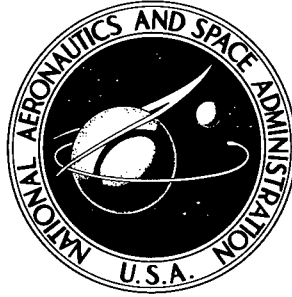


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## THE EFFECT OF VACUUM ON THE FATIGUE AND STRESS-RUPTURE PROPERTIES OF S-816 AND INCONEL 550 AT 1500° F

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# THE EFFECT OF VACUUM ON THE FATIGUE AND STRESS-RUPTURE PROPERTIES OF S-816 AND INCONEL 550 AT 1500° F

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## SUMMARY

The relative effects of vacuum ( $10^{-4}$  to  $10^{-5}$  mm Hg) and air environments on the axial tensile fatigue (cyclic- to mean-stress ratios of 0.667 and 0.125) and stress-rupture properties of a cobalt-base alloy (S-816) and a nickel-base alloy (Inconel 550) were investigated at 1500° F. The range of mean stress was from 15 000 to 40 000 psi.

Life in vacuum was generally greater than life in air for both alloys in both fatigue and stress rupture. This improvement was fairly constant over the entire stress range for S-816, but a convergence was observed at the lower stresses for Inconel 550. The convergence indicates that long time elevated temperature tests in air may not always adequately describe superalloy performance in fatigue or stress rupture under vacuum. Thus, to avoid miscalculations in space vehicle component designs, which could result in premature failures, superalloys should be tested for long time periods in vacuum to more nearly simulate a space environment.

Failures at the highest cyclic- to mean-stress ratio were generally transgranular. As the ratio was decreased and a stress-rupture-type test was more nearly approached, a combination of transgranular and intergranular failure modes was observed. Stress-rupture failures were generally intergranular.

Oxide penetration varied with test life for both materials but did not appear to be affected by either mean or alternating stress. Electron beam microprobe analyses indicated that chromium, of all the elements checked, showed by far the greatest degree of depletion after long test times for both alloys. Chromium depletion was observed in S-816 in both vacuum and air and in Inconel 550 in air.

## INTRODUCTION

There is an increasing need for additional knowledge regarding the behavior of

materials in vacuum since the advent of the space age. Mission requirements for contemplated space vehicles call for long time (10 000 hr and more) exposure of various structural and powerplant components to the vacuum of outer space. Many of these components are also subjected to elevated temperatures as a result of solar heating and heat generated by the propulsion systems. As a consequence, the combined effects of vacuum and elevated temperatures upon the fatigue and stress-rupture properties of metals that may be used in space vehicle applications becomes of major importance to the designer.

*Start* Nickel- and cobalt-base superalloys will be used for intermediate temperature (1300° to 2000° F) applications in some advanced space power systems. Investigations have been made to study the effect of a vacuum environment upon high-temperature mechanical properties of some superalloys. Much of this work is reviewed in reference 1. Most of these investigations were made to determine stress-rupture properties, but only limited fatigue data have been obtained. Also, these studies were made for widely disparate test conditions and specimen configurations. Under certain conditions, stress-rupture and fatigue lives were higher in air than in vacuum. Under other conditions, the reverse was true. Some of these results are reviewed in the following paragraphs.

*A1* In stress rupture, three nickel-base alloys (Hastelloy C, Inconel X, and N-155) and a cobalt-base alloy (S-816) all had longer lives in air than in vacuum at temperatures ranging from 1500° to 1600° F within relatively narrow stress ranges (refs. 2 and 3). *N.B. N.B.* Other investigations (refs. 4 to 7) showed that nickel and some nickel-base alloys had either longer or shorter stress-rupture lives in air than in vacuum depending upon the test temperature and stress. In general, the effect of increasing the temperature of stress-rupture tests was to decrease life in vacuum relative to that in air. The effect of increasing the stress at constant temperature was to increase life in vacuum relative to that in air. Crossover effects were observed in some constant temperature tests where at high stresses the life in vacuum was longer than in air, and the reverse was true at lower stresses. *→ pg 25*

In fatigue, bending tests (constant strain range) have been run with pure nickel at 1500° F (ref. 8). A crossover of air and vacuum data was observed. This investigation also showed that the 1500° F bending fatigue lives of Inconel X and 316 stainless steel in vacuum were approximately 25 times as great as in air at high cyclic strains and only about four times as great as in air at low cyclic strains. Thus, a convergence was indicated, but an actual crossover did not occur within the range of strains considered.

Theories have been advanced to explain the physical phenomena involved. The theory that adsorption of gases to fatigue fracture surfaces facilitates crack propagation was investigated (ref. 9). The results of this study with stainless steel and nickel generally indicated that fatigue life increased with decreasing absolute pressure (less than 1 atm) of the environmental gas. It has also been postulated (refs. 4, 6, and 8) that two major

competing mechanisms are operative in environmental testing which determine material life. By one mechanism, the surface energy of the metal is presumed to be lowered in the presence of air or other gas, as compared to its value in a vacuum, thus facilitating crack propagation or extension of surface and lowering metal strength. If this were the only mechanism operative, metals could always be expected to be stronger in a vacuum environment than in air; however, a competing mechanism, that of metal oxidation, is presumed to act as a strengthener. Its effectiveness apparently depends upon the proper combination of stress and temperature. In general, the higher the test temperature, the greater the amount of oxidation that occurs in air during the course of a test. Also, the lower the stress at a given temperature in air, the longer the test and the longer the time period during which oxidation can take place. It was further suggested (ref. 6) that oxidation may (1) act to strengthen the metal adjacent to, and ahead of a crack, (2) blunt the tip of a crack, thereby reducing the stress concentration at that point, or (3) bridge the crack and increase the load-bearing area. It is conceivable that these mechanisms may be operative either individually or jointly, depending upon the material and the conditions of temperature and stress.

Although some data regarding the effect of vacuum on the fatigue and the stress-rupture properties of metals have been obtained, additional data are needed to determine the effect of a vacuum environment on these properties for typical superalloys that may be used in aerospace applications. An investigation was therefore initiated at the NASA Lewis Research Center to determine the 1500<sup>0</sup> F axial fatigue and stress-rupture properties of two common superalloys, a cobalt-base alloy (S-816) and a nickel-base alloy (Inconel 550). Fatigue and stress-rupture tests were conducted with both alloys in air at atmospheric pressure and at vacuum levels ranging between 10<sup>-4</sup> to 10<sup>-5</sup> millimeter of mercury. Ratios of cyclic to mean stress of 0.667 and 0.125 were maintained in the fatigue tests. The mean stress ranged from 15 000 to 40 000 psi. Metallurgical studies were made with each alloy in order to obtain additional insight into the complex interactions that occur between a metal and its environment as well as within the metal itself when it is subjected to constant loads or repeated load cycling at temperature.

## MATERIALS, APPARATUS, AND PROCEDURE

### Materials Studied

Two wrought materials, a cobalt-base alloy (S-816) and a nickel-base alloy (Inconel 550) were selected for this investigation. Both alloys are of the precipitation-hardening type, although they differ with respect to the precipitating phases that contribute to their strength at elevated temperatures. For example, in Inconel 550, which con-

TABLE I. - CHEMICAL ANALYSIS OF ALLOYS TESTED FOR THIS INVESTIGATION

Alloy	Nominal analysis														
	Nickel	Cobalt	Chromium	Iron	Columbium	Molybdenum	Tungsten	Titanium	Aluminum	Manganese	Silicon	Carbon	Zirconium	Sulfur	Copper
S-816	19.30	41.95	20.13	3.74	3.92	3.84	4.45	----	----	1.34	0.52	0.432	<0.001	0.008	----
Inconel 550	71.99	-----	15.41	6.88	0.91	----	----	2.48	1.24	0.59	0.38	0.05	-----	0.007	0.04

TABLE II. <sup>A11</sup> HEAT TREATMENT OF ALLOYS PRIOR TO MACHINING

Alloy	Heat treatment
S-816	2150° F - 1 hr - Water quench
	1400° F - 16 hr - Air cool
Inconel 550	2150° F - 1 hr - Air cool
	1600° F - 4 hr - Air cool
	1350° F - 4 hr - Air cool

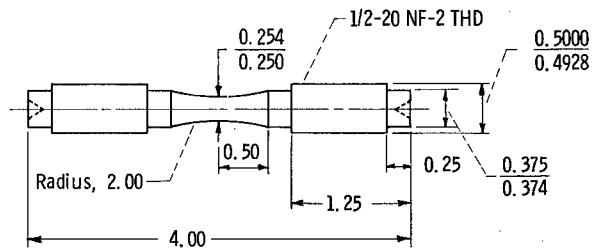


Figure 1. - Test specimen. (All dimensions are in inches.)

tains a relatively large percentage of titanium, precipitation hardening probably occurs through the formation of the Ni<sub>3</sub> (Al, Ti) intermetallic compound ( $\gamma'$  phase) and the Ni<sub>3</sub>Ti intermetallic compound ( $\eta$  phase, (ref. 10)). In S-816, precipitation hardening occurs primarily through carbide precipitation. The nominal compositions of the alloys are listed in table I.

The material was received as 3/4-inch-diameter bar stock. All of the bar stock in each alloy group was from the same heat. The as-received stock of each alloy was cut into 4-inch lengths that were given the heat treatments listed in table II.

### Test Specimens

A cylindrical-type specimen with an hourglass-shaped test section (fig. 1) was chosen for this investigation. In order to reduce any detrimental effects of surface condition on fatigue life, the test section was polished in an axial direction to a finish better than 4 microinches. External threads were provided for gripping. The type of specimens used was identical for both fatigue and stress-rupture tests.

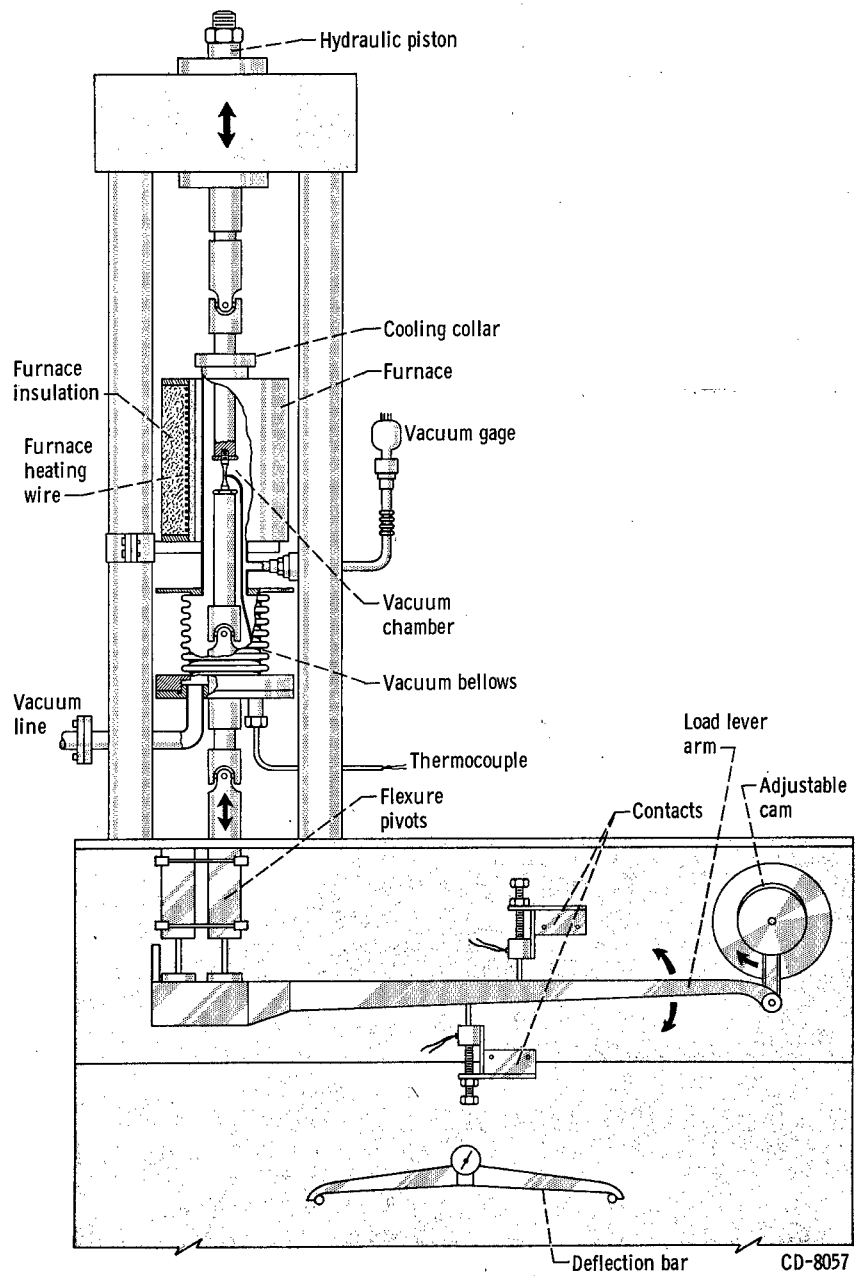


Figure 2. - Fatigue test apparatus.

## Apparatus and Instrumentation

Figure 2 illustrates the fatigue test installation. The major components included a direct tensile stress fatigue testing machine, a vacuum chamber, vacuum pumping station, and a resistance heated tube furnace.

Briefly, the fatigue machine was operated in the following manner. A mean tensile load was applied to the specimen by means of a hydraulic piston. The piston in conjunction with an electronic load controller compensated for changes in specimen length during the test to maintain a constant mean load. Sinusoidal alternating loads were superimposed on the mean load by means of a calibrated, cam-operated lever arm at a frequency of 1970 cycles per minute. A more detailed description of the machine is given in reference 11.

Specimens were enclosed in a tubular vacuum chamber having a  $2\frac{1}{4}$ -inch outside diameter. The vacuum chambers used were made from Inconel or stainless steel. A resistance wound furnace surrounded the vacuum chamber. The lower end of the chamber extended below the furnace and was joined to a brass bellows. The bellows permitted application of the load as well as elongation of the specimen during the test without constraint from the chamber walls. An absolute pressure level ranging from  $10^{-4}$  to  $10^{-5}$  millimeter of mercury was maintained with a 5-inch-diameter diffusion pump backed by a 5-cubic-foot-per-minute mechanical pump. The degree of vacuum was measured with a hot-filament ionization gage connected to the vacuum chamber at a point just above the bellows, approximately 6 inches from the test specimen. Four thermocouples were attached to each test specimen, two at the center of the test section on diametrically opposite sides of the specimen and one on each shoulder. The central thermocouples were wired to the test section; one was used to control the specimen test section temperature within  $\pm 10^{\circ}$  F of the  $1500^{\circ}$  F test temperature, and the other was connected to a continuous strip chart recorder. The shoulder thermocouples were spotwelded in place and also provided a continuous recording of specimen temperature. In this way, the temperature distribution along the length of the specimen was continually monitored. Specimen life was measured in hours by a timer that shut off automatically upon specimen failure.

## Test Procedure

Fatigue and stress-rupture tests. - Two series of tests were conducted with each material, S-816 and Inconel 550. In one series, the specimens were subjected to a vacuum environment; while in the second series, the chamber surrounding the specimen was vented to room air. In both series, specimen test section temperature was maintained at  $1500^{\circ} \pm 10^{\circ}$  F, and tests were conducted for three ratios of cyclic stress to mean stress (0.667, 0.125, and 0) over a range of mean stress from 15 000 to 40 000 psi. The condition of zero cyclic- to mean-stress ratio of course represents a stress-rupture test. The ranges of mean tensile stress applied were determined primarily by the practical limita-

TABLE III. SUMMARY OF FATIGUE DATA FOR  
S-816 TESTED AT 1500° F

Cyclic stress Mean stress	Test environment	Mean stress, psi	Life			
			Cycles	Hours		
0.125	Air	40×10 <sup>3</sup>	0.62×10 <sup>6</sup>	5.2		
		35	1.95	16.5		
		30	7.93	67.0		
		27	22.43	189.6		
		24	43.20	365.3		
	Vacuum	40×10 <sup>3</sup>	1.44×10 <sup>6</sup>	12.2		
		35	3.05	25.8		
		30	10.10	85.4		
		30	13.58	114.8		
		27	46.40	392.4		
		25	69.10	584.7		
		0.667	Air	40×10 <sup>3</sup>	0.059×10 <sup>6</sup>	0.5
				37.5	.20	1.7
				35	.79	6.7
35	1.01			8.5		
30	2.96			25.0		
30	2.98			25.2		
30	3.98			33.7		
26	14.76			124.8		
22	70.10			593.1		
Vacuum	40×10 <sup>3</sup>			0.38×10 <sup>6</sup>	3.2	
	37.5	.84	7.1			
	35	2.67	22.6			
	32	3.63	30.7			
	30	12.80	108.0			
	28	17.0	144.0			
	26	<sup>a</sup> 174.4	1474.8			

<sup>a</sup>Did not fail.

in air and long time (low stress) runs in vacuum and in air for each alloy. The electron beam microprobe analyses were made by an independent laboratory.

## RESULTS

The results of 1500° F fatigue and stress-rupture tests of alloys S-816 and Inconel 550 in vacuum and in air are summarized in tables III to V. These data are also plotted in figures 3 to 5 with least-squares lines drawn through the data. Photomicrographs of

tions of test length. All test conditions are listed in tables III to V.

Metallographic studies. Failed specimens of both alloys were examined metallographically to study the fracture mode as well as the effect of atmospheric environment upon microstructure. Photomicrographs were taken in the region of fracture at magnifications of 100, 500, or 750.

Hardness measurements. - Knoop microhardness measurements with a 1000-gram load were made on representative samples of both vacuum and air tested specimens of each material. Measurements were taken on a polished surface parallel to the longitudinal axis within 1/4 inch of the fracture surface.

Electron beam microprobe analyses. - In order to supplement the metallographic studies, four tested specimens of each material were subjected to electron beam microprobe analysis. The analyses were made to determine if depletion of alloying constituents had occurred during test. Specimens chosen for analysis were representative of short

time (high stress) runs in vacuum and

TABLE IV. SUMMARY OF FATIGUE DATA FOR INCONEL

550 TESTED AT 1500° F

Cyclic stress Mean stress	Test environment	Mean stress, psi	Life		
			Cycles	Hours	
0.125	Air	40×10 <sup>3</sup>	2.15×10 <sup>6</sup>	18.2	
		35	4.56	38.6	
		30	10.90	92.3	
		25	24.63	208.3	
		15	161.5	1366.3	
	Vacuum	40×10 <sup>3</sup>	4.85×10 <sup>6</sup>	41.0	
		35	9.56	80.6	
		30	11.70	98.7	
		30	13.13	110.9	
		30	16.73	141.5	
		25	29.54	249.9	
0.667	Air	40×10 <sup>3</sup>	0.13×10 <sup>6</sup>	1.1	
		35	.92	7.8	
		30	2.73	23.1	
		25	10.80	91.3	
		20	28.33	239.6	
		15	118.0	998.0	
		Vacuum	40×10 <sup>3</sup>	0.38×10 <sup>6</sup>	3.2
			40	2.25	19.0
	40		2.52	21.3	
	37.5		2.48	21.0	
	35		2.96	25.0	
	35		3.80	32.2	
	30		5.95	50.3	
	30		9.40	79.5	
	25		16.93	144.2	
	20	33.10	280.0		
	15	98.0	829.9		

TABLE V. SUMMARY OF STRESS-  
RUPTURE DATA FOR INCONEL

550 AND S-816 AT 1500° F

Alloy	Test environment	Stress, psi	Life, hr	
Inconel 550	Air	40×10 <sup>3</sup>	18.8	
		37	35.0	
		35	56.2	
		30	97.8	
		30	100.2	
		26	219.0	
	Vacuum	15	1980.4	
		15	2173.2	
		40×10 <sup>3</sup>	39.2	
		35	115.3	
		30	143.9	
		26	311.2	
S-816	Air	34×10 <sup>3</sup>	19.4	
		34	21.7	
		30	28.6	
		27	55.7	
		25	163.9	
		22	387.5	
	Vacuum	35×10 <sup>3</sup>	34.2	
		34.5	59.0	
		30	99.9	
		25	464.7	

failed specimens are shown in figures 6 to 9, and the results of electron beam microprobe analyses are presented in figures 10 and 11.

### Fatigue Data

S-816. - The axial tensile fatigue data obtained with S-816 in vacuum and in air are plotted in figure 3. Data were obtained only for the inclined portion of the S-N plot, and no attempt was made to determine endurance limits. Comparison of the least-squares lines indicates that for a ratio of cyclic to mean stress of 0.125 (fig. 3(a)) life in vacuum

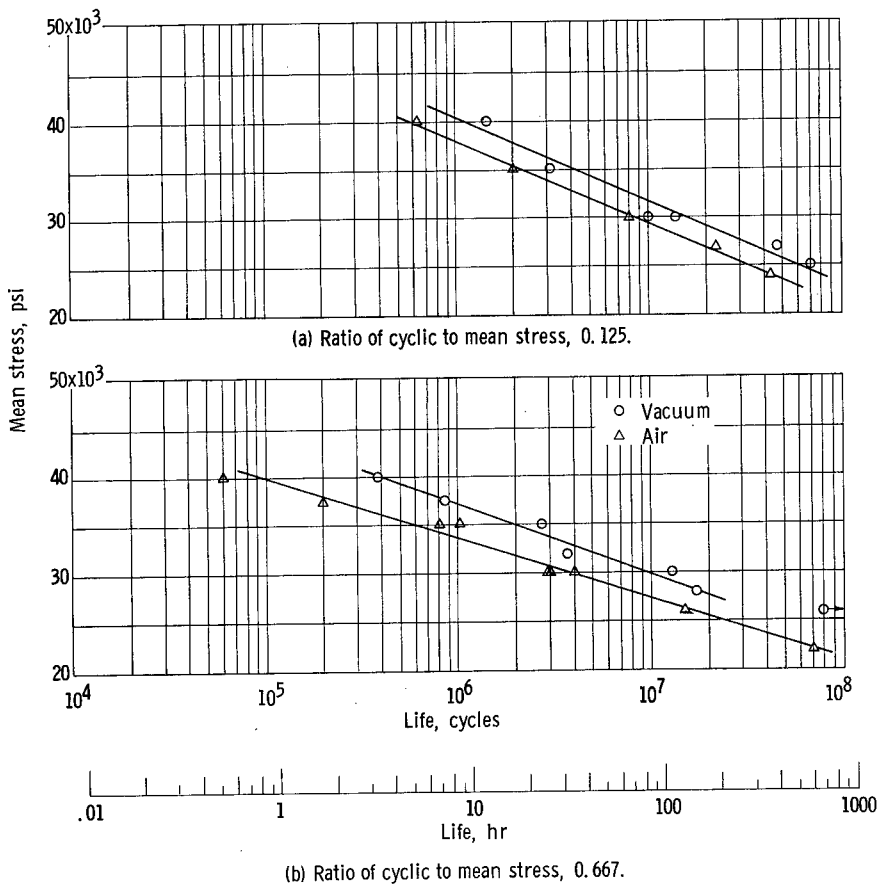


Figure 3. - Axial tensile fatigue properties of S-816 at 1500<sup>o</sup> F in both vacuum and air.

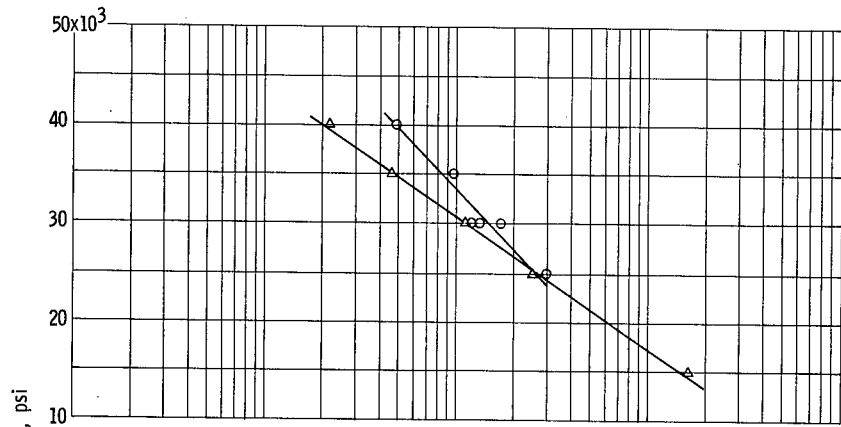
→ Pg 10

was approximately twice that in air over the stress range investigated. A similar comparison for a cyclic- to mean-stress ratio of 0.667 (fig. 3(b)) indicates that life in vacuum was approximately two to four times that in air depending upon the stress level. For example, at the highest stress, 40 000 psi, the curves indicate lives of approximately  $9.0 \times 10^4$  and  $4.0 \times 10^5$  cycles in air and in vacuum, respectively. These values compare with  $7.6 \times 10^6$  and  $1.8 \times 10^7$  cycles at the low stress of 28 000 psi.

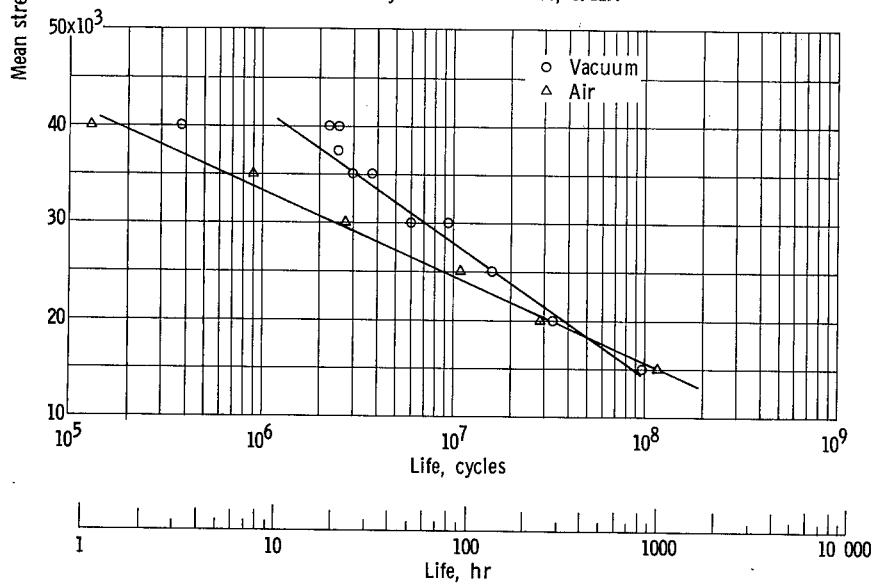
Inconel 550. - Plots of the 1500<sup>o</sup> F axial tensile fatigue data for Inconel 550 (fig. 4) also show that the lives in vacuum are generally longer than those in air although the lines drawn through the vacuum and air data intersect. For a ratio of cyclic stress to mean stress of 0.125, the intersection occurs at 25 000 psi (fig. 4(a)). For a cyclic- to mean-stress ratio of 0.667, it occurs at about 18 000 psi (fig. 4(b)). At the highest stress of 40 000 psi, lives in vacuum are approximately two and eight times those in air for cyclic- to mean-stress ratios of 0.125 and 0.667, respectively.

### Stress-Rupture Data

S-816. - A comparison of the 1500<sup>o</sup> F stress-rupture properties of S-816 in air and



(a) Ratio of cyclic to mean stress, 0.125.



(b) Ratio of cyclic to mean stress, 0.667.

Figure 4. - Axial tensile fatigue properties of Inconel 550 at 1500° F in both vacuum and air.

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pg 11

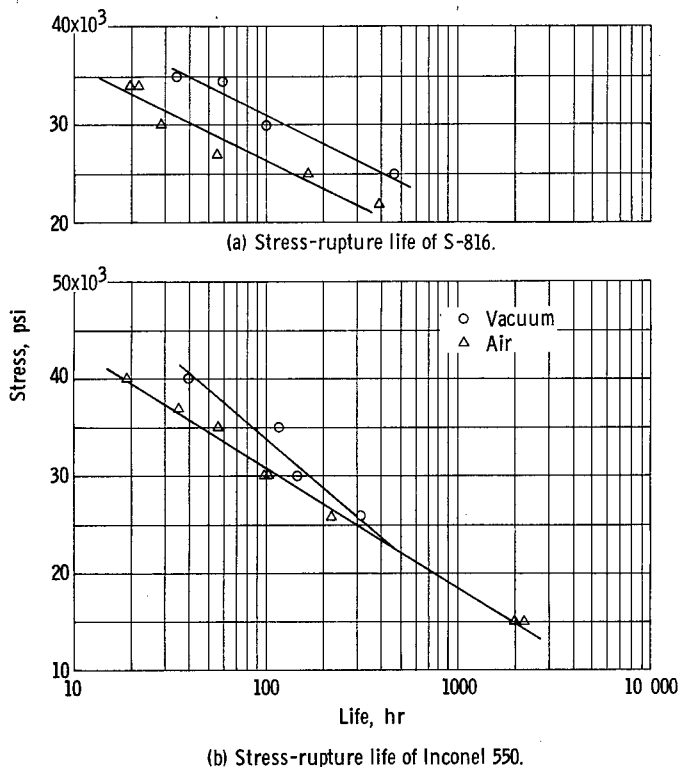


Figure 5. - Stress-rupture properties of S-816 and Inconel 550 at 1500° F in both vacuum and air.

*end*

in vacuum is shown in figure 5(a). Life in vacuum was approximately three times that in air over the entire stress range investigated.

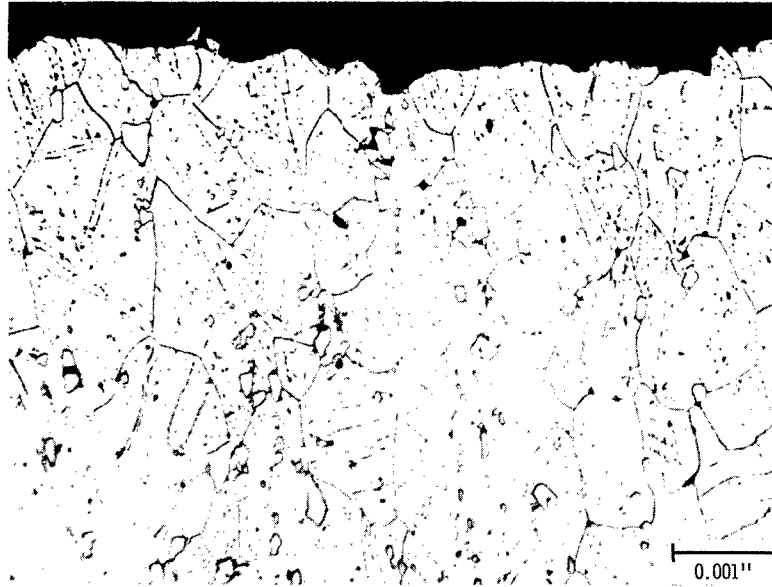
Inconel 550. - Figure 5(b) compares the 1500° F stress-rupture properties of Inconel 550 in vacuum and in air. A convergence of the data similar to that obtained for this material in fatigue tests was observed. The lines drawn through the vacuum and air data appear to converge at approximately 22 000 psi. At the highest stress of 40 000 psi, life in vacuum (44 hr) was approximately twice that in air (18 hr).

## Metallurgical Studies

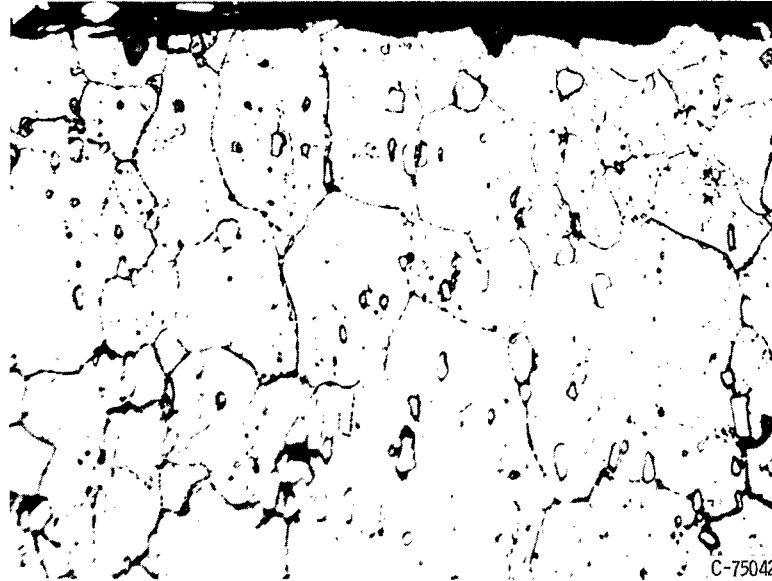
Metallography. - Photomicrographs at magnifications of 500 and 100

of failures typical of those encountered for a cyclic- to mean-stress ratio of 0.667, in both vacuum and air with S-816 and Inconel 550 are shown in figure 6. Similarly, photomicrographs of stress-rupture failures typical of those encountered in vacuum and air are shown in figure 7. It will be noted that the photomicrographs of S-816 are magnified 500 times and those of Inconel 550 are magnified 100 times. Because of the difference in grain size of the two materials, it was necessary to photograph them at different magnifications in order to better illustrate the mode of fracture. Failures for a cyclic- to mean-stress ratio of 0.667 were transgranular in nature for each alloy regardless of test environment, but for a cyclic- to mean-stress ratio of 0.125, the mode of fracture was primarily intergranular although the transgranular mode was also evident. Although the mode of fracture of S-816 in stress rupture is not readily distinguishable from the photomicrographs (fig. 7(a)), microscopic studies indicated that it was primarily intergranular in both vacuum and air tests. Stress-rupture failures in Inconel 550 were also intergranular in both vacuum and in air, as is readily apparent from figure 7(b).

Figures 8 and 9 show photomicrographs of typical tested specimens (fatigue or stress rupture) of each alloy along the external surface within 1/4 inch from the point of fracture. Short, intermediate, and long test times are represented. Figure 8 shows photomicrographs (magnified 500 times) of specimens of both alloys after testing in vacuum. No external scale was apparent on either alloy, regardless of test time; however,



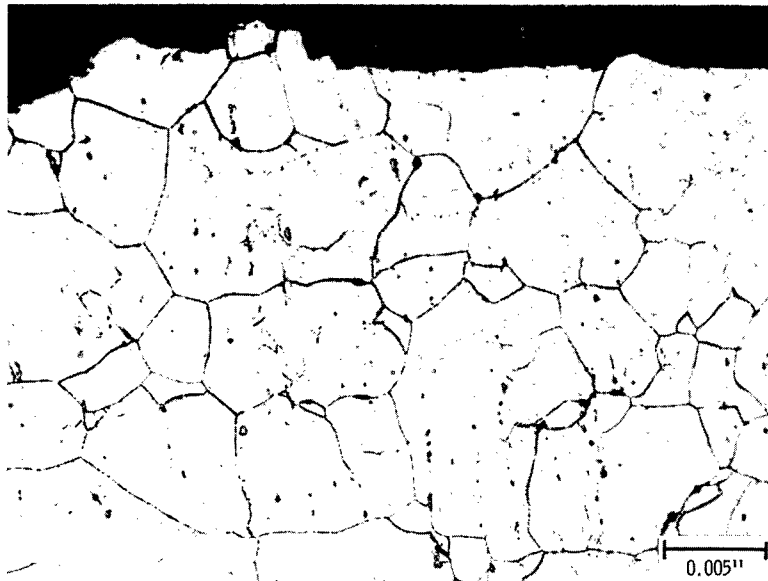
Life in vacuum, 144 hr



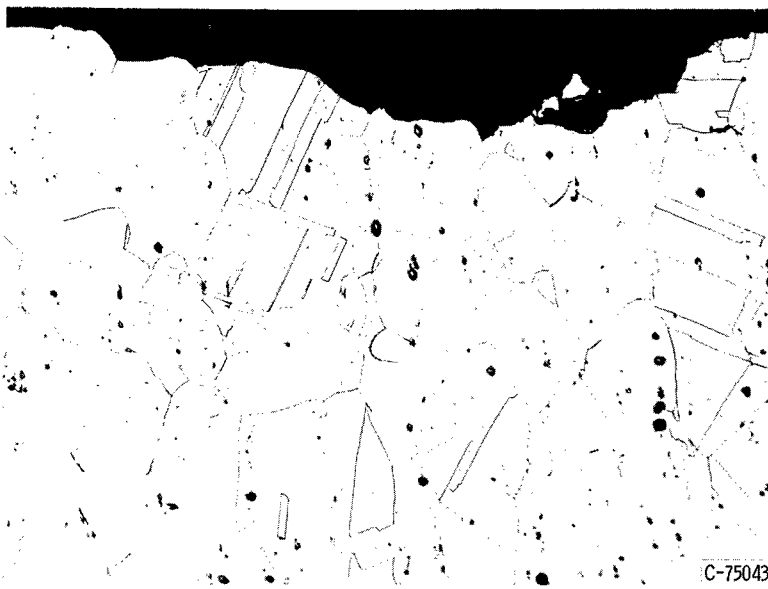
Life in air, 6.7 hr

(a) S-816 alloy. X500.

Figure 6. - Photomicrographs showing transgranular fractures typical of failures encountered in fatigue tests in both vacuum and air. Ratio of cyclic to mean stress, 0.667.



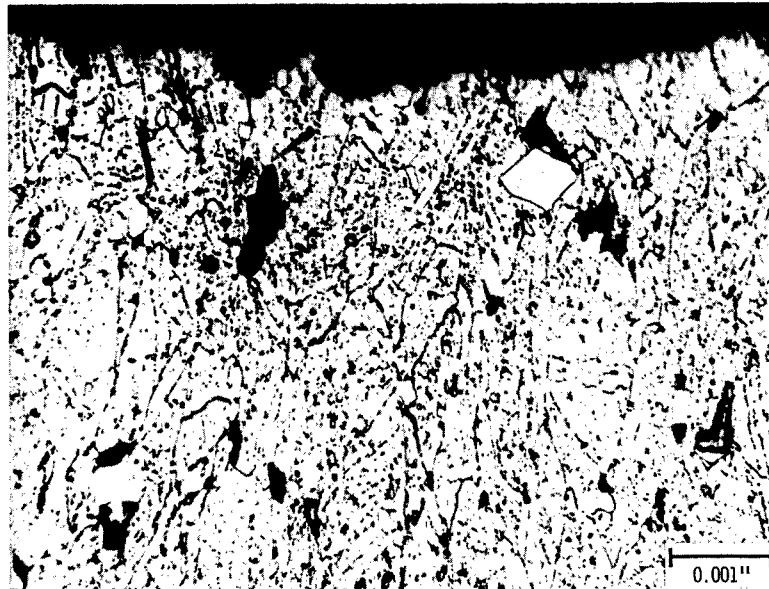
Life in vacuum, 79.5 hr



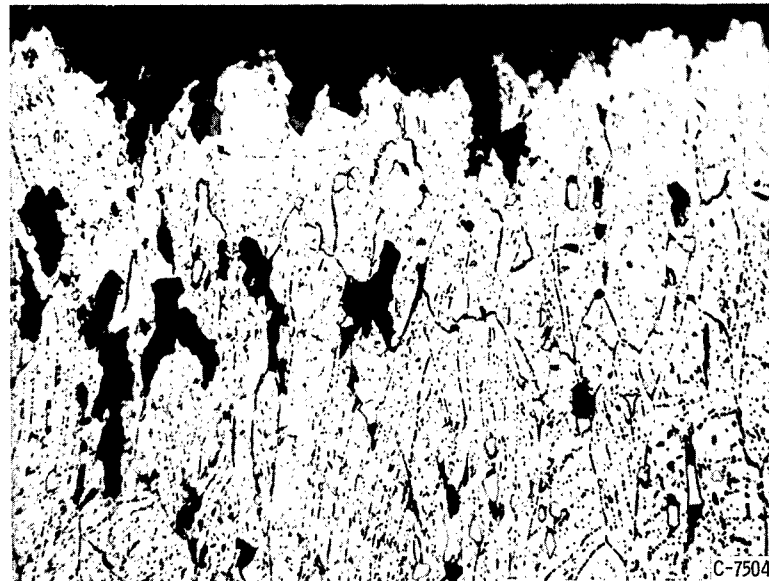
Life in air, 1.1 hr

(b) Inconel 550 alloy. X100.

Figure 6. - Concluded.



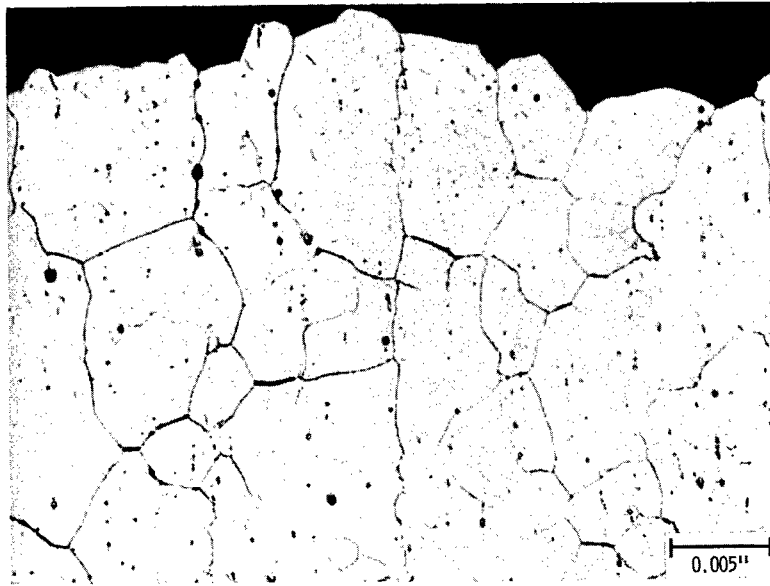
Life in vacuum, 99.9 hr



Life in air, 21.7 hr

(a) S-816 alloy. X500.

Figure 7. - Photomicrographs showing fractures typical of failures encountered in stress-rupture tests in both vacuum and air.



Life in vacuum, 143.9 hr



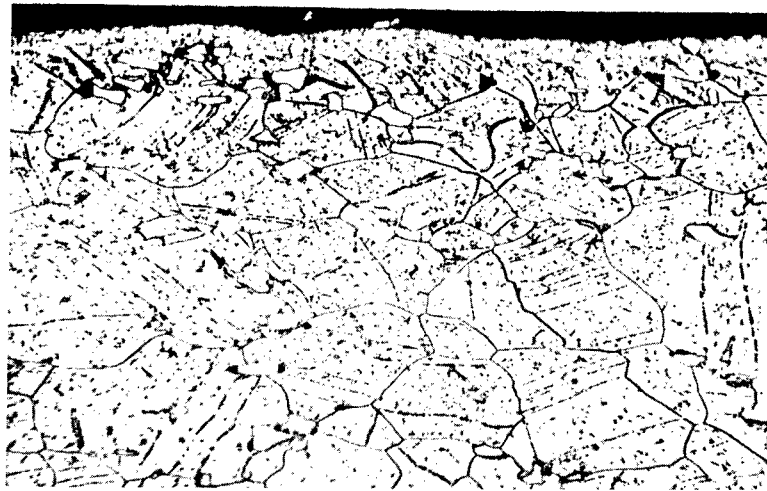
Life in air, 100.2 hr

(b) Inconel 550 alloy. X100.

Figure 7. - Concluded.



Testing period, 7.1 hr



Testing period, 144 hr



Testing period, 1475 hr

(a) Fatigue-tested S-816 alloy.

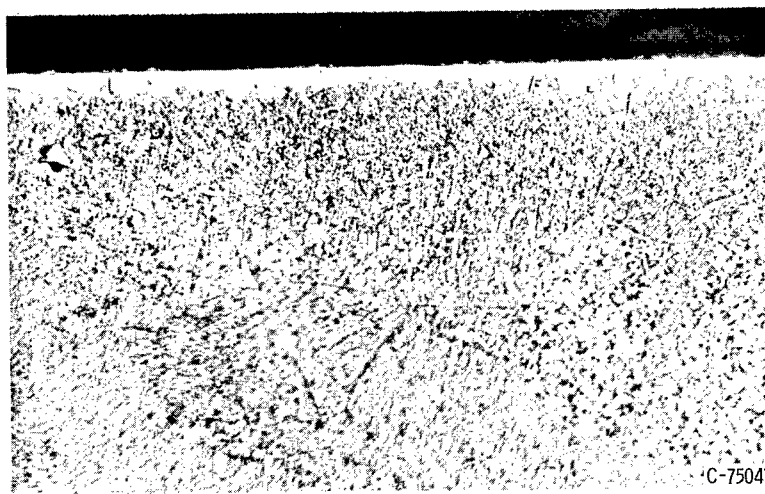
Figure 8. - Photomicrographs showing edges of typical specimens tested in vacuum. Ratio of cyclic to mean stress, 0.667. X500.



Testing period, 32 hr



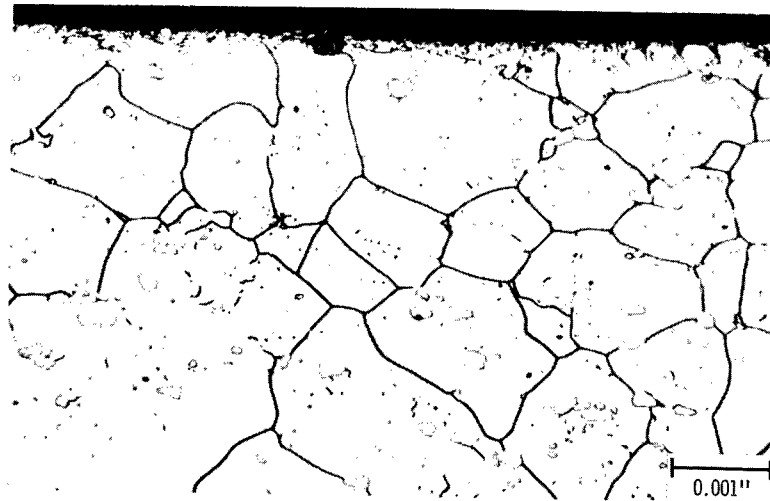
Testing period, 80 hr



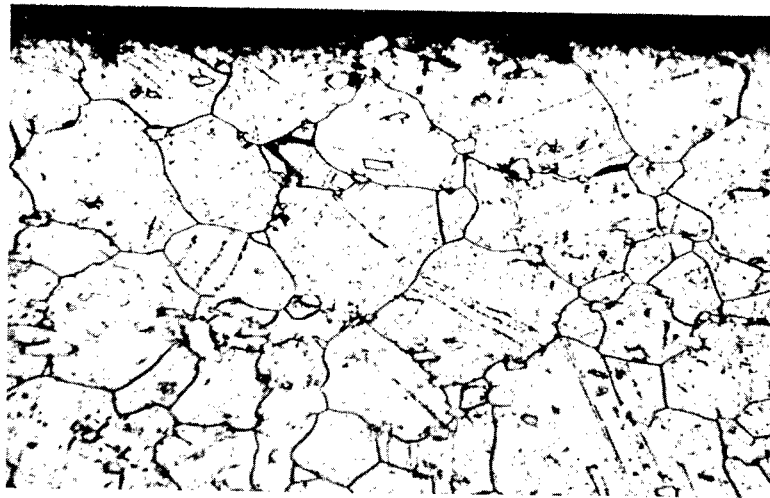
Testing period, 830 hr

(b) Fatigue-tested Inconel 550 alloy.

Figure 8. - Concluded.



Testing period, 0.5 hr



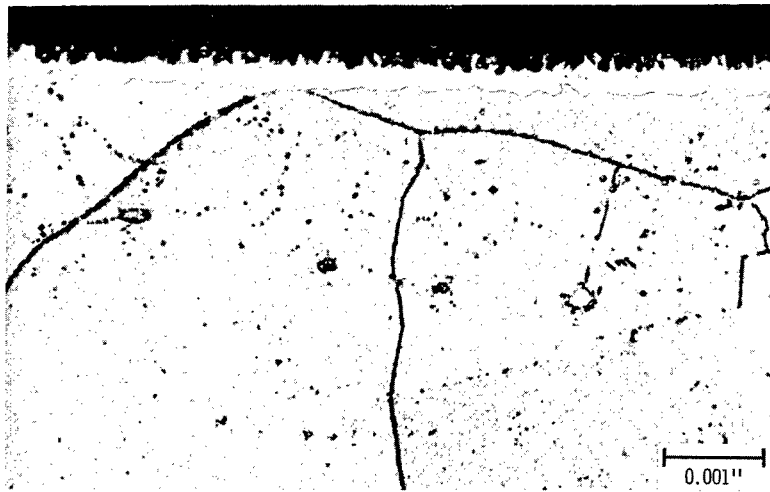
Testing period, 6.7 hr



Testing period, 593 hr

(a) Fatigue-tested S-816 alloy.

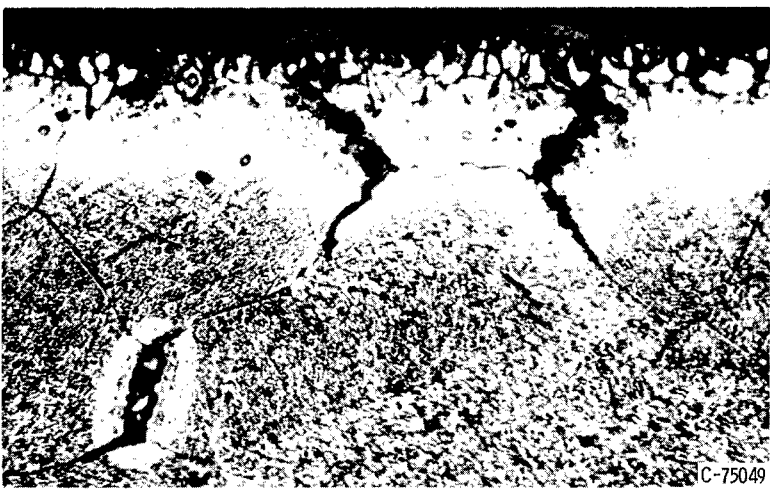
Figure 9. - Photomicrographs showing edges of typical specimens tested in air. Ratio of cyclic to mean stress, 0.667. X500.



Testing period, 35 hr



Testing period, 100.2 hr



Testing period, 1980 hr

(b) Stress-rupture-tested Inconel 550 alloy.

Figure 9. - Concluded.

the microstructure of each alloy appeared to be affected to varying depths beneath the specimen surface. This is not as apparent for S-816 as for Inconel 550. Some depletion of alloying elements apparently occurred in these zones. This will be discussed in connection with the results of electron beam probe analyses in a subsequent section of this report. The affected zone is most evident in Inconel 550 (fig. 8(b)). Figure 9 presents similar photomicrographs of specimens of both alloys after various test times in air. An adherent external scale was formed during test on both alloys. Scale thickness increased with increased test time. The depth of the affected zone in Inconel 550 was greater than in S-816. From a comparison of the scale thickness of the 593-hour test in figure 9(a) and the 100-hour test in figure 9(b), it is evident that more oxidation occurred in Inconel 550 than in S-816. This was also borne out by metallographic studies of most of the tested specimens. From the metallographic evidence, it appears that oxidation occurs at a slower rate in S-816 than in Inconel 550. The photomicrographs of figures 8 and 9 also show aging effects for both materials with increasing test time at 1500<sup>0</sup> F. As test time increased, the size of the precipitates in the regions away from the surface increased. This is particularly evident for S-816 (figs. 8(a) and 9(a)).

Microhardness. - Samples of each alloy were chosen to permit comparison of material hardness after vacuum and air tests at high and low stress levels. A summary of these data is given in table VI. For S-816, the average microhardness of the metal matrix (region away from surface) was higher for vacuum-tested material than air-tested material at both extremes of stress. The average Knoop hardness values were 410 (vacuum tested) against 375 (air tested) at the high stress condition and 392 (vacuum tested) against 368 (air tested) at the low stress condition. For constant stress, these differences in hardness may be attributed to the longer lives obtained in the vacuum tests. Thus longer time was available for increased precipitation and growth of carbides. The

TABLE VI. - SUMMARY OF MICROHARDNESS DATA

Alloy	Test environment	Mean stress, psi	Life, hr	Average Knoop hardness
S-816 (As heat treated)	-----	-----	-----	294
S-816	Vacuum	40×10 <sup>3</sup>	12.2	410
	Air	40	5.2	375
	Vacuum	27	392.4	392
	Air	27	189.6	368
Inconel 550 (As heat treated)	-----	--	-----	344
Inconel 550	Vacuum	40	39.2	324
	Air	40	18.8	329
	Vacuum	15	829.9	281
	Air	15	998.0	287

fact that the hardnesses of specimens tested at the high stress are higher than the ones tested at the low stress probably can be attributed to the relatively large amount of plastic deformation and subsequent work hardening that takes place at high stress.

For Inconel 550 there was no significant difference in hardness for vacuum- and air-tested specimens for constant stress conditions. However, there was a noticeable difference between the hardness of speci-

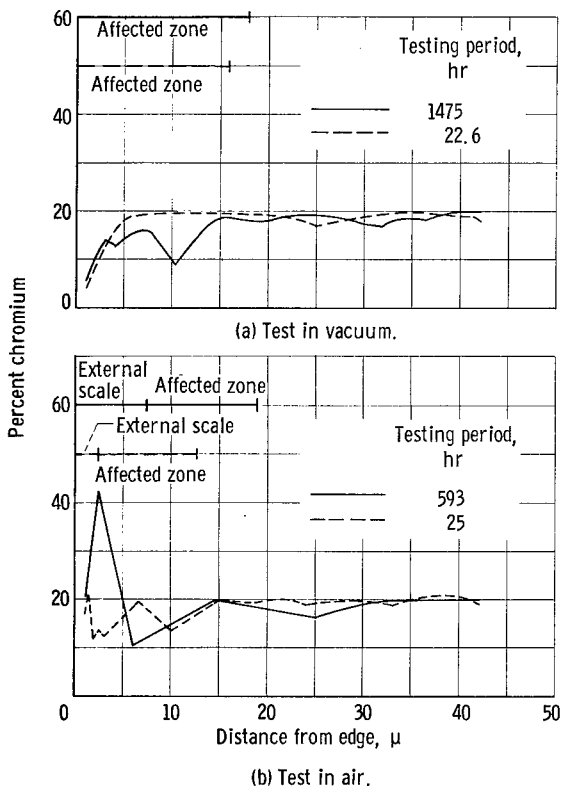


Figure 10. - Comparison of distribution of chromium near surface of S-816 specimens tested in vacuum and air.

mens tested at high and low stress levels regardless of environment. Thus the Knoop hardness values ranged from 324 to 329 for the samples tested at high stress and 281 to 287 for the samples tested at low stress (long life). This difference is probably a reflection of the decrease in hardness attendant to an overaged condition after long time testing with this material. For Inconel 550 there was little apparent difference in plastic deformation between specimens tested at high stress and at low stress.

Attempts were also made to obtain microhardness readings in the affected zone immediately adjacent to the specimen surface for these alloys. These zones, however, were too narrow to permit meaningful readings to be taken.

Electron beam microprobe analyses. - To provide an indication of possible changes in chemical composition, particularly in the region near the specimen surface, electron beam

microprobe analyses were made of specimens of each alloy. Vacuum- and air-tested specimens that had been tested for short times as well as relatively long times were analyzed.

S-816 specimens were analyzed for five elements, tungsten, iron, chromium, nickel, and cobalt. In general, there was some depletion of these elements in the immediate vicinity of the specimen surface, but only the distribution of chromium was markedly affected. Figure 10 provides a comparison of the microprobe analyses results for chromium made on vacuum- and air-tested specimens that were subjected to short and long time tests. The thickness of the external scale and the depth of the affected zone as determined by metallographic inspection are also shown in the figure. In an attempt to account for specimen edge effects in the electron beam microprobe analysis, the curves have been terminated at a distance of 1 micron from the specimen edge. The specimens that were tested for long periods of time in either vacuum (fig. 10(a)) or air (fig. 10(b)) showed a considerable chromium depletion in the affected zone. In the case of the vacuum-tested specimens, however, there was no buildup of a chromium rich external scale as was the case for the air-tested specimens. The large buildup of chromium observed in the external scale of air-tested specimens was probably due to outward diffusion

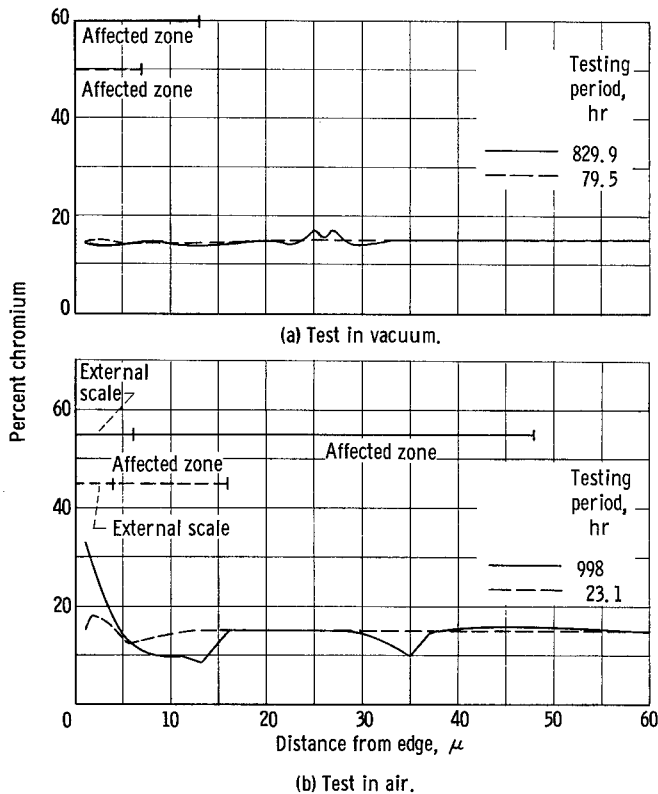


Figure 11. - Comparison of distribution of chromium near surface of Inconel 550 specimens tested in vacuum and air.

of chromium and its subsequent oxidation in the air environment. This effect was not nearly so pronounced for the short time air tests as in the long time air tests.

Representative Inconel 550 specimens were analyzed in the same manner as the S-816 specimens. Analyses were made for six elements, nickel, chromium, iron, titanium, aluminum, and columbium. There was, except for chromium, very little depletion of any of these elements in the affected zone; figure 11 shows the microanalyses results for chromium with both vacuum- and air-tested specimens. There is virtually no difference between the probe trace obtained for the specimen tested for a relatively short time (79.5 hr) and that tested for a long time (829.9 hr) in vacuum. Negligible de-

pletion was observed in the affected zone in either case (fig. 11(a)). The air-tested specimens (fig. 11(b)), showed a substantial chromium buildup in the external scale after both the long and short time tests. In conjunction with this buildup some depletion of chromium occurred in the affected zone adjacent to the external scale as was the case for S-816.

## DISCUSSION OF RESULTS

In the following sections, comparisons are made with data obtained by other investigators; some of the metallurgical factors involved are discussed. It should be noted that the comparisons deal with a variety of tests, specimen configurations, and test conditions.

### Considerations Pertinent to Nickel-Base Alloys

The Inconel 550 data for air and vacuum tests show trends similar to those obtained with nickel and several nickel-base alloys in the investigations of references 2 to 7. For



Figure 12. Evidence of blunting of crack tip by oxide in Inconel 550 specimen fatigue tested for 1366 hours. Ratio of cyclic to mean stress, 0.667. X750.

example, comparisons of air and vacuum data obtained in creep-rupture tests with nickel (ref. 4), Nichrome V (ref. 5), and an aluminum-chromium-nickel base alloy (ref. 6) showed life in vacuum to be greater than life in air at high stress levels. The reverse was generally true at low stress levels. A temperature effect was also observed for Nichrome V and the aluminum-chromium-nickel base alloy (refs. 5 and 6), where the crossover between air and vacuum data occurred at successively lower lives with increasing test temperature. The more limited fatigue data available showed that a similar crossover between life in air and in vacuum occurred at 1500<sup>o</sup> F for nickel (ref. 8). Thus, under steady stress as well as under alternating stress conditions, the results obtained in the present investigation with Inconel 550 tend to agree with those obtained in earlier work with nickel-base materials.

The longer life observed in nickel and nickel-base alloys at low stresses in an air environment as compared to a vacuum environment has been attributed to the strengthening effect of oxidation (refs. 4, 6, and 12). Figure 12 provides evidence that appreciable oxidation occurred in the crack tip of an Inconel 550 specimen tested in fatigue for 1366 hours. It is possible that this oxide could have acted to reduce the stress concentration effect of the crack as suggested in reference 6, thereby tending to increase specimen life. The possibility that the oxide shown in figure 12 could be sufficiently strong and adherent

to act as a strengthener may be inferred from results obtained in the investigation of reference 13. There it was reported that a nickel specimen consisting of two sections joined by sintered nickel oxide was capable of supporting loads comparable to those carried by a homogeneous nickel specimen.

There are, of course, other ways in which oxidation may act to strengthen a material. One of these is by internal oxide formation. No clear-cut evidence of internal oxidation was obtained with either of the alloys investigated, although there was an indication of possible internal oxidation in Inconel 550. Figure 9(b) shows what may be internal oxidation around cracks that extend through the affected zone of a specimen fatigue tested in air for 1980 hours.

Finally, it is of interest to note that longer creep and stress-rupture life has been observed with nickel and nickel-base materials in air than in such diverse environments as hydrogen (ref. 14), nitrogen (ref. 15), argon (refs. 12 and 16), and sodium hydroxide (ref. 12). Although life may have been deleteriously affected by these environments, the fact that longer lives were observed in air than in all of these environments suggests that oxidation is indeed an important strengthening factor for these materials at elevated temperature.

### Considerations Pertinent to Cobalt-Base Alloys

The fact that longer lives were obtained with S-816 in vacuum as compared to air over the entire stress range investigated both in fatigue and stress rupture, does not appear to agree with earlier work using this material (ref. 3). Only partial comparisons are possible, because limited stress-rupture data (two stress levels) were obtained by the investigators of reference 3. Consideration of the metallographic investigation does not provide a clear-cut reason for the results obtained. For example, S-816 was depleted of several alloying constituents (i. e., chromium, nickel, tungsten, and iron) near the surface in long time tests in both vacuum and air. Chromium was the element that was most notably affected, however. The fact that the life of S-816 in a vacuum showed an improvement over life in air indicates that the effect of depletion of the elements for which microprobe traces were made was clearly not overriding in the vacuum environment of this investigation.

It might be expected from a high-chromium-content cobalt-base alloy such as S-816 that appreciable evaporative loss of chromium would occur during long time exposure in a vacuum environment since chromium has a high evaporative loss rate in vacuum (ref. 17). Since chromium makes up 20 weight percent of S-816 the possibility of chromium diffusion to the surface and its subsequent evaporation is clearly suggested. Thus, although chromium depletion was observed in this investigation in air-tested specimens

as well as in the vacuum-tested specimens, it is likely that upon long time (10 000 hr) exposure to the much higher vacuum of space, appreciably greater depletion of this element and subsequent structural deterioration of the alloy would occur. A similar result might be expected from other high-chromium-content (i. e., between 20 and 25 weight percent) cobalt-base alloys.

Greater ductility was observed in S-816 than in Inconel 550 in an earlier study dealing with the thermal fatigue resistance of these two alloys (ref. 18). This was also noted in the present investigation where appreciably greater necking occurred in tested S-816 specimens than in specimens of Inconel 550. This greater elongation during test may have had a tendency to disrupt the buildup of oxides in cracks such as occurred in Inconel 550, thereby destroying the beneficial effect of such oxides in reducing stress concentrations. Finally, the observation that S-816 appeared to oxidize at a slower rate in an air environment than Inconel 550 as noted earlier, may also be a significant consideration. As a result of this, the formation of oxides in growing cracks would be slower, and the tendency to bridge cracks and reduce their stress concentration effect as suggested in reference 6 would be impaired. It is possible that both of these factors may account for the longer life of S-816 in vacuum than in air in contrast to the opposite trend observed for Inconel 550 at low stress levels.

## SUMMARY OF RESULTS

An investigation was conducted to determine the relative effects of vacuum and air upon the 1500° F axial fatigue and stress-rupture properties of two high-temperature alloys, S-816 and Inconel 550. Fatigue and stress-rupture tests were run in air at atmospheric pressure and in vacuum ( $10^{-4}$  to  $10^{-5}$  mm Hg). Fatigue tests were run at ratios of cyclic to mean stress of 0.667 and 0.125. Mean stress ranged from 15 000 to 40 000 psi. The following results were obtained:

1. Life in vacuum was generally greater than life in air for both alloys in both fatigue and stress rupture.
2. The improvement in life was fairly constant for S-816, but a convergence was observed at the lower stresses for Inconel 550. For S-816, as the cyclic- to mean-stress ratio was increased from 0 to 0.125 to 0.667, the life in vacuum exceeded that in air by factors of approximately 3, 2, and 3, respectively. For Inconel 550, at the highest stress investigated (40 000 psi) life in vacuum was approximately twice that in air for the lower ratios and about eight times as great at the highest ratio. Convergence occurred at stresses ranging between 18 000 and 25 000 psi.
3. Fatigue failures at the highest cyclic- to mean-stress ratio of 0.667 were transgranular in both materials whether tested in air or vacuum. As the ratio of cyclic to

mean stress was changed to 0.125 and the conditions of a stress-rupture-type test more nearly approached, specimens failed by a combination of both transgranular and intergranular but primarily intergranular modes. Stress-rupture failures were generally intergranular in nature.

4. Oxide penetration varied with test life for both materials in air-tested specimens but did not appear to be affected by either mean stress or alternating stress. External scale deposits were thicker on the Inconel 550 specimens than on S-816.

5. Chromium depletion was observed in S-816 specimens exposed to long test times in vacuum as well as in air, and after long times in air with Inconel 550. A substantial buildup of chromium was noted in the external scale found after long time air tests with both S-816 and Inconel 550.

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### CONCLUDING REMARKS

The convergence between vacuum and air data with one of the alloys investigated (Inconel 550) suggests that it is not safe to assume that a vacuum environment improves the fatigue and stress-rupture properties of superalloys at all stress levels. Thus, long time elevated temperature tests in air may not always be used to adequately describe superalloy performance in stress rupture or fatigue under vacuum conditions. Long time tests with superalloys should be made in vacuum, thus more nearly simulating a space environment, in order to avoid miscalculations in space vehicle component designs that could result in premature failures.

→ pg 4

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, April 1, 1965.

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