EFFECT OF RELATIVE HUMIDITY ON FATIGUE OF ANODIZED 2024-T351 ALUMINUM IN COMPLETELY REVERSED TORSION

by

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Thesis submitted to the Graduate Faculty of
Virginia Polytechnic Institute and State University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE
in
Mechanical Engineering

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December, 1973
Blacksburg, Virginia

ACKNOWLEDGMENTS

The investigator wants to extend a word of gratitude to his major advisor, Dr. H. H. Mabie, for the suggestion of this topic and assistance provided by him throughout this investigation. Also, thanks is extended to both the co-advisors, Dr. N. S. Eiss, Jr. and Dr. L. D. Mitchell for their suggestions and assistance when called upon.

Special recognition is extended to Mr. C. A. Witherspoon, Supervisor of the Chemical Department at Poly-Scientific (Division of Litton Industries, Blacksburg, Virginia), who gave his advice and showed his patience by anodizing the specimens used in this investigation.

Lastly, the investigator is indebted to the E. I. duPont deNemours and Company, Inc. for the financial support in school which made this work possible.

TABLE OF CONTENTS

APPENDICES

TABLE OF CONTENTS - continued

LIST OF FIGURES

LIST OF FIGURES - continued

LIST OF TABLES

Tables Page

LIST OF TABLES - continued

INTRODUCTION

Fatigue and its mechanical effects on machine components is a very important phenomena that continues to puzzle man today. Many times in various mechanical designs a member fractures abruptly without any previous indication of failure.

One early investigator in the study of fatigue found that different chemical reagents tended to accelerate fatigue. Thus the combined action of corrosion simultaneous with cyclic loading led our predecessors to believe that fatigue of metal is accomplished not only by the mechanical action due to loading, but also the chemical reaction associated with corrosion. This phenomena came to be known as "corrosion fatigue." For almost half a century now, various investigators have embarked on different studies toward answering questions pertaining to reagents effects on metals.

One such test conducted to secure the "true fatigue limit"* was to remove all chemical reagents from air that may affect the metal during the fatigue test. Certain reagents like water vapor could be reasonably removed by creating a vacuum around the specimen during the test. The findings for experiments of this type showed definite increases in fatigue lives** for certain metals when the air was removed.

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[*] Fatigue limit is the stress at which a specimen will endure an infinite number of cycles without failure.

[**] Fatigue lives refer to the number of cycles endured by fatigue specimens under one condition compared to specimens fatigued under other conditions at the same stress level.

One type of metal that experienced such a shift in fatigue life was aluminum and its alloys. Aluminum alloys, which are characterized by their light weight and high strength, are extensively used in the space and aircraft industries.

The present investigation builds on the work of James Wilson (1) who did a study on the effect of relative humidity of fatigue of 2024-T351 aluminum in completely reverse torsion. The present investigator used the same alloy as that used by (1) in his investigation, but having anodic coatings grown on the specimens surfaces to various thicknesses. The specimens in this investigation had the same dimensions (except the diameter was 2 mils smaller) as those used by Wilson. Thus, a direct comparison of the present work with that of Wilson's can be made on the effects of different relative humidity environments on the fatigue lives of the specimens. This comparison has more meaning since the specimens in the two investigations, Wilson and the present one, differ only with the addition of an anodic coating (Wilson tested specimens of the same type with a natural aluminum oxide coating). In several instances the investigators have failed to report the humidity levels in which the metals were fatigued that are known to be affected by the moisture in the air. One common fault of other investigators also is to take too little test data at a given condition.

The objective of this investigation is to study the corrosion fatigue of anodized 2024-T351 aluminum alloy in reversed torsion. The protective coating was grown on the metal substrate (using a conventional sulfuric anodizing process) to two different thicknesses, normally 0.05 and 0.3 mils respectively. This type of coating is brittle but attaches itself tenaciously to the base metal and is well known for its corrosion and wear resistance. The applied stress as a variable in a fatigue study is already known to affect the fatigue life of this metal and therefore will not be studied. The variables of relative humidity, coating thickness, surface finish (polishing), and coating (anodizing) are the variables of interest in this investigation. The latter variables effects are to be studied in relation to their effects on the fatigue lives of the aluminum specimens.

Another mechanism of failure known as stress corrosion also occur in aluminum alloys. This mechanism occurs in corrosion resistant alloys where the material is exposed to a specific corrosive environment when placed under a mean tensile stress. As a rule of thumb this mechanism occurs in a corrosive environment where the material is to be stressed above 50% of its yield strength (2). Since the loading in this investigation is purely cyclic and the applied stresses are below the yield stress in torsion, the mechanism of failure investigated is that of corrosion fatigue.

REVIEW OF LITERATURE

J. Wilson (1) in 1973 experimented with 2024-T351 aluminum alloy stressed in different relative humidity environments for completely reverse torsion. The specimens were run at three relative humidity ranges (20-25%, 50-55%, and 90-95%) for various loading conditions in the "as machined" and "as machined and polished" conditions. Results showed that for the lowest stress level, an increase in humidity from 20-25% to 90-95% gave a decrease in fatigue life of 68.1%. He also found by going from 20-25% to 50-55% that a decrease of about 59% resulted in the fatigue lives of his specimens. Specimens that were machined and polished and run only at 50-55% relative humidity showed no significant differences statistically from the other specimens run at the same humidity level in the "as machined" condition.

J. Odle (3) in 1972 investigated the fatigue of SAE 1018 steel for two relative humidity environments in completely reversed torsion. He used both smooth and notched specimens in his investigation. He found that humidity affected the crack initiation stage in steel and that air of 20-25% relative humidity reduced the fatigue life as compared with tests run in dry air of relative humidity less than 5%. Odle also concluded from other tests that air of 20-25% was just as effective in reducing the fatigue life of the steel as that of 90-95% relative humidity.

As early as 1917 B. P. Haigh (4) noticed that the fatigue limits of metals were often reduced when chemical reagents or even ordinary water were placed on the surfaces of the test pieces during a test. His tests were carried out on a Haigh machine operating in alternating push-pull of known magnitude of stress on different metals for various reagents (fresh water, salt water, diluted ammonia, and acid). The test piece surface was moistened by a pad of cotton wool placed on its surface that was continuously wetted from a liquid supply. Fatigue lives were much shorter on the moistened surfaces with the fatigue limits definitely lower. Some bronzes proved immune when wetted by the various reagents while other metals did not show any immunity. Haigh, believing that air was the prime cause of the reduced fatigue life, followed through on a suggestion made by one of his colleagues to exclude the atmosphere by applying a layer of oil or grease on the test piece. He therefore applied grease, vaseline, and glycerine to the test pieces when the tests were conducted in air to find ratios as much as 2 to 1 for fatigue limit increases. Oil was most effective as a protective layer.

D. J. McAdams (5) in 1927 studied the corrosion fatigue of monel metal with a rotating cantilever beam type machine for different conditions of cold working and heat treatment. Specimens made of a 5 per cent nickel alloy steel showed their fatigue lives when tested in air to be longer than other specimens of the same material fatigued in a water environment (a small stream of water was allowed to contact the specimen's surface during each test). For the other metals tested, chrome-nickel steel and carbon steel, the results of the water stream tests were below those of the tests in air (fatigue lives decrease for water tests). McAdams concluded that slight corrosion simultaneous with fatigue caused a low fatigue resistance of the metals tested, and that the damaging effect is greater the harder the steel.

B. P. Haigh and B. Jones (6) in 1930 studied the effect of fatigue under abnormal conditions in lead for alternating stresses of the direct push and pull type. They found the fatigue life of lead to be greatly increased in an oil bath (Neutraline) or water surroundings. "A thin grease layer (Adcol) would delay fatigue appreciably." Acetic acid baths appeared to increase the fatigue life, but this was not the case when thin films of acetic acid were placed on the test pieces surfaces (i.e., no increases were noticed).

H. J. Gough and D. G. Sopwith (7) in 1932 tested specimens in both bending and tension using the Wohler and Haigh machines, respectively. Tests were run on the four materials as follows: mild steel, annealed copper, annealed brass, and cupro-nickel. They considered these metals to possess uniform mechanical properties. A lanoline grease coating was applied to some samples after machining and used during the fatigue tests but gave no changes in the fatigue limit from tests run in air. Tests carried out in a vacuum (between 0.0005 and 0.001 mm Hg) resulted in fatigue limit increases of 5%, 13%, and 26% for mild steel, annealed copper, and annealed brass, respectively.

Gough and Sopwith (8) in another paper in 1935 tried to explain the increase in fatigue limit in partial vacuum. The reasons why fatigue limits were increased during tests in a vacuum were thought to be the lack of the following:

(a) Oxygen. This is the primary constituent of air in the presence of water vapor.

(b) Atmospheric impurities of acids and alkaline substances.

(c) Impurities. These are mainly dissolved gases reacting within the metal during the fatigue test.

They attempted to study points (a) and (b) with the use of an annealed copper and a 70:30 brass with tests on a Haigh machine in air, partial vacuum, and purified air both wet and dry. Results of these tests discredited point (b) as affecting fatigue but confirmed point (a) to be true. Point (c) was examined by deoxidizing copper with phosphorus and fatiguing in air and partial vacuum. Results on point (c) showed that the tests carried out on oxidized copper and deoxidized copper in air and partial vacuum to have no significant changes in the fatigue limits. Thus, it was concluded that the dissolved gases in this metal has no effect on fatigue limit. The specimens for these tests were fatigued on the Haigh machine and gave 11% and 23% increases in fatigue limits for the vacuum over the air tests for the copper and brass respectively. The wet purified air corresponded to 55% relative humidity and the partial vacuum equaled 0.001 mm Hg.

N. J. Wadsworth (9) in 1958 conducted tests on annealed copper, aluminum polycrystals, and gold by using a different constant strain for each of the previous metals at various chamber pressures. He found reductions in the fatigue lives of the first two metals above by factors of 20 and 10 when the air pressure was increased from 10^{-5} to 760 mm Hg. Gold did not show any change in fatigue life over the same pressure range. Wadsworth created different gas environments (argon, nitrogen, carbon dioxide, wet oxygen, and water) around the specimens in order to distinguish which constituent or constituents of air affected the fatigue lives of the specimens (done for specimens at different pressures for various environments). The copper was annealed and fatigued in vacuum so as not to expose the metal to air. Oxygen had the main effect on copper in fatigue while water vapor had only a slight effect. The aluminum crystals fatigue lives were decreased under those of air tests when more water was added to the environment at 10 mm Hg. Gold, being much less active than each of the above two metals, showed negligible differences between tests in air and in a vacuum. It was concluded that the rate of crack propagation changed in the two environments, the propagation being lower in the vacuum.

H. E. Frankel, J. A. Bennett, and W. L. Holshouser (10) in 1960 performed tests on SAE 4340 steel, 6061-T6 aluminum, 75-A titanium, 17-7 PH stainless steel, copper with 1.75% beryllium, and a magnesium alloy (AZ63-H24). Rotating-beam specimens were tested on the R. R. Moore machine at 2000 rpm. All specimens were polished with 400 Aloxite paper in the longitudinal direction and agitated in three containers of high-purity benzene before testing. All specimens of a given material were tested under one of the following conditions: (a) cleaned and dried, (b) coated or immersed in an oleophobic substance, or (c) immersed in water in either the clean condition or a coated condition of an oleophobic film. The oleophobic substances used in the study were n-Hexadecane, xylene, benzene, octyl alcohol, dodecyl alcohol, dodecylamine, and octadecylamine. The tests for one stress amplitude in water and nonpolar liquids, "either had no significant effect or caused a decrease in fatigue life." Oleophobic films caused an increase in fatigue life of the steel, the magnesium alloy, and the copper-beryllium alloy (as high as 144, 603, and 365%, respectively). Dodecyl alcohol provided optimum conditions for an increase in fatigue life. Xylene and benzene applied on the same metals reduced the fatigue life in the metals. Specimens that were coated by oleophobic films had less scatter than uncoated specimens. The results were the same whether the specimens were immersed or coated with the liquid. The aluminum and the titanium alloys showed no change when the above mentioned liquids were placed on their surfaces, while the stainless steel results were questionable. The investigators suggested that the observed results for these two alloys (aluminum and titanium) are due to the nature of their oxide films which are known to be more adherent and impervious to the surrounding environment than the oxides of iron, copper, and magnesium. The results found for the aluminum in this study that a water environment has no effect on the fatigue life of aluminum is in contrast to other investigators [(1) and (9)]. The investigators concluded the effect of oleophobic coatings were only during the crack initiation stage of the specimen, since it was found that 85 to 90% of the specimens lives were expended in initiation of cracks.

T. Broom and A. Nicholson (11) in 1961 conducted tests on three age hardenable aluminum alloys. A 4% copper aluminum alloy, a Duralumin alloy (B.S. L65) and a D.T.D. 683 alloy were fatigued in air, in vacuum, and in various gaseous environments. All tests were in random sequence under axial loading at a frequency between 155-165 cps. At least six specimens were run at each condition of testing. Fatigue lives of the 4% copper aluminum specimens tested in air at 10^5 cycles to those in well-dried environments were reduced by a factor as much as 8. A butyl rubber coating applied to the specimens surface showed as much as a factor of 10 in increase in the fatigue lives of the specimens over uncoated specimens in air environments at the same stress. A chromic anodic oxidation process produced an oxide coating thickness between 0.05 to 0.1 mils on fatigue specimens made from B.S. L65 and D.T.D 683 aluminum alloys. Comparison of the fatigue tests for anodized and uncoated specimens at the same stresses and environment showed no significant differences between fatigue lives for the two coating conditions. Most of the tests with coatings were done at one stress amplitude except in the case of butyl rubber. The presence of water vapor was again the reason for the reduction in fatigue life in the age hardened aluminum. A zinc chromate paint coating also was ineffective in extending the fatigue life.

A. Hartman (12) investigated the effect of oxygen and water vapor on the propagation of fatigue cracks in 2024-T3 Alcad sheet in 1965. Tests were conducted on a 2-ton Vibrophore machine in tension with a constant mean stress. The crack rate was studied by placing a 1 mm hole in a piece of sheeted material (210 x 70 mm), with a small slot of 0.2 mm wide and 1 mm long, located normal to the direction of loading (slot was placed diametrically across the hole). A mean stress was used in all tests with variable stress amplitudes applied from test to test. At least 2 or 3 specimens were tested at a stress level. A relative humidity of 60% was used in the tests carried out in air. Water vapor was found to have more effect on crack propagation of the specimens than oxygen, when fatigue tests in wet pure argon were found to be shorter than tests run in pure oxygen. Tests conducted in a wet argon environment were considered to show the effect of water vapor only because of the gas inertness. Hartman found that the fatigue life in air continuously increased with a decrease in water content for this metal. He noted an increase of fatigue life of about 6 times in going from saturated air to air of 20 ppm of water vapor. Hartman surmised from his study with 2024-T3 Alcad sheet that the natural aluminum oxide film's affinity for water was a more likely reason for the reduction in fatigue life. Because the oxide have more absorbed water at the crack tip, more water would be available to enter cracks which developed. After the water contacts the metal it is believed that it reacts with the freshly torn metal and forms a thin film of aluminum oxide which does not allow the metal to adhere upon the crack closing (when the load is reversed). This phenomena would then account for an increase in crack rate and reduction in fatigue lives for the specimens.

T. R. Shives and J. A. Bennett (13) in 1965 performed rotating beam fatigue tests on unnotched specimens of AISI 4340 steel, free cutting brass, a titanium alloy (Ti-4AL-4Mn), and a magnesium alloy (AZ61A) which all showed lowered fatigue lives in a wet environment than in a dry one. The wet and dry environments corresponded to relative humidities of greater than 85% and less than 3%, respectively. A coating of dodecyl alcohol was found to increase the fatigue lives in the steel and magnesium alloys. The fatigue specimens were machine finished and polished longitudinally with No. 400 abrasive paper. The increases in fatigue lives for coated specimens of steel and of magnesium alloys showed up in both moist and dry environments. For the magnesium alloy at high humidity, fatigue behavior was more affected at high stress than for the lower stresses used.

E. G. Eeles (14) in 1967 studied the effect of oxide thicknesses on the fatigue behavior of an Alcan 57S aluminum alloy in wet and dry environments. Test pieces were machined out of a 0.05-inch thick sheet to an 11-inch radius toroidal test section of 0.500 inch minimum width. Direct comparison could only be made on specimens of identical machining history since the machining procedure was found to affect the fatigue life of the specimens. All specimens were degreased and finished with dry No. 400 grit silicon carbide paper in a longitudinal direction before testing in pulsating tension. Tests were carried out on a Sonntag SF-1-U machine operating at 1800 cpm. Anodization was accomplished by a tartaric acid process for three applying voltages of 0.5, 2.0, and 10 volts. The films on the surfaces had barrier-layer thicknesses that ranged from a few to not greater than 140 Å. Eeles found that the 0.5-volt-oxidized specimens to differ in some unknown way from the films grown at the other two voltages. He also noted that his probability of failure versus life curves at a given stress changed with environment (i.e., the probability of failure of the specimens decreased in dry environments for the same number of cycles than those in wet environments). The 0.5 and 10 volt samples saw an increase in failure in the wetted environments as opposed to drier environments. He concluded that the scatter experienced is due to strain discontinuities at the metal-oxide interface of the barrier layer instead of in the outer oxide layer that was exposed to the atmosphere.

E. G. Eeles and R. C. A. Thurston (15) in 1970 studied natural oxide films and their effect on cyclic stressing on two commercial aluminum alloys (Alcan 57S and Unclad 2024-T3) in moist and in dry air. Moist saturated air and air of less than 5 per cent relative humidity made up the wet and dry environments respectively. All tests were carried out in pulsating tension with a ratio of minimum to maximum stress of 0.05 on either of two 2-ton Vibrophore machines operating at 2180 cpm or on a Sonntag machine operating at 1725 cpm. Test specimens were essentially the same as those mentioned in reference 14 except that the width of the toroidal section was now 0.475 inches. Differences in the fatigue lives were seen in an earlier investigation (16) when the specimens were mounted and tested directly, from those allowed to set in the test chamber before stressing. This stimulated the investigators to be more consistent in their study and thus they would either refinish the surface of the specimen with abrasive paper just before testing, or allow the specimens to set in the test chamber for approximately 30 minutes before testing (the method depended on the type test desired). Maximum stress used in Alcan 57S and 2024-T3 were 34000 and 45000 lb/in.^{2}, respectively. A thin plastic bag sealed to the grips of the testing machine was the test chamber to which saturated air was admitted. Some specimens were stored in moist air for as long as four weeks to allow oxide stabilization before testing. From these specimens a series of tests were run to study the effect of step-wise increase in the oxide film during fatiguing. Another series were run with the specimens being repolished every 50,000 cycles throughout the test. The latter two tests shortened the lives of the specimens. Specimens that were stored in moist air for a minimum of three weeks after polishing, and before stressing, showed an increase in life when repolished at the first 50,000 cycles and then fatigued to failure. It was concluded that stressing freshly grown oxide was more damaging initially than stressing after a short period where a certain amount of oxidation had taken place. Thick oxide films considerably reduced the fatigue lives of the specimens. Their tests on the oxide film growth gave results which implied that the damaging of the oxide film is considerably reduced at thin films than those of higher thicknesses where higher differential strains in the oxide would be encountered at the metal interface. The ability of the natural oxide to repair itself became more difficult as the film thickness increased. A series of tests were carried out on 2024-T3 and 57S alloys in moist environments whose specimens had been previously pretreated in various solutions of dodecyl alcohol and kerosene for a minimum of two weeks showed the following results: (a) dodecyl alcohol reduced the scatter but had no effect on the mean fatigue life in the 57S alloy, (b) dodecyl alcohol reduced the fatigue life with no effect on the scatter for the 2024-T3 alloy, and (c) the kerosene had no effect on the fatigue life or scatter on the 2024-T3 alloy. Fatigue lives of the 57S alloy specimens in a dry air environment were higher than the fatigue lives of the specimens tested in moist air (for tests less than 10^6 cycles). For tests conducted above 10^6 cycles, the dry environment gave lower fatigue lives than the moist one.

In summary, some metals in general and aluminum alloys in particular have been proven to be affected by the water vapor in the air. The majority of the investigators [(1), (9), (11), (12), (14), and (15)] that experimented with aluminum agree [except (10)] that the above effect exists.

TEST APPARATUS

The test apparatus consisted essentially of four major items: a fatigue machine, test chamber, humidifying unit, and a uniform air source. Other equipment used in the investigation was in the area of instrumentation.

The fatigue machine used in the investigation was a Sonntag model SF-01-U machine as displayed in Fig. 1. The specimens were mounted between two stationery blocks that are rigidly attached to the stationary platen of the machine (see Fig. 2). One of these blocks served as a means for fixing one end of the specimen while the other allowed a through shaft with a coupling attached to secure the other end of the specimen. An arm keyed to the through shaft and connected to the vibrating oscillator cage provided a completely reversed torque of constant amplitude to the specimen. The force was applied by a threaded unbalance mass rotating at operating speed of the driving motor (1800 rpm). Using the force created by the unbalance mass, stresses as calculated for this investigation are within ± 5% of their reported values. A variable transformer connected in parallel with the motor controlled overstressing in the specimen when bringing the rotating mass unbalance up to operating speed. It was by this means that any shock seen by the specimen was kept to a minimum. Limit switches located on the machine were set to cut the machine off once the limiting deflection of 1.15 degrees was attained during a fatigue test.

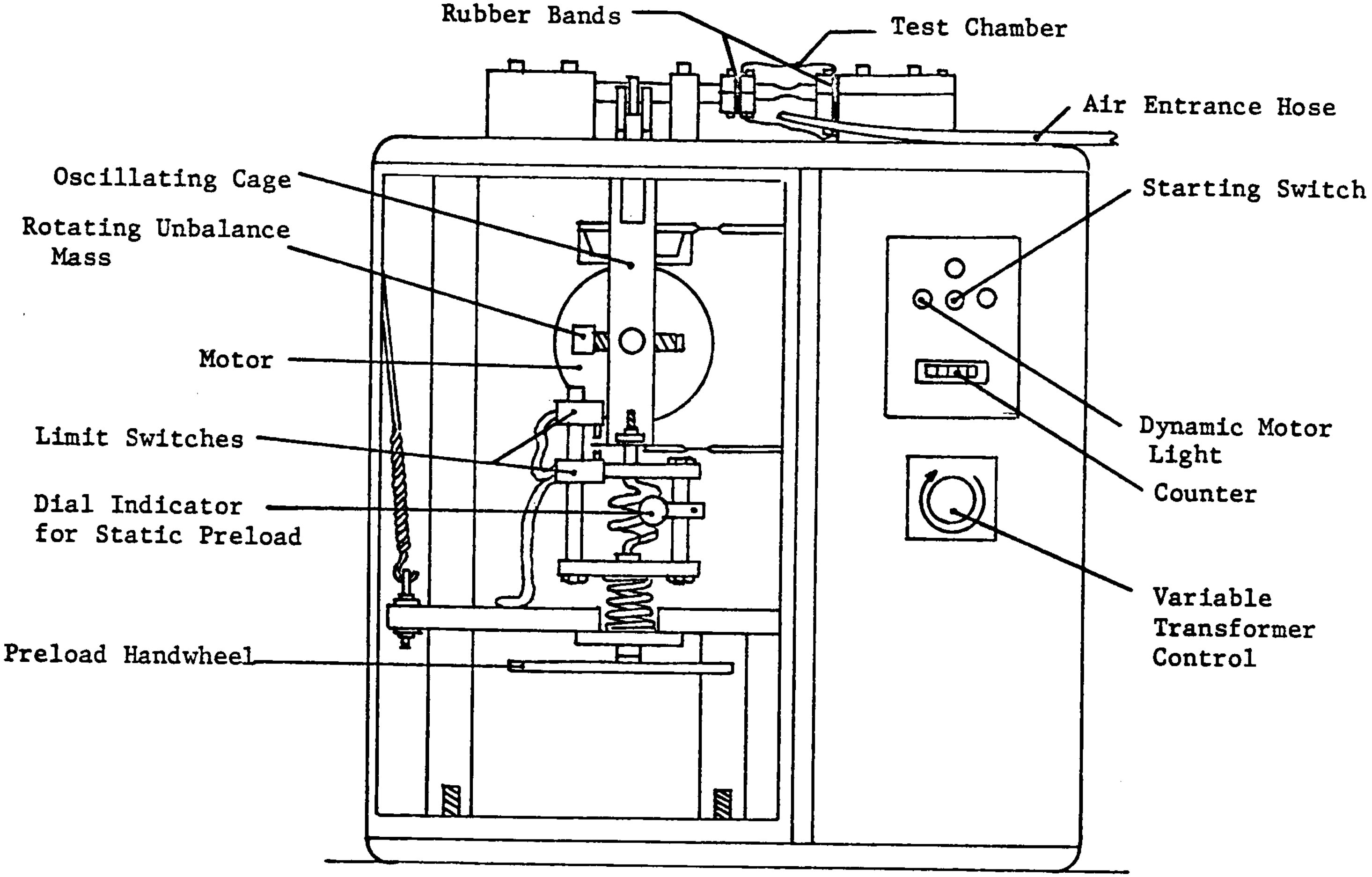

FIGURE 1. SONNTAG MACHINE--COMPONENTS

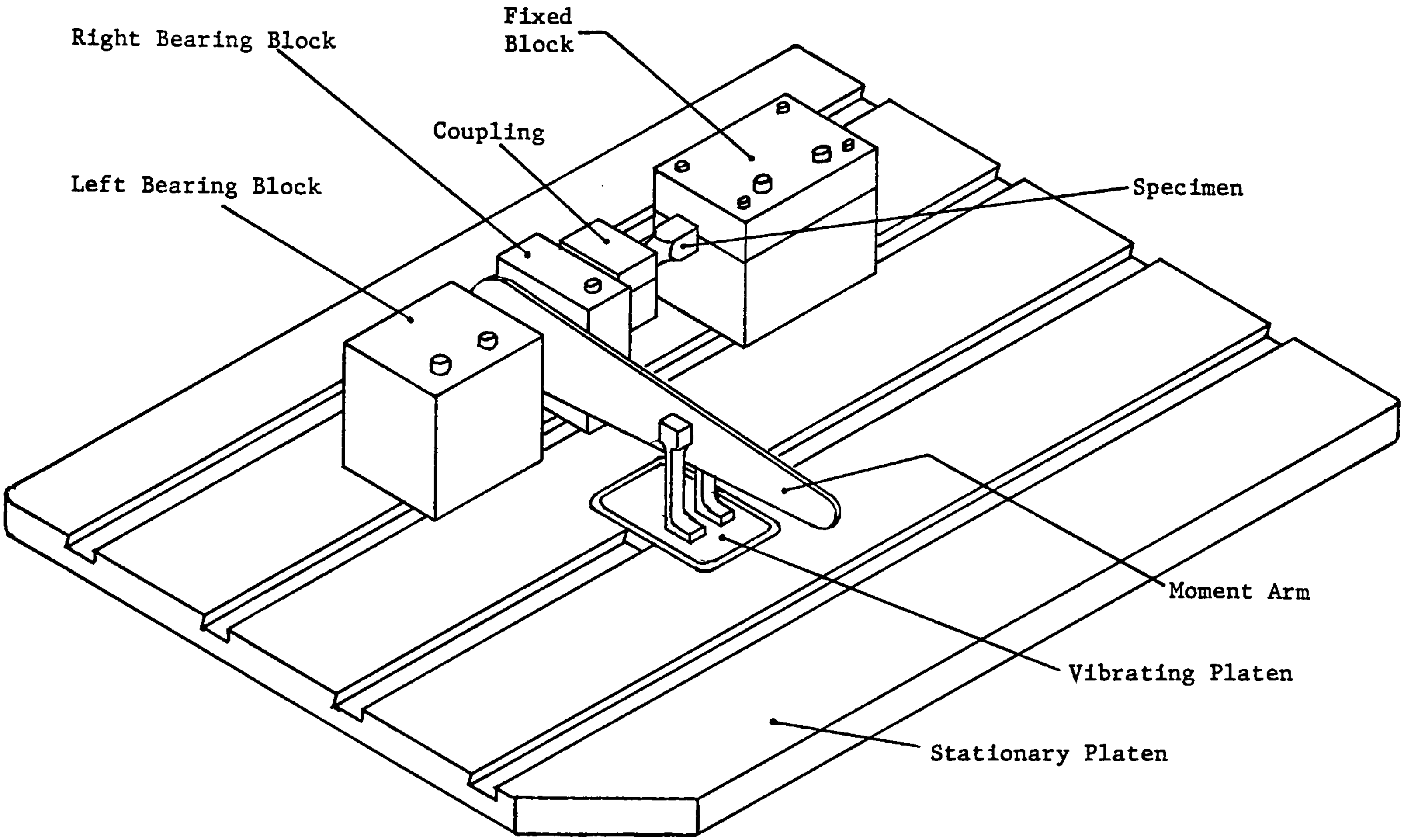

FIGURE 2. TORSION FIXTURE--COMPONENTS

The test chamber was made of heavy plastic material clamped to the grips by thick rubber bands. This arrangement formed the chamber surrounding the specimen. Air of known relative humidity, temperature, and flow entered through a tube from one side of the chamber. The flow rate was measured to be approximately 1.0 cfm.

Humid air was provided by bubbling regulated and filtered laboratory air through a bubble tower filled with distilled water (Fig. 3). The various ranges of relative humidities ran during this investigation (20-25%, low; 55-60%, medium; and 86-91%, high) were accomplished by the heating of the water in the tower for the high humidity, and the cooling of it for the lower ones. A thermostat mounted on the bubble tower regulated the heating and cooling of the water. This regulation held the appropriate humidity range once the correct humidity range was established.

A uniform air source was tapped directly from the laboratory supply. The air was then directed into the bubble tower for bubbling through the distilled water. The humid air leaving the bubble tower passed through two jars used as condenser traps for excess water in the air. The air then passed through several turns of tubing to allow its temperature to return to that of the room. A flowmeter was placed next in the line to measure the flow. After the final trap, a rigid plexiglas cylindrical chamber that housed a hygrometer sensor and thermometer gave continuous readings of humidity and temperature of the air going into the test chamber. The sensor was connected to an electric hygrometer (Fig. 4) whose dial readings could be readily

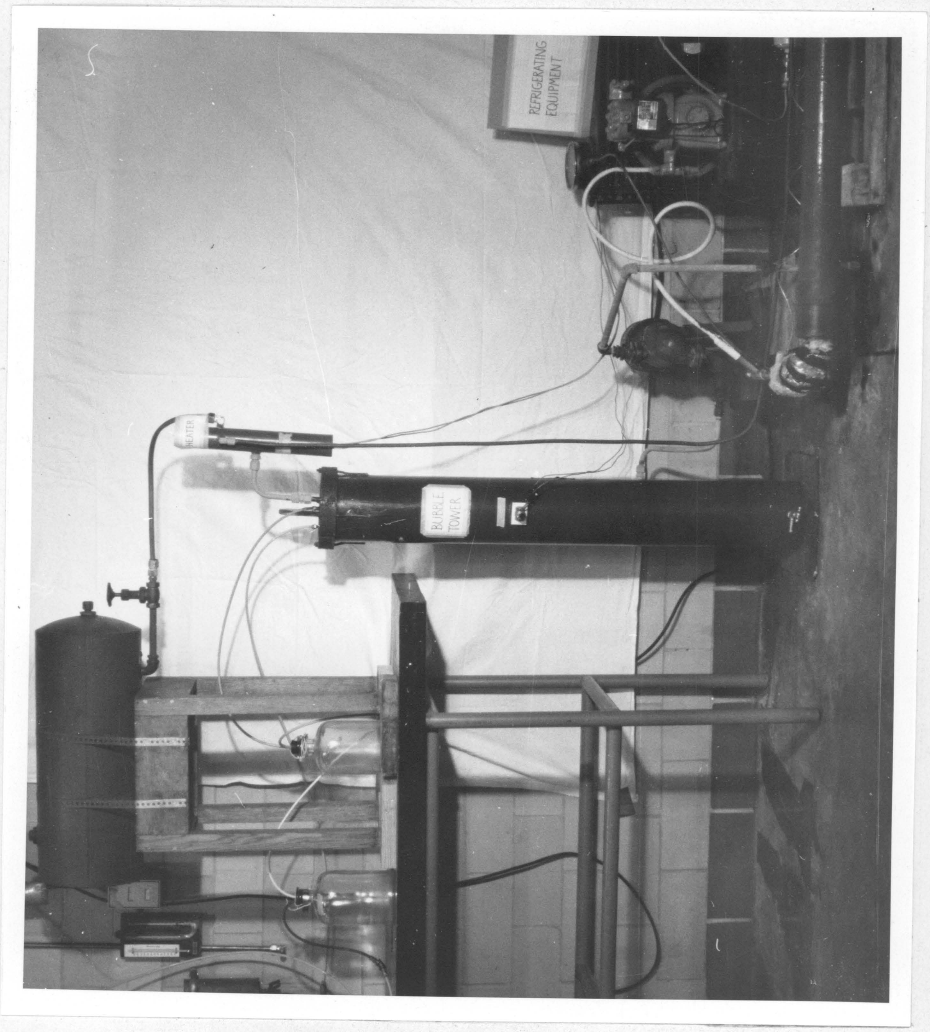

FIGURE 3. HUMIDITY EQUIPMENT

FIGURE 4. ELECTRIC HYGROMETER

converted to relative humidity through the use of supplementary graphs. The temperature in the laboratory was kept at 74 ± 2°F.

TESTS SPECIMENS

Four inch test specimens were cut from extruded 1/2-inch square bars of 2024-T351 aluminum. Properties for this alloy are given in Tables 1 and 2. The specimens upon machining were randomized and placed in lots of 18. Each of the lots are associated with a series that will be discussed later in the investigation. Specimens used for the first three lots in Series A and the first lot in Series B were machined from four, six-foot length bars. The rest of the specimens used in Series B lots two and three were from one six-foot length and one twelve-foot length bar. Lots 2 and 3 of Series B were ordered at a different time from the same company and therefore are of different heats than the other lots used in this investigation. To achieve the dimensions the specimens were machined as shown in Fig. 5 to a minimum diameter of 0.362 inches using a formed tool. This value was periodically checked during machining so as to be within ± 0.0015 inch. The sulfuric acid anodization process required the etching of all specimens for cleaning purposes (removal of scale, paint, foreign matter, etc.). The sodium hydroxide etchant used in this investigation removed approximately 0.5 mils of metal per minute. Since a two-minute etch was used on all the specimens for this study, the minimum diameter in the reduced section was 0.360 inches. The third lot of Series B was polished with No. 400 grit paper in order to make comparisons with unpolished surfaces of lot two of the same series. The circumferential marks left on the unpolished surfaces were removed in lot three so that the remaining marks

TABLE 1 *PROPERTIES OF ALUMINUM ALLOY 2024 - T351

Ultimate Strength	60 Ksi
Yield Strength	44 Ksi
Ultimate Shear Strength	32 Ksi
Modulus of Elasticity	10.5×10^6 lb./in.^{2}
Shear Modulus	4.00×10^6 lb./in.^{2}
Poisson's Ratio	0.33
Weight	0.10 lb./in.^{3}
Elongation	12%
Thermal Conductivity 212°F	81.5 Btu/(hr ft °F)
Thermal Expansion 212°F	12.9×10^{-6} in./in°F

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*Metallic Materials and Elements for Aerospace Vehicle Structures, Military Hand Book (MIL-HDBK-5A), 1966.

TABLE 2 *PERCENTAGE CHEMICAL COMPOSITION LIMITS 2024 - T3 ALUMINUM ALLOY

Silicon	0.50
Iron	0.50
Copper	3.8-4.9
Manganese	0.3-0.9
Magnesium	1.2-1.8
Chromium	0.10
Zinc	0.25
Other	0.15
Aluminum	Remainder

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[*] Alcoa Aluminum Handbook, Aluminum Company of America, 1959. The composition limits on 2024-T3 are the same for 2024-T351.

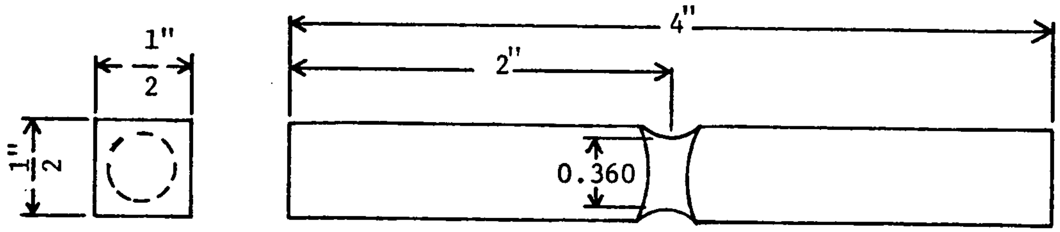

FIGURE 5. TEST SPECIMEN DIMENSIONS

were running in the longitudinal direction to the axes of the specimens. The polishing operation for the above lot was done by hand. After machining, the specimens were put in a plastic bag and placed in a desiccator to await anodization (application of the $AL_2 0_3$ coating). The anodization of the specimens was done by a commercial company. The steps of the anodization process are given in Appendix A. The determination of the nominal coating thicknesses placed on the metal was done through the use of an anodizing graph for conventional sulfuric acid anodizing. This graph plots the nominal coating thickness versus anodizing time for the particular current density, voltage, temperature of electrolyte, and specific electrolyte used (see step 8, Appendix A). The aluminum oxide layer is composed of two layers, a porous layer and a barrier layer. The barrier layer is closest to the metal substrate and when once formed is continuous throughout. The porous layer lies on top of the barrier layer and contains many pores. Sealing, a method by which the pores in the porous layer are closed by the formation of a hydrate on the oxide, is used to maximize the corrosive protection of the oxide to the metal. The sealing process for this investigation consists of boiling the specimens in deionized water for at least 30 minutes. The aluminum oxide hydrate that is formed in the sealing operation forms along the walls of the pores to accomplish the above sealing effect. An oxide coating (17) of less than 0.5 mil thickness may not exhibit cracking until the surrounding temperature exceed 250°F; caused by the fact that the thermal coefficient of expansion is smaller than that of the base metal (approximately 3 times). As will be seen later the 0.3 mil coating showed the above cracking phenomena but the 0.05 mil coating did not after growing and sealing the oxide coating on the specimens. When the cracking does occur, it is found that the crack penetrates through to the base metal (18). The cracks developed in the oxide coating due to fabrication have not been found to diminish the oxide coating protection to the metal (17). The specimens, after anodizing, were individually bagged in small plastic bags. All of the small plastic bags were then placed in a larger plastic bag for storage in the desiccator to await time for testing. Figure 6 is of two specimens in the "before" and "anodized" condition. Only eighteen specimens (one lot) were anodized at a time.

One assumption made with regard to the anodized coating on a specimen was that its strength did not significantly alter the stresses in any way since very thin coatings were used. Properties for aluminum oxide are given in Table 3.

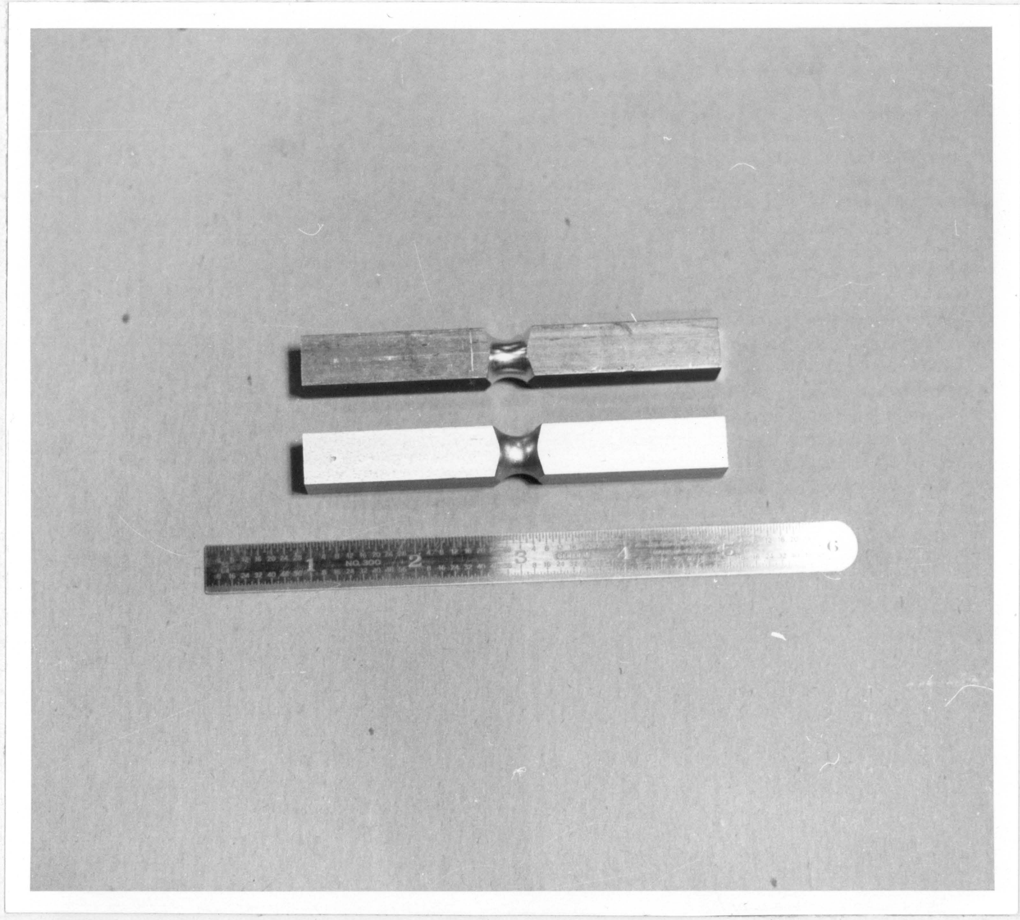

FIGURE 6. SPECIMEN IN BEFORE AND AFTER ANODIZE CONDITION

TABLE 3 *GENERAL PROPERTIES OF ALUMINA (Al_2O_3)

Ultimate Strength**	38 Ksi
Bend Strength	67 Ksi
Impact Strength	1.5 in. - lb.
Modulus of Elasticity**	57 x 10^6 lb./in.^{2}
Shear Modulus**	18-20 x 10^3 lb./in.^{2}
Poisson's Ratio**	0.26
Density	3.98 ± .02 g/cm^3
Melting Point	3720 ± 40 °F
Specific Heat	0.19 Btu/hr °F
Thermal Conductivity	19.4 Btu/(hr ft °F)
Thermal Expansion	4.4 x 10^{-6} in/in °F
Hardness	79 Rockwell C

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[*] Engineering Properties of Selected Ceramic Materials, compiled by Battelle Memorial Institute Columbus Laboratories, Columbus, Ohio, published by American Ceramic Society Columbus, Ohio, 1966.

[**] Property Value at 70°F

INVESTIGATION

Since ASTM (19) recommends that no less than four specimens be tested to estimate the variability of data, six specimens were run at each stress level for each humidity condition (20-25%, low; 55-60%, medium; and 86-91%, high). The specimens were run at stress levels of 13000, 17000, and 20000 lb/in^2. All three stress levels were run for a given coating thickness at each of the three relative humidities in a series. The two thicknesses of anodic coating used (0.05 and 0.3 mils) were arbitrarily chosen for testing purposes. The above number of specimens, stress levels, and relative humidity levels were essentially the same as those used in (1) for which comparison of results will be later made. The source (Alcoa Company) and heat of the material were the same as those used by (1) in his investigation. Since the above material ran out part of the way through this investigation, more had to be ordered which caused for another heat to be used in the latter parts of this investigation. More about this consequence will be mentioned later.

In any fatigue experiment it is important that the specimens be randomized so as to prevent biasing of the data. It was therefore thought appropriate to randomize the specimens for the following uncontrollable factors which could not be totally accounted for in the investigation: (a) inhomogeneity of material, (b) roughness of the reduced section, and (c) tool wear. Factors relating to the humidity equipment and coating did not allow for a totally random experiment.

The humidity equipment capabilities of changing the temperature of the water in the bubble tower to obtain the various relative humidity levels used was very slow and would take a long time. Therefore the relative humidity could not be readily changed from test to test to make complete randomization possible. The anodic coating having been applied outside the laboratory and the preparation necessary for this process also made complete randomization impractical. Therefore it was decided by the present investigator to randomized all specimens based upon manufacture and group them in sequential lots of 18 to be coated and tested in a design as that given in Appendix B. The specimens were numbered on manufacture and randomized accordingly. Unfortunately the supply of alloy stock on hand [the same as that used by (1)] at the start of the investigation ran out and more had to be ordered. The specimens from this new order composed the second and third lots of Series B. These were numbered from the point where the previous supply was exhausted and then randomized. The first six specimens in a lot were run at the highest stress, the second six were run at the medium stress, and the last six at the lowest stress level. It was thought that not randomizing the specimens within the relative humidity levels according to stresses would cause less variation in the results (i.e., it would cause less changing of the unbalanced mass on the fatigue machine). The order of running the lots were Series A (lots one, two, and three) and Series B (lots one, two, and three). Only one lot would be anodized at a time (all anodized to same coating thickness desired).

Series A consisted of running specimens of 0.3 mil coating thickness at the three relative humidities and stress levels. This series was made up of three individual lots.

Series B was run in somewhat of a different manner than Series A. It was thought by the present investigator that more information could be obtained if the medium humidity level would be omitted in this series and replaced by specimens whose surface had been polished. This would determine whether the circumferential marks left on the other specimen surfaces affected the specimens fatigue lives in any way. The consequence of not running a medium humidity lot in this series was thought not too important after studying results of Series A (caused an unbalanced design as shown in Appendix B). At the time it was thought by the present investigator that the same type of shifts in the S-N curves seen in Series A would be seen again in Series B. Since the maximum shifts in the S-N curves were also seen between the specimens tested at high and low humidity in Series A, it was thought that the high and low relative humidity levels would give the most meaningful results (so far as establishing significant differences between specimens fatigue lives at the high and low humidity levels). The specimens of the medium humidity lot was therefore used in obtaining information about the surface condition (polishing) of the specimens to determine this effect on the fatigue lives of the specimens as mentioned above. Series B consisted of specimens with 0.05 mil thick anodic coatings only. Lot one of this series was run at high relative humidity while lot two was at low humidity (both lots in "as machined" condition).

The third lot was in the "as machined and polished" condition and also run at low relative humidity (replaced the medium humidity lot). The polishing of this last lot was done so as to leave the marks longitudinal to the axes of the specimens.

Runout, a term found frequently in fatigue literature, defines a specimen that did not fail before a certain number of cycles. Runout in these tests was set arbitrarily at ten million stress cycles.

An assumption pertaining to the air was that its pH was constant during all tests. Throughout the investigation, a sample of the distilled water was taken from the bubble tower approximately every week (except for lot 1 in Series A where only one pH reading was taken) and checked for its pH value. The check provided the investigator with a means of determining the pH of the incoming air into the bubble tower.

The shear stress was calculated using the well known equation for torsion (all geometrical constants based on the reduced section of the specimen)

$$S = \frac{Tc}{J} \qquad (1)$$

where:

S = shear stress, lb./in.^{2}

T = torque delivered to specimen (product of the moment arm length and force produced by the mass unbalance on fatigue machine), in.-lbs.

c = radius, in.

J = polar moment of inertia of cross-section, in.^{4}

The method of testing each specimen was as follows:

1. Relative humidity range and loading were adjusted on the equipment before start of each test.
2. The specimen to be tested was removed from storage and mounted in the torsional fixture.
3. The plastic chamber with the desired incoming humidity controlled air was sealed to the fixtures of the test machine.
4. A five minute stabilization period was allowed before the start of the test.
5. The machine was started.
6. Once the amplitude in one direction exceeded the set degrees, the machine was stopped.
7. The number of cycles was recorded.
8. The above steps were repeated starting with step 1.

RESULTS

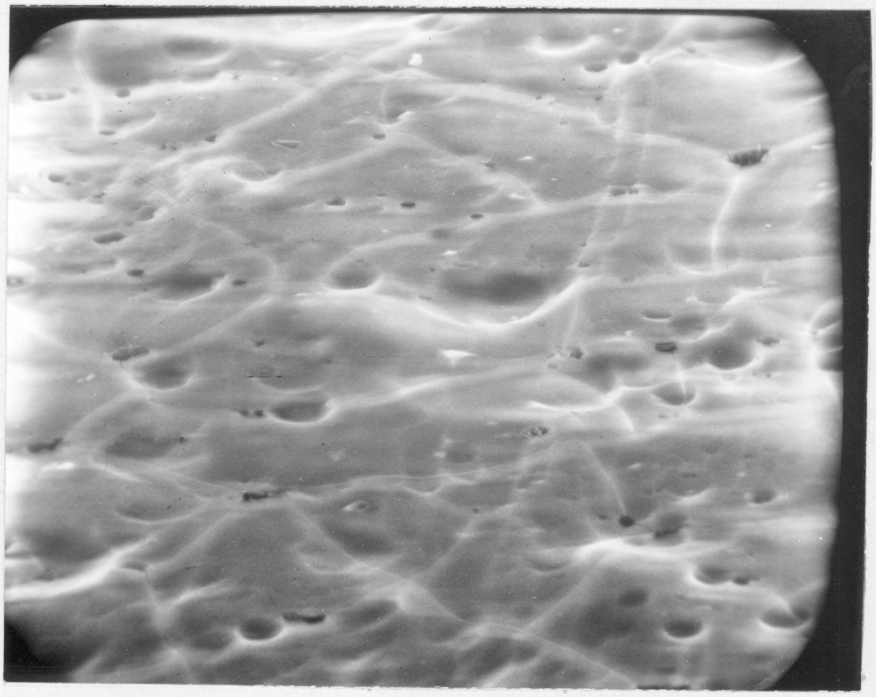
Surface Observations

Scanning electron microscope photographs of the anodized surfaces in various conditions are shown in the figures discussed below. As this study is on the anodic coating and its effects on corrosion fatigue of 2024-T351 with regard to relative humidity, polishing, coating, and thickness, no attempt is being made to report the fatigue mechanism from a metallurgical standpoint.

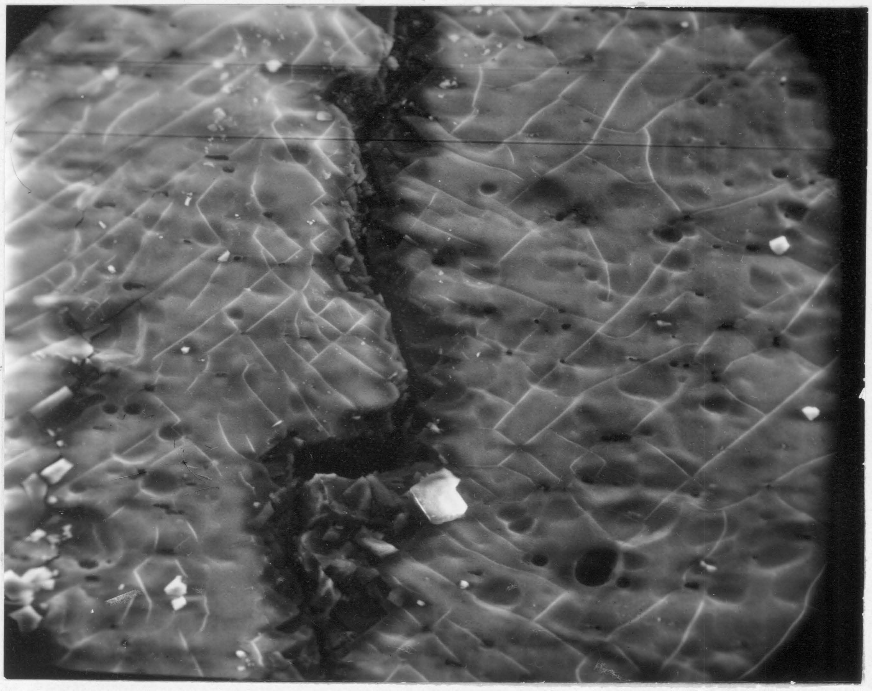
Figure 7 is of the reduced section of a anodized 0.3 mil coated specimen in the before and after test condition. Not the coating cracks in the coating. These cracks are a result of the sealing operation mentioned earlier. The fatigue crack is the darker zig-zag vertical line almost in the center of the photograph. An average crack thickness using four arbitrarily chosen points along the fatigue crack gave a value of approximately 0.28 mils. The specimen was fatigued at high humidity and low stress.

Figure 8 shows a hairline crack in the coating taken at high magnification taken in the same general area as in Fig. 7. The width of the crack in the coating located in the central left portion of this figure was approximately 0.005 mils taking the average of four arbitrary points along its length.

Figure 9 shows the surface of an 0.05 mil coated specimen in the "after" condition for high stress and low humidity. Note the texture



Before. 800X



After. 440X

FIGURE 7. 0.3 MIL ANODIZED SPECIMEN BEFORE AND AFTER TESTING.

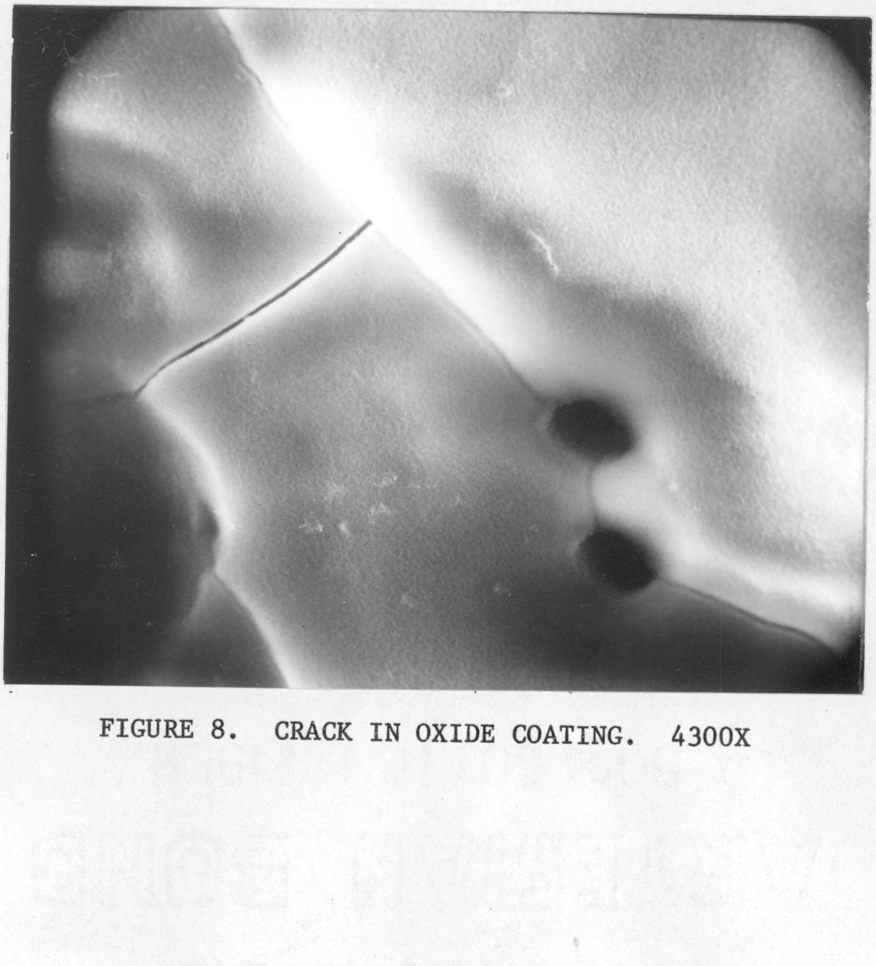


FIGURE 8. CRACK IN OXIDE COATING. 4300X

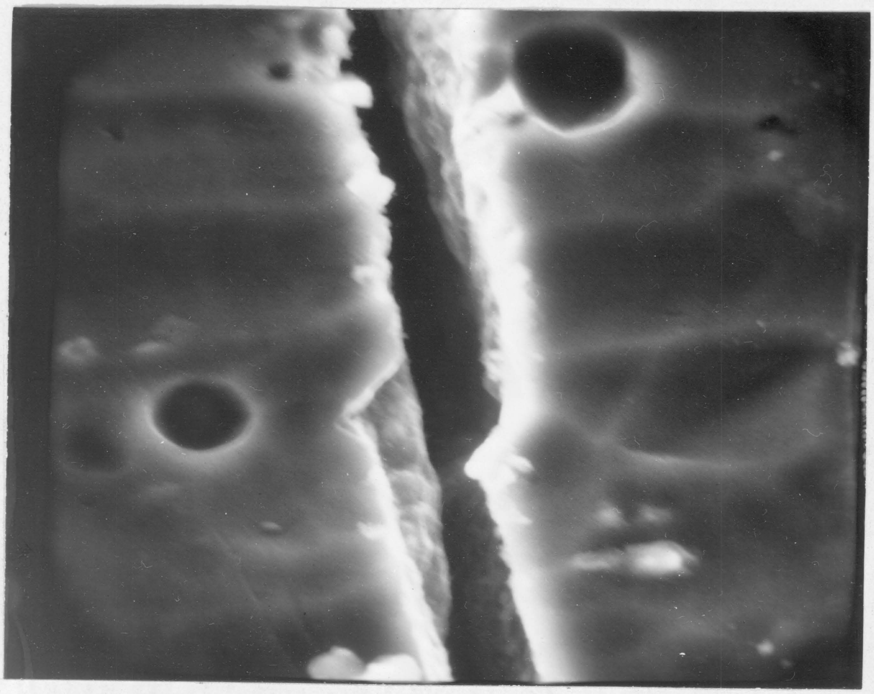


FIGURE 9. FATIGUE CRACK IN A 0.05 MIL SPECIMEN. 3500X

of the coating around the fatigue crack indicating no signs of the coating cracks as seen in Fig. 7. Even though the magnification factors of the two figures are not the same, in order to make the comparison between the two figures justifiable, the investigator observed several other 0.05 mil specimens at various magnifications to see if they possess the coating cracks as exhibited on the 0.3 mil specimens. The result was that no coating cracks could be found before and after fatigue testing the 0.05 mil specimens. The 0.3 mil specimens coating cracks appeared the same before and after fatigue testing.

Figure 10 is an edge view of a specimen that cracked all the way through under the condition of high stress and high humidity. The lighter portion of the figure is that of the anodic coating. The base metal under the coating is dark. The coating itself is a nominal 0.3-mil thickness. Measurement of the coating thickness by choosing four arbitrary points along it gave an average value of approximately 0.24 mils.

Figure 11 is a view of the cross section of the fatigue surface shown in Fig. 10. This figure shows one of the two mating surfaces after the crack propagation stage. Progression of the actual fatigue test can be inferred from this figure also. Note the uniformity of the disoriented patterns. These patterns seem to form concentric circles which converge to a "central core region" located off to the lower right of the center of the figure (the intersection of arrows placed on figure). The pattern displays the presence of the torsional

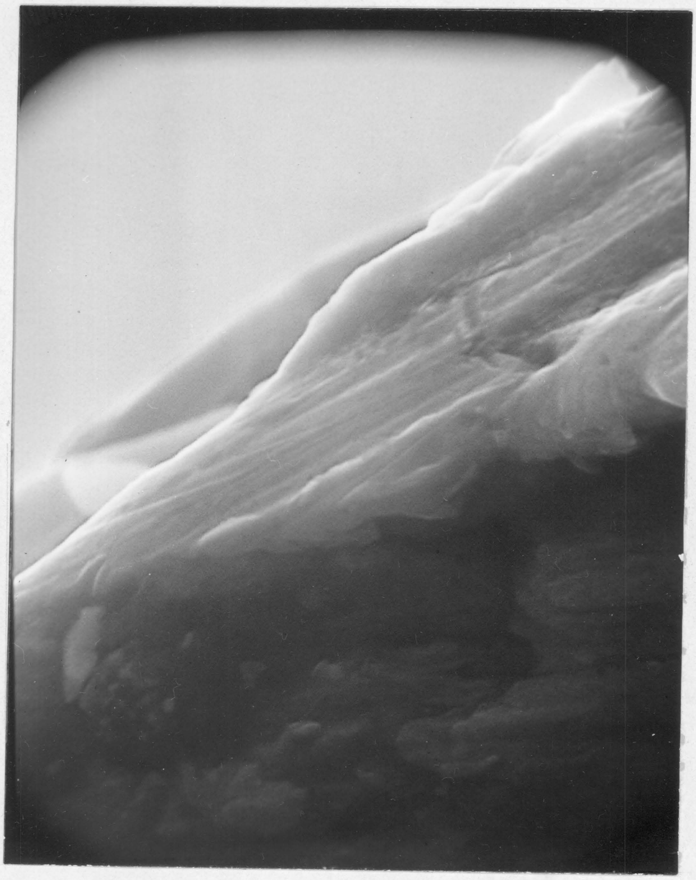


FIGURE 10. ALUMINUM OXIDE COATING. 10000X

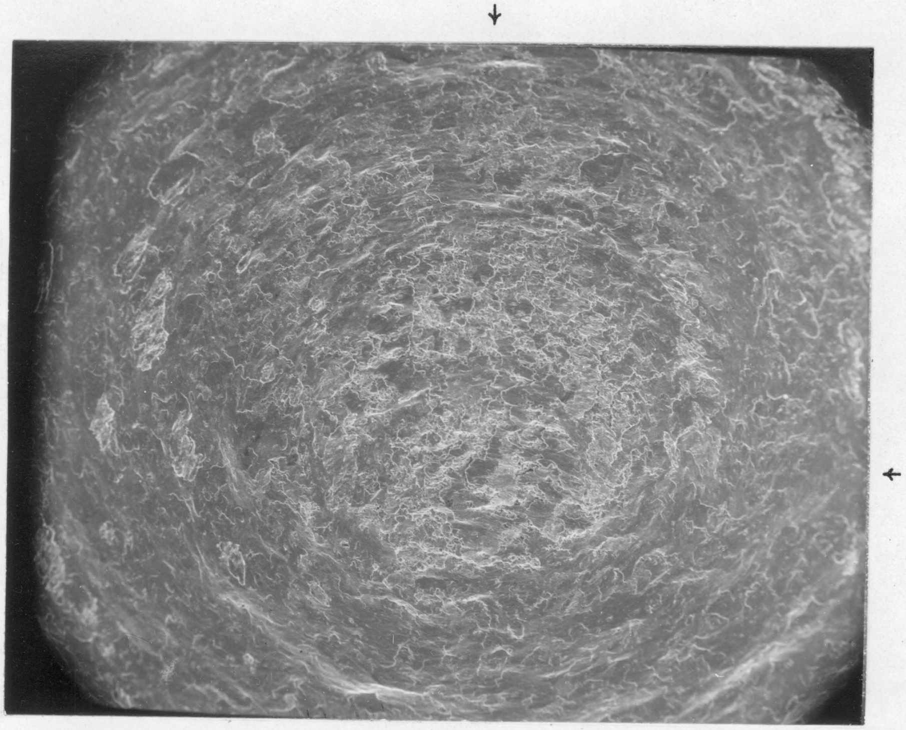


FIGURE 11. CROSS SECTION OF A FATIGUED SURFACE. 42X

mechanism in this study. From the appearance of this figure, the crack moved in from the extremities of the cross-section. Figure 12 is a view of what the above surface looked like in the region approximately midway between the central core and outer edge of Fig. 11. Note the fine lines in the figure caused by the rubbing of the mating surfaces. Figure 13 was taken from the "central core region" of Fig. 11. The pointed peaks shown throughout this figure are characteristics of ductile fractures. The "central core region" of this specimen is believed to be the last area to go before complete fracture.

Surface Roughness

Roughness measurements were attempted for the reduced section by use of a profilometer. Values for the surface roughness are not reported due to the lack of repetition in readings when the samples were checked at various times. The large change in surface roughness reading from one time to another was attributed to the type of stylus used in the profilometer because it was not the optimum type for use on the cylindrical contour of the reduced section. Figures 14 and 15 show photographs of two surfaces in the "as machined" and "as machined and polished" condition. The figures are of the specimens perpendicular to their longitudinal axes showing the edge contour of the reduced section. The "as machined" specimen appears to be rougher in the direction into the figure than the "as machined and polished" specimen (this direction corresponds to the circumferential direction of the specimen). Viewing the two edges in the same two

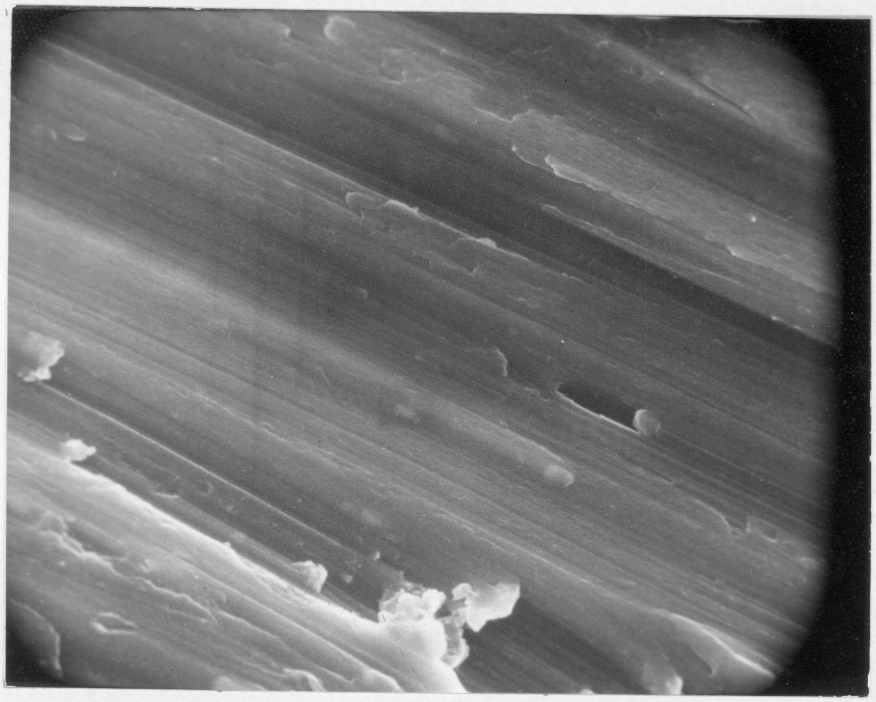


FIGURE 12. BLOW UP OF AN AREA BETWEEN CENTRAL CORE AND OUTER EDGE IN FIGURE 11. 4200X

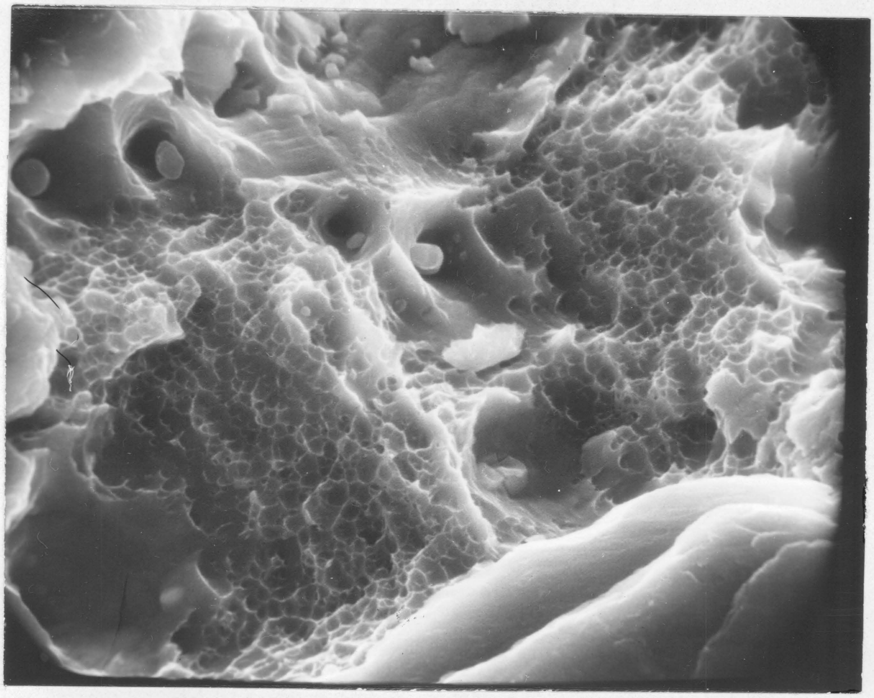


FIGURE 13. BLOW UP OF AREA TAKEN FROM CENTRAL CORE REGION IN FIGURE 11. 4200X

↑ Circumferential ← Longitudinal

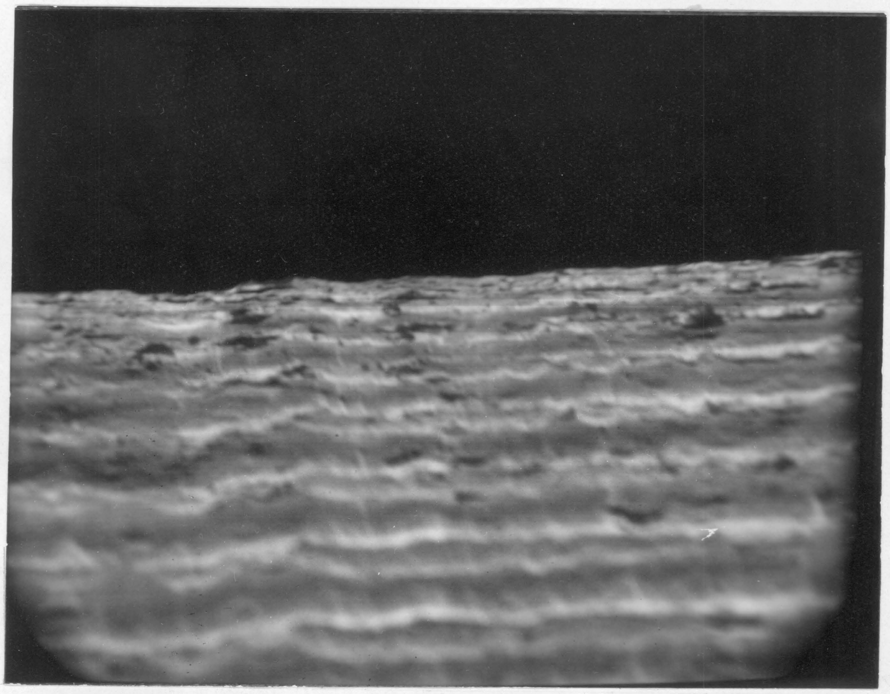


FIGURE 14. AS MACHINED SURFACE. 1300X

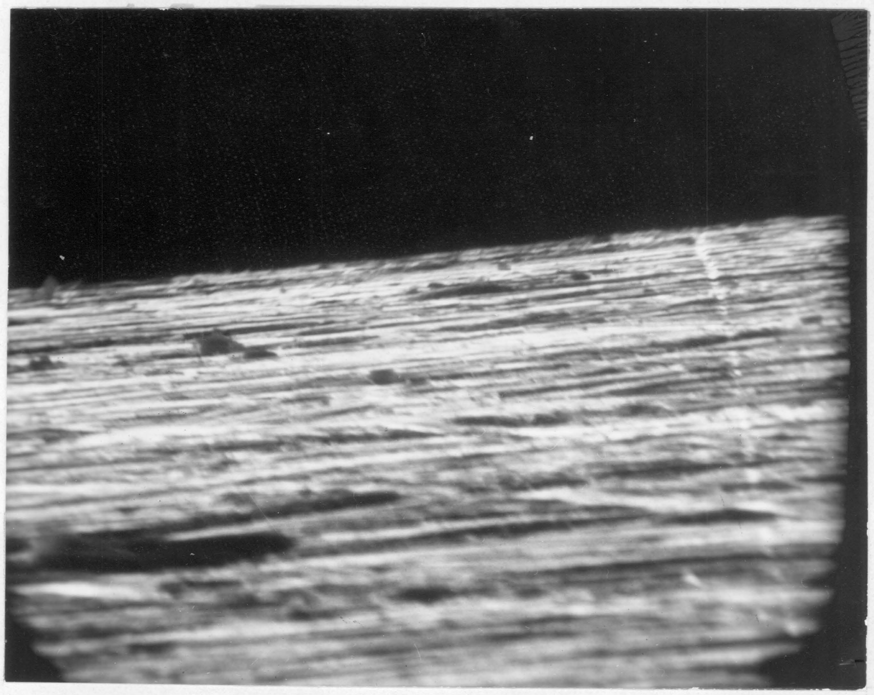


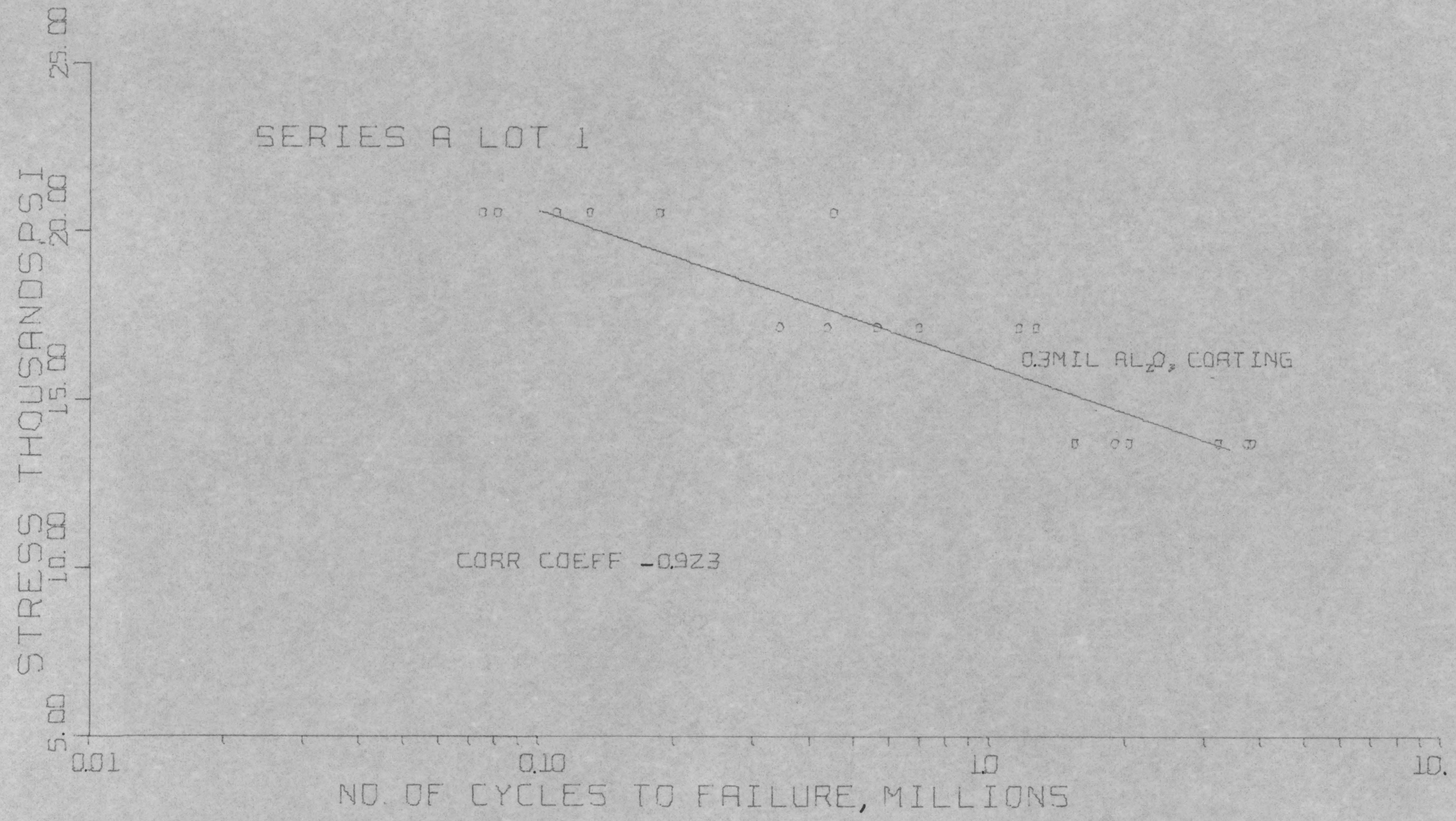
FIGURE 15. AS MACHINED AND POLISHED SURFACE. 1300X

figures in the longitudinal direction the "as machined and polished" specimen is smoother than the "as machined" specimen as before.

Results of Fatigue Tests on 0.3 mil Anodize Specimens

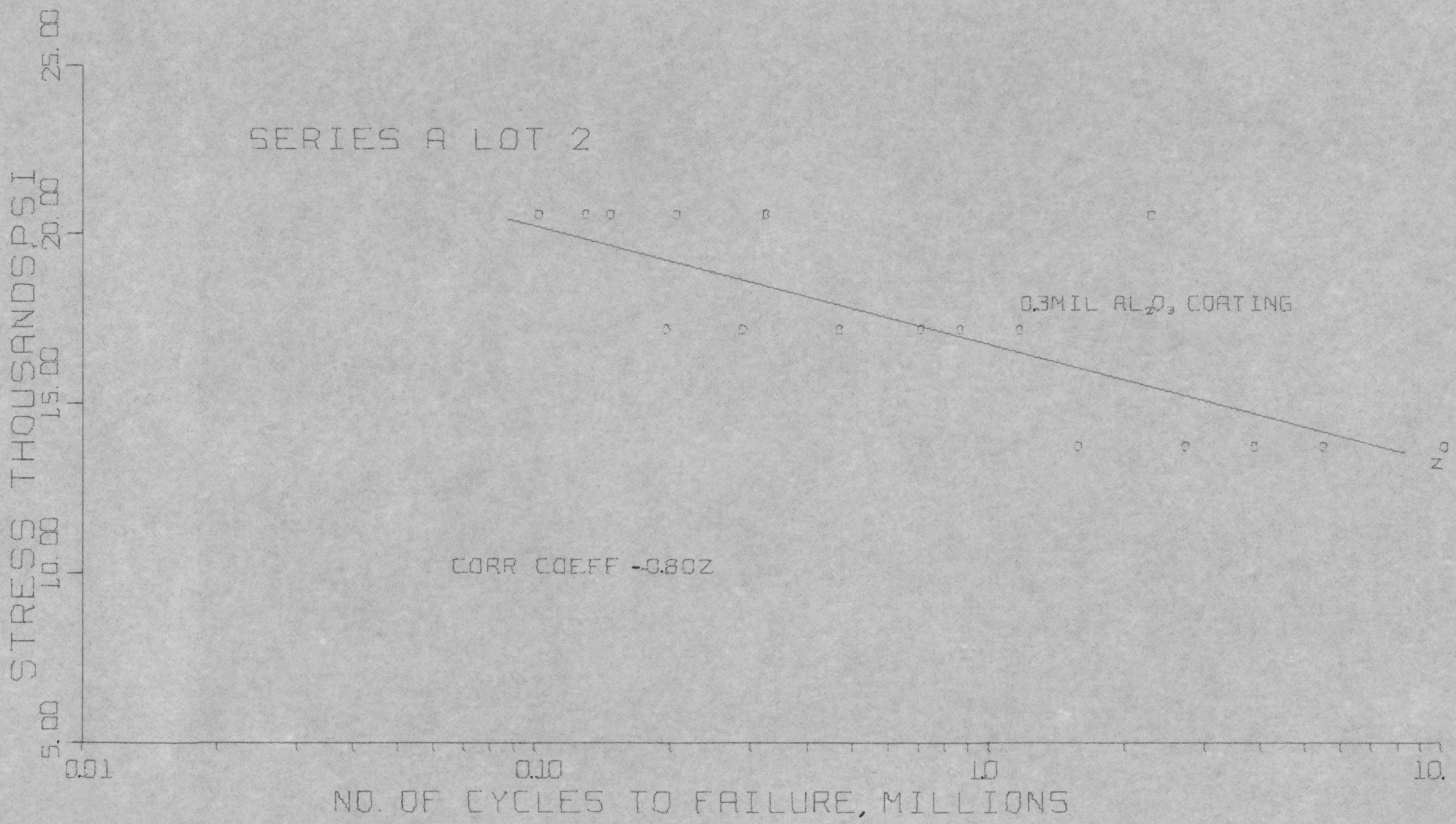
The data of Series A for the three relative humidity levels were fitted to straight lines of the semi-logarithmic type (S-N curves). The method of least squares was used in making the curve fits. The curves are shown in Figs. 16, 17, and 18, with all three curves plotted on Fig. 19 for purposes of comparison. The experimental points and a correlation coefficient are given for each curve on the Figs. 16-18. The specimens of the low relative humidity curve for this series had an increase in fatigue life of 281% over that of the high relative humidity curve at the high stress level. At the low stress level the increase in fatigue life was 194% for the same two curves.

An analysis of variance performed on the data of Series A at the 1% significance level given in Table D-1 of Appendix D, showed the change in humidity to have an effect on the lives of the specimens; i.e., an increase in the relative humidity showed a decrease in the fatigue life of the specimens. The analysis of variance given in Table D-1 are for high and low humidity only, since an analysis of variance performed on lots at high and medium relative humidities showed no humidity effect (Table D-2, Appendix D). The consequences of the relative humidity not having a significant effect at the 1%



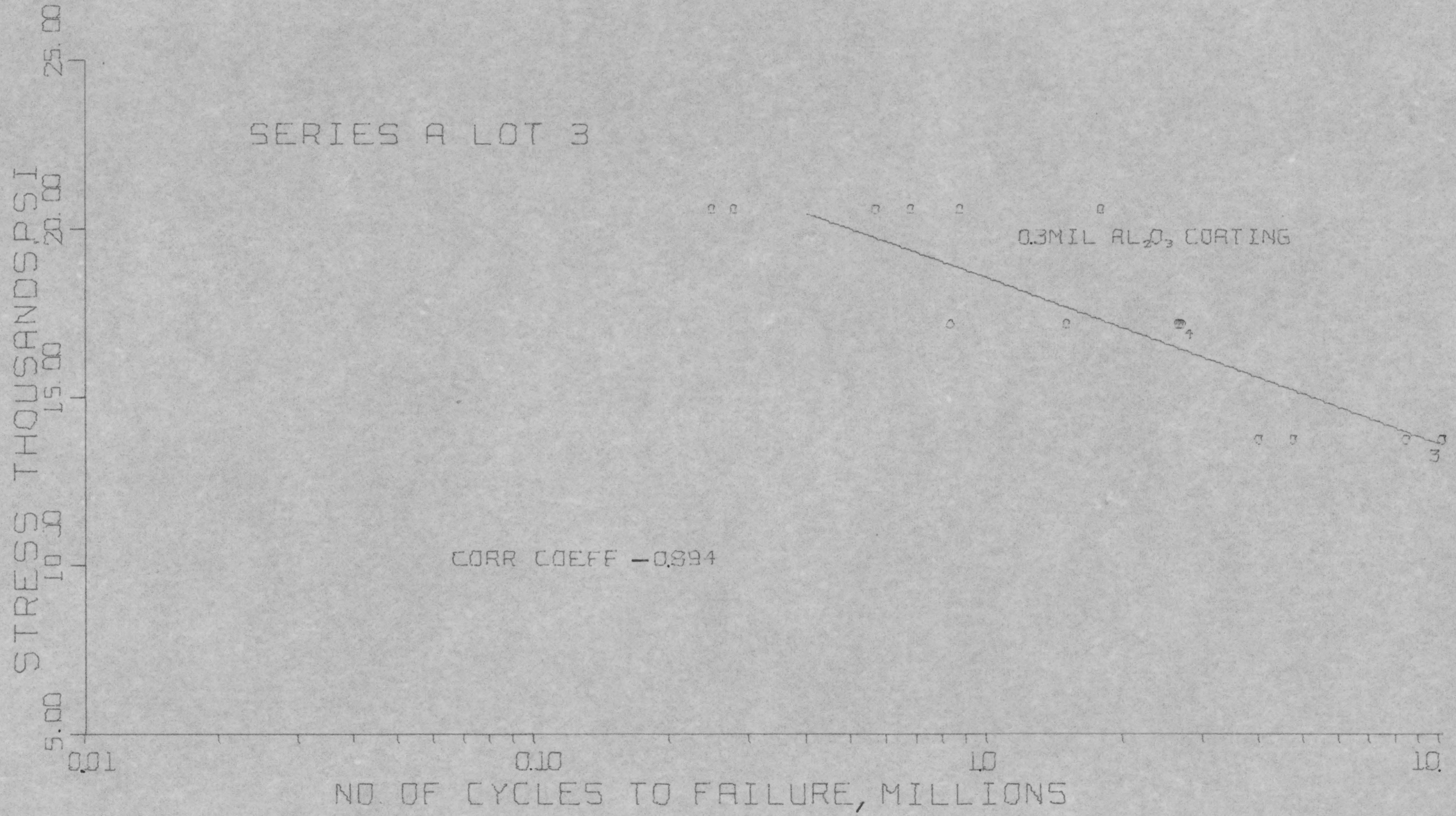
EXPERIMENTAL POINTS FOR S-N CURVE AT HIGH REL. HUMIDITY-SERIES A

FIGURE 16



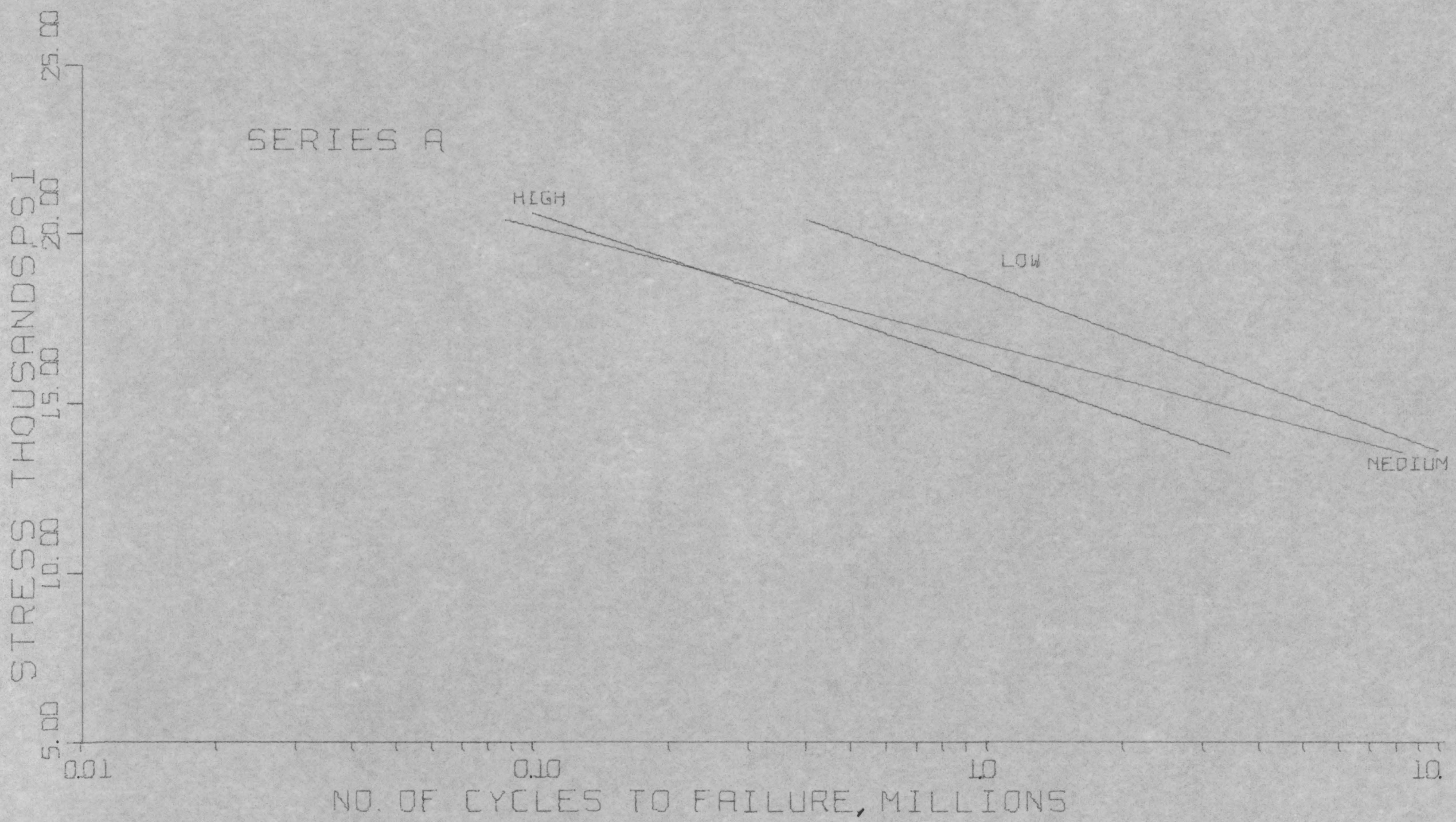
EXPERIMENTAL POINTS FOR S-N CURVE AT MEDIUM REL. HUMIDITY-SERIES A

FIGURE 17



EXPERIMENTAL POINTS FOR S-N CURVE AT LOW REL. HUMIDITY-SERIES A

FIGURE 18



S-N CURVES FOR SERIES A AT LOW , MEDIUM , AND HIGH HUMIDITIES

FIGURE 19

significance level for the high and medium humidities will be discussed later in the Results.

All the data for Series A are given in Table 4 according to their respective lots.

Results of Fatigue Tests on 0.05 mil Anodize Specimens

The curves in Series B were determined in the same manner as those in Series A. Figures 20, 21, and 22 are the S-N curves of Series B at the various humidity levels. Figure 23 is given for comparison of the above curves. As mentioned earlier, this series has a lot that was polished before anodizing (Lot 3). The two "as machined" lots of this series (Lots 1 and 2) which were run at high and low relative humidity respectively, showed the specimens for the low relative humidity curve to have an increase in fatigue life of 1112% at the highest stress level, and 338% at the medium stress level. An analysis of variance was again performed at the 1% significance level for this series involving lots for high and low humidity in the "as machined" condition. The results of the analysis of variance again showed humidity to have an effect on the fatigue lives of the specimens in the same way as before (Table D-3, Appendix D). An analysis of variance of the data for the "as machined" and the "as machined and polished" lots (Lots 2 and 3), found the polished specimens not significantly different from unpolished specimens as seen in Table D-4, Appendix D.

The data recorded for this series is listed in Table 5.

TABLE 4 EXPERIMENTAL DATA - SERIES A

Stress Level, lb/in.^{2}	Cycles to Failure, 10^3 Cycles		
	LOT 1	LOT 2	LOT 3
20000	80	320	1784
	445	145	563
	74	2264	274
	183	101	862
	128	128	672
	108	203	245
17000	338*	464	1486
	690	1160	824
	431	700	2657*
	1253	855	2612
	1146	193	2659
	557	284	2632
13000	1868	10000	10000
	3671	5408*	10000
	3171	3804	3934
	2011	1559	10000
	3755	2683	4687
	1525	10000	8320

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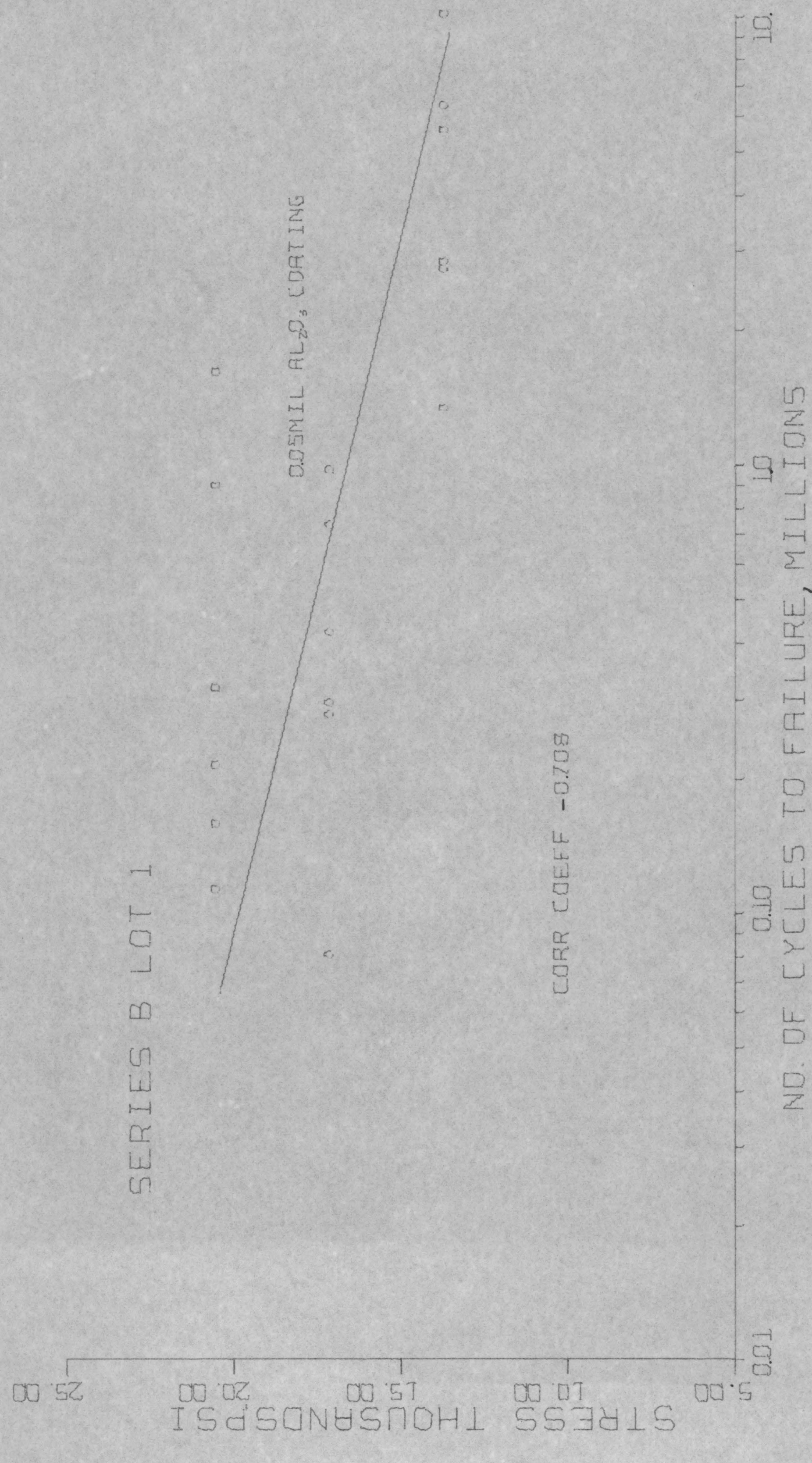
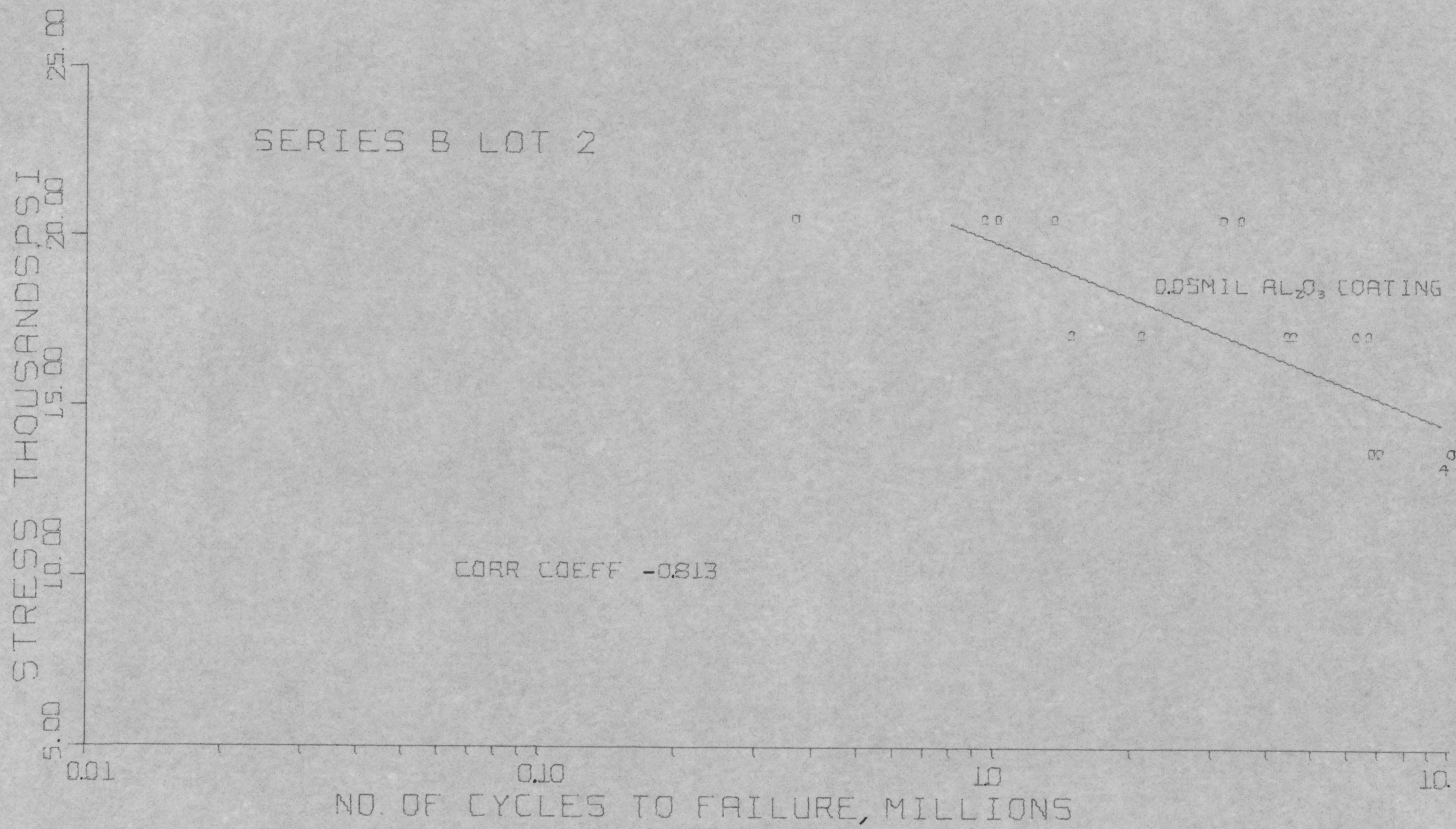
* Humidity went over the desired values less than 3%.



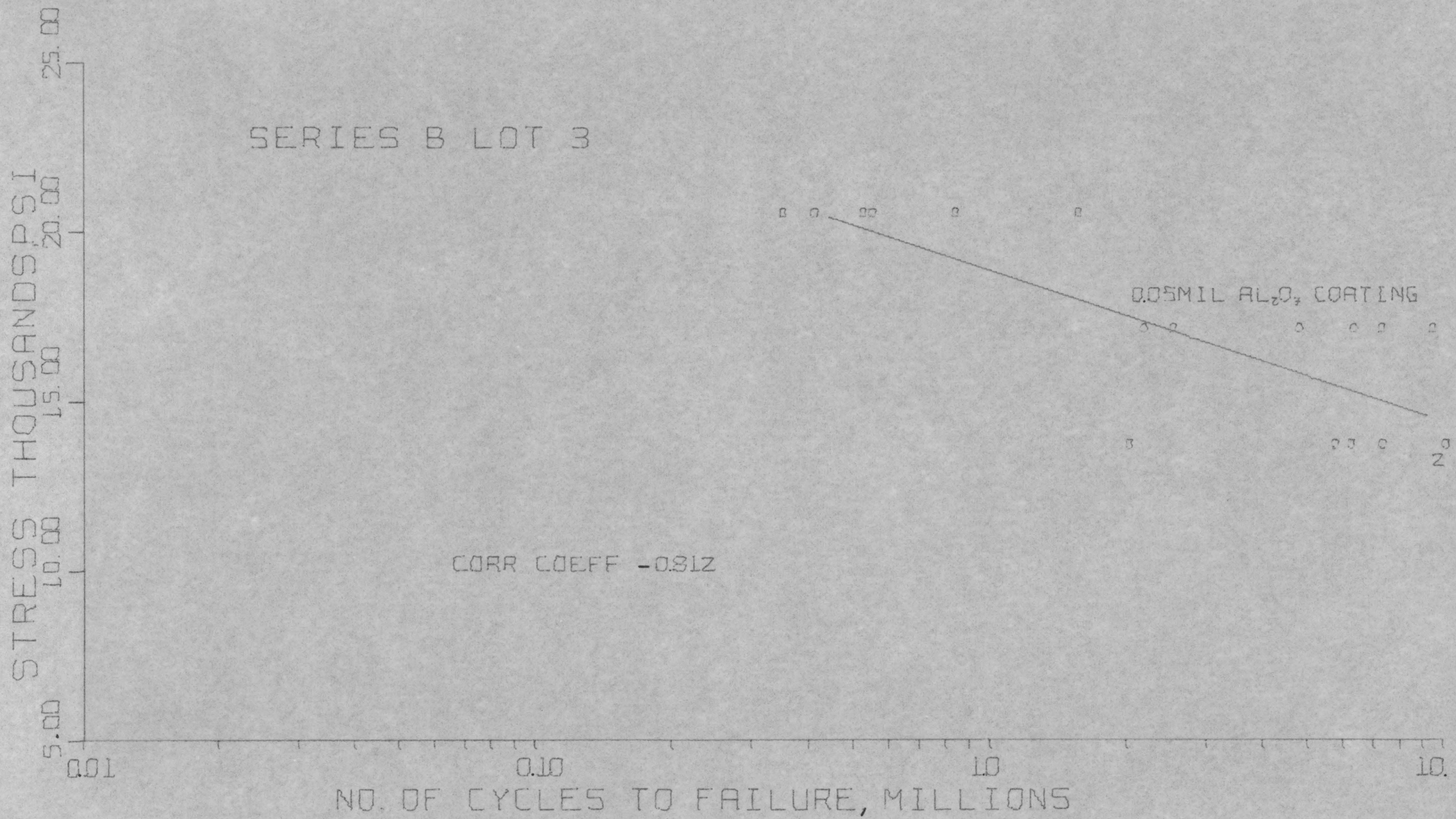
EXPERIMENTAL POINTS FOR S-N CURVE AT HIGH REL. HUMIDITY-SERIES B

FIGURE 20



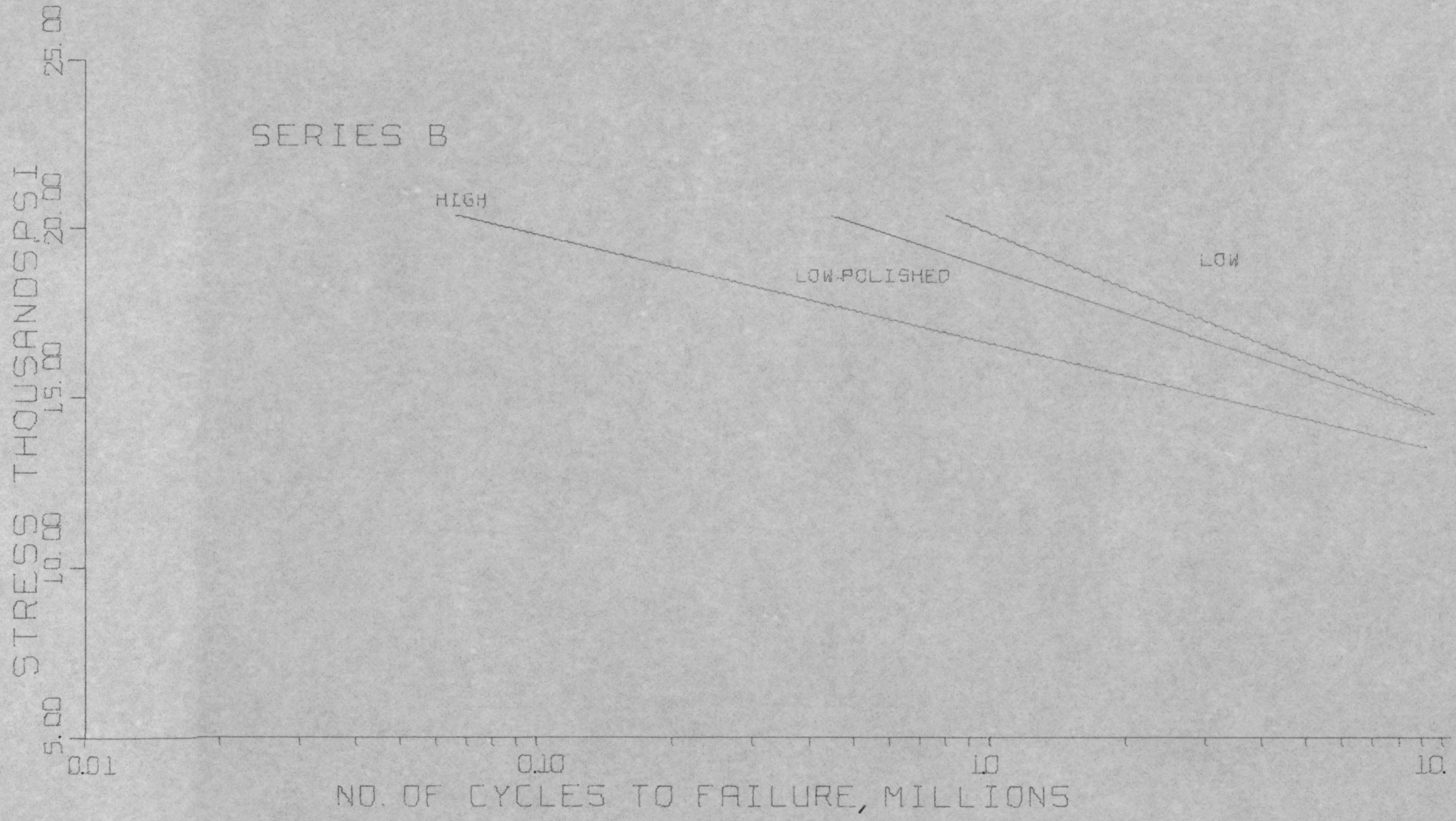
EXPERIMENTAL POINTS FOR S-N CURVE AT LOW REL. HUMIDITY-SERIES B

FIGURE 21



EXPERIMENTAL POINTS FOR S-N CURVE AT LOW REL. HUMIDITY -POLISHED

FIGURE 22



S-N CURVES FOR SERIES B AT HIGH AND LOW REL. HUMIDITIES

FIGURE 23

TABLE 5 EXPERIMENTAL DATA - SERIES B

Stress Level, lb/in.^{2}	Cycles to Failure, 10^3 Cycles		
	LOT 1	LOT 2	LOT 3
20000	315	1008	349
	1598	1340	409
	895	3139	1556
	157*	362	834
	112	3440	522
	212	943*	549
17000	420	6556	2167
	968	6159	7230
	275	1464	2521
	730	2078	4751
	80	4496	6268
	292	4334	9337
13000	5527	10000	6169
	1330	10000	5687
	10000	10000**	7252
	2810	6691	10000
	2707	10000	10000
	6258	6938*	2009

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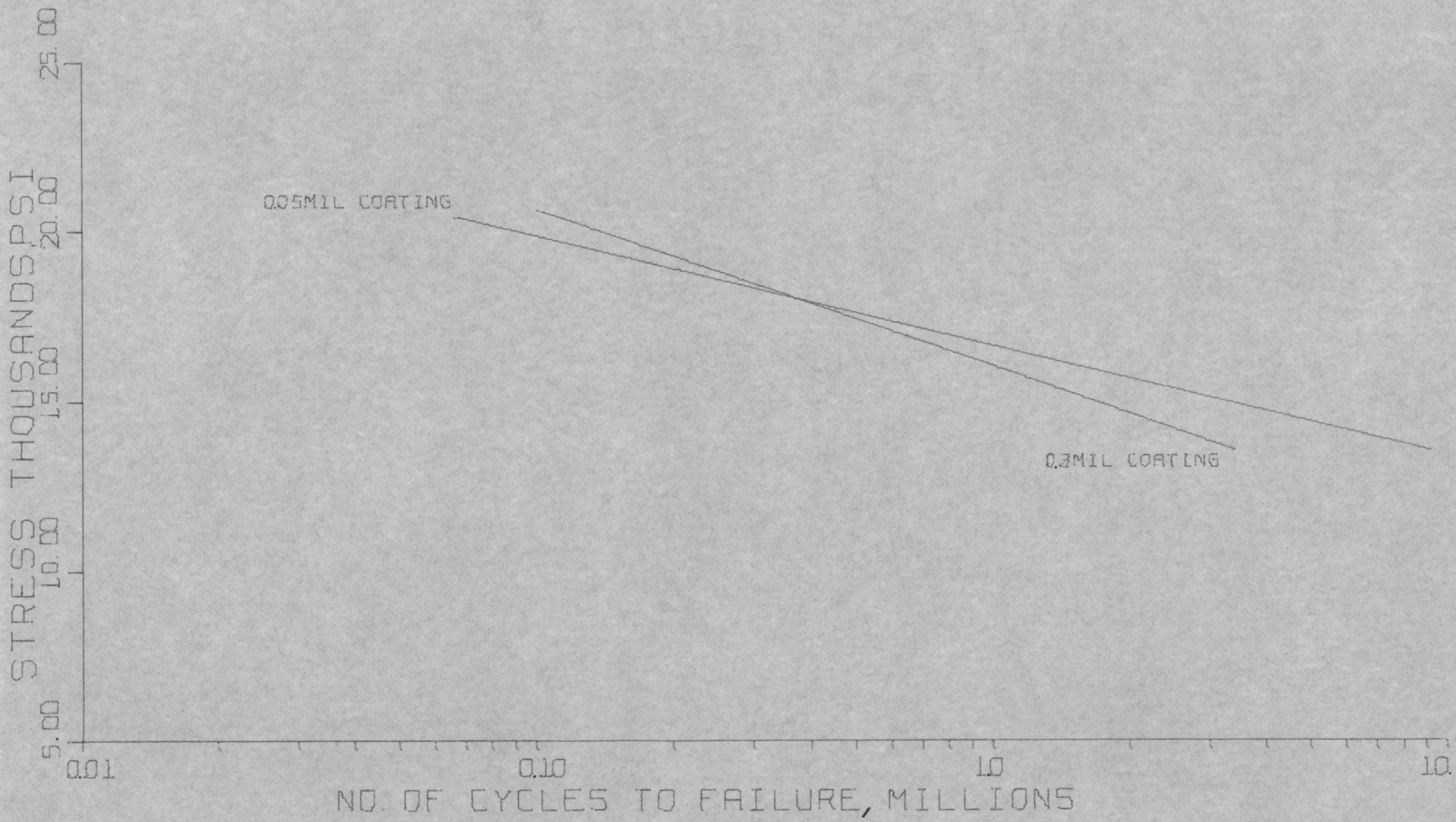
* Humidity went over desired humidity less than 3%.

** Machine running without torquing specimen for approximately 1 million stress cycles.

Results of Fatigue Tests on Coating Thicknesses

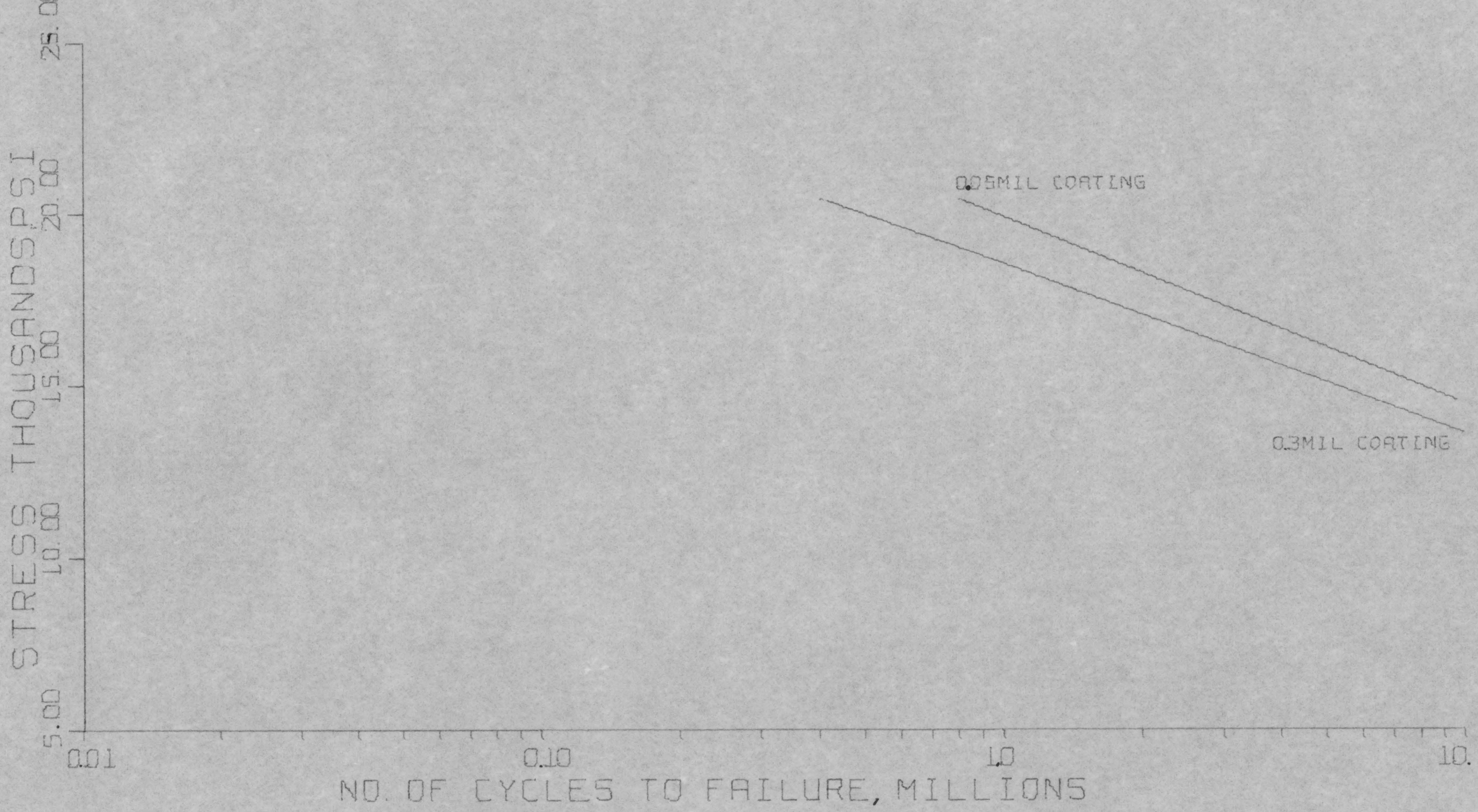
Figure 24 shows two curves for data taken at high relative humidity with anodized coatings of 0.3 and 0.05 mils. Figure 25 is also the same two thicknesses at low relative humidity. Analyses of variances performed at the 1% significance level on the above two groups of data (data taken from tests run at 0.3 and 0.05 mils at the high and low relative humidity levels) showed that the change in coating thickness did not have an effect on fatigue life of the specimens (Tables D-5 and D-6 of Appendix D).

Straight line curves fitted to the data of tests conducted by a previous investigator (1) on uncoated specimens of the same dimensions and material are given in Fig. 26 or Fig. 27 for specimens run at high or low relative humidity, respectively. A corresponding 0.05 mil coating curve for high or low humidity for anodized coated specimens obtained by the present investigator (Series B, Lots 1 or Series B, Lot 2) is also plotted in each of the above figures according to the humidity level in which the specimens were tested. The above allowed for comparison between specimens tested in the unanodized (uncoated) and anodized (coated) condition for the high and low humidity condition. Figure 26 shows the comparison curves for uncoated and coated specimens at high relative humidity and Fig. 27 shows the same for low relative humidity. An analysis of variance performed on the data at the 1% significance level in Fig. 26 showed the coating thickness not to have an effect (Table D-7, Appendix D). Only 16 pieces of data (the first five pieces of data obtained at the highest stress condition,



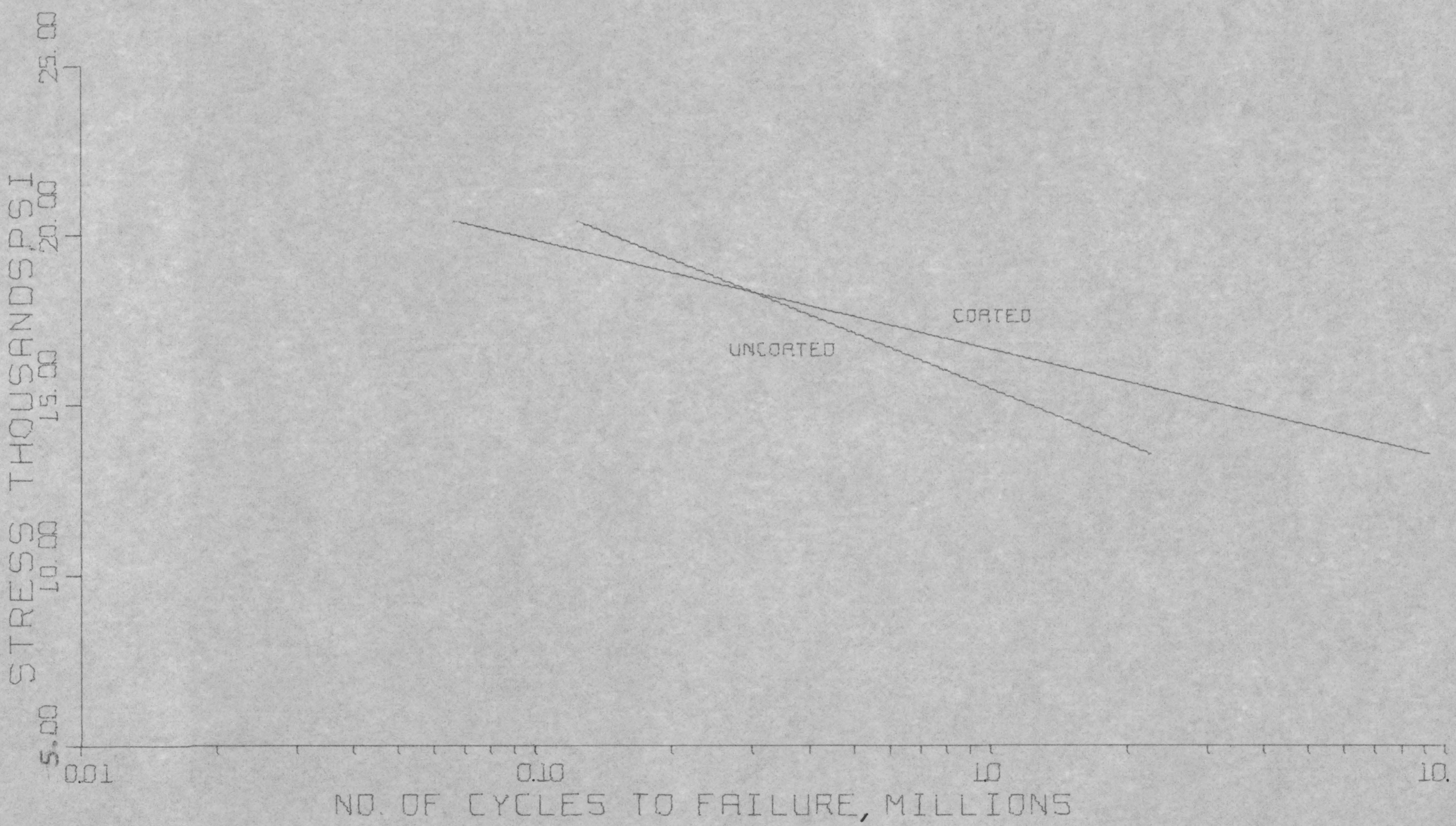
S-N CURVES FOR THICKNESS EFFECT AT HIGH REL. HUMIDITY

FIGURE 24



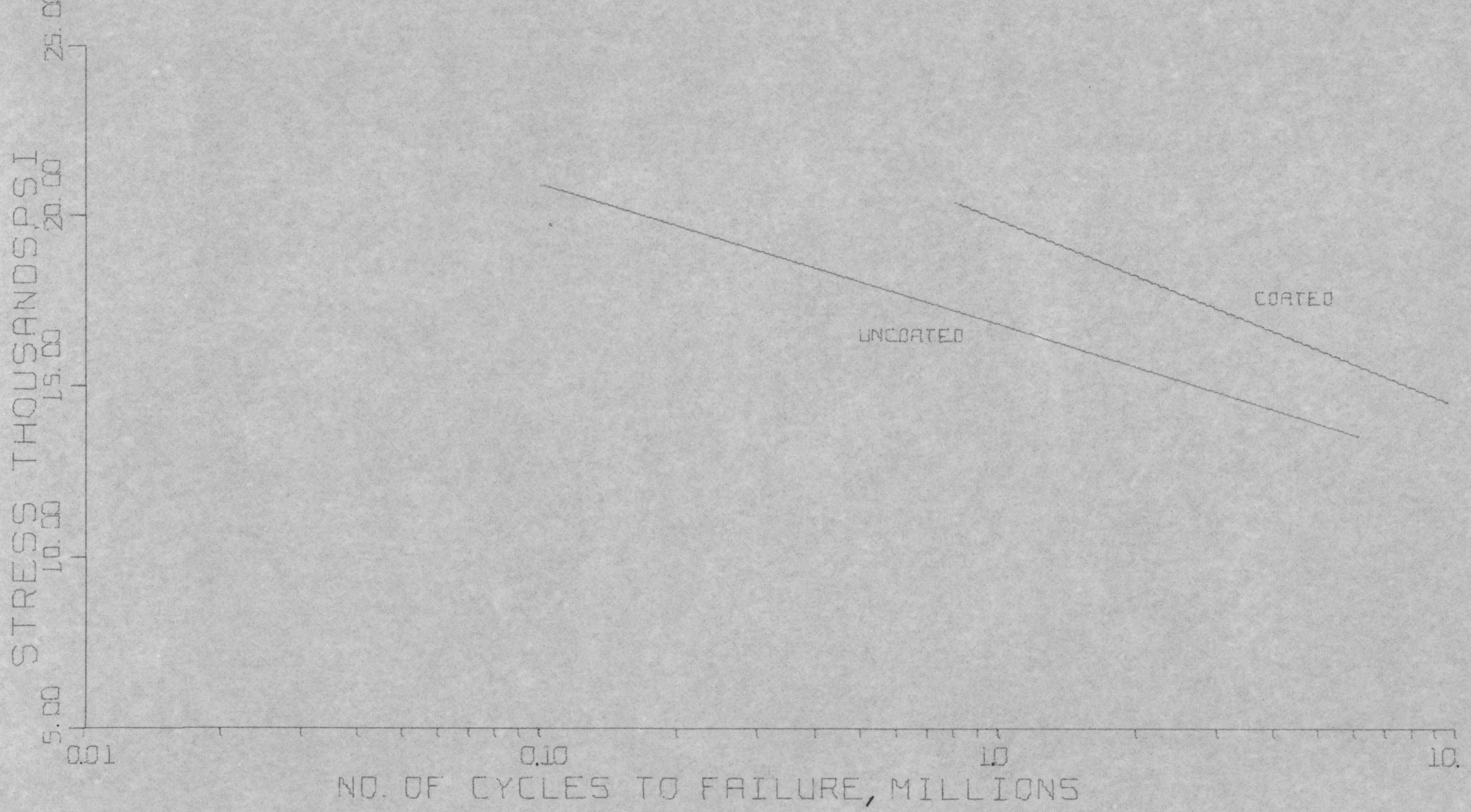
S-N CURVES FOR THICKNESS EFFECT AT LOW REL. HUMIDITY

FIGURE 25



S-N CURVES FOR UNCOATED AND COATED SPECIMENS AT HIGH REL. HUMIDITY

FIGURE 26



S-N CURVES FOR UNCOATED AND COATED SPECIMENS AT LOW REL. HUMIDITY

FIGURE 27

the first five pieces of data obtained at the medium stress condition, and all pieces of data obtained at the lowest stress conditon) were used out of Series B, Lot 1 so as to have a balance design in the analysis of variance when comparing results with (1). This means of analysis for this data was thought appropriate since (1) tested only 16 specimens at the high humidity condition in the same order as already mentioned. Increases in fatigue lives of the specimens in going from uncoated to anodized 0.05 mil coated specimens are 514% and 174% at the highest and lowest stresses, respectively for the low humidity level. An analysis of variance performed on the data of these two curves showed a coating effect (Table D-8, Appendix D) at a level of significance of 1%. The reduced section minimum diameter was 0.362 inches in the uncoated specimens case. This was 2 mils greater than the reduced diameter in this investigation. The anodized coating effect is considered to be the only influencing factor for the increase in the specimens lives. Also, the change in stress applied to the specimens is of the order of 0.55% because of the small variations of the diameters in the two investigations.

Results of Scatter in Data

A measure of the scatter was obtained by taking the variance of each of the cells in the experiment and performing a F_{max} test (see Appendix C for description of this test). The variances were calculated with all the life data in logarithmic form. The F_{max} test was performed at a given stress level when testing a variable's effect on the scatter. The effects of relative humidity, thickness, polishing, and coating showed the variances to be equal in this experiment. The only case where the variance was observed to be different occurred when the present investigator compared results with another investigator (1) at the high stress level (see Table E-6, Appendix E).

The cell variances and F_{max} tests are given in Table 6 and Tables E-1 through E-6 in Appendix E, respectively.

TABLE 6. CELL VARIANCES

SERIES	LOT	STRESS lb/in.^{2}		
		13000	17000	20000
A	1	0.028	0.052	0.083
A	2	0.103	0.088	0.246
A	3	0.034	0.045	0.104
B	1	0.099	0.146	0.203
B	2	0.007	0.069	0.133
B	3	0.066	0.065	0.056
UNCOATED (1)	HIGH	0.069	0.042	0.038
"	LOW	0.058	0.103	0.003

DISCUSSION OF RESULTS

Only indirect inferences can be made when comparing this investigation with others who have done investigations with aluminum. In many cases the other investigators did not study torsional load or the alloy used in this investigation.

A drier environment in this investigation tended to extend the fatigue life of the anodized aluminum. Those who did studies on aluminum in torsion (1), bending (9), and tension [(11), (12), (14), and (15)], found their specimens to have increases in fatigue lives at lower humidity levels. All of the above concluded that the amount of water vapor in the air decreased the fatigue lives of the specimens in a wet environment for their fatigue tests. The results of the present investigation on the fatigue lives of 0.3 mil coated specimens tested at the high and medium relative humidity levels are not significantly different from one another. This phenomena seems to infer that there may be some threshold relative humidity level at which a difference is established for the fatigued specimens since there is a significant difference between specimens run at the high and low relative humidity levels. A significant difference between the fatigue lives of the 0.05 anodized specimens at high and low humidity was also found. Hartman (12) noted a slight difference in the fatigue lives of his specimens between relative humidities of 30 and 100%. The above result at medium and high humidity in this investigation is similar to the one obtained by Frankel, Bennett, and Holshouser (10), where it was found that the fatigue lives of their specimens on a 6061-T6 aluminum alloy did not change from tests in air and water immersed environments. It is also found in the present investigation that there is no significant difference between the fatigue lives of anodized specimens with different oxide coating thicknesses (0.05 and 0.3 mils) grown on their surfaces tested at the high and low relative humidity levels. Thus the two oxide coating thicknesses are just as effective in protecting the metal and maintaining equivalent fatigue lives for the specimens at 86-91% and 20-25% relative humidity. Results of the fatigue tests on uncoated and coated specimens at high humidity showed that the specimens' fatigue lives were not significantly different from one another. The specimens' fatigue lives in the uncoated condition at low humidity was significantly different from coated specimens. These results again support the phenomena already mentioned above [i.e., a threshold relative humidity level exists at which the oxide coating becomes least impervious to a wetted environment and offers no protection to the underlying substrate as it does in drier environments (20-25% relative humidity)].

Broom and Nicholson (11) anodized specimens of a Duralumin alloy and a D.T.D. 683 alloy showed no significant difference from fatigue tests of the unanodized specimens in their investigation. It is probable that they did not see a difference in the specimen fatigue lives because they were running tests at too high a humidity level, and thus experienced the same type of results as the uncoated and coated specimensdid at high humidity as in this investigation. The investigators did not report the humidity environments in which they tested their specimens and thus the above conclusion is speculative only. The same type of consequence mentioned above could have occurred with (10) for them not to get any significant differences in the fatigue lives of their specimens when tested in air and water immersed environments. The maximizing effect of sealing the coating for better corrosion resistance is also lessened at the high humidity levels (55-60% and 86-91% relative humidities). Even though the comparison between uncoated and coated specimens were made between the 0.05 mil and (1) data only, it is also valid for the 0.3 mil coated specimens since they were determined not to be significantly different from the 0.05 mil specimens at high and low relative humidity levels. The phenomena mentioned above infer that anodize parts made from this aluminum alloy for protection against the corrosive environment may only be helpful in a moderately low controlled humidity environment when cyclic loading is present. If the anodized parts made from the alloy are fatigued above a certain threshold humidity level, no increases in the fatigue lives of the parts should be expected.

In agreement with (1), polishing had no effect on the fatigue lives of the specimens in this investigation.

The scatter in this investigation did not vary for the various changes of relative humidity, coating thickness, surface finish (polishing), and coating for the stress levels tested. There was only one instance where the scatter was concluded to be different. This occurred when comparing the 0.05 mil anodized specimens with uncoated ones (1) at the low humidity level for the high stress condition (Table E-6, Appendix E). Comparison of the scatter at the other two stress levels in the above table showed the scatter to be the same between the uncoated and coated specimens. The specimens uncoated and coated at the high humidity level (Table E-5, Appendix E) did not show the scatter to be different at the various stress levels (the results in this case may also be influenced by the phenomena mentioned above).

CONCLUSIONS AND RECOMMENDATIONS

Based on this investigation, the following conclusions and recommendations are:

1. Water vapor did reduce the fatigue lives of the anodic coated aluminum specimens (for both 0.3 and 0.05 mil coatings) when going from fatigue tests at a low relative humidity level (20-25%) to one of higher humidity (86-91%).
2. The fatigue lives of the 0.3 mil specimens tested in a relative humidity environment of 55-60% had no significant difference from specimens tested at 86-91% relative humidity.
3. The coated specimens did not show a significant increase in fatigue life over uncoated specimens at high relative humidity (86-91%), but did at low relative humidity (20-25%). This phenomena and its effects warrants more investigation so as to establish at approximately what level of relative humidity causes a significant increase in the fatigue lives of the anodized specimens.
4. No significant differences occurred between the two thicknesses of oxide films tested (0.05 and 0.3 mil) at high and low relative humidity.
5. There is no difference between specimens that are fatigued in the "as machined" and as "machined and polished" conditions.

6. The scatter experienced in the fatigue test is not caused by changes in relative humidities, coating thicknesses, specimen surface finishes (polishing) or coating (anodizing).

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APPENDIX A *Steps of the Anodization Process

The following steps were carried out according to military specifications MIL - A - 8625 Type II for the thickness of coating desired.

(1) Specimens were racked on titanium racks.

(2) All specimens are vapor degreased with trichloroethane (Chlorothene). The specimens were placed over the boiling solution (168°F) for 5 minutes.

(3) Rinse.

(4) The specimens were etched in a sodium hydroxide type MIL - Etch for 2 minutes at 60°C (140°F). 6 oz./gal. (Wyandottle Chemical Company).

(5) Rinse.

(6) Specimens were deoxidized with chromic sulfuric type Isoprop. 188. (Allied Research Product).

(7) Rinse.

(8) All specimens were anodized with 15% by wt. of technical grade surfuric acid at 25°C (77°F) and 12 amps/ft^2. The agitation was vigorous for the whole time of immersion. The voltage was 17 to 18 volts.

(9) Rinse.

(10) All specimens were then sealed by boiling in deionized water for 30 minutes.

(11) Specimens bagged in plastic bags.

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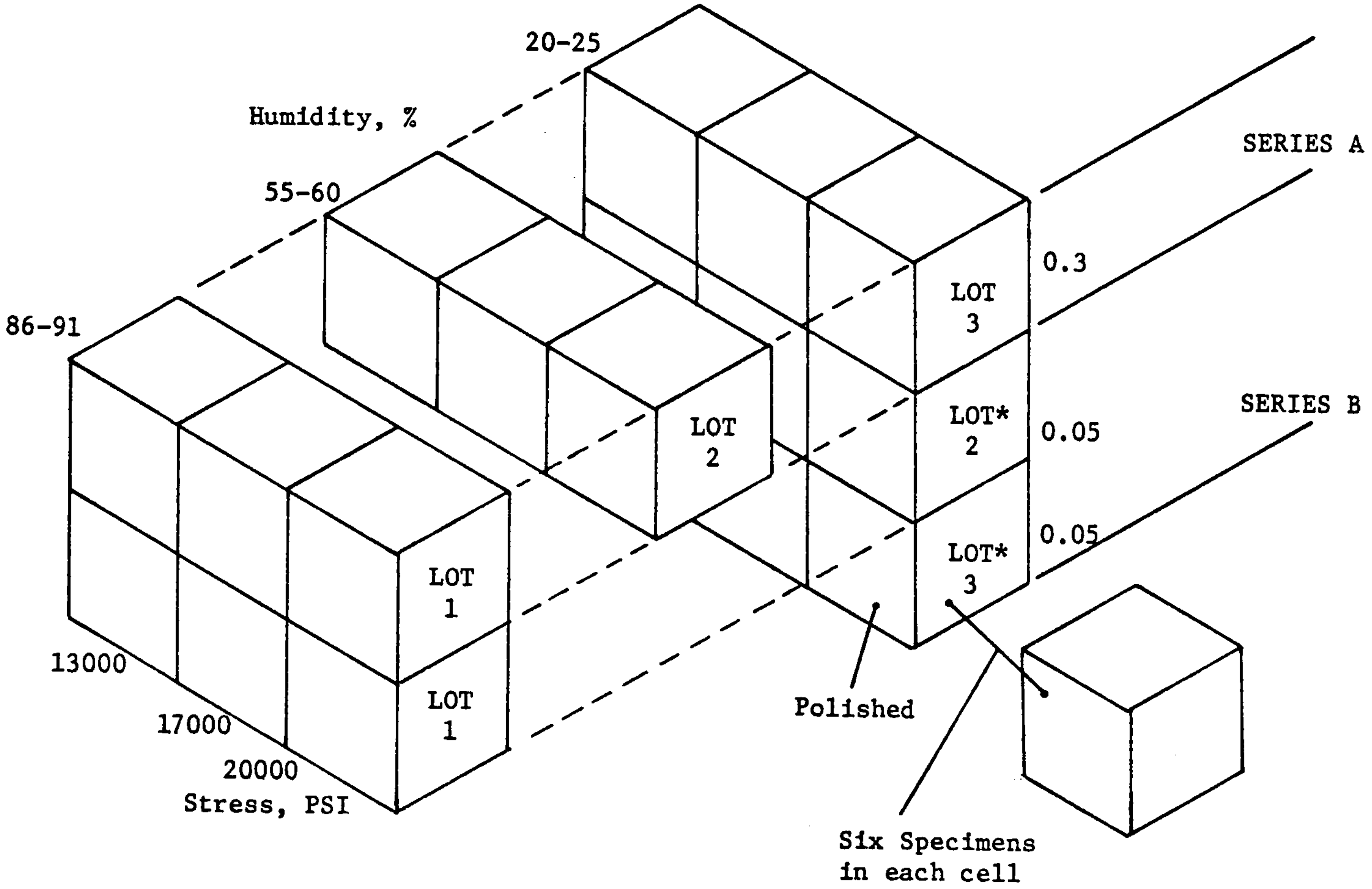
*The steps as listed above are typical for any conventional sulfuric acid anodizing process.

APPENDIX B

EXPERIMENTAL DESIGN



* Series B LOTS 2 and 3 were from a different heats of the same material.

APPENDIX C Statistical Analysis of Results

The following steps were applied to the data which yielded the various conclusions cited in the results.

(1) All the data for the number of cycles to failure in Series A and Series B were converted to their logarithmic values. Fatigue data have been found to follow a normal distribution once the above transformation is made (19).

(2) Several analyses of variances were performed to test the various variable effects according to the groupings of data given below:

(a) Series A [(Lots 1 and 3) and (Lots 1 and 2)].

(b) High Humidity (0.3 mil and 0.05 mil in the "as machined" condition).

(c) Low Humidity (0.3 mil and 0.05 mil in the "as machined" condition).

(d) Low Humidity (0.05 mils in the "as machined" and "as machined and polished" conditions).

(e) Series B (Lots 1 and 2).

(f) Coated and uncoated (at high and low humidity).

The experimental design of each of the above data groupings is of the randomized block design (20) since the specimens were randomized according to treatments (stress) within the various blocks [humidity, thickness, coating, and surface finish (polishing)].

In order to determine an effect of a variable, an F statistic was computed based upon the experimental design. The F statistic as calculated from the data is a ratio of a mean square due to some variable effect over some residual (error) mean square. A null hypothesis is assumed to test the effect under question (i.e., there is no variable effect). In order to determine the truth in the assumed hypothesis, a critical F-value is chosen based on the level of significance to test the null hypothesis. If the calculated value of F is beyond the critical value, reject the null hypothesis and accept the alternative (i.e., the variable has an effect). All tests were performed at a significance level of 1% unless otherwise indicated.

The model used for this investigation is that as follows:

$$x_{ij} = u + B_i + T_j + e_{ij}$$

or

$$x_{ij} = \bar{x}_{..} + (\bar{x}_{i.} - \bar{x}_{..}) + (\bar{x}_{.j} - \bar{x}_{..}) + (x_{ij} - \bar{x}_{i.} - \bar{x}_{.j} + \bar{x}_{..})$$

where

x_{ij} = observed value

u = population mean of all observations

B_j = represents the block effect (humidity, thickness, polishing, and coating)

T_j = represents the treatment effect (stress)

e_{ij} = represents the random error of the experiment

$\bar{x}_{..}$ = sample mean of all observations

$\bar{x}_{i.}$ = i^{th} sample block mean

$\bar{x}_{.j}$ = j^{th} sample treatment mean

$u_{i.}$ = i^{th} true block mean

$u_{.j}$ = j^{th} true treatment mean

The ANOVA (20) for this type experiment is as given in Table C-1 of this appendix. The null hypotheses then to be tested are whether the means of all block effects are equal (i.e., $u_{1.} = u_{2.} = u_{3.}$, etc.) or whether the means of all treatment effects are equal (i.e., $u_{.1} = u_{.2}$, etc.). The F-value for these tests (given in general terms in Table C-1) were then used against the critical value at the 1% significance level to test differences between means.

(3) A linear regression line of the form STRESS = A + B LOG (CYCLES) was fitted to the data using the method of least squares (the number of cycles is in kilocycles). The coefficients A and B are given in Table C-2 of this appendix for the various lots of Series A and Series B. The S-N curve for fatigue data of most metals follow a straight line above the endurance limit (21). An equation of the above type was fitted as it is the form that plots straight on semilogarithmic graphs. These graphs are the usual ones used in representing fatigue data. A correlation coefficient (r) is also given for each curve in Table C-2 of this appendix. The magnitude of this coefficient measures how much association the dependent variable (stress) has with the independent variable (cycles). A value of r equal $\pm$ 1 means that the independent variable is perfectly correlated with the dependent variable (i.e., the regression line explains the data perfectly). An r value of 0 means that the independent variable has no correlation with the dependent variable.

(4) A F_{max} test (22) is used on the data to determine whether the variances between cells are equal. This analysis was done for the

APPENDIX C

TABLE C-1 ANALYSIS OF VARIANCE - RANDOMIZE BLOCK DESIGN

Source	Degrees of Freedom	Sums of Squares	Mean Square	F_{cal}
Blocks (B_i)	$n - 1$	$\sum\limits_{ij}^{nk}\sum (\bar{x}_{i.} - \bar{x}_{..})^2$	$\frac{SS_{blocks}}{n - 1}$	$\frac{SS_{blocks}\ (k - 1)}{SS_{error}}$
Treatments (T_j)	$k - 1$	$\sum\limits_{ij}^{nk}\sum (\bar{x}_{.j} - \bar{x}_{..})^2$	$\frac{SS_{treat}}{k - 1}$	$\frac{SS_{treat}\ (n - 1)}{SS_{error}}$
Error (e_{ij})	$(n-1)(k-1)$	$\sum\limits_{ij}^{nk}\sum (x_{ij} - \bar{x}_{i.} - \bar{x}_{.j} - \bar{x}_{..})^2$	$\frac{SS_{error}}{(n-1)(k-1)}$	
Total	$nk - 1$	$\sum\limits_{ij}^{nk}\sum (x_{ij} - \bar{x}_{..})^2$		

APPENDIX C

TABLE C-2 COEFFICIENTS FOR S-N CURVES

SERIES	LOT	COEFFICIENTS A	COEFFICIENTS B	CORRELATION COEFFICIENT (r)
A	1	29890	-4608	-0.923
A	2	27315	-3509	-0.802
A	3	33467	-4960	-0.894
B	1	26439	-3245	-0.708
B	2	36345	-5447	-0.813
B	3	32477	-4515	-0.812
UNCOATED (1)	HIGH	32041	-5506	-0.893
"	LOW	29246	-4129	-0.923

case where all samples are of uniform size. The number of cells under study determine the number of combinations of ratios that can be generated to give various F-values. The maximum F-value obtained from the ratio of variances is used in the test against a critical F_{max}-value to determine whether there is a difference in the variances. The hypothesis is that no differences exist between the variances of the variable under question. If the maximum calculated F-value is less than the critical F_{max} value, accept the hypothesis (i.e., all the variances are equal). If the calculated maximum F-value is greater than the critical F_{max}-value reject the hypothesis. The critical F_{max}-value was set at the 1% significance level for all analyses performed with the use of this test. The variable effects studied using this type test were humidity, thickness, polishing and coating at various stress levels. This was done in order to get some indication of the variables effects on the scatter in the results. The k given in Appendices E correspond to the number of cells being used in a test at a given stress level. The n is the number of specimens in a cell minus one (degrees of freedom).

APPENDIX D

TABLE D-1 ANALYSIS OF VARIANCE - SERIES A (LOTS 1 AND 3)

SOURCE	SUMS OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE	F_{cal}	F_{crit}
Humidity	2.4686	1	2.4686	46.23	8.40
Stress	9.2346	17	0.5432	10.17	3.31
Error	0.9075	17	0.0534		
Total	12.6107	35			

Results of Analysis of Variance:

Steps in hypothesis testing	Variables of Interest Humidity	Stress
Null Hypothesis	Humidity has no effect.	Stress has no effect.
Alternate	Humidity has an effect.	Stress has an effect.
Test	$F_{cal} > F_{crit.}$	$F_{cal} > F_{crit.}$
Conclusion	Accept Alternate.	Accept Alternate.

APPENDIX D

TABLE D-2 ANALYSIS OF VARIANCE - SERIES A (LOTS 1 AND 2)

SOURCE	SUMS OF SQUARE	DEGREES OF FREEDOM	MEAN SQUARE	F_{cal}	F_{crit}
Humidity	0.1730	1	0.1730	1.25	8.40
Stress	10.5474	17	0.6204	4.49	3.31
Error	2.3477	17	0.1381		
Total	13.0681	35			

Results of Analysis of Variance:

Steps in hypothesis testing	Humidity	Stress
Null Hypothesis	Humidity has no effect.	Stress has no effect.
Alternate	Humidity has an effect.	Stress has an effect.
Test	$F_{cal} < F_{crit}$.	$F_{cal} > F_{crit}$.
Conclusion	Accept null hypothesis.	Accept Alternate.

APPENDIX D

TABLE D-3 ANALYSIS OF VARIANCE - SERIES B (LOTS 1 AND 2)

SOURCE	SUMS OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE	F_{cal}	F_{crit}
Humidity	3.8012	1	3.8012	30.29	8.40
Stress	7.6204	17	0.4483	3.57	3.31
Error	2.1331	17	0.1255		
Total	13.5547	35			

Results of Analysis of Variance:

Steps in hypothesis testing	Variables of Interest	
	Humidity	Stress
Null Hypothesis	Humidity has no effect.	Stress has no effect.
Alternate	Humidity has an effect.	Stress has an effect.
Test	$F_{cal} > F_{crit.}$	$F_{cal} > F_{crit.}$
Conclusion	Accept Alternate.	Accept Alternate.

APPENDIX D

TABLE D-4 ANALYSIS OF VARIANCE - POLISHING EFFECT (LOTS 2 AND 3 SERIES B)

SOURCE	SUMS OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE	F_{cal}	F_{crit}
Polishing	0.1431	1	0.1431	2.28	8.40
Stress	6.5604	17	0.3859	6.14	3.31
Error	1.0685	17	3.5923		
Total	7.7720	35			

Results of Analysis of Variance:

Steps in hypothesis testing	Variables of Interest	
	Polishing	Stress
Null Hypothesis	Polishing has no effect.	Stress has no effect.
Alternate	Polishing has an effect.	Stress has an effect.
Test	$F_{cal} < F_{crit.}$	$F_{cal} > F_{crit.}$
Conclusion	Accept null hypothesis.	Accept alternate.

APPENDIX D

TABLE D-5 ANALYSIS OF VARIANCE FOR HIGH RELATIVE HUMIDITY (LOTS 1 OF SERIES A AND B)

SOURCE	SUMS OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE	F_{cal}	F_{crit}
Thickness	0.1026	1	0.1026	0.80	8.40
Stress	10.0775	17	0.5928	4.64	3.31
Error	2.1732	17	0.1278		
Total	12.3533	35			

Results of Analysis of Variance:

Steps in hypothesis testing	Variables of Interest	
	Thickness	Stress
Null Hypothesis	Thickness has no effect.	Stress has no effect.
Alternate	Thickness has an effect.	Stress has an effect.
Test	$F_{cal} < F_{crit}$.	$F_{cal} > F_{crit}$.
Conclusion	Accept null hypothesis.	Accept alternate.

APPENDIX D

TABLE D-6 ANALYSIS OF VARIANCE FOR LOW RELATIVE HUMIDITY (SERIES A LOT 3 - SERIES B LOT 2)

SOURCE	SUMS OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE	F_{cal}	F_{crit}
Thickness	0.4884	1	0.4884	5.53	8.40
Stress	6.1444	17	0.3614	4.09	3.31
Error	1.5005	17	0.0883		
Total	8.1333	35			

Results of Analysis of Variance:

Steps in hypothesis testing	Variables of Interest	
	Thickness	Stress
Null hypothesis	Thickness has no effect.	Stress has no effect.
Alternate	Thickness has an effect.	Stress has an effect.
Test	$F_{cal} < F_{crit.}$	$F_{cal} > F_{crit.}$
Conclusion	Accept null hypothesis.	Accept alternate.

APPENDIX D

TABLE D-7 ANALYSIS OF VARIANCE-COATING EFFECT AT HIGH HUMIDITY
[Wilson's (1) high humidity and Series B, Lot 1]

SOURCE	SUMS OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE	F_{cal}	F_{crit}
Counting	0.2496	1	0.2496	1.90	8.68
Stress	7.4220	15	0.4948	3.77	3.52
Error	1.9701	15	0.1313		
Total	9.6417	31			

Results of Analysis of Variance:

Steps in hypothesis testing	Variables of Interest	
	Coating	Stress
Null hypothesis	The coating has no effect.	Stress has no effect.
Alternate	The coating has an effect.	Stress has an effect.
Test	$F_{cal} < F_{crit.}$	$F_{cal} > F_{crit.}$
Conclusion	Accept null hypothesis.	Accept alternate.

APPENDIX D

TABLE D-8

ANALYSIS OF VARIANCE-COATING EFFECT AT LOW HUMIDITY
[Wilson's (1) low humidity and Series B, Lot 2]

SOURCE	SUMS OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE	F_{cal}	F_{crit}
Coating	2.8883	1	2.8883	27.75	8.40
Stress	8.2356	17	0.4844	4.65	3.31
Error	1.7703	17	0.1041		
Total	12.8942	35			

Results of Analysis of Variance:

Steps in hypothesis testing	Variables of Interest	
	Coating	Stress
Null hypothesis	The coating has no effect.	Stress has no effect.
Alternate	The coating has an effect.	Stress has an effect.
Test	$F_{cal} > F_{crit.}$	$F_{cal} > F_{crit.}$
Conclusion	Accept alternate.	Accept alternate.

APPENDIX E

HUMIDITY EFFECTS WITHIN SERIES AT GIVEN STRESS LEVELS

TABLE E-1

		Calculated F-values							
	Stress lb/in.^{2}	*S_1^2/S_2^2	S_1^2/S_3^2	S_2^2/S_3^2	S_2^2/S_1^2	S_3^2/S_1^2	S_3^2/S_2^2	k/n	F_{max}
Series A	13000	0.272	0.824	3.029	3.679	1.214	0.330	3/5	22.0
	17000	0.591	1.156	1.956	1.692	0.865	0.511		
	20000	0.337	0.798	2.365	2.964	1.253	0.423		
Series B	13000	14.143			0.071			2/5	14.9
	17000	2.116			0.473				
	20000	1.526			0.655				

Results of above tests:

Steps in hypothesis testing	Variable of Interest Humidity
Hypothesis	All the variances in the above analysis at the given stresses are equal.
Test	All F-values < F_{max} .
Conclusion	Accept hypothesis.

---

*S_1^2 - Read to mean the sample variance of lot 1 at given stress level.

APPENDIX E THICKNESS EFFECTS AT HIGH RELATIVE HUMIDITY FOR GIVEN STRESS LEVELS

TABLE E-2

Stress lb/in.^{2}	Calculated F-values		k/n	F_{max}
	$^*S^2_{A1}/S^2_{B1}$	S^2_{B1}/S^2_{A1}		
13000	0.283	3.536	2/5	14.9
17000	0.356	2.808		
20000	0.409	2.446		

Results of above tests:

Steps in hypothesis testing	Variable of Interest
	Thickness
Hypothesis	All the variances in the above analysis at the given stresses are equal.
Test	All F-values < F_{max} .
Conclusion	Accept hypothesis.

$^*S^2_{1A}$ - Sample variance of lot 1, series A at given stress level.

APPENDIX E THICKNESS EFFECTS AT LOW RELATIVE HUMIDITY FOR GIVEN STRESS LEVELS

TABLE E-3

Stress $lb/in.^2$	Calculated F-values			
	$^*S_{A3}^2/S_{B2}^2$	S_{B2}^2/S_{A3}^2	k/n	F_{max}
13000	4.857	0.206	2/5	14.9
17000	0.652	1.533		
20000	0.782	1.279		

Results of above tests:

Steps in hypothesis testing	Variable of Interest Thickness
Hypothesis	All the variances in the above analysis at the given stresses are equal.
Test	All F values < F_{max} .
Conclusion	Accept hypothesis.

$^*S_{A3}^2$ - Sample variance of lot 3, series A at given stress level.

APPENDIX E
TABLE E-4 POLISHING EFFECTS AT LOW RELATIVE HUMIDITY FOR GIVEN STRESS LEVELS (SERIES B, LOTS 2 AND 3)

Stress lb/in^2	Calculated F-values		k/n	F_{max}
	$*S^2_{B2}/S^2_{B3}$	S^2_{B3}/S^2_{B2}		
13000	0.106	9.434	2/5	14.9
17000	1.062	0.942		
20000	2.375	0.421		

Results of above tests:

Steps in hypothesis testing	Variable of Interest
Hypothesis	All the variables in the above analysis at the given stresses are equal.
Test	All F-values < F_{max}
Conclusion	Accept hypothesis.

$*S^2_{B2}$ - Sample variance of lot 2, series B at given stress level.

APPENDIX E COATING EFFECTS AT GIVEN STRESS LEVELS FOR HIGH RELATIVE HUMIDITY

TABLE E-5

Stress lb/in^2	Calculated F-values			
	$^*S_c^2/S_u^2$	S_u^2/S_c^2	k/n	F_{max}
13000	1.435	0.697		
17000	3.476	0.288	2/5	14.9
20000	5.342	0.187		

Results of above test:

Steps in hypothesis testing	Coating
Hypothesis	All variances in the above analysis at the given stresses are equal.
Test	All F-values < F_{max}
Conclusion	Accept hypothesis

$^*S_c^2$ - Sample variance of coated specimens for a cell (Series B, Lot 1).

S_u^2 - Sample variance of uncoated specimens for a cell [Wilson (1)].

APPENDIX E COATING EFFECTS AT THE GIVEN STRESS LEVELS FOR LOW RELATIVE HUMIDITY

TABLE E-6

Stress lb/in^2	Calculated F-values		k/n	F_{max}
	$*S_c^2/S_u^2$	S_u^2/S_c^2		
13000	0.121	8.286	2/5	14.9
17000	0.670	1.493		
20000	44.333	0.023		

Results of above test:

Steps in hypothesis testing	Variable of Interest
	Coating
Hypothesis	All the variances in the above analysis at the given stresses are equal.
Test	All F-values < F_{max} except at high stress.
Conclusion	Accept hypothesis for all stress except at high stress.

$*S_c^2$ - Sample variance of coated specimens for a cell (Series B, Lot 2).

S_u^2 - Sample variance of uncoated specimens for a cell [Wilson (1)].

TEST EQUIPMENT

1. SONNTAG UNIVERSAL FATIGUE TESTING MACHINE (Model SF-01-U) Sonntag Scientific Corporation, Greenwich Conn.

   Use: Apply reverse torgue to specimen.

2. ELECTRIC HYGROMETER INDICATOR (Model No. 4-5170) American Instrument Company, Inc., Silver Spring, Maryland.

   Use: To indicate the relative humidity of the air flow into test chamber.

3. FISCHER AND PORTER FLOWMETER (Tube No. FP 1/4-20-G-5/81) Fisher and Porter Company, Hatboro, Pa.

   Use: To measure the flow of air into test chamber.

4. VAN-AIR FILTER (Model No. F5), Van Products Company, Erie, Pa.

   Use: Filter the laboratory air.

5. LEHIGH BLU-COLD COMMERCIAL REFRIGERATOR (Model AM 42F FY 12), Lehigh Manufacturing Company, Lancaster, Pa.

   Use: To refrigerate the anti-freeze that cooled the distilled water in bubble tower.

6. BELL AND GOSSET CENTRIFUGAL PUMP (Model No. P 7-4) Bell and Gosset, Inc., Morton Grove, Illinois.

   Use: Circulate the anti-freeze in the bubble tower.

7. HEATING ELEMENT (Model No. D-230-U) Electro-Therm., Inc., Laurel, Maryland

   Use: To heat the circulating anti-freeze in the bubble tower.

8. CORNING EXPANDED SCALE pH METER (Model 10) Corning Scientific Instruments, Corning, New York.

   Use: To measure pH of air bubble distilled water.

9. FISHER RESEARCH pH METER (Model No. 320) Fisher Scientific Company, Pittsburg, Pa.

   Use: To measure pH of air bubble distilled water

10. SCANNING ELECTRON MICROSCOPE (Model No. AMR - 900)
    Advance Metals Research Corporation, Burlington, Mass.

    Use: To make photographs of the metal surface.

EFFECT OF RELATIVE HUMIDITY ON FATIGUE OF ANODIZED 2024-T351 ALUMINUM IN COMPLETELY REVERSED TORSION

by

Ernest F. Womack

(ABSTRACT)

A corrosion fatigue investigation was conducted on anodized 2024-T351 aluminum alloy in reversed torsion. The corrosive environment was provided by varying the relative humidity of air around the specimens during fatigue. The effect of water vapor on fatigue of aluminum (and alloys) have been previously established by other investigators. The aluminum oxide coating was grown on the metal substrate to two thicknesses (0.05 and 0.3 mils). One lot of specimens was polished to determine the effect of this type of surface finish. In all, a total of 108 specimens were run at various combinations of stress, relative humidity, coating thickness, surface finish (polishing), and coating. Six specimens were run at the stress levels of approximately 13,000, 17,000, and 20,000 lb/in^2 respectively. The three relative humidity ranges used were low (20-25%), medium (55-60%), and high (86-91%).

Results of the investigation showed the anodized specimens at low relative humidity to have an increase in fatigue life over specimens of the same type tested at high relative humidity. The increases in fatigue lives were 281% and 194% for the 0.3 mil specimens at the highest and lowest stress levels. Increases in fatigue lives of 1112%

and 338% were also found at the highest and medium stress levels for the 0.05 mil anodized specimens. It was found that the 0.3 mil anodized specimens in a relative humidity environment of 55-60% had no significant difference from those tested in an environment of 86-91%.

The fatigue lives of the anodized specimens compared to results of unanodized specimens of a previous investigator at the low humidity range gave fatigue life increases of 514% and 174% at the high and low stress levels. Another comparison for anodized and unanodized specimens at high humidity showed no differences between the specimens fatigue lives. It is suggested that some phenomena is responsible for these unexpected results. That is, a humidity threshold appears to be necessary to penetrate the anodized barrier so that there is no protection of the substrate after reaching this threshold.

The coating thickness of the oxide and the polishing of the specimens before anodizing showed no effect. The scatter in the investigation did not change with humidity, coating thickness, polishing or coating.