NASA CR-

141696

DEVELOPMENT OF FRACTURE MECHANICS DATA FOR TWO HYDRAZINE APU TURBINE WHEEL MATERIALS

(NASA-CR-141696) DEVELOPMENT OF FRACTURE MECHANICS DATA FOR TWO HYDRAZINE APU TURBINE WHEEL MATERIALS Final Report, Jul. 1973 - Dec. 1974 (AiResearch Mfg. Co., Torrance, Calif.) 76 p HC $4.75 CSCL 20K G3/39

N75-18622

Unclas 12479

George Curbishley
AiResearch Manufacturing Company of California
2525 W. 190th St.
Torrance, Calif. 90509

NAS9-13484

February 1975
Final Report for Period July 1973 – December 1974

Prepared for
LYNDON B. JOHNSON SPACE CENTER
Houston, Texas 77058

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle Development of Fracture Mechanics Data for Two Hydrazine APU Turbine Wheel Materials		5. Report Date 12 February 1975
		6. Performing Organization Code
7. Author(s) G. Curbishley		8. Performing Organization Report No. 74-11081
9. Performing Organization Name and Address AiResearch Manufacturing Company of California 2525 West 190th Street Torrance, Calif. 90509		10. Work Unit No.
		11. Contract or Grant No. NAS9-13484
12. Sponsoring Agency Name and Address Lyndon B. Johnson Space Center Houston, Texas 77058		13. Type of Report and Period Covered Final July 1973-December 1974
		14. Sponsoring Agency Code
15. Supplementary Notes		

16. **Abstract**

The effects of high temperature, high pressure ammonia was measured on the fracture mechanics and fatigue properties of Astroloy and Rene' 41 turbine wheel materials. Also, the influence of protective coatings on these properties was investigated.

Specimens of forged bar stock were subjected to LCF and HCF tests at 950 K (1250°F) and 3.4 MN/m^2 (500 psig) pressure, in ammonia containing about 1.5 percent H_2O. Aluminized samples (Chromizing Company's Al-870) and gold plated test bars were compared with uncoated specimens. Comparison tests were also run in air at 950 K (1250°F), but at ambient pressures. K_{IE} and K_{TH} were determined on surface flawed specimens in both air and ammonia in both the uncoated and gold plated conditions.

Gold plated specimens exhibited better properties than uncoated samples, and aluminized test bars generally had lower properties. The fatigue properties of specimens tested in ammonia were higher than those tested in air, yet the K_{TH} values of ammonia tested samples were lower than those tested in air. However, insufficient specimens were tested to develop significant design data.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Fracture mechanics, High cycle fatigue, low cycle fatigue, Rene' 41, Astroloy, high pressure ammonia	

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price*
Unclassified	Unclassified	78	

*For sale by the National Technical Information Service, Springfield, Virginia 22151

NASA — JSC

JSC Form 1424 (Rev Jul 74)

CONTENTS

	Page
INTRODUCTION	1
PROCEDURE	1
Selection of Test Parameters	1
Material Selection and Procurement	3
Specimen Preparation	4
Test Equipment	5
Test Procedure	6
RESULTS	7
Fracture Mechanics Test Results	7
Fatigue Test Results	7
DISCUSSION	8
Fracture Mechanics Tests	8
Fatigue Tests	9
CONCLUSIONS	10
Fracture Mechanics Tests	10
Fatigue Tests	11
REFERENCES	11
APPENDIX	59

ILLUSTRATIONS

Figure		Page
1	Turbine Disk Temperature Distribution During Startup, Transient and Steady State Operation	12
2	Hydrazine Decomposition Products	13
3	Fracture Mechanics Test Specimen	14

ILLUSTRATIONS (Continued)

Figure		Page
4	Preparation of Rene' 41 Fracture Mechanics Specimens	15
5	Preparation of Astroloy Fracture Mechanics Specimens	16
6	Fracture Mechanics Specimen in Fixture Mounted on Vibrating Table for Precracking by Fatigue	17
7	Finish Machined Fracture Mechanics Specimen, Instrumented and Ready for Testing	18
8	Fatigue Test Specimen	19
9	Preparation of Rene' 41 Fatigue Test Specimens	20
10	Preparation of Astroloy Fatigue Test Specimens	21
11	Finish Machined Fatigue Specimen Instrumented and Ready for Testing	22
12	Schematic Diagram of Tinius Olsen Testing Machine	23
13	Tinius Olsen Testing Machine Used for the Fracture Mechanics Experiments	24
14	Tinius Olsen Testing Machine Used for the Fracture Mechanics Experiments	25
15	Gas Cart and Retort Schematic	26
16	Gas Cart Showing Control Panel	27
17	Gas Cart Showing Internal Mechanism	28
18	Diagram Showing Arrangement of the Bellows Seal	29
19	Specimen Ready for Air Testing	30
20	Schematic Diagram Showing Location of Thermocouples in Fracture Mechanics and Fatigue Tests	31
21	K_i/K_{IE} Values for Rene' 41 at 950 K (1250°F)	32
22	K_i/K_{IE} Values for Astroloy at 950 K (1250°F)	33

ILLUSTRATIONS (Continued)

Figure		Page
23	Fracture Surfaces of Rene' 41 K_{IE} and K_{TH} Test Specimens	34
24	Fracture Surfaces of Astroloy K_{IE} and K_{TH} Test Specimens	35
25	Histogram Showing Relative Fatigue Life for the Various Combinations Tested	36
26	Typical Fatigue Fracture Surface Rene' 41 Specimen	37
27	Typical Fatigue Fracture Surfaces Astroloy Specimen	38
28	Specimen R 32, Gold Plated Before Testing	39
29	Specimen R 32, Gold Plated Tested in Ammonia	40
30	Specimen R 25, Uncoated, Tested in Ammonia	41

TABLES

Table		Page
I	Chemical Analysis of Materials Used	42
II	Age Hardening Procedures for Test Specimens	43
III	Schedule of Fracture Mechanics Testing	43
IV	Schedule of Fatigue Testing	44
V	Search for Optimum Load for Astroloy HCF Tests	44
VI	Search for Optimum Load for Astroloy LCF Tests	45
VII	Search for Optimum Load Rene' 41 HCF Tests	46
VIII	Search for Optimum Load Rene' 41 LCF Tests	46
IX	Results of Fracture Mechanics Testing at 950 K	47
X	Results of Fracture Mechanics Testing at 1250°F	48
XI	HCF Test Results for Astroloy at 950 K (SI Units)	49

TABLES (Continued)

Table		Page
XII	LCF Test Results for Astroloy at 950 K (SI Units)	50
XIII	HCF Test Results for Rene' 41 at 950 K (SI Units)	51
XIV	LCF Test Results for Rene' 41 at 950 K (SI Units)	52
XV	HCF Test Results for Astroloy at 1250°F (U.S. Customary Units)	53
XVI	LCF Test Results for Astroloy at 1250°F (U.S. Customary Units)	54
XVII	HCF Test Results for Rene' 41 at 1250°F (U.S. Customary Units)	55
XVIII	LCF Test Results for Rene' 41 at 1250°F (U.S. Customary Units)	56
XIX	Summary of Fracture Mechanics Testing	57

DEVELOPMENT OF FRACTURE MECHANICS DATA

FOR TWO HYDRAZINE APU TURBINE WHEEL MATERIALS

INTRODUCTION

The proposed use of hydrazine monopropellant fuel to energize the Space Shuttle Auxiliary Power Unit (SSAPU) imposes some potential materials problems. Decomposed hydrazine consists of hydrogen, nitrogen and ammonia, any one of which can cause degradation of mechanical properties of most metals. Little or no data exists that can be used to design turbine wheels to survive the demanding requirements of stress, temperature, life and operating cycles in this environment.

Tests were conducted to measure the effects of exposure to high temperature, high pressure ammonia on the fracture mechanics and fatigue properties of Astroloy and Rene' 41 turbine wheel materials, and to determine the influence of coatings on those properties.

The units used for the physical quantities defined in this report are given in the U.S. Customary Units and in the International System of Units (SI). Measurements and calculations were made in the U.S. Customary Units. Factors relating the two systems are given in reference 1.

PROCEDURE

Selection of Test Parameters

The materials tested, the test environment, and the test procedures were designed to simulate actual SSAPU turbine wheel operating conditions as closely as possible. Previously conducted trade off analyses had determined the optimum operating conditions of the turbine wheel in terms of life, performance and reliability. These studies showed that a wheel run at 75,000 rpm and a turbine inlet temperature (TIT) of 1145 K (1600°F) would satisfy performance parameters. Operating life requirements of 1000 hr and 2000 start-stop cycles would have to be met and the "safe life" design philosophy was used as the criterion for establishing reliability.

Stress analysis had indicated the most critical area of the wheel was adjacent to the bolt holes; here the stresses were at a maximum because of the concentration of the centrifugal and thermal stresses. Thermal analyses conducted on a typical system, similar in requirements to the SSAPU application indicated that 1145 K (1600°F) TIT steady state operating conditions resulted in a metal temperature of 950 K (1250°F) at the bolt hole region of the wheel. Figure 1 shows the calculated temperature profile for the turbine wheel during startup and steady state conditions. The profile shows that a considerable thermal gradient exists throughout the mass of the wheel for several seconds after start up. Consequently, it is imperative that the wheel be able to accomodate such thermal stresses without failure and an understanding of the fracture behavior of the material is important to the design of reliable wheels.

Materials most commonly used for APU turbine wheel applications are the nickel base superalloys. These alloys possess optimum high temperature strength, stability, oxidation, corrosion and erosion resistance. And of the nickel base superalloys, Astroloy and Rene' 41 are the most popular wheel materials because they have the best combination of mechanical properties, manufacturing economy, and operational reliability. Hence, these two materials were selected for the investigation.

Work conducted by AiResearch prior to the start of this program demonstrated that some coatings were beneficial in reducing nickel base superalloys' susceptibility to degradation by exposure to ammonia. As a result of these experiments, two coatings were selected for testing on this contract. They were Chromizing Company's A1-870 a diffused aluminized coating, and 18 μm (0.7 mil) thick gold plating. The coating work is described in the Appendix.

Hydrazine, N_2H_4, is a liquid monopropellant and was selected to fuel the SSAPU. Energy is provided by its decomposition which is usually carried out in a decomposition chamber situated upstream from the turbine. Hydrazine can be decomposed by the application of external energy or by the use of a catalyst. The initial decomposition is exothermic and produces ammonia and nitrogen. Further dissociation of ammonia is endothermic and produces nascent hydrogen and nitrogen. By controlling the fraction of ammonia dissociated through gas generator design and catalyst selection it is possible to control the temperature of the gases and produce a wide variety of gas compositions delivered to the turbine. Figure 2 illustrates the products of decomposition as a function of ammonia dissociated.

Ammonia was used for the test program because it simplified test procedures (hydrazine involves rigid safe-handling techniques) and permitted greater operating flexibility without significantly reducing the data yield. Metallurgically at least, the substitution of hydrazine by ammonia is acceptable since the products of decomposition are the same, vis. nitrogen, hydrogen and ammonia. In practice, where decomposition is confined to the gas generator, any atomic nitrogen or hydrogen formed would probably be recombined to molecular gases before they are presented to the metal surface of the turbine wheel, consequently they are innocuous and only the ammonia component is harmful. At the metal surface the ammonia will dissociate to yield atomic nitrogen and hydrogen which can then penetrate the metal lattic and cause degradation of properties. Ammonia, furthermore, does not present as serious a safety hazard as does hydrazine, and with reasonable precautions can be used without any real danger.

Hydrazine is hygroscopic and readily absorbs moisture from the atmosphere. Consequently the commercial product usually contains about 1 percent water. MIL-P-26536C limits the maximum water content to 1.5 percent. Previous experiments with ammonia conducted by AiResearch showed that this small addition of water significantly improved most alloys' resistance to degradation. Hence, the wheel materials were tested in ammonia containing a nominal 1.5 percent water. A test pressure of 3.4 MN/m^2 (500 psi) was chosen to represent the typical pressure of gases delivered to the turbine. There was no way that the flow rate of gases through the experimental test rig could economically duplicate the mass flow of gasses through the SSAPU, so a nominal flow rate of

7.5×10^{-3} kg/sec (1 lb/hr) of ammonia was selected as being the minimum controllable amount that would give a fairly uniform dissociation ratio throughout the duration of the tests.

To survive the demanding life requirements, the SSAPU turbine wheel must have two basic characteristics. First, it must have good thermal, or low cycle fatigue resistance to accommodate the start up stresses, or, more accurately, the low cycle fatigue properties must be established and design data available for stress analysis calculations, and second, the fracture characteristics must be known and measurable so that a "safe life" wheel can be designed with a high degree of reliability. The fatigue properties and the fracture mechanics threshold intensity parameters were measured in ammonia for both the wheel materials at a temperature of 950 K (1250°F). This program however, was not intended to generate design data, but to indicate which material could be considered, and what influence coatings had on the behavior of the materials exposed to simulated SSAPU operating conditions.

Material Selection and Procurement

The two turbine wheel materials selected were Astroloy and Rene' 41. Both materials have excellent high temperature strength and are suitable for use in forged integral turbine wheels. Ideally, forgings should be used for generating mechanical properties data because the end product will be manufactured from a forging. However, for this program forged bar stock was procured for the following reasons:-

(a) About 130 specimens were required for fatigue and fracture mechanics testing. To machine all of these specimens from the same diametral region of pancake forgings would have been quite expensive.

(b) The main purpose of the program was to compare materials, coatings and atmosphere effects. The difference in properties or behavior between forged bar stock and pancake forgings was considered of secondary importance.

(c) The properties of specimens machined from forged bar stock were considered to be more uniform on a specimen-to-specimen basis, than for those obtained from forged material, thus minimizing data scatter.

Astroloy bars, 75.8 mm (2.985 in.) dia x 1370 mm (54 in.) long and 19.2 mm (0.756 in.) dia x 4900 mm (15 ft) long were purchased from Special Metals Corporation. The alloy was from Heat numbers 8-3154, 75.8 mm (2.985 in. dia) and 8-3155, 19.2 mm (0.756 in. dia) and was processed by the vendor from reforging stock. No heat treatment had been carried out and the bars were delivered in the "as rolled" condition. Test certificates from the mill were received with the bars and chemical analysis was conducted on samples of the material to ensure that the alloy was within the limits of AiResearch Engineering Specification EMS 95355.

Three sizes of Rene' 41 bars were purchased to AMS 5712. The material was from Special Metals Heat Number 9-1855 and was delivered in the solution annealed condition. Analysis of samples of the bars confirmed that the material was within the specified limits.

Chemical analyses of the materials are shown in Table 1.

Specimen Preparation

Fracture Mechanics Specimens. - Astroloy bars, 75.8 mm (2.985 in.) dia were used to make the fracture mechanics test specimen shown in figure 3. The bars were cut to lengths of 215 mm (8.5 in.) and then cut along the longitudinal axis, and in this "D" shaped section the bars were solution annealed at 1935 K (2050°F) for one hour followed by rapid air cooling. Rene' 41 samples were made from 41.3 mm (1.625 in.) dia bars which were delivered in the solution annealed condition, hence, annealing was not carried out by AiResearch. Two thicknesses of test bars were machined; uncoated specimens were 7.8 mm (0.310 in.) thick and the coated samples were 10.5 mm (0.410 in.) thick. The specimens were then EDM machined in the center of one face to produce a slot from which a fatigue crack was grown by a cantilever motion generated by a vibrating table. Age hardening was carried out as shown in Table II. Specimens that were tested in air were heated in air for 100 hr at 950 K (1250°F) prior to testing, and samples tested in ammonia were given a pre-test heat treatment at 950 K (1250°F) for 100 hr in ammonia.

Details of the fracture mechanics specimen preparation are shown in figures 3 through 7.

Fatigue Specimens. - Astroloy bars, 19.2 mm (0.756 in.) dia were centerless ground to 12.7 mm (0.50 in.) dia and cut into convenient lengths for solution annealing at 1395 K (2050°F) for one hour followed by rapid air cooling. The 12.7 mm (0.50 in.) dia Rene' 41 bars were not heat treated by AiResearch because they were delivered in the solution annealed condition. All the test bars were age hardened according to Table II, (except that the Rene' 41 bars were heated in air). After finish machining and coating (where necessary), the specimens were heated for 100 hr at 950 K (1250°F) prior to testing. Air tested specimens were heated in air, and ammonia tested specimens were pretreated in that atmosphere. The purpose of this pre-test heat treatment was to reduce the time differential effects. Low cycle fatigue samples were exposed to the ammonia during testing for only a few hours, whereas high cycle fatigue test bars were exposed for several days to the ammonia environment. Hence, significant differences in properties might have resulted because of the differences in exposure times of the various specimens. By pre-treating all the specimens a buffering effect was produced, and differences in properties could be attributable to the material's reaction to the environment and not just to the time of exposure. Air tested specimens were pretreated for 100 hr in air to ensure that the specimens were in the same metallurgical condition as the ammonia tested specimens.

Details of the fatigue specimen preparation are shown in figures 8 through 11.

Test Equipment

Fracture Mechanics Testing Equipment. - The K_{IE} and the K_{TH} tests were performed on a Tinius Olsen cantilever, dead weight loading machine with a reversible crosshead drive motor. Figure 12 schematically represents the workings of the instrument. Microswitches were added to the end of the lever arm so that changes in specimen dimensions would activate the switches such that the crosshead would be moved to maintain an equilibrium load on the specimen. Loads up to 0.667 MN (150,000 lbs) could be applied to the specimen, which was attached to the machine with universal ball and socket specimen holders which assured alignment. The crosshead was driven mechanically through a gear train by a reversible electric motor.

A 380 mm (15 in.) long, 76 mm (3 in.) dia Marshall furnace was clamped to the frame of the Olsen, and a ventilation duct was installed adjacent to the mouth of the furnace to prevent any escaping ammonia from entering the test cell.

Figures 13 and 14 depicts the Olsen testing machine used for the fracture mechanics experiments.

Fatigue Testing Equipment. - The fatigue testing experiments were conducted at the Engineering Research Center of the California State University at Long Beach using their MTS System Corporation closed loop hydraulic test machine. The machine had a 12.5×10^{-3} m^3/min (55 gal/min) hydraulic supply capacity and was capable of supplying up to 0.31 MN (70,000 lb) load. The unit had a closed loop control system which included three signal conditioners i.e., strain, load and stroke. An AEI-20 analog computer was used to generate sine wave load control during test.

A 380 mm (15 in.) long, 76 mm (3 in.) dia Marshall furnace was clamped to the frame of the MTS machine, and a ventilation duct was installed adjacent to the mouth of the furnace to prevent any escaping ammonia from entering the test cell.

Gas Cart. - Liquid ammonia containing 1.5% H_2O was pressurized to 3.4 MN/m^2 (500 psi) by a small diaphragm pump, preheated in an electric resistance furnace and delivered to the retort at a rate of about 7.5×10^{-3} kg/sec (1 lb/hr). Pressure and flow rate were maintained by adjusting the length of stroke on the pump and a needle valve downstream from the retort. Nitrogen was used to purge the system and to provide pressure for the various safety systems designed to cut off the ammonia flow in case of a major ammonia leak.

The retort was made from Inconel 625 and was aluminized on the inside to minimize the dissociation of ammonia on its surfaces. It was placed inside an electric resistance furnace. Attachment arms were passed through the retort to hold the specimens. These arms were made from Rene' 41 and were also aluminized. A bellows seal was fixed to one end of the retort so that the stress from the crosshead of the testing machine could be transmitted to the specimen inside the retort. Provision was made for extracting gas samples for analysis from as close to the surface of the specimens as possible.

A sheathed chromel-alumel thermocouple was positioned near the center of the specimen inside the furnace, and was coupled to the furnace temperature controller. A second thermocouple was placed outside the retort but in the hot zone of the furnace to monitor furnace temperature and to shut off the current to the furnace in case of specimen thermocouple failure.

The gas cart and retort are shown schematically in figure 15. Figures 16 and 17 show the control panel and internal mechanism of the cart. Figure 18 shows the retort-bellows arrangement.

Test Procedure

Fracture Mechanics Tests. - An unsheathed chromel/alumel thermocouple was wired to the specimen such that the bead was in contact with the surface of the sample adjacent to the fatigue crack. The specimen was then screwed into the specimen attachment holders and inserted into the retort. For ammonia tests the retort was bolted closed using stainless steel clad asbestos gasket seals on the flanges; for air tests, bolts and seals were not used. The retort was permanently fixed inside the furnace. A sheathed chromel/alumel thermocouple was placed inside the retort adjacent to the sample, and the output was fed into the controller to control the specimen temperature. Other chromel/alumel thermocouples were installed in the hot zone between the furnace lining and the retort, and also embedded in the furnace windings. These two thermocouples were wired to Alnor safety cut off devices preset to cut off current to the furnace in case of an overtemperature situation.

Ammonia tests were commenced by purging the gas cart and retort with nitrogen, heating the specimen to 950 K ($1250^{o}F$) and calibrating the sheathed thermocouple with the unsheathed thermocopule inside the retort. Ammonia was then introduced into the retort and its pressure and flow rate stabilized at 3.4 MN/m^2 (500 psig) and 7.5×10^{-3} kg/sec (1.0 lb/hr) respectively. After temperature, pressure and flow rate equilibrium had been reached, the load was applied to the specimen and the timer started. Load was monitored by a strain gage attached to the crosshead fixture, the output from which was fed into a strip chart recorder. Specimen failure automatically stopped the ammonia flow, turned off the furnace and stopped the timer.

For the air tests, the unsheathed thermocuple attached to the specimen was used for temperature control, and the load was applied as soon as temperature equilibrium had been attained. All the specimens were loaded at a strain rate of 15 μm/sec (0.035 in/min).

Figure 19 demonstrates the air testing arrangement. (The furnace has been removed for clarity.) Table III indicates the Fracture Mechanics testing schedule.

Fatigue Tests. - The test set up for fatigue testing was very similar to the procedure described for fracture mechanics testing. An unsheathed chromel/alumel thermocouple was wired to the specimen such that the bead was in contact with the surface of the sample adjacent to the circumferential notch. The sample was then attached to the specimen holders fastened to the MTS machine.

For the ammonia tests the retort was bolted closed using stainless steel clad asbestos gasket seals on the flanges. A sheathed thermocouple was placed inside the retort next to the sample, and fed into the controller to control the furnace temperature. Other chromel/alumel thermocouples in the hot zone between the furnace lining and the retort, and also embedded in the furnace windings. These two thermocouples were wired into Alnor safety cut off devices preset to cut off current to the furnace in case of an overtemperature situation. The thermocouple arrangement is shown in figure 20.

Ammonia tests were commenced by purging the gas cart and retort with nitrogen, heating the specimen to 950 K (1250°F) and calibrating the sheathed thermocouple with the unsheathed sensor inside the retort. Ammonia was then introduced into the retort and its pressure and flow rate stabilized at 3.4 MN/m^2 (500 psig) and 7.5×10^{-3} kg/sec (1.0 lb/hr) respectively. After temperature, pressure and flow rate equilibrium had been reached the load cycle was applied to the specimen. The fatigue testing schedule is shown in Table IV.

A sine wave load control system was selected and the maximum stress adjusted to give failure in 10^3 to 10^4 cycles for LCF testing, and 10^6 to 10^7 cycles for HCF testing. A fatigue load ratio of R = 0.1 was used throughout the experiments, and a frequency of 50 Hz was used for all specimens.

For the air tests, the retort was not used and the unsheathed thermocouple attached to the specimen was used for temperature control. The loading cycle was applied as soon as the temperature of the specimen had reached equilibrium at 950 K (1250°F). At first some difficulty was experienced in selecting the correct load range because two of the first batch of specimens failed in the threaded portion of the test bar instead of at the notch, Reworking the threads by grinding a more generous root diameter and tapering the specimen holders appeared to resolve the problem. The loads attempted and the order of testing the first batch of specimens is shown in Table V. The LCF load cycle search for Astroloy is shown in Table VI, and the Rene' 41 searches in Tables VII and VIII.

RESULTS

Fracture Mechanics Test Results

The results of the Fracture Mechanics tests are shown in Table IX (SI Units) and table X (U.S. Customary Units). Figures 21 and 22 show the K_i/K_{IE} values as calculated for each specimen plotted against the time to rupture. From these graphs the K_{TH} values can be estimated for the various testing conditions. The fractured surfaces are shown in figures 23 and 24.

Fatigue Test Results

HCF and LCF Fatigue test results are tabulated in Tables XI through IV (S1 Units) and XV through XVIII (U.S. Customary Units). A histogram (figure 25) also shows the relative fatigue life of the various combinations of materials, coatings and atmospheres. The fractured surfaces of the fatigue specimens are depicted in figures 26 and 27.

DISCUSSION

Fracture Mechanics Tests

The width (W) and the thickness (B) of the surface flawed specimens were measured before testing, and an attempt was made to measure the crack width (2c) also before testing, but this proved rather difficult to accomplish. Even though the surface around the crack was polished and examined at magnifications as high as 1000X, the ends of the crack were not clearly defined. Of course, no etchants or crack detection fluids were used so that the root of the crack would not become contaminated and introduce an uncontrolled corrosion factor into the experiments. Consequently, the crack length and the crack depth (a) were measured after the test piece had been broken into two pieces. Several of the specimens ruptured during tests, but those that did not were broken in the following manner.

The unbroken test bar was removed from the testing machine after 100 hr and returned to the fatigue fixture on the vibrating table so that the fissure could be extended a little. The sample was then loaded in tension at room temperature until it ruptured. Toward the end of the experiments however, this "fatigue marking" was discontinued because surface discoloration was found to be a reliable guide to the presence of crack growth.

When no crack growth was observed (as in specimens A17-1, A21 and R32) the original fatigue crack surfaces were oxidized and discolored, but the rest of the fracture face (broken at room temperature) was not discolored. On specimens A18, R25 and R30 a second band of discoloration extended beyond the original fatigue crack surface, indicating that the secondary surface had been exposed at high temperature, i.e., the original crack had grown during the test under load at 950 K (1250^{o}F). The final fracture surface, broken at room temperature, was not discolored. Figures 23 and 24, being black and white, do not depict this discoloration very clearly.

Specimens R29, R26, R27 and R31, even though they rupture before the expiration of 100 hr, still showed evidence of a secondary band of discoloration, indicating that the crack growth rate must have been reasonably slow. The secondary band on specimen A20 is not so easily explained since the specimen ruptured after only a few minutes under load. It raises fears that the specimen, in some way, may have been cracked before loading into the test fixture. The gold plated samples had 1.27 mm (0.050 in.) ground from their surfaces, were gold plated and then age hardened in a vacuum furnace, and finally pre-treated for 100 hr at 950 K (1250^{o}F) in ammonia contained in a retort. All this processing was conducted after the specimens had been pre-cracked. Although the test bars were carefully handled, and slowly cooled during heat treatment and preconditioning, the possibility of accidental crack extention can not be overlooked. None of the other gold plate specimens showed any evidence of secondary cracking before testing, except perhaps R31. This specimen was under load for 33.6 hr before it failed, and did follow the pattern observed on all of the Rene' 41 samples that failed in similar times. The uncoated specimens were pre-cracked in the age hardened condition, hence secondary cracking in these cases could not be attributed to the heat treatment cycle.

Crack growth during testing was usually accompanied by crack widening. Figure 28 shows the typical appearance of a gold plated specimen before testing. A specimen which exhibited no growth, and a specimen which did experience some crack growth but did not rupture are illustrated in figures 29 and 30. Note that prior to testing the original fatigue crack is covered by the gold and is all but invisible. In the second case the strain has been sufficient to rupture the gold plating, but no appreciable crack widening was observed. In the third case crack widening is obvious.

Fracture mechanics data points plotted as K_i/K_{IE} values against duration of test under load were shown previously in figures 21 and 22. From these it appears that gold plating improved the K_{TH} value when compared to the uncoated specimens when both were tested in ammonia. For uncoated specimens testing in ammonia causes a lowering of the K_{TH} value when compared to specimens tested in air. The Astroloy data however is not so clearly defined. The same apparent trends exist, but there are not sufficient good data points to be really conclusive. Table XIX summarizes the K_{IE} and the K_i/K_{IE} values observed for the various conditions tested.

Fatigue Tests

Fatigue tests were conducted on 96 specimens. These tests were performed with an MTS closed loop electrohydraulic test machine at the Engineering Research Center of California State University at Long Beach. The specimen size was selected to be compatible with the test equipment (retort size, MTS machine cyclic load), and a circumferential groove was selected as a well defined stress concentration that could be coated uniformly. The specimens were originally to have a nominal stress concentration factor of $K_t = 3.0$, but the initial batch of Astroloy specimens were machined with a stress concentration closer to $K_t = 3.2$. Therefore the decision was made to make all subsequent specimens to the same nominal dimensions, and thus the stress concentration tolerance band for the 96 specimens was $K_t = 3.2 \pm 0.2$. The stress concentration factors given in the tables were calculated (Reference 2) from the groove dimensions before coating.

A fatigue load ratio of R = 0.1 was used for all tests. Fully reversed loads were not used because of the uncertainty in the behavior of the compressive load application to the specimen within the retort. An effort was made to establish loads that would cause failure in air between 10^6 and 10^7 cycles for HCF tests, and between 10^3 and 10^4 cycles for the LCF tests. The same loads were then used for similar specimens tested in ammonia.

The number of variables covered by the tests were:

Two materials (Astroloy and Rene' 41)

Two atmospheres (Air and ammonia)

Three coatings (Uncoated, gold plating and aluminising)

Two load conditions (HCF and LCF)

Since only four specimens were available for each combination of variables, the limitations on the number of test points preclude the generation of S/N curves. However, the test data are represented in the bar chart of figure 22. From this, and the data tabulations, some preliminary comparative evaluations and conclusions can be made, as itemized below:

1) For most test conditions the gold plated specimens exhibited equal or greater fatigue life than the uncoated or diffusion coated specimens. An exception was the slightly higher fatigue life of uncoated Rene' 41 in air when compared with gold plated Rene' 41 tested in air.

2) The fatigue life of the diffiusion coated specimens was less than the fatigue life of uncoated specimens.

3) The HCF life of both materials in the uncoated and gold plated conditions were significantly better in ammonia than in air.

4) Rene' 41 had greater HCF strength than Astroloy tested under the same conditions; i.e., 413 MN/m^2 (60 ksi) vs 328 MN/m^2 (47.5 ksi) for approximately the same cycle life.

It appears that gold plating generally improved the fatigue life, and that diffusion coatings generally reduced it, regardless of the testing atmosphere. This is no doubt caused by the presence of surface stresses introduced into the specimens by the coating techniques, at least for the diffusion coating, which invariably introduces lattice strains as the aluminum diffuses into and reacts with the surface layers of the alloy. In the case of the gold plating though, the apparent beneficial effect may be no more than the presence of the unreactive gold protecting the surface of the specimens' oxidation.

It is difficult to explain the apparently superior fatigue life of uncoated and gold plated specimens tested in ammonia as compared to specimens tested in air. Maybe the number of tests were insufficient to really detect a significant statistical trend; maybe the nitride surface strengthening had some effect; or maybe the nitride layers formed a compressive skin on the surface of the specimens. No analysis was conducted so the statements must remain as speculations rather than conclusions.

CONCLUSIONS

Fracture Mechanics Tests

(1) Specimens gold plated to a depth of 18 μm (0.7 mil) apparently had a higher threshold stress intensity factor than diffusion coated Al-870 specimens.

(2) For Rene' 41, the highest value of K_i/K_{IE} which resulted in no crack growth was 0.65, observed on the gold plated samples tested in ammonia.

(3) Uncoated specimens, tested in ammonia had a maximum no crack growth K_i/K_{IE} ratio of only 0.43.

(4) Definite conclusions were more difficult to establish for Astroloy because of the wide scatter band of data points.

(5) Because of the limited number of specimens (20) to cover combinations of two materials, three coatings (uncoated, aluminized and gold plated), and two environments; fracture mechanics data scatter did not yield meaningful K_{TH} values for any of the fracture mechanics experiments.

Fatigue Tests

(1) For most test conditions the gold plated specimens exhibited equal or better greater fatigue life than the uncoated or diffusion coated samples. An exception was the slightly higher fatigue life of uncoated Rene' 41 in air when compared with air tested gold plated Rene' 41.

(2) The fatigue life of the diffusion coated specimens was less than the fatigue life of the uncoated test bars.

(3) The HCF life of both materials in the uncoated and gold plated conditions were significantly better in ammonia than in air.

(4) Rene' 41 had greater HCF strength than Astroloy tested under the same conditions.

REFERENCES

1. ASTM Committee on Metric Practice: Metric Practice Guide (a guide to the use of SI - the international System of Units). ASTM Standard E380, Vol. 41, 1974.

2. Peterson, R.E.: Stress Concentration Factors. John Wiley and Sons, New York, 1965.

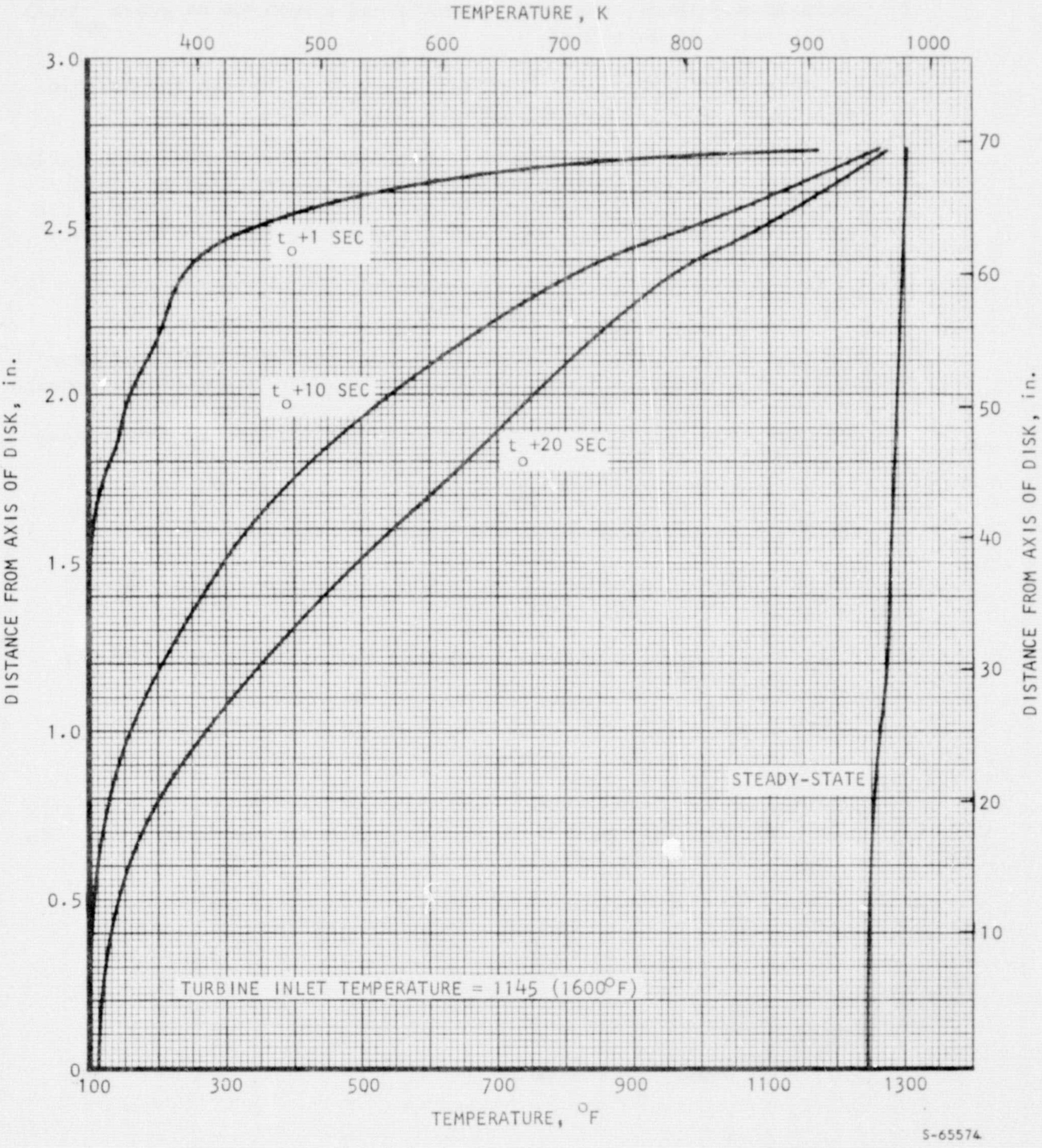


Figure 1. Turbine Disk Temperature Distribution During Startup Transient to Steady-State Operation

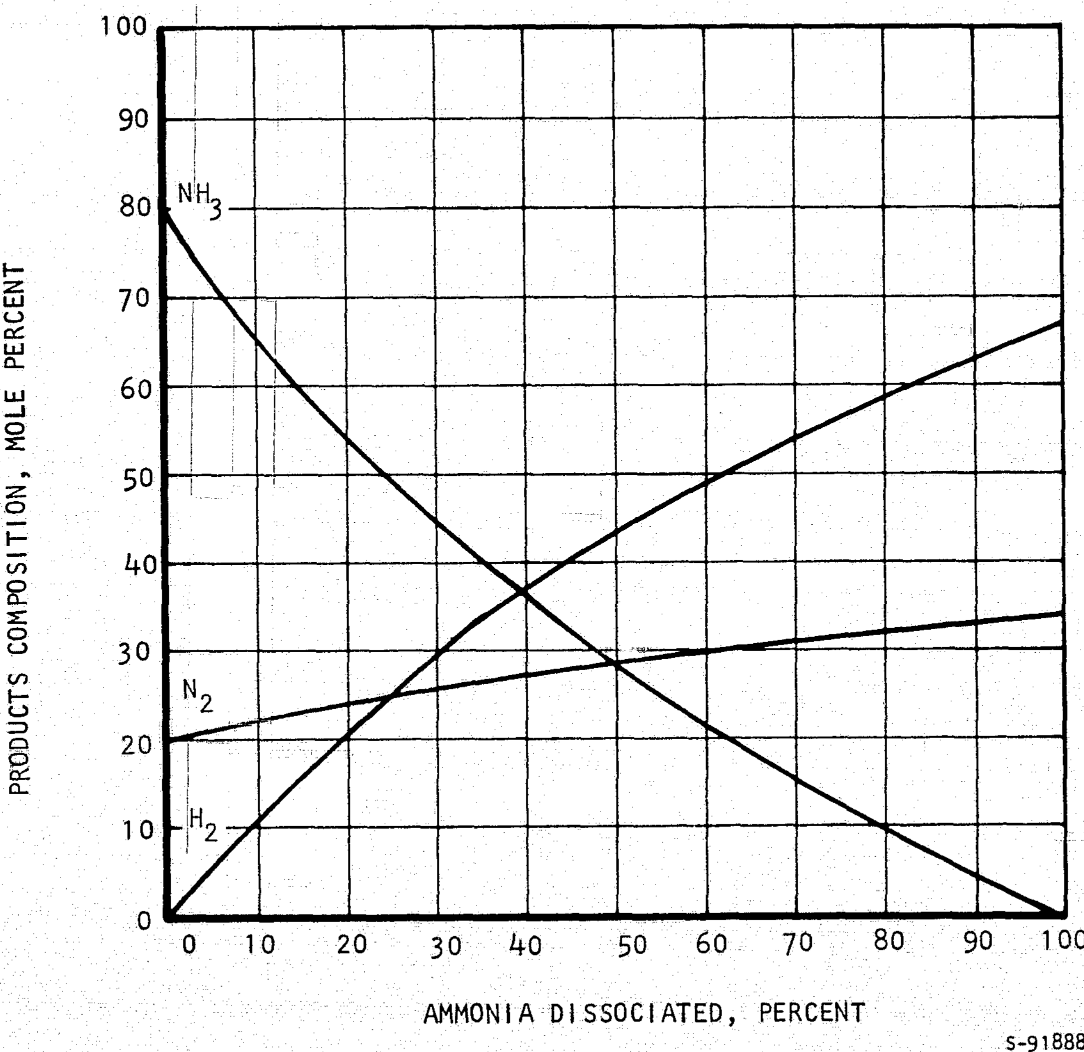


Figure 2. Hydrazine Decomposition Products

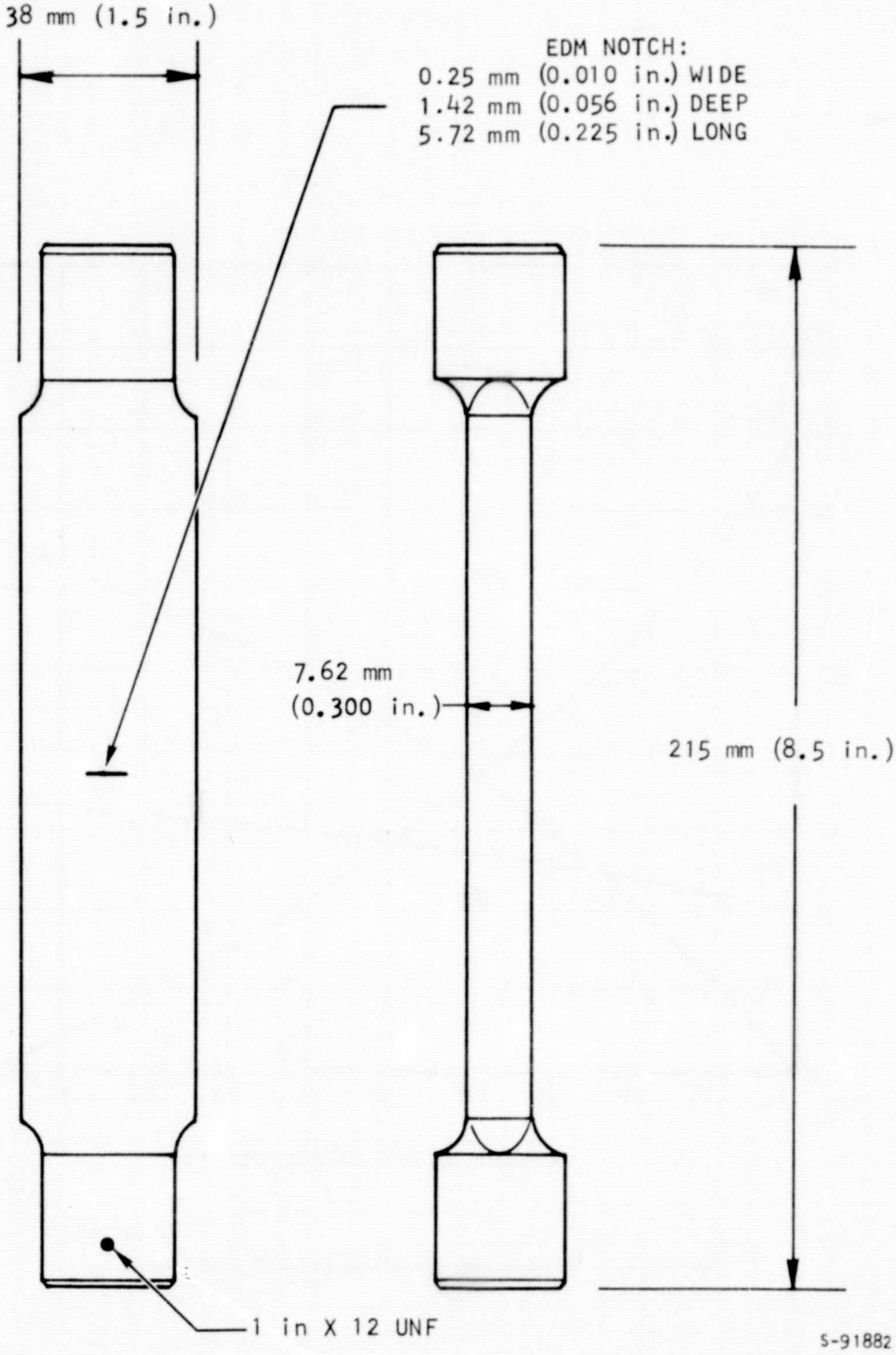


Figure 3. Fracture Mechanics Test Specimen

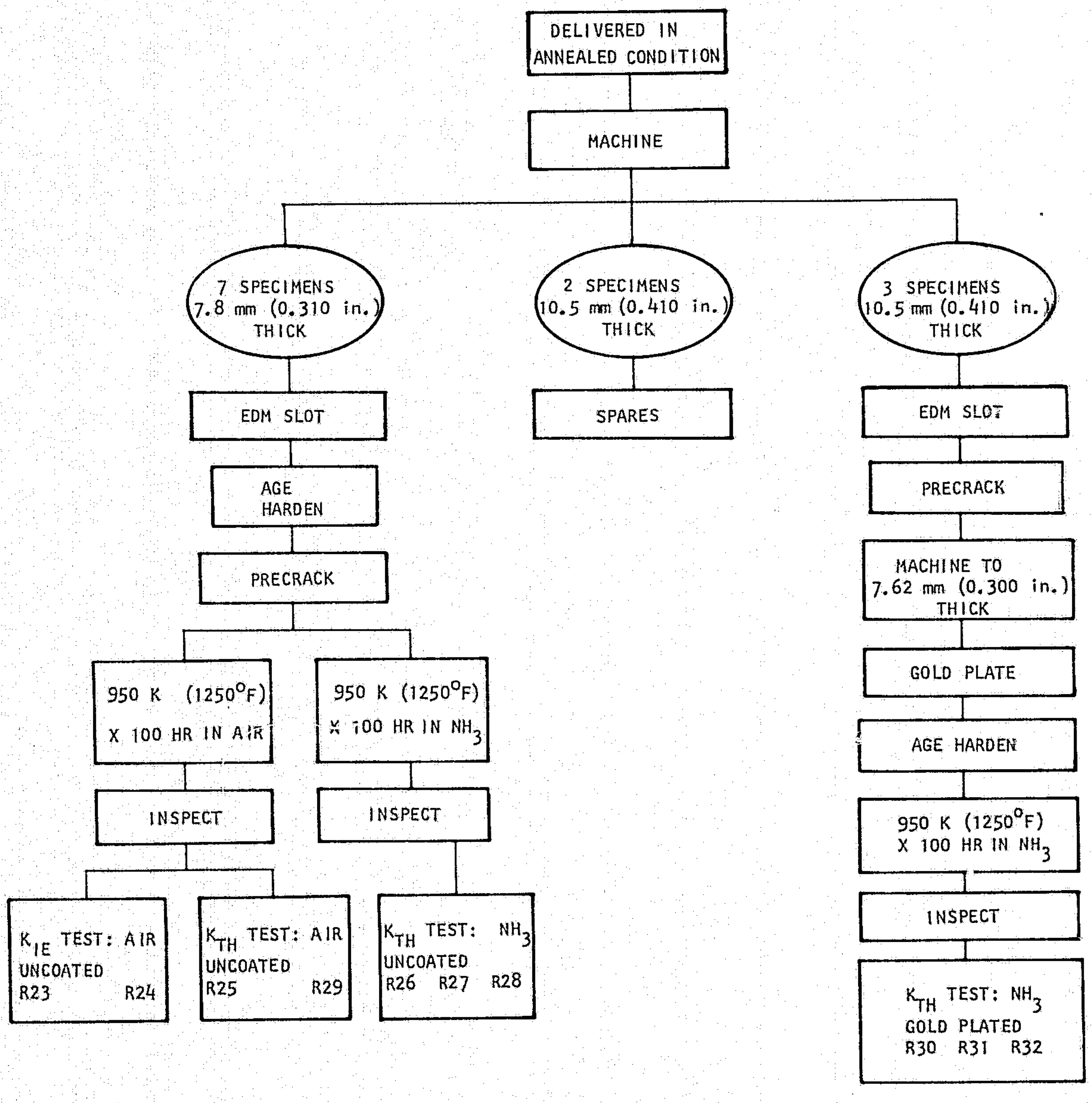


Figure 4. Preparation of Rene' 41 Fracture Mechanics Specimen

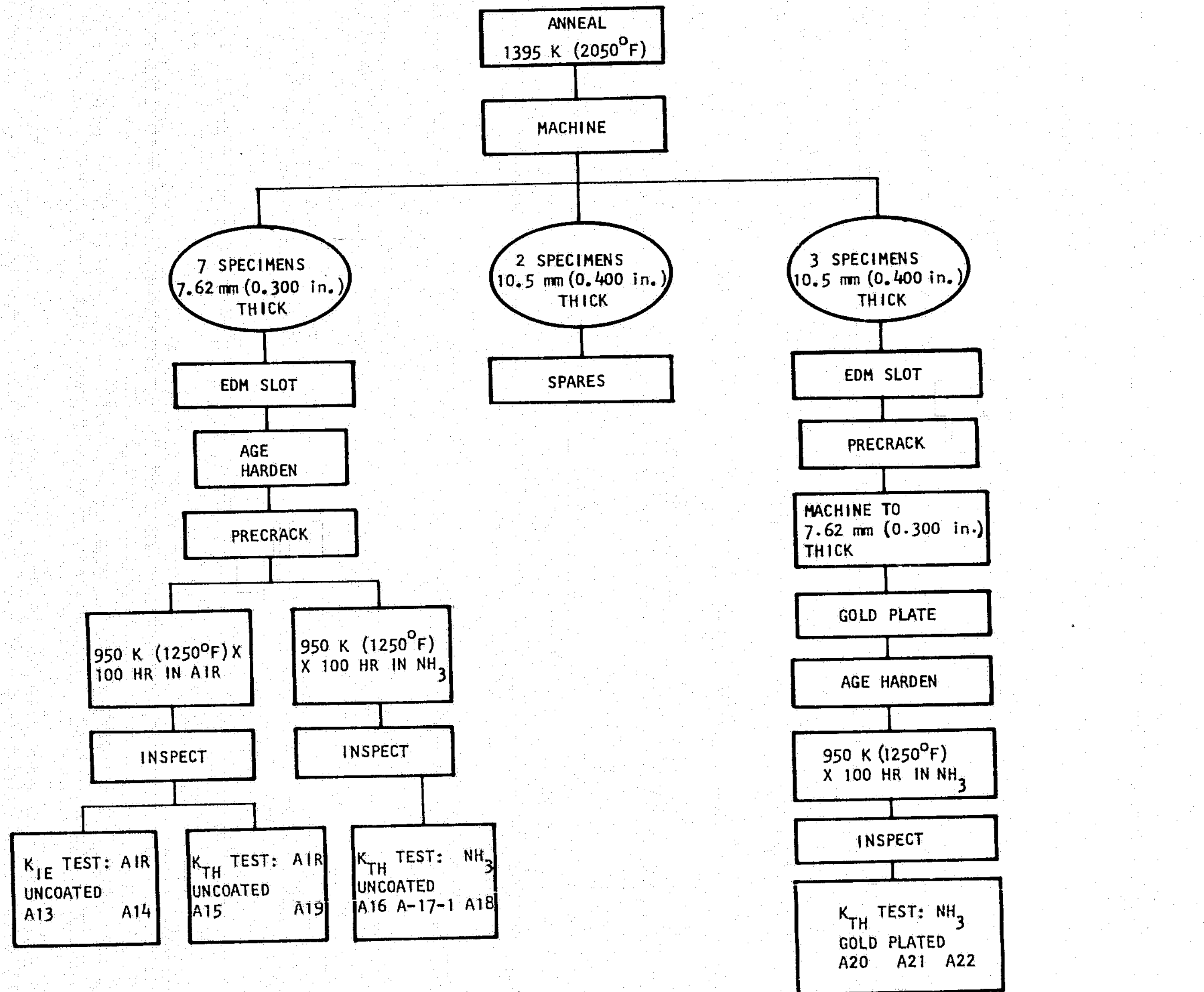


Figure 5. Preparation of Astroloy Fracture Mechanics Specimens

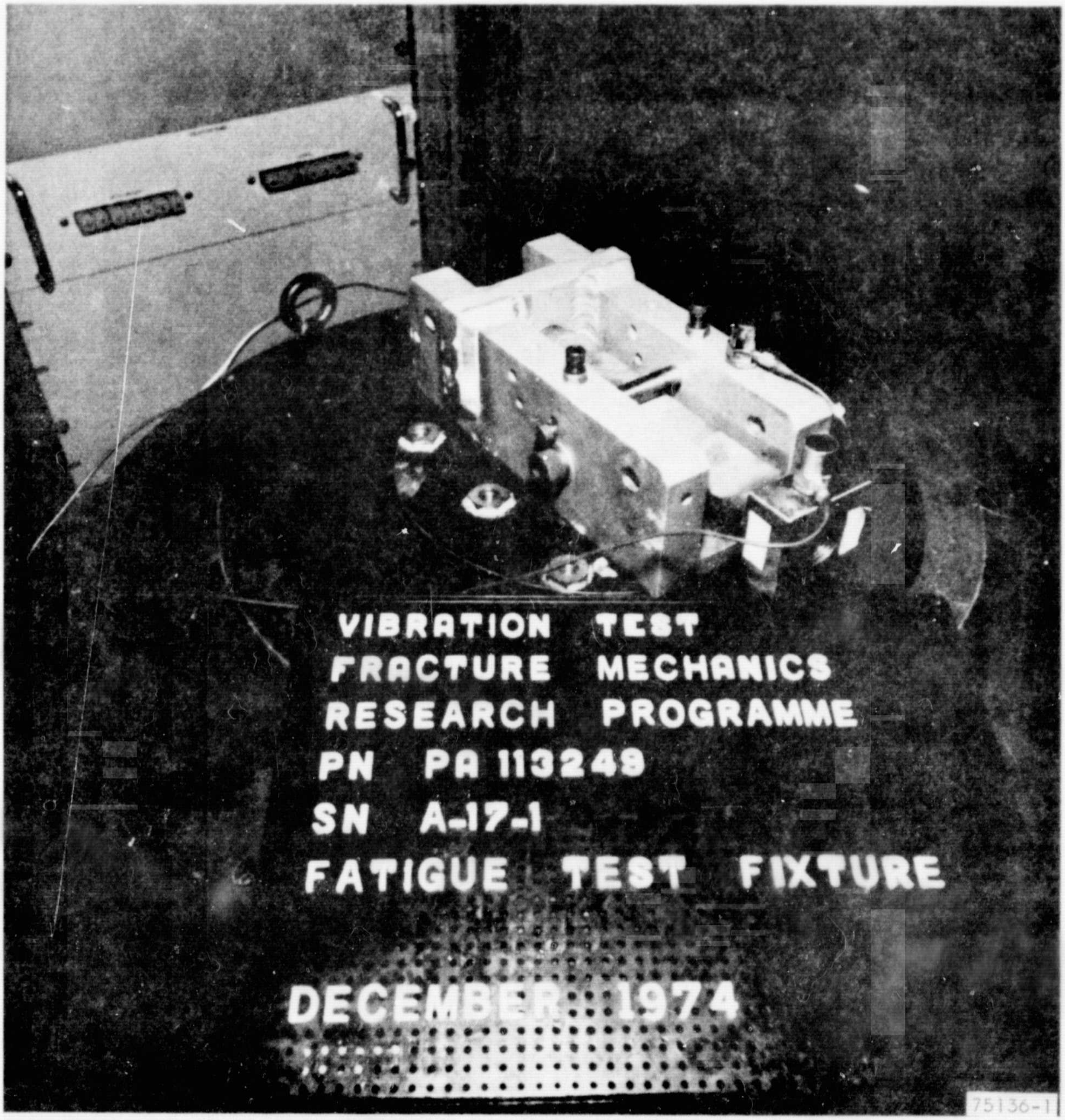


Figure 6. Fracture Mechanics Specimen on Fixture, Mounted on Vibration Table for Precracking by Fatigue

ORIGINAL PAGE IS
OF POOR QUALITY

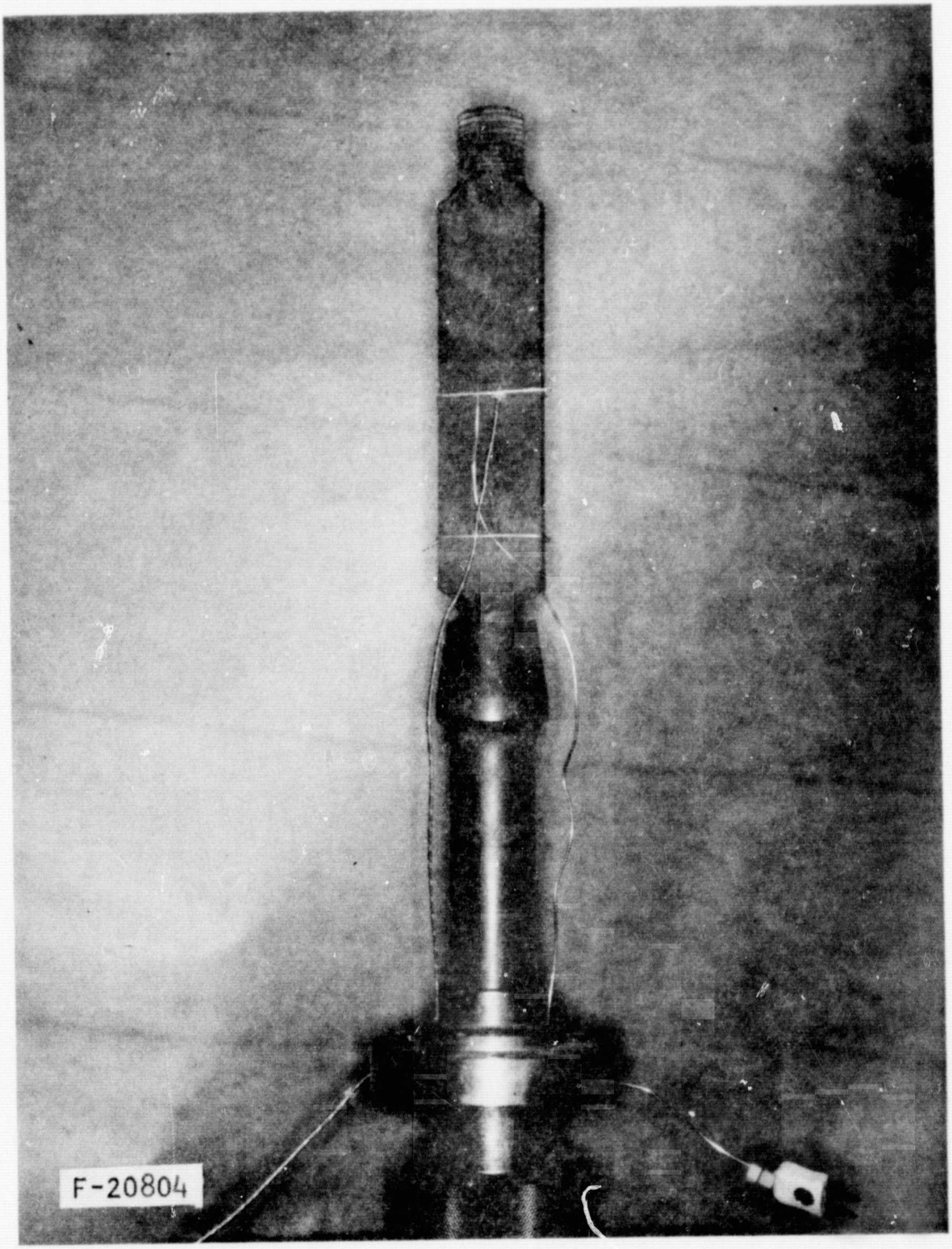


Figure 7. Finish Machined Fracture Mechanics Specimen Instrumented and Ready for Testing.

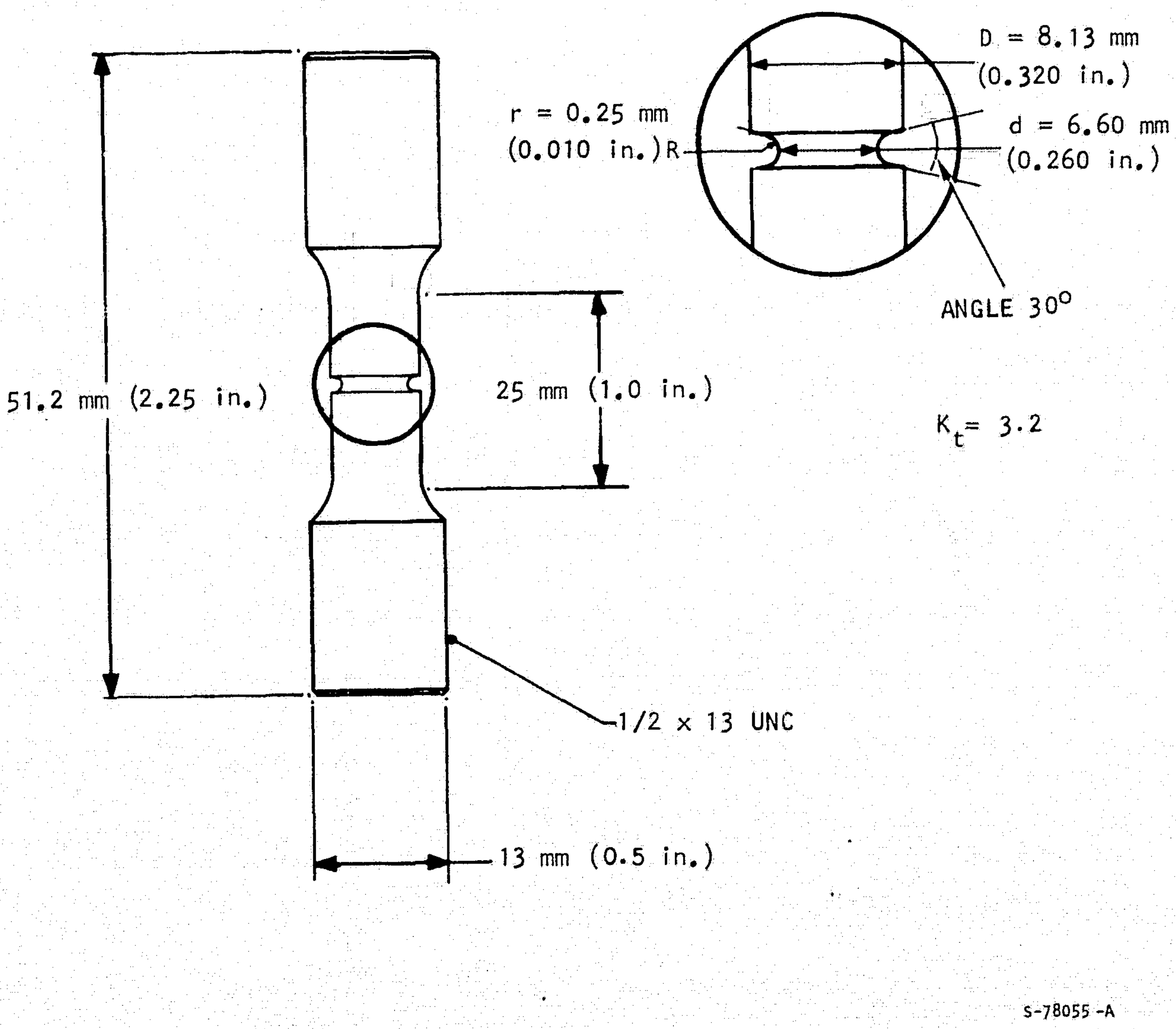


Figure 8. Fatigue Test Specimen

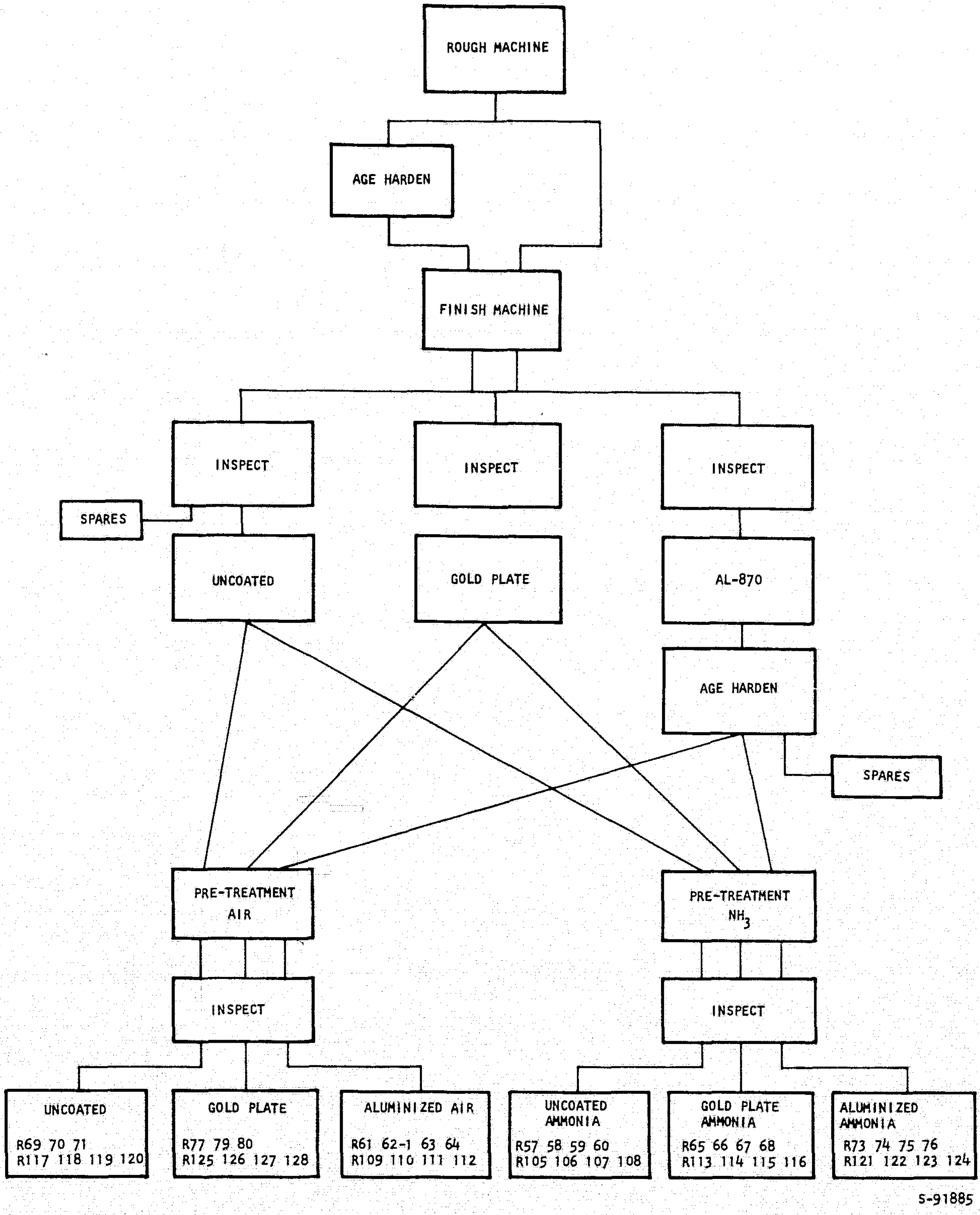


Figure 9. Preparation of Rene' 41 Fatigue Specimens

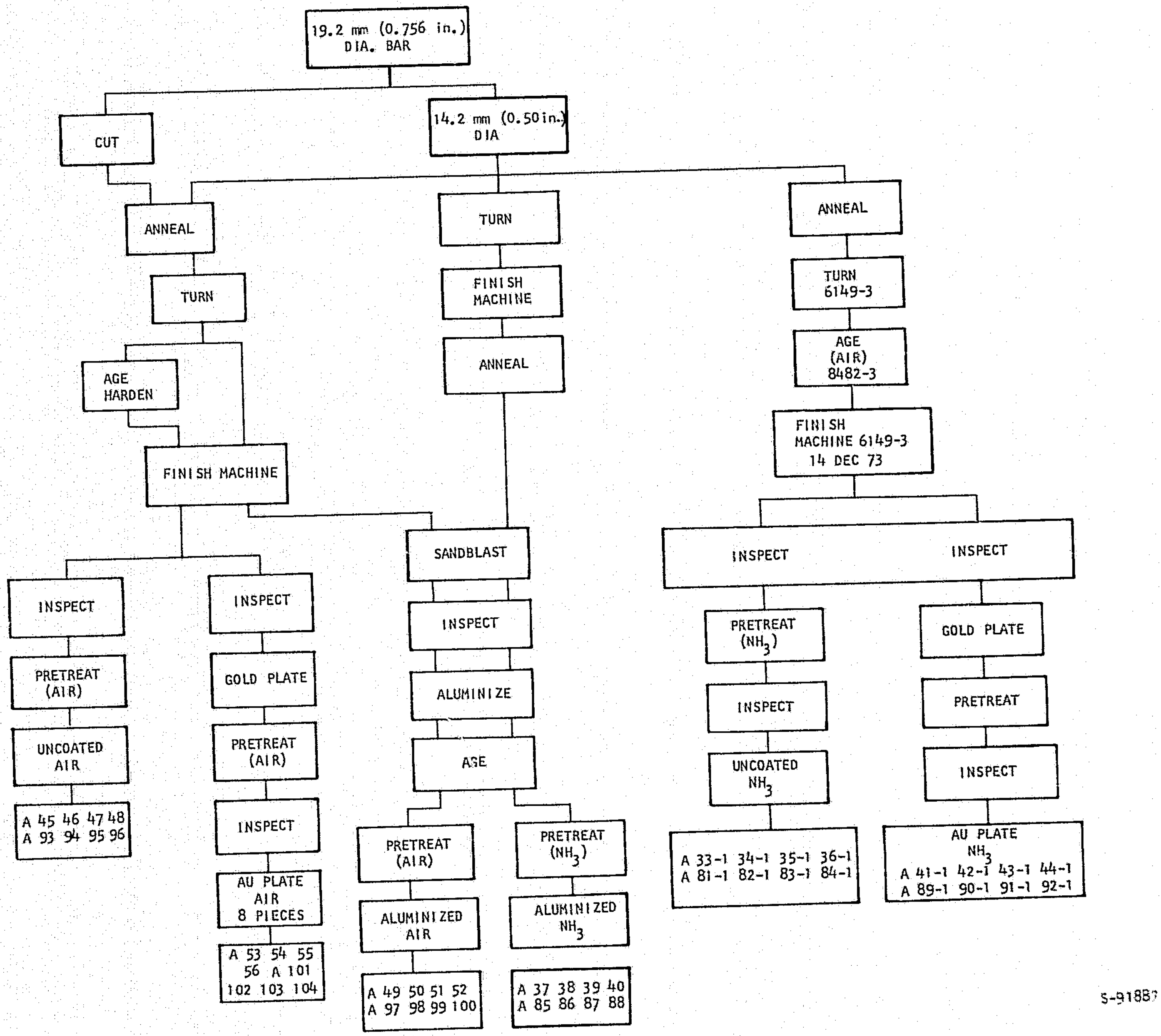


Figure 10. Preparation of Astroloy Fatigue Specimens

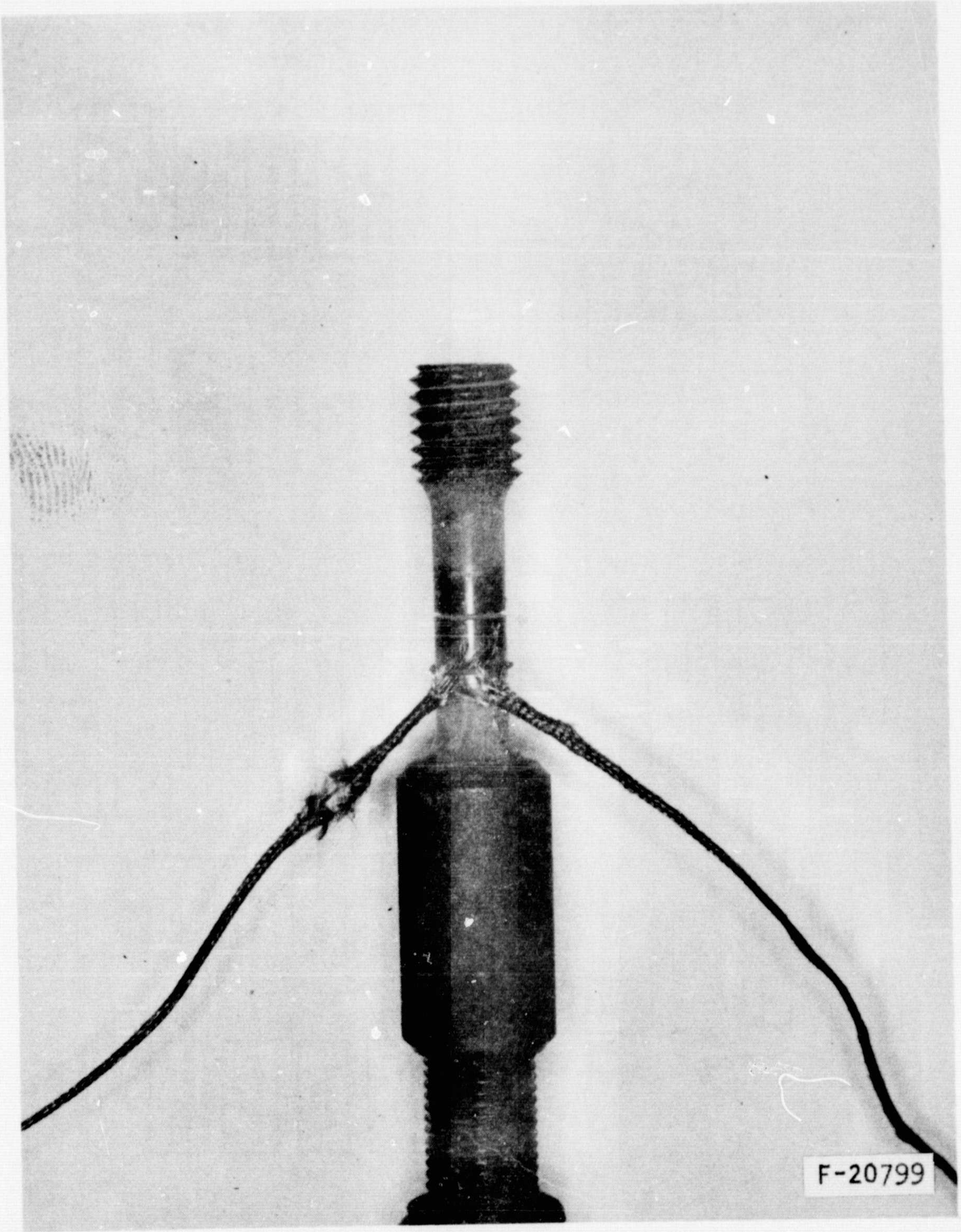
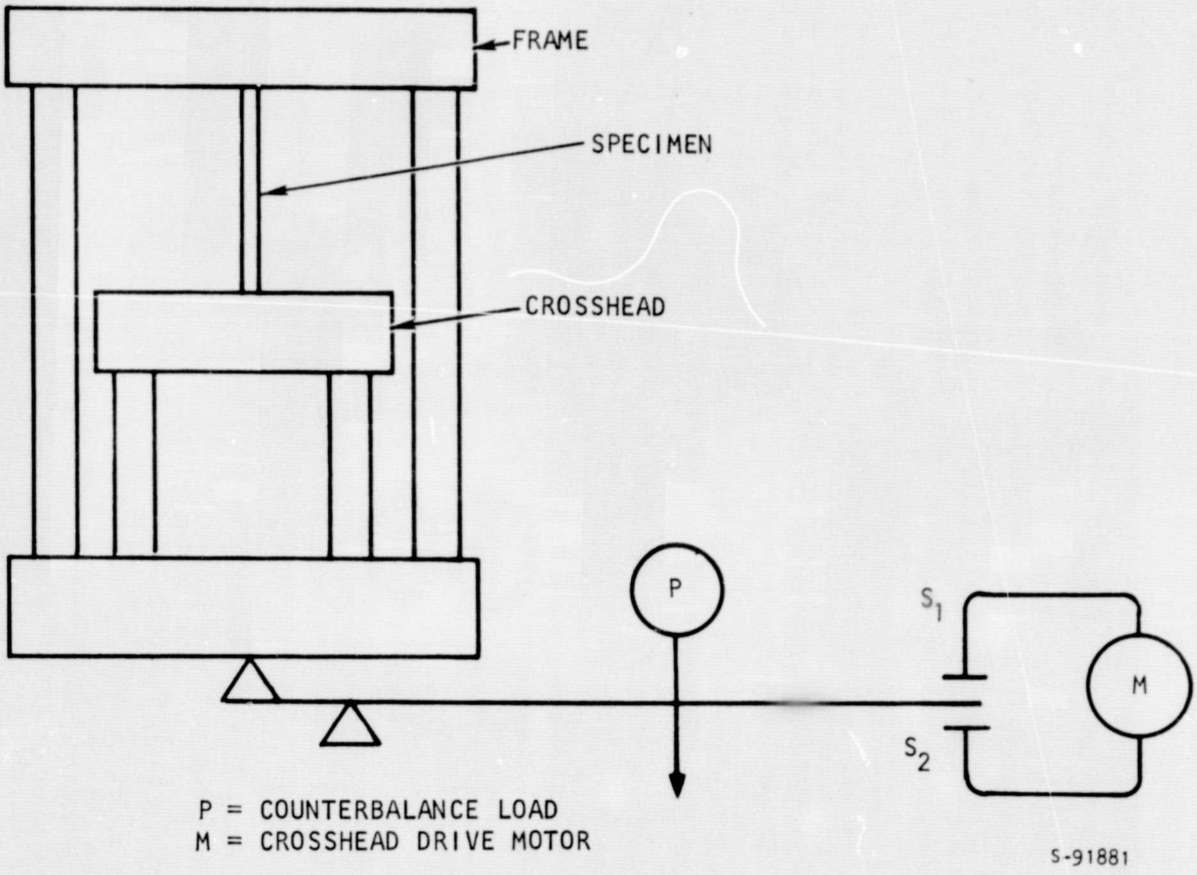


Figure 11. Finish Machined Fatigue Specimen Instrumented and Ready for Testing.



1) Specimen attached between frame and crosshead.

2) Load P is applied which causes the lever arm to drop, thus activating the reversible crosshead drive motor through the micro-switch s_2. The crosshead frame distance increases, applying a tensile load to the specimen. When the tensile load balances the applied load P, the lever arm balances, the motor stops and the machine is in equilibrium with a tensile load applied to the specimen.

3) An increase in the length of the specimen (elastic and plastic strain, thermal-expansion etc.) causes the lever arm to drop, activate the motor and regain the equilibrium load on the specimen.

4) A decrease in the length of the specimen (thermal contraction) causes the lever arm to raise, activating s_1 to reverse the direction of the crosshead movement to maintain an equilibrium load.

Figure 12. Schematic Diagram of Tinius Olsen Testing Machine

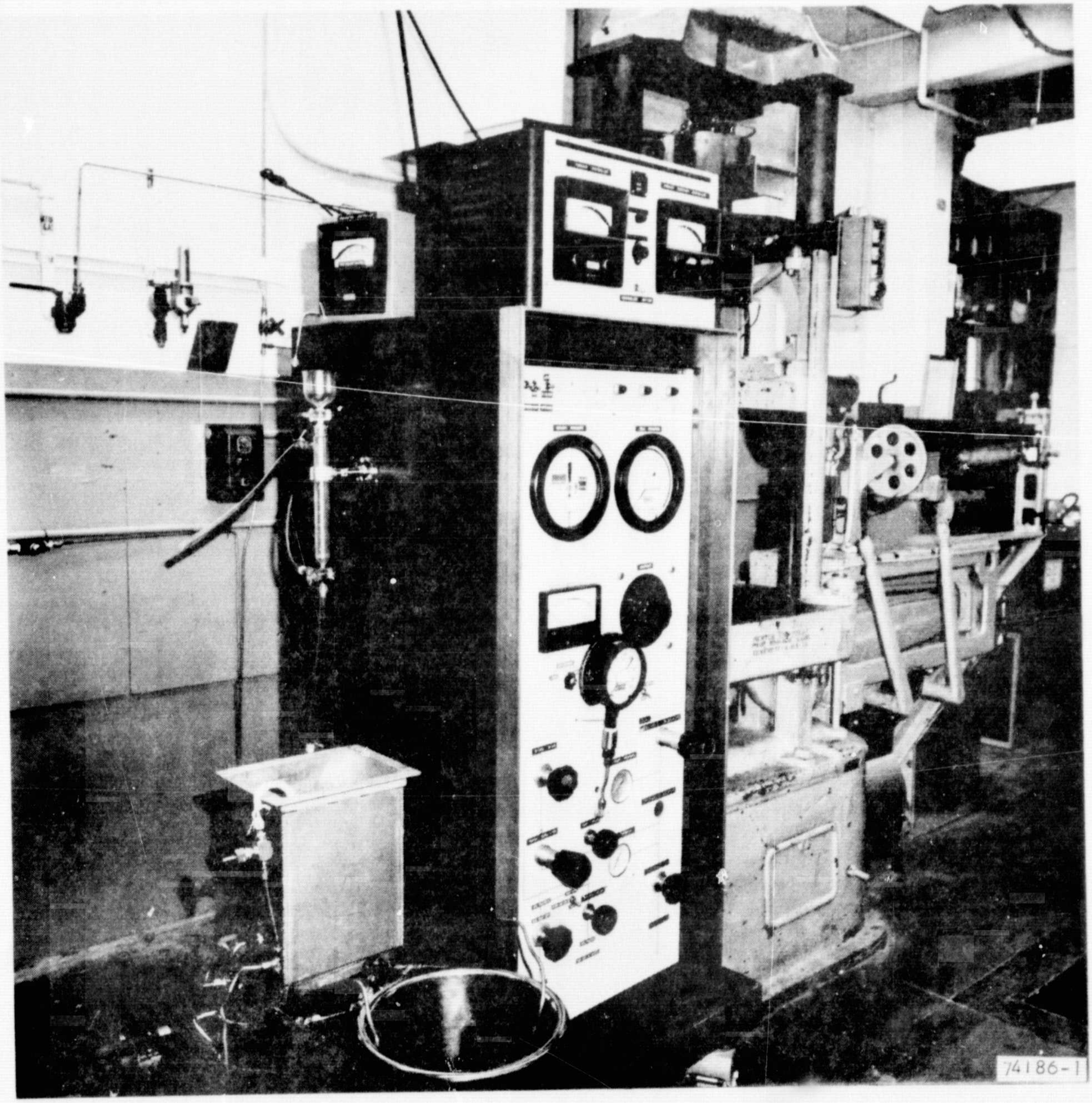


Figure 13. Tinius Olsen Testing Machine Used for the Fracture Mechanics Experiments

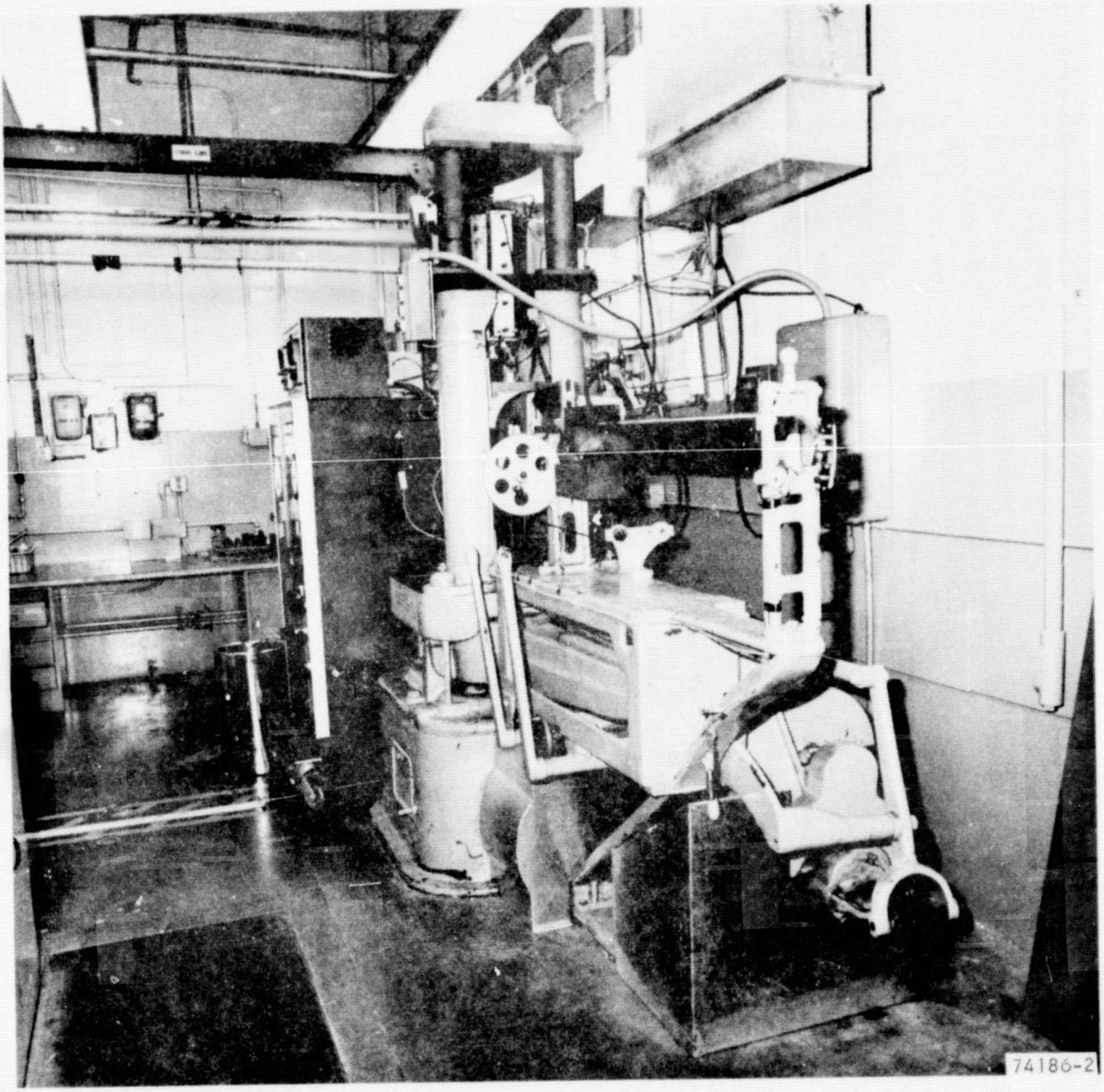


Figure 14. Tinius Olsen Testing Machine, Used for the Fracture Mechanics Experiments

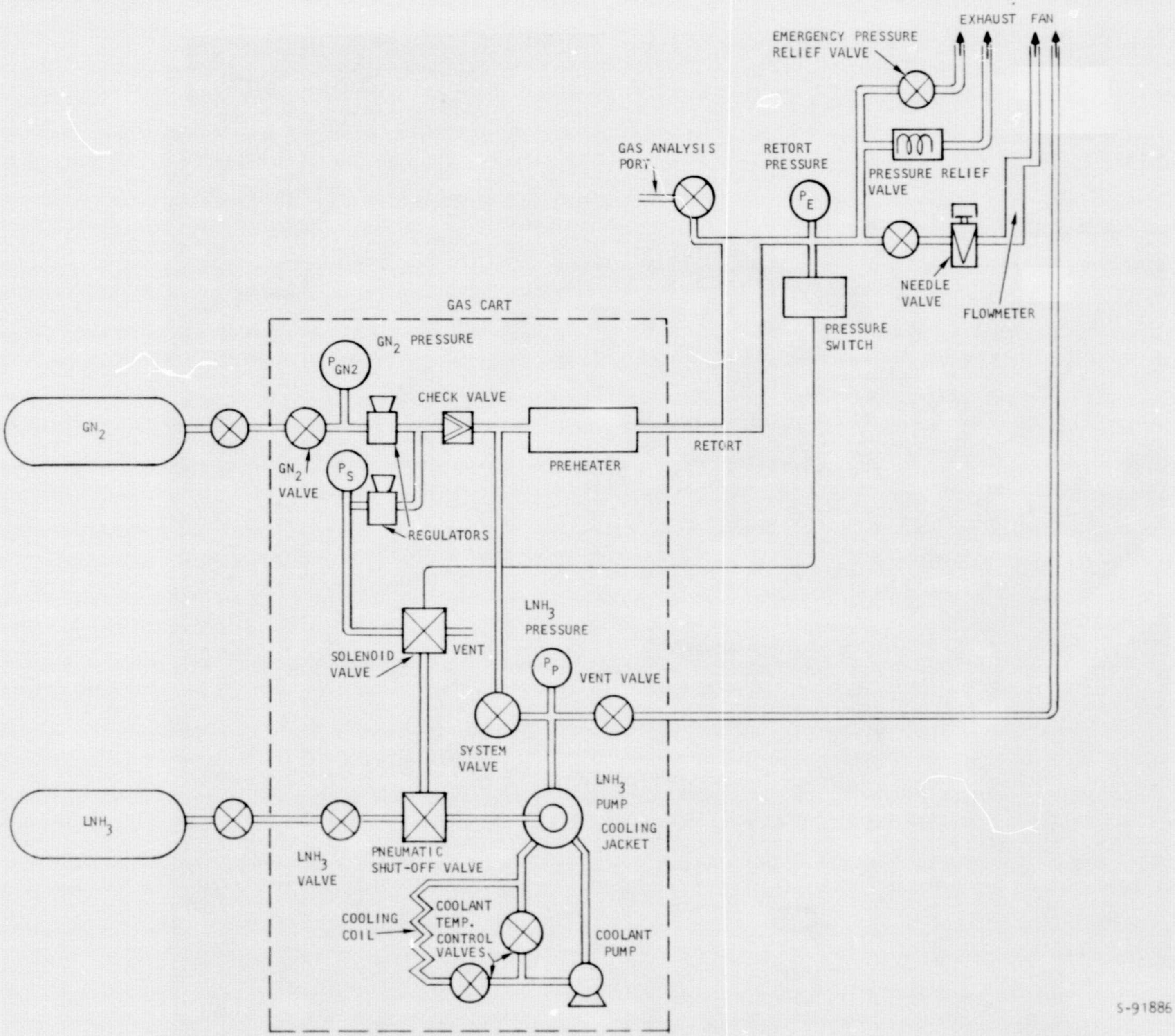


Figure 15. Gas Cart and Retort Schematic

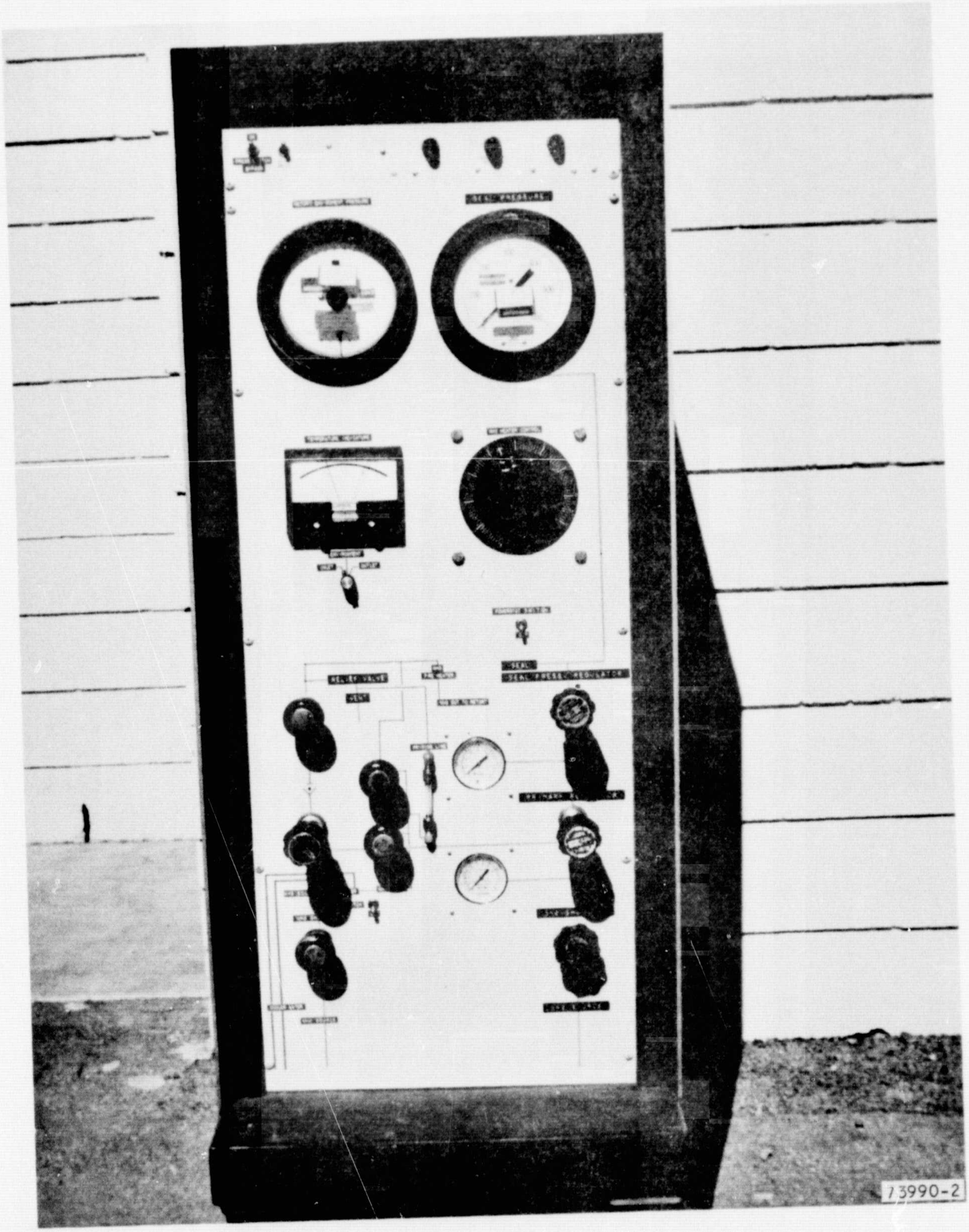


Figure 16. Gas Cart Showing Control Panel

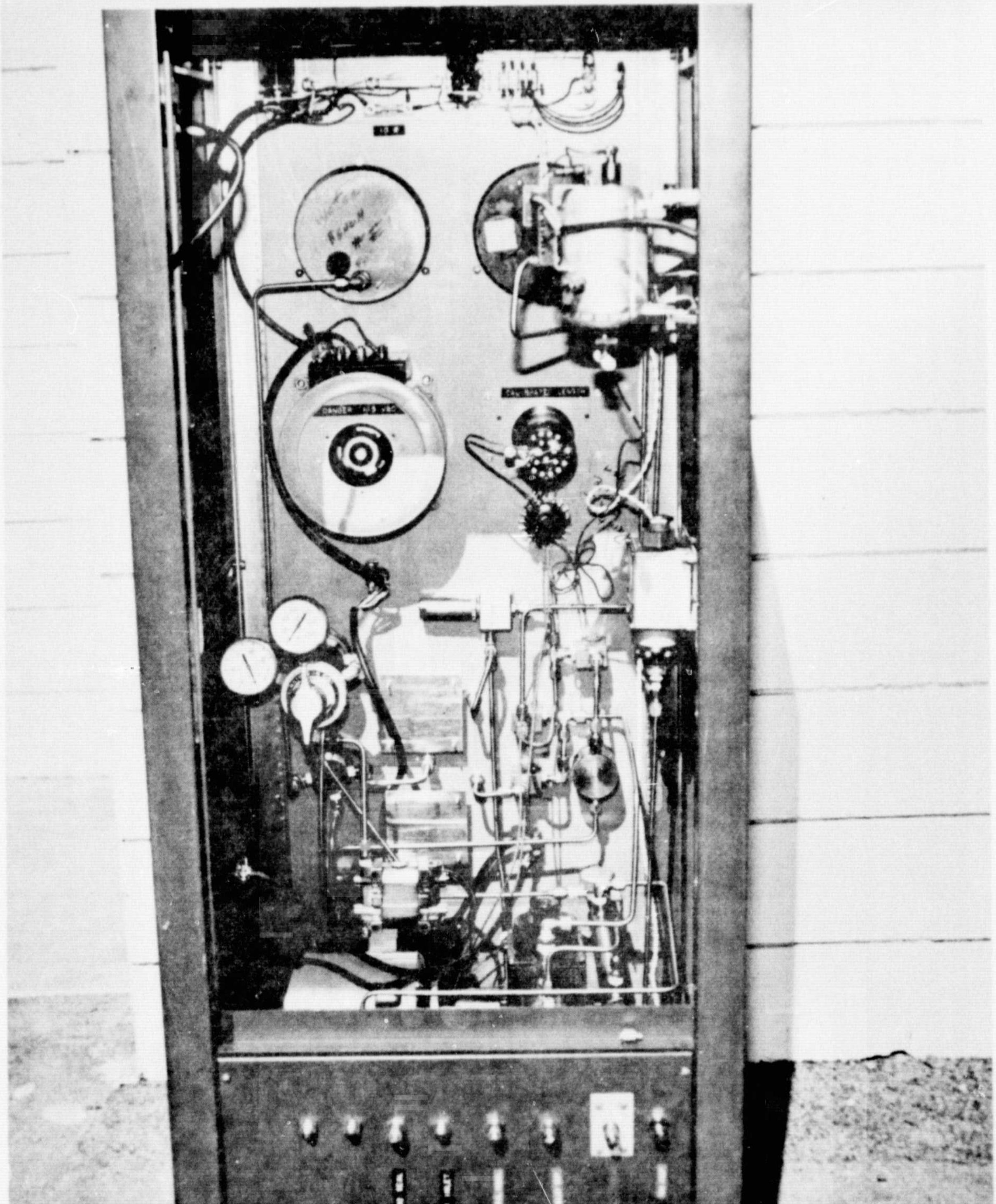


Figure 17. Gas Cart Showing Internal Mechanism

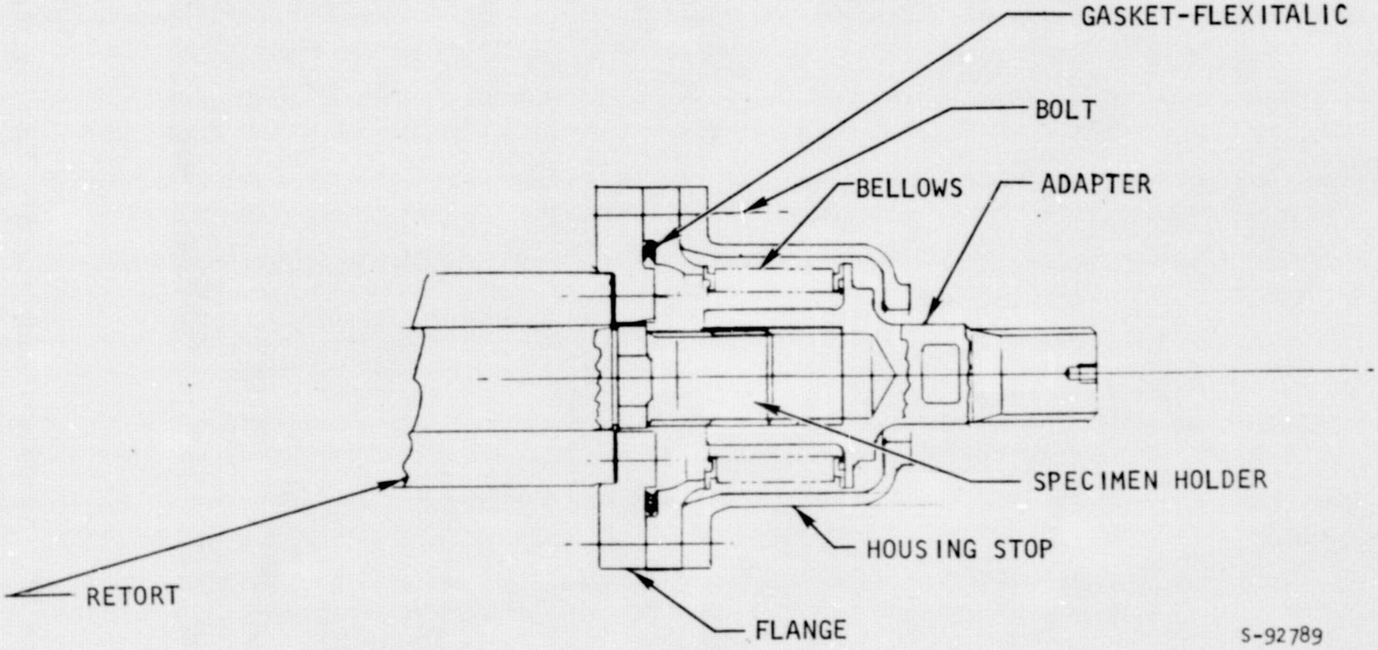


Figure 18. Diagram Showing Arrangement of Bellows Seal

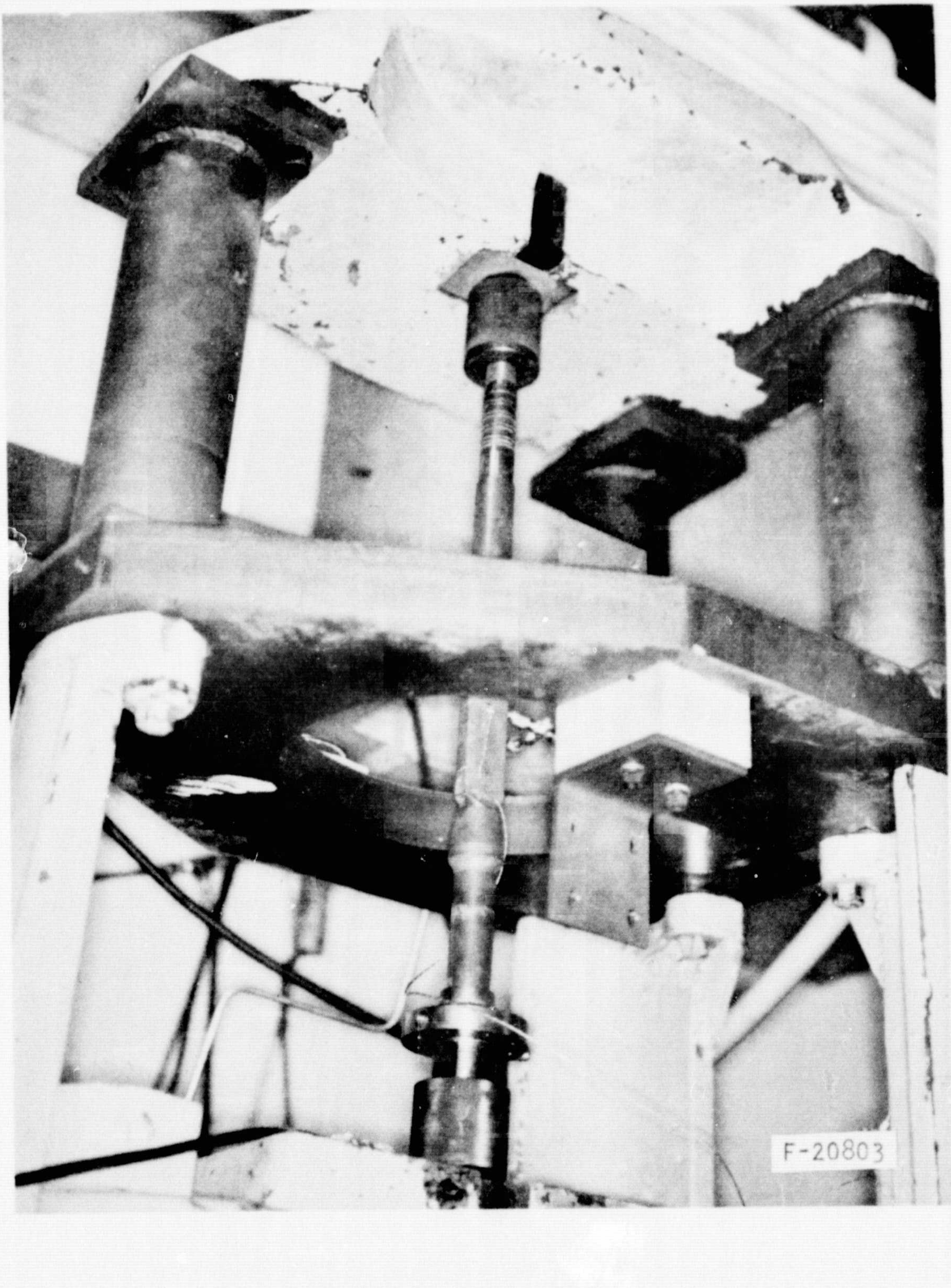


Figure 19. Specimen Ready for Air Testing (Furnace has been removed for clarity).

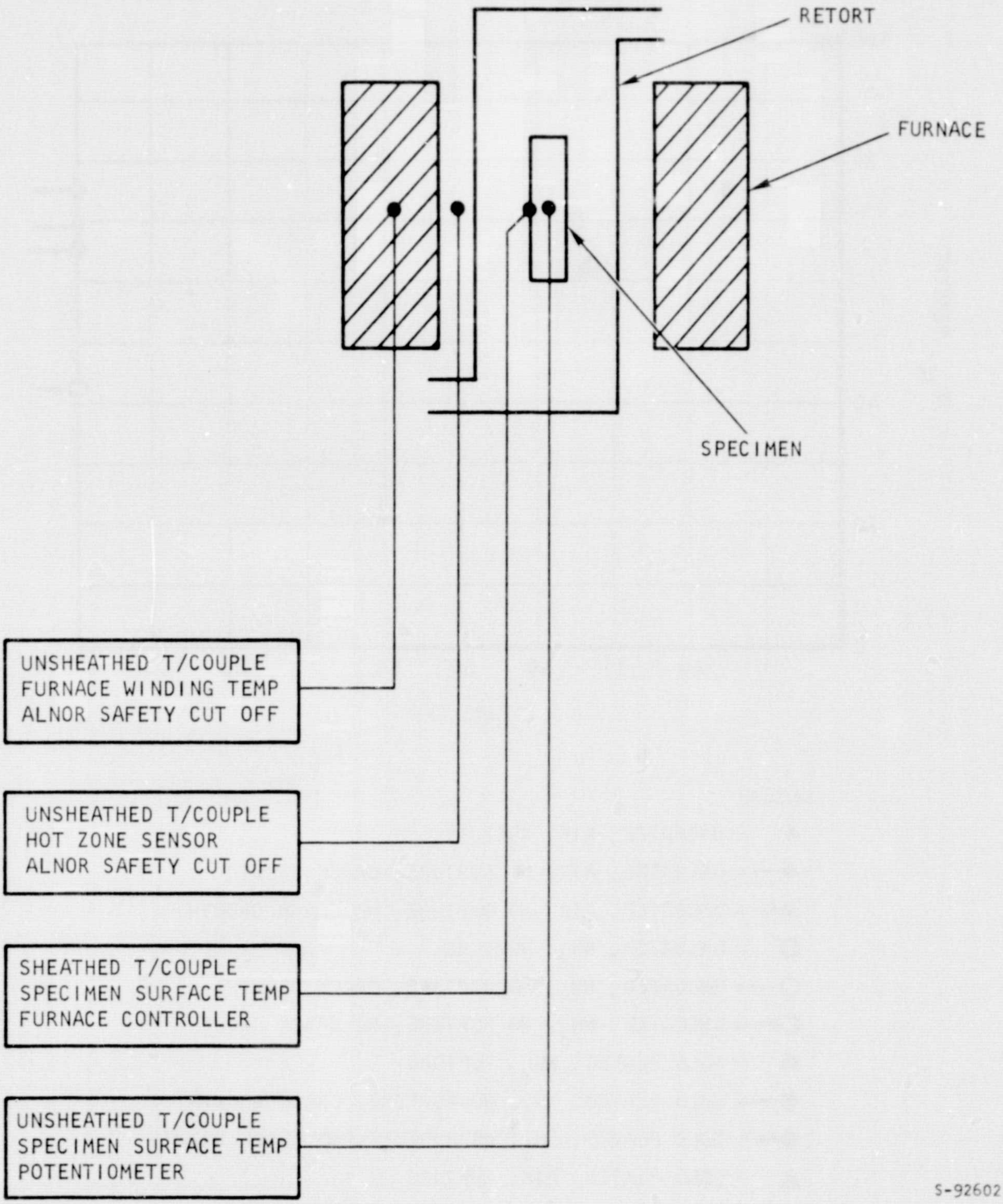


Figure 20. Schematic Diagram Showing Location of Thermocouples in Fracture Mechanics and Fatigue Tests

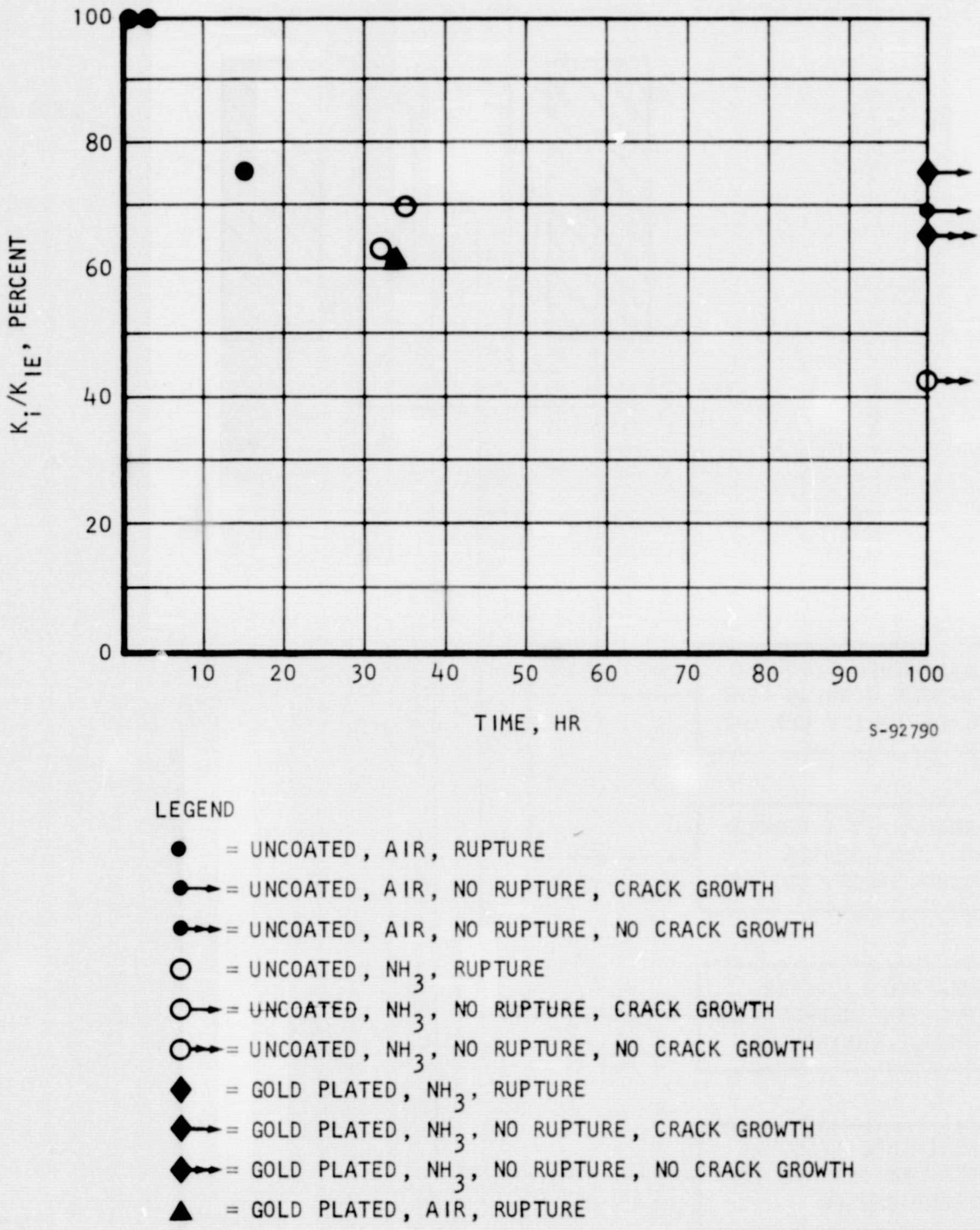
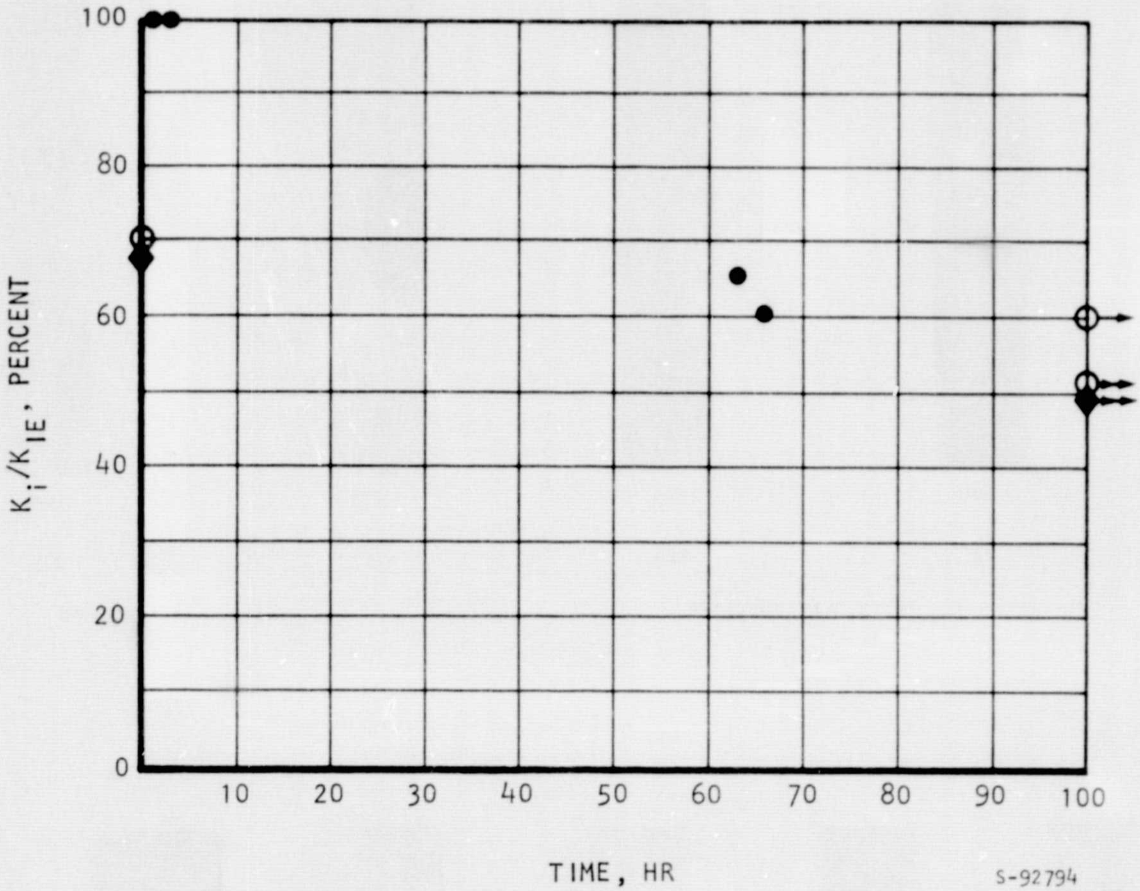


Figure 21. K_i/K_{IE} Values for Rene' 41



LEGEND

● = UNCOATED, AIR, RUPTURE

●→ = UNCOATED, AIR, NO RUPTURE, CRACK GROWTH

●→→ = UNCOATED, AIR, NO RUPTURE, NO CRACK GROWTH

○ = UNCOATED, NH_3, RUPTURE

○→ = UNCOATED, NH_3, NO RUPTURE, CRACK GROWTH

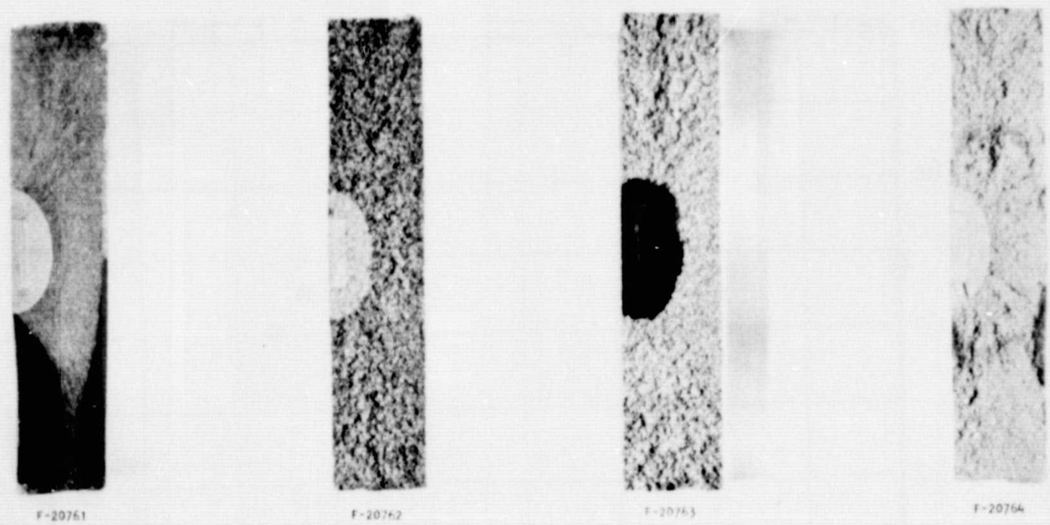
○→→ = UNCOATED, NH_3, NO RUPTURE, NO CRACK GROWTH

◆ = GOLD PLATED, NH_3, RUPTURE

◆→ = GOLD PLATED, NH_3, NO RUPTURE, NO CRACK GROWTH

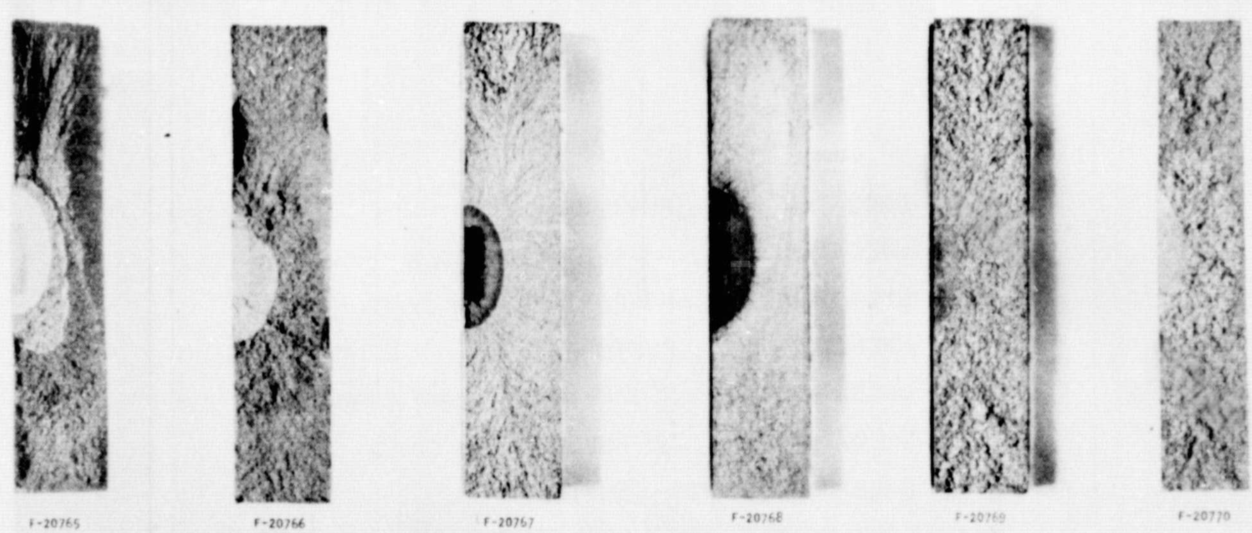
◆→→ = GOLD PLATED, NH_3, NO RUPTURE, CRACK GROWTH

Figure 22. K_i/K_{IE} Values for Astroloy



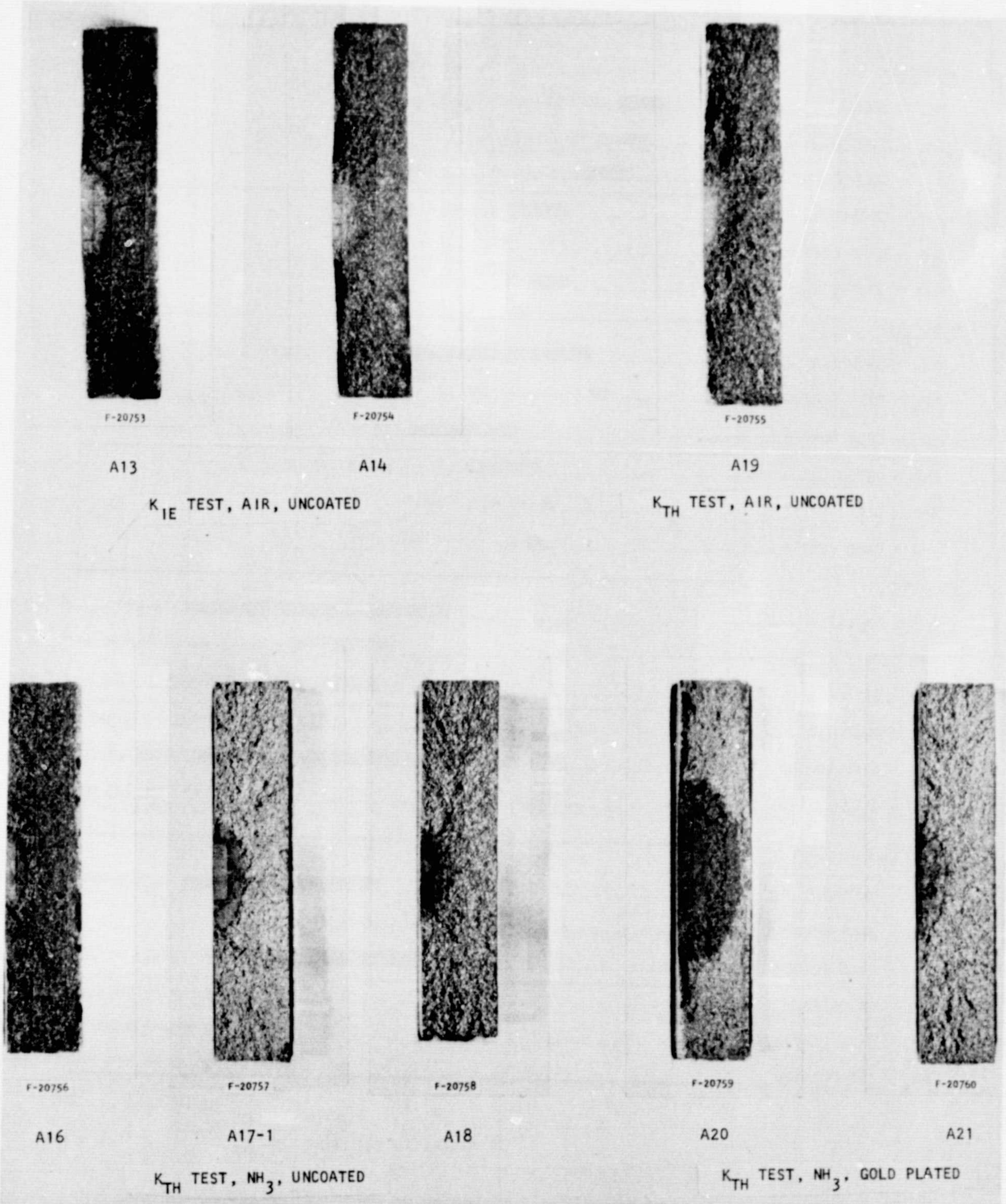
R23 R24 R25 R29

K_{IE} TESTS, AIR, UNCOATED K_{TH} TESTS, AIR, UNCOATED



F-20805

Figure 23. Fracture Surfaces of Rene' 41 K_{IE} and K_{TH} Test Specimens



F-20806

Figure 24. Fracture Surfaces of Astroloy K_{IE} and K_{TH} Test Specimens

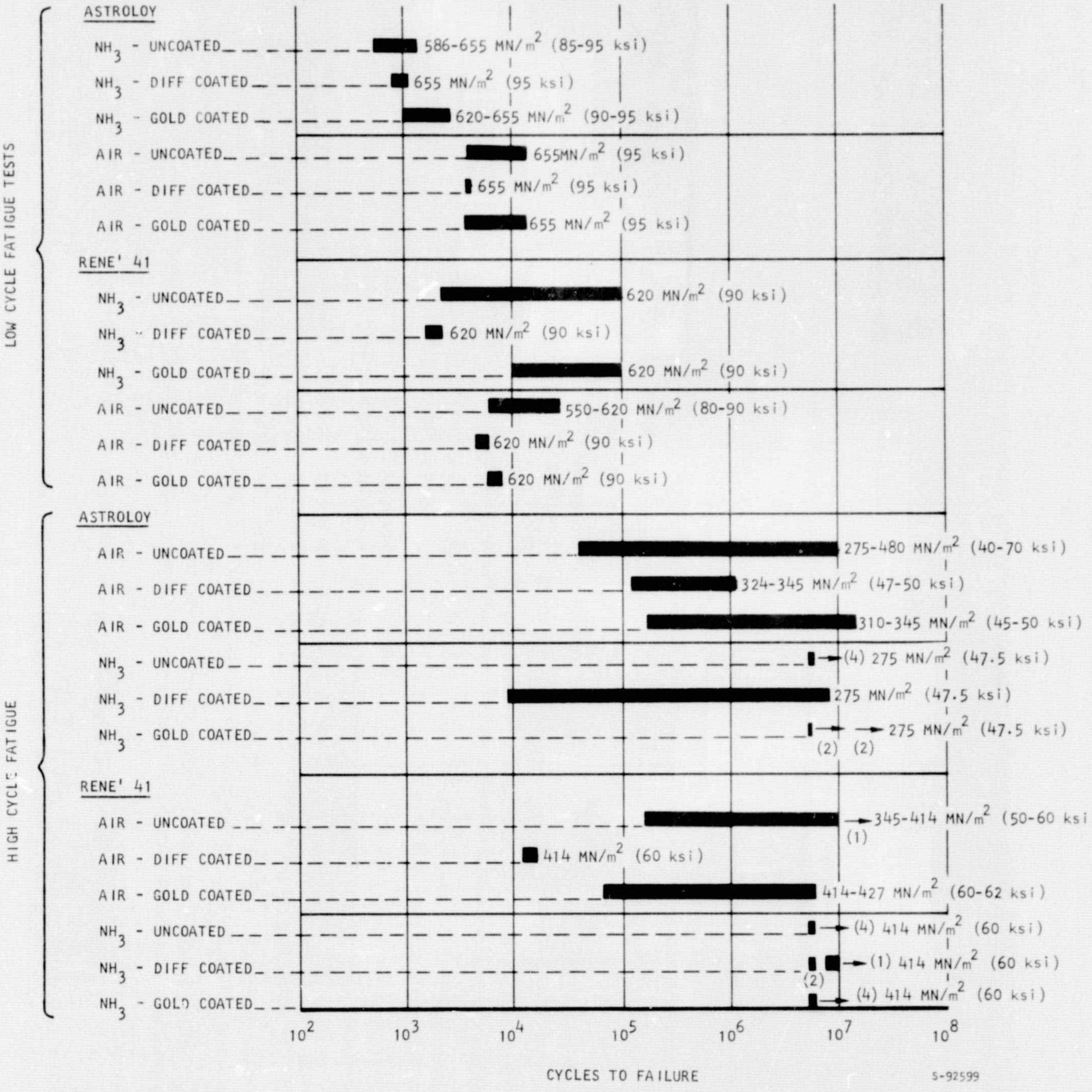


Figure 25. Histogram Showing Relative Fatigue Life for The Various Combinations Tested

Figure 26. Typical Fatigue Fracture Surfaces
Rene' 41 Specimens

Figure 27. Typical Fatigue Fracture Surfaces
Astroloy Specimens

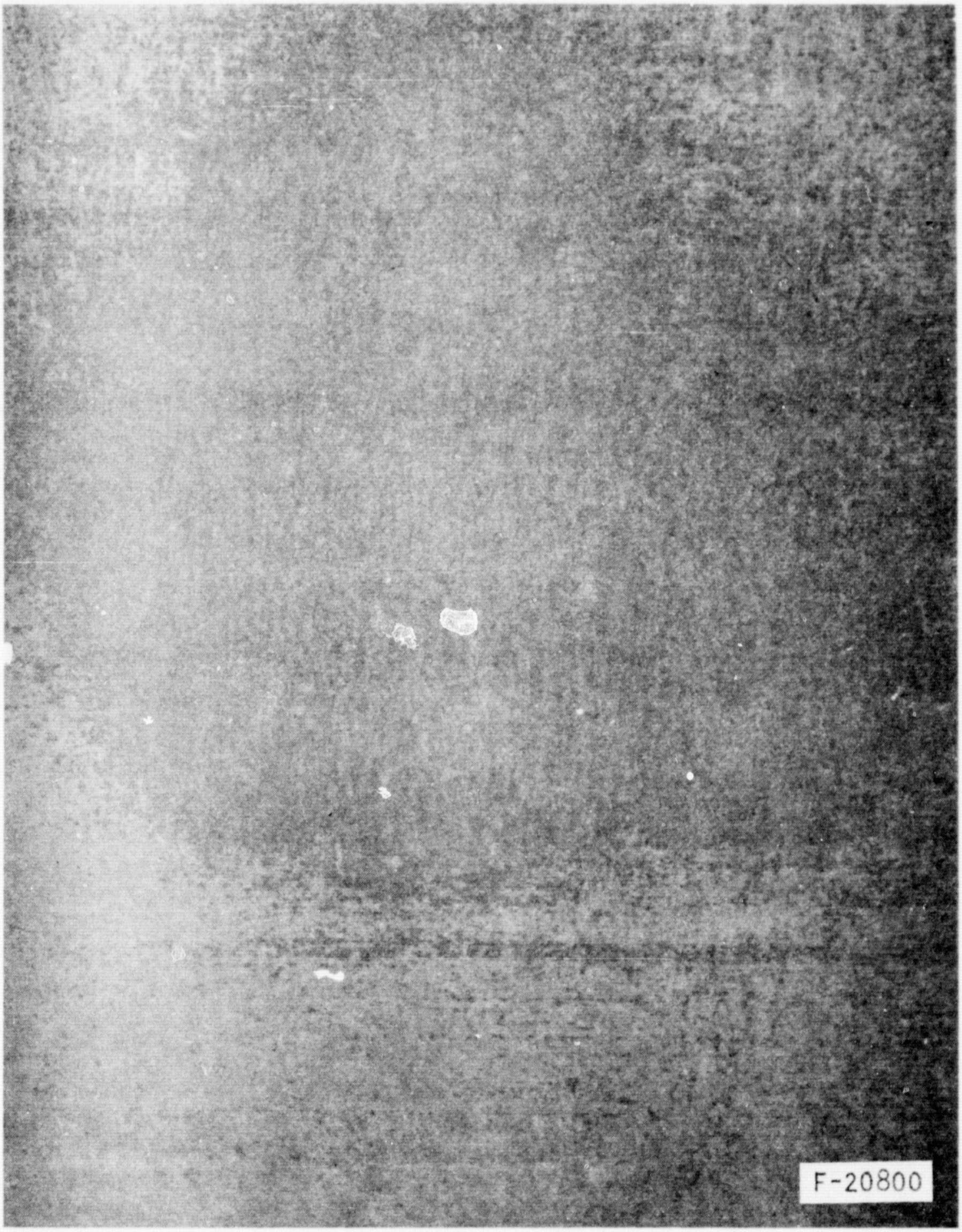


Figure 28. Specimen R32, Gold Plated, Before Testing. The gold plating has so covered the fissure that it has become almost invisible.

Figure 29. Specimen R32, Gold Plated, Tested in Ammonia. The gold plating over the fissure had cracked, but no appreciable crack hardening was detected. Subsequent rupturing revealed that no crack growth had taken place.

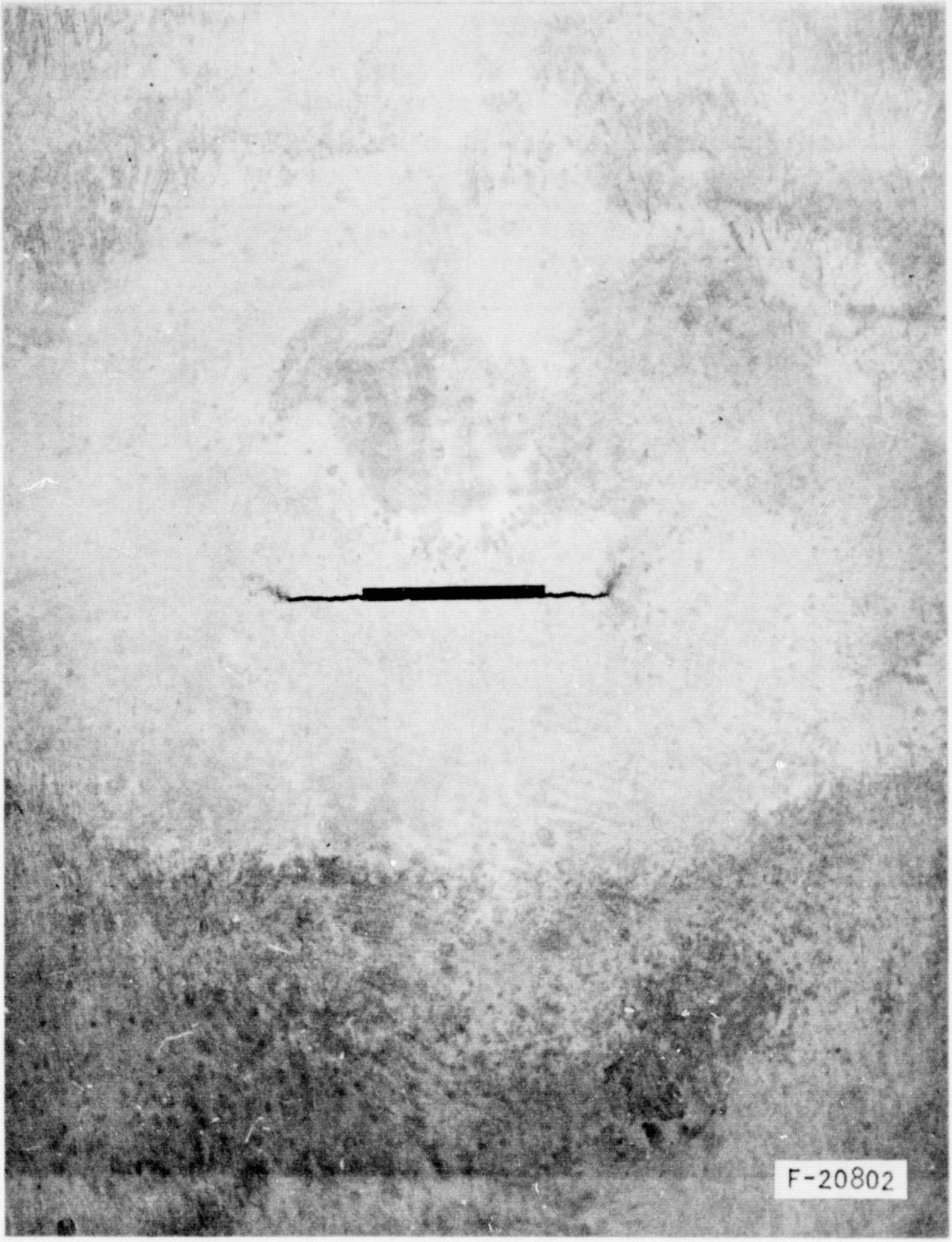


Figure 30. Specimen R25 Uncoated, Tested in Ammonia. There is evidence of crack widening. Subsequent fracturing revealed that the sample had experienced crack growth during testing.

TABLE I. - CHEMICAL ANALYSIS OF MATERIALS USED

Material	Astroloy		Rene' 41
Size	19.2 mm (0.756 in.) dia	75.8 mm (2.985 in.) dia	All Sizes
Heat Nº	8-3155	8.3154	9-1855
C	0.08	0.06	0.09
Mn	< 0.10	< 0.10	< 0.10
Si	< 0.10	< 0.10	< 0.10
Cr	14.9	15.2	18.8
Ni	bal	bal	bal
Co	17.2	17.0	10.9
Fe	0.62	0.18	0.71
Mo	4.60	5.00	10.2
Ti	3.20	3.55	3.25
Al	4.30	4.10	1.52
B	0.028	0.027	0.004
Z	< 0.04	< 0.04	
S	0.003	0.003	0.003
P	-	< 0.001	-
Cu	< 0.10	< 0.10	

TABLE II. - Age Hardening Procedures for Test Specimens

Material	Temperature	Time	Atmosphere
Rene' 41	1030 K (1400°F)	16 hr	Vacuum
Astroloy[1]	1145 K (1600°F)	8 hr	Air
	1255 K (1800°F)	4 hr	Air
	920 K (1200°F)	24 hr	Air
	1030 K (1400°F)	8 hr	Air

[1] Specimens were rapid air cooled to room temperature between each treatment.

TABLE III. - SCHEDULE OF FRACTURE MECHANICS TESTING

Environment	Number of Specimens	
	Astroloy	Rene' 41
950 K (1250°F), Air, uncoated K_{IE}	2	2
950 K (1250°F), Air, uncoated, K_{TH}	2	2
950 K (1250°F), Wet NH_3, uncoated, K_{TH}	3	3
950 K (1250°F), Wet NH_3, gold plated, K_{TH}	3	3

During the course of the experiments, it was agreed that one of the gold plated Rene' 41 specimens should be tested in air, instead of in ammonia.

TABLE IV. - SCHEDULE OF FATIGUE TESTING

Environment		Number of Specimens	
		Astroloy Bar	Rene' 41 Bar
Wet NH_3, uncoated	LCF	4	4
Air, uncoated	LCF	4	4
Wet NH_3, coating A	LCF	4	4
Air, coating A	LCF	4	4
Wet NH_3, coating B	LCF	4	4
Air, coating B	LCF	4	4
Wet NH_3, uncoated	HCF	4	4
Air, uncoated	HCF	4	4
Wet NH_3, coating A	HCF	4	4
Air, coating A	HCF	4	4
Wet NH_3, coating A	HCF	4	4
Air, coating B	HCF	4	4

TABLE V. - SEARCH FOR OPTIMUM LOAD FOR ASTROLOY HCF TEST

Specimen Number	Max Stress MN/m^2 (ksi)	Remarks
A93	320 (46.5)	Failed 3.82×10^4 cycles
A94	230 (33.3)	Failed in threads after 4.23×10^5 cycles
A102	230 (33.3)	Failed 1.64×10^5 cycles
A103	206 (30.0)	Failed 1.10×10^6 cycles
A96	230 (33.3)	Failed in threads after 1.60×10^6 cycles
A95	180 (26.2)	No failure after 1×10^7 cycles

TABLE VI. - SEARCH FOR OPTIMUM LOAD FOR ASTROLOY LCF TESTS

Specimen Number	Max Stress MN/m^2 (kps)	Remarks
A45	275 (40.0)	No cracks after 10^4 cycles
	293 (42.5)	No cracks after 10^4 cycles
	310 (45.0)	No cracks after 10^4 cycles
	327 (47.5)	No cracks after 10^4 cycles
	344 (50.0)	No cracks after 10^4 cycles
	379 (55.0)	No cracks after 10^4 cycles
	413 (60.0)	No cracks after 10^4 cycles
	448 (65.0)	No cracks after 10^4 cycles
	482 (70.0)	No cracks after 10^4 cycles
	517 (75.0)	No cracks after 10^4 cycles
	551 (80.0)	No cracks after 10^4 cycles
	586 (85.0)	Failed after 3.63×10^3 cycles
A46	586 (85.0)	Failed after 1.44×10^4
A54	603 (87.5)	Cracked at 9.5×10^3 cycles
	620 (90.0)	Failed after 2.9×10^2 cycles
A53	654 (95.0)	Failed after 3.89×10^3 cycles
A47	654 (95.0)	Failed after 4.71×10^3 cycles

TABLE VII. - SEARCH FOR OPTIMUM LOAD FOR RENE' 41 HCF TESTS

Specimen Number	Max Stress MN/m^2 (ksi)	Remarks
R117	310 (45)	No cracks after 3.07×10^6 cycles
	344 (50)	No cracks after 1×10^6 cycles
	379 (55)	Failed after 1.79×10^4 cycles
R118	344 (50)	No failure after 1.0×10^7 cycles
R119	413 (60)	Failed in 3.27×10^5 cycles

TABLE VIII. - SEARCH FOR OPTIMUM LOAD FOR RENE' 41 LCF TESTS

Specimen Number	Max Stress MN/m^2 (ksi)	Remarks
R69	482 (70)	No cracks after 1×10^4 cycles
	517 (75)	No cracks after 1×10^4 cycles
	551 (80)	Failed after 6.1×10^3 cycles
R70	586 (85)	No cracks after 1×10^4 cycles
	620 (90)	Failed after 6.4×10^2 cycles

TABLE IX

RESULTS OF FRACTURE MECHANICS TESTING AT 950K (SI UNITS)

Specimen Number	Material	Coating	Atmos-phere[1]	Type of Test	Load P (MN)	Width W (mm)	Thickness B (mm)	Crack Length 2c (mm)	Crack Depth a (mm)	Stress σ (MN/m^2)	Yield σ_y (MN/m^2)	Q	M_k	K_{IE} ($MN/m^{3/2}$)	K_i ($MN/m^{3/2}$)	K_i/K_{IE} (%)	Time on Test (hr)	Remarks
A13	Astroloy	Uncoated	Air	K_{IE}	274	37.97	7.95	9.53	2.68	907	885	1.42	1.03	79.1		-	0.0	Intergranular rupture
A14					293	38.05	7.67	9.65	2.54	1005	947	1.28	1.03	90.1		-	0.0	Intergranular rupture
A15				K_{TH}	-	-	-	-	-	-	-	-	-	-		-	-	Accidently failed at 70°F[3]
A19					178	38.23	7.90	9.91	3.05	589	285	1.59	1.04		52.4	62	67.6	Intergranular rupture
A16			NH_3	K_{TH}	<205	37.85	7.98	9.27	2.54	<680	874	1.36	1.03		59.2	<70	0.0	Failed on loading[4]
A17-1					137	38.10	7.62	9.14	2.79	471	874	1.36	1.03		43.0	50	>100	No rupture, no crack growth
A18					165	35.71	7.85	9.14	2.29	587	874	1.36	1.02		50.3	60	>100	No rupture, crack growth ~ .64mm (.025 in)
A20		18μm (0.7 mil) gold	NH_3	K_{TH}	165	38.10	7.52	12.95	3.30	574	830	1.36	1.05		57.8	68.5	.01	Intergranular rupture
A21					137	38.30	7.62	11.18	2.79	469	830	1.42	1.04		42.2	50	>100	No rupture, no crack growth
A22					150		-	-	-	-	-	-	-	-		-	-	Failed in threads
R23	Rene 41	Uncoated	Air	K_{IE}	253	38.12	7.95	9.65	3.18	833	900	1.56	1.03	75.6			0.0	Transgranular rupture
R24					215	38.05	7.82	9.91	3.43	723	789	1.62	1.04	67.5			0.0	Transgranular rupture
R25				K_{TH}	150	38.20	7.98	9.65	3.30	495	789	1.35	1.03		49.2	68.7	>100	No rupture, crack growth ~ 1,27mm (.050 in)
R29					183	38.12	7.98	9.53	3.05	601	789	1.56	1.035		53.9	75	16.1	Transgranular rupture
R26			NH_3	K_{TH}	150	38.12	7.85	9.78	3.18	504	941	1.68	1.045		44.7	62.5	33.7	Transgranular rupture
R27					161	38.18	7.85	10.16	3.56	539	941	1.74	1.04		49.5	70	36.6	Transgranular rupture
R28					107	38.18	7.98	9.65	3.18	353	941	1.68	1.03		30.8	43	>100	No rupture, no growth
R30		18μm (0.7 mil) gold	NH_3	K_{TH}	161	38.10	7.62	11.18	3.30	556	978	1.52	1.06		53.7	75	>100	No rupture, crack growth ~ .25mm (.010 in)
R32					150	38.10	7.57	10.41	2.29	559	9.78	1.30	1.02		46.75	65	>100	No rupture, no growth
R31			Air	K_{TH}	161	38.05	7.62	9.65	2.03	557	978	1.30	1.02		43.8	61.3	33.6	Transgranular rupture

$K_{IE} = 1.1\,\sigma\,M_K\sqrt{\pi a/Q}$

Mk = Magnification factor based on crack shape and specimen thickness
Q = Flow shape parameter based on crack shape and material strength
σ = Stress (MN/m^2) applied to specimen
a = Crack depth (mm)

σ_y = Yield strength measured on tensile bars machined from failed fracture mechanics specimens numbers A13, A14, A16, A20, R23, R24, R26 and R30

Notes:

1. Air tests were conducted at ambient pressure; ammonia testing was at 3.4 MN/m^2. The ammonia contained 1.5% H_2O
2. Based on average K_{IE} values.
3. Specimen A15 was accidentally broken at room temperature while setting up the machine.
4. Specimen A16 failed before the intended load of 205 MN kips was reached. We have no record of the actual breaking load.

TABLE X

RESULTS OF FRACTURE MECHANICS TESTING AT 1250°F
(U.S. CUSTOMARY UNITS)

Specimen Number	Material	Coating	Atmos-phere (1)	Type of Test	Load P (kips)	Width W (in)	Thickness B (in)	Crack Length 2c (in)	Crack depth a (in)	Stress σ (ksi)	Yield σy (ksi)	Q	M_k	K_{IE} (ksi$\sqrt{in}$)	K_i (ksi$\sqrt{in}$)	K_i/K_{IE} (%)	Time on Test (hr)	Remarks
A13	Astroloy	Uncoated	Air	K_{IE}	61.6	1.495	.313	.375	.105	131.6	128.4	1.42	1.03	71.9		-	0.0	Intergranular rupture
A14					66.0	1.498	.302	.38	.10	145.9	137.5	1.28	1.03	81.9		-	0.0	Intergranular rupture
A15				K_{TH}	-	-	-	-	-	-	-	-	-	-	-	-	-	Accidently failed at 70°F (3)
A19					40.0	1.505	.311	.39	.12	85.5	128.4	1.59	1.04		47.6	62	67.6	Intergranular rupture
A16			NH_3	K_{TH}	<46.2	1.490	.314	.365	.10	<98.8	126.8	1.36	1.03		<53.8	<70	0.0	Failed on loading (4)
A17-1					30.8	1.500	.300	.36	.11	68.4	126.8	1.36	1.03		39.0	50	>100	No rupture, no crack growth
A18					37.0	1.406	.309	.36	.09	85.2	126.8	1.36	1.02		45.7	60	>100	No rupture, crack growth ~ .64mm (.025 in)
A20		18μm (0.7 mil) gold	NH_3	K_{TH}	37.0	1.500	.296	.51	.13	83.3	120.4	1.36	1.05		52.7	68.5	.01	Intergranular rupture
A21					30.8	1.508	.300	.44	.11	68.0	120.4	1.42	1.04		33.4	50	>100	No rupture, no crack growth
A22					33.9	-	-	-	-	-	-	-	-	-	-	-	-	Failed in threads
R23	Rene 41	Uncoated	Air	K_{IE}	56.8	1.501	.313	.38	.125	120.9	130.6	1.56	1.03	68.7			0.0	Transgranular rupture
R24					48.4	1.498	.308	.39	.135	104.9	114.5	1.62	1.04	61.4			0.0	Transgranular rupture
R25				K_{TH}	33.9	1.504	.314	.38	.130	71.8	114.5	1.35	1.03		44.7	68.7	>100	No rupture, crack growth ~ 1.27mm (.050 in)
R29					41.1	1.501	.314	.375	.12	87.2	114.5	1.56	1.035		49.0	75	16.1	Transgranular rupture
R26			NH_3	K_{TH}	33.9	1.501	.309	.385	.125	73.1	136.6	1.68	1.045		40.6	62.5	33.7	Transgranular rupture
R27					36.3	1.503	.309	.40	.14	78.2	136.6	1.74	1.04		45.0	70	36.6	Transgranular rupture
R28					24.2	1.503	.314	.38	.125	51.2	136.6	1.68	1.03		28.0	43	>100	No rupture, no growth
R30		18μm (0.7 mil) gold	NH_3	K_{TH}	36.3	1.500	.300	.44	.13	80.7	142.0	1.52	1.06		48.8	75	>100	No rupture, crack growth ~ .25mm (.010 in)
R32					33.9	1.500	.298	.41	.09	81.2	142.0	1.30	1.02		42.5	65	>100	No rupture, no growth
R31			Air	K_{TH}	36.3	1.498	.300	.38	.08	80.8	142.0	1.30	1.02		39.85	61.3	33.6	Transgranular rupture

$K_{IE} = 1.1\,\sigma\,M_K\sqrt{\pi a/Q}$

Mk = Magnification factor based on crack shape and specimen thickness
Q = Flow shape parameter based on crack shape and material strength
σ = Stress (ksi) applied to specimen
a = Crack depth (in)

σy = Yield strength measured on tensile bars machined from failed fracture mechanics specimens numbers A13, A14, A16, A20, R23, R24, R26 and R30

Notes:

1. Air tests were conducted at ambient pressure; ammonia testing was at 500 psi. The ammonia contained 1.5% H_2O
2. Based on average K_{IE} values.
3. Specimen A15 was accidentally broken at room temperature while setting up the machine.
4. Specimen A16 failed before the intended load of 46.2 kips was reached. We have no record of the actual breaking load.

TABLE XI

HIGH CYCLE FATIGUE TEST RESULTS FOR ASTROLOY AT 950 K (SI UNITS)

Specimen Number	Coating	Atmos-phere[1]	Notch Dimensions D1 mm	D2 mm	R mm	Stress Concentration Factor	Load[2] Maximum N	Minimum N	Nominal Stress at Notch Maximum MN/m^2	Minimum MN/m^2	Cycles to Failure Crack Initiation	Rupture	Time at Temperature Under Load Hour
A81	Uncoated	NH_3	8.146	6.617	0.23	3.3	11263	1126	328	32.8	None	5.00×10^6	21.5
A82	Uncoated	NH_3	8.164	6.642	0.23	3.3	11263	1126	328	32.8	None	5.00×10^6	21.5
A83	Uncoated	NH_3	8.179	6.629	0.23	3.3	11165	1116	328	32.8	None	5.00×10^6	21.5
A84	Uncoated	NH_3	8.156	6.617	0.20	3.4	11218	1122	328	32.8	None	5.00×10^5	21.5
A85	Diffusion	NH_3	8.207	6.655	0.20	3.4	11218	1122	328	32.8	6.52×10^3	8.65×10^3	0.17
A86	Diffusion	NH_3	8.214	6.655	0.20	3.4	11218	1122	328	32.8	6.95×10^3	1.10×10^3	0.20
A87	Diffusion	NH_3	8.209	6.642	0.20	3.4	11218	1122	328	32.8	4.03×10^3	7.35×10^3	0.14
A88	Diffusion	NH_3	8.207	6.629	0.31	3.0	11125	1113	328	32.8	4.30×10^3	8.09×10^5	0.16
A89	Gold Plate	NH_3	8.169	6.629	0.20	3.4	11329	1133	328	32.8	None	1.00×10^7	47.5
A90	Gold Plate	NH_3	8.164	6.629	0.23	3.3	11236	1124	328	32.8	None	1.00×10^7	47.5
A91	Gold Plate	NH_3	8.169	6.629	0.23	3.3	11272	1127	328	32.8	None	5.00×10^6	21.5
A92	Gold Plate	NH_3	8.133	6.617	0.20	3.4	17263	1726	328	32.8	None	5.00×10^6	21.5
A93	Uncoated	Air	8.158	6.629	0.23	3.3	12210	1221	432	43.2	2.38×10^4	3.82×10^4	2.58
A94[3]	Uncoated	Air	8.166	6.617	0.23	3.3	11854	1185	345	34.5	3.20×10^5	4.23×10^5	3.88
A95	Uncoated	Air	8.166	6.579	0.23	3.3	9367	937	276	27.6	None to	1.00×10^7	93.73
A96[3]	Uncoated	Air	8.156	6.604	0.23	3.3	11854	1185	345	34.5	---	1.60×10^6	20.00
A97	Diffusion	Air	8.194	6.642	0.28	3.0	11850	1185	345	34.5	8.54×10^5	8.71×10^5	4.83
A98	Diffusion	Air	8.194	6.642	0.25	3.2	11213	1121	324	32.4	1.08×10^6	1.11×10^6	13.88
A99	Diffusion	Air	8.189	6.629	0.28	3.1	11213	1121	324	32.4	1.25×10^5	1.35×10^5	1.83
A100	Diffusion	Air	8.197	6.642	0.25	3.2	11213	1121	324	32.4	1.02×10^5	1.16×10^5	2.67
A101	Gold Plate	Air	8.197	6.642	0.20	3.4	11218	1122	324	32.4	5.71×10^6	5.73×10^6	29.33
A102	Gold Plate	Air	8.204	6.655	0.20	3.4	11854	1185	345	34.5	1.57×10^5	1.64×10^5	3.00
A103	Gold Plate	Air	8.199	6.642	0.25	3.2	10698	1070	310	31.0	1.03×10^6	1.19×10^6	12.00
A104	Gold Plate	Air	8.489	6.642	0.23	3.3	10662	1066	310	31.0	None to	1.42×10^7	71.83

NOTES: 1 Air tests were at ambient pressure; ammonia tests at 3.4 MN/m^2. The ammonia contained 1.5 percent H_2O.

2 Stress Ratio = 0.1

3 Failed in threads.

TABLE XII

LOW CYCLE FATIGUE TEST RESULTS FOR ASTROLOY AT 950 K
(SI UNITS)

Specimen Number	Coating	Atmosphere[1]	Notch Dimensions D1 mm	D2 mm	R mm	Stress Concentration Factor	Load[2] Maximum N	Minimum N	Nominal Stress at Notch Maximum MN/m^2	Minimum MN/m^2	Cycles to Failure Crack Initiation	Rupture	Time at Temperature Under Load Hour
A33	Uncoated	NH_3	8.164	6.617	0.25	3.2	22521	2252	655	65.5	8.70×10^2	1.23×10^3	10
A34	Uncoated	NH_3	8.179	6.637	0.23	3.3	22628	2263	655	65.5	5.90×10^2	8.00×10^2	5
A35	Uncoated	NH_3	8.148	6.612	0.23	3.3	22593	2259	655	65.5	4.50×10^2	5.40×10^2	5
A36	Uncoated	NH_3	8.146	6.629	0.23	3.3	22593	2259	655	65.5	6.40×10^2	7.90×10^2	5
A37	Diffusion	NH_3	8.209	6.655	0.20	3.4	22437	2244	655	65.5	5.80×10^2	8.20×10^2	5
A38	Diffusion	NH_3	8.199	6.642	0.23	3.3	22437	2244	655	65.5	6.00×10^2	7.40×10^2	10
A39	Diffusion	NH_3	8.209	6.642	0.20	3.4	22347	2235	655	65.5	4.60×10^2	8.00×10^2	55
A40	Diffusion	NH_3	8.219	6.615	0.20	3.4	22486	2249	655	65.5	6.60×10^2	1.06×10^3	10
A41	Gold Plate	NH_3	8.141	6.642	0.20	3.4	22641	2264	655	65.5	2.28×10^3	2.61×10^3	20
A42	Gold Plate	NH_3	8.158	6.629	0.23	3.3	22539	2254	655	65.5	7.10×10^2	9.70×10^2	10
A43	Gold Plate	NH_3	8.156	6.629	0.23	3.3	22574	2257	655	65.5	1.03×10^3	1.38×10^3	10
A44	Gold Plate	NH_3	8.164	6.629	0.23	3.3	22610	2261	655	65.5	8.90×10^2	1.23×10^3	15
A45	Uncoated	Air	8.166	6.617	0.23	3.3			586	58.6	1.88×10^3	3.63×10^3	92
A46	Uncoated	Air	8.156	6.617	0.23	3.3	20195	2020	586	58.6	5.25×10^3	1.44×10^4	70
A47	Uncoated	Air	8.176	6.617	0.25	3.2	22526	2253	655	65.5	2.02×10^3	4.71×10^3	105
A48	Uncoated	Air	8.171	6.617	0.25	3.2	22526	2253	655	65.5	5.29×10^3	8.39×10^3	90
A49	Diffusion	Air	8.186	6.604	0.25	3.2	22348	2235	655	65.5	2.06×10^3	3.64×10^3	150
A50	Diffusion	Air	8.181	6.604	0.25	3.2	22437	2244	655	65.5	2.54×10^3	3.69×10^3	150
A51	Diffusion	Air	8.219	6.629	0.25	3.2	22437	2244	655	65.5	2.18×10^3	3.49×10^3	50
A52	Diffusion	Air	NOT TESTED										
A53	Gold Plate	Air	8.169	6.629	0.23	3.3	22508	2251	655	65.5	2.02×10^3	3.89×10^3	40
A54	Gold Plate	Air	8.186	6.614	0.23	3.3	21262	2126	621	62.1	9.50×10^3	1.24×10^4	150
A55	Gold Plate	Air	8.204	6.629	0.23	3.3	22526	2253	655	65.5	1.90×10^3	3.82×10^3	90
A56	Gold Plate	Air	8.169	6.642	0.25	3.2	22526	2253	655	65.5	1.97×10^3	4.37×10^3	90

NOTES: 1 Air tests were at ambient pressure; ammonia tests at 3.4 MN/m^2
The ammonia contained 1.5 percent H_2O

2 Stress Ratio = 0.1

TABLE XIII

HIGH CYCLE FATIGUE TEST RESULTS FOR RENE' AT 950 K (SI UNITS)

Specimen Number	Coating	Atmos-phere[1]	Notch Dimensions D1 mm	D2 mm	R mm	Stress Concentration Factor	Load[2] Maximum N	Minimum N	Nominal Stress at Notch Maximum MN/m^2	Minimum MN/m^2	Cycles to Failure Crack Initiation	Rupture	Time at Temperature Under Load Hour
R105	Uncoated	NH_3	8.148	6.604	0.24	3.2	14172	1417	414	41.4	None	5.00×10^6	21.5
R106	Uncoated	NH_3	8.001	6.642	0.23	3.3	14225	1423	414	41.4	None	5.00×10^6	21.5
R107	Uncoated	NH_3	8.176	6.655	0.23	3.3	14172	1417	414	41.4	None	5.00×10^6	21.5
R108	Uncoated	NH_3	8.001	6.655	0.27	3.0	14225	1423	414	41.4	None	5.00×10^6	21.5
R109	Diffusion	Air	8.230	6.695	0.23	3.2	14501	1450	414	41.4	6.80×10^3	1.11×10^4	1.83
R110	Diffusion	Air	8.197	6.673	0.23	3.2	14172	1417	414	41.4	9.34×10^3	1.52×10^4	2.25
R111	Diffusion	Air	8.197	6.673	0.23	3.2	14225	1423	414	41.4	8.56×10^3	1.34×10^4	1.75
R112	Diffusion	Air	8.169	6.668	0.23	3.2	14172	1417	414	41.4	9.00×10^3	1.55×10^4	1.92
R113	Gold Plate	NH_3	8.146	6.520	0.28	3.0	14172	1417	414	41.4	None	5.00×10^6	21.5
R114	Gold Plate	NH_3	8.164	6.629	0.31	3.0	14172	1417	414	41.4	None	5.00×10^6	21.5
R115	Gold Plate	NH_3	8.125	6.528	0.25	3.2	14279	1428	414	41.4	None	5.00×10^6	21.5
R116	Gold Plate	NH_3	8.176	6.528	0.25	3.2	14279	1428	414	41.4	None	5.00×10^6	21.5
R117	Uncoated	Air	8.141	6.604	0.25	3.2	12989	1299	379	37.9	1.00×10^6	5.09×10^6	43.75
R118	Uncoated	Air	8.166	6.655	0.23	3.2	11992	1199	345	34.5	None to	1.00×10^7	40.00
R119	Uncoated	Air	8.166	6.617	0.23	3.2	14255	1423	414	41.4	3.27×10^5	3.28×10^5	2.17
R120	Uncoated	Air	8.153	6.604	0.31	3.0	14172	1417	414	41.4	1.55×10^5	1.62×10^5	2.67
R121	Diffusion	NH_3	8.222	6.670	0.20	3.4	14172	1417	414	41.4	6.57×10^6	6.85×10^6	32.0
R122	Diffusion	NH_3	8.214	6.680	0.24	3.2	14225	1423	414	41.4	None	1.00×10^7	47.5
R123	Diffusion	NH_3	8.197	6.657	0.23	3.3	14172	1417	414	41.4	None	5.00×10^6	21.5
R124	Diffusion	NH_3	8.217	6.655	0.28	3.1	14172	1417	414	41.4	None	5.00×10^6	21.5
R125	Gold Plate	Air	8.169	6.642	0.20	3.4	14225	1423	414	41.4	3.21×10^6	3.22×10^6	20.83
R126	Gold Plate	Air	8.158	6.629	0.28	3.0	14759	1476	427	42.7	No Data	4.23×10^5	6.67
R127	Gold Plate	Air	8.174	6.642	0.20	3.4	14225	1423	414	41.4	No Data	6.24×10^4	0.83
R128	Gold Plate	Air	8.176	6.629	0.25	3.2	14172	1417	414	41.4	5.88×10^6	5.93×10^6	66.50

NOTES: 1 Air tests were at ambinet pressure; ammonia tests at 3.4 MN/m^2
The ammonia contained 1.5 percent H_2O

2 Stress Ratio = 0.1

TABLE XIV

LOW CYCLE FATIGUE TEST RESULTS FOR RENE' 41 AT 950 K
(SI UNITS)

Specimen Number	Coating	Atmos-phere[1]	Notch Dimensions D1 mm	Notch Dimensions D2 mm	Notch Dimensions R mm	Stress Concentration Factor	Load[2] Maximum N	Load[2] Minimum N	Nominal Stress at Notch Maximum MN/m^2	Nominal Stress at Notch Minimum MN/m^2	Cycles to Failure Crack Initiation	Cycles to Failure Rupture	Time at Temperature Under Load Hour
R57	Uncoated	NH_3	8.171	6.629	0.28	3.0	21338	2134	621	62.1	None to	2.53×10^4	50
R58	Uncoated	NH_3	8.156	6.629	0.25	3.2	21418	2141	621	62.1	None to	1.00×10^5	115
R59	Uncoated	NH_3	8.146	6.604	0.23	3.3	21254	2125	621	62.1	2.51×10^3	3.14×10^3	15
R60	Uncoated	NH_3	8.171	6.642	0.23	3.3	21582	2158	621	62.1	1.35×10^3	2.00×10^3	15
R61	Diffusion	Air	8.181	6.665	0.23	3.2	21254	2125	621	62.1	3.11×10^3	4.05×10^3	110
R62-1	Diffusion	Air	8.230	6.685	0.23	3.2	21347	2135	621	62.1	3.68×10^3	5.42×10^3	120
R63	Diffusion	Air	8.209	6.680	0.25	3.1	21338	2134	621	62.1	2.92×10^3	5.55×10^3	110
R64	Diffusion	Air	8.204	6.680	0.25	3.1	21338	2134	621	62.1	2.94×10^3	4.35×10^3	95
R65	Gold Plate	NH_3	8.141	6.553	0.25	3.2	21338	2134	621	62.1	None to	1.00×10^5	110
R66	Gold Plate	NH_3	8.146	6.535	0.25	3.2	21338	2134	621	62.1	7.79×10^3	9.73×10^3	30
R67	Gold Plate	NH_3	8.174	6.528	0.28	3.1	21338	2134	621	62.1	None to	1.00×10^5	115
R68	Gold Plate	NH_3	8.169	6.528	0.25	3.2	21254	2125	621	62.1	3.08×10^4	3.20×10^4	50
R69	Uncoated	Air	8.166	6.604	0.25	3.1	18892	1889	621	62.1	2.57×10^4	2.61×10^4	120
R70	Uncoated	Air	8.169	6.617	0.31	3.0	21338	2134	621	62.1	1.00×10^4	1.06×10^4	150
R71	Uncoated	Air	8.161	6.629	0.23	3.2	21418	2142	621	62.1	4.47×10^3	5.57×10^3	90
R72	Uncoated	Air	8.138	6.609	0.25	3.1	21582	2158	621	62.1	6.73×10^3	7.57×10^3	90
R73	Diffusion	NH_3	8.288	6.657	0.20	3.4	21254	2125	621	62.1	1.69×10^3	1.97×10^3	20
R74	Diffusion	NH_3	8.214	6.675	0.23	3.3	21254	2125	621	62.1	1.25×10^3	1.59×10^3	10
R75	Diffusion	NH_3	8.209	6.652	0.25	3.2	21254	2125	621	62.1	1.28×10^3	1.52×10^3	15
R76	Diffusion	NH_3	8.214	6.675	0.28	3.0	21254	2125	621	62.1	1.32×10^3	2.01×10^3	10
R77	Gold Plate	Air	8.186	6.655	0.23	3.2	21418	2142	621	62.1	4.17×10^3	5.57×10^3	75
R78	Gold Plate	Air	8.189	6.629	0.20	3.4	21418	2142	621	62.1	5.83×10^3	7.52×10^3	
R79	Gold Plate	Air	8.161	6.604	0.20	3.4	21338	2134	621	62.1	5.27×10^3	6.31×10^3	95
R80	Gold Plate	Air	8.133	6.617	0.23	3.2	21254	2125	621	62.1	4.97×10^3	6.47×10^3	190

NOTES: 1 Air tests were at ambient pressure; ammonia tests at 3.4 MN/m^2. The ammonia contained 1.5 percent H_2O.

2 Stress Ratio = 0.1

TABLE XV

HIGH CYCLE FATIGUE TEST RESULTS FOR ASTROLOGY AT 1250°F
(U.S. CUSTOMARY UNITS)

Specimen Number	Coating	Atmosphere[1]	Notch Dimensions			Stress Concentration Factor	Load[2]		Nominal Stress at Notch		Cycles to Failure		Time at Temperature Under Load
			D1 in.	D2 in.	R in.		Maximum lb	Minimum lb	Maximum ksi	Minimum ksi	Crack Initiation	Rupture	
A81	Uncoated	NH_3	0.3207	0.2605	0.009	3.3	2532	253	47.5	4.7	None	5.00×10^6	21.5
A82	Uncoated	NH_3	0.3214	0.2615	0.009	3.3	2532	253	47.5	4.7	None	5.00×10^6	21.5
A83	Uncoated	NH_3	0.3220	0.2610	0.009	3.3	2510	251	47.5	4.7	None	5.00×10^6	21.5
A84	Uncoated	NH_3	0.3211	0.2605	0.008	3.4	2522	252	47.5	4.7	None	5.00×10^6	21.5
A85	Diffusion	NH_3	0.3231	0.2620	0.008	3.4	2522	252	47.5	4.7	6.52×10^3	8.65×10^3	.17
A86	Diffusion	NH_3	0.3231	0.2620	0.008	3.4	2522	252	47.5	4.7	6.96×10^3	1.10×10^3	.20
A87	Diffusion	NH_3	0.3232	0.2615	0.008	3.4	2522	252	47.5	4.7	4.03×10^3	7.34×10^3	.14
A88	Diffusion	NH_3	0.3231	0.2610	0.012	3.0	2501	250	47.5	4.7	4.30×10^3	8.09×10^6	.16
A89	Gold Plate	NH_3	0.3216	0.2610	0.008	3.4	2547	255	47.5	4.7	None	1.00×10^7	47.5
A90	Gold Plate	NH_3	0.3214	0.2610	0.009	3.3	2526	252	47.5	4.7	None	1.00×10^7	47.5
A91	Gold Plate	NH_3	0.3216	0.2610	0.009	3.3	2534	253	47.5	4.7	None	5.00×10^6	21.5
A92	Gold Plate	NH_3	0.3202	0.2605	0.008	3.4	2532	253	47.5	4.7	None	5.00×10^6	21.5
A93	Uncoated	Air	0.3212	0.2610	0.009	3.3	3745	375	70	7.0	2.38×10^4	3.82×10^4	2.58
A94[3]	Uncoated	Air	0.3215	0.2605	0.009	3.3	2665	267	50	5.0	3.20×10^5	4.23×10^5	3.88
A95	Uncoated	Air	0.3215	0.2590	0.009	3.3	2106	211	40	4.0	None to	1.00×10^7	93.73
A96[3]	Uncoated	Air	0.3211	0.2600	0.009	3.3	2665	266	50	5.0	---	1.60×10^6	20.00
A97	Diffusion	Air	0.3226	0.2615	0.011	3.0	2664	266	50	5.0	8.54×10^5	8.71×10^5	4.83
A98	Diffusion	Air	0.3226	0.2615	0.010	3.2	2521	252	47	4.7	1.08×10^6	1.11×10^6	13.88
A99	Diffusion	Air	0.3224	0.2610	0.011	3.1	2521	252	47	4.7	1.25×10^5	1.33×10^5	1.83
A100	Diffusion	Air	0.3227	0.2615	0.010	3.2	2521	252	47	4.7	1.02×10^5	1.16×10^5	2.67
A101	Gold Plate	Air	0.3227	0.2615	0.008	3.4	2522	252	47	4.7	5.71×10^6	5.73×10^6	29.33
A102	Gold Plate	Air	0.3230	0.2620	0.008	3.4	2665	266	50	5.0	1.57×10^5	1.64×10^5	3.00
A103	Gold Plate	Air	0.3228	0.2615	0.010	3.2	2405	214	45	4.5	1.03×10^6	1.10×10^6	12.00
A104	Gold Plate	Air	0.3224	0.2615	0.009	3.3	2397	240	45	4.5	None to	1.42×10^7	71.83

NOTES: 1 Air tests were at ambient pressure; ammonia tests at 500 psi. The ammonia contained 1.5 percent H_2O.

2 Stress Ratio = 0.1

3 Failed in threads

TABLE XVI

LOW CYCLE FATIGUE TEST RESULTS FOR ASTROLOGY AT 1250°F
(U.S. CUSTOMARY UNITS)

Specimen Number	Coating	Atmosphere[1]	Notch Dimensions D1 in.	Notch Dimensions D2 in.	Notch Dimensions R in.	Stress Concentration Factor	Load[2] Maximum lb	Load[2] Minimum lb	Nominal Stress at Notch Maximum ksi	Nominal Stress at Notch Minimum ksi	Cycles at Failure Crack Initiation	Cycles at Failure Rupture	Time at Temperature Under Load Minutes
A33	Uncoated	NH_3	0.3214	0.2605	0.010	3.2	5063	506	95	9.5	8.70×10^2	1.23×10^3	10
A34	Uncoated	NH_3	0.3220	0.2613	0.009	3.3	5087	509	95	9.5	5.90×10^2	8.00×10^2	5
A35	Uncoated	NH_3	0.3208	0.2603	0.009	3.3	5079	508	95	9.5	4.50×10^2	5.40×10^2	5
A36	Uncoated	NH_3	0.3207	0.2610	0.009	3.3	5079	508	95	9.5	6.40×10^2	7.90×10^2	5
A37	Diffusion	NH_3	0.3232	0.2620	0.008	3.4	5044	504	95	9.5	5.80×10^2	8.20×10^2	5
A38	Diffusion	NH_3	0.3228	0.2615	0.009	3.3	5044	504	95	9.5	6.00×10^2	7.40×10^2	10
A39	Diffusion	NH_3	0.3232	0.2615	0.008	3.4	5024	502	95	9.5	4.60×10^2	8.00×10^2	55
A40	Diffusion	NH_3	0.3236	0.2620	0.008	3.4	5055	506	95	9.5	6.60×10^2	1.06×10^3	10
A41	Gold Plate	NH_3	0.3205	0.2615	0.008	3.4	5090	509	95	9.5	2.28×10^3	2.61×10^3	20
A42	Gold Plate	NH_3	0.3212	0.2610	0.009	3.3	5067	507	95	9.5	7.10×10^2	9.70×10^2	10
A43	Gold Plate	NH	0.3211	0.2610	0.009	3.3	5075	507	95	9.5	1.03×10^3	1.38×10^3	10
A44	Gold Plate	NH	0.3214	0.2610	0.009	3.3	5083	508	95	9.5	8.90×10^2	1.23×10^3	15
A45	Uncoated	Air	0.3215	0.2605	0.009	3.3			85	8.5	1.88×10^3	3.63×10^3	92
A46	Uncoated	Air	0.3211	0.2605	0.009	3.3	4540	454	85	8.5	5.25×10^3	1.44×10^4	70
A47	Uncoated	Air	0.3219	0.2605	0.010	3.2	5064	506	95	9.5	2.02×10^3	4.71×10^3	105
A48	Uncoated	Air	0.3217	0.2605	0.010	3.2	5064	506	95	9.5	5.29×10^3	8.39×10^3	90
A49	Diffusion	Air	0.3223	0.2600	0.010	3.2	5024	502	95	9.5	2.06×10^3	3.64×10^3	150
A50	Diffusion	Air	0.3221	0.2600	0.010	3.2	5044	504	95	9.5	2.54×10^3	3.69×10^3	150
A51	Diffusion	Air	0.3236	0.2610	0.010	3.2	5044	504	95	9.5	2.18×10^3	3.49×10^3	50
A52	Diffusion	Air	NOT TESTED										
A53	Gold Plate	Air	0.3216	0.2610	0.009	3.3	5060	506	95	9.5	2.02×10^3	3.89×10^3	40
A54	Gold Plate	Air	0.3223	0.2604	0.009	3.3	4780	465	90	9.0	9.50×10^3	1.24×10^4	150
A55	Gold Plate	Air	0.3230	0.2610	0.009	3.3	5064	506	95	9.5	1.90×10^3	3.82×10^3	90
A56	Gold Plate	Air	0.3216	0.2615	0.010	3.2	5064	506	95	9.5	1.97×10^3	4.37×10^3	90

NOTES: 1 Air tests were at ambient temperature; ammonia tests at 500 psi. The ammonia contained 1.5 percent H_2O.

2 Stress Ratio = 0.1

TABLE XVII

HIGH CYCLE FATIGUE TEST RESULTS FOR RENE' 41 at 1250°F
(U.S. CUSTOMARY UNITS)

Specimen Number	Coating	Atmosphere[1]	Notch Dimensions D1 in.	Notch Dimensions D2 in.	Notch Dimensions R in.	Stress Concentration Factor	Load[2] Maximum lb	Load[2] Minimum lb	Nominal Stress at Notch Maximum ksi	Nominal Stress at Notch Minimum ksi	Cycles to Failure Crack Initiation	Cycles to Failure Rupture	Time at Temperature Under Load Hours
R105	Uncoated	NH_3	0.3208	0.2600	0.0095	3.2	3186	319	60	6.0	None	5.00×10^6	21.5
R106	Uncoated	NH_3	0.3215	0.2615	0.009	3.3	3198	320	60	6.0	None	5.00×10^6	21.5
R107	Uncoated	NH_3	0.3219	0.2620	0.009	3.3	3186	319	60	6.0	None	5.00×10^6	21.5
R108	Uncoated	NH_3	0.3215	0.2620	0.0105	3.0	3198	320	60	6.0	None	5.00×10^6	21.5
R109	Diffusion	Air	0.3240	0.2636	0.009	3.2	3260	326	60	6.0	6.80×10^3	1.11×10^4	1.83
R110	Diffusion	Air	0.3227	0.2627	0.009	3.2	3186	319	60	6.0	9.34×10^3	1.52×10^4	2.25
R111	Diffusion	Air	0.3227	0.2627	0.009	3.2	3198	320	60	6.0	8.56×10^3	1.34×10^4	1.75
R112	Diffusion	Air	0.3216	0.2625	0.009	3.2	3186	319	60	6.0	9.00×10^3	1.55×10^4	1.92
R113	Gold Plate	NH_3	0.3207	0.2567	0.011	3.0	3186	319	60	6.0	None	5.00×10^6	21.5
R114	Gold Plate	NH_3	0.3214	0.2610	0.012	3.0	3186	319	60	6.0	None	5.00×10^6	21.5
R115	Gold Plate	NH_3	0.3199	0.2570	0.010	3.2	3210	321	60	6.0	None	5.00×10^6	21.5
R116	Gold Plate	NH_3	0.3219	0.2570	0.010	3.2	3210	321	60	6.0	None	5.00×10^6	21.5
R117	Uncoated	Air	0.3205	0.2600	0.010	3.2	2920	292	55	5.5	1.00×10^6	5.09×10^6	43.75
R118	Uncoated	Air	0.3215	0.2620	0.009	3.2	2696	270	50	5.5	None to	1.00×10^7	40.00
R119	Uncoated	Air	0.3215	0.2605	0.009	3.2	3198	320	60	6.0	3.27×10^5	3.28×10^5	2.17
R120	Uncoated	Air	0.3210	0.2600	0.012	3.0	3186	319	60	6.0	1.55×10^5	1.62×10^5	2.67
R121	Diffusion	NH_3	0.3237	0.2626	0.008	3.4	3186	319	60	6.0	6.57×10^6	6.85×10^6	32.0
R122	Diffusion	NH_3	0.3234	0.2630	0.0095	3.2	3198	320	60	6.0	None	1.00×10^7	47.5
R123	Diffusion	NH_3	0.3227	0.2621	0.009	3.3	3186	319	60	6.0	None	5.00×10^6	21.5
R124	Diffusion	NH_3	0.3235	0.2620	0.011	3.1	3186	319	60	6.0	None	5.00×10^6	21.5
R125	Gold Plate	Air	0.3216	0.2615	0.008	3.4	3198	320	60	6.0	3.21×10^6	3.22×10^6	20.83
R126	Gold Plate	Air	0.3212	0.2610	0.011	3.0	3318	322	62	6.2	No Data	4.23×10^5	6.67
R127	Gold Plate	Air	0.3218	0.2615	0.008	3.4	3198	320	60	6.0	No Data	6.24×10^4	0.83
R128	Gold Plate	Air	0.3219	0.2610	0.010	3.2	3186	319	60	6.0	5.88×10^6	5.93×10^6	66.50

NOTES: 1 Air tests were at ambient pressure; ammonia tests at 500 psi. The ammonia contained 1.5 percent H_2O.

2 Stress Ratio = 0.1

TABLE XVIII

LOW CYCLE FATIGUE TEST RESULTS FOR RENE' 41 AT 1250°F
(U.S. CUSTOMARY UNITS

Specimen Number	Coating	Atmos-phere[1]	Notch Dimensions D1 in.	Notch Dimensions D2 in.	Notch Dimensions R in.	Stress Concentration Factor	Load[2] Maximum lb	Load[2] Minimum lb	Nominal Stress at Notch Maximum ksi	Nominal Stress at Notch Minimum ksi	Cycles to Failure Crack Initiation	Cycles to Failure Rupture	Time Temperature Under Load Minute
R57	Uncoated	NH_3	0.3217	0.2610	0.011	3.0	4797	480	90	9.0	None to	2.53×10^4	50
R58	Uncoated	NH_3	0.3211	0.2610	0.010	3.2	4815	482	90	9.0	None to	1.00×10^5	115
R59	Uncoated	NH_3	0.3207	0.2600	0.009	3.3	4778	478	90	9.0	2.51×10^3	3.14×10^3	15
R60	Uncoated	NH_3	0.3217	0.2615	0.009	3.3	4852	485	90	9.0	1.35×10^3	2.00×10^3	15
R61	Diffusion	Air	0.3221	0.2624	0.009	3.2	4778	478	90	9.0	3.11×10^3	4.05×10^3	110
R62-1	Diffusion	Air	0.3240	0.2632	0.009	3.2	4799	480	90	9.0	3.68×10^3	5.42×10^3	120
R63	Diffusion	Air	0.3232	0.2630	0.010	3.1	4797	480	90	9.0	2.92×10^3	5.55×10^3	110
R64	Diffusion	Air	0.3230	0.2630	0.010	3.1	4797	480	90	9.0	2.94×10^3	4.35×10^3	95
R65	Gold Plate	NH_3	0.3205	0.2580	0.010	3.2	4797	480	90	9.0	None to	1.00×10^5	110
R66	Gold Plate	NH_3	0.3207	0.2573	0.010	3.2	4797	480	90	9.0	7.79×10^3	9.73×10^3	30
R67	Gold Plate	NH_3	0.3218	0.2570	0.011	3.1	4797	480	90	9.0	None to	1.00×10^5	115
R68	Gold Plate	NH_3	0.3216	0.2570	0.010	3.2	4778	478	90	9.0	3.08×10^4	3.20×10^4	50
R69	Uncoated	Air	0.3215	0.2600	0.010	3.1	4247	425	80	8.0	2.57×10^4	2.61×10^4	120
R70	Uncoated	Air	0.3216	0.2605	0.012	3.0	4797	480	90	9.0	1.00×10^4	1.06×10^4	150
R71	Uncoated	Air	0.3213	0.2610	0.009	3.2	4815	481	90	9.0	4.47×10^3	5.57×10^3	90
R72	Uncoated	Air	0.3204	0.2602	0.010	3.1	4852	485	90	9.0	6.73×10^3	7.57×10^3	90
R73	Diffusion	NH_3	0.3263	0.2621	0.008	3.4	4778	478	90	9.0	1.69×10^3	1.97×10^3	20
R74	Diffusion	NH_3	0.3234	0.2628	0.009	3.3	4778	478	90	9.0	1.25×10^3	1.59×10^3	10
R75	Diffusion	NH_3	0.3232	0.2619	0.010	3.2	4778	478	90	9.0	1.28×10^3	1.52×10^3	15
R76	Diffusion	NH_3	0.3234	0.2628	0.011	3.0	4778	478	90	9.0	1.32×10^3	2.01×10^3	10
R77	Gold Plate	Air	0.3223	0.2620	0.009	3.2	4815	481	90	9.0	4.17×10^3	5.57×10^3	75
R78	Gold Plate	Air	0.3224	0.2610	0.008	3.4	4815	481	90	9.0	5.83×10^3	7.52×10^3	
R79	Gold Plate	Air	0.3213	0.2600	0.008	3.4	4797	480	90	9.0	5.27×10^3	6.31×10^3	95
R80	Gold Plate	Air	0.3202	0.2605	0.009	3.2	4778	478	90	9.0	4.97×10^3	6.47×10^3	190

NOTES: 1 Air tests were at ambient pressure ammonia tests at 500 psi. The ammonia contained 1.5 percent H_2O

2 Stress Ratio = 0.1

TABLE XIX. - SUMMARY OF FRACTURE MECHANICS TESTING

Alloy	Coating	Atmos	K_{IE} (min) $MN/M^{2/3}$ ($Ksi\sqrt{in.}$)	K_i/K_{IE} (1)
Rene' 41	Uncoated	Air	67.5 (61.4)	
		NH_3	-	43%
	18 μm (0.7 mil) Gold Plate	NH_3	-	65%
Astroloy	Uncoated	Air	79.1 (71.9)	
		NH_3	-	50%
	18 μm (0.7 mil) Gold Plate	NH_3	-	50%

(1) Highest value observed for zero crack growth

APPENDIX

EVALUATION OF COATINGS ON WASPALOY FOR USE IN HIGH TEMPERATURE AMMONIA

PRECEDING PAGE BLANK NOT FILMED

INTRODUCTION

This report presents results of an AiResearch IR and D program to select two coatings best suited to protect Astroloy and Rene' 41 in a high temperature, high-pressure ammonia environment. With NASA concurrence, the selected coatings will be used to coat these alloys for further evaluation in the program funded under NASA-Houston Contract (NAS9-13484) for the development of fracture mechanics data on two candidate hydrazine APU turbine wheel materials. Because Astroloy and Rene' 41 were not immediately available, the IR and D tests were conducted on sheets of Waspaloy which reacts similarly to coating application techniques and to the ammonia environments. In the test specimens with various diffusion, plated, and other coatings were weighed and then subjected to 1250°F ammonia for 150-hr. Following the exposure, the specimens were reweighed and subjected to bend tests, tensile tests, and metallurgical evaluation.

SUMMARY AND CONCLUSION

Two coatings were selected subject to NASA approval for the fracture mechanics development program: gold plating, 0.5 to 1.0 mil thick, and Chromizing Company's diffusion barrier coating 870. The gold plating afforded the best protection, but its resistance to erosion and adhesion at high rotating speeds require careful evaluation. The results of the bend tests are summarized in Table 1.

In the table, each of the coatings tested are rated on the angle the sample could be bent around the 0.125-dia. mandrel before cracking; the higher the angle, the greater the ductility and therefore the greater the protection. Values obtained ranged from 115 deg for gold plating to 52 deg for nickel plating with 83 deg recorded for an uncoated specimen. Uncoated and unexposed specimens could be bent 180-deg.

An additional criteria for evaluating the coating is the change in weight which is indicative of the degree of reaction with the ammonia and generally increases with the decrease in ductility. These weight changes are also tabulated in Table 1. Electroplated gold and copper, chemical vapor deposited molybdenum and Chromizing Company diffusion coating LCF, 870 and BB all improved the bend ductility of Waspaloy sheet.

Alloy Surfaces coating HI-15, Chromizing Company coating UC, Walbar coating RE-101 and chemical vapor deposited tungsten were marginal in their degree of protection.

AiResearch applied CoCrAl, Metco 450 (nickel aluminide) and nickel coatings decreased the bend ductility of the Waspaloy sheet specimens.

PROCEDURE

Strips of 0.040-in.-thick Waspaloy sheet in the solution annealed condition (AMS 5544) were cut into 2 in. by 1 in. pieces and individually identified. The specimens were then shipped to various coatings vendors where they were cleaned and coated according to each vendor's propriety process. A list of coatings selected for evaluation is tabulated in Table 2.

TABLE 1

RESULTS OF BEND TEST AND INCREASE IN WEIGHT OF SPECIMENS

Coating	Bend Angle (0^{o})	$\Delta\omega$ (mgm)
Uncoated - unexposed	180	-
Gold plating, 0.5 mil	115	3.3
CVD molybdenum	113	10.5
Diffusion coating LCF	112	8.0
Gold plating, 1.0 mil	106	1.9
Diffusion coating 870	102	1.4
Copper plating, 1.0 mil	100	0.7
Copper plating, 2.0 mil	98	1.9
Diffusion coating BB	93	1.7
Uncoated - exposed	83	27.0
Diffusion coating HI-15	82	2.6
Diffusion coating RE-101	79	1.1
Diffusion coating UC	78	1.6
Electroless nickel, 1.0 mil	75	14.3
AiResearch CoCrAl	71	45.5
CVD tungsten	70	2.0
Electroless nickel, 2.0 mil	69	14.5
Uncoated - exposed	67	36.1
Nickel aluminide (Metco 450)	64	33.4
Nickel plate, 1.0 mil	64	36.9
Nickel plate, 2.0 mil	52	23.0

NOTES:

1. The higher the bend angle, the better the degree of protection.
2. The lower the $\Delta\omega$ figure, the less the reaction with ammonia.

TABLE 2

COATINGS EVALUATED

Coating	Vendor	Appearance of Coating as Received from Vendor
Diffusion		
HI-15	Alloy surfaces	Good satin finish, slight marbling
UC	Chromizing Co.	Smooth satin finish
780	Chromizing Co.	Pitted, satin finish
LCF	Chromizing Co.	Pitted, Satin finish
BB	Chromizing Co.	Smooth, satin finish
MDC-1	Howmet	No response from Howmet
MDC-1A	Howmet	No response from Howmet
RE-101	Walbar	Dark, satin finish
Plated		
.5 mil Au	AiResearch	Smooth, even deposit
1 mil Au	AiResearch	Smooth, even deposit
1 mil Cu	AiResearch	Frosty, corners burned
2 mil Cu	AiResearch	Badly stained, oxide blotches
1 mil Ni(E)	(E = electroless)	Very shiny finish
2 mil Ni(E)	(E - electroless)	Very shiny finish
1 mil Ni(P)	(P = plated sulphamate bath)	Satin finish, slightly burned
2 mil Ni(P)	(P = plated sulphamate bath)	Satin finish
Miscellaneous		
CoCrAl	AiResearch	Poor adhesion of coating
Metco 450	Bender Machine	Rough, but uniform surfaces
CVD Mo	Ultramet	Smooth, frosty, burned on one corner
CVD W	Ultramet	Smooth, frosty surfaces

After the specimens had been returned from the vendors, they were ultrasonically cleaned in acetone, dried and the weight and dimensions of each specimen recorded. All of the specimens, along with three uncoated pieces of Waspaloy sheet were threaded onto a wire holder inside a Hastelloy-X retort which was then sealed and purged with argon and heated to 1250°F. Temperature measurement and control were effected by two sheathed thermocouples attached to the specimens. A third thermocouple was attached to the ourside of the retort and set to shut off the power supply to the furnace in case of a serious overtemperature condition. Ammonia was admitted into the retort at a flow rate of 16 - 20 ft^3/hr.

At the beginning of the experiment the ammonia dissociation was measured to be 76 - 77 percent, but after 18 hr it fell to 65 - 66 percent, and remained constant there for the balance of the 150-hr exposure. The retort was then removed from the furnace, ammonia flow discontinued and the specimens cooled to room temperature under a flow of argon. A schematic diagram of the test setup is shown in Figure 1.

Immediately following removal from the retort, the specimens were again cleaned ultrasonically in acetone, dried and reweighed. The increase in weight of the specimens was recorded.

The specimens were sectioned as shown in Figure 2, and the various pieces were subjected for tensile tests, bend tests and metallographic examination.

RESULTS

Increase in Weight

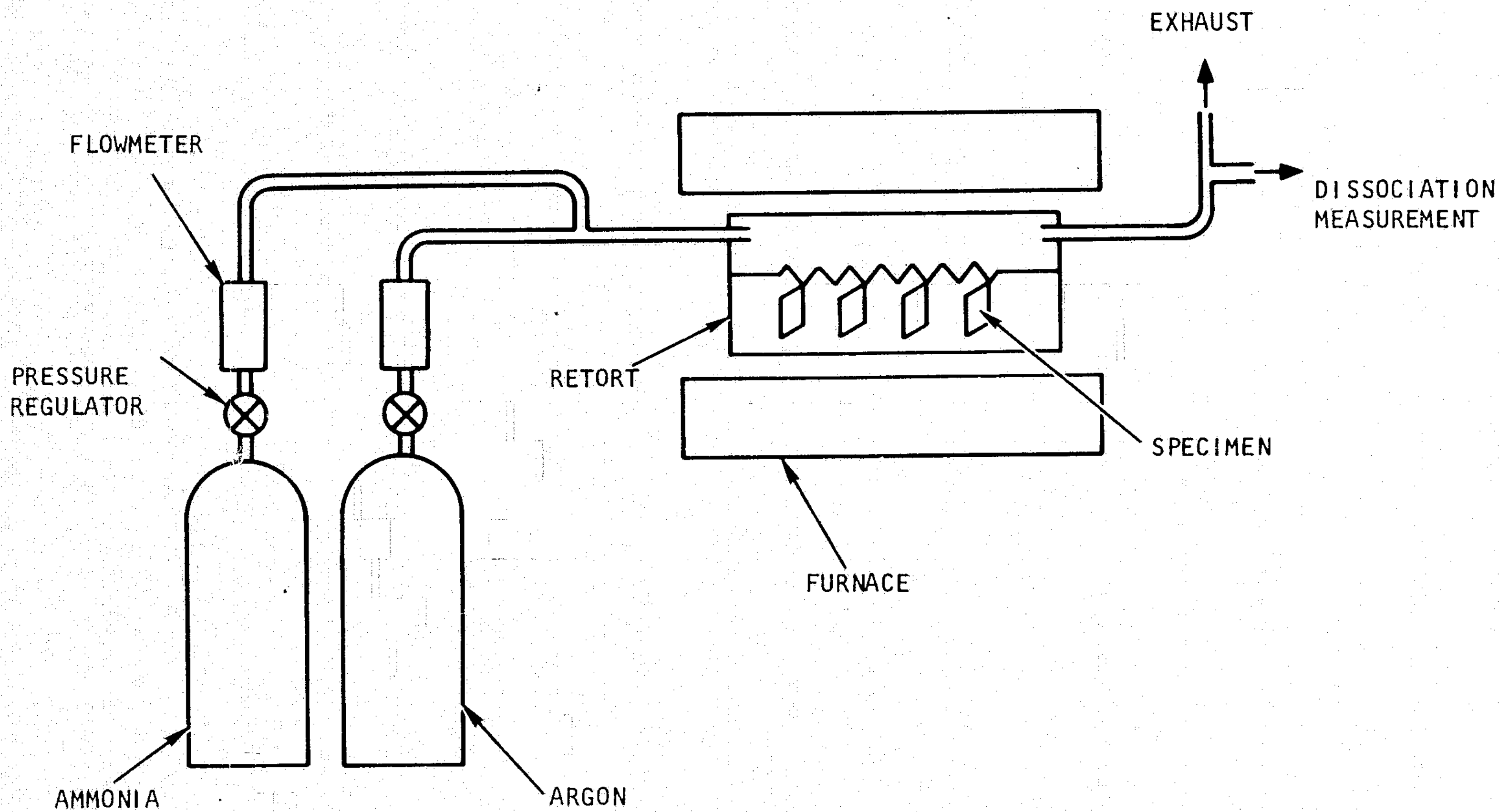
All the specimens were ultrasonically cleaned in acetone immediately before weighing on a analytical beam balance. The change in weight is recorded in Table 1. This increase is attributed to the amount of H_2 and N_2 absorbed by the specimens.

Bend Test

A 0.125-in.-dia mandrel was used to form the bend radius in a bend test fixture shown in Figure 3. The specimens were placed in the fixture and bent by a Baldwin tensile machine. The end point of the test was determined to be at the point where the bending load dropped off abruptly, indicating that the specimen had cracked. The angle through which the specimen could be bent before cracking was taken as a measure of the degree of protection conferred by each coating. (The higher the angle the better).

Tensile Tests

After exposure, small specimens were subjected to tensile testing at room temperature. The results of these tests are shown in Table 3.



S-82057

Figure 1. Schematic Diagram of Test Setup

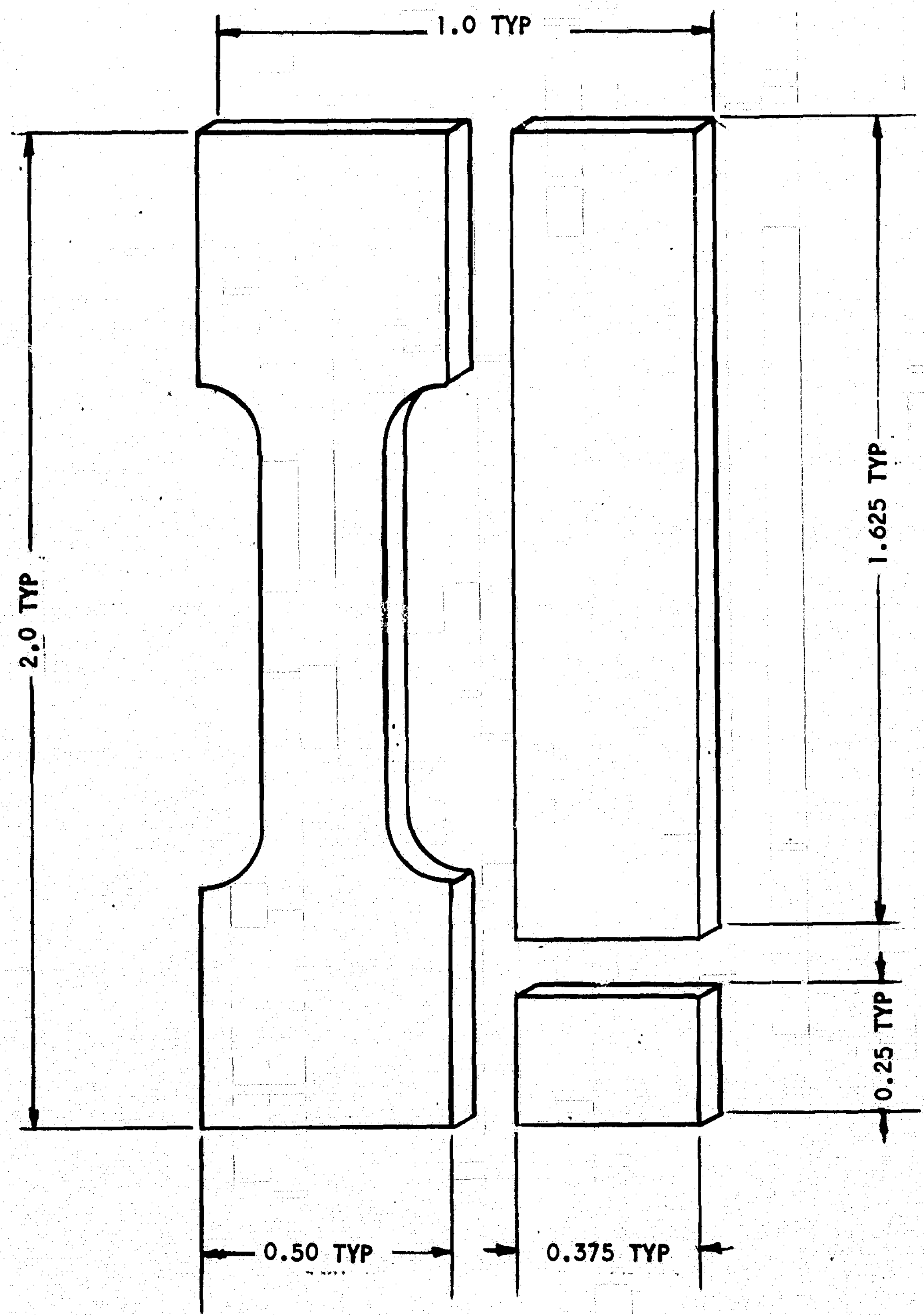


Figure 2. Specimen Sectioned After Exposure Showing Relative Locations of Test Pieces.

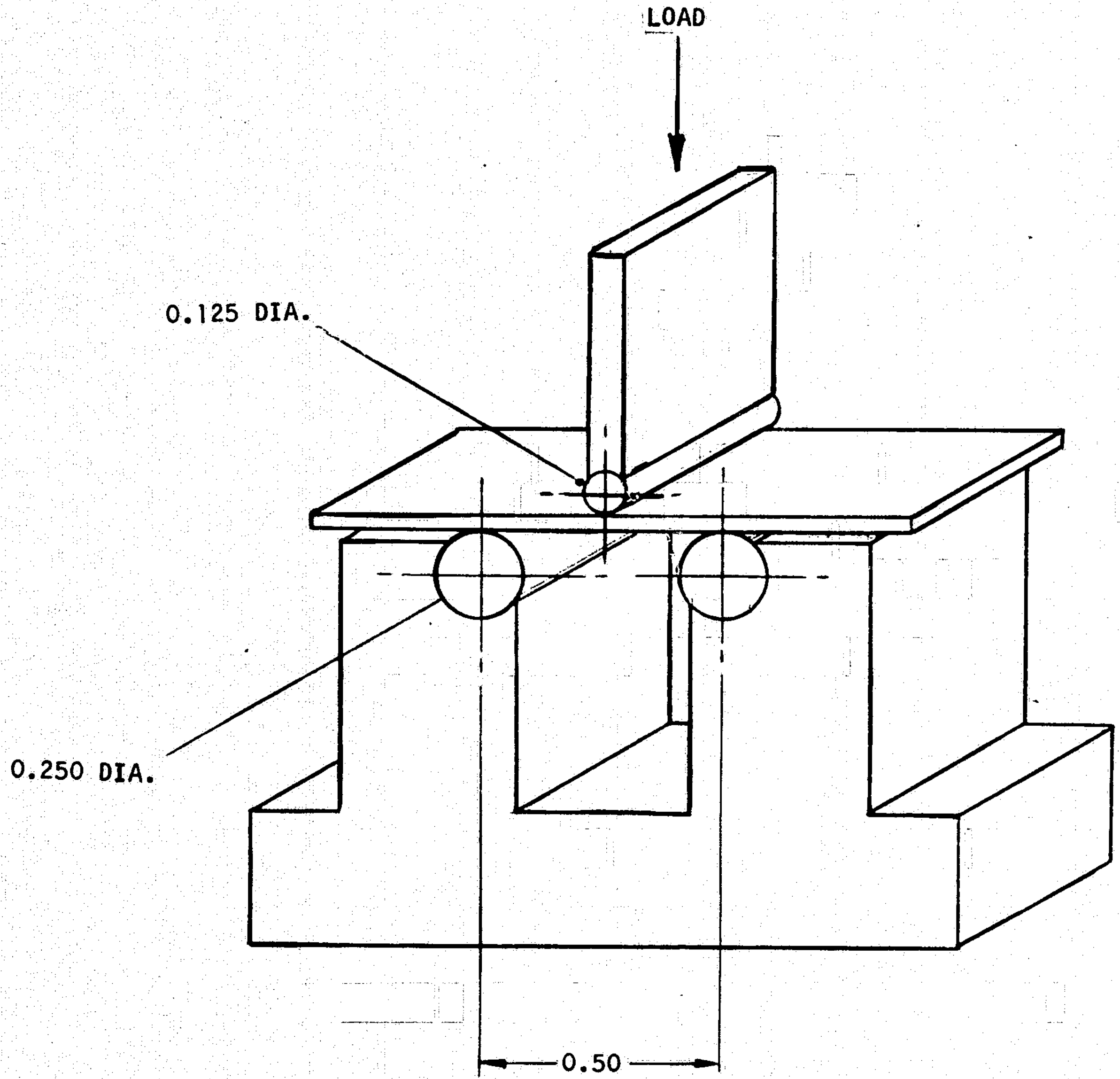


Figure 3. Schematic Diagram of Bend Test Fixture.

TABLE 3

RESULTS OF ROOM TEMPERATURE TENSILE TESTS

Coating	UTS (ksi)	0.2% YS (ksi)	Elongated (percent)	RA (percent)
Uncoated				
Specimen G34	177.9	138.5	28.0	22.3
Specimen G35	174.5	135.8	24.0	21.9
Specimen G36	180.8	144.2	23.0	18.7
Diffusion				
HI-15	155.2	75.2	16.0	21.0
UC	162.9	86.2	17.0	15.8
780	154.7	101.9	27.0	18.7
LCF	166.3	87.5	23.0	17.8
BB	154.6	108.3	-	17.4
RE-101	157.0	86.9	19.0	15.3
Plated				
0.5 mil Au	177.5	-	32.0	34.4
1 mil Au	176.4	-	26.0	21.4
1 MIL-Cu	183.3	136.7	30.0	28.7
2 mil Cu	173.0	126.5	30.0	33.9
1 mil Ni(E)	169.8	130.2	26.0	21.0
2 mil Ni (E)	162.1	123.7	25.0	21.5
1 mil Ni (P)	177.4	135.8	19.0	17.3
2 mil Ni (P)	166.7	132.5	23.0	20.3
Miscellaneous				
CoCrAl	172.5	136.2	16.0	20.6
Metco 450	180.8	150.0	22.0	8.5
CVD Mo	154.7	91.0	27.0	22.2
CVD W	161.1	119.9	23.0	19.2
Uncoated, unexposed, aged typical properties	185.0	115.0	25.0	---

Metallographical Examination

A cross section of each specimen was prepared for metallographical examination of the surface layers. Uncoated Waspaloy has a nitride layer 0.8 - 1.0 mil deep. (See Figure 4.) The diffusion coatings were all approximately 2.0 mil deep, and no nitrides were detected on the Waspaloy substrate in the case of the Chromizing Company's coating LCF. This was only 0.1 to 0.25 mil deep and there appeared to be intergranular nitrides to a depth of about 1 mil below the surface coating (see Figure 5). Figure 6 shows the Chromizing Company's 870 coating as being typical of the diffusion coatings.

Of the electroplated specimens, both gold and copper were effective in preventing nitriding of the Waspaloy. The 0.5 mil gold specimen is shown in Figure 7. However, neither type of nickel coating prevented the formation of nitride layers on the Waspaloy substrate. Figure 8 illustrates the porosity of the nickel coatings which developed during the ammonia exposure.

DISCUSSION

Table 1 shows the order of nitriding prevention of the coatings tested, based on the results of the bend tests. It shows that uncoated, unexposed Waspaloy is capable of being bent 180 deg around a zero diameter bend radius. After nitriding, uncoated Waspaloy specimen could only be bent 67 deg around a .125 in.-dia bend diameter. A second specimen was bent through an 83 deg angle. Therefore, any specimen that could be bent more than 83 deg received some degree of protection from the coating, while those that cracked at less that 67 deg were actually worsened by the presence of the coating.

Gold plating afforded the best protection, although copper plating was also satisfactory. However, copper coatings are not expected to perform as well in wet ammonia (the atmosphere to be used for the NASA program).

Chemical vapor deposited molybdenum also performed exceptionally well, except that the considerable increase in weight indicates that the molybdenum was absorbing nitrogen to form Mo_2N or MoN. However, molybdenum coatings would be removed by high temperature exposure to air (as would happen in a ground tested SSAPU) and consequently these coatings cannot be recommended.

With the exception of diffusion coating UC, the other three Chromizing Company coatings (LCF, 870 and BB) were better than diffusion coatings received from other vendors. Coating LCF did show a moderate increase in weight, indicating nitrogen absorption. Evidence of nitrided grain boundaries also suggest that this coating may not protect the substrate over prolonged periods of time. Alloy Surfaces coating HI-15, Chromizing Co. UC and Walbar RE-101 provided only marginal protection and likewise, may not be suitable for extended periods of time. The remaining specimens generally offering the least degree of protection.

The tensile tests were not very discriminatory in that the processing temperature has a more pronounced effect on the mechanical properties than the influence of nitrides. Thus the plated and uncoated specimens, which were not heated, had higher strengths than the diffusion coated specimens which has been heated to high temperatures during aluminiding. Consequently, no meaningful conclusions could be drawn from these tests.

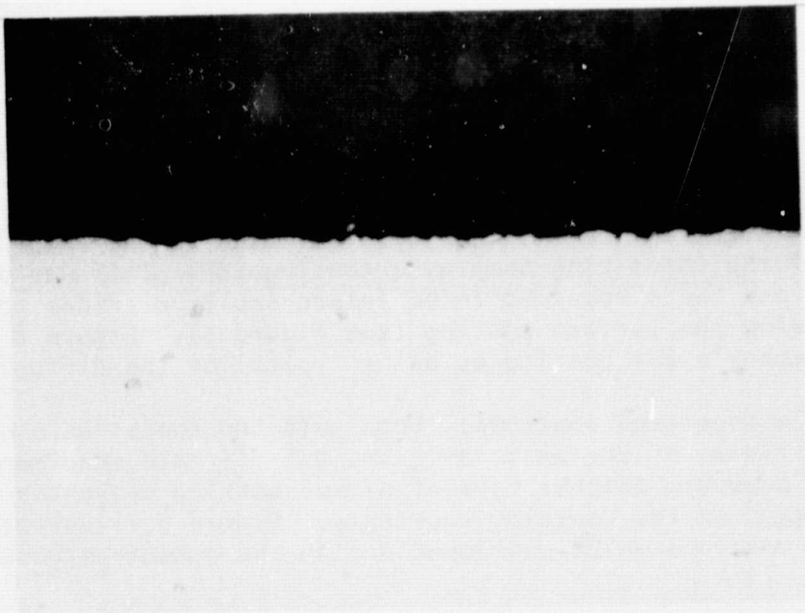


Fig. 4. X250

Uncoated Waspaloy, showing depth of nitride layer to be about 1.0 mil deep.

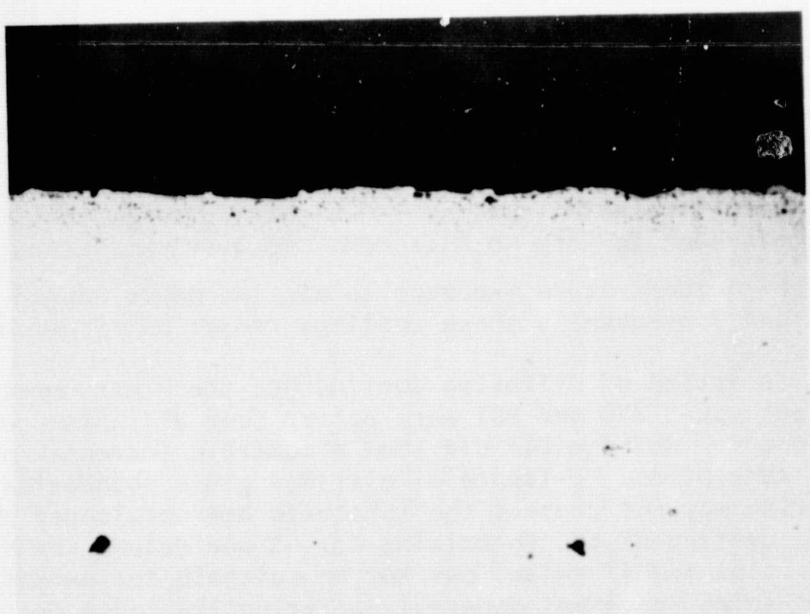


Fig. 5. X250 F-18473

Chromizing Co. LCF coating showing presence of what appear to be intergranular nitrides in the Waspaloy substrate.

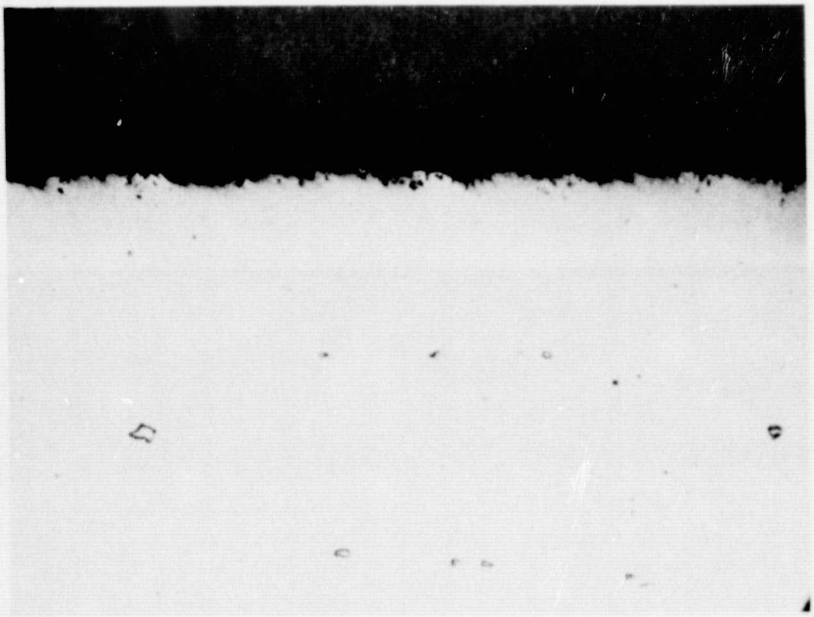
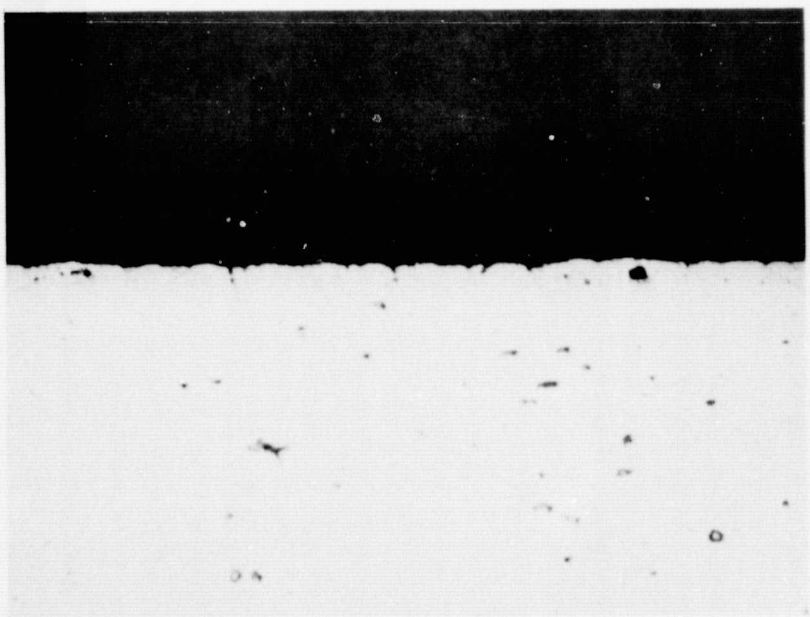


Fig. 6. X250

Chromizing Co. coating 870; typical of the diffusion coatings. Note absence of nitrides in Waspaloy.



F-18472

Fig. 7. X250

Gold plating, .5 mil nominal thickness. Note absence of nitrides; typical of gold and copper coatings.

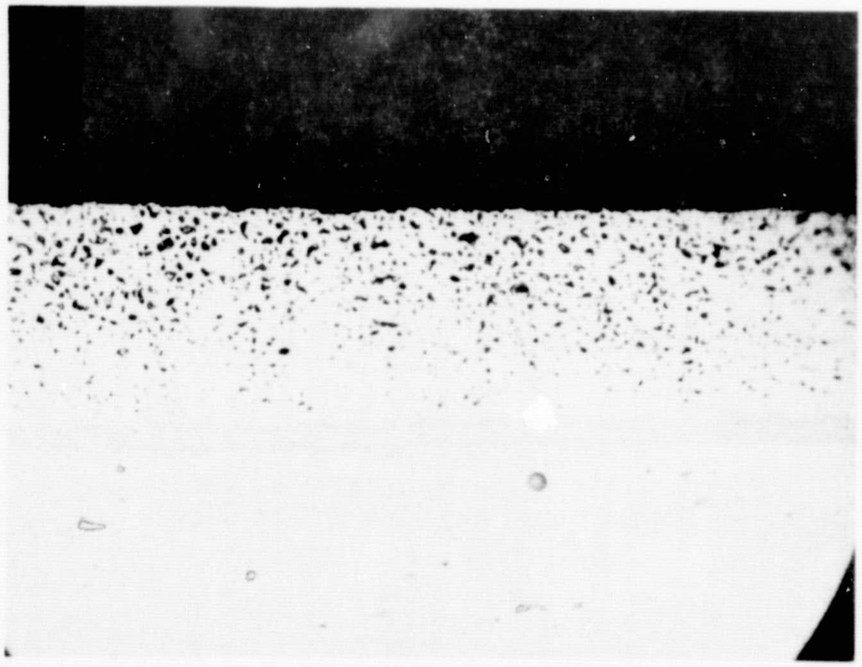


Fig. 8. X250 F-18471

Nickel plating (sulfamate), nominally 2 mil thick (actually 6.5 mil thick!); typical of all nickel plated specimens. Note porosity in coating (developed during exposure to ammonia) and nitride layer on Waspaloy substrate.