EFFECTS OF COMMERCIALLY AVAILABLE PROTECTIVE COATINGS ON STRESS-CORROSION PROPERTIES OF SUPERSONIC-TRANSPORT SKIN MATERIALS

FINAL SUMMARY REPORT

TO

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
For the period between 1 June, 1964, and 31 January, 1966

Contract No. NASr-117

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22 March, 1966
7685-1417-XV

N 23647

SUMMARY

The purpose of the work reported herein was 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 experiments consisted of the exposure of self-stressed specimens, bare and coated, scratch damaged and undamaged, to environments of hot salt at 550° F and humid salt at 95° F for durations up to 7000 hr. The substrates investigated were titanium alloy Ti-8-1-1 (titanium with 8% aluminum, 1% molybdenum, 1% vanadium), AM 350 SCT (a high-strength stainless steel in the sub-zero-cooled and tempered condition), and Rene 41 (a nickel-base superalloy in the heat-treated and aged condition). The coatings investigated on all three substrates were designated as Aluminum-Modified Silicone, Catalytically Cured Silicone, and Zinc in Silicate Vehicle. Two coatings, designated as Electrophoretically Deposited Aluminum and as Flame-Sprayed Aluminum were evaluated on the titanium-alloy substrate only.

The effects of the exposures were evaluated by means of visual examination, bend-ductility tests, and metallographic examination. In addition to the experimental work, a personal-visit survey was made of five leading airframe manufacturers to determine their latest opinions about SST coatings. The experimental results were interpreted with respect to the findings from this survey. A preliminary evaluation was made on five additional coatings that had not been included in this or the previous investigations. Author

The results from this investigation and survey have led to the following conclusions:

1. Duplex annealed Ti-8-1-1 is readily susceptible to stress corrosion from hot salt at 550° F. It is apparently not attacked by humid salt at 95° F within 7000 hr. As measured by bend-ductility shortening, complete deterioration from stress corrosion occurred within 1000 hr of exposure to the hot salt.

2. The AM 350 SCT stainless steel is resistant to stress-corrosion attack from hot salt at 550° F but, depending upon other undefined conditions, can be rapidly attacked by humid salt at 95° F.

3. Rene 41 is apparently resistant to stress-corrosion attack from salt in either the hot condition at 550° F or the humid condition at 95° F. However, its inherent ductility, in the heat-treated condition used for these experiments, is frequently as low as salt-embrittled specimens of the other substrates.

4. Because of the demonstrated potential danger from stress corrosion of titanium alloys in the presence of hot salt, all titanium areas of the SST will probably require protection with a suitable coating regardless of whether coatings are needed for other reasons such as control of IR emissivity.

5. All of the coatings evaluated in this investigation are apparently capable of preventing stress corrosion of the three substrates evaluated. However, because of concomitant requirements for SST service, only Aluminum-Modified Silicone and Catalytically Cured Silicone appear to be satisfactory unless operating temperatures remain below 450° F.

6. If SST operating temperatures remain below 450° F, other coatings previously screened out in earlier investigations of this program (polyimide coatings, for example) might become leading candidates for SST service.

7. Because of the need for coatings to provide high IR emissivity on large areas of the SST outer skin, white-pigmented Catalytically Cured Silicone is the most feasible commercially available coating. In areas not subject to IR control, Aluminum-Modified Silicone is probably superior, especially in engine areas and on engine components exposed to temperatures up to 900° F.

8. Because of slight but significant differences in formulations from one supplier to another, coatings for SST service must ultimately be evaluated on the basis of supplier or trade name rather than on the basis of fundamental type used in this investigation.

9. In order to provide standard requirements and test methods for future evaluations and screening of attempted coating improvements, it would be desirable to develop a target specification for supersonic-aircraft coatings. Because of the difficulties in compromising differences of opinion among the various aircraft manufacturers and coating manufacturers, such a specification must probably be developed by a neutral agency under Government sponsorship.

10. The current attempts to improve the stress-corrosion resistance of titanium alloys by special heat treatments of finished sheet should be supplemented by investigations of the feasibility of providing more homogeneous structures by means of complementary heating and rolling treatments in the early stages of sheet production.

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INTRODUCTION

Purpose:

The purpose of this project was to determine whether certain commercially available coatings, selected on the basis of earlier work at Southern Research Institute (1, 2)[1] will retard or prevent stress corrosion of substrate alloys that are promising candidates for use as the outer skins on supersonic-transport aircraft (SST). This work consituted the third major phase of a larger program, the purpose of which was to determine the feasibility of using commercially available coatings for corrosion protection of SST outer skins.

Scope:

This final report includes the work performed during the period between 1 June, 1964, and 31 January, 1966. Although stress-corrosion susceptibilities as indicated by bend-ductility tests comprise the majority of the data, the report also includes results from a metallographic examination of exposed specimens, an interview survey of appropriate personnel employed by airframe manufacturers, and a preliminary evaluation of additional coatings brought to our attention after the completion of the earlier work. The results are condensed and summarized for clarity in the body of the report, but detailed data has been included in the Appendix for the benefit of those who might wish to obtain the data from individual specimens.

Background:

The development of supersonic-transport aircraft involves several design problems, among which is the frictional heat that is expected to be generated at the skin of the aircraft at cruising altitudes and supersonic speeds. The temperatures generated by the frictional heat were earlier estimated to

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[1] The numbers in parentheses refer to the bibliography.

approach a maximum of 650° F, but more recent designs have reduced the maximum temperature estimate to the vicinity of 500 to 550° F. At these temperatures the use of conventional clad aluminum alloys as wing and fuselage skin materials is impractical. Consequently, substitute materials such as stainless steels, maraging steels, alloy structural steels, and nonferrous heat-resistant alloys (titanium and nickel-base superalloys) are being evaluated as potential SST skin materials. Although these materials can be expected to meet the strength requirements at elevated temperatures up to 650° F, other problems such as harmful discoloration, oxidation, and other forms of corrosion arise. Therefore, if these materials are to be used, some type of protective coating must be employed to prevent corrosion in service.

There are other less critical but potentially important reasons for coating the SST. Coatings will be needed to provide for identification, visibility, and decorative markings, and to protect the leading substrate materials from oxide discolorations that form when they are heated to SST temperatures in air. Although some of these discolored films are reasonably attractive gold or blue colors, it is doubtful that they would remain uniform under the influence of the temperature gradients to which they will be exposed. They will, therefore, have the appearance of the heat tints that they are, and will likely have a discouraging psychological effect on civilian passengers. There is a possibility that the oxide films, or ruptures in them, will also provide the type of discontinuities that can nucleate corrosion pits or cracks. Regardless of the need for corrosion protection, then, coatings might be needed only for providing a satisfactory appearance.

At first thought it would appear that coatings for the SST would not present a difficult problem. Besides the many available corrosion-protective coatings that are effective at weathering temperatures, there are many that have been developed to effectively protect refractory metals at greatly elevated temperatures on the order of 2500 F. The problem arises, however, because the weathering-temperature coatings tend to deteriorate at elevated temperatures and the high-temperature coatings do not perform well for extended periods of time, even in the lower temperature range (-100° to 650° F) in which the SST is expected to operate. Therefore, SST service requirements pose a considerable challenge to coatings technology, and any coating that might meet the corrosion and appearance requirements must also meet the following operational criteria:

1. It must be serviceable for the complete life of the SST or be reasily and economically replaceable during regular overhaul periods. The minimum life of an SST is expected to be ten years, including at least 30,000 hours of flying time.

2. It must withstand temperatures ranging from subzero to approximately 550° F without deteriorating.

3. It must be flexible enough to withstand distortions of the substrate caused by assembly operations, static loading, vibrational loading and exposure to fluctuating temperatures.

4. It must be resistant to abrasion, erosion, and other mechanical damage.

5. It must not deteriorate under exposure to normal weathering and ultraviolet radiation from the sun.

6. If the coating is a type that must be periodically replaced, its application procedure must be simple and economical enough to be practical under field conditions.

7. In addition to the known requirements, the coating will undoubtedly be expected in the future to meet limitations with respect to first cost, weight, emissivity, color, resistance to fuels and cleaners, surface smoothness, and adaptation to manufacturing processes performed on the substrate subsequent to coating.

Early in 1962 the NASA Special Committee on Materials Research for Supersonic Transports recognized the coatings challenge and engaged Southern Research Institute to make a study of the feasibility of using commercially available coatings for protection of skin materials on the SST. In one investigation (1) it was found that several commercially available coatings applied to a nonstainless substrate (18% Ni maraging steel) appeared to furnish adequate corrosion protection under static conditions chosen to simulate the expected service conditions in SST aircraft. The most promising of these coatings were further evaluated (2) for their protective qualities and their compatibility with five types of candidate skin alloys by subjecting the coated substrates to tension tests and simulated service conditions that combined cyclic loading with exposure to heat, a corrosive medium, and an aircraft cleaning compound. These exposures eliminated some of the previously promising coatings from further evaluation and showed that although the titanium, stainless, and nickel-base alloys were resistant to surface corrosion in the bare condition, they were all subject to heat-tint discolorations when heated to 650° F in air. Consequently, it is likely that some form of coating on the aircraft skin will be desirable regardless of whether corrosion protection is necessary.

General surface corrosion, then, is not expected to be a serious problem with the heat-resistant substrates, but investigations made at NASA's Langley Research Center and elsewhere (3, 4) have shown that some of the titanium alloys among those favored for use on critical skin areas of SST aircraft are susceptible to stress-corrosion attack from thick salt deposits (0.004 to 0.006 in.) and temperatures in the vicinity of 550° F. Other investigations in the same laboratory and elsewhere have shown that the stress-corrosion attack can also occur with very thin salt deposits on the order of those caused by human finger prints. Therefore, even though evidence of stress-corrosion failure has not yet been observed on existing military aircraft employing titanium alloys in their engines and structure, hot-salt stress corrosion and its prevention have become important factors in the design considerations for SST aircraft. The stainless steels and superalloys have not yet been found to develop stress corrosion from hot-salt exposures, but stainless steels and many other alloys are known to be susceptible to stress-corrosion attack under various atmospheric conditions.

The promising protective coatings under study at Southern Research Institute would be expected to prevent or reduce stress-corrosion attack at either atmospheric or elevated temperatures while they remain intact. On the other hand, if these coatings become porous or sustain mechanical damage such as cracks, nicks or scratches, they could allow the occurrence of local attack that might initiate stress-corrosion even on materials that are resistant to stress-corrosion in the bare condition. Therefore, it is important to determine the effects of coatings on the stress-corrosion characteristics of the alloys under consideration for SST service.

PROCEDURE

Materials:

The investigation was performed with three substrate materials and five coating materials. The substrate materials included a titanium alloy (Titanium - 8%Aluminum - 1%Molybdenum - 1%Vanadium), a high-strength stainless steel (AM 350), and a nickel-base superalloy (Rene 41). The titanium alloy (Ti-8-1-1) was received in the duplex-annealed condition and the other two substrates in the annealed condition. The titanium alloy was used in the as-received condition and the other two substrates were heat treated at Southern Research Institute after the specimens had been partially constructed. Pertinent data on the three substrates are summarized in Table I.

The five coatings chosen for the stress-corrosion study were those that had shown the most promise from the results of the earlier work previously reported (1, 2). Because of the necessity for holding the number of specimens to a reasonable quantity consistent with the size of the program, only three of the coatings were evaluated on each of the three substrates. The other two coatings were evaluated on the titanium-alloy substrate only. The coatings

Table I

Mill Data for Substrate Materials

Material	Ti-8-1-1	AM 350	Rene 41
Mill Chemical Analysis, wt. %:			
Carbon	0.023	0.087	0.095
Iron	0.06	Bal.	1.13
Nitrogen	0.011	0.098	-
Aluminum	7.8	-	1.55
Vanadium	1.0	-	-
Molybdenum	1.0	2.74	9.46
Hydrogen	0.006	-	-
Titanium	Bal.	-	3.17
Manganese	-	0.84	0.03
Silicon	-	0.30	0.05
Sulphur	-	0.007	0.005
Phosphorous	-	0.023	-
Chromium	-	16.71	18.40
Nickel	-	4.53	Bal.
Boron	-	-	0.0074
Cobalt	-	-	10.48
Thickness Range, In.	0.040 - 0.043	0.036 - 0.040	0.035 - 0.039
Mill Heat Treatment for Mechanical-Property Tests	1450° F 15 min., A. C.	1710° F 45 Min/In. Th., W.Q. -100° F, 3 Hr, Air Warm, 850° F, 2 Hr, A. C.	1975° F 30 Min, A. C. 1400° F 16 Hr, A. C.
Mechanical Properties:			
Hardness, Rockwell C	-	44	37
Ult. Tensile Str., psi	150,100	213,500	152,800
Yield Strength, psi	133,800	176,000	112,500
Elongation, %	14.0	18.0	19.8

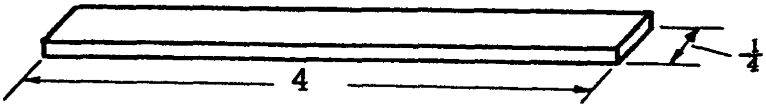
applied to each of the substrates were Aluminum-Modified Silicone, Catalytically Cured Silicone, and Zinc in Silicate Vehicle. Those applied only to the titanium-alloy substrate were Electrophoretically Deposited Aluminum and Flame-Sprayed Aluminum. A brief description of each of these coatings and the additional coatings that were given a preliminary evaluation for meeting SST requirements is included in Table X in the Appendix.

With the exception of Electrophoretically Deposited Aluminum, all of the coatings were applied to the substrates by the coating suppliers subsequent to the construction of the specimens. The specimens coated with Electrophoretically Deposited Aluminum were made from a sample lot of material that had been submitted by the British Iron and Steel Research Association for use in the previous investigation. Although these samples were unsatisfactory in the previous program because of poor adhesion and blistering (the process had not been sufficiently developed for titanium substrates), subsequent experiments performed at Southern Research Institute indicated that the adhesion could be greatly improved by a 1-hour heat treatment at 1115° F. Therefore, the specimens of Electrophoretically Deposited Aluminum were different from the others in several ways: the substrate was from a different lot of material, the substrate was subjected to possibly harmful heat exposures subsequent to its duplex annealing treatment, the edges were exposed because the substrate was coated prior to the construction of the specimens, and small blisters were present in the coating.

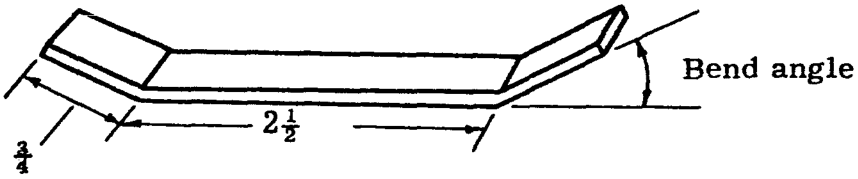
Preparation of Specimens:

Self-loaded-type stress-corrosion specimens as described by Braski and Heimerl (4) were made from each substrate. They had the configuration shown in Figure 1, with the gap opening ("d + 2t" distance) set to produce a load in the outer fibers amounting to 70% of the ultimate tensile strength of the substrate involved. The bend angles necessary to produce the desired load when two identical strips are spot welded as shown in Figure 1 were determined by applying strain gages to sample specimens. Subsequent specimens were then made to the proper configuration as determined on the sample specimens. At least two replicate specimens were evaluated for each set of experimental conditions.

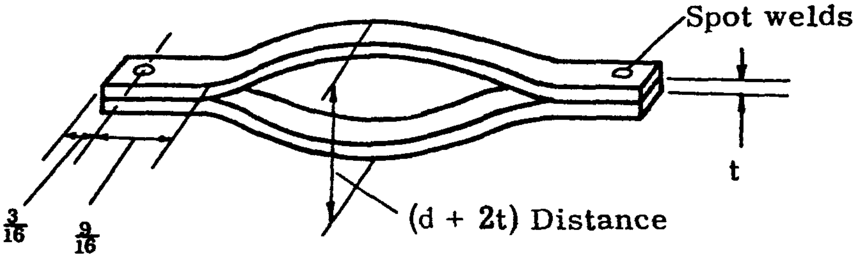
As indicated in Figure 1, the desired stress (within the elastic range) in the outer fibers of the stress-corrosion specimen is produced by bending tabs at each end of the two strips that constitute the complete specimen, and welding these tabs together to produce a bowed specimen. The applied stress is greater as the distance across the bow is greater, and measurement of this distance (d + 2t) provides a facile method for determining the existing stress. The relationships between the stress and the bow distance can be derived from elastic stress-strain formulas and from formulas for simple circular bending as shown in Figure 2.



(a) Machined strip.

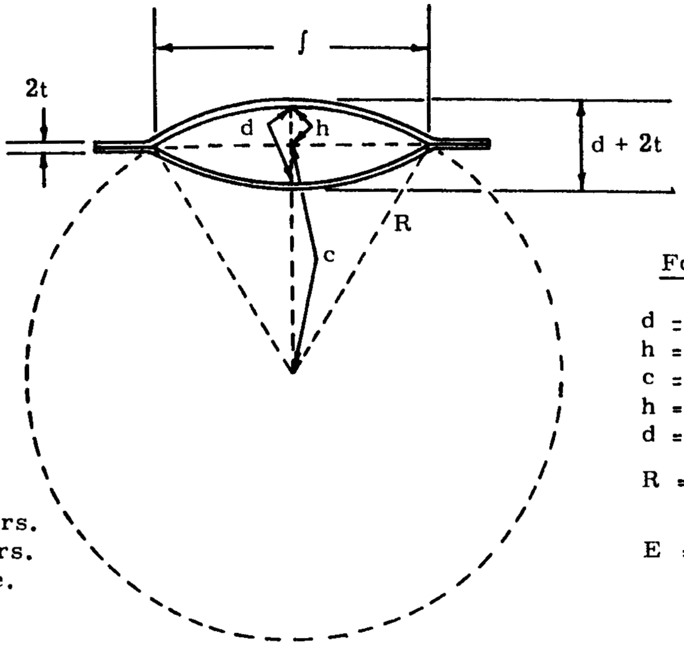


(b) Strip with ends bent.



(c) Completed specimen.

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



Symbols

t = thickness of strip.
d = inside bow distance.
h = rise of the arc.
R = radius of curvature.
c = distance to chord.
$\int$ = chord length.
E = elastic modulus.
σ = tensile stress on outer fibers.
ϵ = tensile strain in outer fibers.
$d + 2t$ = specimen bow distance.

Formulas

$d = 2h$
$h = R - c$
$c = 1/2 \sqrt{4R^2 - \int^2}$
$h = R - 1/2 \sqrt{4R^2 - \int^2}$
$d = 2R - \sqrt{4R^2 - \int^2}$
$R = \frac{tE}{2\sigma}$
$E = \frac{\sigma}{\epsilon}$

CALCULATIONS OF SPECIMEN BOW DISTANCES

Property	AM 350 SCT	Rene 41	Ti-8-1-1
E [1]	30.3×10^6	31.6×10^6	18.5×10^6
σ [2]	107,000 psi	149,500 psi	105,100 psi
ϵ [3]	0.00354	0.00473	0.00569
t [1]	0.0385	0.0365	0.0425
R [3]	5.43	3.86	3.74
$\int$ [4]	2.5	2.5	2.5
d [3]	0.29	0.42	0.43
$d + 2t$	0.367	0.493	0.515

[1] Nominal value for the materials involved.
[2] Chosen to be 70% of nominal ultimate tensile strength.
[3] Calculated by the formulas given. All dimensions are in inches.
[4] Value chosen for the gage length of the specimens.

Figure 2. Relationships between stress and specimen configuration.

Although the bow distance, d + 2t, is subject to simple calculation for a given stress, the corresponding bend angle of the tabs can only be estimated because of springback during the bending of the tabs and the amount of elastic springback occurring at the bend when the tabs are spot welded together. Calculation of the bend angles of the heat-treatable substrates was further complicated because the tabs were bent prior to heat-treatment. Therefore, the bend angles were determined by experiment.

The bend-angle determinations were made with 6 strips from each of the 3 substrate materials. Pairs of strips from each of the substrates were bent to each of 3 angles: 15, 25, and 35 degrees as clamped in the bending fixture (to eliminate consideration of springback).

Type SR-4 strain gages, 1/8-in. long, were attached to the strips, and zero readings of strain were taken before the specimens were assembled. Attempts were made to assemble the specimens in the welding fixture but it was found that the strain gages interfered with the fit in the fixture. While attempting to use the fixture, the two AM 350 specimens with the larger bend angles were strained beyond the yield point as indicated by a slight permanent bow remaining in the strips after they were removed from the fixture unwelded. Therefore, these and the remaining calibration specimens were assembled without welding by carefully aligning the specimens by hand and clamping the tabs together with vise-grip clamps. Strain-gage readings were taken with the specimens in the clamped condition to determine the strain produced in each substrate material by the different tab angles. The strain readings were then converted to stress by multiplying the strain by the modulus of elasticity of each of the materials.

The stresses produced by the various tab bend angles are shown by the plots in Figure 3. The results from the titanium alloy exhibited only slight scatter and indicate a linear relationship between the tab bend angles and the stresses produced. A reading from the best straight line through the plotted points shows that a bend angle of 30.5 degrees should produce the desired stress of 105,100 psi in the titanium alloy. This result is in close agreement with that of Braski and Heimerl (4) in which a stress of approximately 100,000 psi was produced by a bend angle of 30 degrees.

The results from the Rene 41 specimens also had slight scatter, but the largest bend angle caused a permanent set to take place which prevented an accurate determination of the stress by the elastic formula. Therefore, the calibration line was drawn between the two accurate points on the assumption that the linear relationship would apply to the Rene 41 material as it had for the titanium alloy. These results show that a tab bend angle of 21.5 degrees should produce the desired 149,500 psi stress.

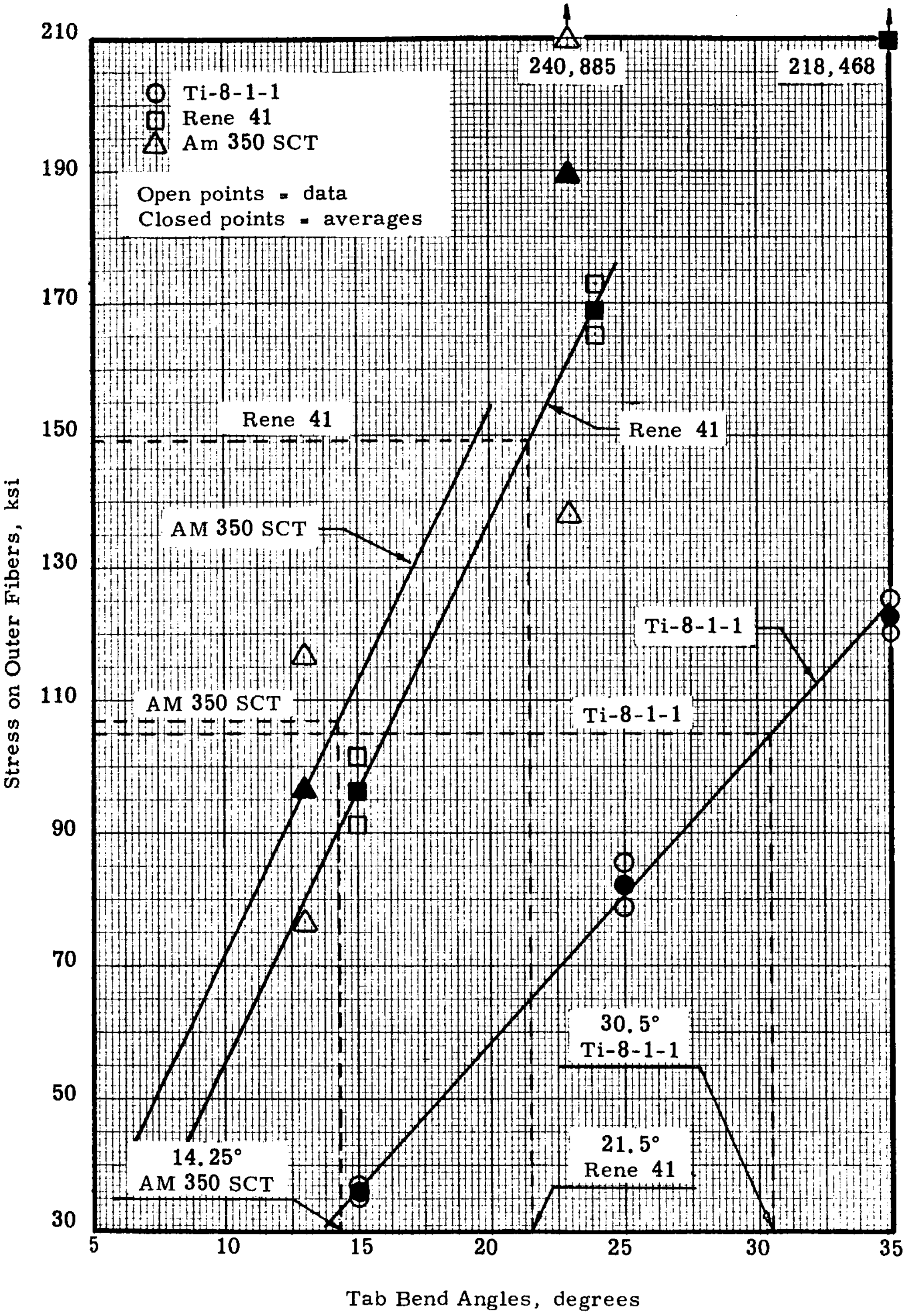


Figure 3. Tab bend angles versus resulting stress in assembled specimens.

Since the intermediate-angle and large-angle AM 350 specimens were strained beyond the elastic limit when attempts were made to weld them, only the small-angle specimen was available for the stress determination. Although the readings from this specimen had wide scatter, the specimen appeared to be satisfactory in all other respects and the average value was considered to be of satisfactory accuracy. Readings were also taken on the overstrained specimens, but the intermediate-angle specimen showed extremely wide scatter and the readings from the large-angle specimen were beyond the scale of the strain-gage instrument. Consequently, the calibration line for this material was drawn through the average value for the small-angle specimen and parallel to the line for Rene 41, the other heat-treat hardened material. Extension of this line shows that the intermediate and large angles (25 and 35 degrees) would produce stresses beyond the elastic limit of the material, probably explaining the difficulty encountered in attempting to fit these specimens in the welding fixture.

Although it would have been desirable to check the results in Figure 3 by constructing another set of specimens, it was decided that the progress of the project should not be subjected to the further delay that repeating the calibration would cause. Specimens constructed on the basis of Figure 3 might be slightly inaccurate, but the chosen stress level (70% of the nominal ultimate stress) is sufficiently high that stress corrosion can occur at considerably lower stress levels. An approximate check of the stresses produced by the chosen angles were obtained when the bow distances (d + 2t) were measured on the evaluation specimens.

Prior to the bending operation, the substrate strips were cleaned with Turco Vitro-Klene in accordance with the precleaning procedures listed in Tables II, III, and IV. These tables show the procedures used for AM 350, Rene 41, and Ti-8Al-1Mo-1V, respectively. The cleaned specimens were kept in clean containers and handled with cotton gloves during the bending to prevent fingerprint contamination of the two alloys that were subsequently subjected to heat treatment.

In accordance with the results of the bending calibration shown in Figure 3, the AM 350 strips were bent to 15 degrees, the Rene 41 to 22 degrees, and the titanium alloy (including those already coated with electrophoretically deposited aluminum) to 31 degrees. The tabs were bent to the next larger whole degree of the values shown in Figure 3 because fractions of a degree could not be accurately measured in the bending fixture.

The bent strips of AM 350 and Rene 41 alloys were heat treated in accordance with the procedures listed in Tables II and III, respectively. For handling convenience, the strips were bound with Chromel thermocouple wire into tight bundles approximately 2-in. wide and 1-1/2-in. thick. The bundles were water quenched after each heating treatment to speed cooling time and to keep the formation of oxide to a minimum.

Table II. Cleaning, Heat Treating, and Pickling Procedures for AM350 Alloy.

A. PRECLEANING:
1. Warm water rinse.[1]
2. Vitro Klene (Solution 1)[2], 75-95° C, 10 minutes
3. Warm water rinse.
4. Cold water rinse.
5. Wipe dry - DO NOT HANDLE WITH BARE HANDS.

B. HEAT TREATING:
1. Bundle parts with chromel thermocouple wire.
2. Adjust furnace to 1710° F and purge 30 minutes with argon at a flow rate of 5 CFH.
3. Place parts bundle in furnace on steel slab.
4. Hold at 1710° F for 45 minutes per inch of bundle thickness.
5. Water quench as rapidly as possible and shut off argon.
6. Subzero cool bundle in dry ice and alcohol (-100° F) for 3 hours.
7. Air warm bundle to room temperature.
8. Adjust furnace to 850° F.
9. Place parts bundle in furnace on steel slab.
10. Hold at 850° F for 2 hours.
11. Cool to room temperature.

C. PICKLING:
1. Scratch brush with stainless-steel brush wheel.
2. Warm water rinse.
3. Vitro Klene (Solution 1), 75-95° C, 10 minutes.
4. Warm water rinse.
5. Permanganate (Solution 2), 75-95° C, 1 hour.
6. Warm water rinse.
7. 20% Sulphuric (Solution 3), 75-95° C, 20 seconds.
8. Warm water rinse.
9. Nitric-hydrofluoric (Solution 4), 40-50° C, 2 minutes.
10. Warm water rinse.
11. Vitro Klene (Solution 1), 75-95° C, 10 minutes.
12. Warm water rinse.
13. 20% Nitric (Solution 5), 75-95° C, 1 minute.
14. Warm water rinse.
15. Cold water rinse.
16. Wipe dry - DO NOT HANDLE WITH BARE HANDS.

D. POSTCLEANING:
1 - 6. Same as steps C10 through C15.
7. Hot distilled-water rinse.
8. Hot-air dry - DO NOT HANDLE WITH BARE HANDS.

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[1] All rinses except distilled water are running-water rinses.

[2] Compositions of solutions are given in Table V.

Table III. Cleaning, Heat Treating, and Pickling Procedures for Rene 41 Alloy.

A. PRECLEANING:
1. Warm water rinse.[1]
2. Vitro Klene (Solution 1)[2], 75-95° C, 10 minutes.
3. Warm water rinse.
4. Cold water rinse.
5. Wipe dry - DO NOT HANDLE WITH BARE HANDS.

B. HEAT TREATING:
1. Bundle parts with chromel thermocouple wire.
2. Adjust furnace to 1975° F and purge 30 minutes with argon at a flow rate of 5 CFH.
3. Place parts bundle in furnace on steel slab.
4. Hold at 1975° F for 45 minutes per inch of bundle thickness.
5. Water quench as rapidly as possible.
6. Adjust furnace to 1400° F and purge 30 minutes with argon at a flow rate of 5 CFH.
7. Place parts bundle in furnace on steel slab.
8. Hold at 1400° F for 16 hours.
9. Water quench as rapidly as possible.

C. PICKLING:
1. Warm water rinse.
2. Vitro Klene (Solution 1), 75-95° C, 10 minutes.
3. Warm water rinse.
4. Permanganate (Solution 2), 75-95° C, 1 hour.
5. Warm water rinse.
6. 20% Sulphuric (Solution 3), 75-95° C, 30 minutes.
7. Warm water rinse.
8. Nitric-hydrofluoric (Solution 4), 40-50° C, 10 minutes.
9. Warm water rinse.
10. Permanganate (Solution 2), 75-95° C, 10 minutes.
11. Warm water rinse.
12. 20% Nitric (Solution 5), 75-95° C, 1 minute.
13. Warm water rinse.
14. Cold water rinse.
15. Wipe dry - DO NOT HANDLE WITH BARE HANDS.
16. Buff with stainless-steel compound and cotton wheel.
17. Wash with soap and water.
18. Hot water rinse.
19. Vitro Klene (Solution 1), 75-95° C, 10 minutes.
20. Hot water rinse.
21. Marble's etch (Solution 6), 40-50° C, 10 minutes.
22. Repeat steps C3 through C15.

D. POSTCLEANING:
1 - 6. Same as steps C9 through C14.
7. Hot distilled-water rinse.
8. Hot-air dry - DO NOT HANDLE WITH BARE HANDS.

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[1] All rinses except distilled water are running-water rinses.

[2] Compositions of solutions are given in Table V.

Table IV. Cleaning Procedure for Titanium-8Al-1Mo-1V.

A. PRECLEANING:
  1. Warm water rinse.[1]
  2. Vitro Klene (Solution 1)[2], 75-95° C, 10 minutes.
  3. Warm water rinse.
  4. Cold water rinse.
  5. Wipe dry - DO NOT HANDLE WITH BARE HANDS.

B. POSTCLEANING:
  1 - 4. Same as steps A1 through A4.
  5. Hot distilled-water rinse.
  6. Hot-air dry - DO NOT HANDLE WITH BARE HANDS.

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[1] All rinses except distilled water are running water rinses.

[2] Compositions of solutions are given in Table V.

Considerable delay in the preparation of specimens was caused by difficulties encountered in removing the thin film of oxide that formed on the AM 350 and Rene 41 alloys during heat treatment. Much time was consumed in experimenting with various pickling procedures found in the literature and suggested by the materials supplier. None of the procedures were found to be completely effective until augmented by mechanical treatments. The AM 350 strips were satisfactorily prepared by a combination of scratch brushing with a stainless-steel brush wheel and the chemical procedure listed in Table II. The compositions of the cleaning and pickling solutions are shown in Table V.

When the Rene 41 strips were scratch brushed, a film of stainless steel from the brush apparently deposited on the surface and stained the strips during the chemical treatments. This problem was overcome by buffing the strips with a stainless-steel cutting and coloring compound on a cotton buff wheel. Since the buffed surface was too smooth for coating adherence and resisted the action of the chemical treatments, the strips were etched in Marbles solution after buffing. The complete procedure for Rene 41 is listed in Table III.

The welding operation consisted of installing strips in each half of a fixture, clamping the fixture together with wing nuts on threaded studs, fitting the bottom half of the fixture over the bottom electrode, inserting the top electrode into the fixture by means of a drill-press crank arm, exerting slight pressure on the electrodes by means of the crank arm, and throwing the switch on a portable welder for sufficient time to complete the weld. The required welding times were 1-1/2 sec for Am 350, 1 sec for Rene 41, and 3/4 sec for Ti-8-1-1.

The welded specimens were cleaned after welding by the postcleaning procedures included in Tables II, III, and IV. The cleaned specimens were handled with clean cotton gloves to prevent contamination from fingerprints, and were packed in clean paperboard pint cartons for shipment to the coating suppliers.

The specimens were shipped to the coating suppliers for application of their coatings. The distribution of the specimens to the coating suppliers is presented in Table VI. All coatings, with the exception of Electrophoretically Deposited Aluminum and Flame-Sprayed Aluminum, were applied to the entire surface of the specimens. The Flame-Sprayed Aluminum coating supplier could not apply a uniform coating to the inner (concave) surfaces of the specimens' bowed members and consequently left them bare. The supplier did, however, apply a uniform coating to the outside (convex) surfaces of the specimens. The specimens coated with Electrophoretically Deposited Aluminum had bare edges because the specimens were constructed from previously coated sheet.

Table V. Compositions of Cleaning and Pickling Solutions.

Solution 1: Vitro Klene

43	grams	Vitro Klene Powder
957	ml	Water

Solution 2: Permanganate

180	grams	Sodium Hydroxide ($NaOH$)
50	grams	Potassium Permanganate (KMnO4)
770	ml	Water

Solution 3: 20% Sulphuric

200	ml	Sulphuric Acid (H_2SO_4)
800	ml	Water

Solution 4: Nitric-Hydrofluoric

100	ml	Nitric Acid (HNO_3)
30	ml	Hydrofluoric Acid (HF)
870	ml	Water

Solution 5: 20% Nitric

200	ml	Nitric Acid (HNO_3)
800	ml	Water

Solution 6: Marble's Etch

91	grams	Copper Sulphate ($CuSO_4$)
454	ml	Hydrochloric Acid (HCI)
455	ml	Water

Table VI. Distribution of Specimens to Coating Suppliers

Substrate Material	Number of Specimens	Coating Supplier	Coating
1. AM350	68	Dow Corning Corp.	Aluminum-Modified Silicone
	69	Humble Oil Co.	Zinc in Silicate Vehicle
	69	Air Force Mat. Lab.	Catalytically Cured Silicone
2. Rene 41	90	Dow Corning Corp.	Aluminum-Modified Silicone
	90	Humble Oil Co.	Zinc in Silicate Vehicle
	89	Air Force Mat. Lab.	Catalytically Cured Silicone
3. Titanium	73	Dow Corning Corp.	Aluminum-Modified Silicone
	72	Humble Oil Co.	Zinc in Silicate Vehicle
	73	Air Force Mat. Lab	Catalytically Cured Silicone
	26	Metco, Inc.	Flame-Sprayed Aluminum
	59[a]	BISRA	Electrophoretically Deposited Aluminum

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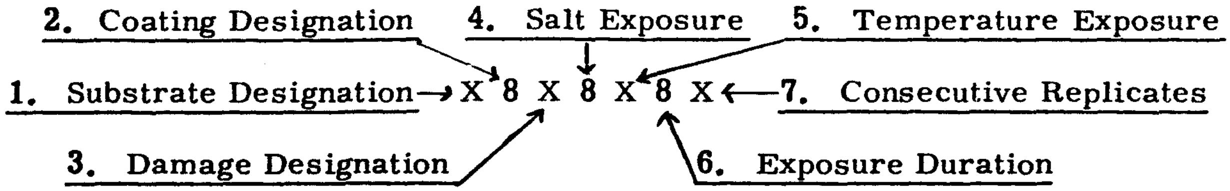
[a] Specimens were made from panels of this coating-substrate combination which were left over from last year's work.

Upon receipt from the coating suppliers, all specimens were identified and appropriate groups of specimens were conditioned for exposure by scratch damage and salt deposition. An alternating letter-number arrangement consisting of four letters and three numbers was assigned to each specimen as a means of identification. The illustration of the arrangement, presented in Figure 4, indicates the proper letter-number sequence and also reveals what each letter and number represents. The letter-number sequences were scribed on tags, which were secured at a tab end of the specimens. Metal (Monel) tags were used for the high-temperature specimens and paper merchandising-type tags were used for those specimens requiring humid exposures.

Appropriate groups of both bare and coated specimens from each substrate material were scratch damaged by forcing the specimens under a Rockwell diamond penetrator mounted in a drill press. The vertical position of the penetrator for each of the substrate materials was determined by placing a bare specimen on a specimen holder with its (d + 2t) distance (refer to Figure 1 c) in the vertical direction. The specimen-holder assembly was then positioned under the penetrator through the use of a guide rail mounted on the table so that the penetrator when lowered would touch a bowed member at its maximum point of stress. As the penetrator was lowered by the crank arm, a dial gage was activated by bearing against a surface mounted on the stationary table. When the penetrator touched the specimen, the dial gage was manually set to zero. The specimen-holder assembly was then removed and the penetrator was lowered 0.020 or 0.025 in. from the zero setting and locked into position. The specimen-holder assembly was then re-positioned along the guide rail and was forced forward under the penetrator, causing a scratch to extend transversely across the member's point of maximum stress. The orientation of the specimen with respect to the penetrator was then changed 90° and the operation repeated in order to produce a longitudinal scratch intersecting the transverse scratch. Once the penetrator was set and locked into position for a particular substrate, all specimens were scratched with the same setting. The 0.020-in. setting was used for all substrates except the substrate coated with Catalytically Cured Silicone. This coating-substrate combination required a 0.025-in. penetrator setting to insure penetration into the substrate. Since the settings were determined on bare substrates, the scratches extended through the coating and into the substrates of the coated specimens. The scratch depths into the substrate were estimated to be approximately 0.005 in. but, because of slight non-uniformity in geometry from specimen to specimen, the scratch depths varied to some extent.

Salt was applied to the appropriate specimens by momentarily hand dipping each specimen into a 20% aqueous salt solution at room temperature. Each specimen was held horizontally by the tab ends with its (d + 2t) distance (refer to Figure 1 c) extending vertically and was lowered into the salt solution so that only one member was wetted at a time. The solution was allowed to



Example: A 0 D 1 L 1 A

1. A = AM350, R = Rene 41, T = Titanium.

2. 0 = Bare, 1 = Aluminum-Modified Silicone, 2 = Catalytically Cured Silicone, 3 = Zinc in Silicate Vehicle, 4 = Electrophoretically Deposited and Rolled Aluminum, and 5 = Flame-sprayed Aluminum.

3. D = Damaged, U = Undamaged.

4. 1 = No Salt, 2 = Salt Deposit.

5. L = 100° F Humid Exposure, H = 550° F Dry Exposure.

6. 1 = 1,000 Hours, 3 = 3,000 Hours, 5 = 5,000 Hours, and 7 = 7,000 Hours.

7. A = First Specimen, B = Second Specimen, C = Third Specimen.

Figure 4. Specimen Identification System

contact the bowed member only on its convex side at the area of maximum stress. The specimen with its wetted member was then placed on a rack over a hot plate and thoroughly dried. After drying, this same procedure was repeated for the other bowed member. The specimens were subjected to the environmental exposures in this condition. Two completed specimens, without salt, one scratched and one unscratched, are shown in Figure 5.

Equipment:

The fixture used for bending tab ends on the specimen strips (refer to Figure 1 b) is shown in Figure 6. It consisted of a stationary base and a sliding member designed so that the assembly could be clamped together within the jaws of a bench vise. As illustrated by Figure 6 c, the bends were made by placing a strip on its edge in the opened fixture and then squeezing it between the sliding member and the anvil of the stationary member by closing the jaws of the vise.

The amount of bend was controlled by adjusting small screws in the face of the sliding part of the fixture. A scale divided in increments of 5 degrees was cemented to the base of the stationary part of the fixture so that the extent of the bend in each strip could be checked while the strip was in the fixture. The angles between multiples of 5 degrees were measured by estimating the appropriate distance between the 5-degree marks. It was observed that the springback of the titanium alloy amounted to approximately 6 degrees and the springback in the AM 350 and Rene 41 alloys (in the solution-annealed condition) to approximately 1 degree. No significant problems were encountered in the bending operation.

The heat treatments were performed in a Kanthal-wound resistance furnace with a silica-tube heating chamber supplied with an argon atmosphere and controlled with a Leeds and Northrop Micromax potentiometer recorder-controller. The subzero treatment (minus 100° F) was accomplished in a bath of dry ice and methyl alcohol contained in a Dewar flask.

The specimens were spot welded together with a Miller Portable Spot Welder, 220 volt, 2.5 KVA, with a 2.5-volt secondary open-circuit voltage. The specimens were clamped for welding in the fixture shown in Figure 7. The fixture was designed so that the adjustable arm at the center of each half of the fixture could be used to bow the strips to the extent necessary to cause the bent tabs to lie flat on the face of the fixture. Then, when the two halves of the fixture were clamped together by means of wing nuts on the threaded studs, the faying surfaces of the tabs would be in flat contact. In practice, however, it was found that the use of the adjustable arms to bow the strips sufficiently to lay the tabs flat caused permanent deformation in the strip as indicated by a

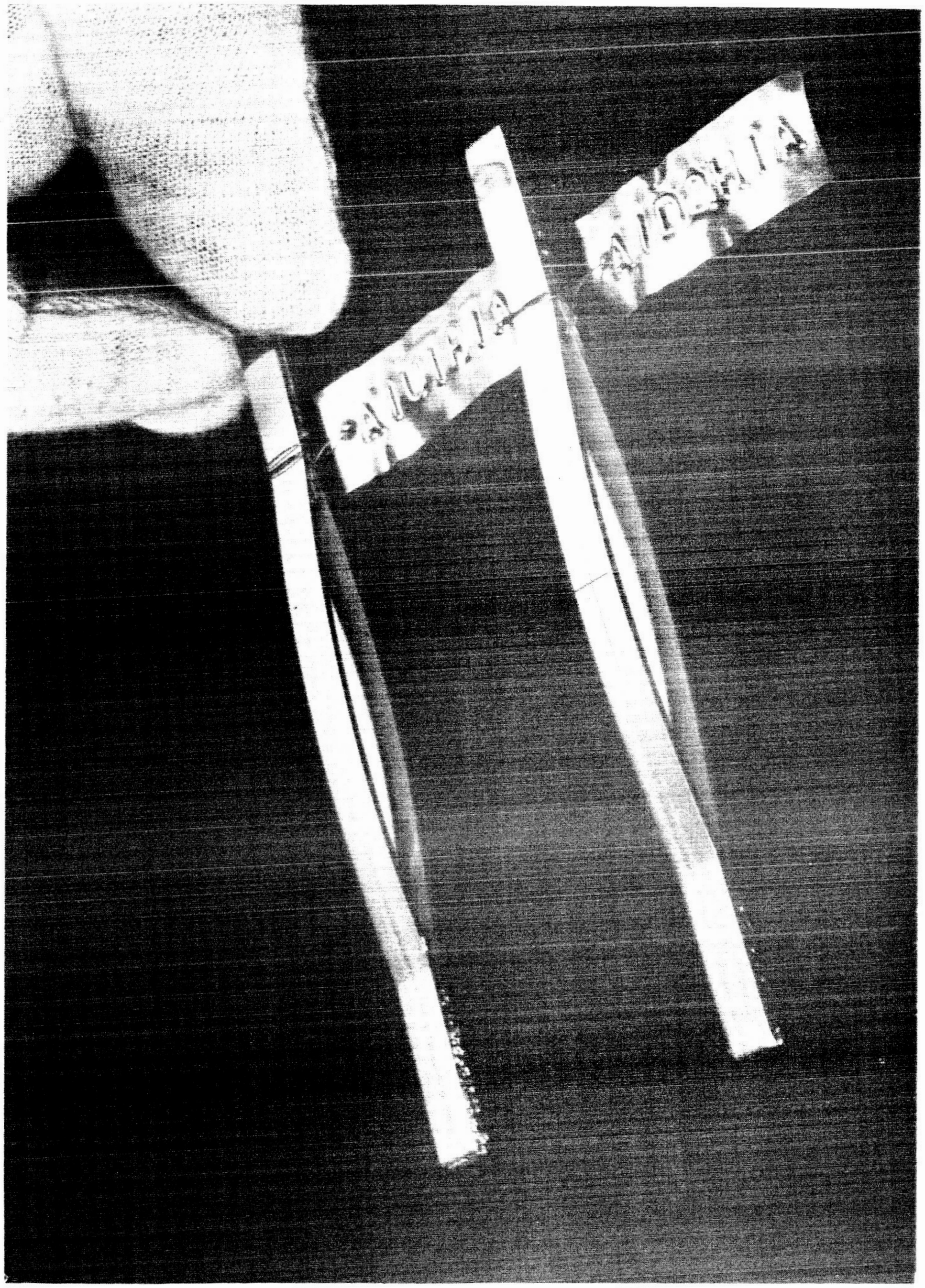
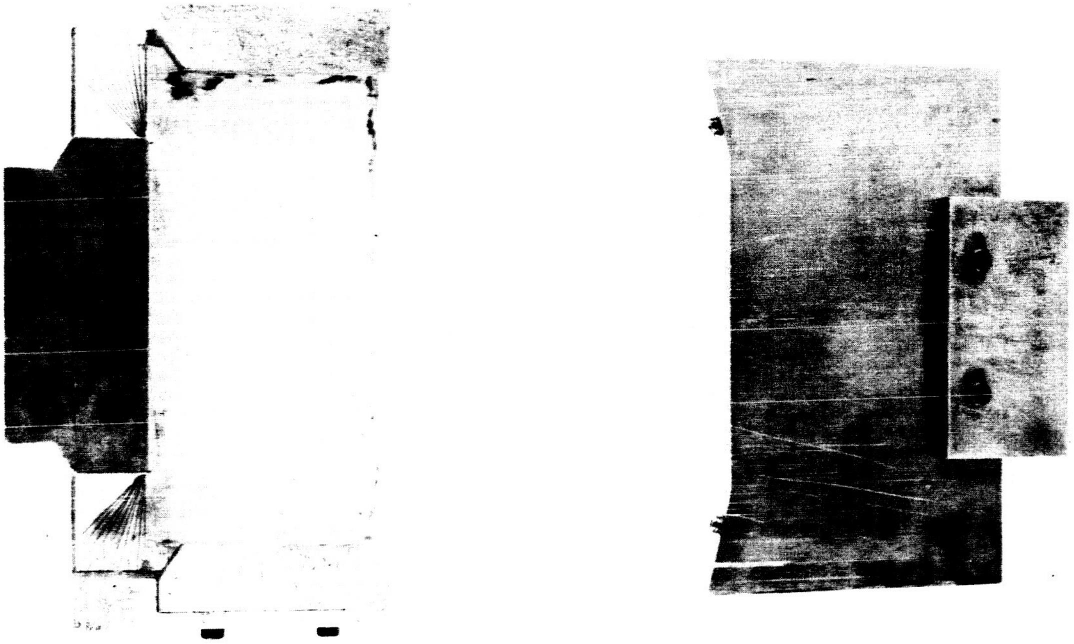
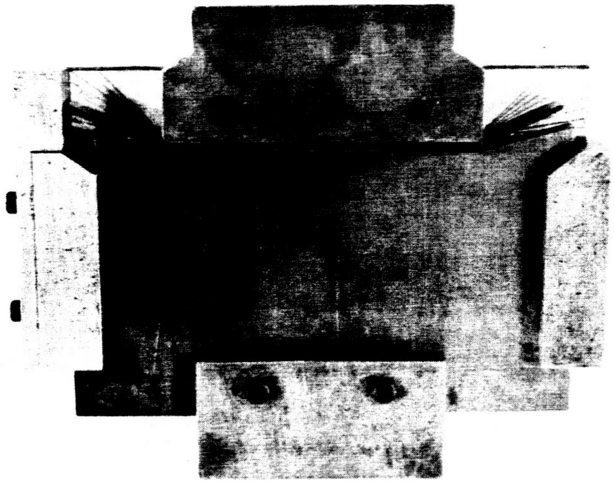


Figure 5. Completed Self-Stressed Specimens.



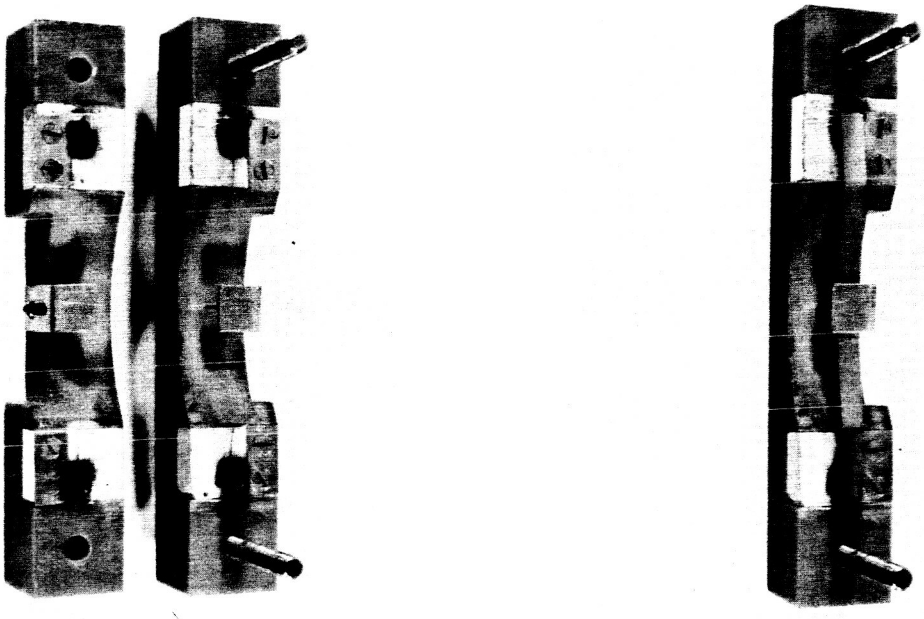
(a)
Stationary Base

(b)
Sliding Member



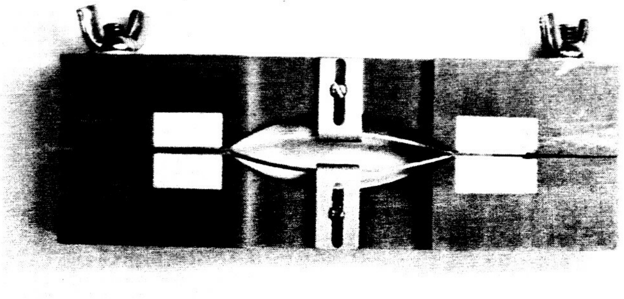
(c)
Assembled Fixture Showing Bent Strip

Figure 6. Bending fixture.



(a)
Disassembled Fixture

(b)
One Side with Strip Installed



(c)
Assembled Fixture with Specimen Installed

Figure 7. Spot-welding fixture.

small amount of retained bow when such strips were released unwelded from the fixture. Therefore, the arms were adjusted to produce only a partial bow, and the tabs were not forced flat until the two halves of the fixture were clamped together.

The body of the fixture was constructed from paper-infiltrated phenolic resin, which began to deteriorate and sink under the specimen tabs after subjection to the heat from the welding of the first trial specimens. The deterioration at that location caused the fixture to fail to force the tabs into close contact at the point of welding, resulting in faulty welds. This problem was overcome by machining a 1-in. wide and 3/8-in. deep slot across the full width of each half of the fixture at the tab locations and installing an insert of Supramica, a ceramic material manufactured by Mycalex Corporation of America. This material deteriorated slowly by charring, cracking, and spalling away of minute pieces, but it did not sink, and remained intact for sufficient time to complete the welding of all of the specimens.

The welding operation was performed in a bench-type drill press which provided a means of moving the upper electrode sufficiently to carry it in and out of the fixture. The electrodes consisted of lengths of copper ground wire that were hand filed to a diameter slightly smaller than the 3/16-in.-diameter openings in the fixture. These electrodes were connected to the electrode arms of the portable welder with braided flexible leads.

A fixture was used to damage appropriate specimens from each coating-substrate combination with a transverse and longitudinal scratch intersecting at the location of maximum stress. The fixture consisted of a bench-type drill-press stand to which a Rockwell diamond penetrator and an Ames 102 dial gage were attached. The Rockwell penetrator was anchored in a copper sleeve, which was fixed to the movable shaft of the drill press. By means of the drill-press crank arm, the movable shaft and the penetrator could be adjusted to any desired vertical position. A stationary table was placed under the penetrator for support of the specimen. One half of the previously described welding fixture was used as a specimen holder. It was ideally contoured to accommodate the bowed portion of each specimen and contained two locator stops at one end which assured repetitive alignment for all specimens.

It was necessary to construct fixtures capable of supporting appropriate groups of each coating-substrate combination during exposure to the elevated-temperature and the warm humid conditions.

A metal stand made from structural aluminum angles, 1/8 in. x 3/4 in. x 3/4 in., was constructed to withstand the elevated-temperature exposures. This stand supported eight sliding frames that held the specimens and made it convenient to remove the completed short-duration specimens without interrupting those specimens requiring longer exposures. The sliding frames were constructed from 1/8-in.-diameter welding rods joined together by spot welding. The assembled metal stand, loaded with specimens, is shown being placed in the exposure oven in Figure 8.

For the humid exposures, four rectangular plastic waste baskets were fitted for use as humidity chambers by means of a framework designed to support an appropriate number of specimens for a particular exposure interval. The framework consisted of two wood planks contoured to stand vertically in the containers, and drilled with holes through which the 1/8-in.-diameter stainless-steel welding rods that supported the specimens could be inserted. The top of each container was sealed with transparent plastic film held in place around the periphery of the container by means of rubber bands.

The bend-ductility evaluations of exposed specimens were performed in a fixture consisting of two clamping members fitted vertically in a manually operated hydraulic press. Both clamping members were mechanically secured to the press, the top clamp to the upper, stationary crosshead of the press by means of a threaded stud, and the bottom clamp to a steel supporting block that was "C" clamped to the lower, movable platen. A dial gage calibrated in 0.001-in. increments was mounted on a ring stand adjacent to the press. The height of the dial gage was adjustable on the ring stand and could be positioned so that the stem could bear against the movable platen of the press.

The configuration of the clamping members is shown in Figure 9. The members consisted of two 3-in. long, 1.25-in.-diameter round steel bars. Both members contained a 1-in.-deep, 0.100-in.-wide slot into which the specimen tab ends could be inserted. The edges of each slot were rounded to insure against breakage of the specimen at that point where the specimen deflected and bore against an edge of the slots. Two dial-pin locators were inserted across each slot in order to insure that lateral alignment of the specimen tab ends could be repeated from specimen to specimen. Four set screws were used in conjunction with each slot for the purpose of anchoring the specimens' tab ends. The set screws were arranged in pairs, with each pair extending through the sides on opposite sides of the slot. The operation of the bend-ductility assembly with a ductile specimen is illustrated in Figure 10.

Figure 8. Metal rack and specimens being placed in the 550° F exposure oven.

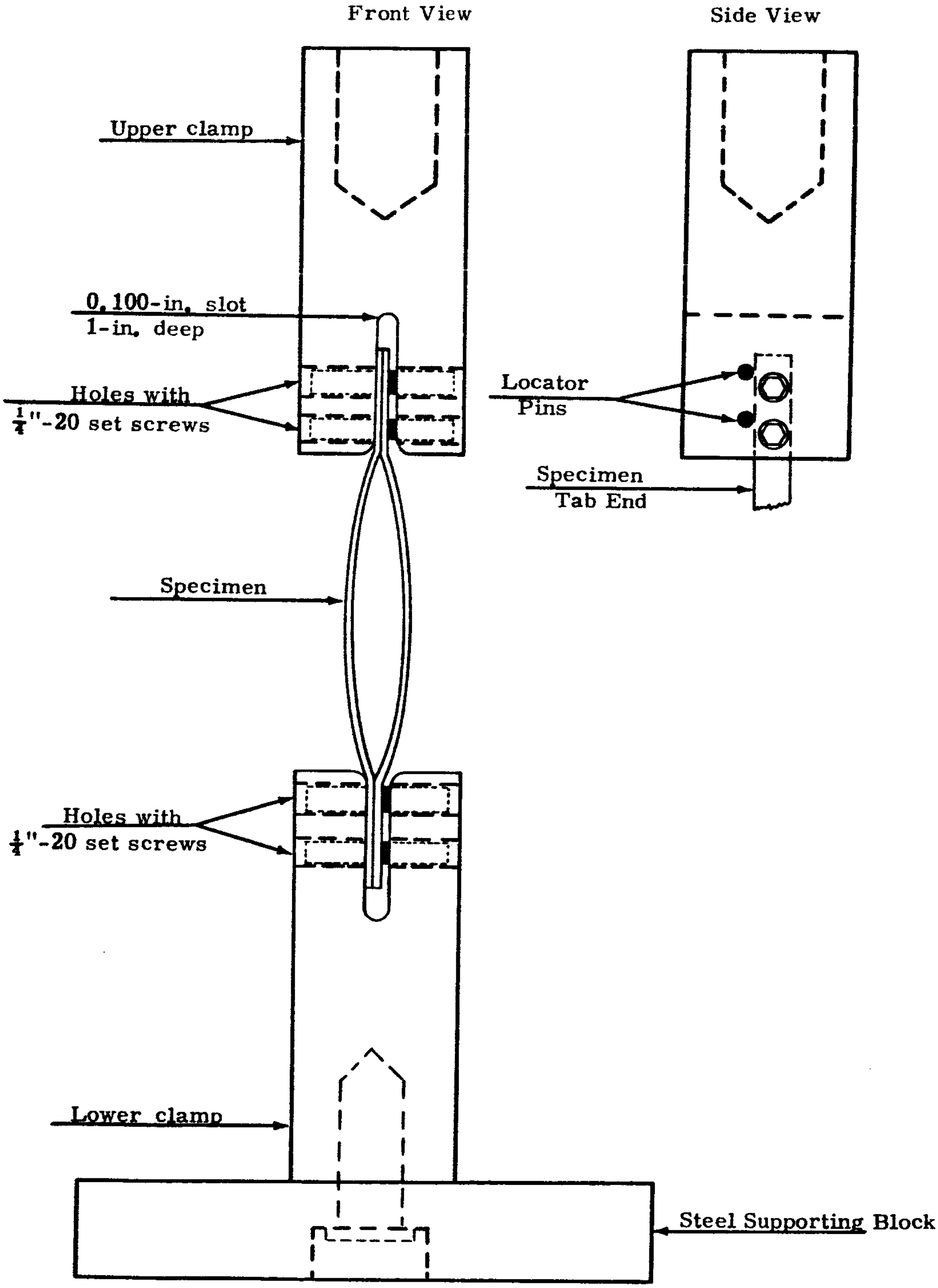


Figure 9. Schematic drawing of clamping members with specimen in place.

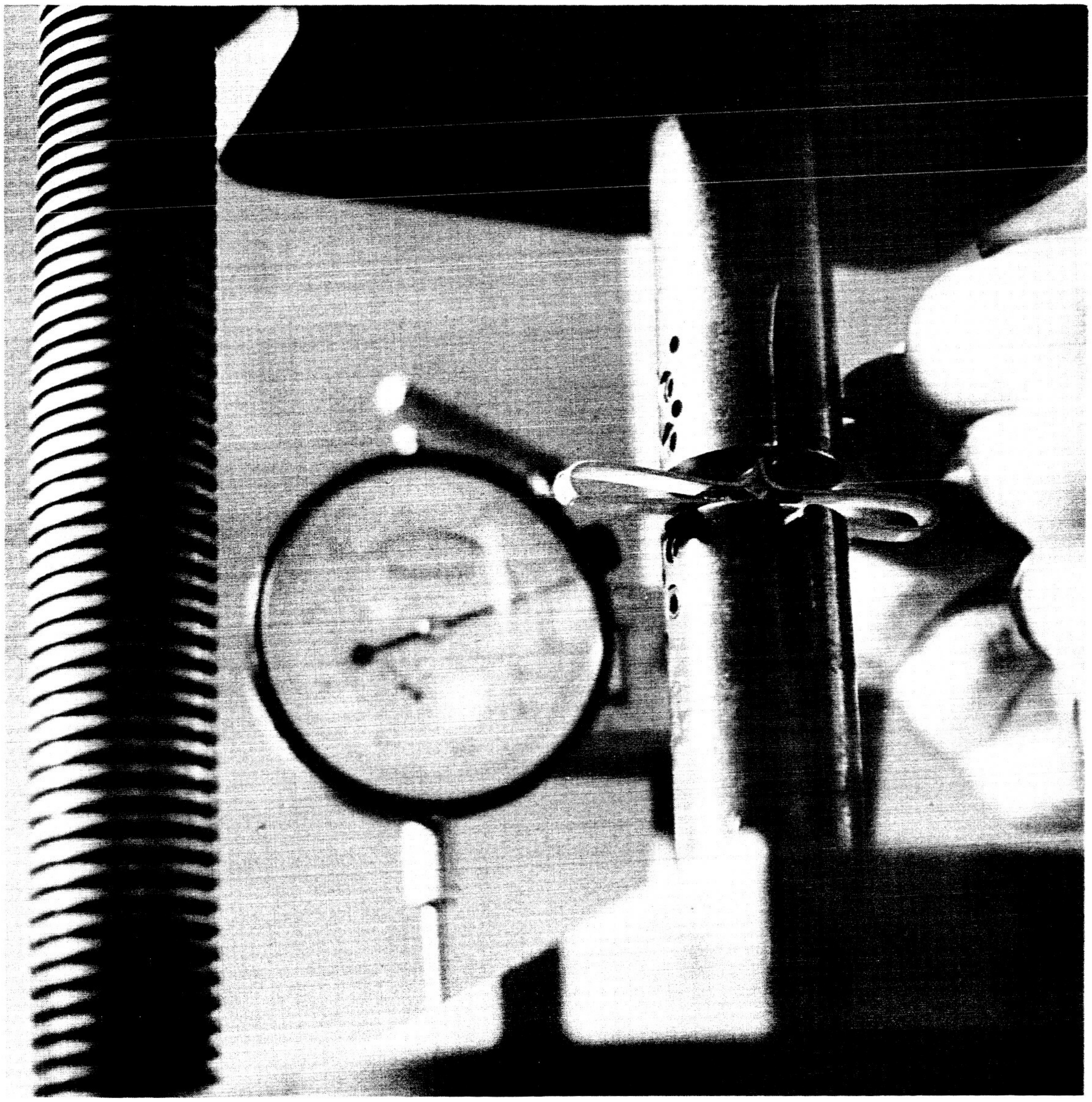


Figure 10. Bend-Ductility Assembly in use with a Ductile Specimen.

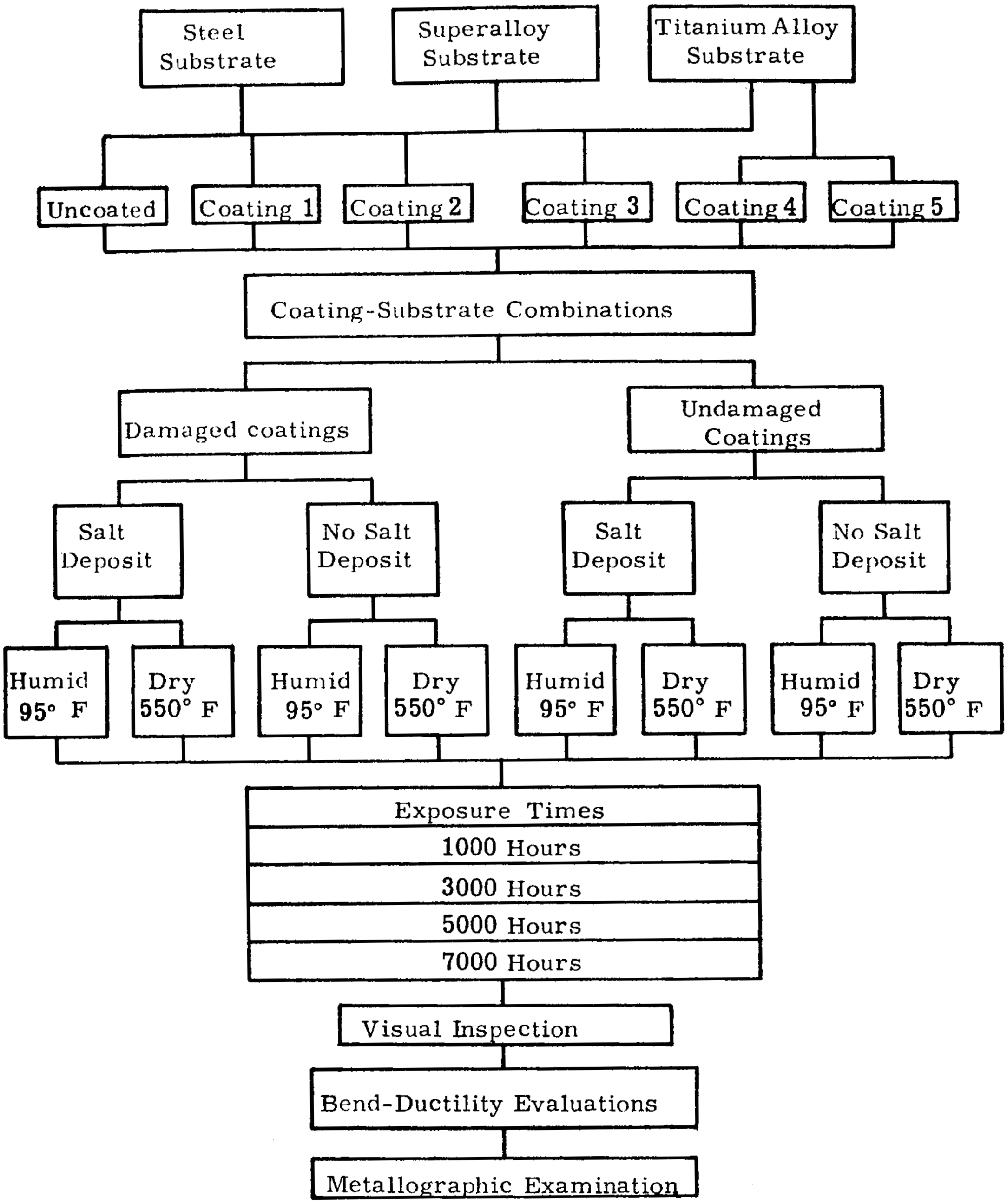
Exposure Environments:

A flow sheet illustrating the distribution of specimens to the experimental conditions is shown in Figure 11. As shown in this flow sheet, the various coating-substrate combinations were first divided into two groups; one group damaged with the previously described cross scratches and the other group remaining undamaged. Each of these two groups was further subdivided to provide groups on which salt was deposited and groups with no salt deposit. Specimens from each of the groups were then subjected to either a saturated-humidity environment at 95° F or a dry-air environment at 550° F.

With few exceptions, each combination of conditions was represented in four exposure durations consisting of 1000, 3000, 5000, and 7000 hr. Where specimen shortages precluded complete representation in all conditions and exposure durations, the available specimens were distributed in the longer exposure durations and in the conditions that were thought to be most detrimental to the stress-corrosion resistance of the substrates. The exposure conditions for each specimen are shown along with the detailed results of the bend-ductility tests in the Appendix in Tables XI through XXVI and in Figures 30 through 41.

All specimens to receive the high-temperature exposure were loaded on the metal frames, two of which were allotted for each exposure interval. The specimens were arranged horizontally in two parallel rows on each frame with each specimen supported at its tab ends. In order to accommodate the large number of specimens within the limited furnace space, the specimens were positioned on the frames with their (d + 2t) distances (refer to Figure 1 c) extending vertically. Only enough space between specimens to prevent contact was allowed. Of the two rows on each frame, one row contained salt-deposited specimens and the other contained unsalted specimens. The loaded frames were placed in the aluminum stand in such a manner that the salt deposited and unsalted specimens were in opposite vertical rows. These precautions were taken to prevent any dislodged salt grains from falling on unsalted specimens. The loaded stand was placed in a circulating-air electric oven set at 550° F.

The specimens for exposure at 95° F were placed on the supporting rods in each container with their (d + 2t) distances (refer to Figure 1 c) extending horizontally. Each specimen was supported near the outer ends of its bowed gage section. By positioning the specimen in this manner, both bowed members were on the same level and would thus share the same conditions during exposure. Vertical positioning of the specimens' (d + 2t) distance would have allowed any condensate that might drip from the top member to collect on the bottom member and produce unequal conditions between the two.



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 11. Flow Sheet of Experimental Conditions

In each container the salt-deposited specimens were placed on the lower level supporting rods to insure against any transference of dripped salt to the unsalted specimens. Distilled water was added to the bottom of each container to a depth of 1/2 in. The top of each container was sealed with a thin, transparent plastic film and then all containers were placed in a room thermostatically controlled at 95° F.

Upon completion of each exposure period, the appropriate specimens were removed from the experimental environment, visually examined, rinsed and dried, and then subjected to bend-ductility tests.

Visual and Bend-Test Evaluations:

The visual examination consisted of a careful inspection of each specimen before it was rinsed and dried for bend-ductility tests and storage. Any changes in the specimen such as fractures, discolorations, corrosion deposits, spalled coatings, or cracked coatings were recorded.

When the visual inspection and rinsing had been completed, the residual bend-ductility of the specimens was determined in the fixture and press illustrated in Figures 9 and 10. As shown in Figure 9, each specimen was placed into the clamping members with its (d + 2t) distance oriented in the horizontal direction. With the specimen installation shown, wherein the tab ends were inserted to their full 3/4-in. length, some specimens tended to fracture at one or more of the fixture radii instead of at the location of maximum bend. Therefore, all specimens except those from the 1000-hr exposure were installed with only 1/2-in. of the tab ends inserted in the fixture. With this installation, the bowed members of ductile specimens were free to deflect to nearly complete compression (maximum shortening of 2.5 in. with contact between opposing tab ends) before bearing against a slot radius in the fixture.

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 the specimen had been positioned and secured in the fixture, the dial gage was placed in contact with the lower movable platen of the press and set to the zero reading. 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 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.

Metallographic Examinations:

Metallographic examinations were made on several specimens selected on the basis of the results obtained from the bend-ductility evaluations. The metallographic study was made to ascertain whether losses in ductility were attributable to stress corrosion or some other cause, and to determine the characteristics of stress-corrosion penetration into the susceptible substrates.

The specimens were mounted in diallyl phthalate along with small chips of ferrochrome that were included to help preserve the edges of the specimens during polishing. The successive steps in the polishing procedure were:

1. Fine grind in a Whirlimet polisher on 240, 320, 400, and 600-grit silicon carbide papers.
2. Hand polish with No. 1 Alumina (5μ particle size) on Microcloth.
3. Hand polish with Alumina No. 2 (0.3μ particle size) on Microcloth.
4. Polish approximately 24 hr on vibratory polisher with Type A Alumina (0.3μ particle size) on Microcloth.
5. Polish approximately 24 hr on vibratory polisher with Gamal abrasive on Microcloth.
6. Examine, etch, repolish, and re-etch until satisfactory structure is obtained.

The etches used with the three substrate materials were:

1. Ti-8-1-1: 1 ml HF, 2 ml HNO_3, 97 ml H_2O
2. AM 350: 4 g $CuSO_4 \cdot 5H_2O$, 20 ml HCl
3. Rene 41: 4 g $CuSO_4 \cdot 5H_2O$, 20 ml HCl

Survey of Airframe Manufacturers:

Although the coating evaluations that had been made since the start of the overall program in 1962 were based on SST design requirements as they were known at the time, later design information indicated that there might be other requirements that had not been given sufficient emphasis. Since these requirements could have significant influence on the relative promise of candidate coatings, it was considered desirable to determine the degree of correlation existing between the accumulated evaluation data and the currently known manufacturing and property requirements that must be met by the airframe manufacturers. Therefore, a survey was made of five airframe manufacturers that had been represented on the NASA Special Committee on Materials Research for Supersonic Transports. Appropriate personnel at the airframe companies were invited to discuss their confidence in the data that had been accumulated; the additional information needed by them to meet their design and manufacturing problems; and the relative importance of such factors as weight, visual appearance, reflectivity, thermal and electrical conductivity, erosion resistance, flame resistance, resistance to fuels and other solvents, edge exposure, joining methods, curing temperatures, and material cost.

To conduct the survey, the airframe manufacturers were visited by the principal investigator, who led a discussion based upon a previously prepared questionnaire. Copies of the questionnaire were submitted to the airframe-company personnel prior to the visit so they would have an opportunity to give some thought to the questions. A copy of the questionnaire has been included in the appendix.

Preliminary Evaluation of Additional Coatings:

Since the completion of the screening evaluation that comprised the first major phase of the coatings evaluations, Southern Research Institute has continued to seek and invite suggestions for other promising coatings that were not included in the original screening. During the course of the work reported herein, samples were obtained of five additional coatings that seemed worthy of preliminary investigation. A brief description of these coatings is included in the Appendix as part of Table X.

Each coating was applied to three small panels, 3 in. x 4 in. x 0.040-in. thick. Two of the three panels were 4130 steel and the other was Ti-8-1-1. The purpose of the steel substrates was to provide a corrodable base for illustrating the protective effectiveness of the coating. The titanium-alloy substrate was used to demonstrate the thermal stability and adhesion characteristics of the coatings to titanium alloys when heated at temperatures up to 650° F.

The coated titanium-alloy substrates were heated to 650° F, held for 1 hr, and air cooled. The operation was repeated for four cycles, except that a water spray provided cooling in the third cycle. The heat-exposed samples were visually inspected to detect any appearance or adhesion changes in the sample.

One of the steel panels for each coating was retained as received and the other was subjected to mechanical damage consisting of 3 Rockwell-C hardness penetrations and a 4 ft-lb impact blow with a 1/8 in.-diameter striking edge. Both panels were then subjected to 150 hr of salt spray from a 20% salt solution.

ASTM method B 117 was used as a guide in the operation of the salt-spray cabinet. The edges of the specimens were covered with plastic electrical tape and they were supported in wooden racks at a 5° angle from the vertical, spaced to prevent condensate from one specimen dripping upon another. The operation of the cabinet was checked daily. The specification conditions and the operating conditions were as follows:

Control Item	Specification Conditions	Operating Conditions
Water-Tower Temp, ° F:	100-112	107-110
Air Pressure, psig:	11.0-12.5	11.0-12.0
Cabinet Temp, ° F:	92-97	84-99
Condensate Collected, ml/hr:	0.5-3.0	3.74-5.40
Sp. Gr. of Condensate:	1.126-1.157	1.134-1.160

The rate of condensate collection was considerably greater than the specified amount, probably because of the difficulty that was encountered in holding the cabinet temperature as high as it should have been. This condition was recognized during the run, but attempts to overcome the condition without major interruptions of the run were unsuccessful. Therefore, the results from these specimens may be compared with each other (because they were subjected to identical conditions) but comparison with previous salt-spray results might be misleading.

RESULTS

Visual Evaluations:

The results from the visual examination are summarized in Table VII for the 550° F exposures and in Table VIII for the humid exposures. Except in a few of the bare AM 350 stainless steel specimens, which fractured completely through one leg of the specimen within 800 hr after the start of the humid-salt exposures, there were no visual indications of stress-corrosion damage.

As shown in Table VII, exposure to 550° F caused discoloration of the bare Ti-8-1-1 and AM 350 whereas the Rene 41 alloy remained unchanged. The hot salt deposits caused visible staining on the Ti-8-1-1 and Rene 41 substrates. These stains were more prominent on the titanium alloy and grew more prominent as the length of exposure increased. The hot salt had no visible effect on the AM 350 substrate.

The visual characteristics of the three coatings that had been applied to all three substrates were apparently unaffected by the substrate material. The Aluminum-Modified Silicone remained unchanged throughout the exposure at 550° F. On the other hand, the Catalytically Cured Silicone cracked and shredded from all three substrates within 48 hours after the start of the exposure. Therefore, the substrates of these specimens had essentially the same visual appearance as the originally bare specimens.

The unsalted specimens coated with Zinc in Silicate Vehicle remained unchanged by the heat exposure but the salted specimens developed white stains that grew more severe and tended to partially turn yellow the longer the exposures progressed. No visual changes occurred at 550° F in the specimens coated with Electrophoretically Deposited Aluminum and Flame-Sprayed Aluminum, which were applied only to the Ti-8-1-1 substrate.

The visual reactions of the substrates and coatings to the humid exposure were more varied, as shown in Table VIII. No changes were observed in any of the bare substrates on unsalted specimens. Rene 41 was not visually affected by the salt deposits, and Ti-8-1-1 was unaffected except for slight staining that became visible after 7000 hr. AM 350, on the other hand, was considerably affected by the humid-salt exposure and developed rust spots that increased in severity with increasing exposure duration. Some of the salted AM 350 specimens were rapidly attacked and fractured at locations approximately midway between the tab ends and the point of maximum bow. These fractures, which were observed after approximately 800 hr of exposure, occurred only on certain specimens within each of the four containers that represented the four exposure durations. Attempts to find a cause for the location of the fracture

Table VII. Visual Evaluation of Specimens Subjected to 550° F Exposure up to 7000 Hours

Coating(1)	Visual Observation
Bare	Ti-8-1-1: Gold-colored discoloration over entire surface of all specimens. Stains on salted areas grew more prominent as exposure extended from 1000 to 7000 hr. AM 350: Dark bronze-colored discoloration over entire surface of all specimens. Rene 41: No change in color. Light stains on salted areas.
AMS	All substrates: No changes.
CCS	All Substrates: Coating shredded and spalled from all specimens within 48 hours.
ZSV	All substrates: No change on unsalted specimens. Salted specimens developed white stains that grew more severe and mixed with yellow stains between 3000 and 7000 hr.
EDA	Ti-8-1-1: No changes.
FSA	Ti-8-1-1: No changes.

(1) AMS is Aluminum-Modified Silicone, CCS is Catalytically Cured Silicone, ZSV is Zinc in Silicate Vehicle, EDA is Electrophoretically Deposited Aluminum, and FSA is Flame-Sprayed Aluminum.

Table VIII. Visual Evaluation of Specimens Subjected to Humid Exposure up to 7000 Hr

Coating(1)		Visual Observation
Bare	Ti-8-1-1:	No changes at 5000 hr. Slight stains on salted areas after 7000 hr.
	AM 350:	No changes on unsalted specimens. Rust spots on salted areas increasing in severity from 1000 to 7000 hr. Some salted specimens fractured within approximately 800 hr.
	Rene 41:	No changes.
AMS	Ti-8-1-1:	No changes on unsalted specimens. Slight stains on salted areas at exposure durations beyond 3000 hr.
	AM 350:	No changes on unsalted specimens. Stains on salted areas grew more prominent as exposure extended from 1000 to 7000 hr. Slight rust corrosion mixed with salt stains beyond 3000 hr, especially on scratch-damaged specimens.
	Rene 41:	No changes on unsalted specimens. Slight stains on salted areas.
CCS	All Substrates:	No changes.
ZSV	Ti-8-1-1:	Brown-colored lustrous patches on all specimens. Zinc-oxide on all salted specimens grew more prominent as exposure extended from 1000 to 7000 hr. Zinc oxide especially prominent at scratched areas of salted specimens.
	AM 350:	Brown-colored lustrous patches on all specimens. Zinc oxide on all salted specimens grew more prominent as exposure extended from 3000 to 7000 hr. Slight zinc oxide appeared on unsalted specimens beyond 5000 hr.
	Rene 41:	Brown-colored lustrous patches on all specimens. Zinc oxide on all salted specimens grew more prominent as exposure extended from 1000 to 7000 hr. Slight zinc oxide appeared on all unsalted specimens.
EDA	Ti-8-1-1:	No specimens exposed for less than 5000 hr. No changes in unsalted specimens. Powdery white corrosion product appeared on salted specimens at 5000 hr and was more severe at 7000 hr.
FSA	Ti-8-1-1:	No specimens exposed for less than 5000 hr. No changes in unsalted specimens. Slight amount of powdery white corrosion product appeared on salted areas.

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(1) AMS is Aluminum-Modified Silicone, CCS is Catalytically Cured Silicone, ZSV is Zinc in Silicate Vehicle, EDA is Electrophoretically Deposited Aluminum, and FSA is Flame-Sprayed Aluminum.

and its occurrence in only some of the otherwise identical specimens were unsuccessful.

Aluminum-Modified Silicone exhibited no visual effects on unsalted specimens of all three substrates. The effects observed on salted specimens, however, were influenced by the substrate. On Ti-8-1-1 the coating was slightly stained at salted areas after exposures of 3000 hr or more. On AM 350 the stains were visible at 1000 hr and grew more prominent as the exposure duration increased. After 3000 hr slight rust corrosion was observed with the stains, especially on scratch-damaged specimens. Slight staining of the coating also occurred on the Rene 41 specimens but the stains did not increase with the exposure duration.

Catalytically Cured Silicone remained unchanged on all three substrates in the humid exposures at 95° F.

All specimens coated with Zinc in Silicate Vehicle and exposed to high humidity at 95° F developed scattered patches of a brown-colored lustrous stain that appeared to be strictly a surface phenomenon of the coating and was unrelated to the substrate. The cause of these stains was not determined. A zinc-oxide corrosion product appeared on all salted specimens of all three substrates and grew more prominent as exposure durations increased. This zinc-oxide product was especially prominent at scratched areas on the salted specimens of the Ti-8-1-1 substrate. Slight zinc-oxide stains also appeared on all unsalted specimens of Rene 41 and on unsalted specimens of AM 350 exposed for 5000 hr or more.

Electrophoretically Deposited Aluminum was applied only to the titanium-alloy substrate, and specimens were available only for the 5000 hr and 7000 hr durations. No changes were visible on the unsalted specimens but a powdery white corrosion product appeared on salted specimens exposed 5000 hr and was more severe at 7000 hr.

Flame-Sprayed Aluminum was applied only to the titanium-alloy substrate and was exposed only at the 5000 hr and 7000 hr durations. The unsalted specimens exhibited no changes but a slight amount of powdery white corrosion product appeared on all salted areas.

Bend-Test Evaluations:

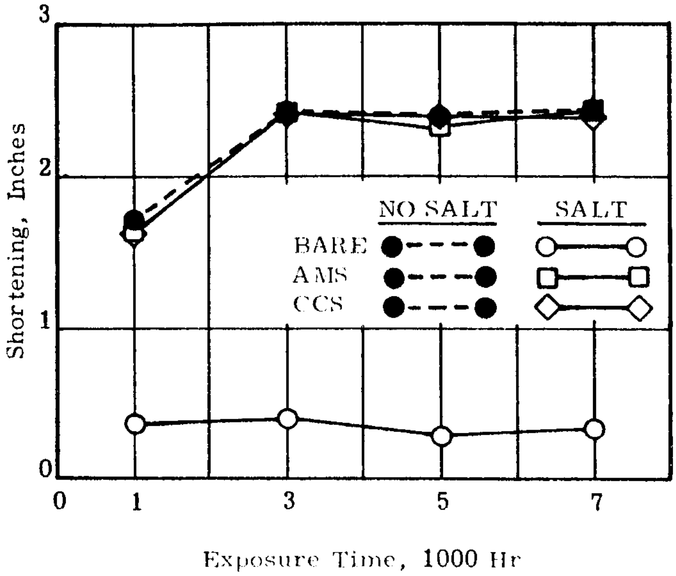
The significance of the bend-test results are illustrated in summary form in Figures 12 through 17. The tabulated results from individual specimens, and bar charts showing average results, are included in the Appendix, Tables XI through XXVI and Figures 30 through 41.

Figure 12 shows the effects of the 550° F dry-salt environment on titanium-alloy specimens that were not previously damaged with scratches. Part (a) of the figure contains the results from bare specimens and from those coated with Aluminum-Modified Silicone (AMS) and with Catalytically Cured Silicone (CCS). All of the specimens within this group retained equivalently high ductilities except the bare specimens exposed to salt, which failed at low values of shortening. There is nothing in these results to confirm the visual observation that the CCS coating crazed and spalled from the specimens within 48 hours after exposure to the 550° F environment, and were essentially bare from that time on. The most logical explanation for the retention of high ductility by these specimens is that the salt deposits were removed along with the spalled coating.

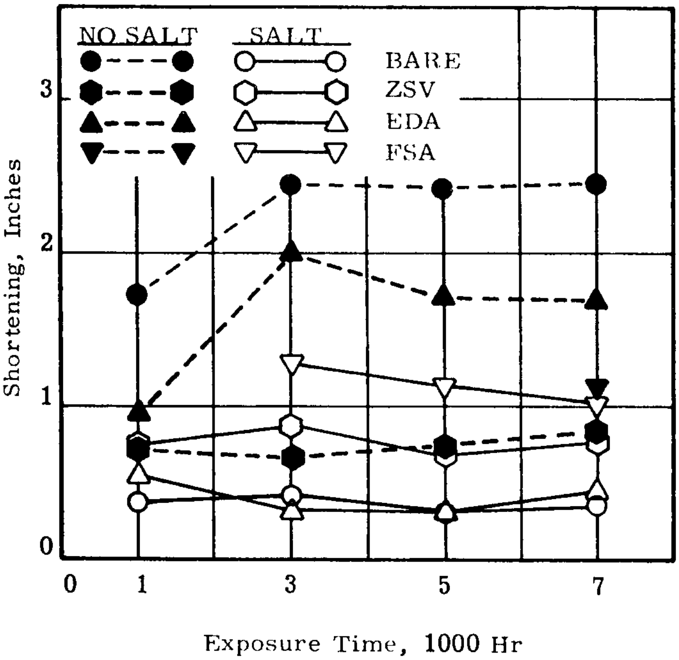
The apparent increase in ductility between 1000 and 3000 hr is a false effect caused by the previously explained change that was made in the location of the specimens within the bending fixture. It is likely that the ductile 1000-hr specimens would have exhibited ductilities equivalent to those from the longer exposures if they had been similarly located in the fixture. The change in location was made while bending the 3000-hr specimens after the bare, AMS, and CCS specimens unexposed to salt had already been tested. Therefore, these specimens appeared to have lower ductilities similar to those from the 1000-hr specimens. Although the actual shortening values obtained for these 3000-hr "no-salt" specimens are reported in the appendix, they were not used in Figure 12 (a) because they presented a distorted value for comparison with the other 3000-hr specimens. For purposes of the plot, the more realistic shortening values obtained with the 3000-hr AMS and CCS specimens exposed to salt were considered to be representative of the "no salt" specimens and were plotted as such. The change of location in the fixture did not significantly effect the results obtained from embrittled specimens.

In Figure 12 (b) the results from the bare specimens are shown compared to those obtained from the specimens coated with Zinc in Silicate Vehicle (ZSV), Electrophoretically Deposited Aluminum (EDA), and Flame-Sprayed Aluminum (FSA). The specimens exposed to salt are shown by open data points connected with solid lines, and the specimens unexposed to salt by closed data points connected with dashed lines.

The results show that the specimens coated with ZSV were similarly embrittled in both the "no salt" and salt-exposed conditions, and produced shortening values ranging between approximately 0.7 to 0.9 in. from all specimens. Subsequent metallographic examination (see Figure 27) indicated that the embrittlement was caused by gross surface roughness that was probably inflicted when the coating supplier prepared the specimens for coating.



(a) Bare and coated with Aluminum-Modified Silicone (AMS) and Catalytically Cured Silicone (CCS).



(b) Bare and coated with Zinc in Silicate Vehicle (ZSV), Electrophoretically Deposited Aluminum (EDA), and Flame-Sprayed Aluminum (FSA).

Figure 12. Effects of 550° F Dry Salt on UNSCRATCHED TITANIUM 8-1-1 as Shown by Bend-Ductility Evaluations.

Specimens coated with FSA were embrittled similarly but to a lesser extent than those coated with ZSV, and although only one "no salt" data point was available for comparison, the lack or presence of salt apparently had insignificant effects on the embrittlement. Metallographic examination revealed that the surface of these specimens had also undergone roughening prior to coating.

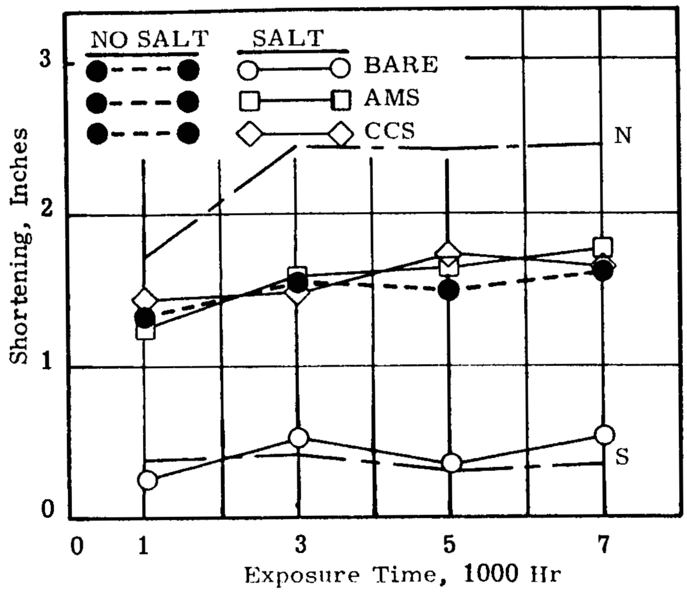
The EDA specimens, on the other hand, exhibited considerable difference between the ductilities of the "no salt" and salt-exposed specimens. The "no salt" specimens were less ductile than the bare specimens, probably because the EDA coating was applied to a different lot of titanium alloy and was given additional heat treatments to improve the adherence of the coating. Because this coating was applied to the substrate in sheet form, the edges of the specimens were bare. The bare edges possibly account for the loss of ductility on salt-exposed specimens, which were embrittled to the same extent as bare specimens.

All results in Figure 12 and its companion figures show that all specimens affected by the salt had achieved their complete embrittlement to bending within the first 1000-hr exposure period, and no further significant losses in ductility occurred beyond that duration.

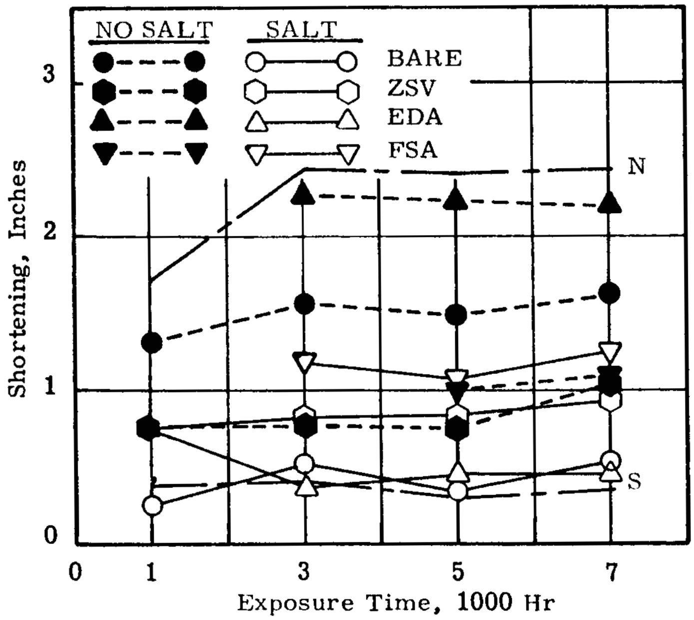
Figure 13 shows the shortening results for titanium-alloy specimens that were damaged with scratches before introduction to the 550° F exposure. Except for the reductions in shortening caused by the scratches themselves, the results are quite consistent with the results from unscratched specimens (Figure 12). The results from unscratched bare specimens have been included in Figure 13 for purposes of comparison.

Figure 13 (a) compares the bare specimens with the Aluminum-Modified Silicone (AMS) and Catalytically Cured Silicone (CCS). The bare specimens not exposed to salt and the coated specimens with and without salt all had similar ductilities on the order of 1.5 in. shortening, which was apparently the ductility level dictated by the presence of the scratches. The bare specimens exposed to salt were further embrittled and had ductilities equivalent to the unscratched bare specimens that were exposed to salt. As with the unscratched specimens, the apparent protection furnished by CCS is not necessarily valid because the coating spalled off of the specimens shortly after heating to 550° F.

Figure 13 (b) shows the results from bare specimens compared to those from specimens coated with Zinc in Silicate Vehicle (ZSV), Electrophoretically Deposited Aluminum (EDA), and Flame-Sprayed Aluminum (FSA). These results were quite similar to the results obtained on the unscratched specimens (Figure 12). All specimens coated with ZSV had equivalent lowered ductilities



(a) Bare and coated with Aluminum-Modified Silicone (AMS) and Catalytically Cured Silicone (CCS). Bare, undamaged results with no salt (N) and with salt (S) shown for comparison.



(b) Bare and coated with Zinc in Silicate Vehicle (ZSV), Electrophoretically Deposited Aluminum (EDA), and Flame-Sprayed Aluminum (FSA). Bare, undamaged results with no salt (N) and with salt (S) shown for comparison.

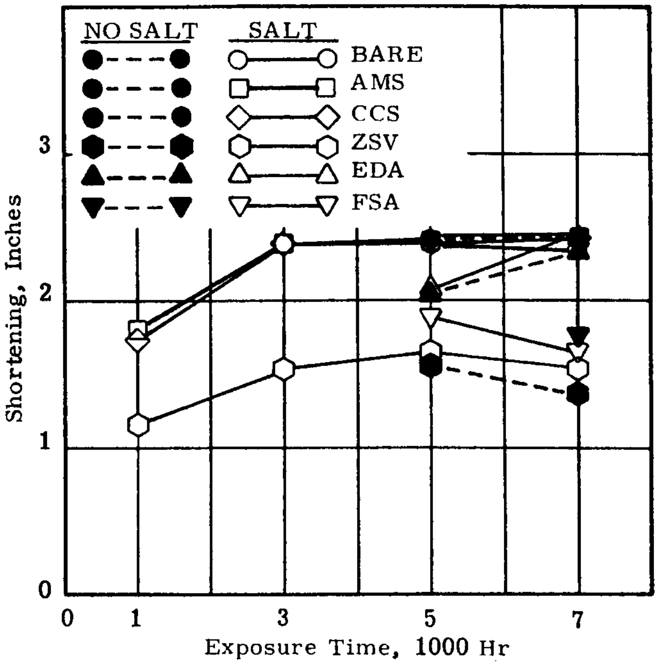
Figure 13. Effects of 550° F dry salt on SCRATCHED TITANIUM 8-1-1 as shown by bend ductility evaluations.

that were apparently not affected by the salt. The results from FSA specimens had a similar relationship but with slightly better ductilities than the ZSV specimens. Specimens coated with EDA had nearly complete ductility in the absence of salt but were completely embrittled in the presence of salt, presumably because of the bare edges.

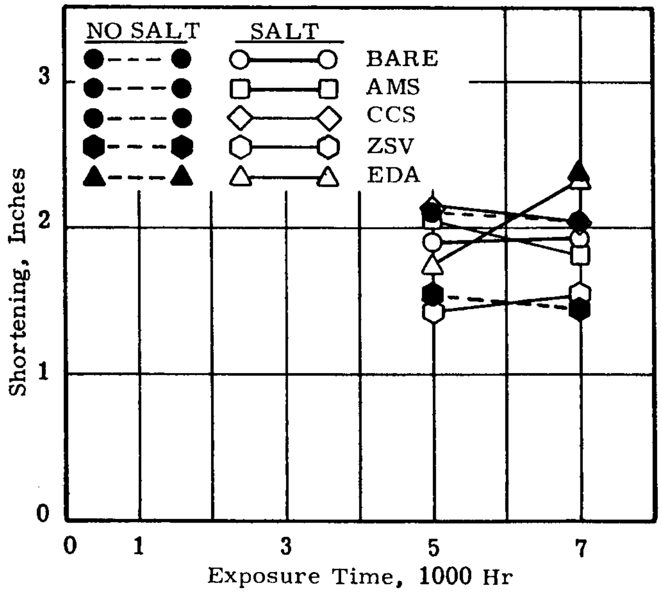
Figure 14 shows the effects of the humid exposure on the bare and coated Ti-8-1-1 substrate. The results from the unscratched specimens are shown in Figure 14 (a). Although fewer data are available for the shorter-duration exposures, enough data are available to indicate the trends. It is evident, for example, that the humid environment, either with or without salt, did not cause any embrittlement of the bare substrate. The two coatings, AMS and CCS, therefore, also exhibited maximum ductility. On the other hand, the surface roughness noted in the previous discussion of the 550° F exposures has apparently caused lower ductilities in the specimens coated with ZSV and with FSA. The specimens coated with EDA retained good ductility even though general corrosion of the aluminum coating itself was increasingly severe with increasing exposure times (see Table VIII). The increase in ductility of the EDA specimens between 5000 and 7000 hr (noted also in the scratched specimens shown in Figure 14 b) could not be explained by any of the known data.

Figure 14 (b) contains the data from the scratched specimens. These data confirm that the presence of salt in this environment had no stress-corrosion effects on the titanium-alloy substrate, either in the bare or coated conditions. All specimens exhibited some loss in ductility because of the presence of the scratches but the losses were not as great as those caused by the scratches in the specimens exposed to the 550° F environment (see Figure 13).

Figure 15 contains a plot of all of the results obtained from the AM 350 substrate—scratched and unscratched specimens in both 550° F and humid exposures. It was practical to summarize most of the data to form the upper curve in the figure because neither the scratches nor the 550° F exposures had any effect on the ductility of AM 350 substrate. Specimens coated with ZSV and exposed to salt had less ductility on the average than the ZSV specimens not exposed to salt. However, reference to the tabular data in the Appendix will show that several of the salt-exposed specimens did retain maximum ductility. The bare specimens exposed to salt also reacted in an erratic manner not revealed by the average shortening values. As shown in Table VIII and the tabulated data in the Appendix, the bare specimens exposed to salt either fractured completely within 800 hr after the start of exposure or remained intact and exhibited maximum ductility at the end of the exposure. Therefore, the plotted points are averages between zero shortening and maximum shortening, with the average value depending upon the relative number of

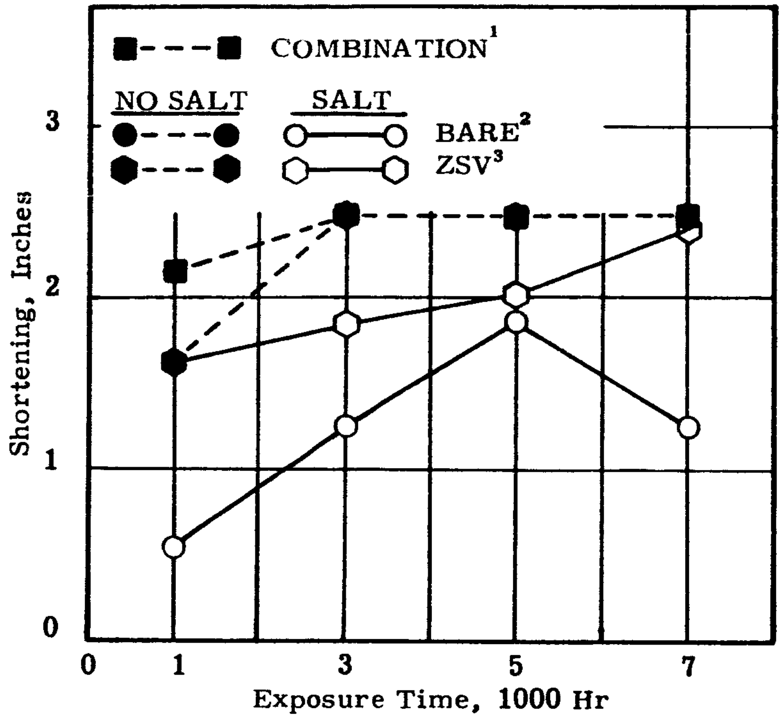


(a) Unscratched, Bare and Coated with Aluminum-Modified Silicone (AMS), Catalytically Cured Silicone (CCS), Zinc in Silicate Vehicle (ZSV), Electrophoretically Deposited Aluminum (EDA), and Flame-Sprayed Aluminum (FSA).



(b) Scratched, Bare and Coated with Aluminum-Modified Silicone (AMS), Catalytically Cured Silicone (CCS), Zinc in Silicate Vehicle (ZSV), and Electrophoretically Deposited Aluminum (EDA).

Figure 14. Effects of Humid Salt on TITANIUM 8-1-1 as shown by bend-ductility evaluations.



[1] Average of all data from the 550° F exposure and from the humid-exposure specimens coated with Aluminum-Modified Silicone and Catalytically Cured Silicone.

[2] Data from all bare specimens in the humid exposure.

[3] Data from all of the humid-exposure specimens coated with Zinc in Silicate Vehicle (ZSV).

Figure 15. Effects of 550° F Dry Salt and of Humid Salt on AM 350 SCT STAINLESS STEEL as shown by bend-ductility evaluations.

specimens having zero shortening. No explanation could be found to account for the erratic behavior of the bare and ZSV-coated substrates in the humid-salt environment.

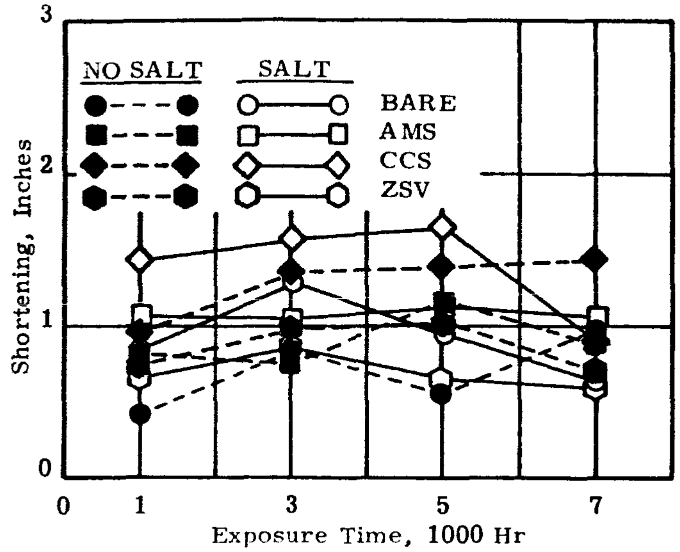
The results from Rene 41 specimens exposed at 550° F are shown in Figure 16. The erratic pattern of the plotted results, in which the salt-exposed specimens were often more ductile than the "no-salt" specimens, indicates that the Rene 41 substrate was not affected by the hot salt. The results shown in Figure 17 indicate the same lack of effects from the humid salt. Both figures show that the specimens coated with ZSV had consistently less ductility than the other specimens, probably because of the previously mentioned surface roughness found in all of the metallographic specimens involving the ZSV coating.

Metallographic Examinations:

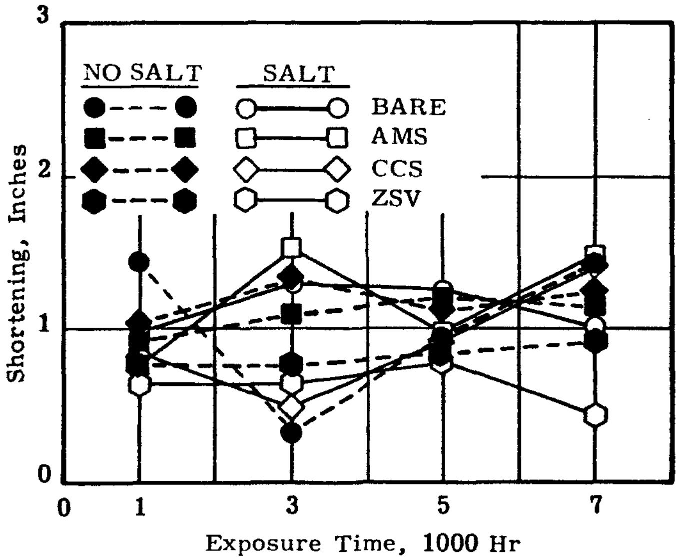
Although a thorough and comprehensive metallographic study was beyond the scope of this program, certain selected specimens were subjected to metallographic examination in an effort to help explain the results from the corrosion exposures. The significant microstructures observed are reproduced in Figures 18 through 29.

Figure 18 shows the microstructure of the Ti-8-1-1 alloy in the as-received, duplex-annealed condition. As shown in Figure 18 (a), the longitudinal structure at 100X magnification and bright-field illumination consisted of a fine-grained α matrix with small β particles scattered throughout the section. The α matrix itself, however, consists of two types of α grains. One type of α grain is characterized by a clear white appearance, prominently etched grain boundaries, an absence of β particles in the grain interiors, and retention of the elongation produced in rolling the material to sheet form. The other type of α grain is characterized by a darker and mottled appearance, less prominent grain boundaries, the presence of β particles in the grain interiors, and a more equiaxed structure. For purposes of discussion, the clear, elongated grains were designated primary α and the other secondary α .

The differences between the primary and secondary α , and the appearance of the β particles is more clearly seen in the 500X bright-field view in Figure 18 (b). This view shows that the β particles appear in the grain boundaries of either primary or secondary α but in the grain interiors of only the secondary α . These structural phenomena are emphasized under polarized illumination of the same field, as shown in Figure 18 (c). Under polarized illumination the primary α grains appear clear and black, the secondary α grains are a mottled dark gray, and the β particles have a bright crystalline appearance.

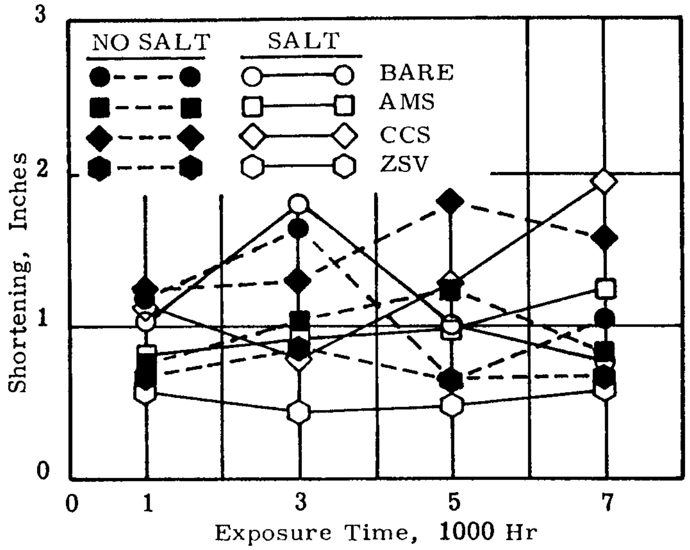


(a) Results from UNSCRATCHED Specimens Bare and Coated with Aluminum-Modified Silicone (AMS), Catalytically Cured Silicone (CCS), and Zinc in Silicate Vehicle (ZSV).

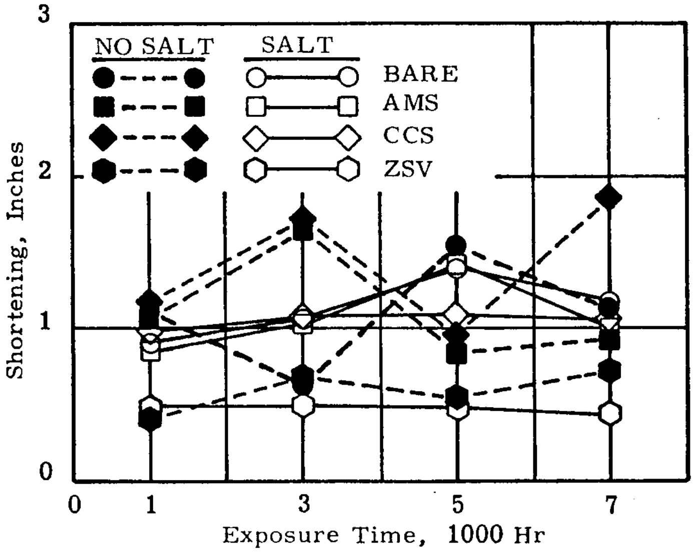


(b) Results from SCRATCHED Specimens Bare and Coated with Aluminum-Modified Silicone (AMS), Catalytically Cured Silicone (CCS), and Zinc in Silicate Vehicle (ZSV).

Figure 16. Effects of 550° F Dry Salt on RENE 41 as shown by bend-ductility evaluations.

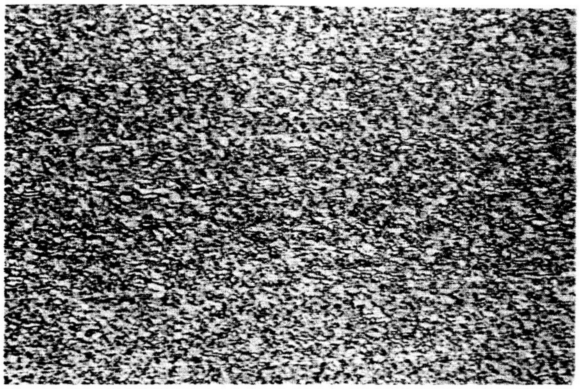


(a) Results from UNSCRATCHED Specimens Bare and Coated with Aluminum-Modified Silicone (AMS), Catalytically Cured Silicone (CCS), and Zinc in Silicate Vehicle (ZSV).



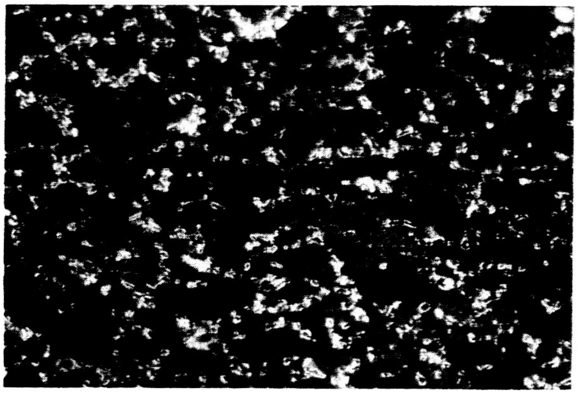
(b) Results from SCRATCHED Specimens Bare and Coated with Aluminum-Modified Silicone (AMS), Catalytically Cured Silicone (CCS), and Zinc in Silicate Vehicle (ZSV).

Figure 17. Effects of Humid Salt on RENE 41 as shown by bend-ductility evaluations.



(a) Bright field.
100X

(b) Bright field.
500X



(c) Polarized light.
500X

Figure 18. Ti-8-1-1 original structure prior to exposure. Shows α matrix with small β particles and elongated islands of "primary α." Longitudinal section.

The microstructures shown in Figure 18 indicate that there was segregation of the α and b stabilizers during the preparation of the alloy. It was not conclusively determined whether this structure has significant influence on the susceptibility of Ti-8-1-1 and other titanium alloys to dry-salt stress corrosion, but the possibility that it does is worthy of further investigation.

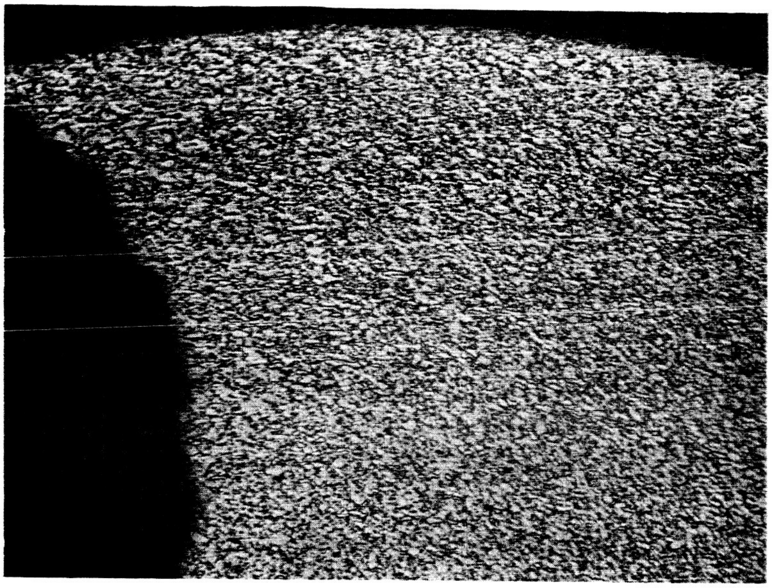
Figure 19 presents a comparison of the surface and fracture appearances of two bare Ti-8-1-1 specimens subjected to the 550° F exposure. The specimen shown in Figure 19 (a) was exposed for 5000 hr without salt and exhibited full ductility in the bend test. The specimen shown in Figure 19 (b) was subjected to the 550° F exposure for 1000 hr with salt and had extremely poor ductility. The microstructures of these two specimens appear to be identical except for the surface cracks visible in the salt-exposed specimens.

The surface cracks in the embrittled specimen were observed along the surface at areas remote from the fracture, indicating that the cracks were produced by stress corrosion and were not opened up by the bending operation. In fact, the most severe cracking occurred remote from the fracture. This area of most severe cracking is shown in Figure 20 (a) at the same magnification used for Figure 19 (b).

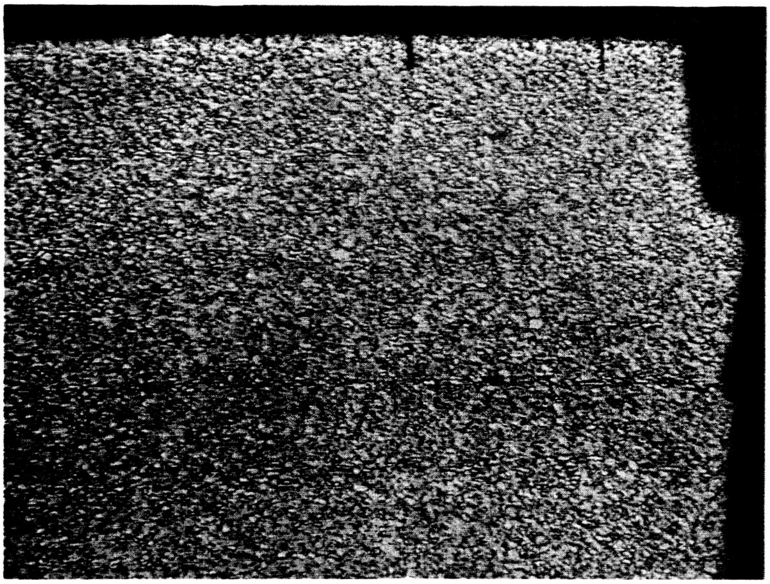
Some of the characteristics of the cracks are revealed at both 100X and 500X magnifications in Figure 20. One characteristic is the blunt configuration of the ends of the cracks. In view of the apparent intergranular path of the cracks, these blunt ends are somewhat surprising. and no suggested explanation appears to be completely satisfactory. Although the stresses in the self-loaded specimen would be expected to decrease below the threshold for stress corrosion with increasing depth below the surface, it is unlikely that this phenomenon would produce the blunt ends until all cracks had reached approximately the same depth.

Another characteristic of the cracks is that many of them are discontinuous in the plane of the cross section as shown in Figure 20 (b). Such apparent discontinuities are undoubtedly a further indication of the intergranular path of the cracks, but it is interesting to note that the discontinuities tend to occur at intersections with the primary α grains, providing an indication that the primary α might be more resistant to stress-corrosion cracking.

Figure 21 shows microsections of bare Ti-8-1-1 specimens that were purposely damaged with scratches prior to 500 hr exposures at 550° F. The specimen in Figure 21 (a) exposed without salt was embrittled by the scratch (1.68 in. shortening) and the specimen in Figure 21 (b) was further embrittled by salt (0.21 in. shortening). Yet, no cracks were

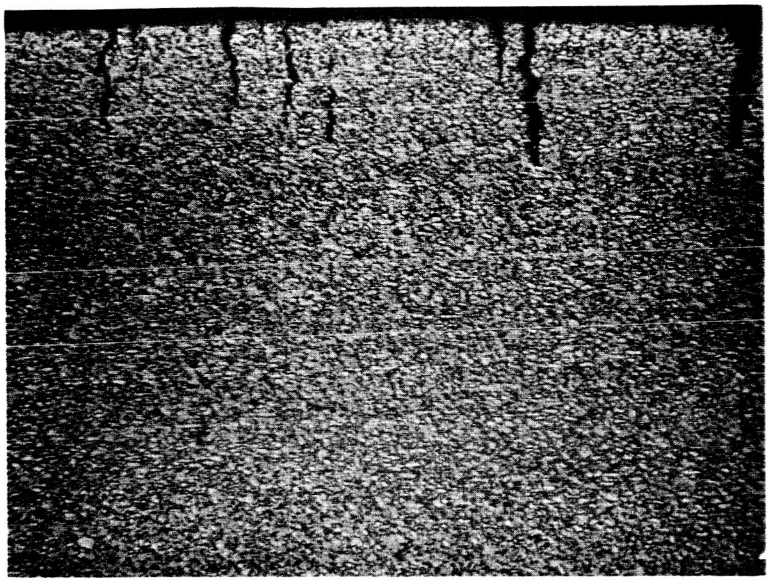


(a) 5000 hr with no salt. 100X

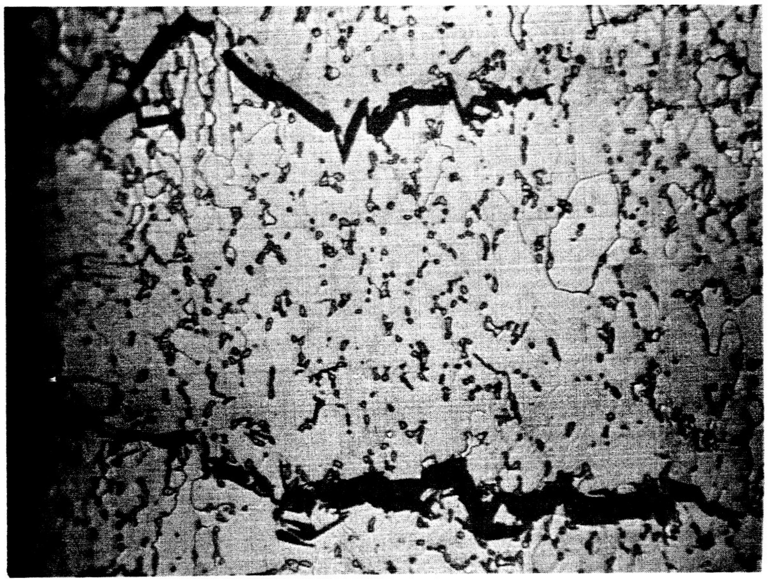


(b) 1000 hr with salt. 100X

Figure 19. Ti-8-1-1 ductile and brittle fractures after exposure at 550° F. (a) Specimen T0U1H5A, 5000 hr with no salt and (b) Specimen T0U2H1C, 1000 hr with salt. Shows stress-corrosion cracks on surface of embrittled specimen (b).

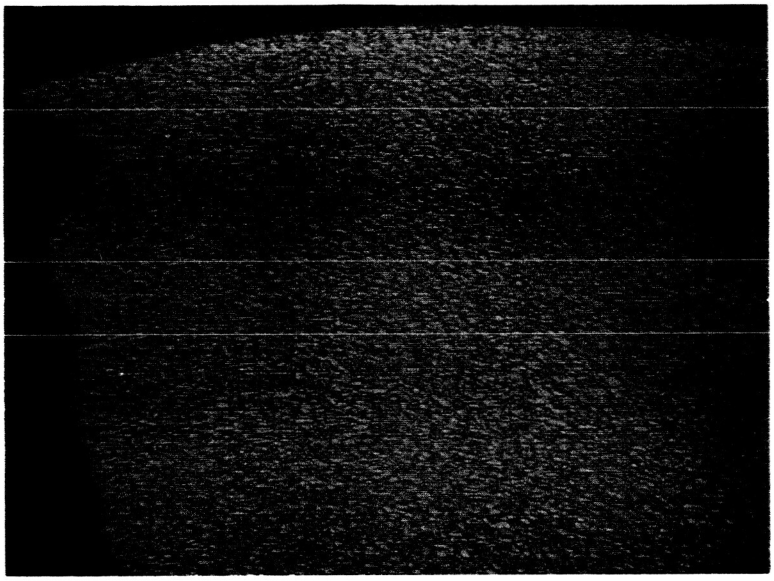


(a) 100X

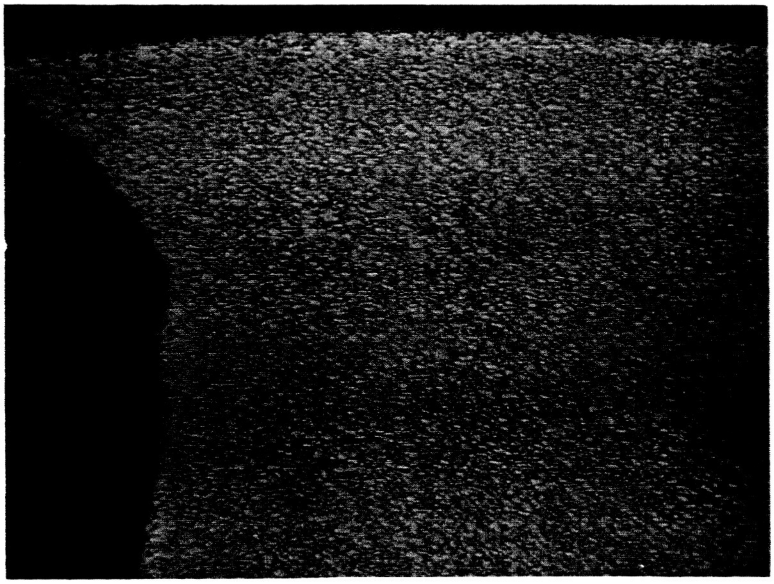


(b) 500X

Figure 20. Ti-8-1-1 stress-corrosion cracks. Most severe cracks in bare specimen T0U2H1C subjected to 1000 hr at 550° F with salt. Most severe cracking was at location away from fracture.



(a) No salt. 100X



(b) With salt. 100X

Figure 21. Ti-8-1-1 fracture surfaces of scratched bare specimens after 5000 hr exposure at 550° F.
(a) Specimen T0D1H5A, no salt, 1.68 in. shortening.
(b) Specimen T0D2H5B, with salt, 0.21 in. shortening.

found in the surface of the salt-embrittled specimen. It is indicated, therefore, that either all surface cracks were accidentally missed in the plane of the cross section or that the stress-corrosion attack was confined to the scratches.

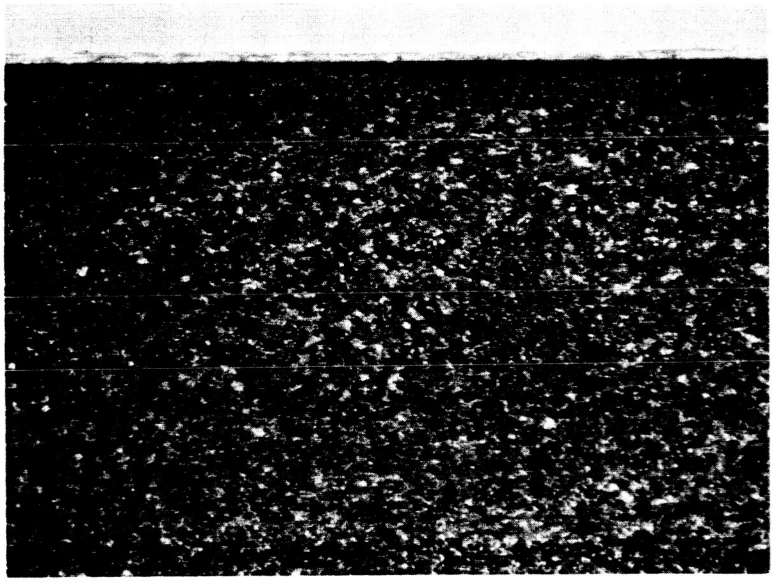
Sections of Ti-8-1-1 specimens coated with Aluminum-Modified Silicon (AMS) and with Zinc in Silicate Vehicle (ZSV) are shown in Figure 22. No stress-corrosion cracks were found in either specimen even though the AMS specimen, Figure 22 (a), was ductile and the ZSV specimen, Figure 22 (b), was embrittled. Figure 22 reveals that the surface of the embrittled specimen has been roughened and grossly disturbed, apparently in preparation for the ZSV coating. The surface under the ductile specimen coated with AMS was smooth and undisturbed. Both sections are shown under polarized illumination in order to make the coating more visible.

Specimens coated with Catalytically Cured Silicone were not metallographically examined because the coating spalled from the substrate in the 550° F exposure, and all specimens from all exposures were ductile in the bend test.

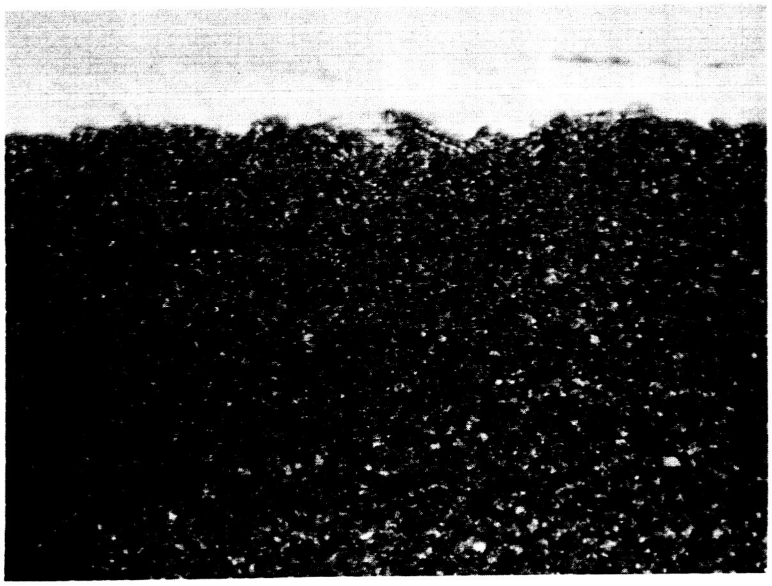
Microsections of a Ti-8-1-1 specimen coated with Electrophoretically Deposited Aluminum (EDA) and exposed to 550° F for 5000 hr with salt are shown in Figure 23. A view of the brittle fracture is shown in Figure 23 (a). No outstanding stress corrosion cracks were found in any part of this longitudinal section. At higher magnification, 500X in Figure 23 (b), quite small openings perpendicular to the substrate surface were observed. These openings were so small that their significance is doubtful, but they were unique to this specimen. Since the edges of the EDA specimens were exposed and the section shown is a longitudinal section through the interior of the specimen, the small openings might be lateral extensions of deeper edge cracks. Figure 23 (c) shows the appearance of the coating at 100X magnification under polarized light.

Figure 24 shows microsections of a Ti-8-1-1 specimen coated with Flame-Sprayed Aluminum (FSA) and exposed with salt to 550° F for 5000 hr. Figure 24 (a) shows the area near the brittle fracture and Figure 24 (b) is a polarized-light view showing the appearance of the coating and the disturbed substrate surface. No stress-corrosion cracks were observed and the embrittlement was probably caused by the substrate surface conditions.

Several specimens of Ti-8-1-1 exposed to the humid atmosphere were examined but not photographed because there were no significant changes in the basic structure of the substrate. All of the titanium-alloy specimens exposed to the humid atmosphere were ductile unless embrittled by scratched or roughened surfaces. The AM 350 SCT substrate, on the other hand, was

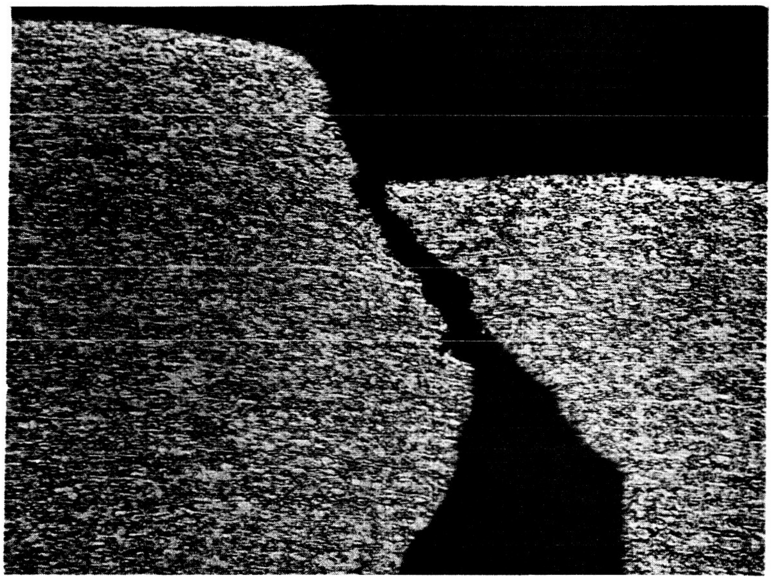


(a) AMS coating. 100X

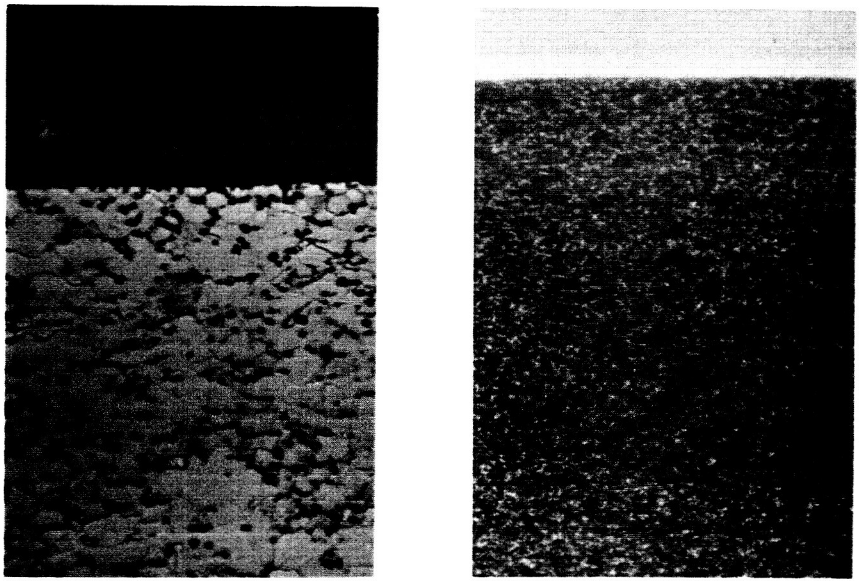


(b) ZSV coating. 100X

Figure 22. Coated Ti-8-1-1 exposed with salt at 550° F for 5000 hr. (a) Ductile specimen T1U2H5B coated with Aluminum-Modified Silicone (AMS).
(b) Brittle specimen T3U2H5C coated with Zinc in Silicate Vehicle (ZSV). Taken with polarized light.



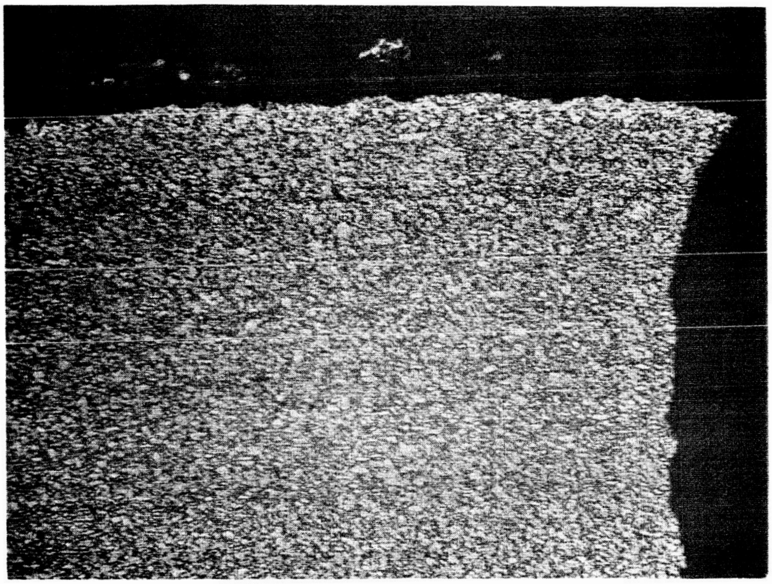
(a) Fracture. 100X



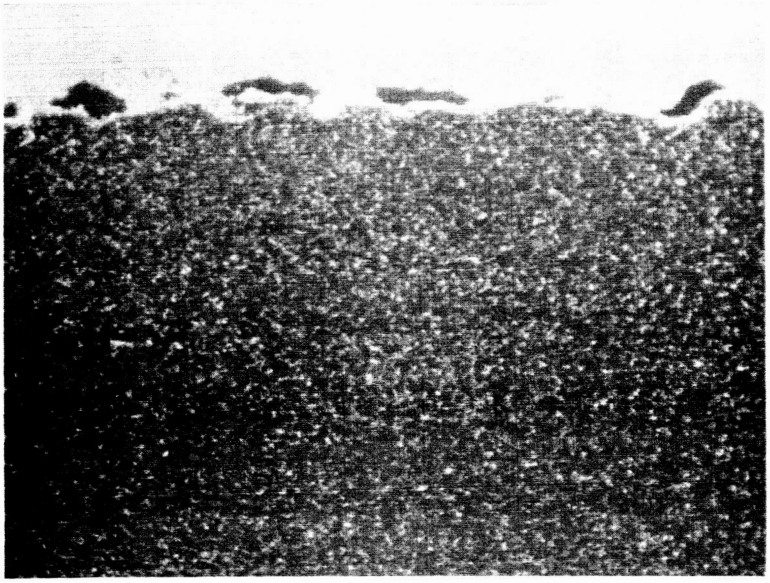
(b) Fine surface cracks. 500X

(c) Coating shown with polarized light. 100X

Figure 23. Ti-8-1-1 coated with Electrophoretically Deposited Aluminum (EDA). Specimen T4U2H5B exposed 5000 hr with salt at 550° F. Brittle failure.



(a) Fracture. 100X



(b) Coating shown with polarized light. 100X

Figure 24. Ti-8-1-1 coated with Flame-Sprayed Aluminum (FSA). Specimen T5U2H5B. Exposed 5000 hr with salt at 550° F. Brittle failure.

resistant to corrosion at 550° F but did undergo attack in some specimens in the humid atmosphere.

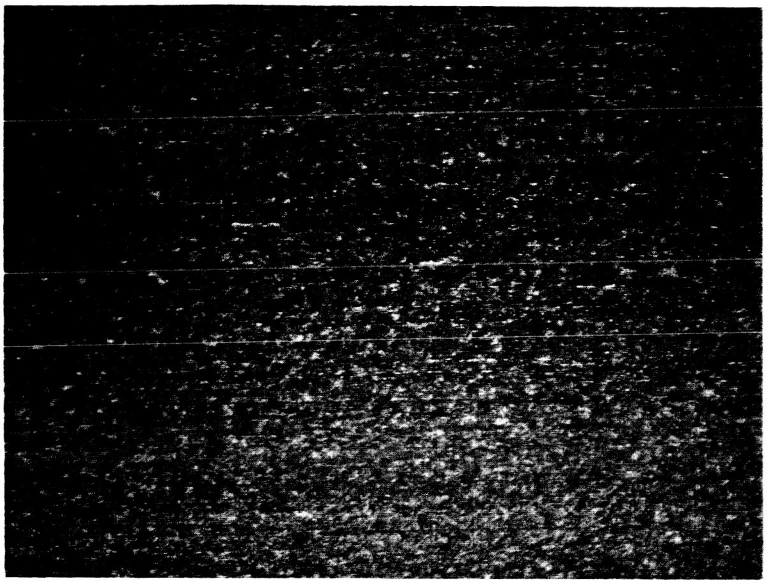
The structure of the SCT heat-treated AM 350 prior to corrosion exposures is shown in Figure 25 at 100X (a) and 500X (b) magnifications. The structure consisted of small elongated ferrite grains in a fine martensitic matrix.

As previously shown in the results plotted in Figure 15, humid salt exposures had an average embrittling effect on bare AM 350. The actual attack, however, was concentrated only on certain specimens and caused fractures to occur within 800 hr at a location approximately 3/8 in. from a tab end while the specimens were still on the exposure rack. Other specimens, apparently identical in preparation and exposure, withstood the full exposure times and retained full ductility in the bend test.

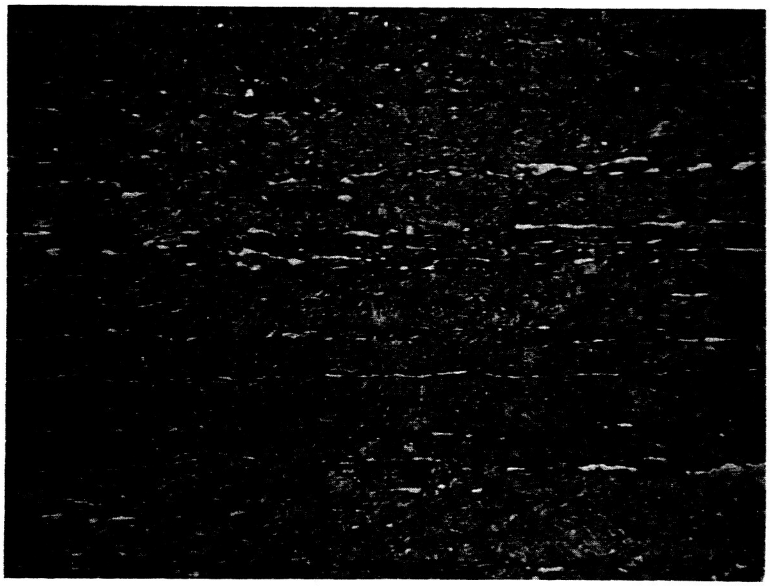
Microsections in the vicinity of the fracture in one of the failed AM 350 specimens are shown in Figures 26 (a) and 26 (b). The section in Figure 26 (a) shows a large crack extending angularly between the fracture surface and the surface of the specimen. Neither end of the crack extends to the surface in the plane of the microsection. Figure 26 (b) shows a portion of the crack at higher magnification and indicates that it is predominently intergranular, although the poor definition of the grain boundaries makes it difficult to be certain. The lack of perpendicular surface cracks and the position and direction of the large crack indicate that the attack was not stress corrosion but stress-accelerated general corrosion. Further evidence of the general corrosion was found in the specimen shown in Figure 26 (c). This view shows small corrosion cracks extending into the specimen from the concave surface of one of the ductile bend specimens. The location of the cracks on the surface subjected to compressive stresses during the exposures indicates that tensile stresses from external sources were not necessary for corrosive attack when it occurred.

The only coated specimens of AM 350 that had reduced ductility were some that were coated with Zinc in Silicate Vehicle and exposed to humid salt. Metallographic examination revealed no corrosive attack from the salt but did show that the substrate surfaces of ductile and embrittled specimens had been considerably distorted, apparently in preparation for the coating. As shown in Figure 27, the maximum surface roughness was greater in the embrittled specimen than in the ductile specimen. The metallographic examination did not reveal, however, why the reduced ductilities were confined to specimens exposed to salt.

The results from the bend-ductility tests of Rene 41 specimens (Figures 16 and 17) indicated that the inherent bend ductility of that substrate was lower

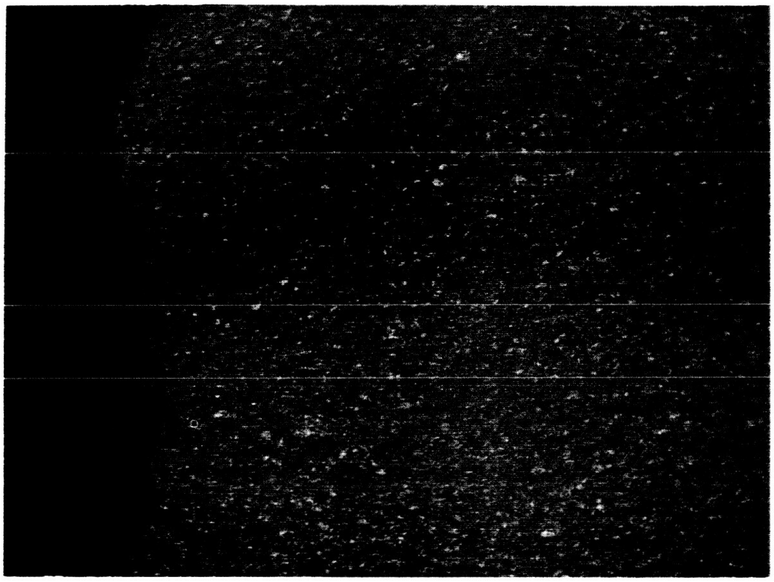


(a) 100X

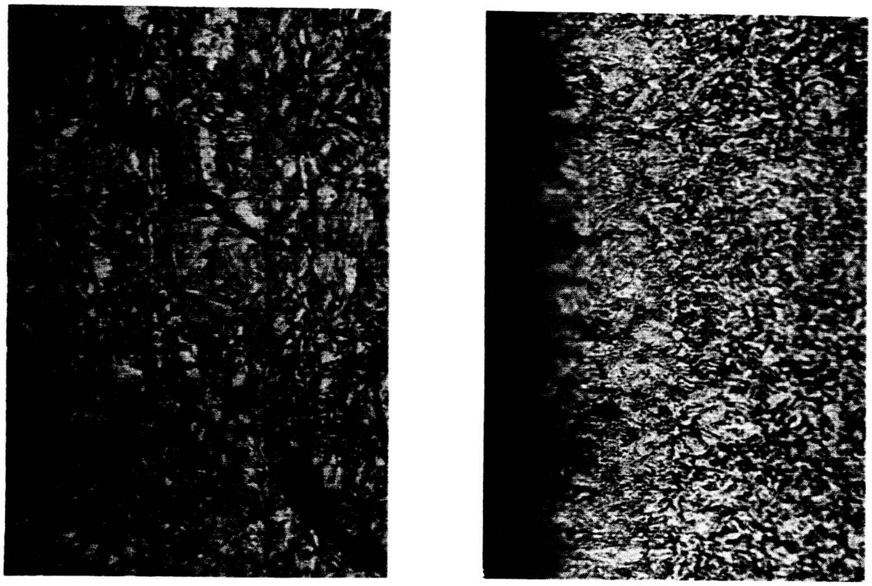


(b) 500X

Figure 25. AM 350 SCT original structure prior to exposure. Shows elongated ferrite in martensitic matrix.



(a) Internal crack. 100X



(b) Internal crack. 500X

(c) Cracks in concave surface. 500X

Figure 26. Cracks in bare unscratched AM 350 SCT exposed to humid salt at 95° F. (a) and (b), Specimen A0U2L5A, show internal crack at angle to brittle-fracture surface. (c) shows small cracks at concave surface on full-ductility specimen A0U2L5B.

(a) Ductile specimen. 500X

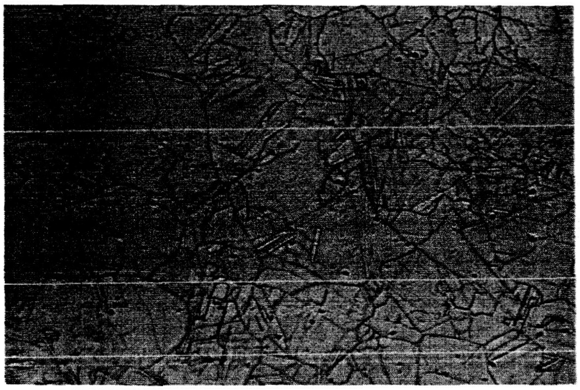
(b) Brittle Specimen. 500X

Figure 27. Maximum surface roughness of AM 350 SCT substrate coated with Zinc in Silicate Vehicle (ZSV). (a) Specimen A3U2L1A with good bend ductility. (b) Specimen A3D2L1B with poor bend ductility. Both specimens exposed to humid salt at 95° F for 1000 hr.

and more erratic than the bend ductility of the other two substrates. The microstructure of Rene 41 in the heat-treated-and-aged condition prior to the exposures is shown in Figure 28. The view at 100X magnification in Figure 28 (a) shows the structure to consist of a matrix with small carbides precipitated at the grain and twin boundaries and more massive carbides scattered at random. The matrix, however, appears to consist of two sets of grains: one "old" set of large grain size on which the fine carbides are precipitated, and one set of "new" twins and small grains or subgrains on which carbides are not precipitated. The distinction between the old and new grains is shown in Figure 28 (b), which is a higher magnification view of the subgrain complex at the 8 o'clock position on the large grain in the center of Figure 28 (a). Figure 28 (c) is an identical view taken with polarized illumination to emphasize the carbide locations. The structure indicates that the final solution heat treatment partially recrystallized the previous grain structure but did not dissolve the carbides for further precipitation on the new twin and grain boundaries during the subsequent aging treatment. Further investigation would be necessary to determine whether structure was influential in producing the wide scatter observed in the bend-ductility results.

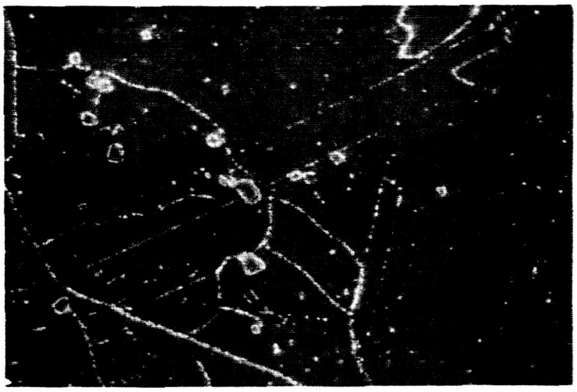
The only consistent feature of the Rene 41 bend-ductility results was that the specimens coated with Zinc in Silicate Vehicle (ZSV) exhibited ductilities at the lower boundary of the scatter band. The metallographic examination failed to reveal the cause of lower ductilities in the ZSV specimens, but it may be logically surmised that they were probably caused by the surface-roughening treatments observed to produce reduced bend ductilities in the other two substrates.

Figure 29 shows the structures at the fracture surfaces of two specimens exposed for 5000 hr at 550° F. The specimen in Figure 29 (a) was exposed with salt but produced no evidence of attack by the salt. The fracture in the unsalted specimen shown in Figure 29 (b) was quite similar to that of the salt-exposed specimen. Some surface cracks like those visible in Figure 29 (b) were present in the unsalted specimen but they were located only near the fracture and were probably opened up during the bend-ductility tests.



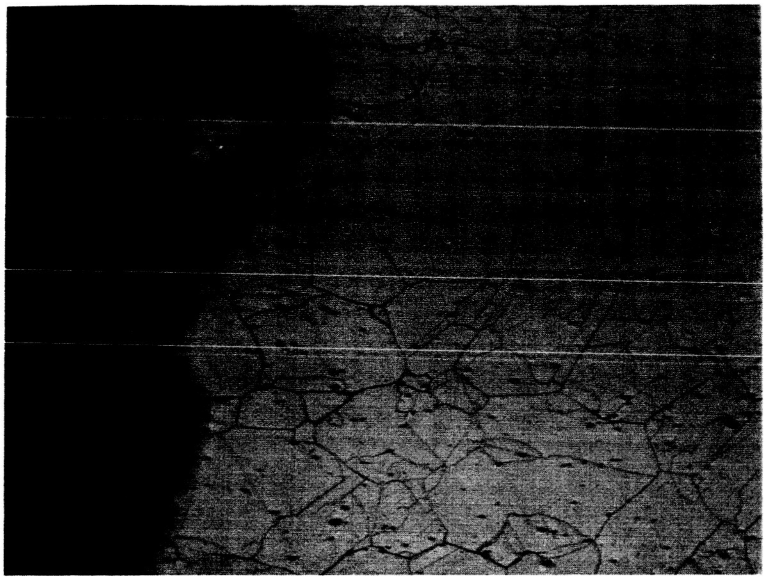
(a) General view.
100X

(b) Bright-field view of old and new twins.
500X

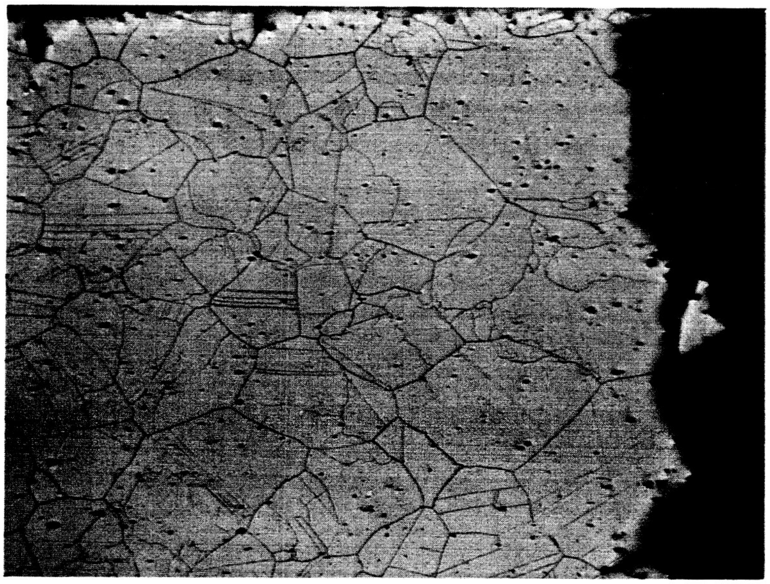


(c) Polarized-light view of old and new twins.
500X

Figure 28. Rene 41 original structure prior to exposure. Shows precipitated carbides in old grain and twin boundaries, and new grain and twin boundaries without precipitated carbides.



(a) With salt. 100X



(b) No salt. 100X

Figure 29. Rene 41 brittle fractures after exposure to 550° F for 5000 hr. Shows intergranular fractures in (a) Specimen R0U2H5A with salt and (b) Specimen R0U1H5B without salt.

Survey of Airframe Manufacturers:

Although the results from the survey of five airframe manufacturers were conflicting in several details, they did provide a valuable overall evaluation of the coatings program and assessment of the needs for the future. A recapitulation of the questionnaire used in the survey, and of the answers given, are included in the Appendix. The following is a summary of the prevalent opinions obtained from the formal interviews with the five airframe manufacturers and from an informal interview (questionnaire was not used as the basis for the discussion) with one engine manufacturer.

The conclusions reached by Southern Research Institute with respect to the most promising coatings, that is, Aluminum-Modified Silicone and Catalytically Cured Silicone were considered to be generally correct. However, because of SST requirements for high emissivity in the infra-red (IR) range, Aluminum-Modified Silicone, the first choice on the basis of the experimental program, has been given little consideration for skin areas of the SST. Catalytically Cured Silicones with white pigmentation are, however, the most promising formulations currently available. The spalling problem encountered with Catalytically Cured Silicone in the experimental program has been overcome in the formulations developed by various paint manufacturers working with the airframe companies. In engine areas, where IR emissivity is not a critical consideration and temperature exposures might be on the order of 800 to 900° F, Aluminum-Modified Silicones appear to be most advantageous.

Within the scope of the experimental program, no coatings are known to be superior to those found most promising by Southern Research Institute. On the other hand, slight changes in requirements, or specific requirements for local areas, could increase the feasibility of some of the types of coatings that have been screened out of the promising list. For example, it appears possible that more refined SST design will allow large portions of the skin to operate at maximum temperatures on the order of 450° F. At this maximum temperature, polyimide coatings appear promising if improvements can be made toward room-temperature curing and better color stability in pigments. Cermet types of coatings offer some possibilities in local engine areas where temperature or abrasion requirements are too great for the Aluminum-Modified Silicones. Metallic coatings such as aluminum cladding offer some possibilities but, since IR emissivity control is a critical consideration, top coatings would still be necessary on the aluminum surface.

Among the properties not included in this investigation, the IR emissivity to absorptivity ratio is considered to be of primary importance. High emissivity (0.8 or greater) is a necessity not only for reducing the temperature exposure of skin and coating materials but, more importantly, for weight reductions in insulation and cooling requirements for the interior of the aircraft. Therefore, the suitability of a skin coating will be largely determined by the temperature reductions the coating can provide by means of IR radiation. Solar reflectivity was considered to be of much less importance for temperature control.

A general consensus was not reached on the relative importance of other coating properties. These other properties, listed more or less in descending order of majority opinion of their importance, were:

1. Ultraviolet, weathering, and thermal-cycling resistance.
2. Resistance to hydraulic oils, fuels, and similar fluids.
3. Abrasion resistance.
4. Resistance to combinations of elevated temperature and reduced pressure.
5. Resistance to rain erosion.
6. Aerodynamic smoothing.
7. Flexibility at high and low temperatures.
8. Electrical conductivity.
9. Hardness before, during, and after temperature exposures.

In answer to the specific question pertaining to the importance of resistance to rain erosion, most of respondents considered it to be of minor importance in the SST program. Most of the skin areas of the SST will present a low angle of attack to the rain and will not be subject to erosion. Rain erosion will be a problem on leading edges but the SST designs do not call for coatings at these areas.

The airframe manufacturers do not consider problems with hot-salt stress corrosion and with heat tinting of skin surfaces to be serious enough in themselves to require coatings. It was unanimously agreed that the lack of known stress-corrosion effects in current aircraft using titanium alloys, the apparent ability of titanium alloys to resist stress corrosion from cyclic exposures, and the improvements in notch ductility produced by special heat treatments will make coatings unnecessary for stress-corrosion protection. Heat tinting discolorations of SST skins would be acceptable from a technical standpoint, but it was generally agreed that airlines would probably require coatings for the sake of appearance.

Opinions were somewhat divided on the probable effects of differences in pressure between ground conditions and flight conditions. The majority opinion was that serious adhesion and stability problems would arise with most coatings when they are heated to the 450-500° F temperature range in the reduced pressures existing at 65,000 to 75,000 ft. (30 mm Hg). Therefore, candidate coatings must be evaluated under these conditions. In the minority opinion, however, coatings that meet temperature requirements at ambient pressures will meet the same temperature requirements in the partial vacuum at cruising altitudes, provided that the coating has been completely cured before subjection to the reduced pressure.

Titanium alloys, particularly Ti-8-1-1, are the leading candidates for the skin material on SST aircraft. Both the Ti-8-1-1 and Ti-6Al-4V, the second leading candidate, will probably be used with special heat treatments for improving notch ductility and resistance to stress corrosion and wet crack propagation. Other titanium alloys mentioned were Ti-4Al-3Mo-1V and Ti-5Al-2.5Sn. Ti-8-1-1 appears to be the leading candidate for engine applications also.

There was a wide range of opinions on whether it would be valuable for the Government to sponsor development of coatings, primers, and surface preparations specifically oriented toward SST requirements. The majority opinion was that the sponsorship of development of improved resins and polymers would probably be valuable, whereas sponsorship of surface preparation and primer studies would be of doubtful value. There were several expressions, however, of the need for the development of a target or tentative specification that could be used by coating suppliers for evaluating innovations in vehicles, fillers, and formulations.

Preliminary Evaluation of Additional Coatings:

The results from the preliminary evaluation of additional coatings not included in earlier parts of the program are listed in Table IX. These coatings are identified by general terms in the table, but more specific identification is available in Table X in the Appendix. On the basis of the preliminary evaluation and visual observation of the as-received samples, none of the additional coatings would be expected to be superior for SST service in comparison with Aluminum-Modified Silicone or Catalytically Cured Silicones.

The Phenolic-Vinyl-Metallic Compound has some desirable characteristics in that it was reported to have high emissivity properties at 500° F, and it provided excellent protection of the substrate in salt spray. Its adherence to the Ti-8-1-1 substrate was poor, however, and this shortcoming must be corrected before it could be given further consideration for SST service.

The Ceramic-Bonded Aluminum adhered well to the titanium-alloy substrate and provided good protection in salt spray. In comparison with Aluminum-Modified Silicone, however, it requires excessively high curing

Table IX. Effects of Temperature Cycling(1) and Salt Spray(2) on Additional Coatings

Temperature Cycling(1)	Salt Spray(2)
Phenolic-Vinyl-Metallic Compound, 0.7 mils thick, dull black color.	
Coating lost adherence and spalled from substrate during first cycle at temperature.	Undamaged Specimen: No rust or significant change in coating appearance Damaged Specimen: Rust streaks from the damaged spots but no changes otherwise.
Ceramic-Bonded Aluminum, 650° F Cure, 2-3 mils thick, dull light-gray color.	
Color changed slightly to darker gray. Adherence not affected.	Undamaged Specimen: No rust or significant change in coating appearance. Damaged Specimen: 3 pin-point spots of rust apparently caused by external contamination. Damaged spots showed no rust but had slight amount of white corrosion product from the coating.
Ceramic-Bonded Aluminum, 1000° F Cure, 2-3 mils thick, dull light-gray color.	
No discernible changes.	Undamaged Specimen: No rust, but color darkened and fine white corrosion product formed. Damaged Specimen: No rust, but color darkened and much fine white corrosion product formed, particularly at damaged spots.
Silicate-Bonded Zinc + Silicate Stainless, 5-6 mils thick, dull gray color.	
Color changed toward light straw color. Adherence not affected.	Undamaged Specimen: 3 pin-point spots of rust apparently caused by external contamination. Some staining from fine white corrosion product. Damaged Specimen: 8 pin-point spots of rust apparently caused by external contamination. Light rust-colored stains and staining from fine white corrosion product. Light rusting at impact damage but none at penetrator damage.
Silicate Stainless, 5-6 mils thick, dull gray color.	
Color changed toward straw color. Adherence not affected.	Undamaged Specimen: General rusting over complete surface. Damaged Specimen: General rusting over complete surface but more severe below the damaged spots. Light rusting at impact damage but none at penetrator damage.

(1) Tested on Ti-8-1-1 panels. Heated to 650° F for 1 hr, air cooled, for 4 cycles except that panels were spray quenched after 3rd heating cycle.

(2) Tested on 4130 steel panels, 1 as received, and 1 damaged at 1 spot with 4 ft-lb impact blow and at 3 spots with Rockwell "C" hardness penetrations.

temperature for color retention and is not as smooth and attractive in appearance. In comparison with Catalytically Cured Silicone, it has low emittance as well as the high curing temperature.

The double coating of Silicate-Bonded Zinc plus Silicate Stainless adhered well and provided adequate corrosion protection, but its color stability when heated to 650° F on titanium-alloy was poor. Also, its original appearance is not as smooth or attractive as the leading coatings. When used alone, the Silicate Stainless provided inadequate corrosion protection and discolored when heated to 650° F.

DISCUSSION

Substrates:

Among the three substrates included in this evaluation, the Ti-8-1-1 alloy was apparently the only one that was consistently susceptible to stress corrosion in one of the environmental conditions included in the program. Rene 41 was apparently unaffected by salt, either in the humid or 550° F exposures, although its inherently erratic bend ductility resulted in some shortening values on the same order as those from the salt-embrittled titanium alloy. The AM 350 SCT substrate exhibited a consistent immunity to corrosion in the hot-salt environment (550° F) but was inconsistently attacked in the humid-salt environment. Although the few specimens that were attacked might have failed by stress-corrosion or stress-accelerated-corrosion mechanisms, the complete immunity of the other replicate specimens indicates that some uncontrolled factor (such as, possibly, fortuitous mode of contact with the welding-rod supports in the exposure racks) was necessary in addition to the presence of humid salt. In accordance with prior expectations, the Ti-8-1-1 substrate was readily susceptible to stress corrosion from dry salt at 550° F but was resistant to embrittlement from humid salt even when previously damaged with cross scratches. Apparently the notch formed by these scratches was not severe enough to cause crack initiation and propagation to parallel the reduced fracture toughness that has been reported (5) for pre-cracked high-strength titanium alloys in the presence of wet salt.

The interviews with personnel employed by airframe manufacturers make it clear that Ti-8-1-1 or other high-strength titanium alloys are, by far, the most likely materials for use on the outer skin of SST aircraft. It is also clear that these personnel do not consider the stress-corrosion problem to be a critical one, despite the great susceptibility the favored alloys have shown in laboratory tests in hot-salt environment under sustained loads on the order of 8 hr or more. This confidence in the ability of titanium to withstand the stress-corrosion threat is mostly based on the following factors:

1. Special heat treatments improve the notch ductility of the titanium alloys and reduce susceptibility to stress corrosion.

2. Stress corrosion has not arisen as a problem in existing supersonic aircraft using titanium alloys.

3. Laboratory tests using cyclic exposures similar to the cyclic conditions expected in SST service have not produced stress-corrosion cracking.

4. The "working" coatings necessary for temperature control of the outer skin will simultaneously protect the substrate from exposure to dry salt.

5. The maximum skin temperature expected to occur with the latest SST designs has been decreased to near the threshold temperature (approximately 450° F) below which stress corrosion of titanium alloys has not been found to occur.

Regardless of the above arguments to the contrary, the many laboratory demonstrations of the rapid deterioration of stressed titanium alloys when exposed to small quantities of hot salt make it imperative that the stress-corrosion problem be given serious consideration. In addition, reasonable arguments can be given to counter those used to depreciate the potential danger of stress-corrosion failures. Examples of such arguments are:

1. Special heat treatments that completely eliminate the susceptibility to stress corrosion have not yet been developed for the titanium alloys most favored for SST service.

2. Existing supersonic aircraft are not subject to the same operational, maintenance, and exposure conditions that will be imposed in commercial SST service.

3. Some SST flight schedules, particularly in future generations, are likely to exceed the cyclic exposure times that have been investigated in the laboratory, and could approach the shortest times that have produced stress corrosion in steady-state laboratory tests.

4. The high-emissivity "working" coatings will protect from stress corrosion only if they are applied and maintained on all areas subjected to stress corrosion exposure regardless of whether temperature control is required in those areas.

5. Maximum skin temperatures in the latest SST designs are still above the threshold temperature for stress-corrosion failures and there is no assurance that the design temperatures will not sometimes be exceeded on operating aircraft.

Embrittlement Mechanisms:

The several investigations that are underway to determine the mechanism of stress-corrosion cracking have not yet resulted in a satisfactory explanation (6). It is well known that stress corrosion cracks in the titanium alloys are intergranular, and there has been some indication that segregations of some of the alloying elements might contribute to the cracking.

The metallographic examinations made in this investigation confirm the intergranular path of the stress-corrosion cracks. As shown in Figures 18 and 20, there is also some indication that segregations might be involved. The crack interruptions shown in Figure 20 appear to occur at grains of the elongated "primary α."

There is a possibility that the improvement in notch ductility (and stress-corrosion resistance) brought about by special heat treatments is due, at least in part, to greater homogeneity in the heat-treated structure. However, unless the heat treatments eliminate the characteristically elongated "primary α," complete homogeneity will not be achieved. It would appear, therefore, that special homogenizing treatments before or during the reduction from billet to sheet form might be more effective than special heat treatments of the finished sheet. Homogenizing treatments at early stages in processing might make it possible to achieve stress-corrosion resistance with conventional heat treating of the final sheet, and without compromising the optimum strength characteristics. Investigation of the possible improvements from early homogenizing treatments would probably be worthwhile.

SST Coating Requirements:

In the opinions of the interviewed airframe representatives, high emissivity in the IR range will be the ruling criterion for choice of an SST skin coating from among those coatings that can withstand the exposure temperatures and that can be cured at room temperature, preferably, or at reasonable elevated temperatures. The results of this and other investigations indicate that protection from stress corrosion might be of equal importance. Fortunately, the same basic type of coating can serve to meet both of these criteria in the temperature range of interest.

From the standpoint of stress corrosion, it appears that any paint-type coating that is impervious to wet salt will protect titanium substrates from stress corrosion if it remains stable and retains adherence at the temperatures involved. Aluminum-Modified Silicone is the only coating that has consistently withstood the exposures involved in the investigation at Southern Research Institute. Catalytically Cured Silicone has also performed well until the current investigation, in which it shredded and spalled from the substrate when exposed to 550° F. However, the airframe companies have found similar coatings to perform well and, in this investigation, the coating had withstood heating to 650° F in previous exposures on other substrates. It can be assumed, then, that Catalytically Cured Silicones are satisfactory if care is taken to assure that the application technique and formulation used will provide adherence to titanium alloys. Because of the many concomitant requirements that have been mentioned previously in this report, it is doubtful that any of the other types of coatings involved in this investigation can successfully compete for SST service in their present form.

From the standpoint of IR emissivity, Aluminum-Modified Silicone, even if formulated to cure catalytically, is not a satisfactory coating because of the low emissivity characteristics of the aluminum pigment. The white-pigmented Catalytically Cured Silicones, on the other hand, possess attractively high IR emissivities. Therefore, Catalytically Cured Silicone, properly pigmented and formulated for elevated-temperature adherence to titanium alloys, is judged to be the most promising coating for major areas of the skin of SST aircraft. In local areas where IR emissivity is not the controlling factor, coatings should be employed for stress-corrosion protection. Aluminum-Modified Silicone will probably provide superior protection for these areas if it can be formulated to cure catalytically without compromising the properties available in the bake-cured coatings. Unpublished results obtained in an investigation by a leading engine manufacturer have shown that a baked Aluminum-Modified Silicone provided meaningful stress-corrosion protection of engine components at temperatures up to 900° F. The baked coatings are feasible for the relatively small engine components since their size makes oven-baking practical.

CONCLUSIONS

1. Duplex annealed Ti-8-1-1 is readily susceptible to stress corrosion from hot salt at 550° F. It is apparently not attacked by humid salt at 95° F within 7000 hr. As measured by bend-ductility shortening, complete deterioration from stress corrosion occurred within 1000 hr of exposure to the hot salt.

2. The AM 350 SCT stainless steel is resistant to stress-corrosion attack from hot salt at 550° F but, depending upon other undefined conditions, can be rapidly attacked by humid salt at 95° F.

3. Rene 41 is apparently resistant to stress-corrosion attack from salt either in the hot condition at 550° F or the humid condition at 95° F. However, its inherent ductility, in the heat-treated condition used for these experiments, is frequently as low as salt-embrittled specimens of the other substrates.

4. Because of the demonstrated potential danger from stress corrosion of titanium alloys in the presence of hot salt, all titanium areas of the SST will probably require protection with a suitable coating regardless of whether coatings are needed for other reasons such as control of IR emissivity.

5. All of the coatings evaluated in this investigation are apparently capable of preventing stress corrosion of the three substrates evaluated. However, because of concomitant requirements for SST service, only Aluminum-Modified Silicone and Catalytically Cured Silicone appear to be satisfactory unless operating temperatures remain below 450° F.

6. If SST operating temperatures remain below 450° F, other coatings previously screened out in earlier investigations of this program (polyimide coatings, for example) might become leading candidates for SST service.

7. Because of the need for coatings to provide high IR emissivity on large areas of the SST outer skin, white-pigmented Catalytically Cured Silicone is the most feasible commercially available coating. In areas not subject to IR control, Aluminum-Modified Silicone is probably superior, especially in engine areas and on engine components exposed to temperatures up to 900° F.

8. Because of slight but significant differences in formulations from one supplier to another, coatings for SST service must ultimately be evaluated on the basis of supplier or trade name rather than on the basis of fundamental type used in this investigation.

9. In order to provide standard requirements and test methods for future evaluations and screening of attempted coating improvements, it would be desirable to develop a target specification for supersonic-aircraft coatings. Because of the difficulties in compromising differences of opinion among the various aircraft manufacturers and coating manufacturers, such a specification must probably be developed by a neutral agency under Government sponsorship.

10. The current attempts to improve the stress-corrosion resistance of titanium alloys by special heat treatments of finished sheet should be supplemented by investigations of the feasibility of providing more homogeneous structures by means of complementary heating and rolling treatments in the early stages of sheet production.

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Birmingham, Alabama
March 23, 1966
7685-1417-XV

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APPENDIX

Table X

Description of Coatings

Type of Coating	Trade Name	Supplier	Description
Coatings Included in the Stress-Corrosion Evaluation			
* Aluminum-Modified Silicone	* XP-310 Aluminum Paint	Dow Corning Corp.	A smooth and bright aluminum-colored coating applied by painting techniques. Usual thickness is 1 to 2 mils. Cost about 2¢/ft². Formulation in lb per 100 gal: 409.2 lb Dow Corning 805 resin (50% NVM), 175.6 lb Dow Corning resin 806A (50% NVM), 256.0 lb Alcoa No. 240 aluminum paste, 69.2 lb Xylene. Requires curing at 480°F for 1 hr. Can be air cured by adding Dow Corning Z-6020 in 1 wt. part to 100 wt. parts XP-310. Pot life of Z-6020 catalyzed paint can be extended to about 8 hr by adding 10 parts Butanol to 100 parts of paint.
* Catalytically Cured Silicone	* Formulation No. 58-5	Air Force Materials Lab. (Code MANE)	A smooth white glossy coating applied by painting techniques. Usual thickness 4 to 5 mils including P-4 primer. Cost data not reported. Formulation not reported. Catalyst is dissolved in Toluene and added prior to coating. Air cures for re-coating in about 1 hr. In-service heating develops full properties.
* Zinc in Silicate Vehicle	* Rust-Ban 191 Galvicon Compound	Humble Oil and Refining Co. Galvicon Corporation	A dull gray coating applied by painting techniques. Usual thickness 3 to 5 mils. Cost, including surface preparation and application, about 30¢/ft². Formulation not reported. Composed of metallic zinc in a silicate vehicle. Air cures in 1 hr, fully cured in 8 hr.
* Electrophoretic Aluminum	* Elphal	BISRA	A smooth bright metallic-aluminum coating applied to sheet or strip substrates by electrophoretic deposition, rolling and heating. Fully developed only for mild-steel substrates. Thickness can be controlled between about 10 to 50 microns. Cost expected to be comparable to galvanizing (on steel).
* Flame-Sprayed Aluminum	* None None	Metco, Inc. Metallizing Co. of L. A.	A relatively rough but bright aluminum-colored coating applied by flame-spraying techniques. Coating can be made smoother and more protective by sanding and sealing with aluminum silicone. Usual thickness about 2 mils. Cost less than 10¢/ft².

* Denotes coatings that were evaluated by Southern Research Institute and the supplier that prepared the samples.

Table X (Continued)

Description of Coatings

Type of Coating	Trade Name	Supplier	Description
Coatings Eliminated after the Simulated - SST - Exposures Evaluation			
* Aircraft White	* A-6110-50	Rust-Oleum Corp.	A smooth glossy coating applied by painting techniques, and similar to catalytically cured silicone. Usual thickness about 2 mils per coat, 2 coats required. Cost about 10¢/ft^2 for 2 coats. Formulation not reported. Requires cure at 500°F for 1 hr.
* Hot-Dip Zinc (Galvanizing)	* None	Metal Coating Corp.	A smooth bright spangled coating of metallic zinc applied by immersion of the substrate in molten zinc. Usual thickness about 3.5 mils. Cost data not reported
* Electroplated Ni and Cd	* None	Commercial platers	A smooth dark coating applied by electroplating and diffusion techniques. Usual thickness about 0.5 mil nickel plus 0.2 mil cadmium. Cost data not reported. Diffusion treatment is 620°F to 640°F for 30 minutes minimum. Specification AMS 2416B.
* Electroplated and Rolled Ni	* None	International Nickel Co.	A smooth bright nickel coating applied by electroplating nickel on billets prior to hot rolling to final thickness. Usual thickness 3 to 5 mils. Cost about 8¢/ft^2 per mil thickness. Fully developed only for steel substrates. Hot rolled at 2250°F (for steel).
Coatings Eliminated after the Experimental-Screening Evaluation			
* Teflon in Silicone Resin	* EC-1697E	Acheson Colloids Co.	A smooth soft coating of purple color applied by spray-painting techniques. Usual thickness 1 mil or less. Cost data not reported. Formulation not reported. Cures at 450°F for 1 hr.
* Zinc Silicate	* ZRAS Zincilate 101 Dimetcote No. 4 (No. 1731)	Koppers Co., Inc. Industrial Metal Protectives, Inc. Amercoat Corp.	A dull gray coating applied by painting techniques and similar to Zinc in Silicate Vehicle. Usual thickness 2 to 3 mils. Cost about 7¢/ft^2. Formulation not reported. Material in form of inorganic zinc silicate. Air cures to dry in 20 min, to handle in 3 hr, and completely in 3 days.
* Flame-Sprayed Aluminum Oxide	* Rokide A	Norton Co., C. M. C. Corp.	A hard speckled coating applied by flame-spray techniques. Usual thickness is 10 to 100 mils. Cost about 35¢/ft^2 for 10 mils. Submitted coating consisted of undercoat of 60Ni-15Cr-25Fe, then Rokide A, then a silicone-resin sealing coat.

* Denotes coatings that were evaluated by Southern Research Institute and the supplier that prepared the samples.

Table X (Continued)

Description of Coatings

Type of Coating	Trade Name	Supplier	Description
Coatings Eliminated after the Experimental-Screening Evaluation (Continued)			
*Heat-Resistant Black	*57X50	Benjamin Foster Co.	Dull black coating applied by painting techniques. Usual thickness is 10 mils. Cost about 30¢/ft^2. Formulation not reported. Will air cure, but for best results requires 250°F to 300°F for 1 hr.
*High-Temperature Varnish	*Pyre-ML Varnish	DuPont	A smooth resinous amber-colored coating applied by painting techniques. Usual thickness is 0.5 to 5 mils. Cost is about 4¢/ft^2 per mil thickness plus application cost. Polyamic acid solution convertible to a polyimide coating. Requires baking between coatings for 10 to 15 min at 150°C and final cure at 200°C for 20 min, 300°C for 3 min, or 400°C for 1 min.
*Aluminized Paint	*Markal DA-9	Markal Company	A smooth bright aluminum-colored coating applied by painting techniques and similar to Aluminum-Modified Silicone. Usual thickness about 1.5 to 1.75 mils. Cost about 3¢/ft^2. Formulation reported only as a modified silicone epoxy thinned with Xylene. Cures at 300°F for 20 to 30 min or 400°F for 10 to 20 min.
*Silicon-Nitrogen Polymer	*None	Southern Research Institute	A slightly amber but almost clear resinous coating applied by painting techniques. Usual thickness 0.2 to 0.7 mils. Cost is about 5¢/ft^2. Formulation: 9 parts of 10 wt/o solution of by-product from preparation of hexaphenylcyclotrisilizane in benzene, to 1 part of 10 wt/o ethylenediamine silizane solution in benzene.
*Fused Minerals	*Korok A-19	Enamel Products Co.	A dull charcoal-gray fused enamel coating applied by painting techniques and fused at 1700°F. Usual thickness 1 to 2 mils. Cost is about 35¢/ft^2. Formulation was not reported.
*White Silicone Resin	*No. 15966 (PV 100X)	Vita-Var Co.	A smooth white glossy coating applied by painting techniques and similar to Catalytically Cured Silicone. Usual thickness is 2 to 5 mils. Cost data not reported. Formulation described only as a white-pigmented 100% silicone resin vehicle. Cures at 275°F for 25 min.

*Denotes coatings that were evaluated by Southern Research Institute and the supplier that prepared the samples.

Table X (Continued)

Description of Coatings

Type of Coating	Trade Name	Supplier	Description
Coatings Eliminated after the Experimental-Screening Evaluation (Continued)			
* Vacuum-Deposited Aluminum	* None None	Ethyl Corporation National Research Corp.	Satin-finish aluminum coating. Usual thickness 0.5 to 2 mils. Cost data not reported but expensive. Deposited by thermal decomposition of organoaluminum on metallurgically clean substrate at 250°C in absence of air or moisture.
* Electroless Nickel	* Kanigen Nickel Alloy	Gen. American Transportation	Glossy metallic coating of nickel-phosphorous alloy. Usual thickness is 3 to 5 mils. Cost data not reported. Deposited chemically without electric current. Composition of chemical-plating bath not reported.
* Thermosetting Polymer	* GIC-805	Nat. Glaco Chemical Corp.	Semigloss white coating. Application procedure not reported. Usual thickness about 2 mils. Cost about \$1.75 to \$3.00 per ft^2. Formulation not reported.
* Diffused Aluminum-Iron	* Coating C-10 Alonizing	Haynes Stellite Co. Alon Processing, Inc.	Diffused metallic coating. Usual thickness about 5 mils. Cost data not reported. Applied by pack-cementation in an aluminum particulate material within a sealed metal retort and heated to an unreported elevated temperature for diffusion.
Additional Coatings Eliminated after Preliminary Evaluations			
* Hardened Teflon in Silicone	* GP-797-A	Acheson Colloids Co.	A dark coating produced by adding hardening pigments to the EC-1697E (TFE-Silicone) coating listed previously. Usual thickness, cost, and formulation data not reported. Cures at 400°F for 30 min.
* Tungsten Disulphide Phenolic	* GP-713	Acheson Colloids Co.	A dull black coating. Usual thickness 0.3 to 0.5 mil per coat. Cost data not reported. Composition is tungsten disulphide and graphite in a phenolic resin. Contains 56% solids content. Best diluted with special solvent blend obtainable from supplier. Cures in 1 hr at 300°F after 10 min air dry to avoid trapping of solvents
* Metallic Gold	* B-25 or B-26	Barrier, Inc.	Metallic gold coating applied by a proprietary process. Usual thickness 0.5 mil. Cost about \$1.85/ft^2. Requires bake at 400-900°F.
* Aluminum-Modified CCS	* None	AFML (Code MANE)	Same coating as Catalytically Cured Silicone except that it contains some aluminum pigment. Coating has white glossy appearance but slightly rougher than CCS.

* Denotes coatings that were evaluated by Southern Research Institute and the supplier that prepared the samples

Table X (Continued)

Description of Coatings

Type of Coating	Trade Name	Supplier	Description
Additional Coatings Eliminated after Preliminary Evaluations (Continued)			
*Complex Phenolic-Vinyl	*An-Cor 13-7	George E. Swett and Co.	Smooth semi-gloss black coating applied by painting techniques. Usual thickness about 0.5 mil. Cost about 3¢/ft². Formulation described only as a metallic derivative dispersed in a complex mixture of phenolics and vinyls. Cures at 300°F for 30 min.
*Aluminum Inorganic	*Serme Tel (Type W)	Teleflex, Inc.	Dull light-gray coating that is applied by painting techniques and can be polished to a bright finish. Usual thickness is 1.5 to 3 mils (2 coats). Cost for 2 coats about 15¢/ft². Formulation descibed only as a water-base inorganically (ceramic) bonded aluminum. Cures in about 15 min at 650°F.
*Aluminum Inorganic (1000)	*Serme Tel (Type W)-1000	Teleflex, Inc.	Same as previous coating except post-cured for 90 min at 1000°F to make the coating fully conductive.
*Inorganic Zinc Silicate	*Plasite 1000	Wisconsin Prot. Coating Corp.	Undercoat for Plasite 1010 stainless
*Stainless Zinc Silicate	*Plasite 1010	Wisconsin Prot. Coating Corp.	Relatively rough dull-gray coating applied by painting techniques. Usual thickness combined with Plasite 1000 is 5 to 6 mils. Cost data not reported. Formulation not reported. Air cures.
Coatings Eliminated after the Industrial-Survey Evaluation			
Hot-Dip Aluminum	Aluminized Steel Type II	Armco Steel Corporation	Usual thickness 2 mils. Cost is $9.25 per 100 lbs plus extras.
Diffused Aluminum-Nickel	Coating C-3	Haynes Stellite Co.	Applied by pack cementation and diffusion. Usual thickness about 5 mils. Cost data not reported.
Diffused Chromium	Chromalloy Type G Alphatizing	Chromalloy Corporation Alloy Surfaces Company	Applied by pack cementation (Chromalloy) or gas process (Alphatizing) and diffusion treatments. Usual thickness 0.5 to 3 mils. Cost about 6¢/ft².
Fused Nickel-Chromium	Nicrocoat	Wall Colmonoy Corp.	Applied by coating with slurry by painting techniques and fused at high temperature (1800-2150°F). Thicknesses used range between 0.1 to 10 mils. Cost for material about 25¢/ft² per mil.
Explosively Bonded Metals	None	DuPont (Explosives Dept.)	Corrosion-resistant metals bonded by explosive techniques. Minimum thickness 5 mils. Cost data not developed.

*Denotes coatings that were evaluated by Southern Research Institute and the supplier that prepared the samples.

Table X (Continued)

Description of Coatings

Type of Coating	Trade Name	Supplier	Description
Coatings Eliminated after the Industrial-Survey Evaluation (Continued)			
Chemically Cured Epoxy	Super Koropon 521-002	DeSoto Chemical Coatings, Inc.	Glossy white applied by painting techniques. Usual thickness is 1.8 to 2.2 mils. Cost about 4¢/ft^2. Used with curing solution No. 910-037.
Epoxy Resin	Vibro-Flo E-201	Armstrong Resins, Inc.	Applied by fluidized-bed, spray, or flow processes. Usual thickness 5 to 30 mils. Cost data not reported
Teflon in Phenolic Resin	Emralon 310	Acheson Colloids Co.	Applied by painting techniques. Usual thickness 1 mil or less. Cost data not reported. Cures 1 hr at 300°F.
Acrylic Copolymer	SX-228	Reichhold Chemicals, Inc.	Applied by painting techniques. Usual thickness about 6 mils. Cost 50¢ to 75¢/ft^2 for 6 mils. Cures at 300°F in 30 min.
Epoxy Polymer	Resiweld No. 224	H. B. Fuller Co.	Applied by painting techniques. Usual thickness 1 to 1.5 mils. Cost about 1.5¢/ft^2. Cures in 2 hr at 140°F to 5 min at 400°F
Chemically Cured Polyurethane	Polyurethane 821-001	DeSoto Chemical Coatings, Inc.	White enamel applied by painting techniques. Usual thickness is 1.8 to 2.2 mils. Cost about 5¢/ft^2 for 2 mils. Used with curing solution 910-028.
Si-N-Phenyl Polymer	None	Southern Research Institute	A diphenyl silazane polymer. Usual thickness is 0.2 to 0.7 mils. Cost is about 5¢/ft^2. Cures at 200°F for 5 hr or 570°F for 2 hr.
Organophosphorous-Metal Oxide	None	Southern Research Institute	A development coating. No information available.
Aluminum-Ceramic	Alcermet S1177	Solar Aircraft Co.	Applied by flame-spray techniques and firing. Usual thickness is 3 to 8 mils. Cost data not reported. Fired at 1450 to 1550°F.
Non-Vitreous Ceramic	None	Ceramco, Inc.	Applied by flame spraying. Usual thickness 2 to 4 mils. Cost not reported.

Table X (Continued)

Description of Coatings

Type of Coating	Trade Name	Supplier	Description
Coatings Eliminated after the Industrial-Survey Evaluation (Continued)			
Non-Heat-Tint Film	Connerkote	Allegheny Ludlum Steel Corp.	Applied by painting techniques and baking. Usual thickness is 0.5 to 1 mil. Cost and curing data not reported.
Humidity-Sealing Paint	Humiseal Type 1C40	Columbia Technical Corp.	Applied by dipping and baking. Usual thickness about 1 mil. Cost about 16¢/ft^2. Cures 8 hr at 250°C.
High-Temp. Elastomeric	3M EC-1937 B/A	3 M Company	Applied by painting techniques. Usual thickness 3 to 5 mils. Cost about 85¢/ft^2 for 3 mil to \$1.38/ft^2 for 5 mil. Complex cure.
Bonded Paint	Titanine 805 Series	Seagrave Corp.	Application procedure not reported. Usual thickness about 1 mil. Cost 5¢ to 7¢/ft^2. Cures at 450°F for 1 hr.
Black Passivating Treatment	Black Magic	Mitchell-Bradford Co.	Applied by dipping procedures. Usual thickness 0.1 mil or less. Cost about 1¢/ft^2.
Colloidal Graphite	Aquadag	Acheson Colloids Co.	Colloid graphite in water applied by painting techniques. Usual thickness 1 mil or less. Cost data not reported.

QUESTIONNAIRE
ON SST COATINGS

The following questionnaire was used as the basis for personal interviews with technical personnel of five leading airframe manufacturers identified herein as companies A through E. The summary answers provided by the interviewed personnel are listed immediately following each question. Of the five aircraft companies involved, companies A, C, and E have had extensive manufacturing or design experience with supersonic aircraft and companies B and D have not.

1. The results from the investigations at Southern Research Institute show that resin coatings of the silicone type (Aluminum-Modified Silicone and Catalytically cured silicone) show the most promise among existing coatings for SST service. Based on your experience, what is your opinion of these results?

☐ Concur completely   [B] [C] [E] Substantially correct   [A] Inconclusive

☐ Disagree   [D] Other comment

Other Comment:

A. Polyimide coatings, cermet coatings offer possibilities.
B. Air-cured coatings necessary.
C. Desire catalytically cured system, stable decorative colors, good far-infra-red emittance. Amount and choice of fillers would be guided by effects on IR emissivity.
D. No experience in this requirement range.
E. Flat white catalytically cured silicone is the best at present.

2. Are you aware of a more promising coating not covered in the investigation at Southern Research Institute? If so, what?

A. Polyimide (up to 450°F).
B. No.

C. No. However, roll cladding or dip-coating with aluminum is a possibility. Paint would still be needed for IR emittance. Polyimides have some promise except for curing temperatures and pigment discoloration.

D. No.

E. Flat white catalytically cured silicone (also available in gloss and semi-gloss).

3. Of the properties not investigated by Southern Research Institute, which do you consider the most important for future investigation? Indicate order of importance by 1, 2, 3, etc.

B1 C9 D4	Rain-erosion resistance
A3 B1 C7 D2	Abrasion resistance
A1 C1 D3 E1	IR Absorptivity and emissivity
A2 C8 D1 E1	Ultraviolet resistance
A4 B2 C4 D3 E1	Hydraulic fluids and fuels resistance
D3	Electrical conductivity

Others:

C2. Flexibility at -65°F and at 500°F.
C3. Reduced pressure (30 mm Hg).
C5. Hardness before, during, and after temperature exposure.
C6. Weathering and thermal cycling.
E1. Weight loss at elevated temperature and reduced pressure.
E1. Aerodynamic smoothing.

4. Do you expect resistance to rain erosion to be important in a coating for SST service?

☐	Primary importance
B	Significant importance
A C D E	Insignificant importance
☐	No importance

Other comment:

A. Might be important on radomes.
C and E. Important only at leading edges where more effective materials might be used.

5. In the investigations at Southern Research Institute, comparisons have purposely been restricted mostly to coatings of different types. Do you believe comparisons should also be made between competitive coatings (trade names) that fall within the same type?

A			
B	Yes	D	No
C			
E			

Other:

B. Suppliers should work to pre-specified requirements.
C. Yes, particularly for paint coatings.
E. Many suppliers important to aircraft industry were not included in the original survey.

6. Do you expect hot-salt stress corrosion to be such a serious problem that protective coatings will be required for that reason alone, if for no other reason?

		A	
☐	Yes	B	No
		C	
		D	
		E	

Other:
A. Wet crack propagation important in engine areas.
B. Not convinced it is a serious problem but possibility of problem should be recognized.
D. Some possibility of problem, especially wet crack propagation.

7. Do you expect heat-tinting to be such a serious problem that protective coatings will be required for that reason alone, if for no other reason:

B E	Yes	A C D	No

Other:

C. Airline customers might need convincing, however.
E. Coatings needed for appearance even if heat-tinting is not a problem.

8. Do you expect the differences in pressure between ground conditions and flight conditions to be significant in the performance of coatings?

B C E	Yes	A D	No

Other:

B. Effects on adhesion.
D. But should be considered.

9. In your poinion what are the three leading candidates for the skin material on SST aircraft?

	A	B	C
1.	Ti-8-1-1	Ti-8-1-1	Ti-6-4 (Special Heat Treatment)
2.	Ti-6-4	Ti-6-4	Ti-4Al-3Mo-1V (Special Heat Treatment
3.	-	Ti-5Al-2.5Sn	Ti-8-1-1 (Mill Annealed)

	D	E
1.	Ti-8-1-1 or Ti-6-4	—
2.	PH Stainless Steels	—
3.	INCO 718	—

10. In your opinion would it be valuable for the Government to sponsor coating development specifically oriented toward SST coating requirements?

B C	Extremely valuable	C D E	Valuable
A C	Doubtfully valuable	☐	No value

Comment:

C. Opinion divided, depending upon type of development.
D. Only in context of the question and current approach to SST development.
E. But problem not critical.

11. In your opinion would it be valuable for the Government to sponsor a study of surface preparation and primers to improve the adherence (and, possibly, the performance) of the more promising coatings?

B	Extremely valuable	A	Valuable
C D E	Doubtfully valuable	☐	No value

12. From the standpoint of aircraft manufacture, which type of coating would be most preferable? Designate order of preference with 1, 2, 3, etc.

☐ A solid, rolled, metal coating (such as electrophoretically deposited aluminum) that would be obtained on the original sheet, that would be formed along with the sheet, that would require edge protection only, and that would not require replacement for the life of the aircraft.

☐ A paint-type resin coating that would be sprayed on after assembly, that would require heat curing at temperatures up to 450°F, that would require replacement at 2-year overhaul periods, and that would require occasional touch-up repair.

[A1] [B1] [C1] [E1] A paint-type resin coating that would be sprayed on after assembly, that would cure at room temperature, that would require replacement annually, and that would require more frequent touch-up repair.

☐ An electroplated metallic coating that would be deposited after forming but before assembly, that would require replacement at 5-year periods, and that would require infrequent touch-up repair by a Dalic-type process.

☐ A flame-sprayed ceramic coating that would be applied after assembly, that would require replacement at 5-year periods, and that would require occasional touch-up repair.

☐ A flame-sprayed metallic coating that would be applied after assembly, that would require replacement at 2-yr overhaul periods, and that would require occasional touch-up repair.

Additional Comment:

A1 and E1. Answer should be altered to need replacement every few years and occasional repair.
C1. Best listed answer, but subject to numerous qualifications because of assumptions made in the choices.
D. Opinions too divided to summarize.

13. In your opinion, should the NASA Special Committee on Materials Research for SST establish a suggested standard procedure (based upon the work at Southern Research Institute) for screening evaluation of present and future candidate coatings for SST service?

[D] Yes [A] No

Other Comment:

B. Aircraft companies could probably not get together on requirements but tentative specification would be helpful.
C. Doubtful of SRI specimen for wide use. General approach by SRI was good, but current SST designs indicate 550°F temperature is too high.
E. Need target specification that should be met before coatings are submitted to aircraft manufacturers.

14. What additional comments, suggestions, or information do you have to offer on the SST coatings problem in general, the work performed by Southern Research Institute, and the work remaining to be done in the future? We would appreciate your candid comments on your confidence in the data already accumulated; the additional information needed to meet design and manufacturing problems; and the relative importance and restrictions for such factors as weight, visual apparance, reflectivity, thermal and electrical conductivity, erosion resistance, flame resistance, resistance to fuels and other solvents, edge exposure, joining methods, curing temperatures, material cost, and application costs.

A. Interior coatings will be required to obtain corrosion resistance, acid resistance, etc. (problems from coffee, urine, alcohol).

The temperature regime will be on the order of 550°F rather than 650°F. Erosion resistance and thermal conductivity will be required on leading edges. Resistance to oils, fuels, and cleaning materials will be required. Material and application costs are important but not as critical as obtaining satisfactory coatings.

Changes in absorptivity and emissivity for exterior coatings due to temperature or aging will be critical.

Ambient curing of coatings is very important.

Exterior coatings should not be more than two mils in total thickness to minimize weight. Temperature cycling tests from -65°F to 550°F will be required.

Surface preparation such as descaling, anodizing, or surface treating of titanium materials to obtain adhesion will be critical.

B. Have full confidence in the data from Southern Research Institute. All factors mentioned are influential for coatings but their importance varies depending upon the mission requirement.

Relative importance of the various vactors must be assessed as the design proceeds.

C. The hot-salt corrosion problem does not seem to be so severe a problem with the notch-ductile SST skin candidates now under consideration. The materials are more resistant, and the more probable exposure temperature is 425°F with occasional excursions to 500°F. SRI work was with continuous stress exposure to salt at 550°F. Our experience indicates no problems (on Ti-8-1-1) under cycle exposure to salt and temperature.

A very important consideration is IR emittance of the coating. Emittance must exceed 0.8. Reflectivity is relatively unimportant, although a high solar reflectance is desirable. Weight is a very important consideration, as is visual appearance and resistance to fuels and solvents. Material cost and application cost are rather important. Curing cycles and temperatures are quite important. Room-temperature curing is very desirable.

Joining methods are important only if the coating would be applied prior to final assembly. In case the material was applied prior to final assembly, interference with weldability would be critical. Edge exposure would be important in such cases also, with erosion of secondary importance. Thermal and electrical conductivity are important only if the material is applied prior to assembly and if the tendency of the coating to spall is affected.

Additional information needed is:

1. Flexibility of coatings at high and low temperatures both before, during, and after aging.
2. Resistance of coating to deterioration by weather, moisture, fuels, and solvents, thermal cycling, ultraviolate exposure, and possibly ozone.
3. Effects of reduced pressure.

4. Ease of application.
5. Cost.
6. Appearance before and after aging.
7. IR emittance and solar reflectance.
8. Hardness and abrasion resistance.

Concerning confidence in data gathered by SRI:

1. Question remains - how does continuous exposure to hot salt correlate with expected service exposure?
2. The data presented appears valid within the rather limited scope of the examination.
3. Why - when the catalytically cured silicone coating spalled off early in exposure - was exposure of these specimens continued?
4. What attempts were made to determine the reason for the spalling?
5. What surface pretreatment was used for each paint type of coating?

D. None.

E. Optimum thickness (or weight) of the coating is very important.

Our coating (flat white catalytically cured silicone) probably meets the requirements for outer skin but more critical problems are posed by materials for aerodynamic smoothing and for rain erosion.

A tentative general specification would be valuable for vendors to use as a guide or goal for developing suitable formulations. Our own specification should provide a good start for a tentative general specification.

Emissivity is one of the most important characteristics in a coating because these must be "working" coatings that hold down the temperatures as much as possible.

Table XI

Bend-Ductility Data from UNSCRATCHED Ti-8-1-1
After Exposure to 550°F WITHOUT SALT for Durations Shown

1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
Bare							
TOU1H1A	1.484	TOU1H3A	2.135	TOU1H5A	2.360	TOU1H7A	2.465
TOU1H1B	1.765	TOU1H3B	2.200	TOU1H5B	2.380	TOU1H7B	2.460
TOU1H1C	1.885	TOU1H3C	2.120	TOU1H5C	2.315	TOU1H7C	2.390
Coated with Aluminum - Modified Silicone							
T1U1H1A	1.819	T1U1H3A	1.630	T1U1H5A	2.460	T1U1H7A	2.325
T1U1H1B	1.285	T1U1H3B	1.635	T1U1H5B	2.365	T1U1H7B	2.465
T1U1H1C	1.828	T1U1H3C	1.781	T1U1H5C	2.465	T1U1H7C	2.440
Coated with Catalytically Cured Silicone(1)							
T2U1H1A	1.790	T2U1H3A	1.690	T2U1H5A	2.450	T2U1H7A	2.385
T2U1H1B	1.794	T2U1H3B	1.670	T2U1H5B	2.460	T2U1H7B	2.470
T2U1H1C	1.730	T2U1H3C	1.686	T2U1H5C	2.395	T2U1H7C	2.455
Coated with Zinc in Silicate Vehicle							
T3U1H1A	0.719	T3U1H3A	0.670	T3U1H5A	0.890	T3U1H7A	0.798
T3U1H1B	0.748	T3U1H3B	0.660	T3U1H5B	0.835	T3U1H7B	0.885
T3U1H1C	0.660	T3U1H3C	0.695	T3U1H5C	0.540	T3U1H7C	0.800
Coated with Electrophoretically Deposited Aluminum							
T4U1H1A	0.554	T4U1H3A	2.100	T4U1H5A	1.490	T4U1H7A	2.460
T4U1H1B	1.566	T4U1H3B	1.710	T4U1H5B	1.700	T4U1H7B	0.960
T4U1H1C	0.780	T4U1H3C	2.200	T4U1H5C	1.940	T4U1H7C	1.690
Coated with Flame-Sprayed Aluminum							
						T5U1H7A	1.290
						T5U1H7B	0.950

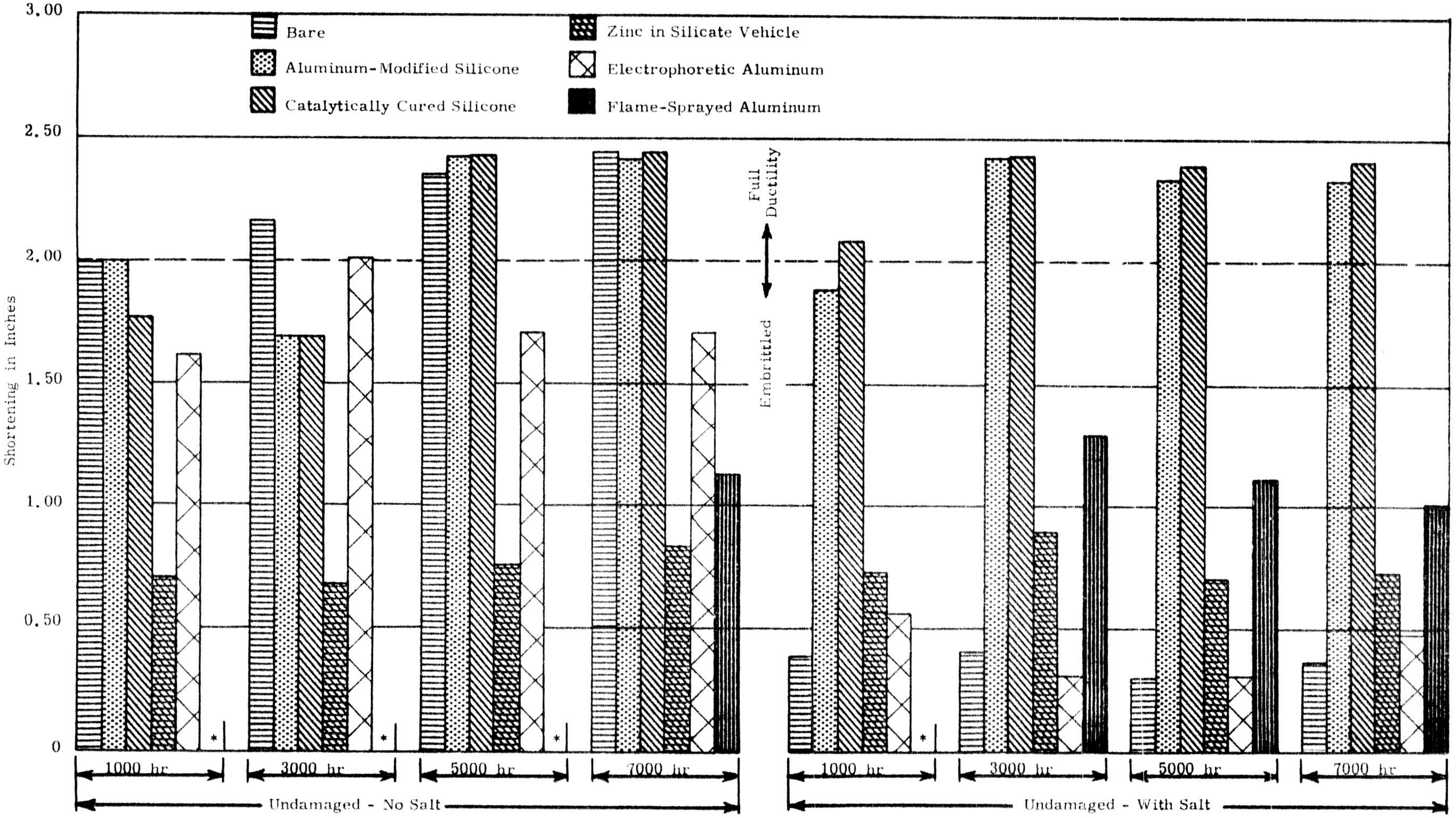
(1) Catalytically Cured Silicone spalled from substrate within 48 hr after exposure to 550°F.

Table XII

Bend-Ductility Data from UNSCRATCHED Ti-8-1-1
After Exposure to 550°F WITH SALT for Durations Shown

1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
Bare							
TOU2H1A	0.315	TOU2H3A	0.497	TOU2H5A	0.350	TOU2H7A	0.580
TOU2H1B	0.530	TOU2H3B	0.365	TOU2H5B	0.320	TOU2H7B	0.280
TOU2H1C	0.334	TOU2H3C	0.362	TOU2H5C	0.230	TOU2H7C	0.225
Coated with Aluminum - Modified Silicone							
T1U2H1A	1.810	T1U2H3A	2.336	T1U2H5A	2.110	T1U2H7A	2.420
T1U2H1B	1.405	T1U2H3B	2.481	T1U2H5B	2.450	T1U2H7B	2.255
T1U2H1C	1.790	T1U2H3C	2.470	T1U2H5C	2.470	T1U2H7C	2.300
Coated with Catalytically Cured Silicone(1)							
T2U2H1A	1.700	T2U2H3A	2.400	T2U2H5A	2.290	T2U2H7A	2.465
T2U2H1B	1.450	T2U2H3B	2.455	T2U2H5B	2.470	T2U2H7B	2.312
T2U2H1C	1.815	T2U2H3C	2.463	T2U2H5C	2.430	T2U2H7C	2.455
Coated with Zinc in Silicate Vehicle							
T3U2H1A	0.700	T3U2H3A	0.990	T3U2H5A	0.950	T3U2H7A	0.580
T3U2H1B	0.720	T3U2H3B	1.000	T3U2H5B	0.540	T3U2H7B	0.895
T3U2H1C	0.775	T3U2H3C	0.688	T3U2H5C	0.620	T3U2H7C	0.895
Coated with Electrophoretically Deposited Aluminum							
T4U2H1A	0.320	T4U2H3A	0.290	T4U2H5A	0.265	T4U2H7A	0.280
T4U2H1B	0.894	T4U2H3B	0.283	T4U2H5B	0.320	T4U2H7B	0.300
T4U2H1C	0.480	T4U2H3C	0.355	T4U2H5C	0.345	T4U2H7C	0.820
Coated with Flame-Sprayed Aluminum							
		T5U2H3A	1.259	T5U2H5A	1.185	T5U2H7A	1.120
		T5U2H3B	1.305	T5U2H5B	0.975	T5U2H7B	1.060
				T5U2H5C	1.190	T5U2H7C	0.890

(1) Catalytically Cured Silicone spalled from substrate within 48 hr after exposure to 550°F



* No specimen available for exposure.

Figure 30. Bend-Ductility Results from UNSCRATCHED Ti-8-1-1 After Exposure to 550° F.

Table XIII

Bend-Ductility Data from SCRATCHED Ti-8-1-1
After Exposure to 550°F WITHOUT SALT for Durations Shown

1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
Bare							
TOD1H1A	1.450	TOD1H3A	1.170	TOD1H5A	1.680	TOD1H7A	1.690
TOD1H1B	0.955	TOD1H3B	1.710	TOD1H5B	1.770	TOD1H8B	1.265
TOD1H1C	1.345	TOD1H3C	1.597	TOD1H5C	1.230	TOD1H7C	1.545
Coated with Aluminum - Modified Silicone							
T1D1H1A	1.561	T1D1H3A	1.160	T1D1H5A	1.300	T1D1H7A	1.440
T1D1H1B	1.254	T1D1H3B	1.590	T1D1H5B	1.270	T1D1H7B	1.880
T1D1H1C	1.214	T1D1H3C	1.400	T1D1H5C	1.485	T1D1H8C	1.510
Coated with Catalytically Cured Silicone(1)							
T2D1H1A	1.365	T2D1H3A	1.660	T2D1H5A	1.400	T2D1H7A	1.345
T2D1H1B	1.152	T2D1H3B	1.910	T2D1H5B	1.480	T2D1H7B	1.600
T2D1H1C	1.510	T2D1H3C	1.900	T2D1H5C	1.780	T2D1H7C	2.175
Coated with Zinc in Silicate Vehicle							
T3D1H1A	0.748	T3D1H3A	0.778	T3D1H5A	0.800	T3D1H7A	1.100
T3D1H1B	0.750	T3D1H3B	0.795	T3D1H5B	0.920	T3D1H7B	0.945
T3D1H1C	0.779	T3D1H3C	0.770	T3D1H5C	0.500	T3D1H7C	1.060
Coated with Electrophoretically Deposited Aluminum							
		T4D1D3A	2.275	T4D1H5A	2.365	T4D1H7A	2.460
		T4D1H3B	2.283	T4D1H5B	1.960	T4D1H7B	2.450
				T4D1H6C	2.295	T4D1H7C	1.700
Coated with Flame-Sprayed Aluminum							
				T5D1H5A	1.060	T5D1H7A	1.055
				T5D1H5B	0.930	T5D1H7B	1.110

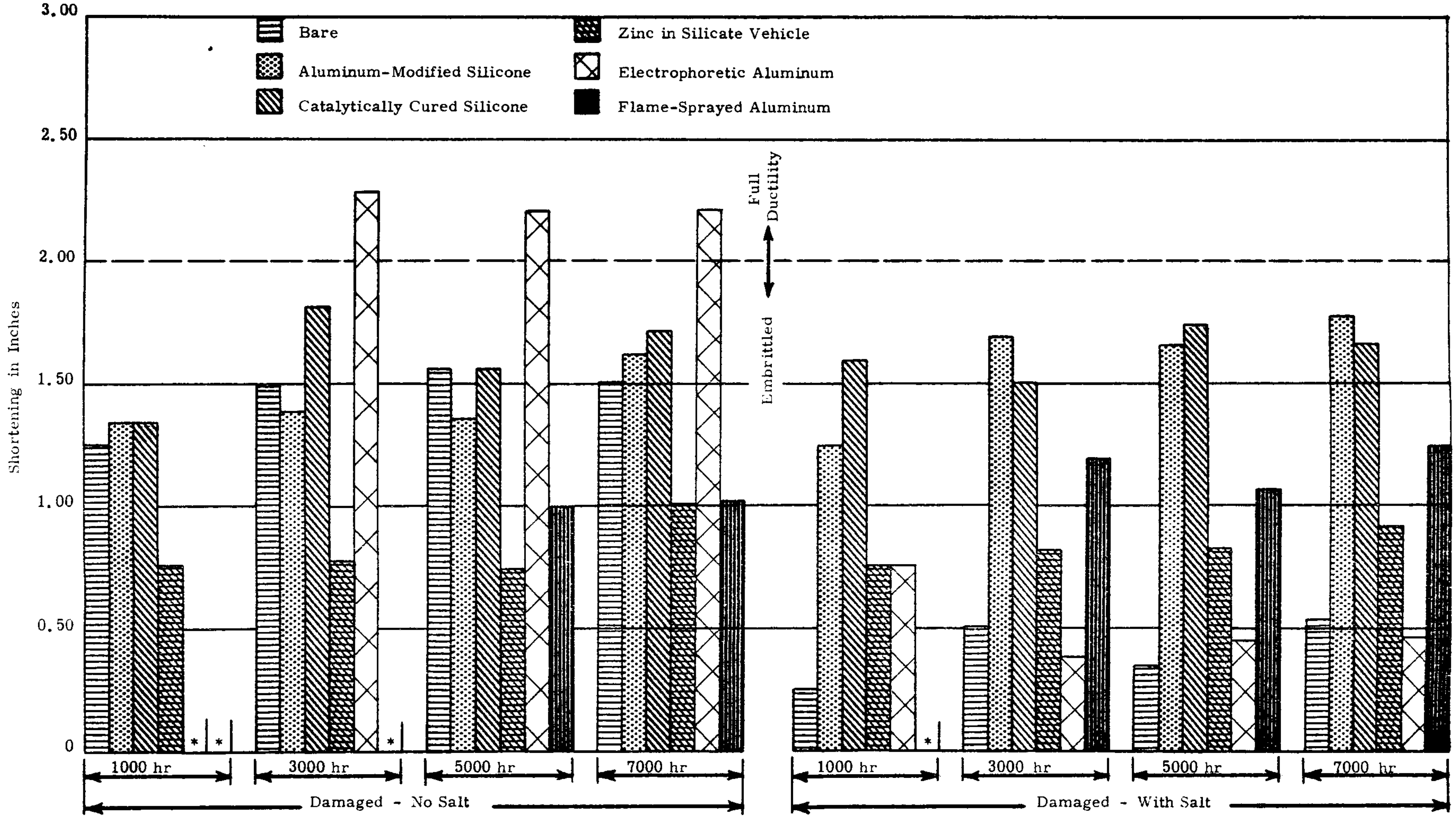
(1) Catalytically Cured Silicone spalled from substrate within 48 hr after exposure to 550°F

Table XIV

Bend-Ductility Data from SCRATCHED Ti-8-1-1
After Exposure to 550°F WITH SALT for Durations Shown

1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
Bare							
TOD2H1A	0.236	TOD2H3A	0.590	TOD2H5A	0.205	TOD2H7A	0.730
TOD2H1B	0.305	TOD2H3B	0.286	TOD2H5B	0.335	TOD2H7B	0.550
TOD2H1C	0.230	TOD2H3C	0.670	TOD2H5C	0.500	TOD2H7C	0.310
Coated with Aluminum - Modified Silicone							
T1D2H1A	1.348	T1D2H3A	1.710	T1D2H5A	1.560	T1D2H7A	1.540
T1D2H1B	1.167	T1D2H3B	1.960	T1D2H5B	1.730	T1D2H7B	1.665
T1D2H1C	1.230	T1D2H3C	1.410	T1D2H5C	1.670	T1D2H7C	2.120
Coated with Catalytically Cured Silicone(1)							
T2D2H1A	1.444	T2D2H3A	1.135	T2D2H5A	2.000	T2D2H7A	1.335
T2D2H1B	1.442	T2D2H3B	1.440	T2D2H5B	1.470	T2D2H7B	1.555
T2D2H1C	1.469	T2D2H3C	1.925	T2D2H5C	1.750	T2D2H7C	2.080
Coated with Zinc in Silicate Vehicle							
T3D2H1A	0.630	T3D2H3A	0.580	T3D2H5A	0.830	T3D2H7A	0.910
T3D2H1B	0.815	T3D2H3B	0.925	T3D2H5B	0.860	T3D2H7B	1.040
T3D2H1C	0.834	T3D2H3C	0.950	T3D2H5C	0.790	T3D2H7C	0.800
Coated with Electrophoretically Deposited Aluminum							
T4D2H1A	0.470	T4D2H3A	0.280	T4D2H5A	0.390	T4D2H7A	0.390
T4D2H1B	0.340	T4D2H3B	0.445	T4D2H5B	0.600	T4D2H7B	0.600
T4D2H1C	1.480	T4D2H3C	0.420	T4D2H5C	0.345	T4D2H7C	0.390
Coated with Flame-Sprayed Aluminum							
		T5D2H3A	1.290	T5D2H5A	0.910	T5D2H7A	1.260
		T5D2H3B	1.090	T5D2H5B	1.245	T5D2H7B	1.230

(1) Catalytically Cured Silicone spalled from substrate within 48 hr after exposure to 550°F



* No specimen available for exposure.

Figure 31. Bend-Ductility Results from SCRATCHED Ti-8-1-1 After Exposure to 550° F.

Table XV

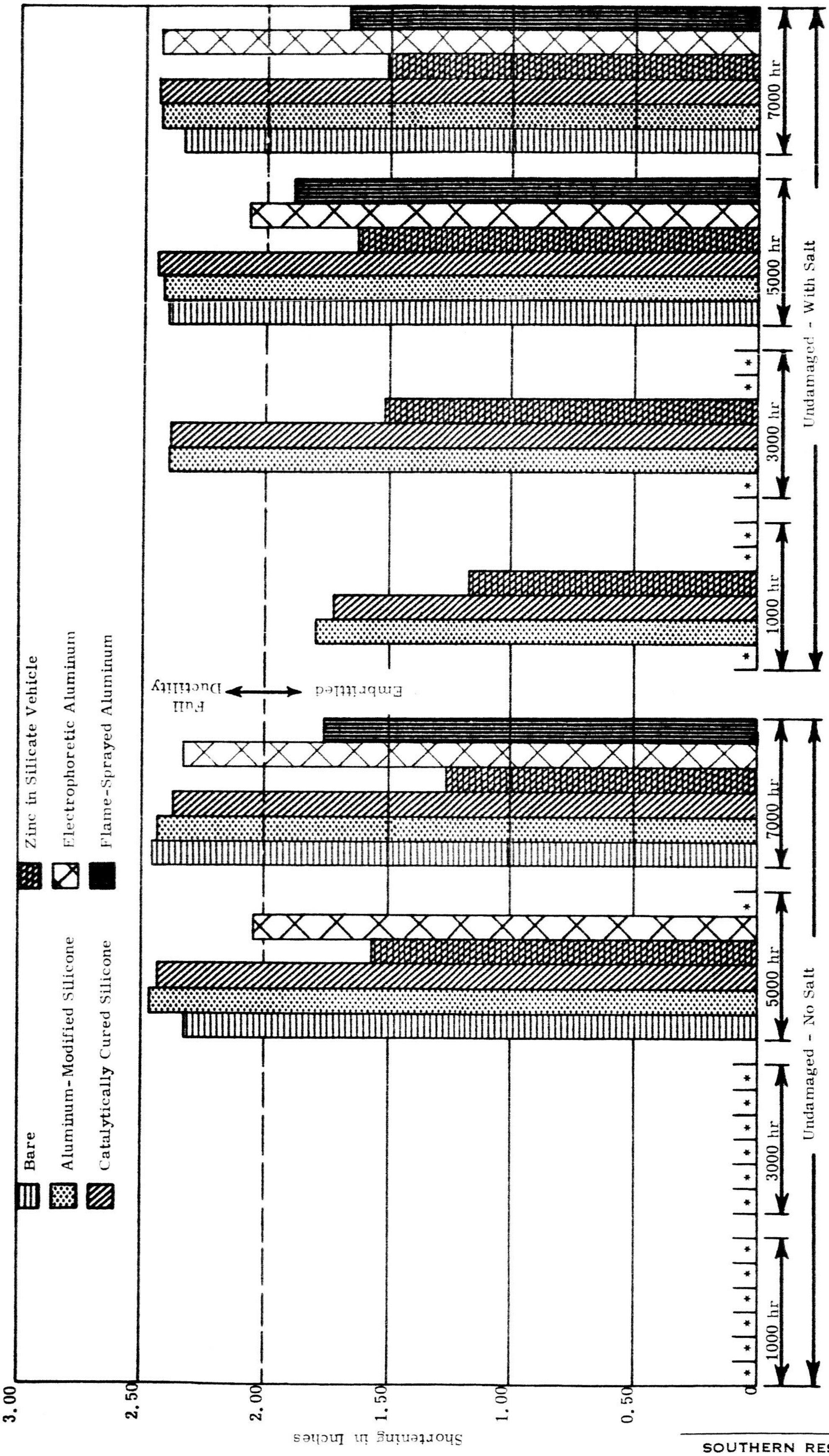
Bend-Ductility Data from UNSCRATCHED Ti-8-1-1 After Exposure to HUMID conditions WITHOUT SALT for Durations Shown

1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
Specimen	**In.**	**Specimen**	**In.**	**Specimen**	**In.**	**Specimen**	**In.**
Bare							
				TOU1L5A	2.215	TOU1L7A	2.460
				TOU1L5B	2.430	TOU1L7B	2.450
Coated with Aluminum - Modified Silicone							
				T1U1L5A	2.462	T1U1L7A	2.455
				T1U1L5B	2.460	T1U1L7B	2.420
						T1U1L7C	2.430
Coated with Catalytically Cured Silicone							
				T2U1L5A	2.430	T2U1L7A	2.428
				T2U1L5B	2.425	T2U1L7B	2.415
						T2U1L7C	2.270
Coated with Zinc in Silicate Vehicle							
				T3U1L5A	1.530	T3U1L7A	1.420
				T3U1L5B	1.600	T3U1L7B	1.310
Coated with Electrophoretically Deposited Aluminum							
				T4U1L5A	2.365	T4U1L7A	2.370
				T4U1L5B	1.720	T4U1L7B	2.290
Coated with Flame-Sprayed Aluminum							
						T5U1L7A	1.790
						T5U1L7B	1.720

Table XVI

Bend-Ductility Data from UNSCRATCHED Ti-8-1-1 After Exposure to HUMID Conditions WITH SALT for Durations Shown

1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
Bare							
				TOU2L5A	2.330	TOU2L7A	2.435
				TOU2L5B	2.465	TOU2L7B	2.250
Coated with Aluminum - Modified Silicone							
T1U2L1A	1.750	T1U2L3A	2.470	T1U2L5A	2.440	T1U2L7A	2.375
T1U2L1B	1.838	T1U2L3B	2.340	T1U2L5B	2.360	T1U2L7B	2.455
T1U2L1C	1.795	T1U2L3C	2.370	T1U2L5C	2.457	T1U2L7C	2.460
Coated with Catalytically Cured Silicone							
T2U2L1A	1.500	T2U2L3A	2.440	T2U2L5A	2.450	T2U2L7A	2.430
T2U2L1B	1.760	T2U2L3B	2.445	T2U2L5B	2.437	T2U2L7B	2.440
T2U2L1C	1.925	T2U2L3C	2.285	T2U2L5C	2.429	T2U2L7C	2.442
Coated with Zinc in Silicate Vehicle							
T3U2L1A	1.100	T3U2L3A	1.450	T3U2L5A	1.675	T3U2L7A	1.545
T3U2L1B	1.225	T3U2L3B	1.490	T3U2L5B	1.675	T3U2L7B	1.530
T3U2L1C	1.162	T3U2L3C	1.620	T3U2L5C	1.530	T3U2L7C	1.480
Coated with Electrophoretically Deposited Aluminum							
				T4U2L5A	2.060	T4U2L7A	2.425
				T4U2L5B	2.070	T4U2L7B	2.430
Coated with Flame-Sprayed Aluminum							
				T5U2L5A	1.775	T5U2L7A	1.680
				T5U2L5B	1.990	T5U2L7B	1.660



* No specimen available for exposure.

Figure 32. Bend-Ductility Results from UNSCRATCHED Ti-8-1-1 After Exposure to HUMID Conditions.

Table XVII

Bend-Ductility Data from SCRATCHED Ti-8-1-1 After Exposure to HUMID Conditions WITHOUT SALT for Durations Shown(1)

1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
Bare							
				TOD1L5A	2.140	TOD1L7A	1.940
				TOD1L5B	2.200	TOD1L7B	2.040
Coated with Aluminum - Modified Silicone							
				T1D1L5A	2.000	T1D1L7A	1.615
				T1D1L5B	1.740	T1D1L7B	1.870
Coated with Catalytically Cured Silicone							
				T2D1L5A	2.260	T2D1L7A	2.385
				T2D1L5B	2.330	T2D1L7B	2.325
Coated with Zinc in Silicate Vehicle							
				T3D1L5A	1.475	T3D1L7A	1.500
				T3D1L5B	1.580	T3D1L7B	1.380
Coated with Electrophoretically Deposited Aluminum							
						T4D1L7A	2.200
						T4D1L7B	2.465
						T4D1L7C	2.460

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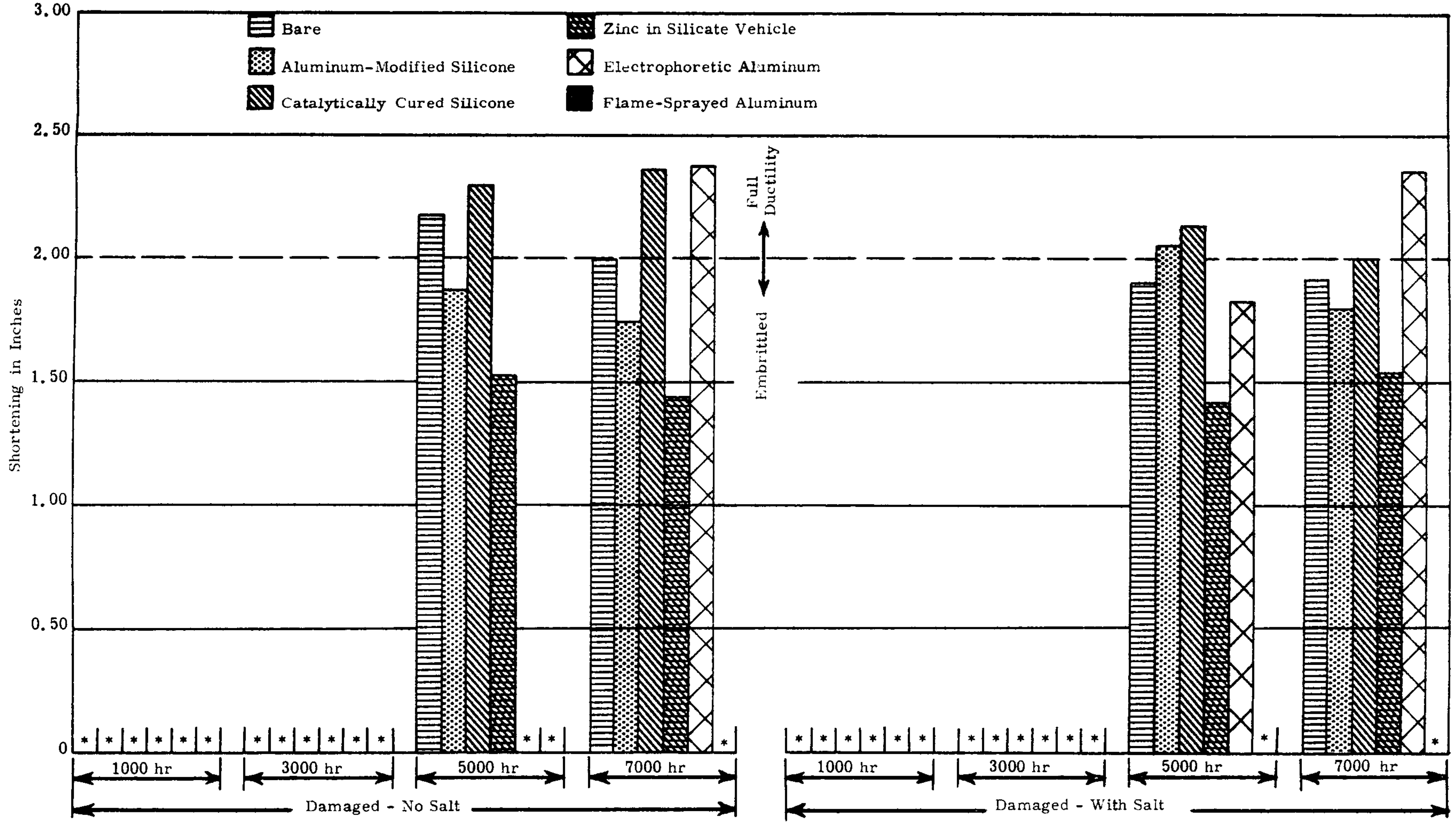
(1) No specimens Coated with Flame-Sprayed Aluminum were included in these exposures.

Table XVIII

Bend-Ductility Data from SCRATCHED Ti-8-1-1
After Exposure to HUMID Conditions WITH SALT for Durations Shown(1)

1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
Bare							
				TOD2L5A	1.820	TOD2L7A	1.885
				TOD2L5B	1.980	TOD2L7B	1.950
Coated with Aluminum - Modified Silicone							
				T1D2L5A	1.890	T1D2L7A	1.755
				T1D2L5B	2.215	T1D2L7C	1.845
Coated with Catalytically Cured Silicone							
				T2D2L5A	2.030	T2D2L7A	2.135
				T2D2L5B	2.230	T2D2L7B	1.925
Coated with Zinc in Silicate Vehicle							
				T3D2L5A	1.300	T3D2L7A	1.620
				T3D2L5B	1.540	T3D2L7B	1.460
Coated with Electrophoretically Deposited Aluminum							
				T4D2L5A	1.350	T4D2L7A	2.285
				T4D2L5B	2.130	T4D2L7B	2.425

(1) No specimens Coated with Flame-Sprayed Aluminum were included in these exposures.



* No specimens available for exposure.

Figure 33. Bend-Ductility Results from SCRATCHED Ti-8-1-1 After Exposure to HUMID Conditions.

Table XIX

Bend-Ductility Data from UNSCRATCHED AM 350 SCT
After Exposure to 550°F for Durations Shown

	1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
	Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
WITHOUT SALT	Bare							
	AOU1H1A	2.168	AOU1H3A	2.500	AOU1H5A	2.500	AOU1H7A	2.490
	AOU1H1B	2.167	AOU1H3B	2.504	AOU1H5B	2.480	AOU1H7B	2.495
	Coated with Aluminum - Modified Silicone							
	A1U1H1A	2.168	A1U1H3A	2.500	A1U1H5A	2.490	A1U1H7A	2.490
	A1U1H1B	2.163	A1U1H3B	2.500	A1U1H5B	2.483	A1U1H7B	2.485
	Coated with Catalytically Cured Silicone(1)							
	A2U1H1A	2.161	A2U1H3A	2.502	A2U1H5A	2.485	A2U1H7A	2.497
	A2U1H1B	2.168	A2U1H3B	2.500	A2U1H5B	2.490	A2U1H7B	2.495
	Coated with Zinc in Silicate Vehicle							
	A3U1H1A	2.161	A3U1H3A	2.500	A3U1H5A	2.481	A3U1H7A	2.482
	A3U1H1B	2.159	A3U1H3B	2.504	A3U1H5B	2.482	A3U1H7B	2.492
WITH SALT	Bare							
	AOU2H1A	2.166	AOU2H3A	2.500	AOU2H5A	2.490	AOU2H7A	2.500
	AOU2H1B	2.169	AOU2H3B	2.500	AOU2H5B	2.490	AOU2H7B	2.494
	Coated with Aluminum - Modified Silicone							
	A1U2H1A	2.165	A1U2H3A	2.499	A1U2H5A	2.490	A1U2H7A	2.493
	A1U2H1B	2.165	A1U2H3B	2.500	A1U2H5B	2.480	A1U2H7B	2.491
	A1U2H1C	2.166	A1U2H3C	2.500	A1U2H5C	2.488	A1U2H7C	2.496
	Coated with Catalytically Cured Silicone(1)							
	A2U2H1A	2.163	A2U2H3A	2.500	A2U2H5A	2.485	A2U2H7A	2.494
	A2U2H1B	2.160	A2U2H3B	2.488	A2U2H5B	2.488	A2U2H7B	2.489
			A2U2H3C	2.486	A2U2H5C	2.488	A2U2H7C	2.500
	Coated with Zinc in Silicate Vehicle							
	A3U2H1A	2.144	A3U2H3A	2.500	A3U2H5A	2.484	A3U2H7A	2.492
	A3U2H1B	2.145	A3U2H3B	2.500	A3U2H5B	2.483	A3U2H7B	2.480
					A3U2H5C	2.482	A3U2H7C	2.480

(1) Catalytically Cured Silicone spalled from substrate within 48 hr after exposure to 550°F

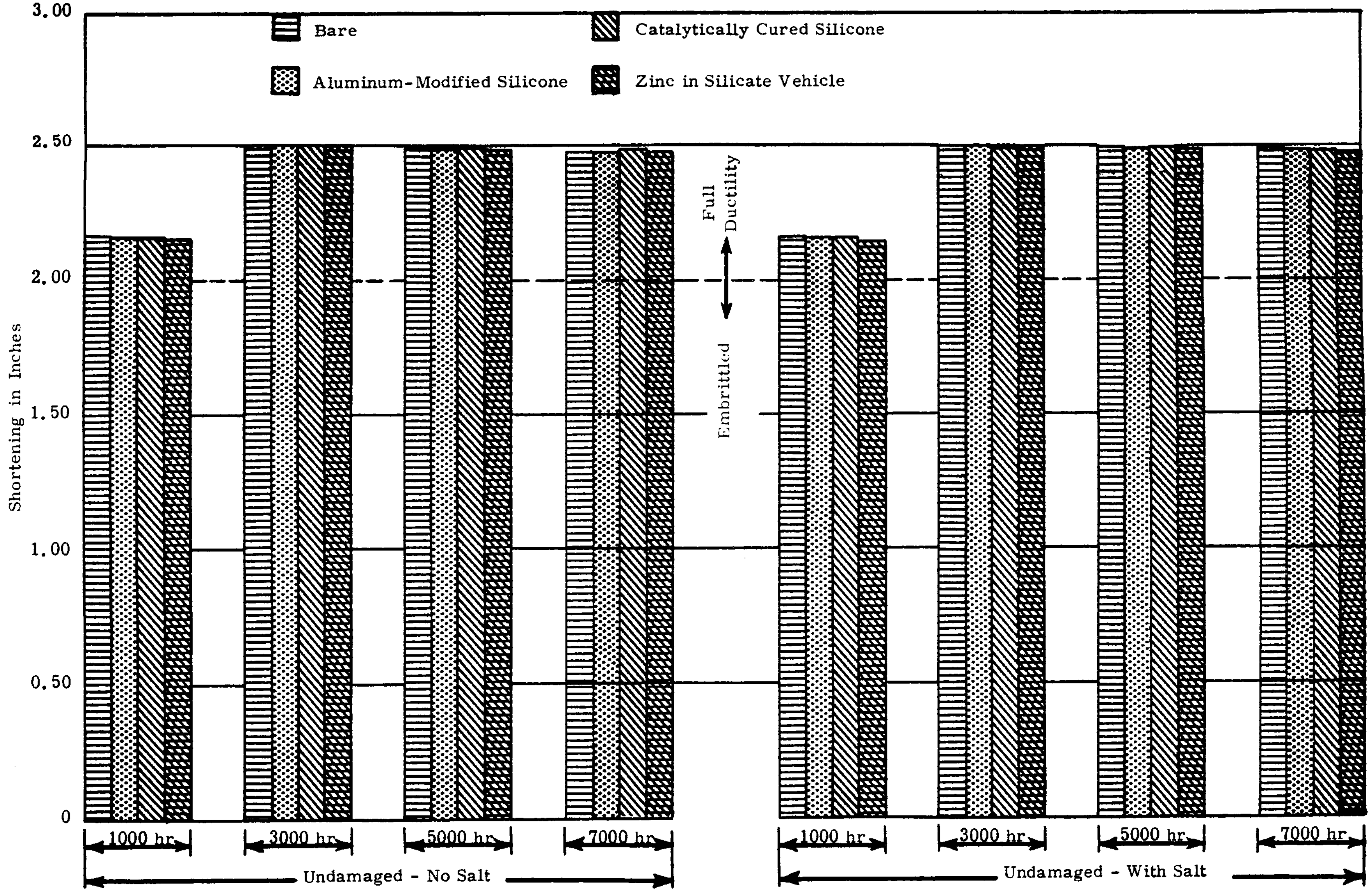


Figure 34. Bend-Ductility Results from UNSCRATCHED AM 350 SCT After Exposure to 550° F.

Table XX

Bend-Ductility Data from SCRATCHED AM 350 SCT
After Exposure to 550°F for Durations Shown

	1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
	Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
WITHOUT SALT	Bare							
	AOD1H1A	2.166	AOD1H3A	2.502	AOD1H5A	2.485	AOD1H7A	2.493
	AOD1H1B	2.061	AOD1H3B	2.503	AOD1H5B	2.490	AOD1H7B	2.495
	Coated with Aluminum - Modified Silicone							
	A1D1H1A	2.145	A1D1H3A	2.500	A1D1H5A	2.487	A1D1H7A	2.493
	A1D1H1B	2.163	A1D1H3B	2.500	A1D1H5B	2.484	A1D1H7B	2.491
	Coated with Catalytically Cured Silicone(1)							
	A2D1H1A	2.075	A2D1H3A	2.500	A2D1H5A	2.485	A2D1H7A	2.500
	A2D1H1B	1.964	A2D1H3B	2.500	A2D1H5B	2.490	A2D1H7B	2.492
	Coated with Zinc in Silicate Vehicle							
	A3D1H1A	2.162	A3D1H3A	2.490	A3D1H5A	2.470	A3D1H7A	2.492
	A3D1H1B	2.163	A3D1H3B	2.500	A3D1H5B	2.484	A3D1H7B	2.478
WITH SALT	Bare							
	AOD2H1A	2.155	AOD2H3A	2.500	AOD2H5A	2.485	AOD2H7A	2.490
	AOD2H1B	2.170	AOD2H3B	2.496	AOD2H5B	2.480	AOD2H7B	2.495
	Coated with Aluminum - Modified Silicone							
	A1D2H1A	2.167	A1D2H3A	2.500	A1D2H5A	2.490	A1D2H7A	2.492
	A1D2H1B	2.068	A1D2H3B	2.500	A1D2H5B	2.490	A1D2H7B	2.498
	Coated with Catalytically Cured Silicone(1)							
	A2D2H1A	2.165	A2D2H3A	2.500	A2D2H5A	2.490	A2D2H7A	2.500
	A2D2H1B	2.161	A2D2H3B	2.500	A2D2H5B	2.490	A2D2H7B	4.493
	Coated with Zinc in Silicate Vehicle							
	A3D2H1A	2.155	A3D2H3A	2.495	A3D2H5A	2.480	A3D2H7A	2.493
	A3D2H1B	2.123	A3D2H3B	2.488	A3D2H5B	2.480	A3D2H7B	2.488

(1) Catalytically Cured Silicone spalled from substrate within 48 hr after exposure to 550°F

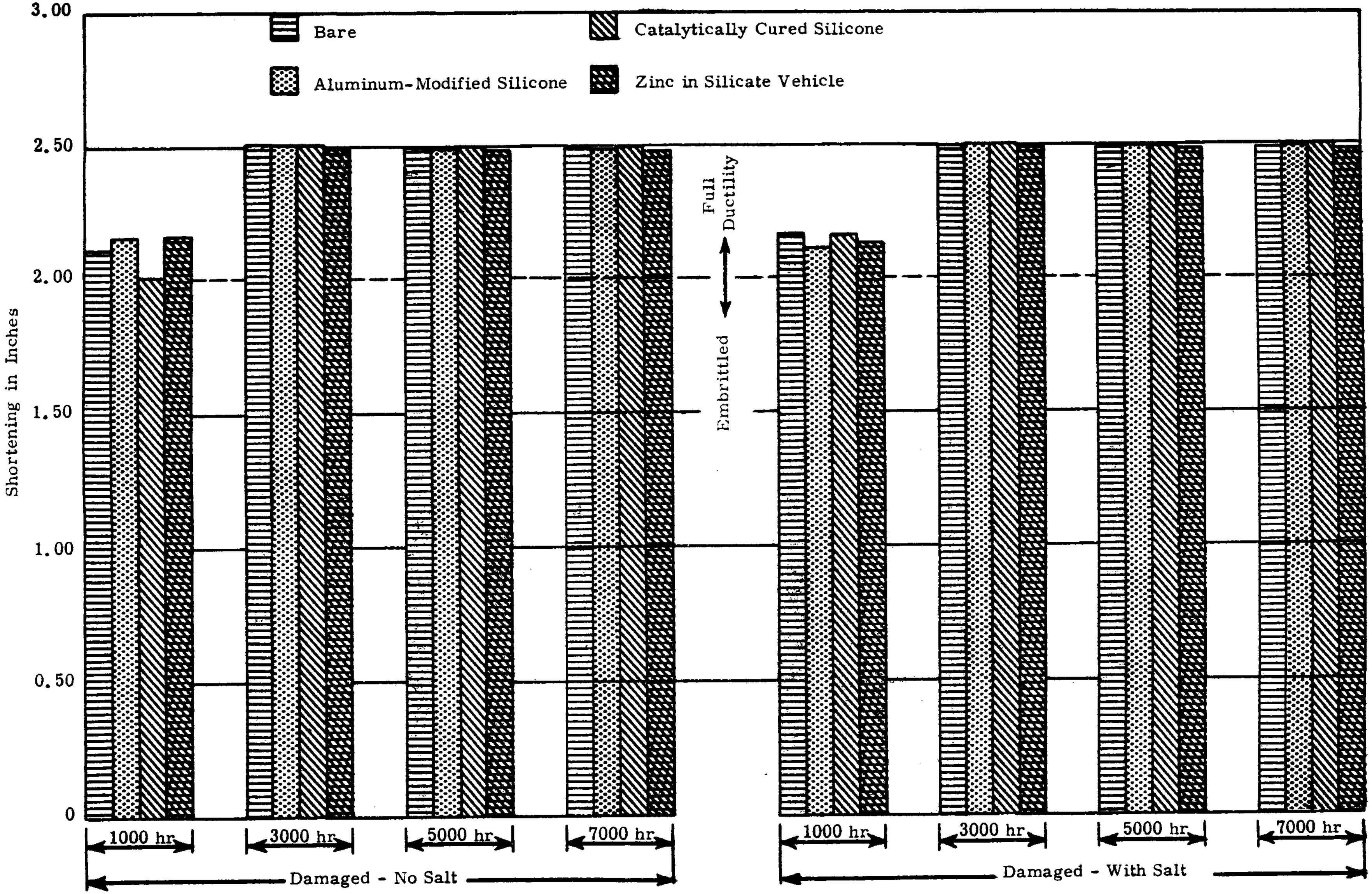


Figure 35. Bend-Ductility Results from SCRATCHED AM 350 SCT After Exposure to 550° F.

Table XXI

Bend-Ductility Data from UNSCRATCHED AM 350 SCT
After Exposure to HUMID Conditions for Durations Shown

	1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
	Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
WITHOUT SALT	Bare							
	AOU1L1A	2.194	AOU1L3A	2.500	AOU1L5A	2.490	AOU1L7A	2.490
	AOU1L1B	2.218	AOU1L3B	2.500	AOU1L5B	2.486	AOU1L7B	2.490
	Coated with Aluminum - Modified Silicone							
	A1U1L1A	2.155	A1U1L3A	2.500	A1U1L5A	2.486	A1U1L7A	2.490
	A1U1L1B	2.163	A1U1L3B	2.500	A1U1L5B	2.487	A1U1L7B	2.490
	Coated with Catalytically Cured Silicone							
	A2U1L1A	2.165	A2U1L3A	2.460	A2U1L5A	2.450	A2U1L7A	2.468
	A2U1L1B	2.164	A2U1L3B	2.470	A2U1L5B	2.455	A2U1L7B	2.453
	Coated with Zinc in Silicate Vehicle							
	A3U1L1A	1.950	A3U1L3A	2.498	A3U1L5A	2.480	A3U1L7A	2.476
	A3U1L1B	1.360	A3U1L3B	2.495	A3U1L5B	2.480	A3U1L7B	2.485
WITH SALT	Bare							
	AOU2L1A	0(1)	AOU2L3A	2.500	AOU2L5A	0(1)	AOU2L7A	0(1)
	AOU2L1B	2.171	AOU2L3B	2.500	AOU2L5B	2.487	AOU2L7B	2.490
	Coated with Aluminum - Modified Silicone							
	A1U2L1A	2.174	A1U2L3A	2.500	A1U2L5A	2.480	A1U2L7A	2.486
	A1U2L1B	2.165	A1U2L3B	2.500	A1U2L5B	2.477	A1U2L7B	2.482
	Coated with Catalytically Cured Silicone							
	A2U2L1A	2.159	A2U2L3A	2.470	A2U2L5A	2.450	A2U2L7A	2.455
	A2U2L1B	2.165	A2U2L3B	2.465	A2U2L5B	2.450	A2U2L7B	2.455
							A2U2L7C	2.470
	Coated with Zinc in Silicate Vehicle							
	A3U2L1A	2.155	A3U2L3A	1.685	A3U2L5A	1.800	A3U2L7A	2.478
	A3U2L1B	1.965	A3U2L3B	2.495	A3U2L5B	2.482	A3U2L7B	2.455
							A3U2L7C	2.475

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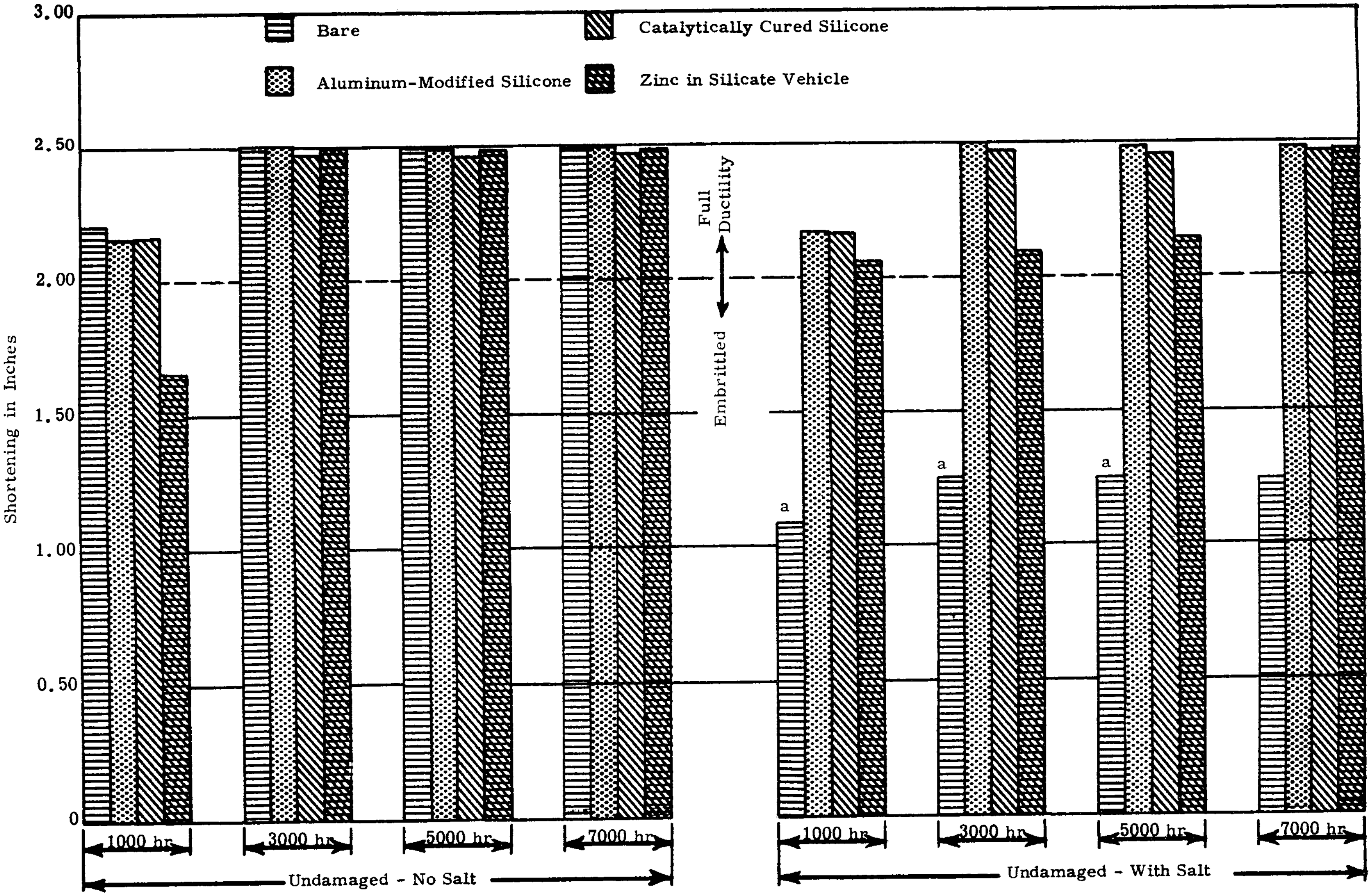
(1) Specimen fractured spontaneously within the first 800 hr of exposure.



[a] Average between one specimen that had no significant change in ductility and one specimen that fractured during exposure (zero shortening).

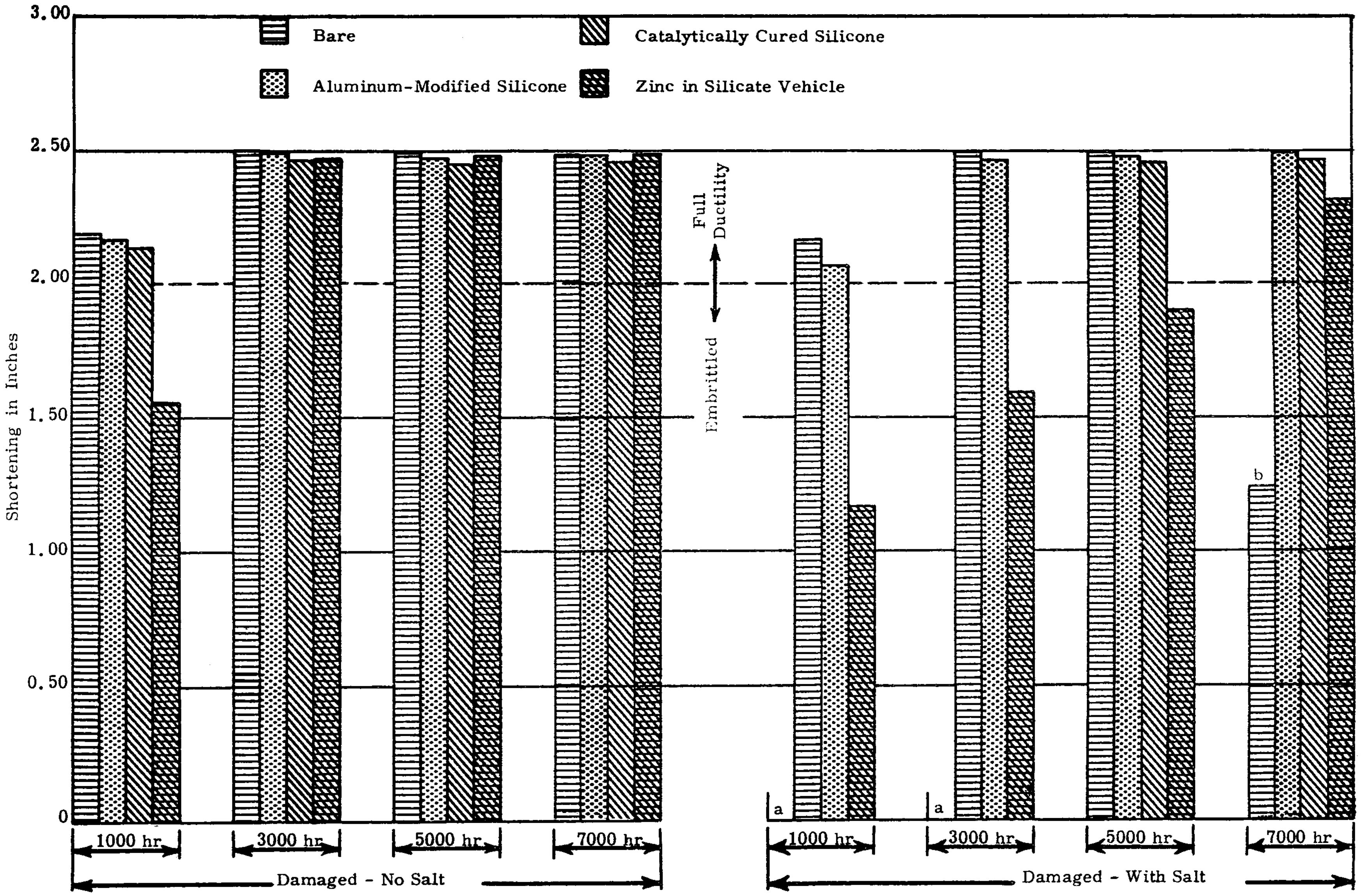
Figure 36. Bend-Ductility Results from UNSCRATCHED AM 350 SCT After Exposure to HUMID Conditions.

Table XXII

Bend-Ductility Data from SCRATCHED AM 350 SCT
After Exposure to HUMID Conditions for Durations Shown

	1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
	Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
WITHOUT SALT	Bare							
	AOD1L1A	2.177	AOD1L3A	2.500	AOD1L5A	2.486	AOD1L7A	2.490
	AOD1L1B	2.180	AOD1L3B	2.500	AOD1L5B	2.486	AOD1L7B	2.487
	Coated with Aluminum - Modified Silicone							
	A1D1L1A	2.170	A1D1L3A	2.494	A1D1L5A	2.474	A1D1L7A	2.481
	A1D1L1B	2.167	A1D1L3B	2.500	A1D1L5B	2.480	A1D1L7B	2.487
	Coated with Catalytically Cured Silicone							
	A2D1L1A	2.167	A2D1L3A	2.460	A2D1L5A	2.445	A2D1L7A	2.456
	A2D1L1B	2.110	A2D1L3B	2.462	A2D1L5B	2.450	A2D1L7B	2.450
	Coated with Zinc in Silicate Vehicle							
	A3D1L1A	1.440	A3D1L3A	2.450	A3D1L5A	2.475	A3D1L7A	2.487
	A3D1L1B	1.705	A3D1L3B	2.488	A3D1L5B	2.480	A3D1L7B	2.482
WITH SALT	Bare							
	AOD2L1A	0(1)	AOD2L3A	0(1)	AOD2L5A	2.487	AOD2L7A	0(1)
	AOD2L1B	0(1)	AOD2L3B	0(1)	AOD2L5B	2.490	AOD2L7B	2.490
	Coated with Aluminum - Modified Silicone							
	A1D2L1A	2.170	A1D2L3A	2.495	A1D2L5A	2.477	A1D2L7A	2.488
	A1D2L1B	2.165	A1D2L3B	2.495	A1D2L5B	2.480	A1D2L7B	2.490
	Coated with Catalytically Cured Silicone							
	A2D2L1A	1.975	A2D2L3A	2.457	A2D2L5A	2.450	A2D2L7A	2.452
	A2D2L1B	2.161	A2D2L3B	2.460	A2D2L5B	2.450	A2D2L7B	2.456
	Coated with Zinc in Silicate Vehicle							
	A3D2L1A	1.330	A3D2L3A	1.390	A3D2L5A	2.110	A3D2L7A	2.477
	A3D2L1B	1.000	A3D2L3B	1.795	A3D2L5B	1.690	A3D2L7B	2.140

(1) Specimen fractured spontaneously within the first 800 hr of exposure.



[a] Specimens fractured during exposure (zero shortening).
[b] Average between one specimen that had no significant change in ductility and one specimen that fractured during exposure (zero shortening).

Figure 37. Bend-Ductility Results from SCRATCHED AM 350 SCT After Exposure to HUMID Conditions.

Table XXIII

Bend-Ductility Data from UNSCRATCHED RENE 41
After Exposure to 550°F for Durations Shown

	1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
	Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
WITHOUT SALT	Bare							
	ROU1H1A	0.485	ROU1H3A	0.750	ROU1H5A	0.580	ROU1H7A	0.345
	ROU1H1B	0.319	ROU1H3B	0.900	ROU1H5B	0.555	ROU1H7B	1.620
	Coated with Aluminum - Modified Silicone							
	R1U1H1A	1.014	R1U1H3A	0.724	R1U1H5A	1.515	R1U1H7A	0.640
	R1U1H1B	0.970	R1U1H3B	0.544	R1U1H5B	0.745	R1U1H7B	1.275
	R1U1H1C	0.445	R1U1H3C	1.045	R1U1H5C	1.225	R1U1H7C	0.730
	Coated with Catalytically Cured Silicone							
	R2U1H1A	1.030	R2U1H3A	1.540	R2U1H5A	1.630	R2U1H7A	0.530
	R2U1H1B	0.824	R2U1H3B	1.310	R2U1H5B	1.530	R2U1H7B	1.035
	R2U1H1C	1.000	R2U1H3C	1.115	R2U1H5C	1.020	R2U1H7C	1.280
	Coated with Zinc in Silicate Vehicle							
	R3U1H1A	0.640	R3U1H3A	1.030	R3U1H5A	0.740	R3U1H7A	0.750
	R3U1H1B	0.753	R3U1H3B	0.935	R3U1H5B	1.120	R3U1H7B	0.550
	R3U1H1C	0.875	R3U1H3C	0.980	R3U1H5C	1.210	R3U1H7C	0.860
WITH SALT	Bare							
	ROU2H1A	1.284	ROU2H3A	1.585	ROU2H5A	0.500	ROU2H7A	0.460
	ROU2H1B	0.400	ROU2H3B	0.980	ROU2H5B	1.410	ROU2H7B	0.770
	Coated with Aluminum - Modified Silicone							
	R1U2H1A	1.750	R1U2H3A	0.775	R1U2H5A	1.180	R1U2H7A	0.605
	R1U2H1B	0.738	R1U2H3B	1.240	R1U2H5B	1.060	R1U2H7B	1.570
	R1U2H1C	0.715	R1U2H3C	1.090	R1U2H5C	1.140	R1U2H7C	1.040
	Coated with Catalytically Cured Silicone							
	R2U2H1A	1.784	R2U2H3A	1.835	R2U2H5A	0.750	R2U2H7A	0.985
	R2U2H1B	1.148	R2U2H3B	1.832	R2U2H5B	2.230	R2U2H7B	0.710
	R2U2H1C	1.400	R2U2H3C	1.055	R2U2H5C	1.940	R2U2H7C	0.990
	Coated with Zinc in Silicate Vehicle							
	R3U2H1A	0.558	R3U2H3A	0.745	R3U2H5A	0.640	R3U2H7A	1.040
	R3U2H1B	0.744	R3U2H3B	1.085	R3U2H5B	0.780	R3U2H7B	1.020
	R3U2H1C	0.670	R3U2H3C	0.777	R3U2H5C	0.600	R3U2H7C	0.775

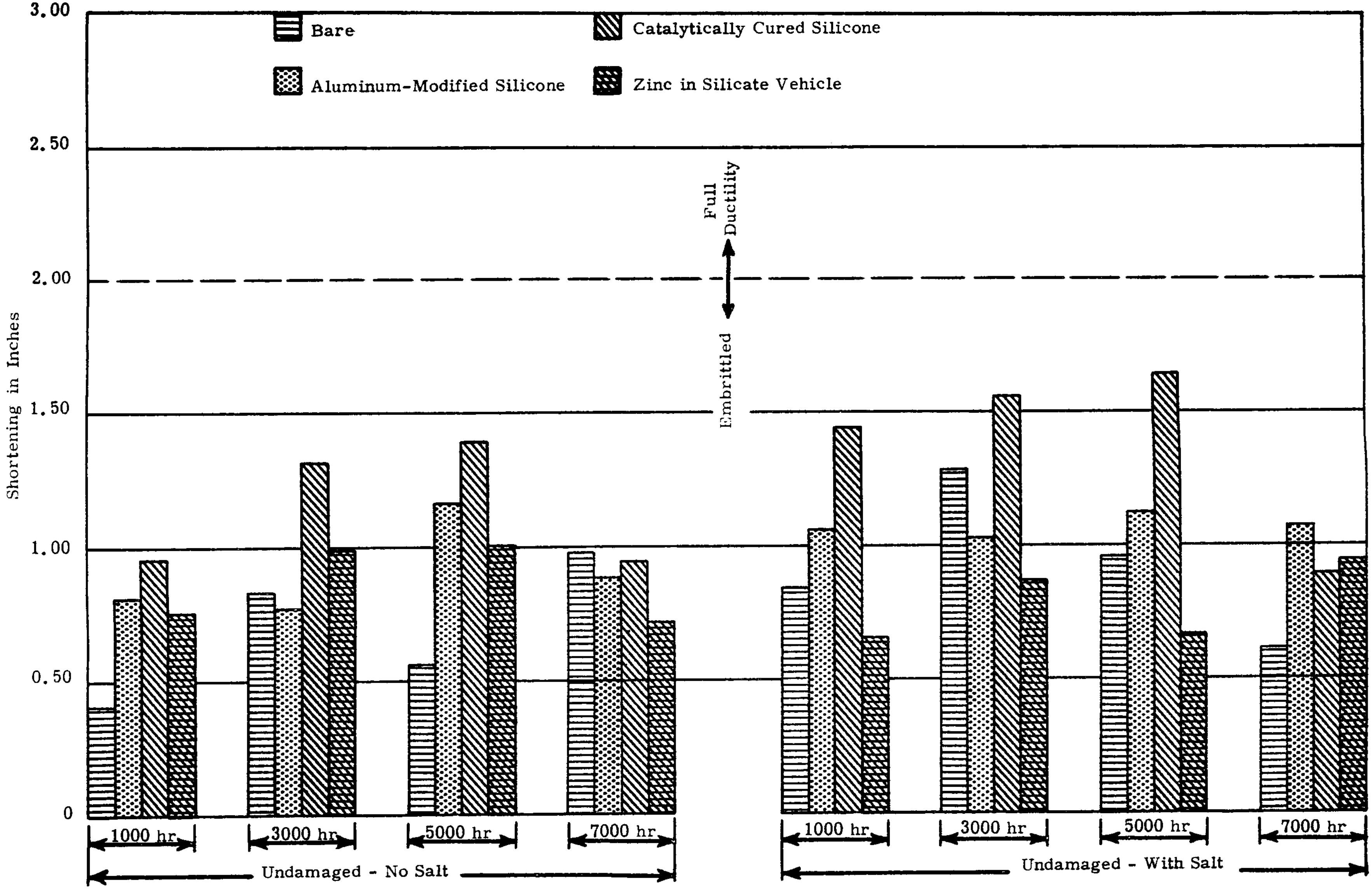


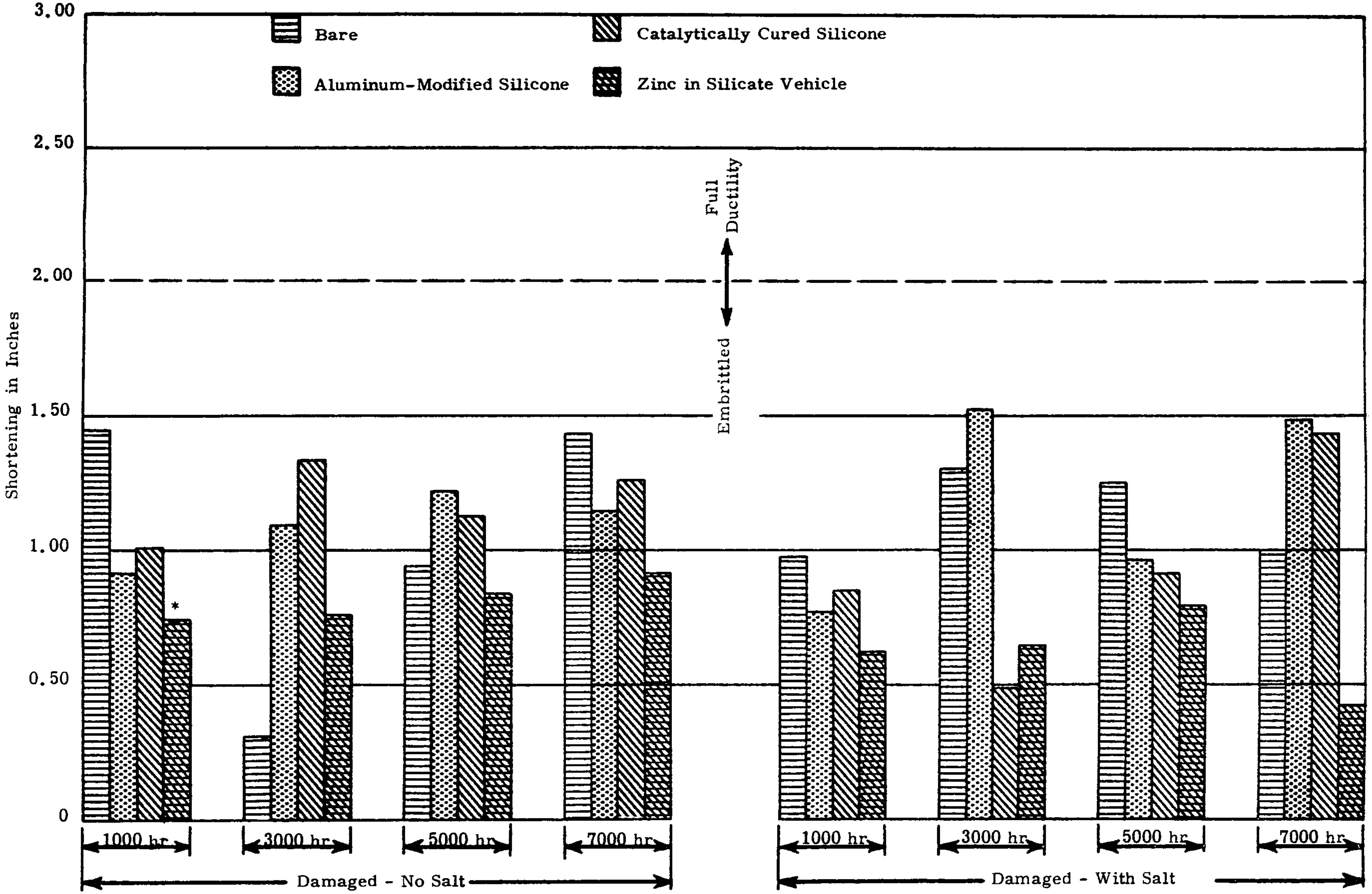
Figure 38. Bend-Ductility Results from UNSCRATCHED RENE 41 After Exposure to 550° F.

Table XXIV

Bend-Ductility Data from SCRATCHED RENE 41 After Exposure to 550°F for Durations Shown

	1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
	Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
WITHOUT SALT	Bare							
	ROD1H1A	1.264	ROD1H3A	0.360	ROD1H5A	0.740	ROD1H7A	0.960
	ROD1H1B	1.620	ROD1H3B	0.270	ROD1H5B	1.145	ROD1H7B	1.910
	Coated with Aluminum - Modified Silicone							
	R1D1H1A	0.980	R1D1H3A	1.180	R1D1H5A	0.840	R1D1H7A	1.415
	R1D1H1B	0.858	R1D1H3B	1.010	R1D1H5B	1.100	R1D1H7B	0.880
					R1D1H5C	1.730	R1D1H7C	1.145
	Coated with Catalytically Cured Silicone							
	R2D1H1A	1.232	R2D1H3A	1.495	R2D1H5A	1.120	R2D1H7A	1.160
	R2D1H1B	0.800	R2D1H3B	1.175	R2D1H5B	1.130	R2D1H7B	1.290
							R2D1H7C	1.340
	Coated with Zinc in Silicate Vehicle							
			R3D1H3A	0.655	R3D1H5A	1.135	R3D1H7A	1.050
	R3D1H1B	0.748	R3D1H3B	0.865	R3D1H5B	0.750	R3D1H7B	0.630
					R3D1H5C	0.640	R3D1H7C	1.080
WITH SALT	Bare							
	ROD2H1A	0.795	ROD2H3A	0.575	ROD2H5A	2.080	ROD2H7A	0.370
	ROD2H1B	1.155	ROD2H3B	2.040	ROD2H5B	0.420	ROD2H7B	1.650
	Coated with Aluminum - Modified Silicone							
	R1D2H1A	0.645	R1D2H3A	2.225	R1D2H5A	1.010	R1D2H7A	1.190
	R1D2H1B	0.904	R1D2H3B	0.325	R1D2H5B	0.900	R1D2H7B	1.745
	R1D2H1C	0.755	R1D2H3C	2.025	R1D2H5C	0.990	R1D2H7C	1.530
	Coated with Catalytically Cured Silicone							
	R2D2H1A	1.035	R2D2H3A	(1)	R2D2H5A	1.145	R2D2H7A	0.990
	R2D2H1B	0.745	R2D2H3B	0.250	R2D2H5B	0.785	R2D2H7B	1.580
	R2D2H1C	0.769	R2D2H3C	0.725	R2D2H5C	0.810	R2D2H7C	1.735
	Coated with Zinc in Silicate Vehicle							
	R3D2H1A	0.540	R3D2H3A	0.800	R3D2H5A	0.960	R3D2H7A	0.360
	R3D2H1B	0.687	R3D2H3B	0.355	R3D2H5B	0.520	R3D2H7B	0.500
	R3D2H1C	0.660	R3D2H3C	0.790	R3D2H5C	0.900	R3D2H7C	0.420

(1) Weld parted during exposure.



* One specimen only.

Figure 39. Bend-Ductility Results from SCRATCHED RENE 41 After Exposure to 550° F.

Table XXV

Bend-Ductility Data from UNSCRATCHED RENE 41
After Exposure to HUMID Conditions for Durations Shown

	1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
	Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
WITHOUT SALT	Bare							
	ROU1L1A	1.465	ROU1L3A	1.820	ROU1L5A	0.875	ROU1L7A	0.455
	ROU1L1B	0.910	ROU1L3B	1.500	ROU1L5B	0.460	ROU1L7B	1.625
	Coated with Aluminum - Modified Silicone							
	R1U1L1A	0.966	R1U1L3A	1.180	R1U1L5A	1.630	R1U1L7A	0.760
	R1U1L1B	0.634	R1U1L3B	1.030	R1U1L5B	0.840	R1U1L7B	0.730
	R1U1L1C	0.700	R1U1L3C	0.870	R1U1L5C	1.300	R1U1L7C	0.950
	Coated with Catalytically Cured Silicone							
	R2U1L1A	1.400	R2U1L3A	0.850	R2U1L5A	1.710	R2U1L7A	1.970
	R2U1L1B	1.380	R2U1L3B	2.443	R2U1L5B	2.030	R2U1L7B	1.890
	R2U1L1C	0.945	R2U1L3C	0.620	R2U1L5C	1.700	R2U1L7C	0.870
	Coated with Zinc in Silicate Vehicle							
	R3U1L1A	0.490	R3U1L3A	0.630	R3U1L5A	0.865	R3U1L7A	0.650
	R3U1L1B	0.805	R3U1L3B	0.965	R3U1L5B	0.580	R3U1L7B	0.880
	R3U1L1C	0.735	R3U1L3C	1.030	R3U1L5C	0.510	R3U1L7C	0.560
WITH SALT	Bare							
	ROU2L1A	1.085	ROU2L3A	2.195	ROU2L5A	1.645	ROU2L7A	0.575
	ROU2L1B	0.956	ROU2L3B	1.410	ROU2L5B	0.340	ROU2L7B	1.005
	Coated with Aluminum - Modified Silicone							
	R1U2L1A	0.730	R1U2L3A	0.810	R1U2L5A	0.785	R1U2L7A	1.900
	R1U2L1B	0.985	R1U2L3B	1.080	R1U2L5B	0.880	R1U2L7B	0.975
	R1U2L1C	0.705	R1U2L3C	0.850	R1U2L5C	1.275	R1U2L7C	0.820
	Coated with Catalytically Cured Silicone							
	R2U2L1A	1.682	R2U2L3A	1.110	R2U2L5A	0.755	R2U2L7A	1.920
	R2U2L1B	1.010	R2U2L3B	0.320	R2U2L5B	1.630	R2U2L7B	1.930
	R2U2L1C	0.748	R2U2L3C	0.980	R2U2L5C	1.400	R2U2L7C	2.020
	Coated with Zinc in Silicate Vehicle							
	R3U2L1A	0.651	R3U2L3A	0.405	R3U2L5A	0.350	R3U2L7A	0.430
	R3U2L1B	0.645	R3U2L3B	0.375	R3U2L5B	0.650	R3U2L7B	0.810
	R3U2L1C	0.440	R3U2L3C	0.540	R3U2L5C	0.470	R3U2L7C	0.530

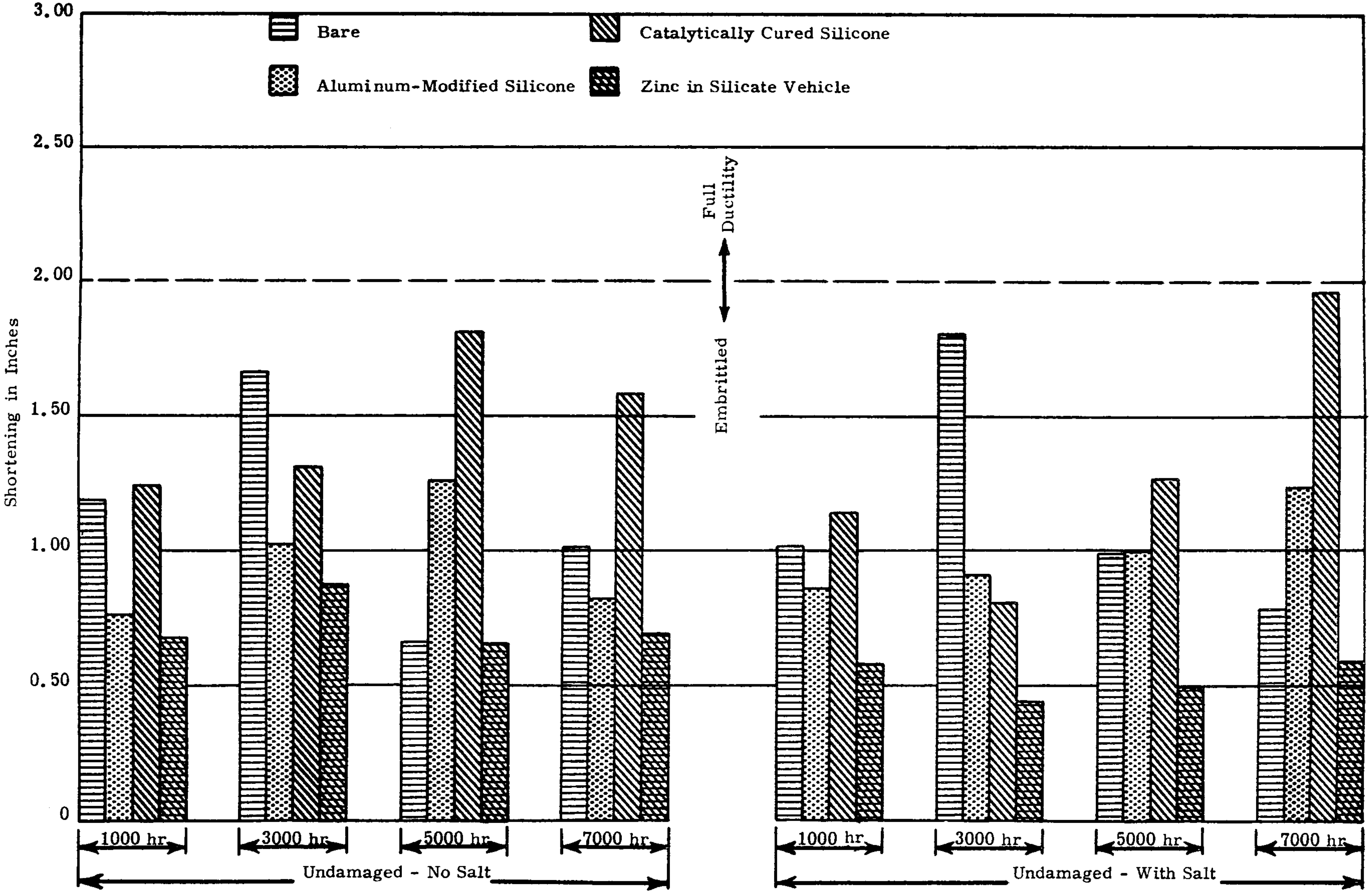


Figure 40. Bend-Ductility Results from UNSCRATCHED RENE 41 After Exposure to HUMID Conditions.

Table XXVI

Bend-Ductility Data from SCRATCHED RENE 41 After Exposure to HUMID Conditions for Durations Shown

	1000 Hr Shortening, Inches		3000 Hr Shortening, Inches		5000 Hr Shortening, Inches		7000 Hr Shortening, Inches	
	Specimen	In.	Specimen	In.	Specimen	In.	Specimen	In.
WITHOUT SALT	Bare							
	ROD1L1A	1.465	ROD1L3A	0.800	ROD1L5A	1.510	ROD1L7A	1.360
	ROD1L1B	0.910	ROD1L3B	0.455	ROD1L5B	1.540	ROD1L7B	0.900
	Coated with Aluminum - Modified Silicone							
	R1D1L1A	1.350	R1D1L3A	1.060	R1D1L5A	0.690	R1D1L7A	0.540
	R1D1L1B	0.813	R1D1L3B	2.210	R1D1L5B	0.970	R1D1L7B	1.320
	Coated with Catalytically Cured Silicone							
	R2D1L1A	1.040	R2D1L3A	1.720	R2D1L5A	1.190	R2D1L7A	2.030
	R2D1L1B	1.250	R2D1L3B	1.720	R2D1L5B	0.750	R2D1L7B	1.690
	Coated with Zinc in Silicate Vehicle							
	R3D1L1A	0.355	R3D1L3A	0.670	R3D1L5A	0.480	R3D1L7A	0.690
	R3D1L1B	0.453	R3D1L3B	0.690	R3D1L5B	0.590	R3D1L7B	0.735
WITH SALT	Bare							
	ROD2L1A	1.390	ROD2L3A	1.280	ROD2L5A	1.850	ROD2L7A	1.300
	ROD2L1B	0.434	ROD2L3B	0.900	ROD2L5B	0.960	ROD2L7B	1.060
	Coated with Aluminum - Modified Silicone							
	R1D2L1A	1.100	R1D2L3A	0.735	R1D2L5A	1.355	R1D2L7A	1.020
	R1D2L1B	0.720	R1D2L3B	1.020	R1D2L5B	1.600	R1D2L7B	0.780
	R1D2L1C	0.795	R1D2L3C	1.440	R1D2L5C	1.310	R1D2L7C	1.250
	Coated with Catalytically Cured Silicone							
	R2D2L1A	1.710	R2D2L3A	1.510	R2D2L5A	1.030	R2D2L7A	0.990
	R2D2L1B	1.249	R2D2L3B	0.750	R2D2L5B	0.790	R2D2L7B	1.520
	R2D2L1C	1.000	R2D2L3C	0.925	R2D2L5C	1.465	R2D2L7C	0.680
	Coated with Zinc in Silicate Vehicle							
	R3d2L1A	0.550	R3D2L3A	0.550	R3D2L5A	0.410	R3D2L7A	0.420
	R3D2L1B	0.469	R3D2L3B	0.480	R3D2L5B	0.555	R3D2L7B	0.400
	R3D2L1C	0.423	R3D2L3C	0.455	R3D2L5C	0.485	R3D2L7C	0.510

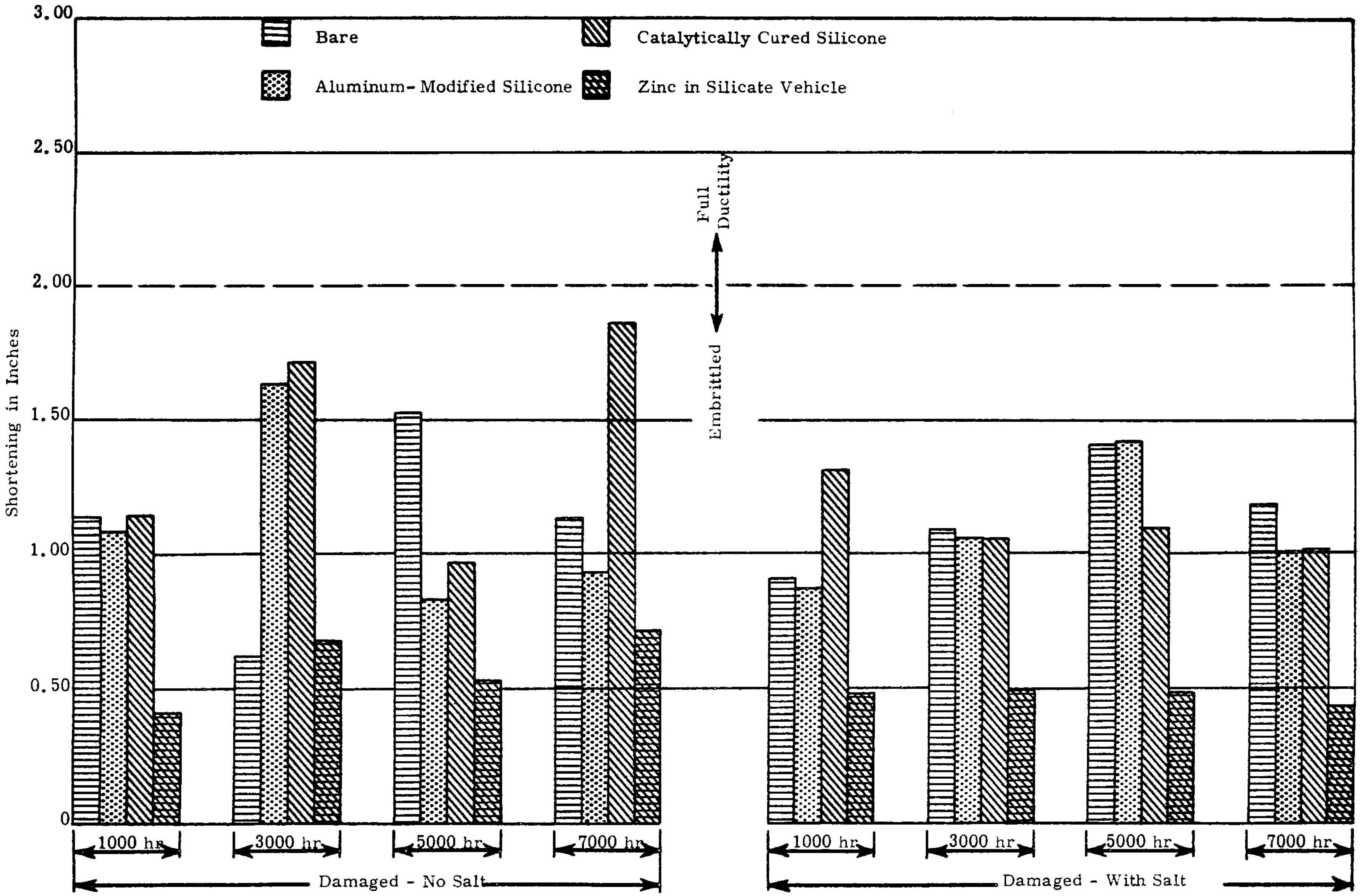


Figure 41. Bend-Ductility Results from SCRATCHED RENE 41 After Exposure to HUMID Conditions.