fusion, hydrogen gas is produced at the metal/Teflon interface by the reaction of the highly acidic Teflon formulations with the magnesium alloy. Unfortunately, the Teflon clear finish (pH 4.5) did not produce a contrasting coating for observation of pores under magnification of the coated specimens; a reduced number of pores would be expected. Chrome pickling of surfaces of the magnesium alloy reduces the rate of attack by forming a protective film which prevents the acidic attack by the Teflon formulation. The less reactive aluminum alloys do not react under the conditions used; if any gas evolution does occur, it does not cause a deteriorative effect on the Teflon coating.

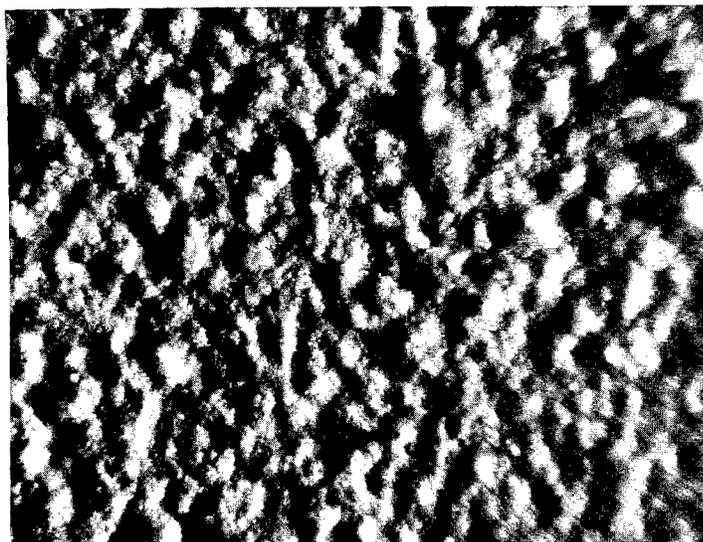
A method to reduce the reactivity of the magnesium alloy toward the acidic Teflon one-coat enamel is suggested by the reduction of the number of pores in the Teflon coating of a specimen with a chrome-pickled surface; a pretreatment film on the magnesium alloy surface can prevent the rapid attack by the acid formulations as well as act as a binder between the metal surface and the protective coating (Fig. 10). The pretreatment film which was chosen for study was selected because of its chemical nature when combined with magnesium ions; the magnesium methacrylate complexes exhibited non-conducting



(a) Before fusion

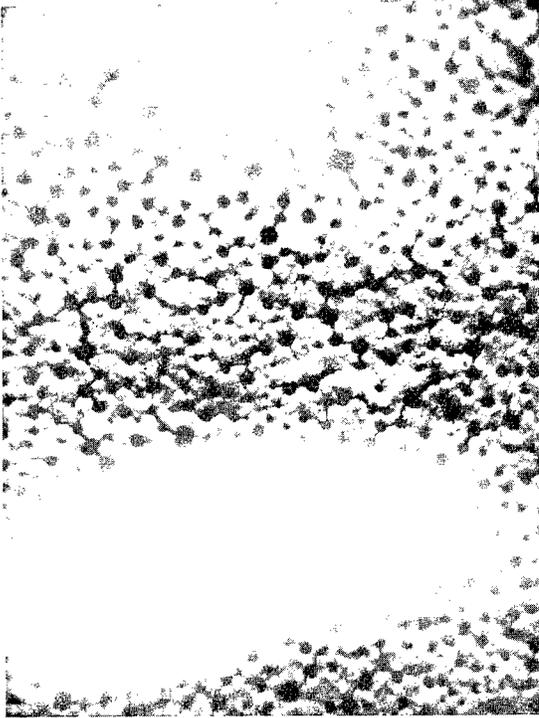


(b) Flame fused

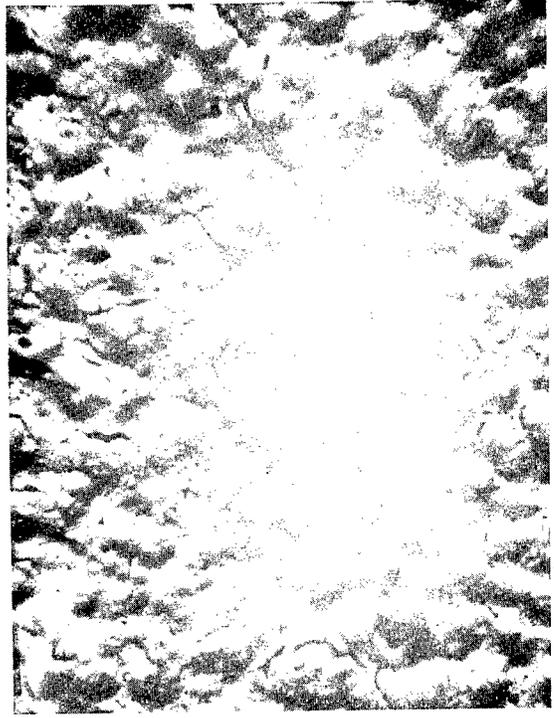


(c) Flame-fused thick coating

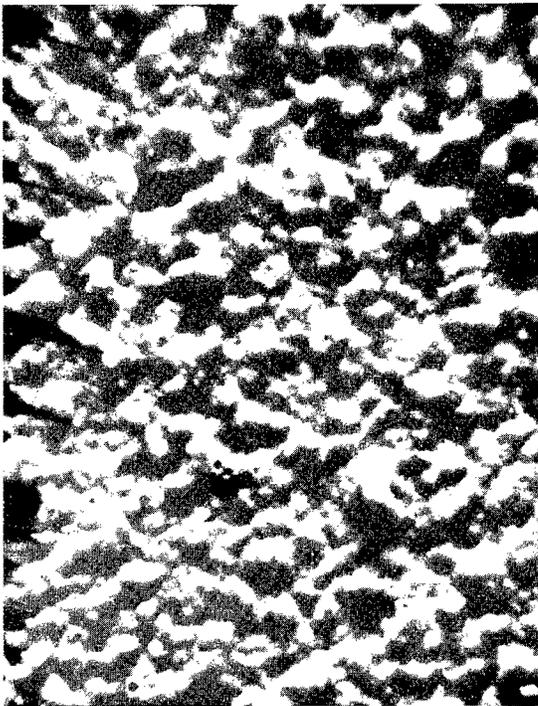
Fig. 7 - Typical Teflon surfaces on aluminum alloy 2024-T3 (original magnification, 90X)



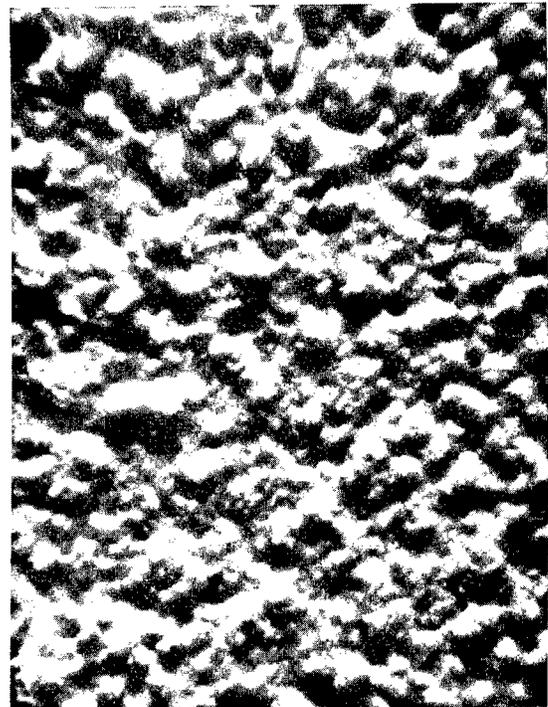
(a) Fused Teflon green primer  
(du Pont No. 850-204)



(b) Fused Teflon green primer (du Pont  
No. 850-204), chrome-pickled alloy

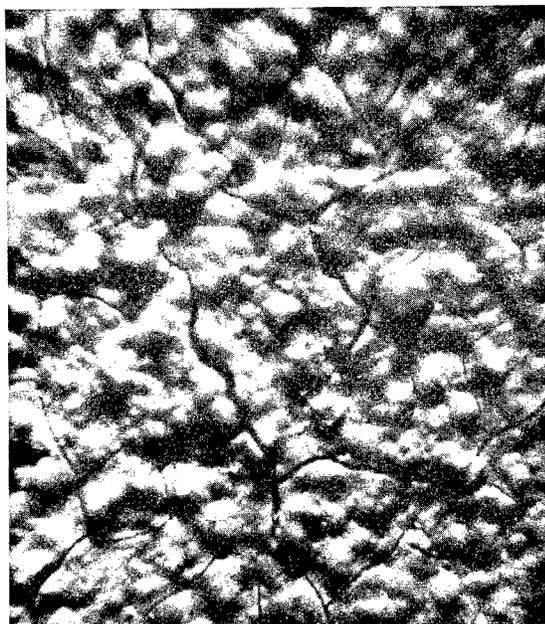


(c) Fused Teflon one-coat enamel  
(du Pont No. 851-204)



(d) Unfused Teflon one-coat enamel (du Pont  
No. 851-204), chrome-pickled alloy

Fig. 8 - Typical Teflon surfaces on magnesium alloy AZ31B-H24  
(original magnification, 90X)



(a) Original magnification, 90X



(b) Original magnification, 189X

Fig. 9 - Fused Teflon enamel on magnesium alloy AZ31B-H24, chrome pickled, thick coating

properties and were water insoluble (12). The apparent polymerization of glacial methacrylic acid during its redox reaction with the magnesium alloy causes a gummy material to form on the surface of the magnesium; a large poly(anion) formation is indicated according to the following reaction:

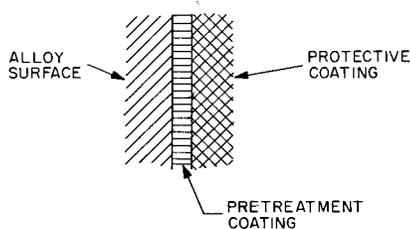
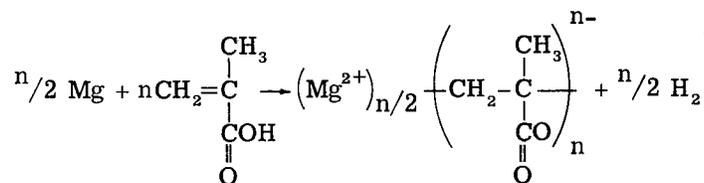
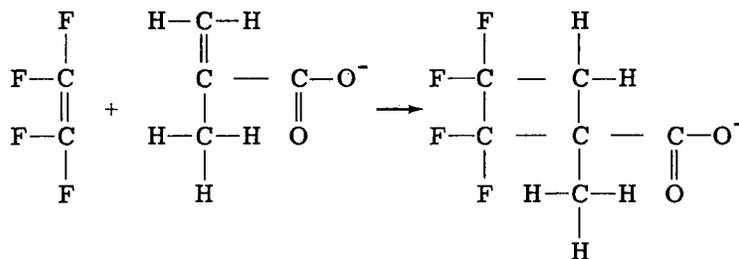
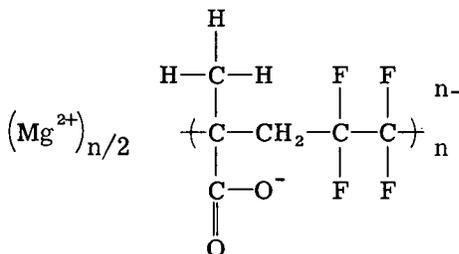


Fig. 10 - Pretreatment and protective coating

The redox polymerization of methacrylic acid (13) and acrylic esters (14) has been reported. The methacrylic pretreatment film may also act as a scavenger for tetrafluoroethylene gas formed during the fusion of the Teflon. A reaction similar to one reported by Barrick and Cramer (15,16) is postulated. A cycloalkyl structure could be formed by the reaction between tetrafluoroethylene and monomeric methacrylate according to the following reaction.



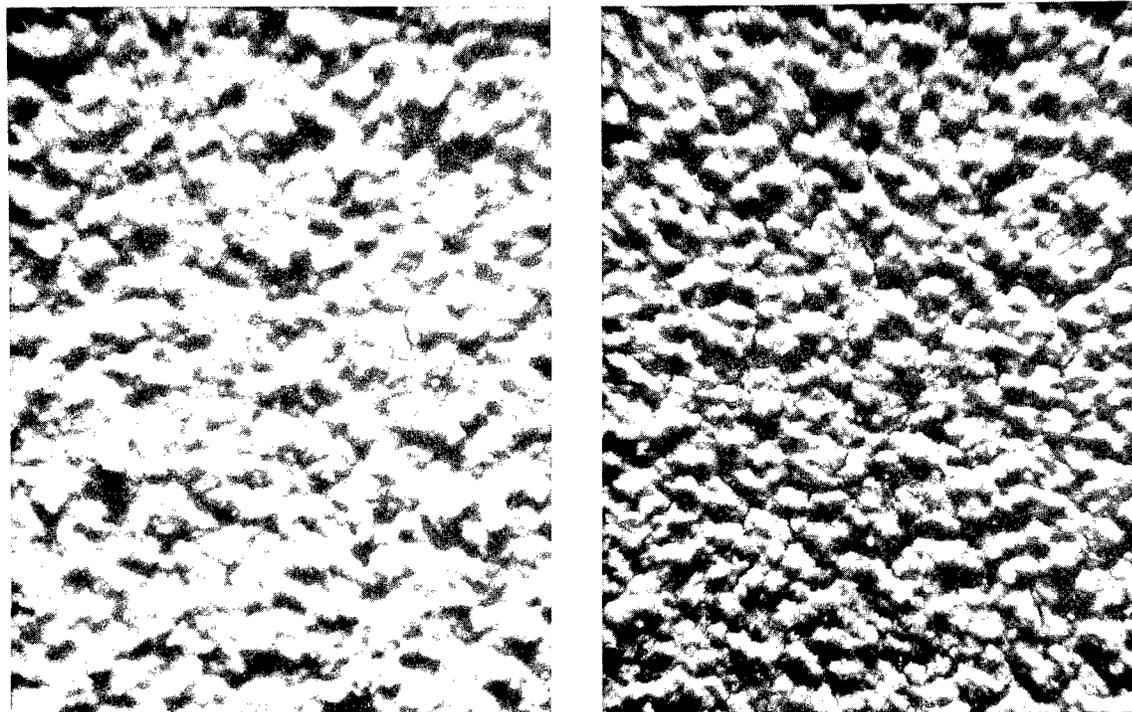
The most reactive systems reported to react with tetrafluoroethylene are 1,3-butadiene, acrylonitrile, methyl methacrylate, and styrene. Copolymer formation may also be postulated between the pyrolysis products, tetrafluoroethylene and monomer methacrylate; the postulated copolymer



would add to the adhesion of the coating to the metal surface.

Magnesium alloy AZ31B-H24 which was pretreated with glacial methacrylic acid showed an absence of pores when Teflon one-coat enamel was applied; however, small cracks were present in the coating (Fig. 11a). After fusion, the Teflon surface was characterized by a profusion of small cracks and a clumping together of the coating (Fig. 11b). Aluminum alloy 2024-T3 pretreated with glacial methacrylic acid showed little difference in the Teflon coating before (Fig. 12a) or after fusion (Fig. 12b). The hair-line cracks observed in the fused Teflon coating were less numerous and of finer dimensions than cracks observed in the surface of a thick Teflon coating (Fig. 7c). Although the pores are believed to be caused by the liberation of hydrogen gas by the reaction of aluminum and magnesium alloys with the acidic Teflon formulations, the occurrence of the hair-line cracks and other larger cracks after flame fusion of the Teflon may be caused in part by volatile gaseous products from the thermal decomposition of the Teflon. The thermal degradation of the bulk poly(tetrafluoroethylene) has been reported (17-22); however, the thermal decomposition products obtained from thin Teflon films on various substrates have not yet been examined.

By use of mass spectral data, thermal decomposition products from thin films of Teflon on magnesium alloy AZ31B-H24 and glass were compared with the decomposition products from bulk Teflon. The Teflon one-coat green enamel was used on glass and magnesium surfaces, and the volatile products evolved at 500°C ( $\pm 50^\circ\text{C}$ ) were compared with the volatile products which were obtained under the same conditions from a piece of Teflon sheet (Table 1, Figs. 13, 14). The data in Fig. 13 show that the volatile decomposition products are the same, within experimental error, regardless of the substrate with which the Teflon is in contact. Furthermore, the Teflon sheet and the reported mass spectrum of tetrafluoroethylene (20) are the same as the thin films of Teflon in contact with magnesium alloy AZ31B-H24 and Pyrex glass. Although the Teflon on the latter two surfaces became black at the experimental degradation temperature, the sheet polymer



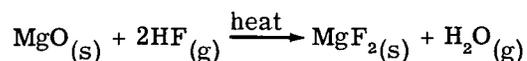
(a) Unfused Teflon surface

(b) Fused Teflon surface

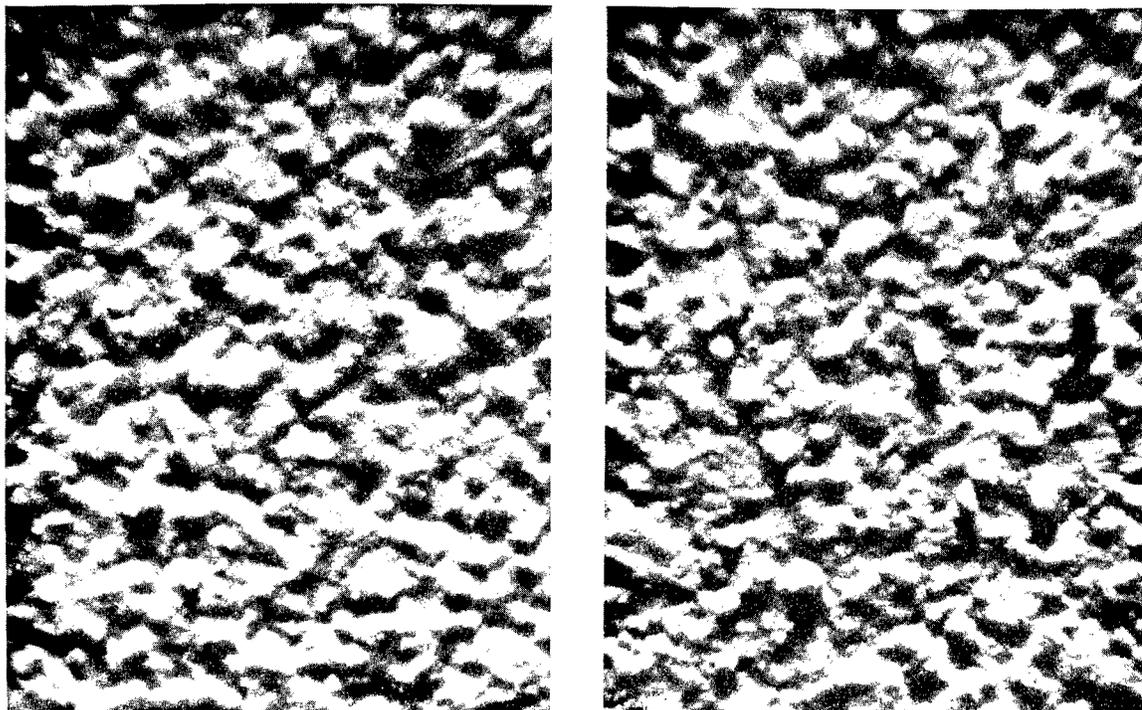
Fig. 11 - Teflon one-coat enamel (du Pont No. 851-204) on magnesium alloy AZ31B-H24, pretreated with glacial methacrylic acid (original magnification, 90X)

was still white but the quantity of polymer was considerably reduced by volatilization. The volatile products not related to the Teflon decomposition (Fig. 14) show a larger amount of CO and N<sub>2</sub> from the sheet Teflon than the thin films on the substrates. This result may be explained by absorbed gases (CO<sub>2</sub>, N<sub>2</sub>, and O<sub>2</sub>) or occluded gases in the manufactured Teflon sheet. Within experimental error, the abundances of other ions appear to be independent of presence or absence of the Teflon film and of the substrate in contact with the Teflon.

Two explanations for the similar mass spectral data may be considered. First, the majority of the volatile products originate from the Teflon surface and not from the interface between the Teflon and substrate. Second, any reaction between the decomposition products and the substrate may be so slight that the differences are not observed in the mass spectral data. Under the conditions studied, tetrafluoroethylene is the major volatile thermal-decomposition product from Teflon; the products are the same at the air/Teflon interfaces regardless of the substrate. The evolution of the tetrafluoroethylene could be a contributing source of the cracks in the fused Teflon coatings on the aluminum and magnesium alloys. The formation of magnesium fluoride at the Teflon/magnesium alloy interface, according to the reaction



is not supported by the mass spectral data; the small amount of F<sup>+</sup> (Fig. 13) and the same relative abundances, regardless of substrate, may be cited as evidence.



(a) Unfused Teflon surface

(b) Fused Teflon surface

Fig. 12 - Teflon one-coat enamel (du Pont No. 851-204) on aluminum alloy 2024-T3, pretreated with glacial methacrylic acid (original magnification, 90X)

In general, when the specimens were placed in the salt solution and immediately withdrawn, only a few droplets of water remained on the Teflon surface. After a day or two, this phenomenon was no longer observed for that portion of the sample that was totally immersed. Evidently, during immersion, water had penetrated the Teflon coating. Several samples, however, retained this nonwetting property for the duration of the test period, but no difference could be detected under microscopic examination between a wetting and non-wetting surface within the same series of test specimens.

Magnesium alloy AZ31B-H24, which was coated with fused Teflon green primer (du Pont 850-204), was immediately attacked by sodium chloride solution; vigorous gassing and accumulation of white solid on the bottom of the jar were noted shortly after immersion in the test solution. Within 10 days two of the three coated specimens of AZ31B-H24 were completely consumed leaving the green Teflon coating, white solid, and a small amount of black solid. The remaining specimen (Fig. 15a) was badly pitted and approximately one half of the immersed area consumed; the area above the immersion line was extensively corroded also. The uncoated magnesium alloy AZ31B-H24 specimens (Fig. 15c) were more severely attacked than the chrome-pickled specimens (Fig. 15b) but less corroded than the fused Teflon-coated specimens. These results are related to the excessive number of pores (Fig. 8a) in the Teflon-coated unpickled specimens compared with the Teflon-coated chrome-pickled specimens (Fig. 8b). The large number of pores act to increase the number of local cells on the alloy and cause an increase in the corrosion rate. The use of the highly porous coating to increase the efficiency of magnesium used as sacrificial anodes or as battery electrodes should be investigated further.

Table 1  
Relative Abundances (%)\*

Ion m/e	Polymer-Derived Ions										Non-Polymer-Derived Ions				
	C	F	CF	CF <sub>2</sub>	CF <sub>3</sub>	C <sub>2</sub> F <sub>3</sub>	C <sub>2</sub> F <sub>4</sub>	C <sub>2</sub> F <sub>5</sub>	C <sub>3</sub> F <sub>6</sub>	O	OH	H <sub>2</sub> O	N <sub>2</sub> :CO	CO <sub>2</sub>	
	12	19	31	50	69	81	100	131	150	16	17	18	28	44	
Teflon on Magnesium Alloy AZ31B-H24	10.3	1.9	100	24.1	10.6	54.6	35.0	4.26	1.42	8.1	24.7	100	45.8	76.1	
Teflon on Glass Beads	9.2	1.8	100	25.9	7.92	56.9	38.1	3.66	1.22	10.5	26.0	100	59.8	71.0	
Teflon Sheet	6.4	1.2	100	25.4	7.0	58.4	38.0	3.50	0.88	12.8	19.5	61.5	100	33.3	
Tetrafluoroethylene†	12.6	2.77	100	29.5	2.83	63.1	33.8	-	-	-	-	-	-	-	
Uncoated Magnesium Alloy AZ31B-H24	-	-	-	-	-	-	-	-	-	7.9	25.3	100	23.4	30.5	
Sample Tube	-	-	-	-	-	-	-	-	-	6.6	24.0	100	( † )	21.8	

\*Based on the largest ion current observed set at 100%

†Ref. 20.

(†) Unable to calculate peak height

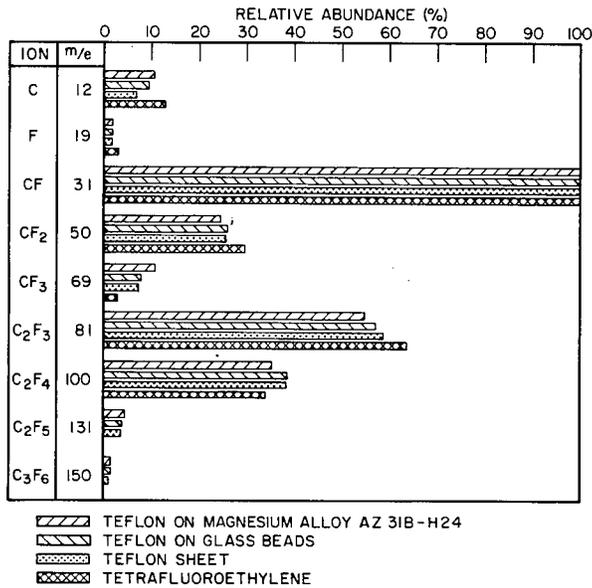
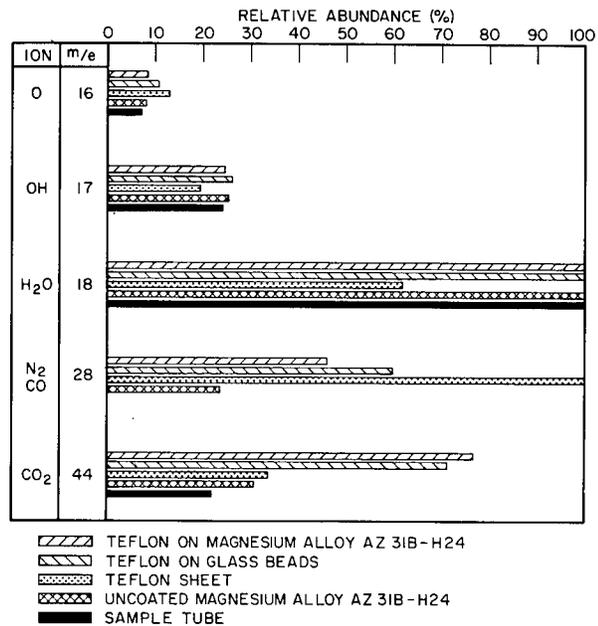


Fig. 13 - Mass spectral data for Teflon-related ions

Fig. 14 - Mass spectral data for products not related to Teflon decomposition

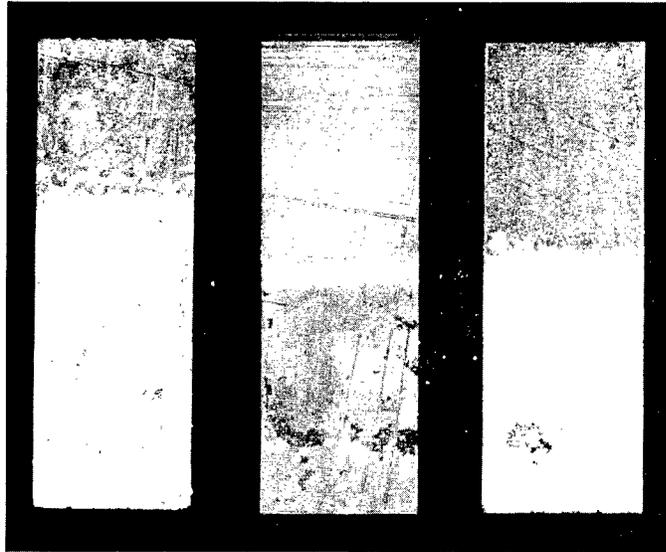


Clad aluminum alloy 2024-T3 specimens which were coated with the fused Teflon primer showed only slight attack after five months of immersion in 3% sodium chloride (Fig. 16a); the decreased reactivity of the aluminum alloy and the more uniform nature of the Teflon coating (Fig. 16b) contribute to these results.

The magnesium alloy AZ31B-H24, coated with a fused Teflon clear finish (du Pont No. 852-201), was immediately attacked when the specimens were brought in contact with 3% sodium chloride solution. Using the spherical-joint method (Fig. 3) the test specimens



(a) Fused green primer on AZ31B-H24

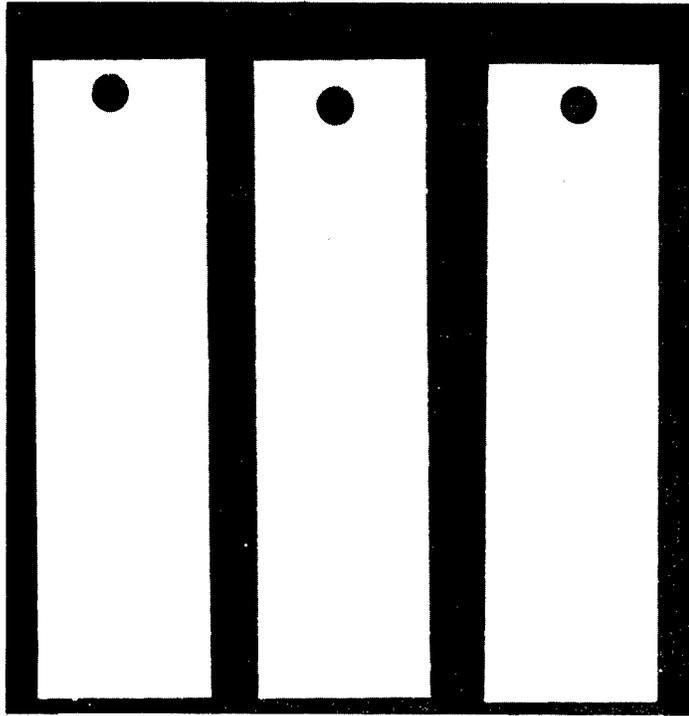


(b) AZ31B-H24 with chrome pickle



(c) AZ31B-H24 without chrome pickle

Fig. 15 - Magnesium alloy AZ31B-H24; specimens after 1 month immersion in 3% sodium chloride solution



(a) Specimens after 5 months of immersion in 3% sodium chloride solution



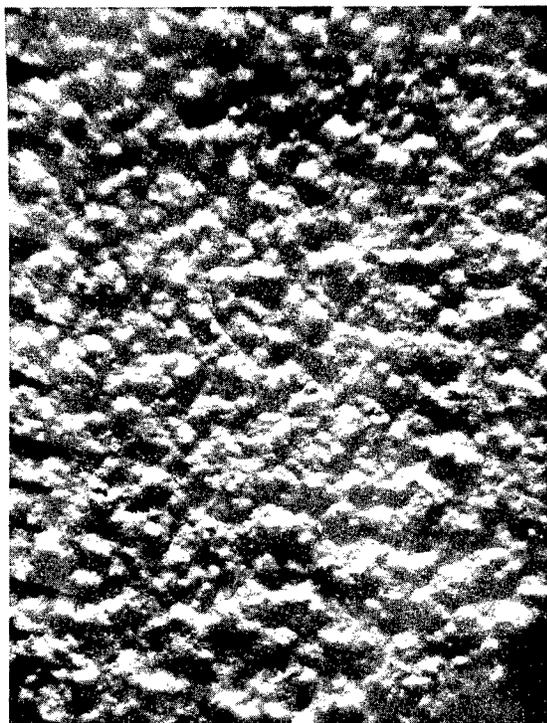
(b) Specimen surface before testing (original magnification, 90X)

Fig. 16 - Clad aluminum alloy 2024-T3 coated with fused Teflon resin green primer (du Pont No. 850-204)

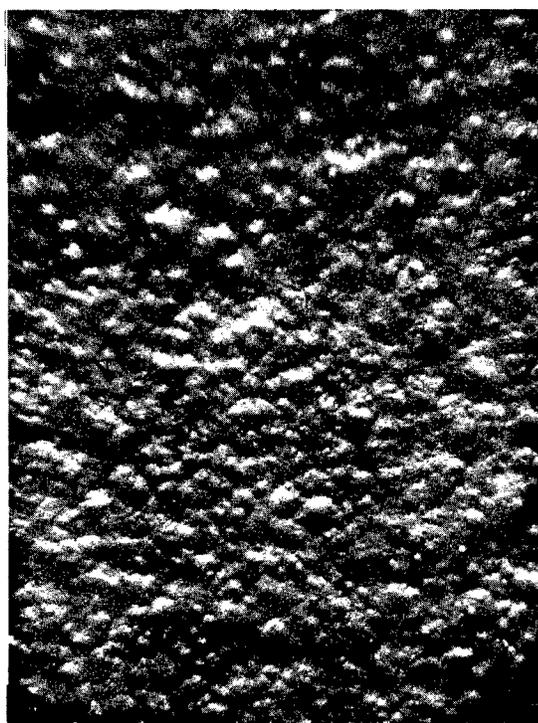
showed extensive corrosion after eight days, at which time the salt solution penetrated through the specimen (Fig. 25f). The clear finish was easily removed by a thumbnail scratch test, and under the microscope it was noted to be highly crazed. Further use of this coating was abandoned.

Coating magnesium alloy specimens with only a thin film of glacial methacrylic acid did not change the amount of corrosion with respect to untreated control specimens. As a pretreatment before the application of a Teflon formulation, the methacrylic acid treatment on magnesium alloy specimens did show protection against pore formation and cracking of the fused Teflon coatings; the one-coat enamel (du Pont 851-204) was found to form the best coating. A good coating of fused Teflon one-coat enamel on magnesium alloy specimens with or without the methacrylic acid pretreatment showed only a few cracks in the coatings (Fig. 17); the hair-line cracks which were observed on the surfaces were different than the pores (Fig. 8a) and cracks (Fig. 8b) observed when fused Teflon primer (du Pont 850-204) was applied to magnesium alloy without chrome pickling and with chrome pickling, respectively.

After a 14-day immersion in 3% sodium chloride solution, the corrosion of magnesium alloy AZ31B-H24 specimens pretreated with glacial methacrylic acid and coated with fused Teflon one-coat green enamel was compared with the corrosion of chrome-pickled specimens coated with Teflon one-coat enamel and of uncoated control specimens. The control specimens were only slightly attacked; the greatest amount of corrosion was noted on the test specimen without the chrome pickle (Fig. 18a). The specimens having fused Teflon

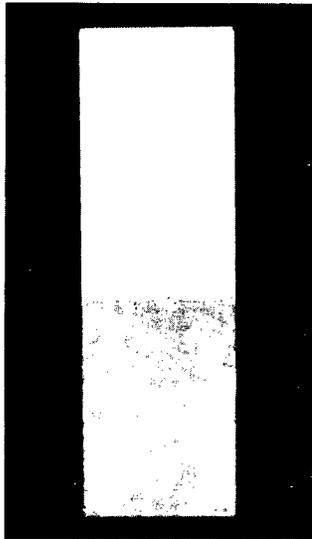


(a) Specimen pretreated with methacrylic acid



(b) Specimen without pretreatment

Fig. 17 - Fused Teflon one-coat enamel (du Pont 851-204) on magnesium alloy AZ31B-H24 (original magnification, 90X)



(a) Control specimens,  
bare alloy



(b) Control specimen,  
chrome pickled



(c) Chrome-pickled specimens  
coated with fused Teflon



(d) Specimens coated with fused  
Teflon only

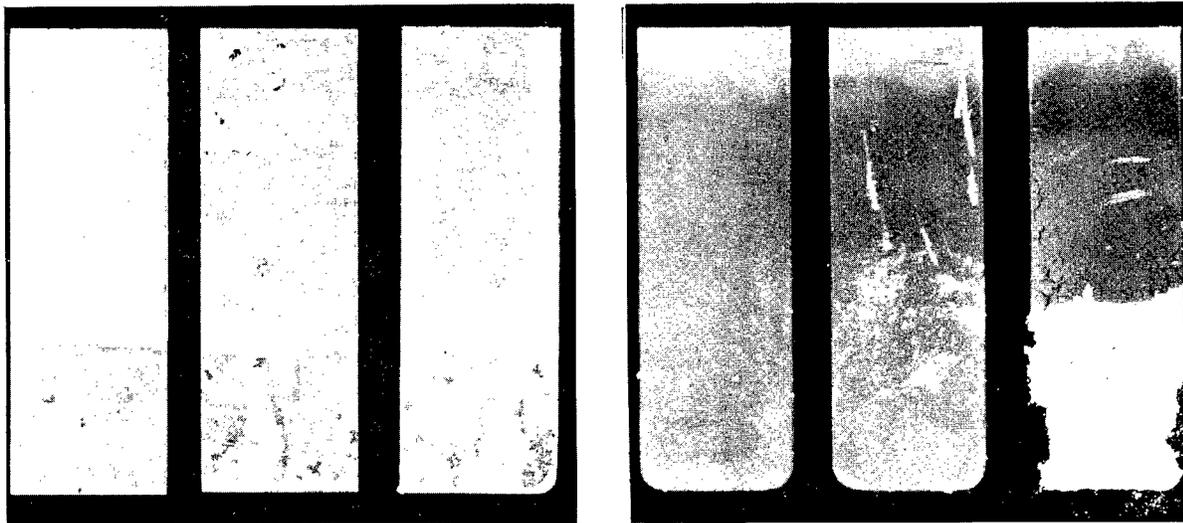


(e) Specimens pretreated with methacrylic acid  
and coated with fused Teflon

Fig. 18 - Magnesium alloy AZ31B-H24 coated with Teflon one-coat green enamel (du Pont 851-204) after 10 days of immersion in 3% sodium chloride solution

over a chrome-pickled surface (Fig. 18c) were more resistant to corrosion than the specimens without the chrome pickle (Fig. 18d); however, one specimen showed considerably more attack along the edges than the second specimen of the series. Specimens having methacrylic acid pretreatment prior to the application of the Teflon showed (Fig. 18e) greater amounts of corrosion than the chrome-pickled Teflon-coated specimens, but approximately the same amount of corrosion as the unpickled samples. Although the presence of the methacrylate coating appears to protect the magnesium alloy during the application of the acidic Teflon formulations, the pretreatment does not significantly increase the corrosion resistance of the Teflon-coated magnesium alloy.

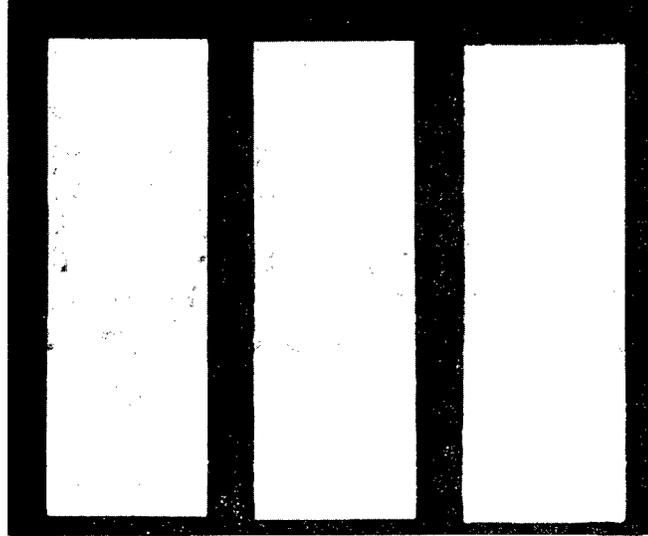
Magnesium alloy specimens with and without chrome pickle and coated with Teflon one-coat enamel were rerun to determine the reproducibility of the above results. The uncoated chrome-pickled specimens (Fig. 19a) showed far more corrosion resistance than the uncoated unpickled specimens (Fig. 20a). The Teflon-coated unpickled specimens were more readily corroded than the uncoated unpickled specimens (Fig. 20b versus Fig. 20a); however, the Teflon-coated chrome-pickled specimens (Fig. 19b) showed less corrosion than any other magnesium alloy specimens tested. The corrosion resistance depended on a properly applied and fused Teflon coating. For example, Specimen No. 351, Fig. 19b, was deliberately prepared with an extra thick Teflon coating and over-heated during the fusion process. A large amount of cracking was noted on the surface of the test specimen before immersing the specimen in the salt solution. After 19 days in 3% sodium chloride solution, the immersed portion of the improperly coated sample was extensively corroded; the other two specimens of the series showed evidence of slight corrosion compared with the chrome-pickled control specimens. The Teflon-coated chrome-pickled specimens which showed the greatest corrosion resistance during the test period were not wetted by the test solution; the nonwetting property of the Teflon coating appears related to the quality of the fused Teflon coating.



(a) Uncoated specimens,  
26 days of immersion

(b) Flame-fused Teflon-coated specimens,  
19 days of immersion

Fig. 19 - Magnesium alloy AZ31B-H24 chrome-pickled specimens after immersion in 3% sodium chloride solution



(a) Uncoated specimens, 7 days  
of immersion



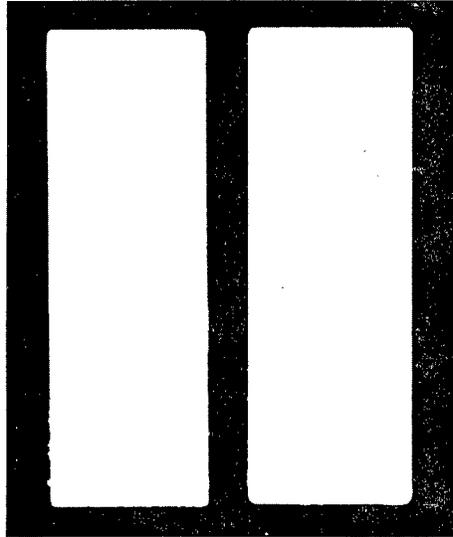
(b) Flame-fused Teflon-coated specimens,  
3 days of immersion

Fig. 20 - Magnesium alloy AZ31B-H24 after  
immersion in 3% sodium chloride solution

The effects of the methacrylic acid pretreatment applied to aluminum alloy 2024-T3 specimens were also investigated. Control specimens of clad and unclad aluminum alloy were compared with flame-fused Teflon-coated clad, unclad, and methacrylic acid-pretreated specimens. After 10 days and 30 days of immersion in 3% sodium chloride solution, none of the Teflon-coated test specimens showed evidence of corrosion; however, after two months of immersion some edge corrosion was noted on the Teflon-coated clad aluminum alloy specimens (Fig. 21a). To a lesser extent some edge corrosion was observed on the methacrylic acid-pretreated samples (Fig. 21b), but no corrosion was noted on the unclad Teflon-coated specimens (Fig. 21c). The clad and unclad control specimens were extensively corroded during the same period (Fig. 21d and e). All of the test specimens exhibited the formation of a small amount of white powdery solid on the areas above the immersion line. The white powdery material easily rubbed off and no evidence of pitting or rupture of the Teflon coating was noted under the deposits. The amount of corrosion on the upper areas of the control specimens was quite large with many pitted regions. The test results indicated that the fused Teflon coating does provide corrosion protection to the aluminum alloy 2024-T3; the clad or methacrylate undercoating does not enhance the corrosion protection.

Aluminum alloys 2024-T3, 5052-H32, and 6061-T6 were used to compare the effect of different tempers on the corrosion resistance of specimens coated with fused Teflon. Teflon-coated and uncoated specimens were tested by the immersion method over a 26-day test period (Figs. 22, 23, 24). The corrosion of the uncoated control specimens appeared to be confined to pitting and edge corrosion (Figs. 22a, 23a, and 24a). Uncoated 2024-T3 specimens showed the greatest amount of corrosion, and uncoated 5052-H32 specimens showed the least amount of corrosion. Although the corrosion was slight for uncoated 5052-H32 and 6061-T6 specimens tested, the Teflon-coated specimens after exposure showed an enhanced corrosion resistance. At first, the Teflon-coated specimens were not wetted by the salt solution; however, after 7 days the surfaces were wetted and a small quantity of clear crystalline granules appeared on the submerged Teflon surfaces. The Teflon-coated 2024-T3 specimens showed evidence of corrosion on only two test specimens after 10 days. The third specimen, which was not wetted by the salt solution until the 18th day of immersion (Sample 337, Fig. 22b), showed the least corrosion of the 2024-T3 specimens. For the series of aluminum alloys tested, Teflon-coated 6061-T6 specimens showed the greatest corrosion.

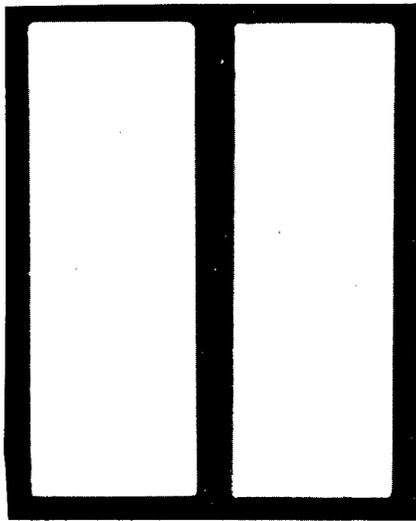
The use of the spherical joints, as shown in Fig. 3, afforded a simpler procedure than the immersion method for observing the effect of 3% sodium chloride solution on the test specimens. Figure 25 shows a few examples of the test results which were in accord with the earlier findings of immersion tests. Using different areas on the same specimen also showed similar results. By treating half of the specimen in a different manner, two tests could be run simultaneously (Fig. 25a) or a different corroding solution run at a later time. An interesting feature of the method is that penetration of the corroding solution through the specimens provides an observable phenomenon to end the test and compare rates of corrosion. The two test areas used on Sample 36, Fig. 25c, were penetrated by the salt solution in 33 days for the lower area and 34 days for the upper area. In general, the rate of corrosion using the spherical-joint apparatus showed a decrease as compared with the immersion test, although the final results were the same. The decrease in the corrosion rate is explained by the pH of the salt solution reaching its limiting value, pH 10.3 to 10.7, at a faster rate for the spherical-joint method than for the immersion method due to the small volume of solution (7 ml) used in the spherical joint. The inability to agitate the solutions is also a factor. The white material collected in the arms of the spherical joints was analyzed for magnesium using EDTA reagent and Erio-T indicator. The percentage magnesium varied considerably, but was approximately 38%, indicating a possible mixture of magnesium oxide and magnesium carbonate.



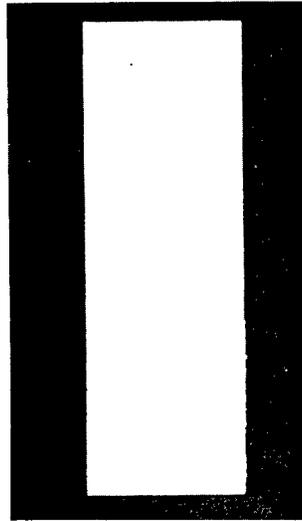
(a) Clad specimens coated with fused Teflon



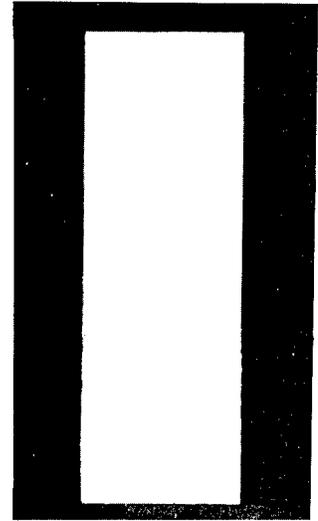
(b) Specimens pretreated with methacrylic acid coated with fused Teflon



(c) Unclad specimens coated with fused Teflon

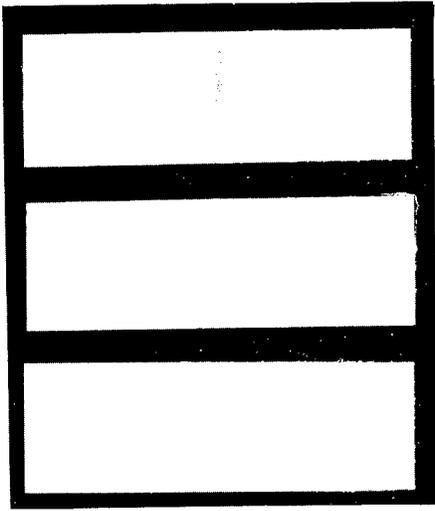


(d) Control specimen, unclad

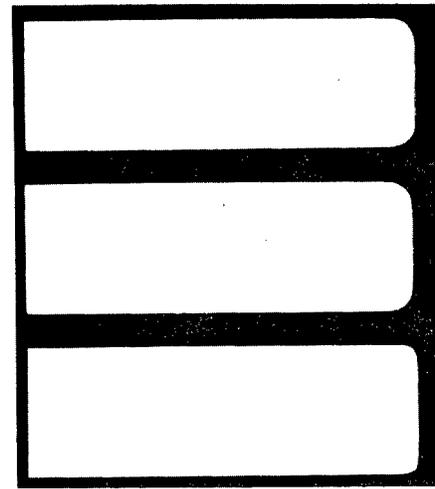


(e) Control specimen, clad

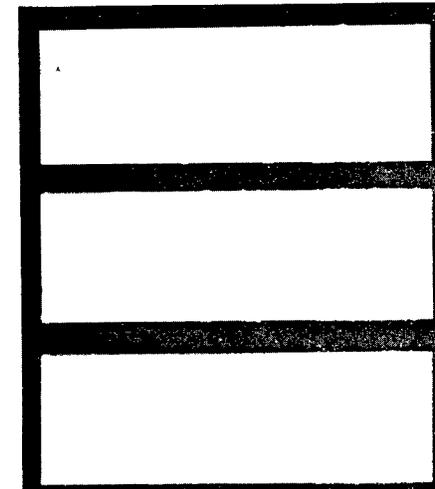
Fig. 21 - Aluminum alloy 2024-T3 coated with fused Teflon one-coat enamel (du Pont 851-204) after 60 days of immersion in 3% sodium chloride solution



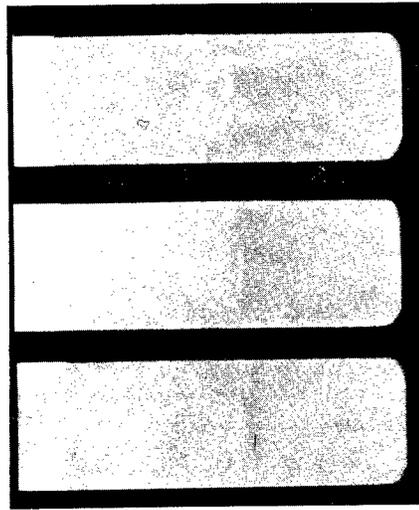
(a) Uncoated specimens



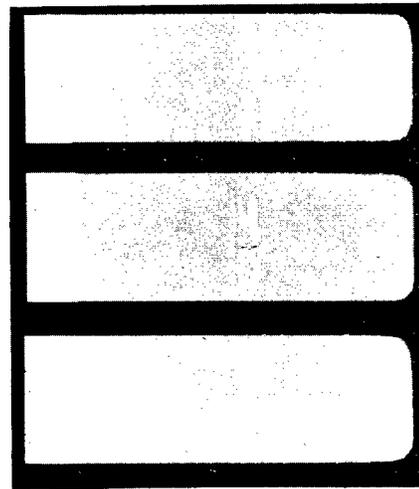
(a) Uncoated specimens



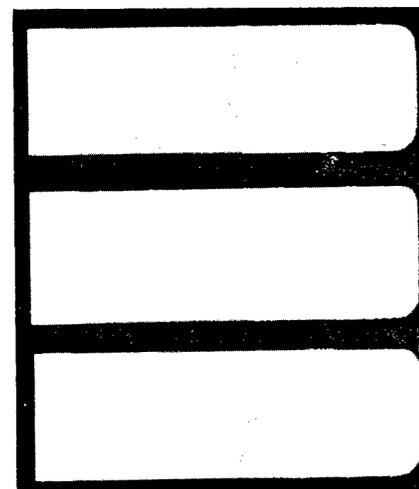
(a) Uncoated specimens



(b) Flame-fused Teflon one-coat enamel coated specimens



(b) Flame-fused Teflon-coated specimens

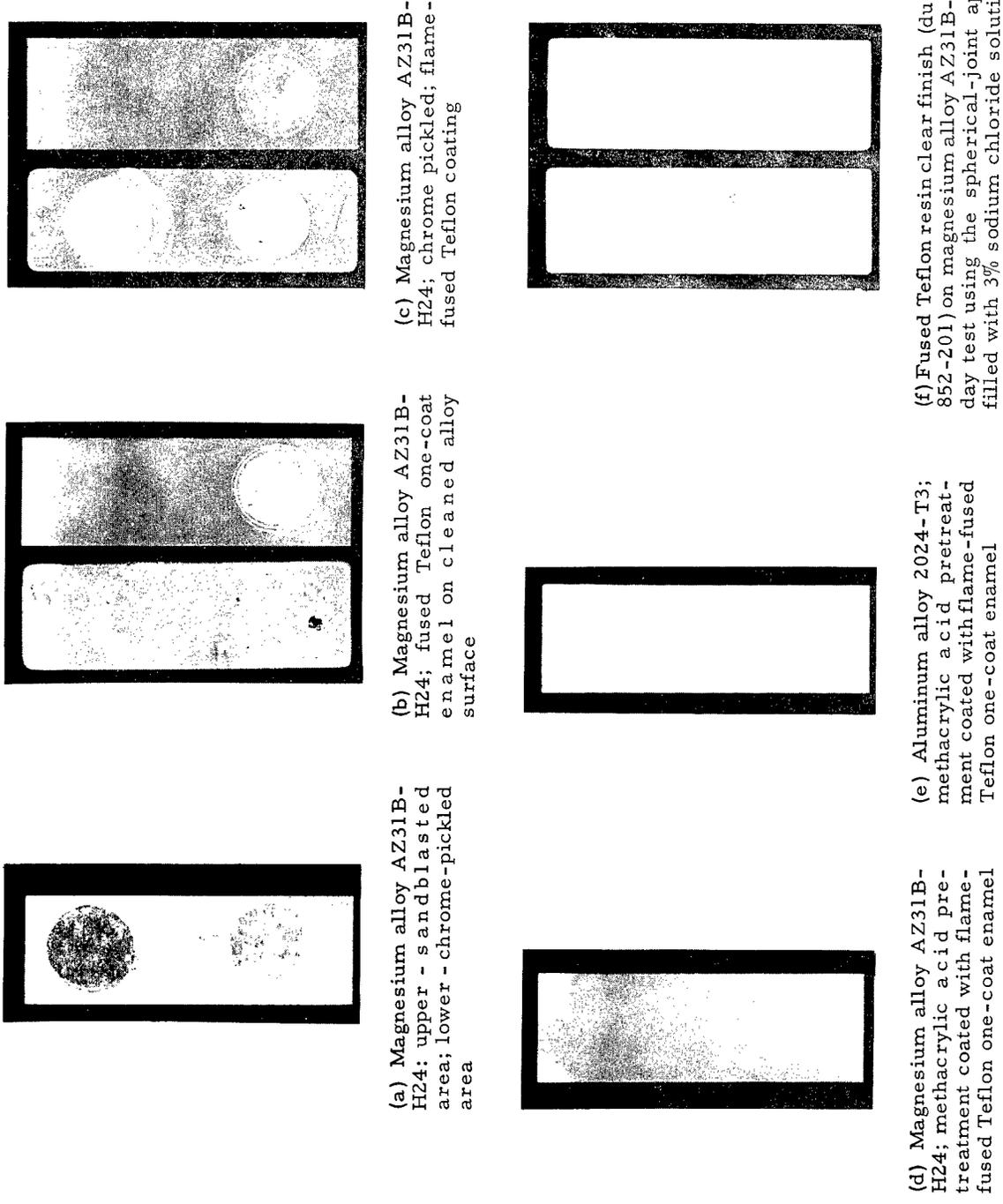


(b) Flame-fused Teflon-coated specimens

Fig. 24 - Aluminum alloy 6061-T6 after 26 days of immersion in 3% sodium chloride solution

Fig. 23 - Aluminum alloy 5052-H32 after 26 days of immersion in 3% sodium chloride solution

Fig. 22 - Aluminum alloy 2024-T3 specimens after 26 days of immersion in 3% sodium chloride solution



(a) Magnesium alloy AZ31B-H24; upper - sandblasted area; lower - chrome-pickled area

(b) Magnesium alloy AZ31B-H24; fused Teflon one-coat enamel on cleaned alloy surface

(c) Magnesium alloy AZ31B-H24; chrome pickled; flame-fused Teflon coating

(d) Fused Teflon resin clear finish (du Pont No. 852-201) on magnesium alloy AZ31B-H24; 10-day test using the spherical-joint apparatus filled with 3% sodium chloride solution

(e) Aluminum alloy 2024-T3; methacrylic acid pretreatment coated with flame-fused Teflon one-coat enamel

(f) Magnesium alloy AZ31B-H24; methacrylic acid pretreatment coated with flame-fused Teflon one-coat enamel

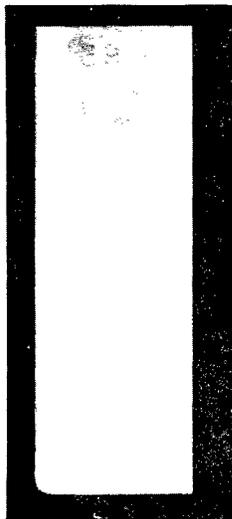
Fig. 25 - Results of spherical-joint method of testing coated and uncoated alloy specimens

A more satisfactory corrosion test method than the immersion or spherical-joint method was the salt droplet test method (9,10) (Fig. 5). The method, which will be used in future corrosion tests, was found to give the same results as the other two test methods; however, the method appears to be less drastic and more closely aligned to the type of spray action that would be encountered by aircraft components exposed to a marine atmosphere. More pitting than edge corrosion was observed in the samples tested (Fig. 26).

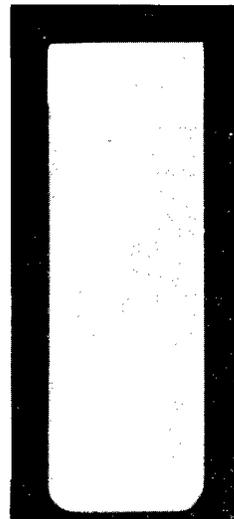


(a) Uncoated AZ31B-H24 controls

(b) AZ31B-H24, chrome pickled



(c) AZ31B-H24 pretreated with methacrylic acid; flame-fused Teflon one-coat enamel



(d) 2024-T3 pretreated with methacrylic acid; flame-fused Teflon one-coat enamel

Fig. 26 - Teflon-coated and uncoated specimens tested by the salt droplet method; test period - 14 days

The change in solution resistance as a measure of coating porosity was investigated by use of the spherical joint test apparatus described in Fig. 4. When measured in the apparatus without a coated panel between the spherical joints, the resistance of a dilute sodium chloride solution used in preliminary tests was  $2.00 \times 10^4$  ohms at  $25 \pm 0.01^\circ\text{C}$ . An aluminum 2024-T3 specimen coated with fused Teflon was inserted between the electrodes, and a reading of  $6.00 \times 10^4$  ohms was initially obtained; the value slowly decreased to  $3.00 \times 10^4$  ohms in approximately 1 hour. Further investigation of the procedure is needed using standard sodium chloride solution and using various surface coatings of known condition. The procedure may provide a test that will expeditiously reveal the continuity of a coating on a test specimen.

## CONCLUSIONS AND FUTURE WORK

The flame method has been used successfully in the fusing of Teflon on a metal surface; the smaller changes in mechanical properties than produced by oven heating (1) suggest a concentrated heat method as a means for thermally curing coatings on structural metals. Although the current investigation was not meant to study a protective system of Teflon, per se, fused Teflon-coated test specimens of magnesium and aluminum alloys were evaluated for corrosion resistance by 3% sodium chloride solution to determine some of the problems to be encountered in future investigations.

Although pore formation on magnesium alloys is attributed to the highly acidic nature of the Teflon formulations used, the thermal decomposition of Teflon, as studied by use of the mass spectrometer, suggests that the formation of cracks in the surface of the Teflon coatings is caused in part by the evolution of tetrafluoroethylene during flame fusion. The possibility of hydrogen fluoride formation was refuted by the mass spectral data, although the air/Teflon interface rather than Teflon/substrate interface was the prime observable decomposing surface. Therefore, magnesium fluoride formation appears unlikely at the Teflon/magnesium alloy interface. The use of poly(vinyl fluoride) under the same conditions as reported in this investigation may produce hydrogen fluoride for magnesium fluoride formation at the magnesium alloy surface and will be studied.

The Teflon resin green primer coating (No. 850-204) caused a considerable amount of pore formation when applied to the magnesium alloys. The high acidity and low solid content (39% solid by weight) of the formulation are considered the cause of the excessive pore formation; therefore, this formulation was not extensively investigated. The Teflon resin clear finish (No. 852-201) was not found to be applicable to the flame fusion method because of the absence of a visible color change on fusion and because of the poor adhesion to the alloys. The Teflon one-coat green enamel (48% solids by weight) was found to be the best formulation used; it was compatible with both magnesium and aluminum alloys. Care must be taken to prevent an excessively thick Teflon coating from being applied to the test specimens; such thick films exhibited mudlike cracking after flame fusion.

An important factor in the ability of Teflon coatings to decrease the corrosion of magnesium alloy AZ31B-H24 was found to be the continuity of the fused Teflon coating; in this respect, the Teflon resin one-coat enamel (du Pont 851-204) gave the best results. When applied over a chrome-pickled alloy specimen, the Teflon coat greatly reduced the rate of corrosion as compared with uncoated control specimens. Methacrylic acid-pretreated specimens and untreated alloy specimens showed accelerated corrosion when the fused Teflon coating was applied. The accelerated corrosion rate could be of value in the use of magnesium for batteries and for sacrificial anodes where an increase in the efficiency of the electrochemical consumption of the magnesium is needed.

The uncoated aluminum alloy 5052-H32, cold worked and one-quarter strain hardened, was most resistant to corrosion by 3% sodium chloride solution; the uncoated 2024-T3, solution heat treated, naturally aged, and cold worked showed the greatest rate of corrosion.

All of the aluminum alloys tested showed an enhanced corrosion resistance when coated with a flame-fused Teflon film. The nonwetting character of the applied Teflon coating is related to the observed decreased corrosion rate of the aluminum specimens. Specimens of 6061-T6 coated with fused Teflon one-coat green enamel showed the least amount of corrosion, although the mechanical properties were affected most extensively compared with the other aluminum alloys tested using the flame fusion method (1). No relationship between the temper and the corrosion rate, before and after flame fusion, could be made from the data.

The cladding on the aluminum alloy 2024-T3 did not affect the corrosion resistance of the Teflon-coated specimens, as the chrome pickling did on the magnesium alloy AZ31B-H24. The corrosion resistance of aluminum alloy 2024-T3 was not enhanced by pretreating the test specimens with methacrylic acid prior to the Teflon application.

The three corrosion test methods evaluated during the investigation indicated that the immersion or spherical-joint contact method, using 3% sodium chloride solution, gave similar results but at different rates. The salt droplet test method, although used in only a few instances during the investigation, appeared to be most reliable by alleviating excessive edge corrosion on test specimens and by providing conditions more closely related to the marine atmosphere conditions to which aircraft components are subjected. The salt droplet test method will be used in all future coating evaluations.

Although the use of Teflon as a greatly improved protective coating system for magnesium or aluminum alloys was not suggested by the results, the utility of the flame method of applying a protective coating has been demonstrated. Several new formulations based on the findings described in this report and the studies pertaining to the properties of the magnesium ion (12) will be used to formulate new protective systems which will be evaluated in the future.

#### ACKNOWLEDGMENTS

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13. ABSTRACT The corrosion resistance toward 3% sodium chloride solution of flame-fused Teflon-coated magnesium alloy AZ31B-H24 and aluminum alloys 2024-T3, 5052-H32, and 6061-T6 has been studied. The formation of pores in the Teflon coatings applied to magnesium alloy AZ31B-H24 is attributed to hydrogen gas which is evolved from the reaction between the highly acidic Teflon formulations and reactive magnesium alloy. Although a methacrylic acid pretreatment of the magnesium test specimens did not reduce pore formation, the presence of a chrome-pickle film on the magnesium alloy specimens and the use of Teflon one-coat green enamel (du Pont 851-204) alleviated pore formation. Cracks which form when the Teflon is flame fused, especially in thick Teflon coatings on aluminum and magnesium alloys, are attributed in part to the volatile tetrafluoroethylene formed when the Teflon is thermally degraded during the flame fusion. Mass spectral data indicate that the major decomposition product, tetrafluoroethylene, is not altered by the magnesium or glass substrate on which it is present during the thermal decomposition. Teflon one-coat green enamel, which has been applied in thin films, 0.0002 to 0.0004 in. thick, and which has been properly flame fused, will not be wet by the 3% sodium chloride solution during the test period; specimens having such Teflon coatings exhibit enhanced corrosion resistance. Three methods were evaluated as accelerated corrosion tests: an immersion test, a salt droplet test, and a spherical-joint contact test which could be modified to measure the relative resistance of the coated specimens. Magnesium alloy AZ31B-H24, chrome pickled, was most resistant to corrosion when coated with flame-fused Teflon one-coat green enamel; magnesium alloy specimens treated otherwise exhibited enhanced corrosion rates. Aluminum alloy 6061-T6, Teflon coated and flame fused, exhibited the greatest corrosion resistance when compared with the other aluminum alloys tested. No correlation was found between the various aluminum alloy tempers studied and the corrosion rate. In general, the corrosion tests indicate that the flame-fused Teflon coating afforded some protection against corrosion as compared to the uncoated specimens.			

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