EFFECT OF PROTECTIVE COATINGS ON THE STRESS-CORROSION PROPERTIES OF SUPERSONIC-TRANSPORT SKIN MATERIALS

ELEVENTH QUARTERLY STATUS REPORT

to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

For the Period Between 1 June, 1965, and 31 August, 1965

Contract No. NASr-117

SOUTHERN RESEARCH INSTITUTE

2000 9th Avenue S. Birmingham 5, Alabama

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ABSTRACT

This report summarizes the work performed under NASA Contract No. NASr-117 during the period between 1 June and 31 August, 1965. The purpose of the project is to determine whether certain commercially available coatings, selected on the basis of earlier work, will retard or prevent stress corrosion of substrate alloys that are promising candidates for use as the outer skins on supersonic-transport aircraft (SST). The program entails the exposure, for durations of 1000, 3000, 5000, and 7000 hr, of self-loaded specimens to various combinations of scratch damage, hot salt at 550° F, and humid salt at 95° F. Susceptibility to damage from stress corrosion was evaluated by means of a bend test that revealed the residual ductility of the exposed specimens.

This report presents the results from the 3000-hr exposures, and compares them to the results from the previously reported 1000-hr exposures. Tentative conclusions derived from these results are:

1. The AM 350 SCT stainless steel substrate will require protection from stress corrosion in salt-laden humid environments.

2. The inherent ductility of the solution-treated-and-aged Rene 41 used in these experiments is inconsistent to the extent that its vulnerability to stress corrosion within 3000 hr is obscured.

3. Duplex annealed Ti-8Al-1Mo-1V alloy will require protection from stress corrosion when exposed to dry salt at 550° F.

4. Aluminum-Modified Silicone provides excellent protection for at least 3000 hr in either hot-salt environments at 550° F or in humid-salt environments at 95° F.

5. Catalytically Cured Silicone provides excellent protection for at least 3000 hr in humid-salt environments at 95° F but it quickly shredded from each of the substrates in the hot-salt environment at 550° F.

6. Zinc in Silicate Vehicle apparently has a large deleterious effect on the ductility of Rene 41 and Ti-8-1-1 regardless of the exposure conditions. It provides some protection on AM 350 but is not as effective as Aluminum-Modified Silicone or Catalytically Cured Silicone.

7. Electrophoretic Aluminum, which was evaluated in a blistered condition and on specimens with exposed edges, did not provide significant protection for Ti-8-1-1 in hot salt at 550° F, which was the only substrate-environment combination in which it was evaluated.

8. Flame-Sprayed Aluminum, which was evaluated on specimens with the inside surfaces essentially uncoated, provided some protection (more than Electrophoretic Aluminum or Zinc in Silicate Vehicle, but considerably less than Aluminum-Modified Silicone) on Ti-8-1-1 in hot salt at 550° F, which was the only substrate-environment combination in which it was evaluated.

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REPORT ON

EFFECT OF PROTECTIVE COATINGS ON THE STRESS-CORROSION PROPERTIES OF SUPERSONIC-TRANSPORT SKIN MATERIALS

INTRODUCTION

This report summarizes the progress made during the fifth quarter of a project being performed by Southern Research Institute under Contract No. NASr-117. This quarter consisted of the period between 1 June, 1965, and 31 August, 1965.

The purpose of this research project is to determine whether selected coatings will protect metal substrates from stress-corrosion. These data will provide needed additional information on the feasibility of using commercially available protective coatings to prevent corrosion of the skins of supersonic-transport aircraft (SST). The coatings and substrates to be evaluated were chosen from the results of earlier work on this contract (1, 2)[1].

Pertinent background information and a detailed description of the specimen preparation and environmental exposures, along with the general evaluation procedure, were presented in earlier progress reports and will not be repeated here. Described briefly, the program consists of various stress-corrosion exposures applied to self-loading type specimens constructed as shown in Figure 1. The substrates, coatings, exposure conditions and evaluation methods are charted in Figure 2.

WORK PERFORMED

During this quarter the second group of exposed specimens (3000-hr exposure) was removed from the exposure atmospheres, visually examined, rinsed, and subjected to compressive loading for bend-ductility evaluations. We also re-evaluated the bend ductility of seven 1000-hr-exposure specimens which had fractured prematurely while bending around a radius of the clamping fixture. We found that, with proper clamping, these specimens could be reloaded so that the fracture would occur at the proper location–near the center of one or both bowed members. The original ductility data reported for these specimens have been amended in accordance with the new ductility readings, but these corrections caused no significant changes in the comparative status of the coatings and substrates involved.

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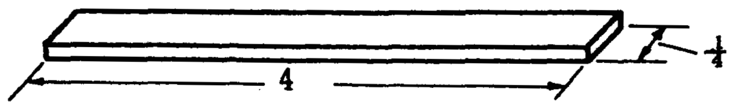
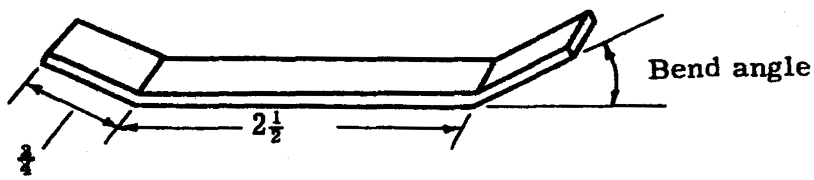
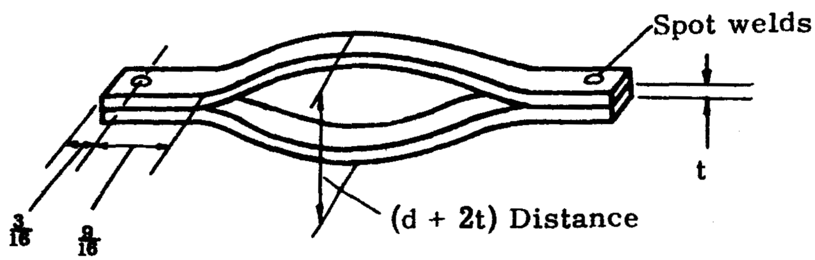
[1] The numbers in parentheses refer to the references at the end of the report.



(a) Machined strip.

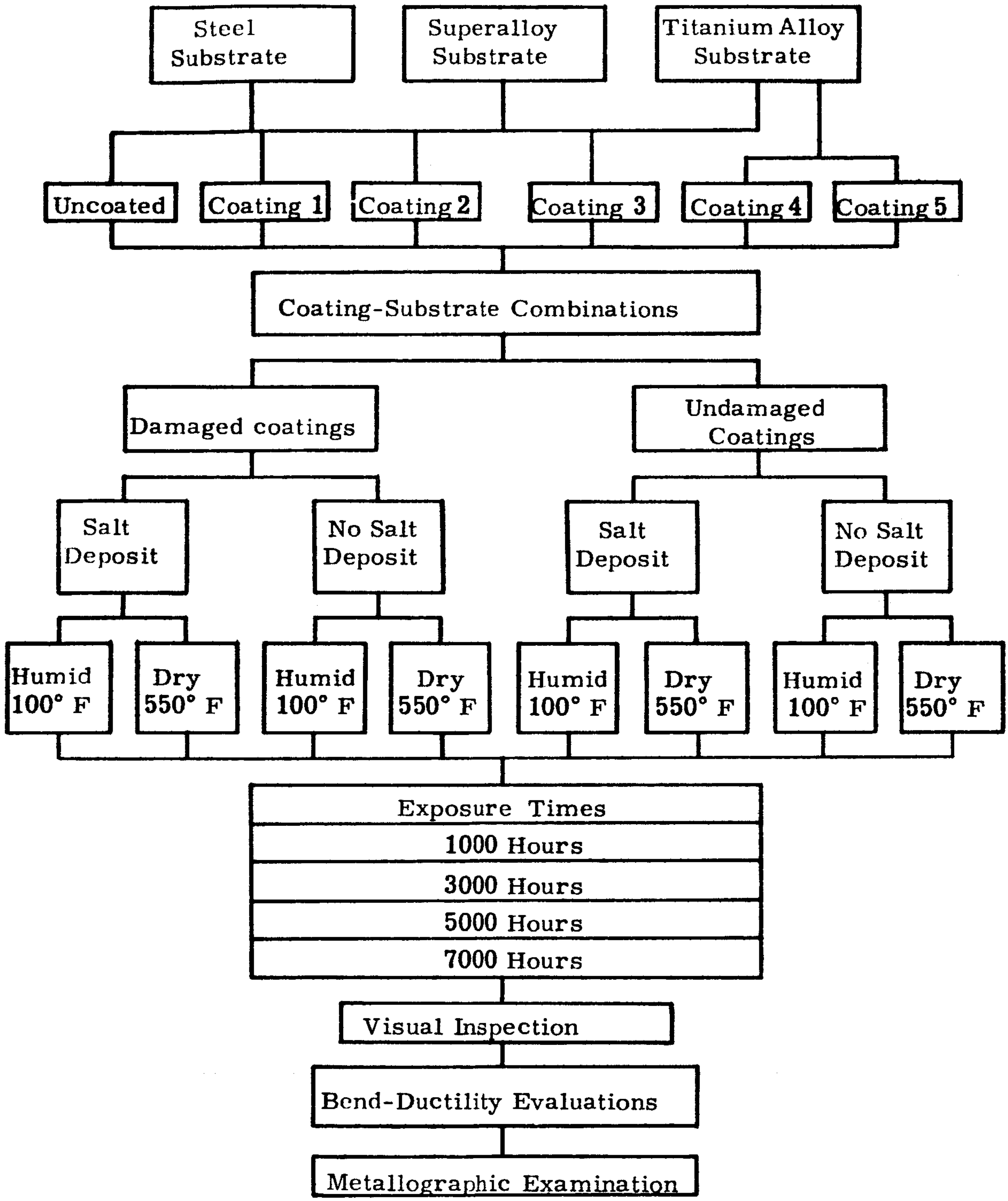


(b) Strip with ends bent.



(c) Completed specimen.

Figure 1. Construction of the self-stressed specimen. (All dimensions are in inches).



Coating 1 - Aluminum-Modified Silicone
Coating 2 - Catalytically Cured Silicone
Coating 3 - Zinc in Silicate vehicle
Coating 4 - Electrophoretically Deposited Aluminum
Coating 5 - Flame-Sprayed Aluminum

Figure 2. Flow Sheet of Experimental Conditions

A detailed description of the compressive loading fixture used in the bend-ductility evaluations was presented in the previous progress report and will not be repeated here. Described briefly, the fixture consists of two clamping members fitted vertically in a manually operated hydraulic press. The configurations of the clamping members with a specimen in place are shown in Figure 3. With the specimen installation shown, some specimens tended to fracture at one or more of the fixture radii. This was corrected by installing specimens so that the tab ends extended approximately 1/4-in. beyond the fixtures.

A dial gage calibrated in 0.001-in. increments is employed to provide a reading of the shortening that occurs in the specimen during compression.

PROCEDURES

Following the visual examination of the exposed specimens and the removal of salt from appropriate specimens by water rinsing, we loaded each specimen into the clamping members with its (D + 2t) distance (refer to Figure 1c) extending horizontally. The lower movable platen of the press was raised to a position where approximately 1/2-in. of the specimen tab ends would extend into the slots of both clamping members. Previously, the tab ends had been inserted to their full 3/4-in. length as shown in Figure 3, but this tended to cause some specimens to fracture at a fixture radius. By inserting only a portion (1/2-in.) of the specimen tab ends, we provided freedom for the bowed members of ductile specimens to deflect to almost complete compression before bearing against a slot radius.

Once inside the slots, the tab ends were positioned against the dial-pin locator stops and also against a common slot side in both clamping members. The specimen was then locked into position by tightening the set screws against the flat face of the tab ends.

After positioning and securing the specimen in the fixture, we placed the dial gage in contact with the lower movable platen and set it to the zero position. The specimen was then compressed by raising the lower platen with the hydraulic pump. Specimen compression was continued until fracture occurred, or until complete compression (maximum specimen shortening with contact between the tab ends) was attained. The dial gage, activated by the upward movement of the lower platen, provided a reading of the bend-ductility or shortening that occurred in the specimen.

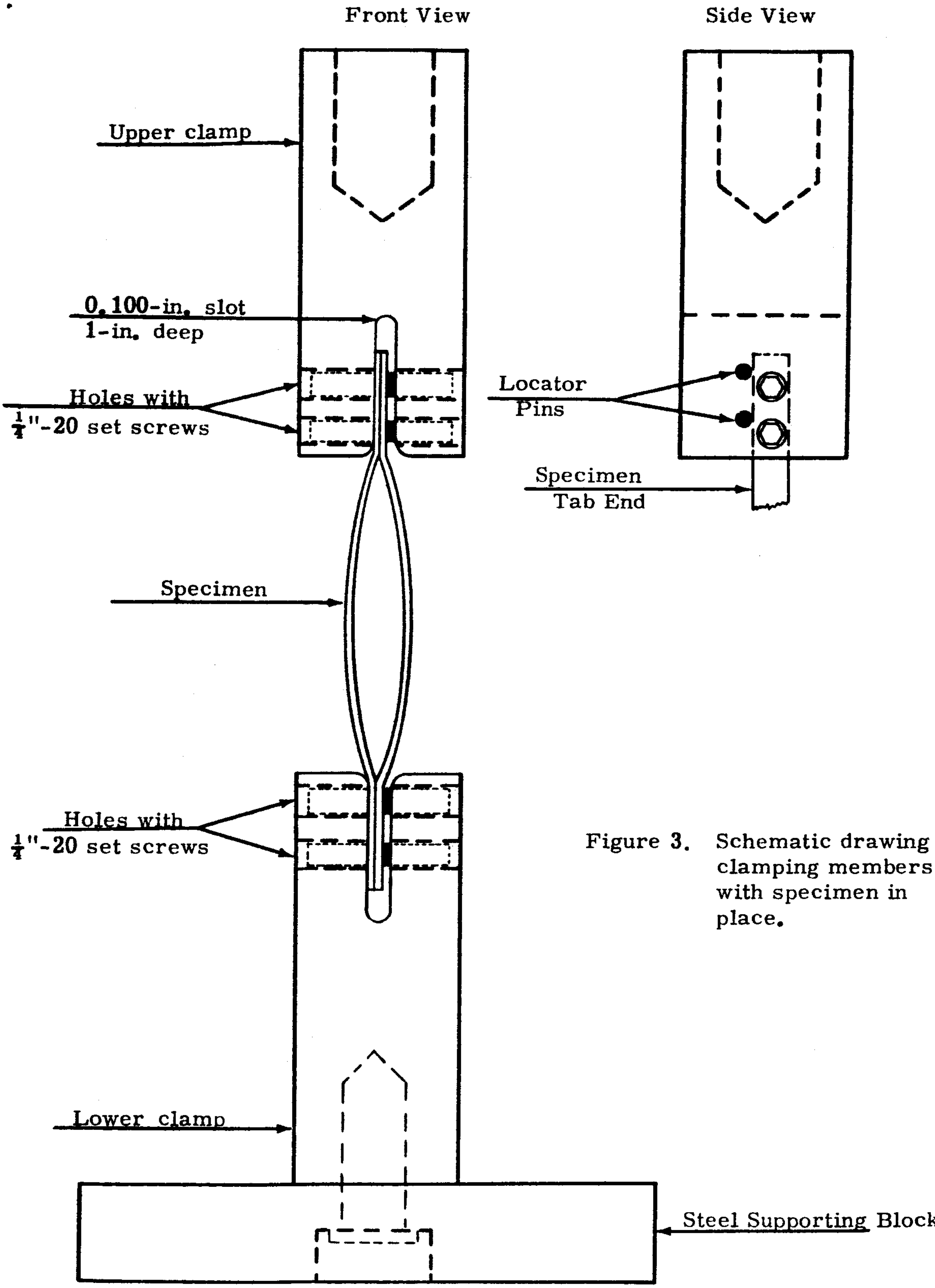


Figure 3. Schematic drawing of clamping members with specimen in place.

RESULTS AND DISCUSSION

Visual Examination

The results of the visual examinations of the 3000-hr-exposed specimens are listed in Table I. This table lists general observations on the appearance of each substrate-coating combination after exposure to the dry, 550° F, or humid, 95° F, atmosphere.

After 3000 hr of exposure to the dry, 550° F atmosphere, the visual appearance of the bare Rene 41 substrate remained unchanged whereas the bare substrates of AM 350 and titanium were discolored. The humid, 95° F exposure caused no visual changes in the bare substrates of titanium and Rene 41, but did, however, cause significant rusting of the bare AM 350 specimens which had been deposited.

The 550° F exposure had no visual effect on any of the substrates coated with Aluminum-Modified Silicone. However, in the 95° F exposure of the Aluminum-Modified-Silicone-coated specimens, slight rusting occurred on salt laden AM 350 specimens and pin-point corrosion appeared on salt laden and damaged Rene 41 specimens.

The Catalytically-Cured-Silicone-coated specimens showed no visual change during the humid, 95° F exposure. However, in the dry, 550° F exposure, this coating shredded and spalled completely from all specimens of each substrate.

Specimens from each substrate material coated with Zinc in Silicate Vehicle contained spotted areas of a brownish-gray discoloration after exposure to the humid, 95° F atmosphere. A grayish-white oxide, appearing in combination with these brownish-gray discolorations, developed on the salt-deposited specimens. The grayish-white oxide was especially severe on the salt deposited Rene 41 specimens. The dry, 550° F atmosphere caused no visual changes in any of the zinc coated specimens in the absence of salt. However, in the salt-deposited specimens a grayish-white oxide formed and appeared only at the immediate areas where the salt was in contact with the coating.

No visual changes were observed in the Electrophoretic-Aluminum and Flame-Sprayed-Aluminum-coated specimens, but these were exposed only to the 550° F atmosphere.

In general, the visual appearance of the 3000-hr-exposed specimens showed no marked differences in comparison with the 1000-hr-exposed specimens. There were slight differences noted, however, in the specimens with

Table I

Visual Examination of 3000-Hr Exposed Specimens

Coating	Substrate	Exposure	Visual Observations after Exposure
Bare	AM 350	550° F	Dark brownish-bronze color over entire surface.
		95° F	Rust spots on specimens with salt.
Aluminum-Modified Silicone	AM 350	550° F	No change.
		95° F	Slight rust and salt stains on salt deposited specimens—no change in unsalted specimens.
Catalytically Cured Silicone	AM 350	550° F	Coating spalled and shredded over entire surface within 48 hr.
		95° F	No change.
Zinc in Silicate Vehicle	AM 350	550° F	Grayish-white oxide only on specimens with salt. No change in specimens without salt.
		95° F	Spotted areas of brownish-gray discolorations on all specimens exposed. Grayish-white oxide noted only on specimens with salt.
Bare	Titanium	550° F	Yellowish gold color over entire surface.
		95° F	No specimen exposed.
Aluminum-Modified Silicone	Titanium	550° F	No change.
		95° F	Salt stains—otherwise no change. No unsalted specimen exposed.
Catalytically Cured Silicone	Titanium	550° F	Coating spalled & shredded over entire surface within 48 hr.
		95° F	No change. Only salt-deposited specimens exposed.
Zinc in Silicate Vehicle	Titanium	550° F	Grayish-white oxide only on specimens with salt. No change in specimens without salt.
		95° F	Only salt deposited specimens exposed—spotted areas of grayish-white oxide.
Electrophoretic Aluminum	Titanium	550° F	No change.
		95° F	No specimens exposed.
Flame-Sprayed Aluminum	Titanium	550° F	Only salt deposited specimens exposed. No change.
		95° F	No specimens exposed.
Bare	Rene 41	550° F	Slight salt stains—otherwise no change.
		95° F	No change.
Aluminum-Modified Silicone	Rene 41	550° F	No change.
		95° F	No change in unsalted specimens. On salt-deposited specimens there were slight salt stains on the undamaged and heavy salt stains with several areas of pin-point corrosion on the damaged specimens.
Catalytically Cured Silicone	Rene 41	550° F	Coating spalled & shredded over entire surface within 48 hr.
		95° F	No change.
Zinc in Silicate Vehicle	Rene 41	550° F	Grayish-white oxide only on specimens with salt. No change in specimens without salt.
		95° F	Spotted areas of brownish-gray discoloration on all specimens. Severe grayish-white oxide coating on specimens with salt.

coatings of Zinc in Silicate Vehicle and Aluminum-Modified Silicone. These differences were:

1. Under humid, 95° F conditions the grayish-white oxide on the 3000-hr zinc-coated and salt-deposited specimens was much heavier than the oxide layer on similar specimens from the 1000-hr exposure. This oxide layer was more prominent on the Rene 41 substrate than on the other substrates.

2. Under humid, 95° F conditions the Aluminum-Modified-Silicone coating on AM 350 and Rene 41 had greater amounts of rusting and pin-point corrosion after the 3000-hr exposure. This corrosion was prominent only on the salt deposited and damaged specimens.

Bend-Ductility

The complete results of the bend-ductility evaluations on the 3000-hr-exposed specimens are listed in Tables II, III and IV. The data from these tables, in combination with similar data from the 1000-hr-exposure specimens, are graphically illustrated in Figures 4, 5, 6, 7, 8, and 9 (550° F exposure) and in Figures 10, 11, 12, 13, and 14 (95° F exposure). In each figure the bend-ductility (shortening) data is presented in bar-chart form with each bar representing an average shortening value for two or more replicate specimens of a particular substrate-coating combination.

Within each temperature exposure (95° F and 550° F), each substrate-coating combination is subjected to four exposure conditions. These conditions are: undamaged, no salt; damaged, no salt; undamaged, with salt; and damaged, with salt. At each temperature, two figures are allotted to each substrate, with each figure containing only two of the exposure conditions.

For each substrate, the data from the undamaged specimens are presented in one figure, and the other figure contains the data from the damaged specimens. Grouped within each of the exposure conditions are separate bars that represent the average bend-ductility results from each substrate-coating combination for the 1000-hr and 3000-hr exposure intervals. Sufficient space has been reserved for future additions of the 5000-hr and 7000-hr data.

The bars for each substrate-coating combination are made with a different pattern so that particular combinations can be easily followed from one figure to the next. The dashed line at 2.0-in. shortening in each figure was established on the basis of specimen and fixture geometry, and represents the transition from full ductility to an embrittled condition. Ductility values below this line were considered to indicate significant embrittlement, whereas values at or above the line indicated full ductility. All specimens with shortening

Table II

AM 350 Bend-Ductility Data—
3000-Hr Exposure to Dry, 550° F,
and Humid, 95° F, Atmosphere

Coating	SPECIMEN NUMBERS[1]				SHORTENING IN INCHES			
	Undamaged No Salt	Damaged No Salt	Undamaged With Salt	Damaged With Salt	Undamaged No Salt	Damaged No Salt	Undamaged With Salt	Damaged With Salt
				AM 350—3000 Hr, 550° F, Dry Atmosphere				
Bare	A0U1H3A	A0D1H3A	A0U2H3A	A0D2H3A	2.500	2.502	2.500	2.500
Bare	A0U1H3B	A0D1H3B	A0U2H3B	A0D2H3B	2.504	2.503	2.500	2.496
Aluminum-Modified Silicone	A1U1H3A	A1D1H3A	A1U2H3A	A1D2H3A	2.500	2.500	2.499	2.500
Aluminum-Modified Silicone	A1U1H3B	A1D1H3B	A1U2H3B	A1D2H3B	2.500	2.500	2.500	2.500
Aluminum-Modified Silicone			A1U2H3C				2.500	
Catalytically Cured Silicone	A2U1H3A	A2D1H3A	A2U2H3A	A2D2H3A	2.502	2.500	2.500	2.500
Catalytically Cured Silicone	A2U1H3B	A2D1H3B	A2U2H3B	A2D2H3B	2.500	2.500	2.488	2.500
Catalytically Cured Silicone			A2U2H3C				2.486	
Zinc in Silicate Vehicle	A3U1H3A	A3D1H3A	A3U2H3A	A3D2H3A	2.500	2.490	2.500	2.495
Zinc in Silicate Vehicle	A3U1H3B	A3D1H3B	A3U2H3B	A3D2H3B	2.504	2.500	2.500	2.488
				AM 350—3000 Hr, 95° F, 95% Humidity				
Bare	A0U1L3A	A0D1L3A	A0U2L3A	A0D2L3A	2.500	2.500	2.500	0[2]
Bare	A0U1L3B	A0D1L3B	A0U2L3B	A0D2L3B	2.500	2.500	2.500	0[2]
Aluminum-Modified Silicone	A1U1L3A	A1D1L3A	A1U2L3A	A1D2L3A	2.500	2.494	2.500	2.495
Aluminum-Modified Silicone	A1U1L3B	A1D1L3B	A1U2L3B	A1D2L3B	2.500	2.500	2.500	2.495
Catalytically Cured Silicone	A2U1L3A	A2D1L3A	A2U2L3A	A2D2L3A	2.460	2.460	2.470	2.457
Catalytically Cured Silicone	A2U1L3B	A2D1L3B	A2U2L3B	A2D2L3B	2.470	2.462	2.465	2.460
Zinc in Silicate Vehicle	A3U1L3A	A3D1L3A	A3U2L3A	A3D2L3A	2.498	2.450	1.685	1.390
Zinc in Silicate Vehicle	A3U1L3B	A3D1L3B	A3U2L3B	A3D2L3B	2.495	2.488	2.495	1.795

1 These specimen numbers relate to the shortening values that are located in the same relative position.
2 Fracture during exposure within 800 hours.

Table III

Rene 41 Bend-Ductility Data—
3000-Hr Exposure to Dry, 550° F,
and Humid, 95° F, Atmosphere

Coating	SPECIMEN NUMBERS[1]				SHORTENING IN INCHES			
	Undamaged No Salt	Damaged No Salt	Undamaged With Salt	Damaged With Salt	Undamaged No Salt	Damaged No Salt	Undamaged With Salt	Damaged With Salt
				Rene 41—3000 Hr, 550° F, Dry Atmosphere				
Bare	R0U1H3A	R0D1H3A	R0U2H3A	R0D2H3A	0.750	0.360	1.585	0.575
Bare	R0U1H3B	R0D1H3B	R0U2H3B	R0D2H3B	0.900	0.270	0.980	2.040
Aluminum-Modified Silicone	R1U1H3A	R1D1H3A	R1U2H3A	R1D2H3A	0.724	1.180	0.775	2.225
Aluminum-Modified Silicone	R1U1H3B	R1D1H3B	R1U2H3B	R1D2H3B	0.544	1.010	1.240	0.325
Aluminum-Modified Silicone	R1U1H3C		R1U2H3C	R1D2H3C	1.045		1.090	2.025
Catalytically Cured Silicone	R2U1H3A	R2D1H3A	R2U2H3A	R2D2H3A	1.540	1.495	1.835	– [a]
Catalytically Cured Silicone	R2U1H3B	R2D1H3B	R2U2H3B	R2D2H3B	1.310	1.175	1.832	0.250
Catalytically Cured Silicone	R2U1H3C		R2U2H3C	R2D2H3C	1.115		1.055	0.725
Zinc in Silicate Vehicle	R3U1H3A	R3D1H3A	R3U2H3A	R3D2H3A	1.030	0.655	0.745	0.800
Zinc in Silicate Vehicle	R3U1H3B	R3D1H3B	R3U2H3B	R3D3H3B	0.935	0.865	1.085	0.355
Zinc in Silicate Vehicle	R3U1H3C		R3U2H3C	R3D2H3C	0.980		0.777	0.790
				Rene 41—3000 Hr, 95° F, 95% Humidity				
Bare	R0U1L3A	R0D1L3A	R0U2L3A	R0D2L3A	1.820	0.800	2.195	1.280
Bare	R0U1L3B	R0D1L3B	R0U2L3B	R0D2L3B	1.500	0.455	1.410	0.900
Aluminum-Modified Silicone	R1U1L3A	R1D1L3A	R1U2L3A	R1D2L3A	1.180	1.060	0.810	0.735
Aluminum-Modified Silicone	R1U1L3B	R1D1L3B	R1U2L3B	R1D2L3B	1.030	2.210	1.080	1.020
Aluminum-Modified Silicone	R1U1L3C		R1U2L3C	R1D2L3C	0.870		0.850	1.440
Catalytically Cured Silicone	R2U1L3A	R2D1L3A	R2U2L3A	R2D2L3A	0.850	1.720	1.110	1.510
Catalytically Cured Silicone	R2U1L3B	R2D1L3B	R2U2L3B	R2D2L3B	2.443	1.720	0.320	0.750
Catalytically Cured Silicone	R2U1L3C		R2U2L3C	R2U2L3C	0.620		0.980	0.925
Zinc in Silicate Vehicle	R3U1L3A	R3D1L3A	R3U2L3A	R3D2L3A	0.630	0.670	0.405	0.550
Zinc in Silicate Vehicle	R3U1L3B	R3D1L3B	R3U2L3B	R3D2L3B	0.965	0.690	0.375	0.480
Zinc in Silicate Vehicle	R3U1L3C		R3U2L3C	R3D2L3C	1.030		0.540	0.455

[1] These specimen numbers relate to the shortening values that are located in the same relative position.

a A welded tab end sprang apart during exposure

Table IV

Ti-8Al-1Mo-1V Bend-Ductility Data–
3000-Hr Exposure to Dry, 550° F,
and Humid, 95° F, Atmosphere

Coating	SPECIMEN NUMBERS[1] Undamaged No Salt	Damaged No Salt	Undamaged With Salt	Damaged With Salt	SHORTENING IN INCHES Undamaged No Salt	Damaged No Salt	Undamaged With Salt	Damaged With Salt
Titanium—3000 Hr, 550° F, Dry Atmosphere								
Bare	T0U1H3A	T0D1H3A	T0U2H3A	T0D2H3A	2.135	1.170	0.497	0.590
Bare	T0U1H3B	T0D1H3B	T0U2H3B	T0D2H3B	2.200	1.710	0.365	0.286
Bare	T0U1H3C	T0D1H3C	T0U2H3C	T0D2H3C	2.120	1.597	0.362	0.670
Aluminum-Modified Silicone	T1U1H3A	T1D1H3A	T1U2H3A	T1D2H3A	1.630	1.160	2.336	1.710
Aluminum-Modified Silicone	T1U1H3B	T1D1H3B	T1U2H3B	T1D2H3B	1.635	1.590	2.481	1.960
Aluminum-Modified Silicone	T1U1H3C	T1D1H3C	T1U2H3C	T1D2H3C	1.781	1.400	2.470	1.410
Catalytically Cured Silicone	T2U1H3A	T2D1H3A	T2U2H3A	T2D2H3A	1.690	1.660	2.400	1.135
Catalytically Cured Silicone	T2U1H3B	T2D1H3B	T2U2H3B	T2D2H3B	1.670	1.910	2.455	1.440
Catalytically Cured Silicone	T2U1H3C	T2D1H3C	T2U2H3C	T2D2H3C	1.686	1.900	2.463	1.925
Zinc in Silicate Vehicle	T3U1H3A	T3D1H3A	T3U2H3A	T3D2H3A	0.670	0.778	0.990	0.580
Zinc in Silicate Vehicle	T3U1H3B	T3D1H3B	T3U2H3B	T3D2H3B	0.660	0.795	1.000	0.925
Zinc in Silicate Vehicle	T3U1H3C	T3D1H3C	T3U2H3C	T3D2H3C	0.695	0.770	0.688	0.950
Electrophoretic Aluminum	T4U1H3A	T4D1H3A	T4U2H3A	T4D2H3A	2.100	2.275	0.290	0.280
Electrophoretic Aluminum	T4U1H3B	T4D1H3B	T4U2H3B	T4D2H3B	1.710	2.283	0.283	0.445
Electrophoretic Aluminum	T4U1H3C		T4U2H3C	T4D2H3C	2.200		0.355	0.420
Flame-Sprayed Aluminum			T5U2H3A	T5D2H3A			1.259	1.290
Flame-Sprayed Aluminum			T5U2H3B	T5D2H3B			1.305	1.090
Titanium—3000 Hr, 95° F, 95% Humidity								
Aluminum-Modified Silicone			T1U2L3A				2.470	
Aluminum-Modified Silicone			T1U2L3B				2.340	
Aluminum-Modified Silicone			T1U2L3C				2.370	
Catalytically Cured Silicone			T2U2L3A				2.440	
Catalytically Cured Silicone			T2U2L3B				2.445	
Catalytically Cured Silicone			T2U2L3C				2.285	
Zinc in Silicate Vehicle			T3U2L3A				1.450	
Zinc in Silicate Vehicle			T3U2L3B				1.490	
Zinc in Silicate Vehicle			T3U2L3C				1.620	

1 These specimen numbers relate to the shortening values that are located in the same relative position.

values less than 2.0-in. fractured in one or both bowed members. Some specimens with shortening values between 2.0 and 2.5 in. fractured also, but these fractures were considered to be insignificant because the shortening values were within the maximum ductility range.

Because of the type of exposures involved, we assumed that any significant reduction in shortening was a result of stress corrosion unless explainable by other causes. In our analysis of the results, ductility reductions of 0.2 in. or more were considered to be significant. The apparent improved ductility of the fully ductile 3000-hr specimens can be attributed to the previously explained change in the technique used for installing specimens in the fixture. This installation change provided a greater original distance between the clamping members of the fixture and thereby allowed fully ductile specimens to attain a greater shortening value.

Figures 4 and 5, which present the results from the AM 350 bare and coated specimens exposed to the dry, 550° F environment , show that, after 3000-hr exposures, damaged and undamaged specimens sustained no significant losses in ductility, either in salted or unsalted conditions. It is indicated that the AM 350 alloy remains insensitive to hot-salt after 3000 hr of exposure. Should this insensitivity continue to prevail throughout the longer-duration exposures, it would indicate that protection from stress corrosion would not be necessary for AM 350 in this environment, but that protective coatings would not be harmful if needed for other reasons.

The ductility results from the Rene 41 specimens exposed to the dry, 550° F atmosphere are presented in Figures 6 and 7. The results have continued to be erratic for both bare and coated specimens through the 3000-hr exposure. For example, the bare, undamaged specimens with salt exhibited greater ductility than those undamaged without salt, and the bare, damaged specimens with salt were more ductile than those without salt. The coated Rene 41 specimens exhibited similarly erratic and unexplainable ductility patterns. Therefore, it is indicated that the effects of the coatings have been obscured by the erratic ductility of the substrate.

Figures 8 and 9 contain the ductility results from bare and coated Ti-8Al-1Mo-1V specimens after exposure to the dry, 550° F atmosphere. After 3000 hr the bare specimens without salt continued to retain good ductility in the undamaged condition, but a considerable ductility decrease occurred in those with previously inflicted mechanical damage. However, the bare specimens with salt exhibited large ductility losses in both the 1000-hr and 3000-hr exposures. The similar ductilities produced by both exposure times indicate that the 550° F-dry-salt condition causes its maximum ductility damage within 1000 hr. Therefore, it remains evident that coatings or some other form of protection will be needed for this substrate if it is subjected to this type of exposure in service.

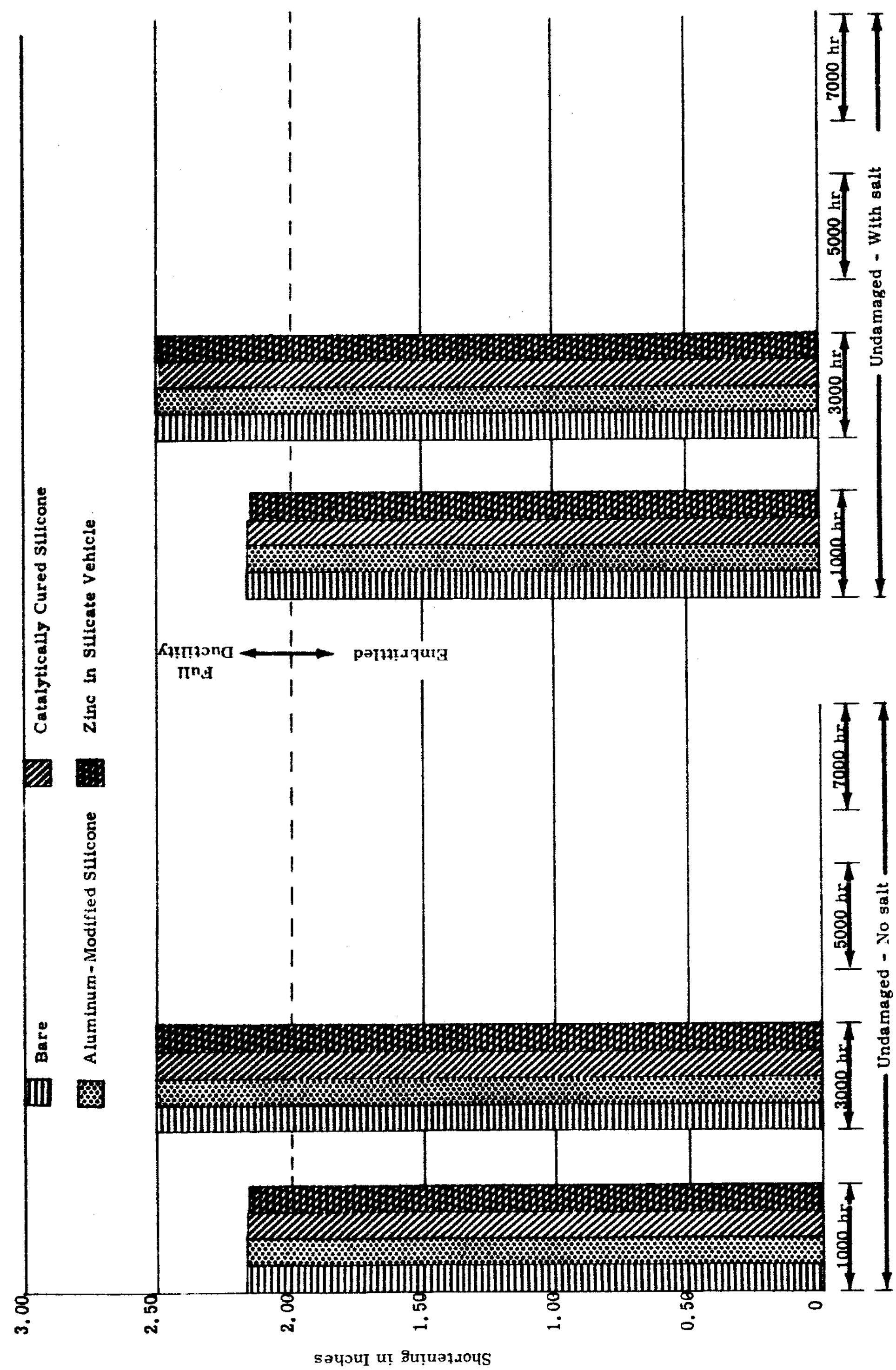


Figure 4. Bend-Ductility Results from Undamaged AM 350 after 3000-Hr Exposure at 550° F.

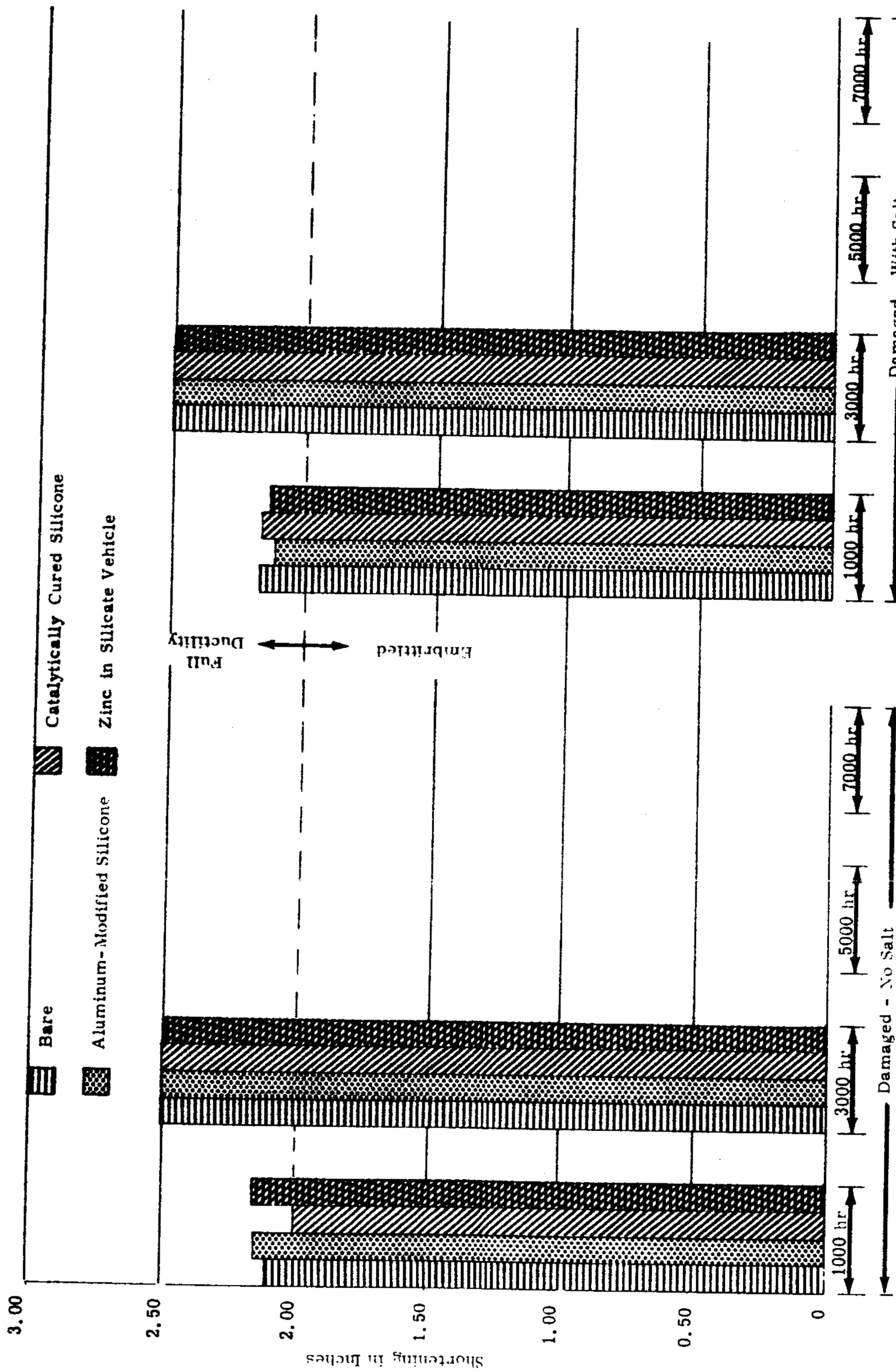


Figure 5. Bend-Ductility Results from Damaged AM 350 after 3000-Hr Exposure at 550° F.

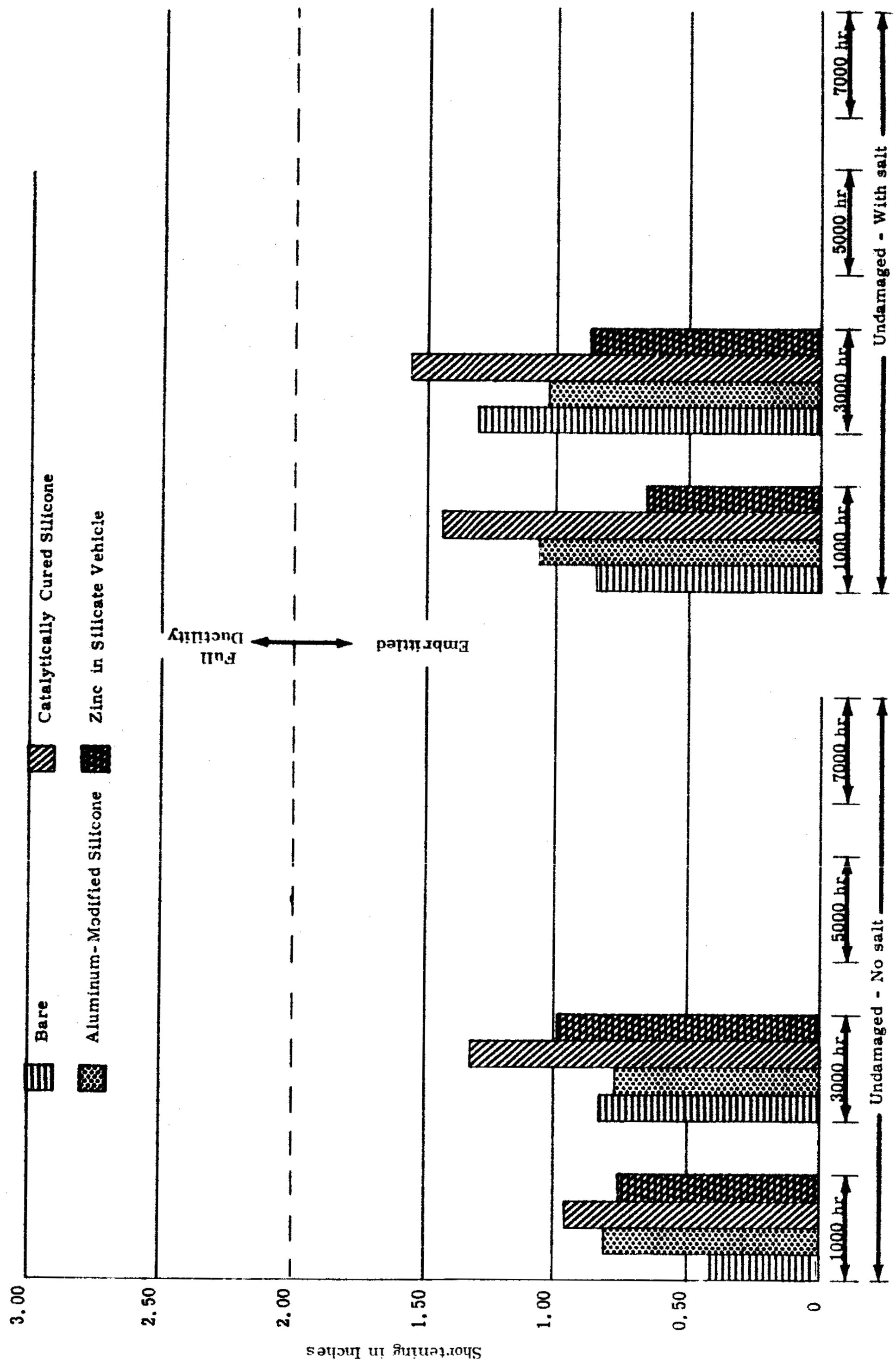
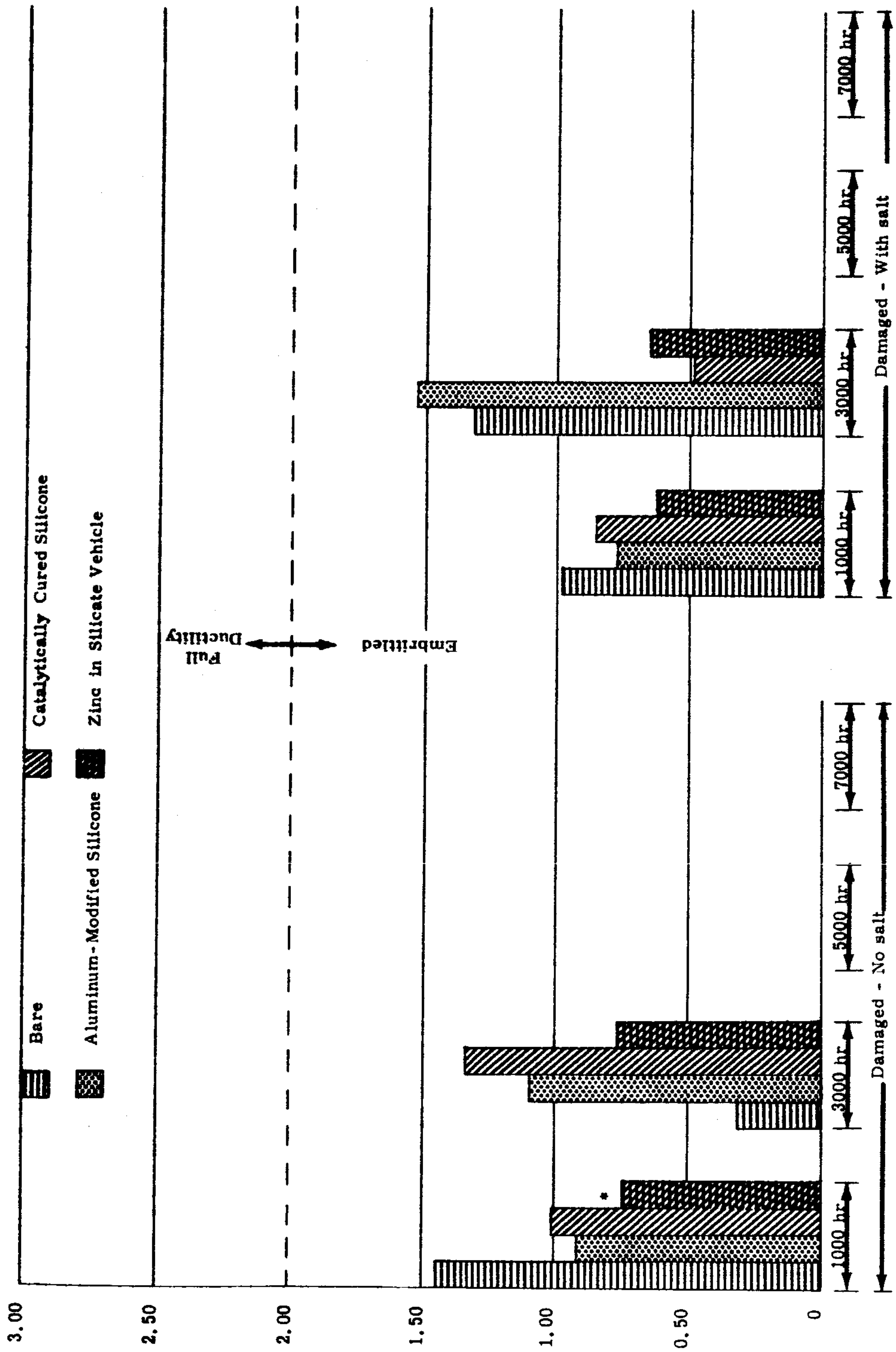
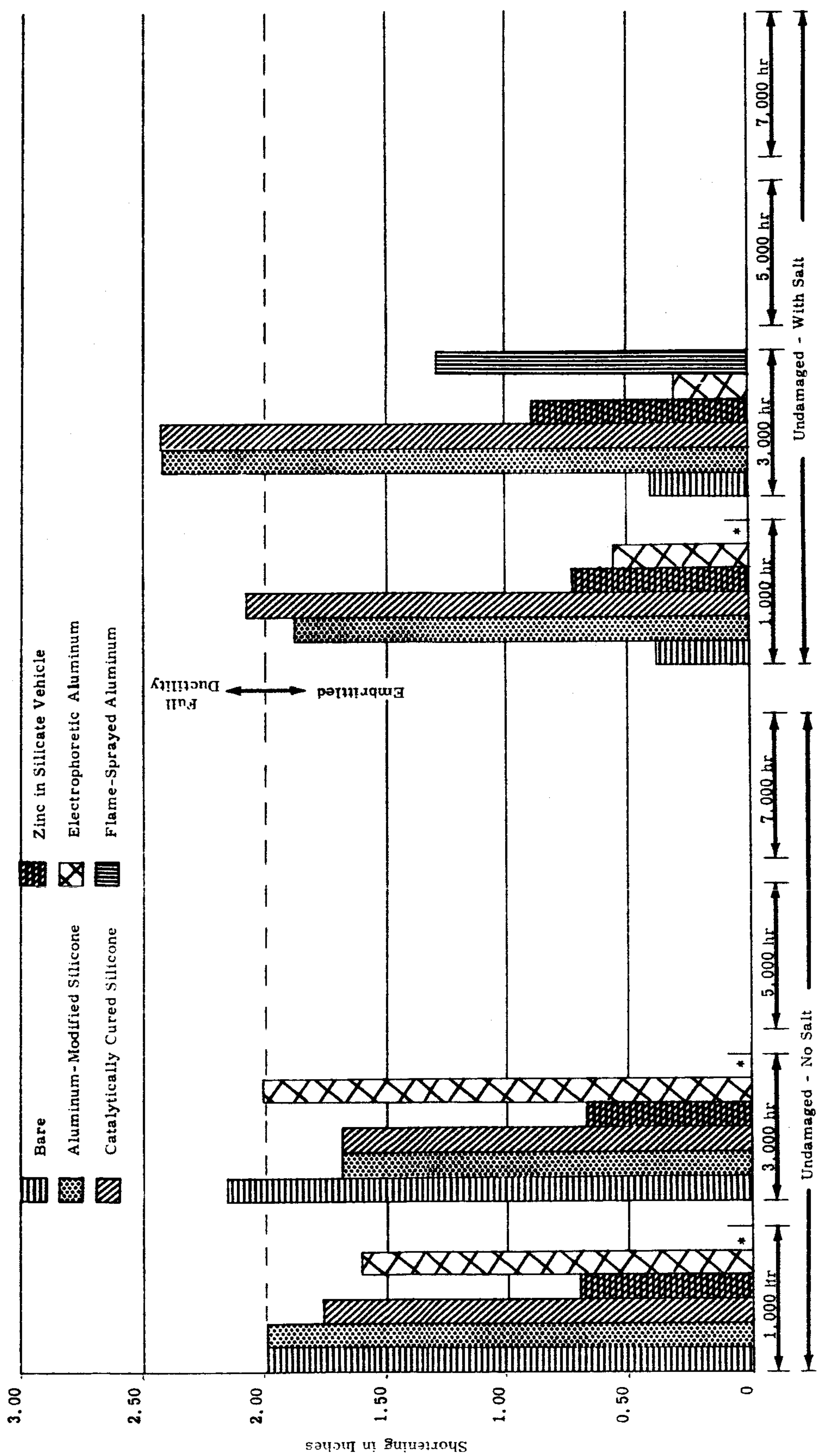


Figure 6. Bend-Ductility Results from Undamaged Rene 41 after 3000-Hr Exposure at 550° F.



* One specimen only

Figure 7. Bend-Ductility Results from Damaged Rene 41 after 3000-Hr Exposure at 550° F.



* No specimen available for exposure

Figure 8. Bend-Ductility Results from Undamaged Ti-8Al-1Mo-1V after 3000-Hr Exposure at 550° F.

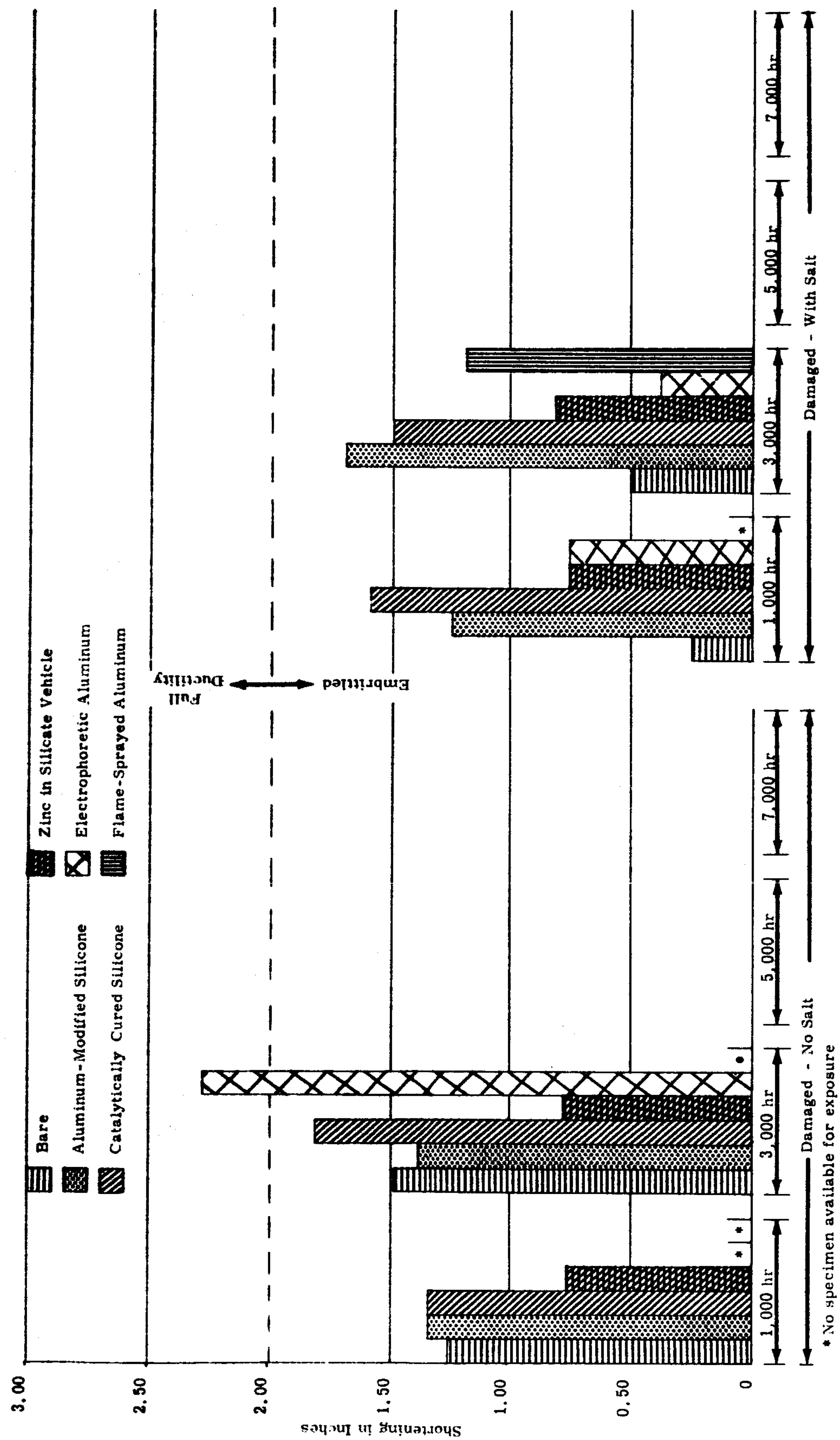


Figure 9. Bend-Ductility Results from Damaged Ti-8Al-1Mo-1V after 3000-Hr Exposure at 550° F.

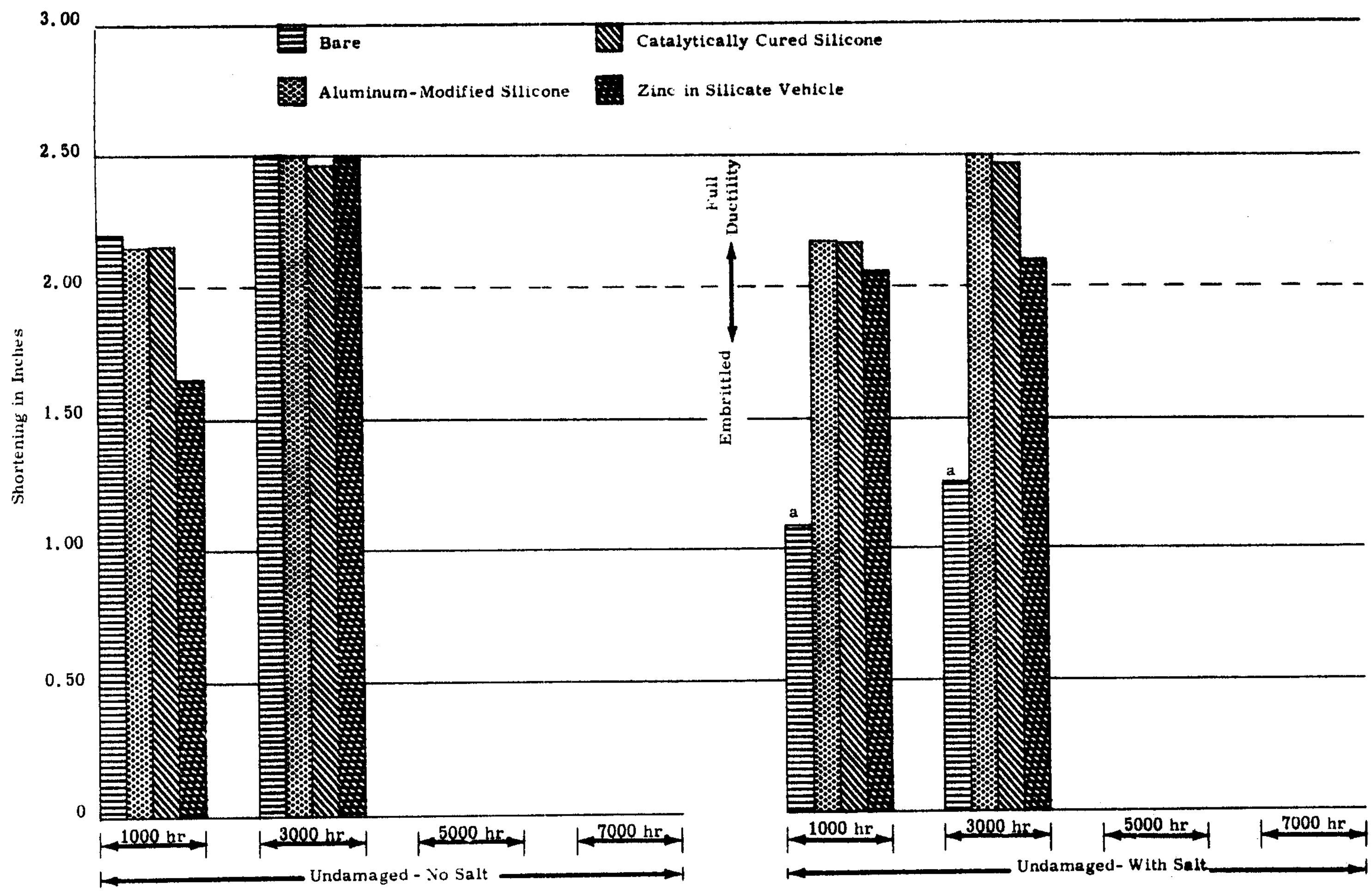
The results from the Aluminum-Modified-Silicone and Catalytically-Cured-Silicone coatings indicate that they continue to provide protection after 3000 hr. The undamaged specimens exposed to salt showed no losses in ductility, although some anomalous loss was exhibited by the undamaged specimens not exposed to salt. The ductility losses in the damaged specimens, in both the salted and unsalted conditions, were equivalent to the losses produced by the mechanical damage alone, showing that the two coatings provided protection from the hot-salt exposure. The apparent protection provided by the Catalytically-Cured-Silicone coating is misleading because it spalled from the substrate within 48 hr after the start of the exposure. The resulting bare specimens probably retained their ductility because the salt deposit was removed by the spalled coating.

The ductilities of the specimens coated with Zinc in Silicate Vehicle continued to remain uniformly low after 3000 hr of exposure. Since the ductilities of these zinc coated specimens in both the 1000-hr and 3000-hr exposures have been most uniform under all conditions of exposure, the low ductilities must be attributed to the coating itself. The reasons for this behaviour of the zinc coating are still undetermined.

The Electrophoretic-Aluminum-coated specimens were within the full ductility range in both the undamaged and damaged conditions when no salt was involved. However, in the presence of salt, both the undamaged and damaged specimens exhibited low ductilities. The ductilities of these salted specimens were in the same range as those ductilities exhibited by the bare, salt-deposited specimens. These ductility losses might have occurred because the edges of the specimens were not coated, or because the coating was in poor condition from the blisters that developed when the coating was applied by an experimental technique.

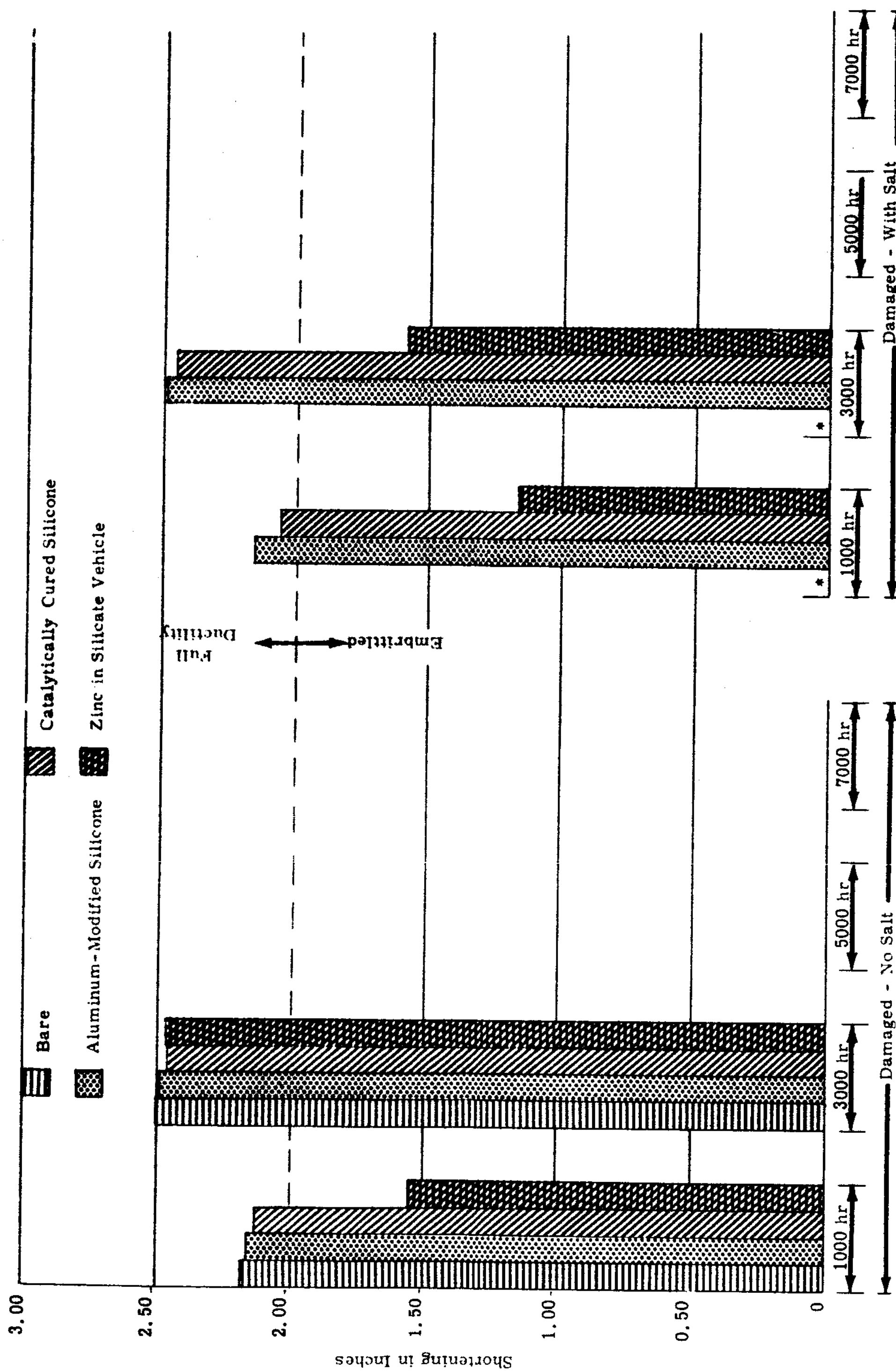
The only Flame-Sprayed-Aluminum-coated specimens exposed at 550° F were those exposed to salt in the undamaged and damaged conditions. The ductility values of both the undamaged and damaged specimens were well below the maximum ductility range. However, no unsalted specimens were involved in this exposure interval. Therefore, the cause of the reduced ductility will be in doubt until additional specimens from the longer-duration exposures become available.

As shown by the results plotted in Figures 10 and 11, which pertain to the humid, 95° F exposure on AM 350 specimens, the bare specimens without salt retained good ductility in both the undamaged and damaged conditions. Most of the bare specimens with salt deposits, however, were rapidly attacked and fractured long before the 1000-hr exposure had been completed. On the other hand, two undamaged specimens, one within the 1000-hr group and one within the 3000-hr group, did not fracture during the exposure and retained full ductility in the bend test.



a. Average between one specimen that had no significant change in ductility and one specimen that fractured during exposure (Zero Shortening).

Figure 10. Bend-Ductility Results from Undamaged AM 350 after 3000-Hr Exposure at 95% Humidity.



*Specimen fractured during exposure (Zero Shortening)

Figure 11. Bend-Ductility Results from Damaged AM 350 after 3000-Hr Exposure at 95% Humidity.

In each damaged and salt-exposed specimen, all of which failed prematurely, the fracture occurred in both bowed members. In each of these specimens, one bowed member fractured near its center at the location of maximum stress (and scratch damage) whereas the other bowed member failed near its tab end. We were unable to discern which bowed member fractured first. The premature failure in the undamaged specimen occurred near the tab end of only one bowed member.

Although two of the undamaged specimens retained full ductility, the results from the majority of specimens indicate that salt and a humid atmosphere can cause rapid stress corrosion in the AM 350 material. The anomalous ductile behavior of two of the undamaged specimens indicate that the attack might be dependent upon obscure critical conditions that were not controlled in these experiments.

The full ductility shown by the specimens of Aluminum-Modified-Silicone and Catalytically-Cured-Silicone coatings on AM 350 remained unchanged after 3000 hr of humid exposure, indicating that these coatings provided adequate stress-corrosion protection. The specimens coated with Zinc in Silicate Vehicle and exposed for 3000 hr appeared to regain some of the ductility losses exhibited by the 1000-hr specimens. Whereas the 1000-hr specimens were fully ductile only in the undamaged and salt-exposed condition (an anomaly in itself), the 3000-hr specimens were ductile in all conditions except the damaged and salt-exposed condition (an apparently reasonable result). Although the 3000-hr results indicate that Zinc in Silicate Vehicle provides a significant amount of protection in humid and salt-laden environments, the overall results indicate that this coating has an erratic effect on the ductility of the substrate.

The ductility results from bare and coated Rene 41 specimens after exposure to the humid, 95° F atmosphere are presented in Figures 12 and 13. Although the 1000-hr results from bare specimens had a reasonable pattern of reduced ductilities as related to the exposure conditions, the 3000-hr results reflected the erratic ductility that is apparently characteristic of the Rene 41 substrate. Similarly erratic patterns were developed with the 1000-hr and 3000-hr coated specimens. Therefore, the protective qualities of the coatings could not be accurately evaluated.

The ductilities of the titanium specimens exposed to the humid, 95° F atmosphere are presented in Figure 14. Because of a shortage of specimens, no bare specimens and only a few coated specimens were exposed with the 1000-hr and 3000-hr groups. These few specimens represented only one condition (undamaged, with salt). The specimens coated with Aluminum-Modified Silicone and Catalytically Cured Silicone, after 3000-hr exposure, attained full ductility in contrast to the deteriorated ductility that resulted from 1000-hr exposure. The greater ductility drop in the 1000-hr specimens coated with Zinc in Silicate Vehicle was apparently diminished by the 3000-hr exposure, but not to the level of complete ductility. Further interpretation of these results might be possible when additional specimens from the longer exposure become available.

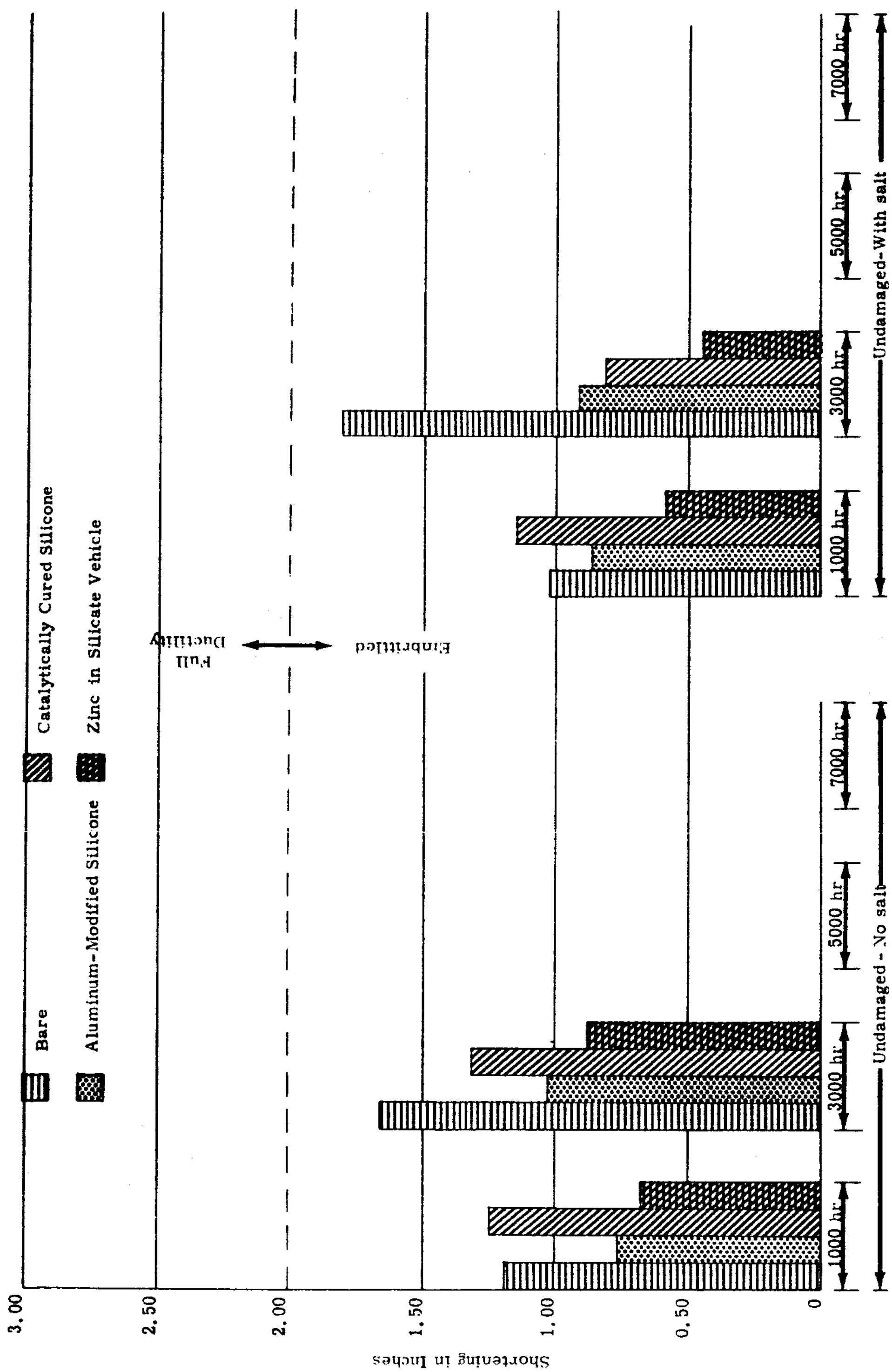


Figure 12. Bend-Ductility Results from Undamaged Rene 41 after 3000-Hr Exposure at 95% Humidity.

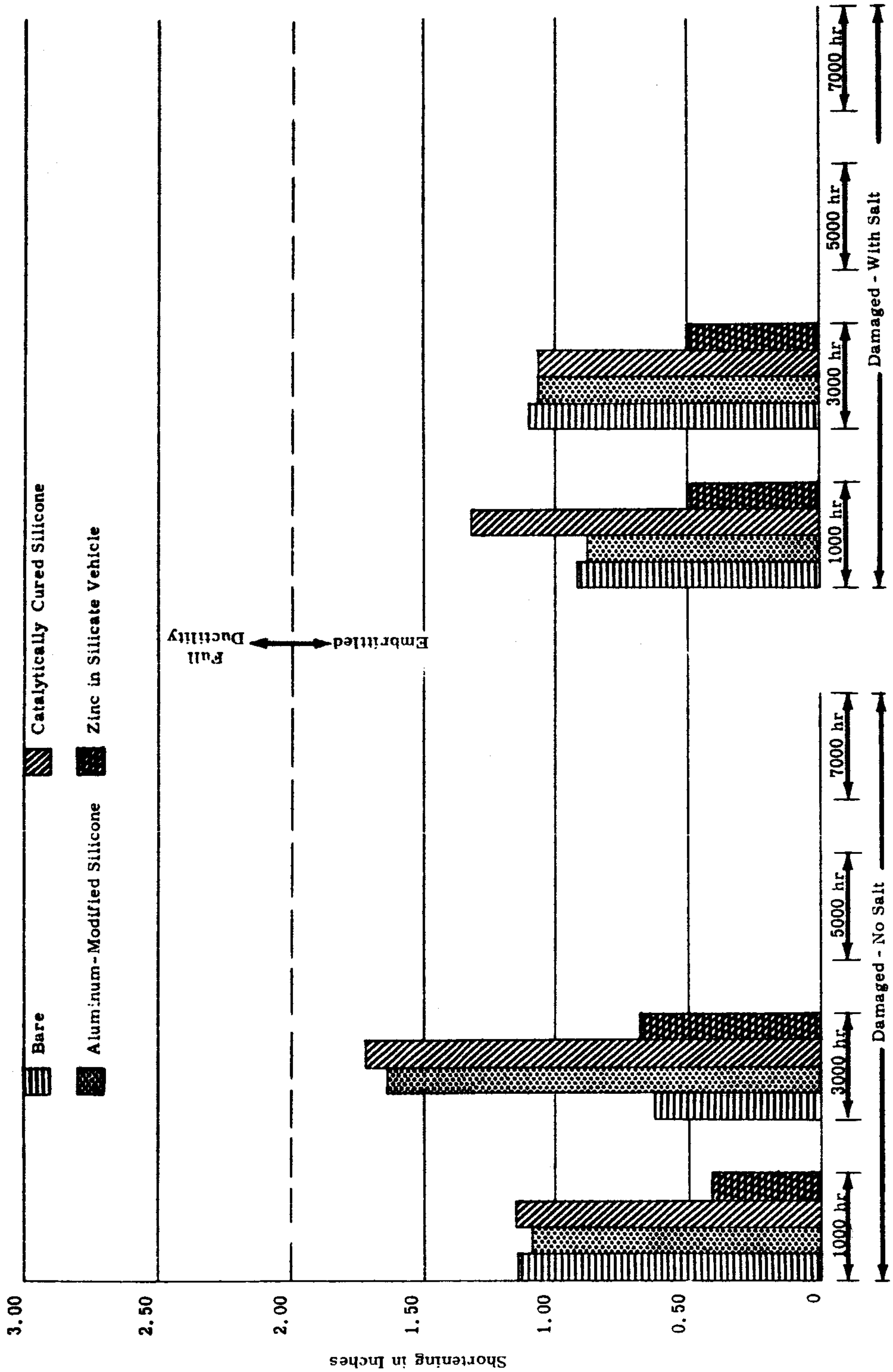


Figure 13. Bend-Ductility Results from Damaged Rene 41 after 3000-Hr Exposure at 95% Humidity.

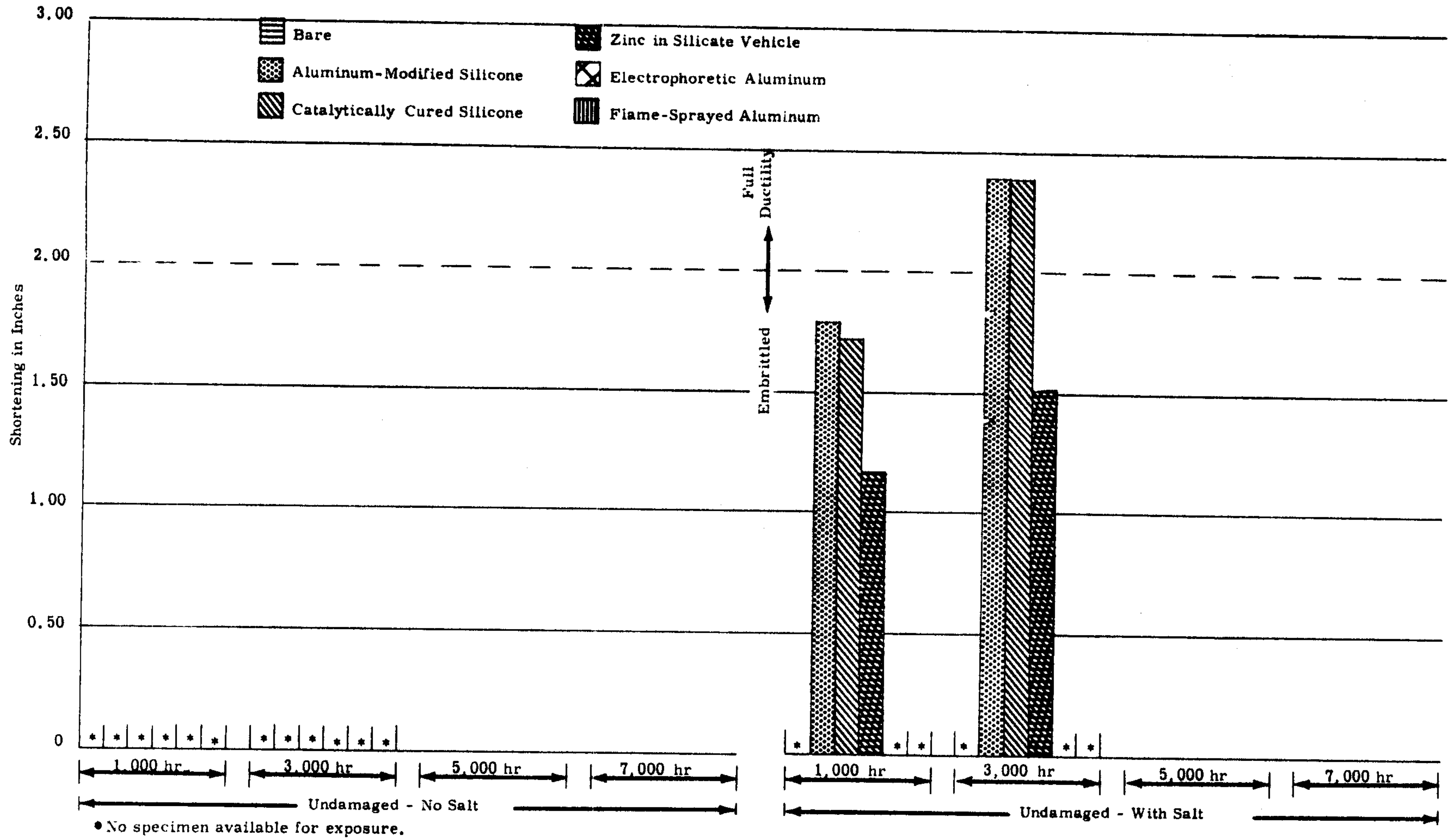


Figure 14. Bend-Ductility Results from Undamaged Ti-8Al-1Mo-1V after 3000-Hr Exposure at 95% Humidity.

CONCLUSIONS

On the basis of the results from the 1000-hr and 3000-hr exposures, we draw the following conclusions:

1. The AM 350 SCT stainless steel substrate will require protection from stress corrosion in salt-laden humid environments.

2. The inherent ductility of the solution-treated-and-aged Rene 41 used in these experiments is inconsistent to the extent that its vulnerability to stress corrosion within 3000 hr is obscured.

3. Duplex annealed Ti-8Al-1Mo-1V alloy will require protection from stress corrosion when exposed to dry salt at 550° F. Its vulnerability to stress corrosion in salt-laden humid environments has not yet been determined in this program.

4. Aluminum-Modified Silicone on AM 350 and Ti-8-1-1 substrates provides excellent protection against stress corrosion for 3000 hr, either under dry, 550° F conditions or humid, 95° F conditions. It probably provides protection for Rene 41 superalloy also, but its effects were obscured because of inconsistencies in the inherent ductility of this substrate.

5. Catalytically Cured Silicone provides excellent protection on AM 350 and Ti-8-1-1 substrates in the humid, 95° F environment. Its apparently excellent protective qualities at 550° F are misleading because it quickly shredded from all three substrates when exposed to the elevated temperature. Its effects on Rene 41 superalloy were obscured by the inconsistencies in the inherent ductility of this substrate.

6. Zinc in Silicate Vehicle apparently has a large deleterious effect on the ductility of the Rene 41 and Ti-8-1-1 substrates regardless of the exposure conditions. It provides some protection on AM 350 in the humid, 95° F environment but is not as effective as Aluminum-Modified Silicone and Catalytically Cured Silicone, especially in the presence of scratch damage. In the dry, 550° F environment the zinc coating has no deleterious effects on AM 350, but its protective qualities are obscure because this environment was not harmful to the bare substrate.

7. The Electrophoretic-Aluminum coating did not provide significant protection for Ti-8-1-1 in the 550° F hot-salt environment. However, the failure to protect might have been caused by the uncoated edges or the blistered condition of the coating. This coating was not evaluated on other substrates or under other exposure conditions.

8. The Flame-Sprayed Aluminum coating provided some protection for Ti-8-1-1 in the 550° F hot-salt environment. It was more effective in this regard than Electrophoretic Aluminum or Zinc in Silicate Vehicle but much less effective than Aluminum-Modified Silicone or Catalytically Cured Silicone. However, its effectiveness might have been reduced because the inside surfaces of the specimens were essentially uncoated. This coating was not evaluated on the other substrates or under other exposure conditions.

FUTURE WORK

During the next quarter, bend-ductility evaluations will be performed on specimens from the 5000-hr exposure interval which ended on 14 September. When necessary for additional clarification of results, we shall make metallographic examinations of selected specimens from the 1000-hr, 3000-hr, and 5000-hr exposures. In addition, we shall collect samples and perform a preliminary evaluation of new coatings that have been brought to our attention. As soon as the 5000-hr specimens have been evaluated, we shall arrange to visit approximately three of the leading aircraft manufacturers to discuss all aspects of the coatings problem with them.

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24 September, 1965
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