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ADVANCED GAS COOLED NUCLEAR REACTOR MATERIALS EVALUATION AND DEVELOPMENT PROGRAM

PROGRESS REPORT
FOR PERIOD

January 1, 1980 - March 31, 1980

MASTER

BY
PROJECT STAFF

GENERAL ELECTRIC COMPANY
ENERGY SYSTEMS PROGRAMS DEPARTMENT
SCHENECTADY, NEW YORK 12345

GENERAL  ELECTRIC

June 25, 1980

Prepared for

THE U.S. DEPARTMENT OF ENERGY
Under Contract No. EY-76-C-02-2975

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FOREWORD

The Advanced Gas Cooled Nuclear Reactor Materials Evaluation and Development Program is being conducted for the Nuclear Power Development Division, U.S. Department of Energy, by the Energy Systems Programs Department, General Electric Company, Schenectady, New York 12345 under Contract DE-AC02-76ET34202. The Program is under the direction of Mr. J.E. Fox, DOE Program Manager, and is being managed for the General Electric Company by Mr. R.G. Frank, Manager, Materials Technology. The Principal Investigator, with responsibility for overall technical coordination of the Program is Dr. O.F. Kimball, Manager, Gas Reactor Materials, assisted by Dr. J.F. Stubbins, Materials Engineer, Gas Reactor Materials. Major contributors to the Program are:

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Section 1.0

INTRODUCTION

ABSTRACT

This report presents the results of work performed from January 1, 1980 through March 31, 1980 on the Advanced Gas-Cooled Nuclear Reactor Materials Evaluation and Development Program. The objectives of this program are to evaluate candidate alloys for Very High Temperature Reactor (VHTR) Nuclear Process Heat (NPH) and Direct Cycle Helium Turbine (DCHT) applications, in terms of the effect of simulated reactor primary coolant (helium containing small amounts of various other gases), high temperatures, and long time exposures, on the mechanical properties and structural and surface stability of selected candidate alloys. A second objective is to select and recommend materials for future test facilities and more extensive qualification programs.

Work covered in this report includes the activities associated with the status of the simulated reactor helium supply system, testing equipment and gas chemistry analysis instrumentation and equipment. The progress in the screening test program is described. This includes: screening creep results and metallographic analysis for materials thermally exposed or tested at 750, 850 and 950°C (1382, 1562 and 1742°F). In addition, the status of the data management system is described.

OTHER REPORTS IN THIS SERIES

QUARTERLY PROGRESS REPORTS

September 23, 1976 to December 31, 1976	C00-2975-3
January 1, 1977 to March 31, 1977	C00-2975-10
April 1, 1977 to June 30, 1977	C00-2975-13
July 1, 1977 to September 30, 1977	C00-2975-17
October 1, 1977 to December 31, 1977	C00-2975-20
January 1, 1978 to March 31, 1978	C00-2975-21
April 1, 1978 to June 30, 1978	C00-2975-22
July 1, 1978 to September 30, 1978	C00-2975-25
October 1, 1978 to December 31, 1978	C00-2975-28
January 1, 1979 to March 31, 1979	C00-2975-31
April 1, 1979 to June 30, 1979	C00-2975-34
July 1, 1979 to September 30, 1979	C00-2975-37
October 1, 1979 to December 31, 1979	C00-2975-40

TOPICAL REPORTS

1. Selection of Candidate Alloys	C00-2975-16
Volume 1 - Advanced Gas Cooled Reactor Systems Definition by M. Marvin	October 31, 1978
Volume 2 - Selection of Alloys for Screen- ing Creep and Structural Stability Studies by R.M. Goldhoff, R.V. Hillery, and S. Yukawa	December 31, 1978
Volume 3 - Selection of Surface Coating/ Substrate Systems for Creep and Structural Stability Studies by D.A. Grey	To be released.

ADVANCED GAS COOLED NUCLEAR REACTOR
MATERIALS EVALUATION AND DEVELOPMENT PROGRAM

1.0 INTRODUCTION

The Very High Temperature Reactor (VHTR) Nuclear Process Heat (NPH) and Direct Cycle Helium Turbine (DCHT) Systems are being developed to provide an expanded energy capability and to facilitate conservation of or substitution for scarce energy resources. In both cases, the use of high temperature helium-cooled reactors will require a knowledge of the long term properties of reactor primary coolant containment materials and of the materials used in constructing related hardware. Because of the very high temperatures required for NPH and DCHT applications, the need is evident for materials testing in the proper helium environment in the temperature range from 750^o - 1050^oC (1382^o - 1922^oF) on those alloys which might serve as materials of construction for heat exchangers, ducting, turbine blades and vanes and other high temperature structural components.

The objectives of the Advanced Gas Cooled Nuclear Reactor Materials Evaluation and Development Program are to:

1. Evaluate candidate alloys for Very High Temperature Reactor (VHTR) Nuclear Process Heat (NPH) and Direct Cycle Helium Turbine (DCHT) applications, in terms of the effect of the primary coolant and thermal exposure on:
 - Surface stability ("corrosion" resistance) in 10,000 hours.
 - Structural stability in 10,000 hours - changes in microstructure and tensile, impact and fatigue properties.
 - Creep - rupture properties in 15,000 hours.
 - Fatigue properties
 - Low Cycle 10³ to 10⁵ cycle
 - High Cycle 10⁵ to 10⁷ cycle
2. Select and recommend materials for future test facilities and more extensive qualification programs.

The data base will permit the selection of materials of construction and should provide a reliable level of design data for the construction of various items of development hardware leading to the successful development of NPH and DCHT systems. The present lack of an accurate measure of materials capabilities under these high temperature helium environments is a factor which is currently limiting the development of the NPH and DCHT systems. This program is intended to provide such initial information.

2.0 PROGRAM STATUS

The Advanced Gas Cooled Reactor Materials Evaluation and Development Program is organized into nine (9) tasks which are designed to accomplish the program objectives listed in Section 1.0 of this report. The task structures (Program Work Breakdown Structure) and a brief description of each task (Program Work Statement) have been included in the first Quarterly Report covering the period September 23, 1976 - December 31, 1976, report number COO-2975-3.

2.1 TASK 1 - SELECTION OF CANDIDATE ALLOYS

The selection of alloys for the screening phase of the program has been completed. A list of the alloys selected for the program was presented in topical report number COO-2975-16, Volume 2, dated December 31, 1978 and entitled: Selection of Alloys for Screening Creep and Structural Stability Studies.

2.2 TASK 2 - MATERIALS PROCUREMENT AND CHARACTERIZATION

2.2.1 Direct Cycle Helium Turbine (DCHT) Alloys

2.2.1.1 Materials Procurement

The procurement of all DCHT materials for the Phase 1 screening test program was completed by June 1978.

The procurement of DCHT alloys for the Phase 2 intensive screening tests continued during the quarter. Delivery of the materials is set for mid-April 1980.

2.2.1.2 Materials Characterization

This subtask was completed for the Phase 1 screening test program and the final results reported in the Progress Report for July 1, 1978 - September 30, 1978, report number COO-2975-25.

2.2.2 Nuclear Process Heat (NPH) Alloys

2.2.2.1 Materials Procurement

The procurement of all NPH materials, sufficient for both testing phases, was completed by September 1978.

2.2.2.2 Materials Characterization

The materials acceptance and characterization testing was completed in the quarter ending in December 1978.

2.2.2.3 Materials Joining Studies

The final results for Vareststraint tests were covered in the quarterly report for the period October 1, 1978 - December 31, 1978, report number COO-2975-28.

2.3 TASK 3 - TESTING AND ANALYSIS FACILITIES AND EQUIPMENT

2.3.1 Multi-Specimen Creep (MSC) Equipment

During the report period, three multispecimen creep stand furnaces were revised. The revision consisted of replacement of original windings with shorter elements designed to shorten the hot zone and relieve the axial temperature gradient believed to have been a principal contributor to earlier ceramic tube failures in these stands. Half of one furnace had previously been modified. The modified furnaces will be devoted to 950 and 1050°C (1742 and 1922°F), helium tests where ceramic retort tubes are involved. This furnace revision reduces the uniform hot zone length in these stands to 78.7 cm (31 inches).

The test equipment for the 1050°C (1922°F) helium tests has been calibrated and assembled. This particular test should be able to be started early next quarter.

2.3.2 Thermal Stability [Aging/Corrosion (A/C)] Test Equipment

During the report period, the final cleanliness verification tests on the A/C retorts were completed. All residual traces of P were successfully removed from these retorts and the retorts were certified to be acceptable for further use in specimen exposure.

In an attempt to bring a 1050°C (1922°F) A/C stand on line, high levels of N₂ were measured in the retort outlet gas. This phenomenon was thought to be associated with a possible air leak. Since all retort supply lines, fittings, end caps and fixtures accessible outside the furnace were thoroughly leak-checked before the furnace was brought to temperature, the ceramic retort tube itself was suspect as a potential source of the leak. This A/C retort and several others were removed from their furnaces and carefully inspected. The A/C retorts are made from high purity (99.8%) alpha-alumina. Two (2) of the eight (8) tubes which had seen service for He testing were found to have severe flaws which prohibited their further use. One (1) of these (2) tubes were associated with the 1050°C (1922°F) test and had two large and two small surface cracks between 2.54 and 5.08 cm (1 to 2 inches) in length. These cracks ran around the circumference of the tube, normal to the tube axis, but could not be seen to penetrate through the total wall thickness. This tube was not found to leak when checked at room temperature, but did give an indication of a leak when vacuum leak checked at 1050°C (1922°F). The other unacceptable retort had a through crack in the longitudinal direction emanating from a chip at the extreme lower end of the tube. This crack is believed to have been caused by damage during the retort assembly process.

The cause of the circumferential cracks in the first tube could not be established a priori, but might be due to a combination of thermally and mechanically induced stresses in the tube wall during testing. If this were so, it would be a significant concern, since the distribution and size of flaws in large ceramic components is known to drastically lower the components' strength. While the other ceramic retorts did not show significant surface cracks, in several cases, surface pits or check marks were observed. These may be unavoidable and are possibly due to the fabrication process. It is presently uncertain whether or not these small surface flaws could lead to tube failure, and if so, what conditions would be conducive to failure. This problem is presently under investigation.

During the investigation, it was found that high N₂ levels in the retort outlet gas, formerly always connected with air in leakage, is actually due to a bake-out phenomenon. Initially, high N₂ levels (30 to 60 μatm) decay to background levels (2 to 6 μatm) in about 1/2 to 1 day at temperatures above 950°C (1472°F). Also, it should be

noted that other gas constituents, especially CO, are high during retort heat-up, but decay to control ranges more rapidly than does N₂. Furthermore, the high N₂ levels occur only when a retort which has seen use in testing is restarted on test. The phenomenon was not observed when these tubes were brought to test temperature for the first time.

Since two (2) of the eight (8) ceramic retorts are unfit for further use, a decision was made to replace them with two (2) metallic retorts. Inconel 601 will be used as the retort material and since significant experience with similar Inconel 601 retorts has been gained in 750° and 850°C (1382 and 1562°F) multispecimen creep testing, the use of metallic retorts for A/C testing should not present a problem. These retorts will be used only for 750°C (1382°F) testing and will be preoxidized or conditioned before they are used for testing.

2.3.3 Single Specimen Creep-Rupture (ST) Equipment

The experience gained with the ten (10) ST stands already in use for screening creep testing has raised some concerns. Testing at elevated temperatures, especially 950° and 1050°C (1742 and 1922°F) has taken a larger toll on test equipment than was anticipated. Several furnace and thermocouple failures have occurred during testing in these stands. Over 40,000 test hours have been accrued between nine (9) of these ten (10) stands. One major factor influencing the longevity of furnace components is the thermal cycling they see in service. The screening creep tests require that all tests be cycled to room temperature every thousand hours. This, along with the fact that currently no ramp-down temperature control capability exists, may result in excessive thermal shock in the furnace components. Solutions to this problem are under investigation.

An additional concern has arisen regarding the precision creep-rupture extensometry. Because the metallic rod and tube extensometers are exposed to the same atmosphere and temperature as the test specimen, there may exist a problem with the corrosion resistance and dimensional changes of the extensometer materials during use in testing. This is also being investigated.

2.3.4 Fatigue Test Equipment

Two (2) closed-loop control, electro-hydraulic test stands for use in testing are in place. The two (2) environmental test chambers which attach to the test stands, have been checked out for compliance to specifications and have been received from the vendor.

A major portion of the hardware for the fatigue test gas supply system (shown in the Quarterly Report for the period for October 1, 1979 to December 31, 1979, report number C00-2975-40) has been received. Construction and commissioning of this system will be completed in the next quarter.

2.3.5 Temperature Control System

An uninterruptible power supply for the computerized temperature control system was purchased with GE funds and installed during the report period. This power supply will buffer the computer system from minor voltage excursions and short term power interruptions to which the system had been very susceptible.

2.4 TASK 4 - SIMULATED REACTOR HELIUM ENVIRONMENT

2.4.1 Loop No. 1 Operation

The average gas composition of the controlled purity He supplied to the retorts during the report period is shown in Table 2.1 Three excursions outside normal chemistry ranges were encountered during the report period. Again, these excursions were only minor deviations from the normal gas control ranges for only short periods of time, usually less than (1) hour; also, they all were associated with retort tie-in to the loop.

2.4.2 Pumping Purification and Analysis Station (PP&A) No. 1

There were no significant revisions or additions to this system during the report period.

2.4.3 Pumping, Purification and Analysis Station (PP&A) No. 2

The gas addition micrometering valves were cleaned and installed during the quarter.

TABLE 2.1

LOOP NO. 1 GAS CHEMISTRY HISTORY
JANUARY 1, 1980 THROUGH MARCH 31, 1980

<u>Constituent</u>	<u>Target Chemistry and Normal Limits (μatm)</u>	<u>January</u>	<u>February</u>	<u>March</u>
H ₂	400 \pm 75	390.0	391.0	383.0
H ₂ O	2.0 \pm 1.0	2.0 (a)	2.0 (a)	2.0 (a)
CO	40 \pm 10	40.4	40.3	40.5
CO ₂	0.2 \pm 0.15	0.18	0.19	0.21
CH ₄	20 \pm 5	19.6	19.2	19.4
N ₂	6 \pm 4	9.0	10.9	8.6

(a) These numbers are determined from the EG&G dew point hygrometer and are believed to be the most accurate measurements available in the High Temperature Reactor Materials Test Laboratory.

Leak checking on the entire PP&A No. 2 continued and is presently approximately 90% complete.

2.4.4 Instrumentation

An oxygen analyzer equipped with an yttria stabilized zirconia (YSZ) element in place of the original calcia stabilized zirconia (CSZ) has been installed in the gas analysis station of PP&A No. 1. Performance to date indicates that this cell output is generally higher than the CSZ cell currently serving as the Loop Monitor. This difference may be due to a lower level of inherent leakage in the cell itself. It also appears the the YSZ cell is susceptible to the gradual loss of output previously observed in CSZ cells. Thus, at the present time, it is not possible to obtain direct measurements of the oxidation potential of the simulated VHTR helium. This means that the YSZ cell provides no significant improvement in the ability to accurately quantify the extremely low O_2 levels in the controlled purity He. Nevertheless, these units are useful for alarm purposes, since they quickly indicate changes in the O_2 levels.

The EG&G Dew Point Hygrometer was returned from the vendor after correction of electronic faults and was reinstalled in Loop No. 1 monitoring position. For many weeks, its response was somewhat erratic. Discussion with the vendor and other instrument users suggested that the observations were consistent with moisture inleakage to the sensor head. Repeated attempts to locate the leak or leaks with the helium mass spectrometer leak detector failed, but the problem remained. Finally, the entire sensor head, with all its connecting coolant, sample gas and electrical lines, was sealed in an inverted plastic bag which was then filled and continuously purged with welding grade He, which typically contains <1 ppm H_2O . This treatment apparently improved the situation and stable readings have been obtained, although the electronic noise superimposed on the basic signal remains. The best estimate of the loop moisture level at the end of the report period was $2.2 \mu\text{atm}$ when the Shaw probes indicated $1.4 \mu\text{atm}$ according to their June, 1979 calibration curves. The current approach to controlling loop H_2O level is to aim for 1.1 to $1.2 \mu\text{atm}$ as indicated by the Shaw probes. This level corresponds to $2 \mu\text{atm}$ on the EG&G hygrometer, the more reliable indication of H_2O , and is consistent with the desired H_2O level of $2 \mu\text{atm}$ in the test gas.

Two new portable gas analysis stations were constructed and placed in service during the report period. These units, in addition to the one already on hand, are designed to be connected to a retort outlet during start up to monitor the composition of the effluent gas. Each unit is equipped with a Carle gas chromatograph, two Shaw hygrometers and the necessary valves, flow meters, pressure gages, etc. These units will greatly aid test startup since a given unit may be required for as much as 2 to 3 days in each retort start up.

2.5 TASK 5 - MECHANICAL PROPERTIES EVALUATION

2.5.1 Test Specimen Machining

Machining of the test specimens required for the screening test program was completed by September, 1978.

Machining of all replacement test specimens for the screening aging/corrosion exposures was completed during the quarter. All specimens were fluorescent penetrant inspected and passed a dimensional check. Specimens requiring coating were coated and also were ready for test by the end of the quarter.

The number of specimens required for Phase 2 intensive screening testing have been machined for the nuclear process heat alloys. These specimens received dimensional and fluorescent penetrant inspection. During the dimensional inspection, it was discovered all of the Charpy specimens were slightly out of tolerance. These have been returned for rework and will be received back shortly.

The machining of the direct cycle helium turbine alloys for the Phase 2 intensive screening tests will have to await the procurement of the alloys.

2.5.2 Selection of Stresses for Screening Creep, and Creep-Rupture Testing

Stresses for multi-specimen creep (MSC) testing in the screening phase of the program have been selected and were reported for the quarterly report for the period April 1, 1979 to June 30, 1979, report number C00-2975-34.

Based on the stress verification testing performed prior to the start of the screening program, stress levels for intensive screening testing on the four (4) nuclear process heat alloys were selected. 1,000-, 5,000- and 15,000-hour

creep-rupture tests will be run in controlled purity He and 1,000- and 10,000-hour tests will be run in air. (300-hour creep-rupture tests, as well as some 1,000-hour tests, were run in air as part of the stress verification testing.) The stresses for these tests are listed in Table 2.2.

The stresses for similar tests on the direct cycle helium turbine alloys will be selected when those alloys are received. Since new heats will be used, a limited number of short term air creep-rupture tests will be run to determine where the heats fall on the creep and rupture curves already on hand.

2.5.3 Screening Creep Testing

Multi-specimen creep results for 2,000 and 3,000 hours at 750°C (1382°F). 3,000 hours at 850°C (1562°F) and 1,000 hours at 950°C (1742°F) were obtained during the report period. The results of room temperature strain measurements for those test conditions are given in Table 2.3 for the nuclear process heat alloys and Table 2.4 for the direct cycle helium turbine alloys. These tables include screening creep data which were reported previously.

No excessive creep strains were noted in this set of data. One specimen at 850°C (1562°F) was removed from test after 2000 hours. This IN-519 specimen (1800 SC01), scheduled for 6,000 hours testing, was removed when a small notch in the gage length near the radius was found during post-exposure visual examination at the specimen. This specimen has been replaced on test.

2.5.4 Phase 2 - Intensive Screening Creep-Rupture Testing

Nine (9) of the twelve (12) 10,000-hour air tests for the nuclear process heat alloys were started during the quarter. The balance of the nuclear process heat air creep-rupture tests, as well as several of the controlled purity He tests, will be initiated during the next quarter.

2.6 - TASKS 6 & 7 - THERMAL STABILITY EVALUATION AND PRIMARY COOLANT CORROSION RESISTANCE

2.6.1 Dimensional Change Measurements

As a result of the ORNL experimental evidence of dimensional changes due to controlled purity He exposure, dimensional measurement will now be made on some specimens exposed in aging/corrosion tests. The length measuring device designed to measure creep strain for multi-specimen creep specimens has been

TABLE 2.2

STRESSES FOR INTENSIVE SCREENING PHASE

CREEP-RUPTURE TESTS NPH ALLOYS

Temp/Time ^(a)	Alloy 80CH		Inconel 617		Hastelloy X		MA-956	
750C/1,000 hr	76 MPa	11.0 ksi	—	—	—	—	—	—
750C/5,000 hr	62	9.0	—	—	—	—	—	—
750C/10,000 hr	58	8.4	—	—	—	—	—	—
750C/15,000 hr	55	8.0	—	—	—	—	—	—
850C/1,000 hr	39	5.6	79 MPa	11.5 ksi	52 MPa	7.5 ksi	79 MPa	11.5 ksi
850C/5,000 hr	30	4.4	62	9.0	40	5.8	76	11.1
850C/10,000 hr	27	3.9	56	8.2	36	5.2	76	11.0
850C/15,000 hr	25	3.6	52	7.6	32	4.7	74	10.7
950C/1,000 hr	18	2.6	38	5.5	23	3.3	69	10.0
950C/5,000 hr	13	1.9	29	4.2	17	2.4	68	9.8
950C/10,000 hr	11	1.65	25	3.7	14	2.0	65	9.5
950C/15,000 hr	11	1.55	24	3.5	13	1.9	65	9.4
1050C/1,000 hr	—	—	14	2.1	9.0	1.3	62	9.0
1050C/5,000 hr	—	—	13	1.95	5.4	0.78	58	8.4
1050C/10,000 hr	—	—	11	1.55	4.1	0.6	56	8.2
1050C/15,000 hr	—	—	9.6	1.4	3.4	0.5	56	8.1

(a) Note: Air Tests for 1,000 and 10,000 hours
He Tests for 1,000, 5,000, and 15,000 hours

TABLE 2.3
SCREENING (MULTISPECIMEN) CREEP RESULTS FOR THE
NUCLEAR PROCESS HEAT ALLOYS

Alloy	Temp °C	Exposure Time-Hrs.	Specimen Number	Stress		%ε Strain			
				MPA	KSI	1000 Hrs	2000 Hrs	3000 Hrs	
Alloy 800H	750	10,000	1000SC3	21	3	.00210	.0127	-.00847	
		3,000	1000SC1	48	7	.386	.910	1.512 comp (a)	
		6,000	1000SC2	48	7	.440	1.100	1.700	
	850	3,000	1000SC6	7	1	-.00952	-.00318	-.0116 comp.	
		10,000	1000SC7	7	1	-.00275	.00423	.0159	
		6,000	1000SC5	21	3	.396	.811	1.255	
	950	10,000	1000SC11	0.7	0.1	-.038			
		3,000	1000SC10	6	0.9	-.030			
	Alloy 800H coated	750	3,000	1001SC1	21	3	.0160	.053	.0739 comp.
			6,000	1001SC2	21	3	.0180	.0666	.0782
10,000			1001SC3	21	3	.0180	.0380	.0613	
850		3,000	1001SC6	7	1	.00950	.00422	.0264 comp.	
		6,000	1001SC7	7	1	.0105	.0190	.0370	
		10,000	1001SC8	7	1	.00950	.00950	.0264	
950		6,000	1001SC12	0.7	0.1	-.018			
		10,000	1001SC13	0.7	0.1	+.0042			
		3,000	1001SC11	6	0.9	.053			
Incoloy 802		750	10,000	1100SC3	48	7	-.0495	-.0296	-.0138
	3,000		1100SC1	76	11	-.0260	.0106	.0233 comp.	
	850	10,000	1100SC7	21	3	-.0220	.0244	.0434	
		3,000	1100SC5	34	5	-.0420	-.0349	-.0154 comp.	
	950	10,000	1100SC10	6	.9	-.036			
		6,000	1100SC11	9	1.25	-.015			
		3,000	1100SC9	14	2	-.012			

(a) comp. - complete; these specimens have reached their planned exposure time and have been removed from test.

TABLE 2.3 (Continued)
 SCREENING (MULTISPECIMEN) CREEP RESULTS FOR THE
 NUCLEAR PROCESS HEAT ALLOYS

Alloy	Temp °C	Exposure Time-Hrs.	Specimen Number	Stress		%ε Strain		
				MPA	KSI	1000 Hrs	2000 Hrs	3000 Hrs
Incoloy 807	750	10,000	1200SC3	21	3	.0100	.00528	.00740
		3,000	1200SC1	62	9	.437	1.252	1.845 comp.
		6,000	1200SC2	62	9	.0740	.174	.250
	850	10,000	1200SC8	7	1	-.00634	-.00740	.00630
		3,000	1200SC6	21	3	.0380	.0623	.0876 comp.
		6,000	1200SC7	28	4	.0580	.0919	.114
	950	10,000	1200SC13	0.7	0.1	-.0074		
		6,000	1200SC12	6	0.9	.030		
		3,000	1200SC11	14	2	.59		
Incoloy DS	750	3,000	1300SC1	21	3	-.0630	.000	.000 comp.
		10,000	1300SC2	21	3	.00739	.0159	.0222
	850	6,000	1300SC4	7	1	.00212	-.0116	.00630
		3,000	1300SC5	7	1	-.00635	-.00424	.00740 comp
		10,000	1300SC6	7	1	.0106	-.00530	.00950
	950	6,000	1300SC10	0.7	0.1	.011		
		10,000	1300SC11	0.7	0.1	.015		
		3,000	1300SC9	6	0.9	.031		
	HD-556	750	10,000	1400SC3	21	3	-.024	-.00635
6,000			1400SC2	62	9	.1215	.159	.192
3,000			1400SC1	76	11	.200	.268	.293 comp.
850		10,000	1400SC7	21	3	-.0130	-.0116	-.00420
		6,000	1400SC8	21	3	-.00850	-.0116	-.00420
		3,000	1400SC31	34	5	.035	.0749	.113 comp.
950		10,000	1400SC11	6	0.9	-.051		
		3,000	1400SC12	17.5	2.5	.070		
		6,000	1400SC13	17.5	2.5	-.0117		

TABLE 2.3 (Continued)
 SCREENING (MULTISPECIMEN) CREEP RESULTS FOR THE
 NUCLEAR PROCESS HEAT ALLOYS

Alloy	Temp °C	Exposure Time-Hrs.	Specimen Number	Stress		%ε Strain		
				MPA	KSI	1000 Hrs	2000 Hrs	3000 Hrs
Manurite 36X	750	10,000	1500SC3	21	3	.0265	.0454	.0676
		6,000	1500SC1	48	7	.297	.484	.585
		3,000	1500SC2	48	7	.300	.495	.605 comp.
	850	10,000	1500SC7	21	3	.0180	.0338	.772
		6,000	1500SC8	28	4	.0620	.0920	.173
		3,000	1500SC6	34	5	.174	.305	.520 comp.
	950	10,000	1500SC12	6	0.9	-.0053	.0529	
		3,000	1500SC11	14	2			
	HK-40	750	10,000	1700SC3	48	7	.0370	.0510
10,000			1700SC4	48	7	.0455	.0850	.930
6,000			1700SC1	62	9	.095	.169	.245
3,000			1700SC2	62	9	.0645	.144	.212 comp.
850		10,000	1700SC8	21	3	-.0260	-.0095	.106
		6,000	1700SC9	28	4	.0210	.0243	.0876
		3,000	1700SC7	34	5	.0360	.0993	.197 comp.
950		10,000	1700SC13	6	0.9	-.024		
		3,000	1700SC12	14	2	.014		
IN-519	750	10,000	1800SC8	48	7	.138	.220	.261
		3,000	1800SC2	62	9	.1825	.428	.586 comp.
		6,000	1800SC1	76	11	.680	1.490*	---
		(Replaced SC1 after 2000 hours)	1800SC30	76	11	.520		
	850	6,000	1800SC7	28	4	-.0180	.0160	.425
		10,000	1800SC8	28	4	-.00320	.00212	.499
		3,000	1800SC6	34	5	.0340	.0730	.147 comp.

* replaced do to ding found in gage section.

TABLE 2.3 (Continued)
 SCREENING (MULTISPECIMEN) CREEP RESULTS FOR THE
 NUCLEAR PROCESS HEAT ALLOYS

Alloy	Temp °C	Exposure Time-Hrs.	Specimen Number	Stress		%ε Strain		
				MPA	KSI	1000 Hrs	2000 Hrs	3000 Hrs
IN-519	950	10,000	1800SC12	6	0.9	-.0446		
		3,000	1800SC11	14	2	.0095		
		6,000	1800SC13	17.5	2.5	.0053		
Hastelloy X	750	10,000	1900SC2	21	3	-.0318	.0498	.0413
		6,000	1900SC3	48	7	.228	.280	.288
		3,000	1900SC1	62	9	.378	.471	.544 comp.
	850	10,000	1900SC8	7	1	-.00104	-.0074	.00210
		6,000	1900SC6	21	3	.126	.140	.192
		3,000	1900SC7	28	4	.238	.340	.498 comp.
	950	10,000	1900SC12	0.7	0.1	-.0079		
		6,000	1900SC11	6	0.9	.055		
		3,000	1900SC10	14	2	1.27		
Inconel 617	750	10,000	1A00SC3	62	9	-.143	-.0148	.0159
		3,000	1A00SC1	76	11	-.0260	.0150	.0423 comp.
		6,000	1A00SC2	76	11	-.00750	.0320	.0761
	850	10,000	1A00SC8	28	4	-.0286	-.00636	.00420
		3,000	1A00SC6	34	5	.000	.0201	.0401 comp.
		6,000	1A00SC7	34	5	-.0110	.00105	.0200
	950	10,000	1A00SC11	14	2	.034		
		6,000	1A00SC12	17.5	2.5	.070		
		3,000	1A00SC13	17.5	2.5	.041		
Inconel 617 coated	750	10,000	1A01SC3	21	3	-.0220	-.0117	.00529
		6,000	1A01SC2	48	7	.0180	-.0110	-.00319
		3,000	1A01SC1	76	11	-.00530	.0230	.0602 comp.
	850	10,000	1A01SC8	7	1	-.0243	-.00952	.00210
		6,000	1A01SC7	21	3	.031	.0592	.121
		3,000	1A01SC6	34	5	1.333	2.523	3.530 comp.

Table 2.3 (Continued)

SCREENING (MULTISPECIMEN) CREEP RESULTS FOR THE
NUCLEAR PROCESS HEAT ALLOYS

Alloy	Temp °C	Exposure Time-Hrs.	Specimen Number	Stress		%ε Strain		
				MPA	KSI	1000 Hrs.	2000 Hrs.	3000 Hrs.
Inconel 617 coated	950	10,000	1A01SC13	0.7	0.1	.016		
		6,000	1A02SC12	9	1.25	.050		
		3,000	1A01SC11	14	2	.16		
Mo-Re 2	750	10,000	1C00SC3	48	7	.304	.360	.375
		3,000	1C00SC7	76	11	.680	.790	.836 comp.
		6,000	1C00SC1	76	11	.703	.811	.870
	850	10,000	1C00SC11	28	4	.166	.191	.224
		6,000	1C00SC39	34	5	.166	.222	.290
		3,000	1C00SC8	34	5	.198	.236	.299 comp.
	950	10,000	1C00SC14	14	2	.146		
		3,000	1C00SC15	17.5	2.5	.176		
		6,000	1C00SC16	17.5	2.5	.143		
MA-956	850	6,000	1D00SC1	42	6	.0100	.0220	.0235
		10,000	1D00SC2	42	6	.029	.0412	.0412
		3,000	1D00SC3	55	8	.00880	.0293	.0293 comp.
	950	6,000	1D00SC7	34	5	.041		
		10,000	1D00SC8	34	5	.018		

TABLE 2.4
SCREENING (MULTISPECIMEN) CREEP RESULTS FOR THE
DIRECT CYCLE HELIUM TURBINE ALLOYS

Alloy	Temp °C	Exposure Time-Hrs.	Specimen Number	Stress		%ε Strain		
				MPA	KSI	1000 Hrs	2000 Hrs	3000 Hrs
TZM	750	10,000	CO1SC16	345	50	.0439	.0548	.0495
		6,000	CO1SC17	345	50	.0580	.0713	.0799
		3,000	CO1SC19	345	50	.0410	.0524	.0609 comp. ^(a)
	850	6,000	CO1SC1	152	22	.0225	.0183	.0183
		3,000	CO1SC2	152	22	.00857	.00714	.00129 comp.
		10,000	CO1SC3	152	22	.00288	.00144	.0072
MA-754	850	6,000	BOOSC1	110	16	.0660	.0663	.0945
		10,000	BOOSC2	110	16	.0660	.0692	.109
		3,000	BOOSC3	110	16	.0636	.0537	.0876 comp.
	950	6,000	BOOSC6	48	7	.018		
		10,000	BOOSC7	48	7	.0226		
		3,000	BOOSC8	48	7	.0085		
MM-0011	750	10,000	100SC1	262	38	.0130	.0356	.0541
		10,000	100SC31	262	38	.0100	.0428	.0257
		6,000	100SC7	345	50	.0584	.0869	.1367
		3,000	100SC19	345	50	.0385	.0898	.1012 comp.
		6,000	100SC32	345	50	.0370	.0754	.0869
		850	10,000	100SC5	110	16	.029	.063
	850	6,000	100SC2	152	22	.0650	.0896	.141
		3,000	100SC3	152	22	.0560	.0915	.150 comp.
		3,000	100SC4	152	22	.0830	.106	.154 comp.
		950	10,000	100SC12	34	5	.030	
	3,000	100SC11	48	7	.044			

(a) comp. - complete; these specimens have reached their planned exposure time and have been removed from test.

TABLE 2.4 (Continued)

SCREENING (MULTISPECIMEN) CREEP RESULTS FOR THE
DIRECT CYCLE HELIUM TURBINE ALLOYS

Alloy	Temp °C	Exposure Time-Hrs.	Specimen Number	Stress		%ε Strain		
				MPA	KSI	1000 Hrs	2000 Hrs	3000 Hrs
MM-0011DS	750	10,000	120SC1	345	50	.0286	.0414	.0657
		6,000	120SC7	345	50	.0472	.0429	.0830
		3,000	120SC19	345	50	.0370	.0654	.0853 comp.
	850	6,000	120SC3	152	22	.0390	.0859	.129
		3,000	120SC4	152	22	.0370	.0840	.124
		10,000	120SC23	152	22	.0340	.0840	.116
	950	6,000	120SC9	48	7	.054		
		3,000	120SC10	48	7	.066		
		10,000	120SC11	48	7	.060		
Rene' 80	750	10,000	200SC1	262	38	.0350	.0709	.123
		10,000	200SC26	262	38	.044	.0937	.140
		6,000	200SC6	345	50	.275	.408	.479
		3,000	200SC16	345	50	.269	.402	.477 comp.
	850	10,000	200SC4	110	16	.0397	.0694	.108
		6,000	200SC2	152	22	.0426	.172	.240
		3,000	200SC3	152	22	.0990	.168	.263 comp.
	950	10,000	200SC11	17	2.5	.024		
		3,000	200SC10	34	5	.021		
6,000		200SC9	48	7	.038			
Rene'100	750	10,000	300SC32	124	18	-.0140	.00285	.00568
		10,000	300SC1	262	38	-.00850	-.00143	.00426
		10,000	300SC31	262	38	-.00570	.00993	.00283
		6,000	300SC7	345	50	.0640	.114	.161
		3,000	300SC19	345	50	.0497	.108	.128 comp.

TABLE 2.4 (Continued)
 SCREENING (MULTISPECIMEN) CREEP RESULTS FOR THE
 DIRECT CYCLE HELIUM TURBINE ALLOYS

Alloy	Temp °C	Exposure Time-Hrs.	Specimen Number	Stress		%ε Strain			
				MPA	KSI	1000 Hrs	2000 Hrs	3000 Hrs	
Rene' 100	850	10,000	300SC5	110	16	-.00850	-.00570	.0114	
		6,000	300SC2	152	22	.00330	.0600	.0880	
		3,000	300SC3	152	22	.0260	.0583	.0853 comp.	
		3,000	300SC4	152	22	.0130	.0600	.194 comp.	
	950	10,000	300SC13	17	2.5	-.055			
		3,000	300SC12	34	5	-.016			
		6,000	300SC11	48	7	.0398			
	Alloy 713LC	750	10,000	400SC26	124	18	-.00995	.00284	.00852
			10,000	400SC27	124	18	-.0140	-.00995	.00853
10,000			400SC1	262	38	.0485	.102	.134	
3,000			400SC16	262	38	.0327	.0668	.102 comp.	
6,000			400SC6	345	50	.24	.39	.570	
850			10,000	400SC4	55	8	.0028	.00852	.0185
850		3,000	400SC2	110	16	.0380	.1067	.135 comp.	
		6,000	400SC3	110	16	.0410	.0994	.133	
		950	10,000	400SC11	1.4	0.2	-.034		
950		3,000	400SC9	17	2.5	.041			
		6,000	400SC10	17	2.5	.013			
		IN-738	750	10,000	500SC32	124	18	-.0185	-.0114
10,000	500SC1			152	22	-.0130	-.00700	.0127	
6,000	500SC7			262	38	.0283	.0991	.130	
3,000	500SC19			262	38	.0300	.109	.173 comp.	

TABLE 2.4 (Continued)

SCREENING (MULTISPECIMEN) CREEP RESULTS FOR THE
DIRECT CYCLE HELIUM TURBINE ALLOYS

Alloy	Temp °C	Exposure Time-Hrs.	Specimen Number	Stress		%ε Strain			
				MPA	KSI	1000 Hrs	2000 Hrs	3000 Hrs	
IN-738	850	10,000	500SC4	55	8	-.0028	.000	-.00280	
		3,000	500SC2	110	16	.0430	.0625	.102 comp.	
		6,000	500SC3	110	16	.0430	.0784	.135	
	950	10,000	500SC11	1.4	0.2	-.0071			
		6,000	500SC9	17	2.5	.0071			
		3,000	500SC10	17	2.5	-.030			
U-720	750	10,000	A00SC1	152	22	.0270	.00570	.0327	
		6,000	A00SC6	262	38	.0410	.0641	.114	
		3,000	A00SC16	262	38	.0540	.0811	.111 comp.	
	850	10,000	A00SC4	55	8	.0170	-.00285	.0100	
		3,000	A00SC2	110	16	.031	.0555	.0812 comp.	
		6,000	A00SC3	110	16	.028	.0313	.0697	
	950	10,000	A00SC10	0.7	0.1	-.024			
		3,000	A00SC9	17	2.5	.0014			
	NX-188	750	10,000	600SC1	152	22	.0930	.1030	.130
			10,000	600SC26	152	22	.102	.119	.126
			6,000	600SC6	262	38	.224	.307	.341
			3,000	600SC16	262	38	.242	.317	.383 comp.
850		10,000	600SC2	69	10	.140	.170	.209	
		6,000	600SC3	110	16	.184	.236	.236	
		3,000	600SC4	110	16	.200	.254	.315 comp.	
950		3,000	600SC12	17	2.5	.099			
		10,000	600SC13	17	2.5	.098			
		6,000	600SC9	28	4	.117			

modified so that it can also measure distances between fiducial points on tensile test specimens. Pre- and post-exposure length measurements will be made on all tensile test bars exposed in the program. Tensile tests are included for all of the alloys tested in screening creep program. However, tensile specimens are not included for some of the developmental alloys which are in the screening A/C tests for exposure only. These are: Manaurite 900, Inconel 618E, M-21, YD-Ni and YD-NiCrAl. For all other alloys in the program, dimension changes with exposure time and temperature will be determined.

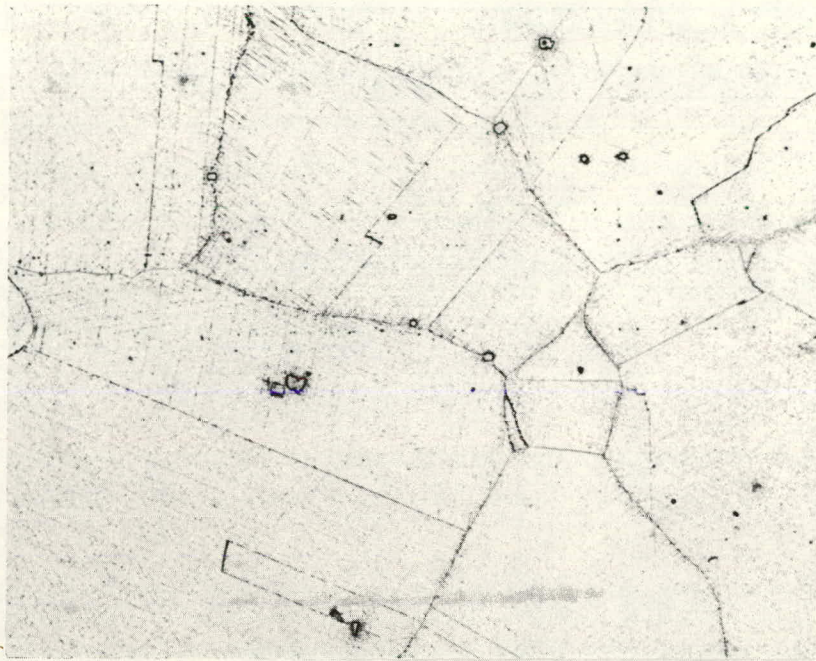
2.6.2 Metallography of Exposed Specimens

Metallography on specimens exposed at 750°C (1382°F) for 1,000 hours and at 850°C (1562°F) for 1104 hours has been completed. Corrosion pins, for which the weight change measurements were reported in the last quarterly report, report number C00-2975-40, were sectioned perpendicular to their longitudinal axis. The right circular cross-section of each pin was examined in both the as-polished and the etched condition. Edge retention was maintained by electro-depositing nickel, after a small vapor deposit of silver, on the specimen surface prior to sectioning.

2.6.2.1 Nuclear Process Heat Alloys

Alloy 800H

Representative optical photomicrographs of the internal microstructure of Alloy 800H after approximately 1000 hr exposure to controlled purity He at 750°C and 850°C (1382 and 1562°F) are shown in Figure 2.1. The microstructure after 750°C (1382°F) aging consisted of a mixture of coarse primary carbides like those observed in the as-received alloy and intermediate to fine precipitated carbides appearing both intra- and intergranularly and on twin boundaries. The large, blocky precipitates are assumed to be TiN and/or Ti (C,N), while the finer precipitated phase is presumably $M_{23}C_6$, probably chromium carbide. Note that the intragranular carbides frequently appeared to be preferentially precipitated around primary carbide particles or along certain crystallographic directions. The 850°C (1562°F) exposed structure was similar in appearance to the lower temperature age material; however, the overall degree of carbide precipitation appeared somewhat reduced. Intragranular carbide precipitation around primary carbide particles appeared reduced rather significantly for the 850°C (1562°F) aged alloy.



(a)

Oxalic Acid

250X



(b)

Oxalic Acid

250X

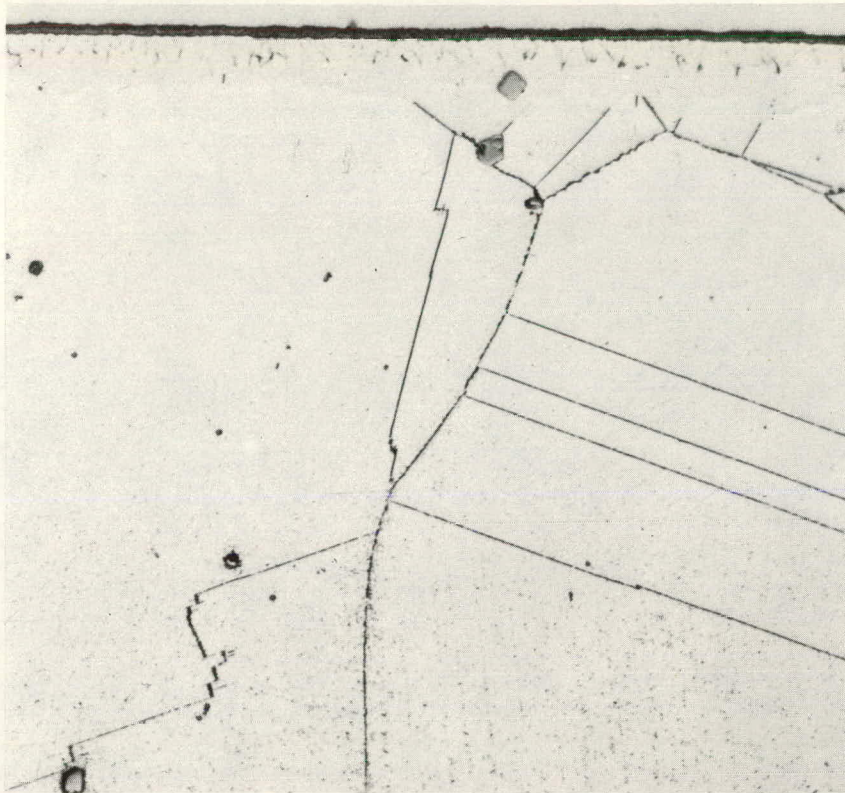
Figure 2.1 Optical Photomicrographs Showing Etched Internal Microstructure of Alloy 800H after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.

After 750°C (1382°F) aging, the alloy revealed a continuous single-layered surface scale (shown in Figure 2.2), approximately 2 μm (0.08 mils) thick. Fine, light-etching particles (probably carbides) were intermittently observed within the scale. An internally oxidized zone was observed to a depth of about 6 μm (0.24 mils) below the scale. Grain boundaries were depleted of precipitates to a depth of about 16 μm (0.63 mils); a lack of intragranular precipitation was evident to a depth of about 96 to 104 μm (3.8 to 4.1 mils). Although not shown, some preferentially oriented carbide precipitation also was evident to a depth of about 21 μm (0.83 mils). After 850°C (1562°F) exposure, the surface scale was much more complex and was composed of primarily two layers with an overall thickness of 7 μm (0.28 mils). Beneath the scale, an internally oxidized zone extended to a depth of 10 to 18 μm (0.39 to 0.71 mils). An alloy depleted zone was also in evidence below the scale to a depth of 18 to 26 μm (0.71 to 1.00 mils). Grain boundaries near the periphery of the sample seemed to contain larger carbides than grain boundaries in the interior of the sample.

The microstructure of the 750° and 850°C (1382 and 1562°F) aged coated specimens of Alloy 800H was quite similar to the uncoated alloy. The degree of intragranular carbide precipitation around the large primary carbides was reduced for the coated 750°C (1382°F) exposed sample, however, as was the degree of carbide precipitation along crystallographic directions. Gray, fairly continuous surface scales with a thickness of about 1 μm (0.04 mils) for the 750°C (1382°F) aged alloy and 2 μm (0.08 mils) for the 850°C (1562°F) aged material were observed. Interestingly, the apparent platinum-chromium-aluminide coating thickness appeared somewhat increased relative to the as-coated thickness. In the as-received condition, the coating was 89 to 102 μm (3.5 to 4.0 mils) thick. However, after 750°C (1382°F) and 850°C (1562°F) aging, the apparent thicknesses were 96 to 104 (3.8 to 4.0 mils) and 122 to 131 μm (4.8 to 5.2 mils), respectively.

Incoloy 802

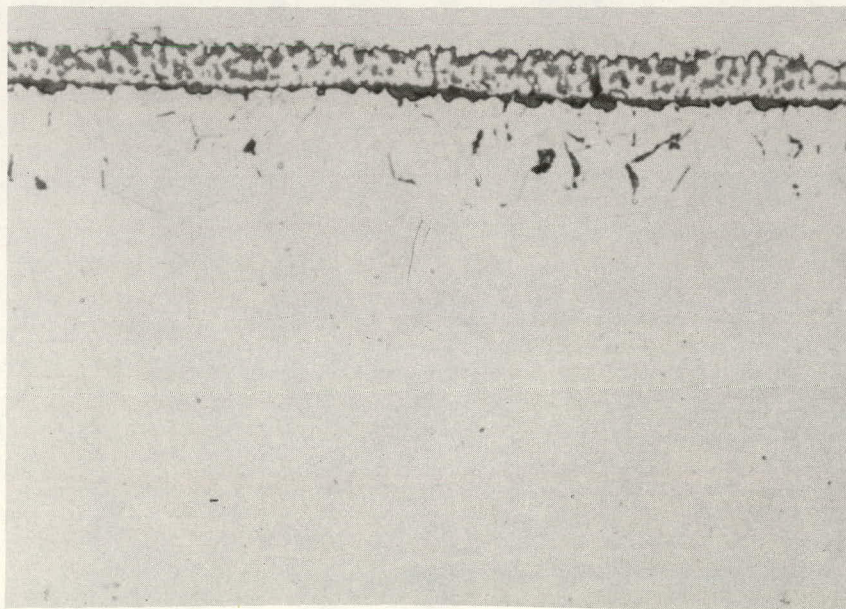
The photomicrographs shown in Figure 2.3 are representative of the microstructures of Incoloy 802 after approximately 1000-hr aging in controlled purity He at 750° and 850°C (1382 and 1562°F). The microstructures were similar and consisted of coarse primary carbides like those observed in the as-received alloy and copious amounts of intermediate to fine precipitated carbides appearing both



(a)

Oxalic Acid

500X



(b)

Unetched

1000X

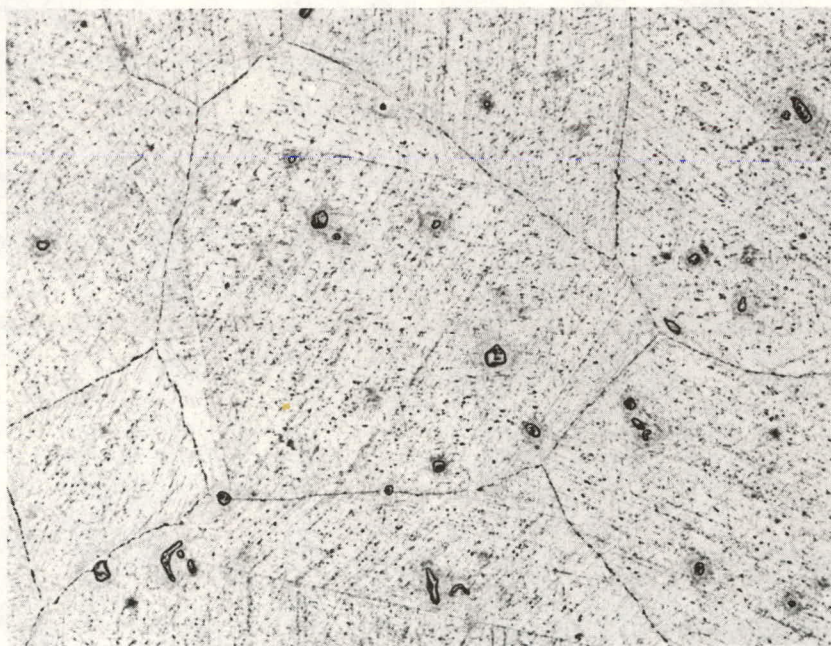
Figure 2.2 Optical Photomicrographs Showing Surface Condition and Subsurface Microstructure of Alloy 800H after Approximately 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C.



(a)

Oxalic Acid

250X



(b)

Oxalic Acid

250X

Figure 2.3 Optical Photomicrographs Showing Etched Internal Microstructure of Incoloy 802 after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.

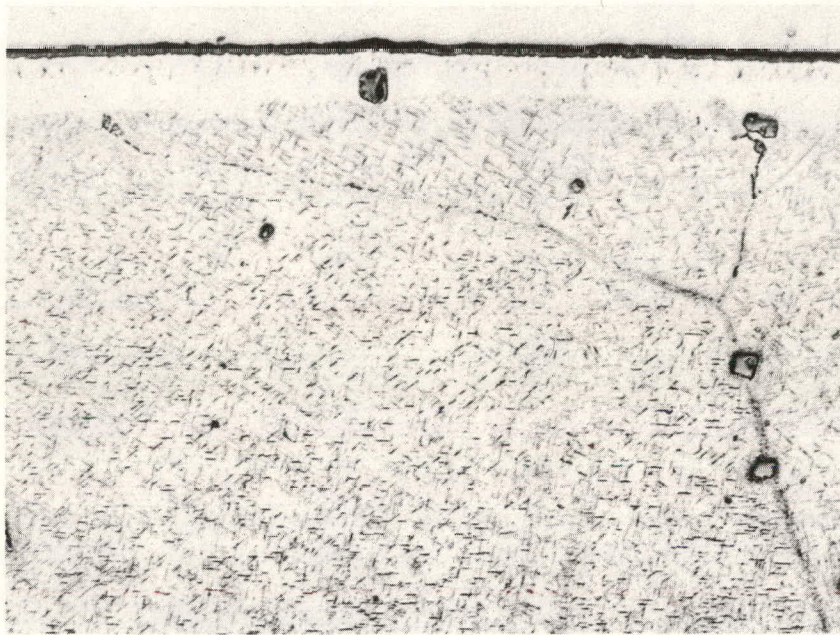
intra- and inter-granularly and on twin boundaries. The large blocky precipitates are presumably TiN, TiC or Ti (C,N). The finer precipitates, which appear as fine spherical or elongated particles, are assumed to be $M_{23}C_6$, probably

A higher concentration of precipitated carbides was evident around the larger primary carbides for a distance up to about 5 μm . (0.20 mils). Additionally, the intragranular carbides frequently appeared to be preferentially precipitated along various crystallographic directions. This alloy exhibited a greater degree of carbide precipitation relative to Alloy 800H for equivalent aging conditions, probably because of its higher carbon content.

The surface of Incoloy 802 after exposure at 750°C (1382°F) was covered with a single-layered dark gray-etching, continuous scale approximately 2 μm (0.08 mils) thick as illustrated in Figure 2.4. The scale contained fine, light-etching particles, probably carbides. Below the scale and extending 6 μm (0.24 mils) into a 14 μm (0.55 mils) deep alloy depleted region were numerous dark gray particles or fingers which were presumably Al-rich oxides like those observed in Alloy 800H. The surface scale after 850°C (1562°F) aging, Figure 2.4, was much thicker 6 to 13 μm (0.24 to 0.50 mils), continuous and quite irregular in nature relative to the scale observed after 750°C (1382°F) exposure. The scale was primarily gray with a high density of unconnected white particles scattered throughout (presumably an oxide/carbide mixture). Below the scale, a 10 to 15 μm (0.39 to 0.59 mils) deep internally oxidized zone extended into a 30 to 40 μm (1.2 to 1.6 mils) deep alloy depleted region.

Incoloy 807

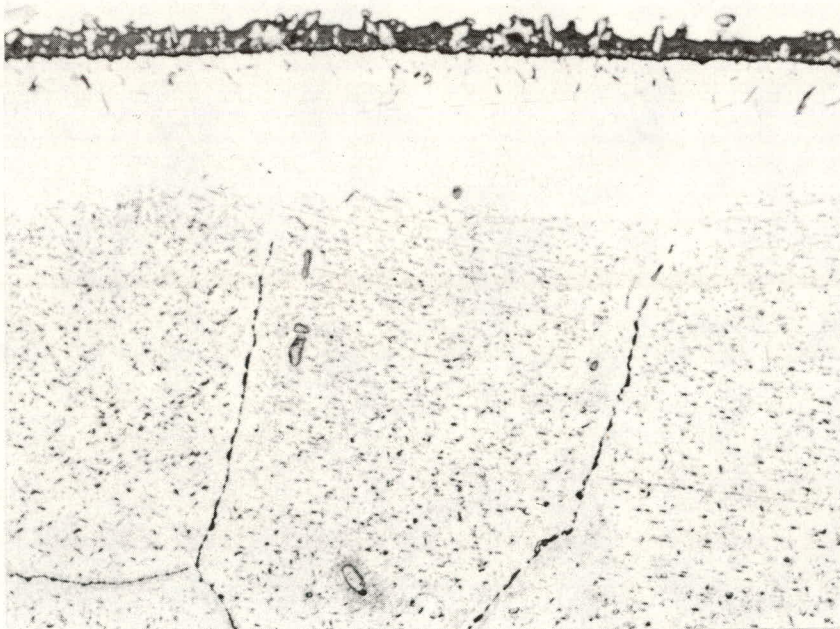
Optical photomicrographs representative of the internal microstructure after approximately 1000-hr exposure to controlled purity He at 750°C and 850°C (1382 and 1562°F) are shown in Figure 2.5. After 750°C (1382°F) aging, the microstructure consisted of a mixture of coarse primary carbides similar to those observed in the as-received alloy and intermediate to fine precipitated carbides appearing both intra- and intergranularly and on twin boundaries. The microstructure after 850°C (1562°F) aging was quite similar in appearance to the 750°C (1382°F) aged materials; however, some coalescence of the grain boundary carbides appeared evident.



(a)

Oxalic Acid

500X

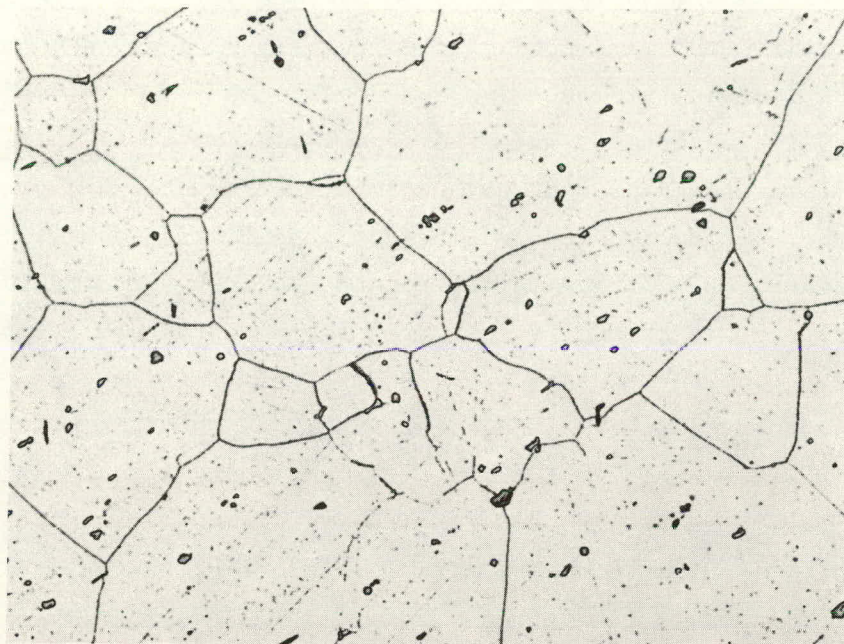


(b)

Oxalic Acid

500X

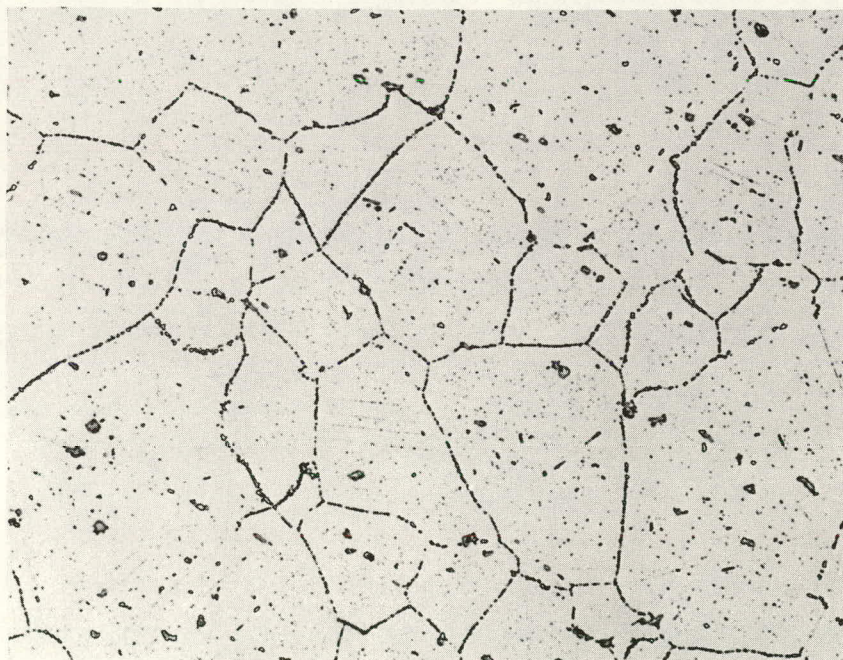
Figure 2.4 Optical Photomicrographs Showing Surface Condition and Etched Subsurface Microstructure of Incoloy 802 after Approximately 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C.



(a)

Oxalic Acid

250X



(b)

Oxalic Acid

250X

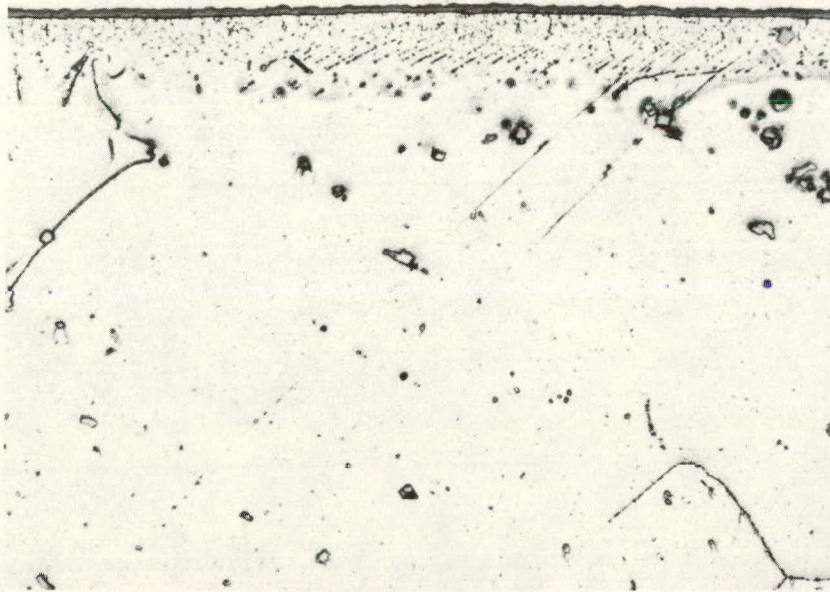
Figure 2.5 Optical Photomicrographs Showing Etched Internal Microstructure of Incoloy 807 after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.

Subsequent to 750°C (1382°F) aging, the surface scale, Figure 2.6, was on the order of 2 μm (0.08 mils) thick, continuous, and gray in color. A region of internal oxidation was evident which extended to about 4 μm (0.16 mils), below the scale. The internally oxidized region was contained in a 10 to 18 μm (0.39 to 0.71 mils) alloy depleted zone. Additionally, areas of preferentially oriented carbide precipitation were visible to a depth of 28 μm (1.1 mils). After 850°C (1562°F) aging, the surface scale, Figure 2.6, was much thicker 8 to 12 μm (0.31 to 0.47 mils), more irregular and more complex in nature relative to the scale after 750°C (1382°F) exposure. The scale was primarily gray and contained many white, unconnected particles and a few dark gray particles (presumably a complex oxide/carbide mixture). A thin, dark gray layer was evident just below this multiphase layer. Particles and fingers indicating internal oxidation were evident to a depth of 10 to 20 μm (0.39 to 0.79 mils) below the scale within an alloy depleted region of 24 to 28 μm (0.94 to 1.10 mils).

Incoloy DS

Typical optical photomicrographs showing the internal microstructure of Incoloy DS after approximately 1000-hr exposure to controlled purity He at 750°C and 850°C (1382 and 1562°F) are shown in Figure 2.7. As evident, the microstructures were quite similar after both aging treatments and consisted of a mixture of coarse primary carbides akin to those observed in the as-received structure and finer precipitated carbides appearing both intra- and intergranularly and on twin boundaries. Generally, the grain boundary carbides were non-continuous and somewhat larger in size than the intragranular precipitate. A few silicide or silicate inclusions also were in evidence.

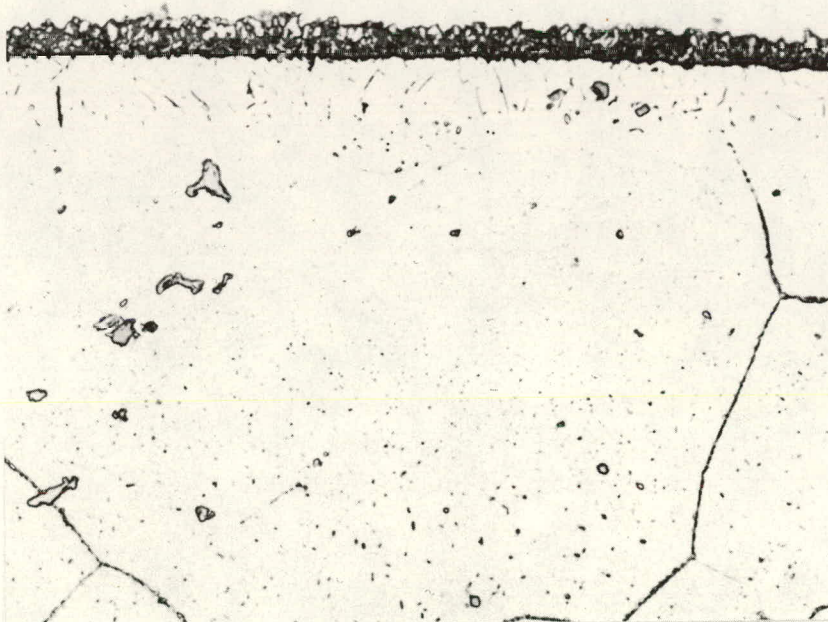
After 750°C (1382°F) aging, the surface of the alloy revealed a discontinuous or partially spalled, 1 μm (0.04 mils) thick, gray scale, Figure 2.8. No evidence of internal oxidation was observed. An alloy depleted region was revealed below the scale, which extended to a depth of 10 μm (0.39 mils). The scale after 850°C (1562°F) exposure, 3 to 6 μm (0.12 to 0.24 mils) thick, was also discontinuous or partially spalled. As with the 750°C (1382°F) exposure, no evidence of internal oxidation was observed; however, an alloy depleted zone beneath the scale extended to a depth of 22 to 32 μm (0.87 to 1.26 mils).



(a)

Oxalic Acid

500X



(b)

Oxalic Acid

500X

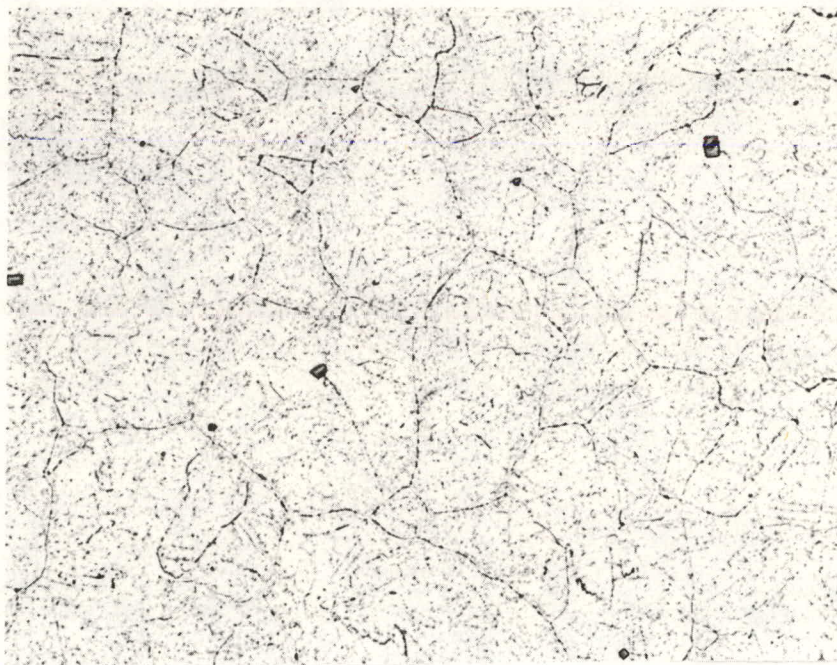
Figure 2.6 Optical Photomicrographs Showing Surface Condition and Etched Subsurface Microstructure of Incoloy 807 after Approximately 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C.



(a)

Oxalic Acid

250X

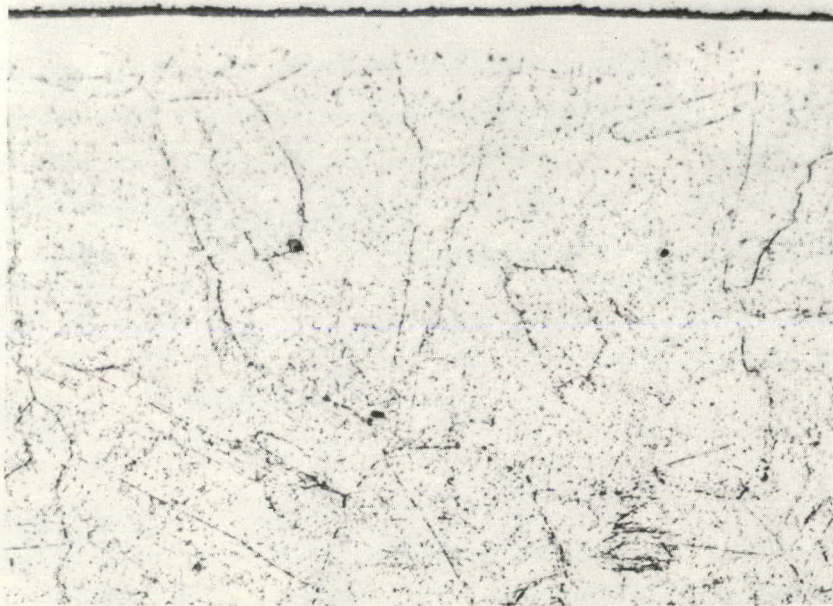


(b)

Oxalic Acid

250X

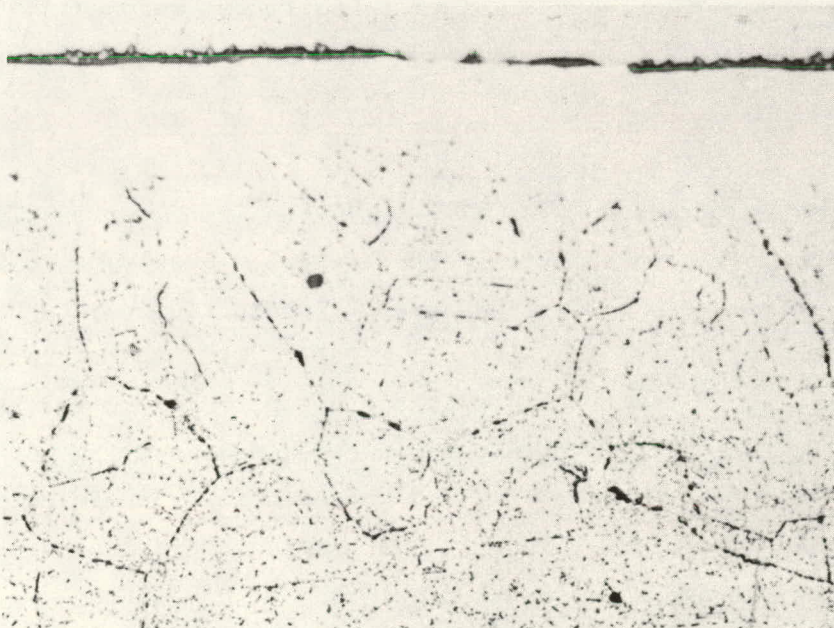
Figure 2.7 Optical Photomicrographs Showing Etched Internal Microstructure of Incoloy DS after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

Oxalic Acid

500X



(b)

Oxalic Acid

500X

Figure 2.8 Optical Photomicrographs Showing Surface Condition and Etched Subsurface Microstructure of Incoloy DS after Approximately 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C.

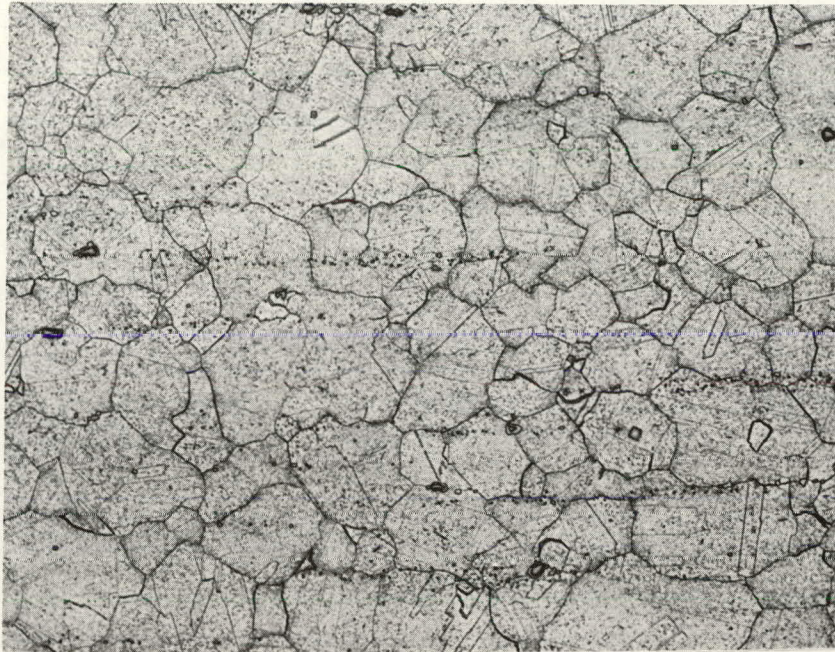
HD-556

Typical optical photomicrographs showing the internal microstructure of HD-556 after approximately 1000-hr exposure to controlled purity He at 750°C and 850°C (1382 and 1562°F) are presented in Figure 2.9. The figure reveals that the microstructures were similar and consisted of coarse primary carbides like those observed in the as-received alloy and intermediate to fine precipitates which were seen both intra- and intergranularly and on twin boundaries.

The surface of HD-556 after 1000-hr exposure at 750°C (1382°F) is shown in Figure 2.10. A 1 to 2 μm (0.04 to 0.08 mils), continuous gray scale containing small, unconnected white particles was visible on the surface of the alloy. Below the scale, a shallow, sparsely populated zone of internal oxidation was evident to a depth of 2 to 3 μm (0.08 to 0.12 mils). A 8 μm (0.31 mils) deep alloy depleted region was also observed. Indications of preferentially-oriented carbide precipitation were observed below the alloy depleted region. After 850°C (1562°F) aging, the surface scale was gray, thicker 3 to 6 μm (0.12 to 0.24 mils) and contained more of the white phase relative to the 750°C (1382°F) aged alloy. It appeared as though the gray surface scale (probably internal oxide) intruded into the substrate in places. Discrete particles were evident to a depth of about 3 to 4 μm (0.12 to 0.16 mils) in a 10 to 16 μm (0.39 to 0.63 mils) deep denuded zone.

Inconel 617

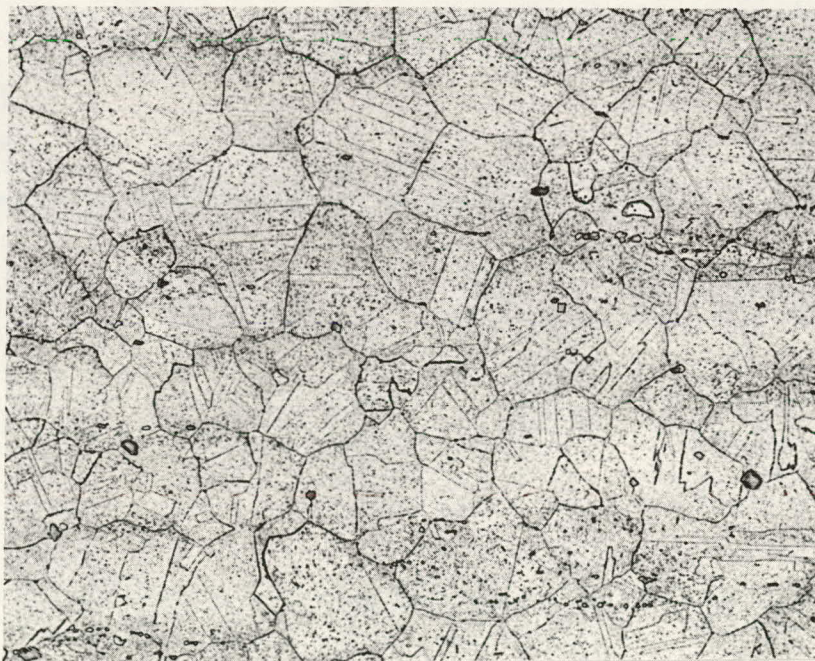
Optical photomicrographs which typify the internal microstructure of Inconel 617 after approximately 1000-hr exposure to controlled purity He at 750°C and 850°C (1382 and 1562°F) are shown in Figure 2.11. The microstructures consisted of a mixture of primary carbides like those observed in the as-received alloy, and the intermediate to fine precipitated carbides appearing both intra- and intergranularly. In many grains, carbides appeared to be preferentially precipitated along certain crystallographic directions. The degree of precipitation was much greater at 750°C (1382°F) than at 850°C (1562°F). Additionally, at 750°C (1382°F), precipitation of fine carbides in the vicinity of grain boundaries and around coarse carbide particles was more predominate than precipitation in the interior of the grain. In contrast, at 850°C (1562°F) a discrete film of intermediate-sized globular carbides was evident at the grain boundaries.



(a)

Kalling's Etch

250X

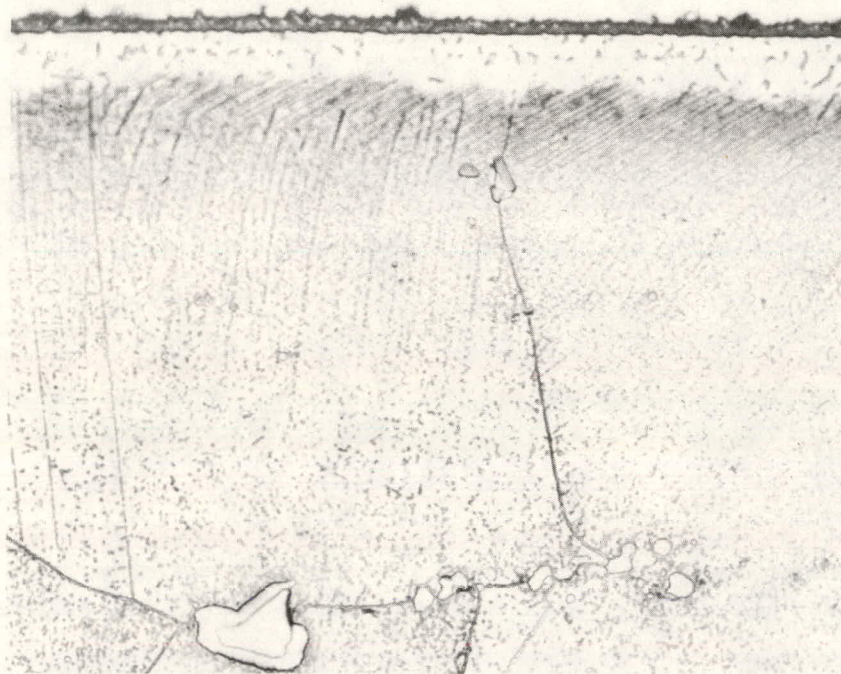


(b)

Kalling's Etch

250X

Figure 2.9 Optical Photomicrographs Showing Etched Internal Microstructure of HD-556 after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

Kalling's Etch

1000X

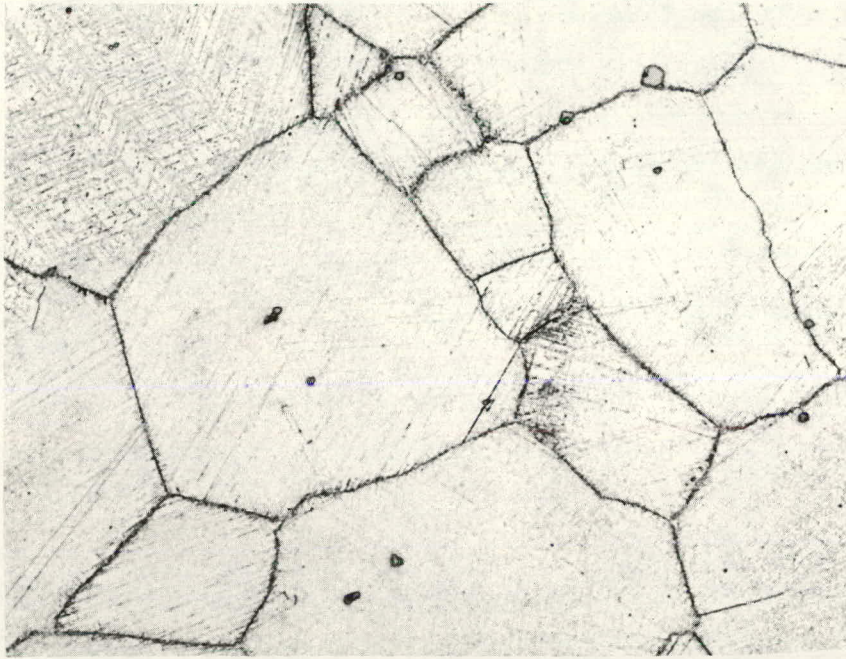


(b)

Kalling's Etch

500X

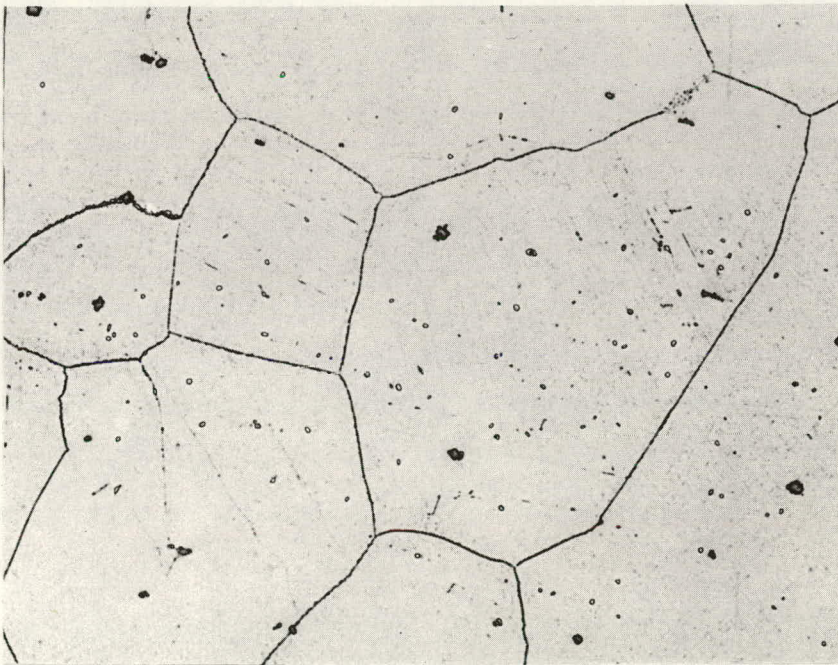
Figure 2.10 Optical Photomicrographs Showing Surface Condition and Etched Subsurface Microstructure of HD-556 after Approximately 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C.



(a)

Oxalic Acid

250X



(b)

Oxalic Acid

250X

Figure 2.11 Optical Photomicrographs Showing Etched Internal Microstructure of Inconel 617 after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 hours at 850°C.

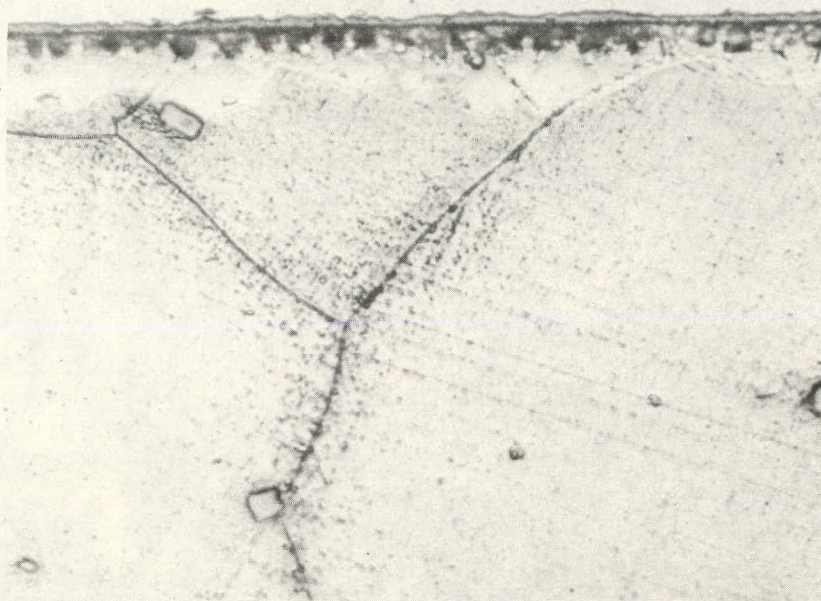
The surface microstructure of Inconel 617 after 750°C (1382°F) aging is shown in Figure 2.12 and was characterized by a 1 μm (0.04 mils) thick, fairly continuous, gray scale containing widely scattered small white particles. Beneath the scale, gray finger-like particles were seen in a 4 μm (0.16 mils) deep internally oxidized region. Additionally, an alloy depleted zone was observed to a depth of 6 to 12 μm (0.24 to 0.47 mils). Carbides within the grain boundaries were larger near the periphery of the sample than at the interior of the specimen. After 850°C (1562°F) aging, the morphology of the surface products was similar to that observed on the 750°C (1382°F) specimen, Figure 2.12; however, the scale formation, internal oxidation and alloy depletion were more extensive. The surface scale thickness was found to be 2 to 5 μm, (0.08 to 0.20 mils), the internally oxidized zone was 5 to 14 μm (0.20 to 0.55 mils), and the depth of alloy depletion was found to be about 10 μm (0.39 mils). An increase of fine carbide precipitation was also noted to a depth of about 37 to 59 μm (1.5 to 2.3 mils).

Subsequent to aluminide coating and 750°C (1382°F) aging in controlled purity He, the internal microstructure was similar to the uncoated and 750°C (1382°F) aged alloy except that precipitation of carbides in the vicinity of grain boundaries was reduced, Figure 2.13. Additionally, there was little difference in internal microstructure between the uncoated and coated samples of Inconel 617 aged at 850°C (1562°F). Coating thickness after 750°C (1382°F) aging was comparable to the 69 μm (2.7 mils) coating thickness measured on the as-coated alloy. However, after 850°C (1562°F) aging, an apparent coating thickness of about 84 μm (3.3 mils) was determined, suggesting additional interdiffusion between coating and substrate. Below the coating some fine to intermediate carbide precipitation was evident. This zone had been denuded of carbides by the coating process.

Surface scale thicknesses of 1 μm (0.04 mils) and 2 to 5 μm (0.08 to 0.20 mils) were revealed after 750°C and 850°C (1382°F and 1562°F) aging, respectively.

Inconel 618E

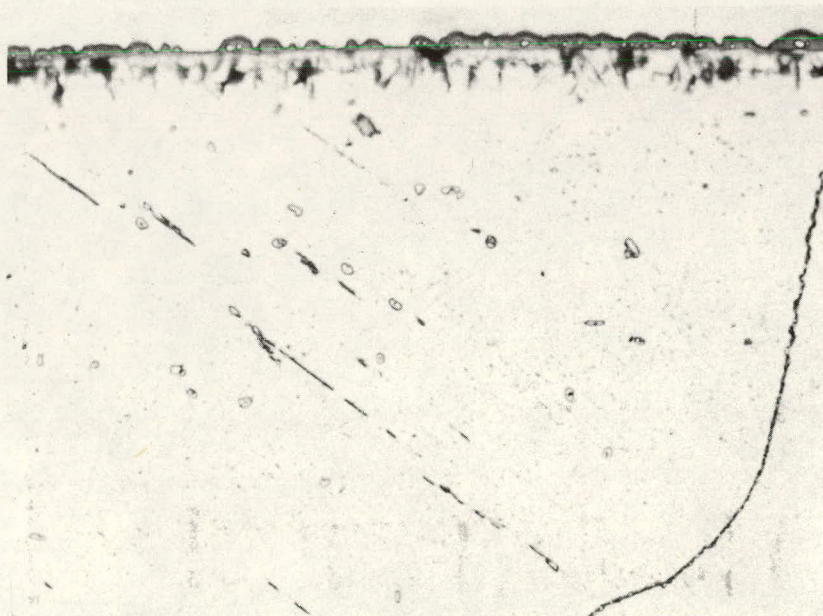
Typical optical photomicrographs of the internal microstructure of Inconel 618E after approximately 1000-hr exposure to controlled purity He at 750°C and 850°C (1382°F and 1562°F) are shown in Figure 2.14. The microstructures were essentially



(a)

Oxalic Acid

1000X



(b)

Oxalic Acid

500X

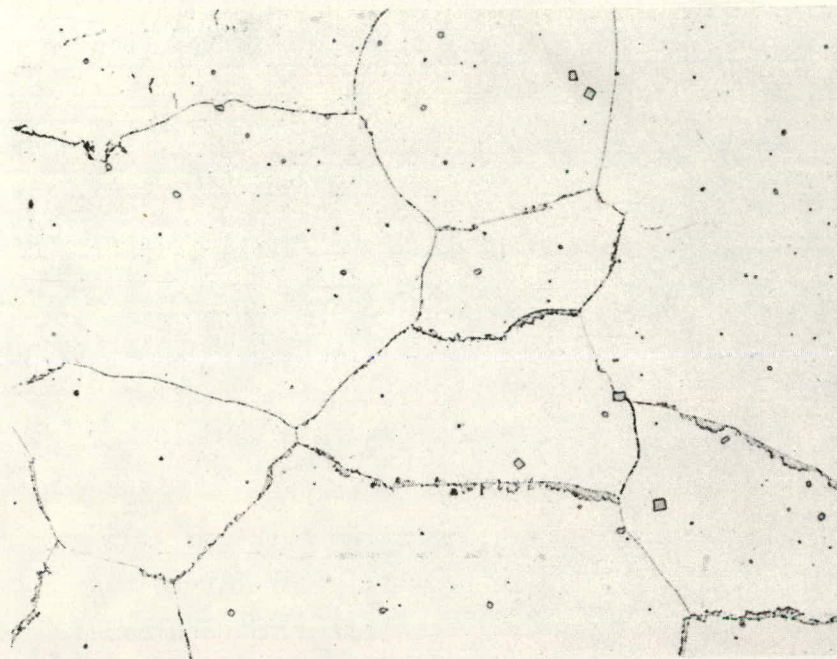
Figure 2.12 Optical Photomicrographs Showing Surface Condition and Etched Subsurface Microstructure of Inconel 617 after Approximately 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C.



Oxalic Acid

200X

Figure 2.13 Optical Photomicrograph Showing Surface Condition and Etched Substrate Microstructure of Coated Inconel 617 after 1000 Hour Exposure in Controlled Purity He at 750°C. Note the fine precipitation in the formerly ~ 560 μm deep denuded zone below the coating.



(a)

Oxalic Acid

250X



(b)

Oxalic Acid

250X

2.14 Optical Photomicrographs Showing Etched Internal Microstructure of Inconel 618E after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.

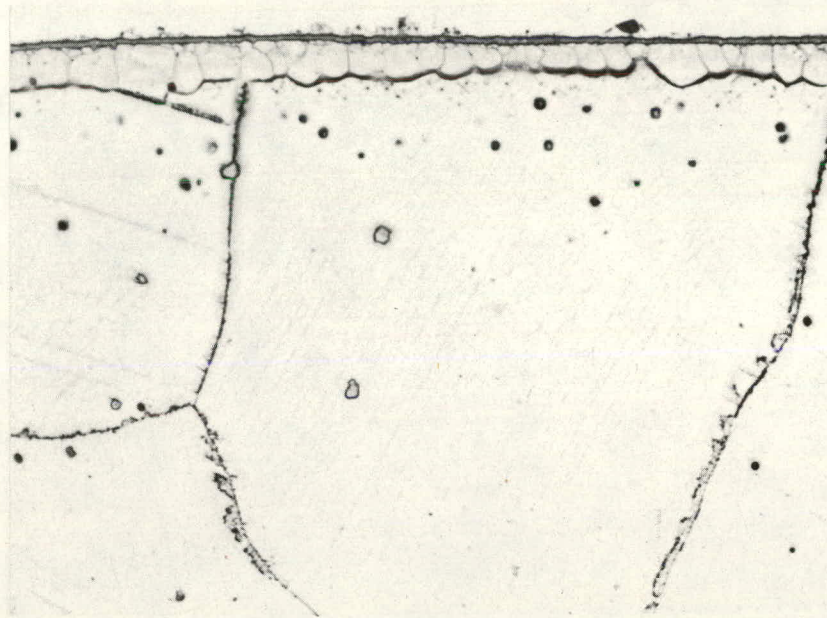
equivalent and consisted of a mixture of coarse primary carbides (which were also observed in the as-received alloy) and intermediate to fine precipitated carbides appearing both intra- and intergranularly.

The surface microstructure of Inconel 618E after exposure at 750°C and 850°C (1382 and 1562°F) is shown in Figure 2.15. After 750°C (1382°F) aging, the surface microstructure consisted of a fairly uniform and continuous 2 μm (0.09 mils) thick gray scale which contained a few white particles (probably carbides). Increased carbide precipitation was observed to a depth of about 18 μm (0.71 mils). The scale morphology was similar after 850°C (1562°F) aging; a thickness of 2 to 7 μm (0.08 to 0.28 mils) was determined. No evidence of spallation was observed. In both cases, no internal oxidation was evident. Alloy depleted zones of 6 to 14 μm (0.24 to 0.55 mils) at 750°C (1382°F) and at 850°C (1562°F) were found. It is of interest to note that the alloy depleted zones appeared to be recrystallized in both cases.

Hastelloy X

The photomicrographs shown in Figure 2.16 are typical of the microstructure of Hastelloy X after approximately 1000-hr aging in controlled purity He at 750° and 850°C (1382 and 1562°F). Microstructures were morphologically similar and consisted of a mixture of large globular carbides plus fine to intermediate precipitated carbides which appeared within grains and at grain and twin boundaries. Areas adjacent to grain boundaries appeared denuded after 750°C (1382°F) exposure.

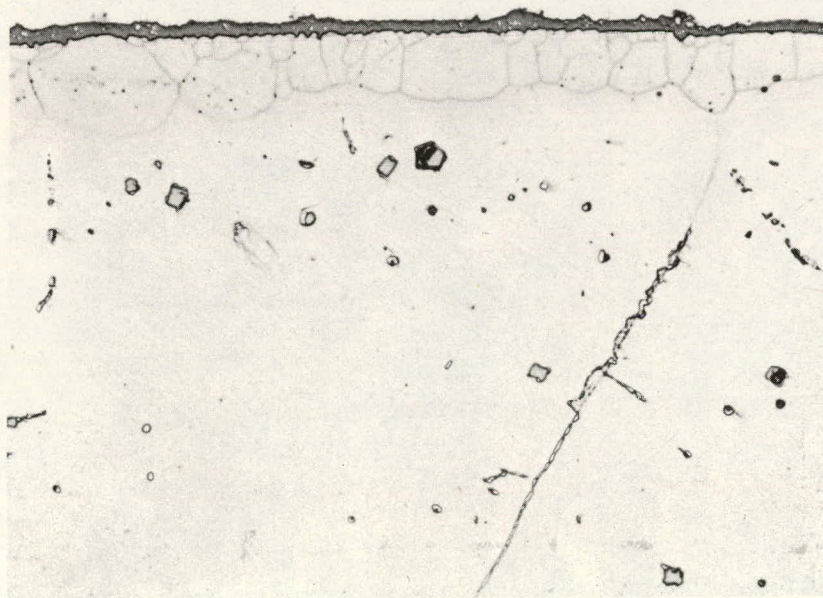
The surface conditions after exposure at 750° and 850°C (1382 and 1562°F) are shown in Figure 2.17. The surface after 750°C (1382°F) exposure was covered with a continuous gray scale, 2 μm (0.08 mils) thick, containing a number of fine, white, unconnected particles. Below the scale, a few gray, globular particles, presumably oxides, were observed and a number of grain boundaries appeared to be preferentially oxidized to a depth of about 10 μm (0.39 mils). This is of significance since preferential oxidation of grain boundaries can have a more deleterious effect on mechanical properties than surface oxidation. A 4 to 10 μm (0.16 to 0.39 mils) deep alloy depleted zone was also in evidence with increased fine carbide precipitation observed to a depth of about 36 μm (1.4 mils). After 850°C (1562°F) aging, the surface scale, 2 to 3 μm (0.08 to



(a)

Oxalic Acid

500X



(b)

Oxalic Acid

500X

Figure 2.15 Optical Photomicrographs Showing Surface Condition and Etched Subsurface Microstructure of Inconel 618E after Approximately 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C.



(a)

Oxalic Acid

250X

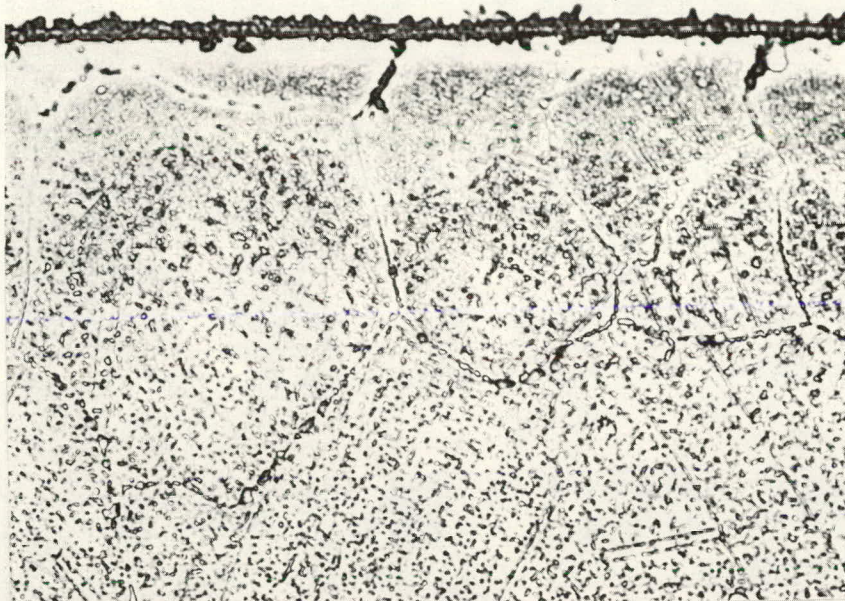


(b)

Oxalic Acid

250X

Figure 2.16 Optical Photomicrographs Showing Etched Internal Microstructure of Hastelloy X after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

Oxalic Acid

1000X



(b)

Oxalic Acid

500X

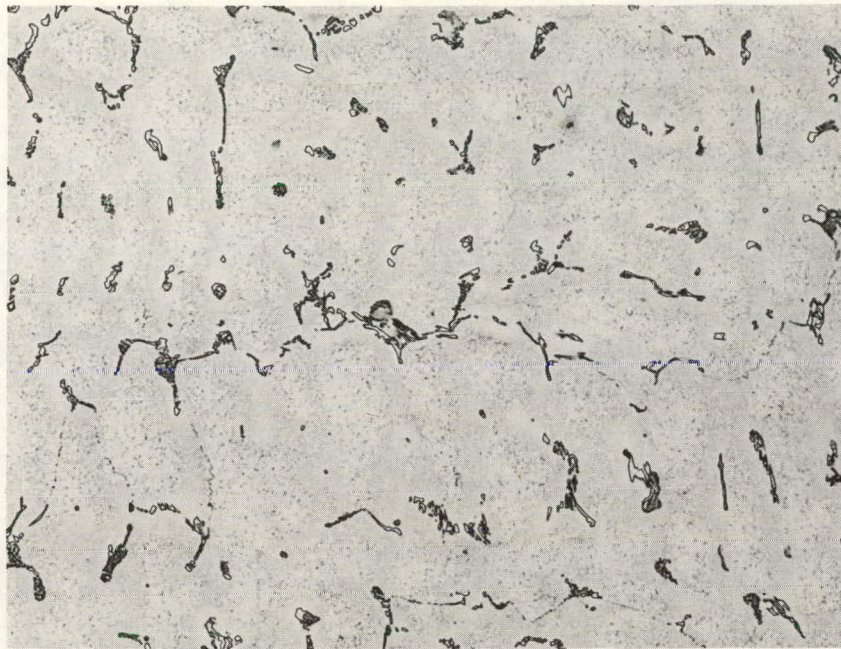
Figure 2.17 Optical Photomicrographs Showing Surface Condition and Etched Subsurface Microstructure of Hastelloy X after Approximately 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C.

0.12 mils) thick, was similar to the scale on the 750°C (1562°F) exposed specimen. An internally oxidized zone about 10 μm (0.39 mils) deep with mainly globular particles in evidence was contained within a 10 to 14 μm (0.39 to 0.55 mils) deep alloy depleted region. Near the surface of the 850°C (1382°F) aged alloy and to a depth of about 120 μm (4.7 mils), the precipitation of carbides seemed significantly increased, probably as a result of carburization. In this region, etched grain boundaries and twin boundaries appeared thicker and more continuous, and the density of precipitated intragranular carbides was increased relative to that observed in the center of the specimen.

HK-40

Optical photomicrographs showing the internal microstructure of HK-40 after approximately 1000-hr aging in controlled purity He at 750°C and 850°C (1382 and 1562°F) are presented in Figure 2.18. Exposure of HK-40 at 750°C and 850°C (1382 and 1562°F) resulted in the copious precipitation of fine, roughly spherical carbide particles. These particles are often referred to as secondary carbides. The cored structure is quite apparent after aging at the lower exposure temperature, whereas aging at 850°C (1562°F) has virtually eliminated evidence of coring.

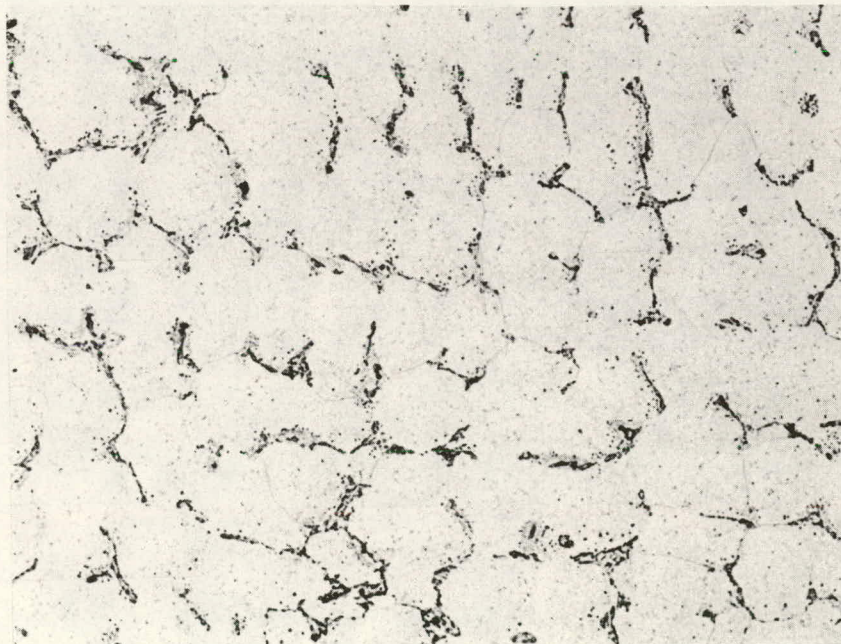
The surface features of HK-40 were quite similar after both the 750°C and 850°C (1382 and 1562°F) aging treatments. The scales appeared to be discontinuous or spalled and were duplex in nature with an outer medium gray layer and a secondary, relatively thin, dark gray layer, Figure 2.19. Both layers contained a very fine white particulate phase in some areas. Overall thickness of the scale on the 750°C (1382°F) aged specimen was 2 μm (0.08 mils), while the specimen exposed at 850°C (1562°F) revealed a scale thickness of 2 to 7 μm (0.08 to 0.28 mils). No evidence of internal oxidation was observed; however, the thin, dark gray layer is believed to be a surface oxide scale. Below the surface scale, a depleted region extended 8 μm (0.31 mils) deep in the 750°C (1382°F) specimen and 8 to 10 μm (0.31 to 0.39 mils) in the 850°C (1562°F) exposed specimen. The depleted zone was void of the small secondary (precipitated) carbides.



(a)

Murakami's Etch

250X

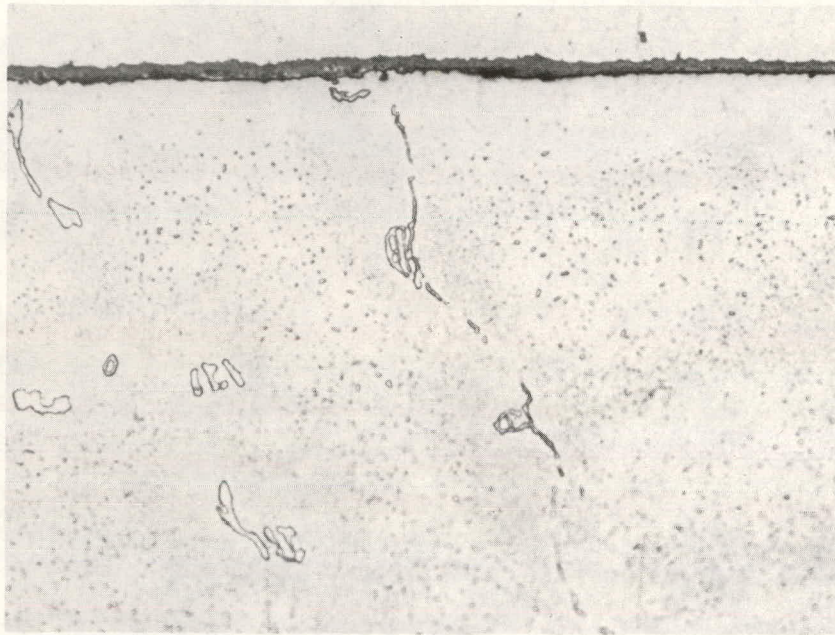


(b)

Murakami's Etch

250X

Figure 2.18 Optical Photomicrographs Showing Etched Internal Microstructure of HK-40 after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

Murakami's Etch

1000X



(b)

Unetched

1000X

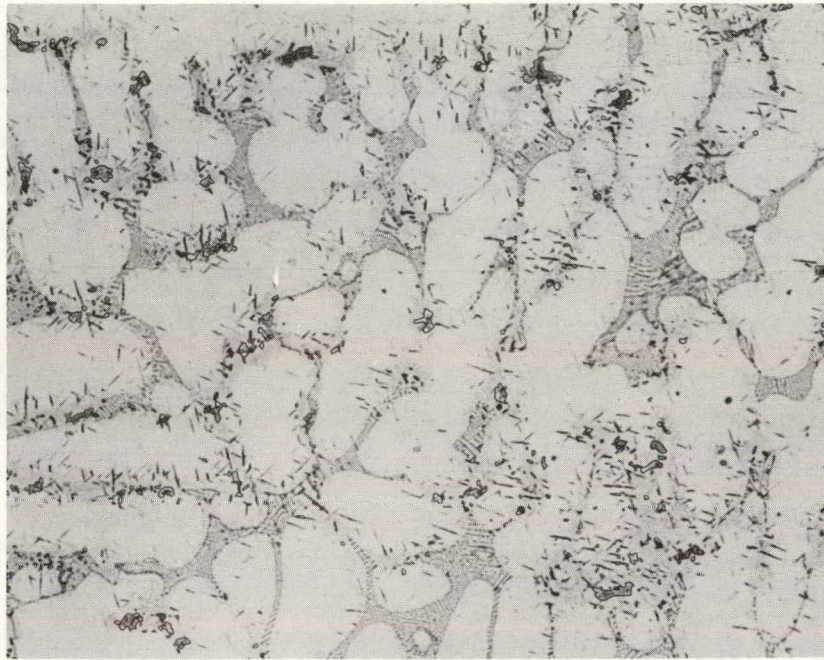
Figure 2.19 Optical Photomicrographs Showing Surface Condition (a) and (b), and Etched Subsurface Microstructure, (a) Only, of HK-40 after ~1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C.

IN-519

The internal microstructure of IN-519 after approximately 1000-hr aging in controlled purity He at 750°C and 850°C (1382 and 1562°F) is typified by the optical photomicrographs shown in Figure 2.20. As with the other centrifugally cast alloys, exposure of IN-519 at 750°C and 850°C (1382 and 1562°F) resulted in precipitation of secondary carbides. At 750°C (1382°F), secondary carbide precipitation was more pronounced than at 850°C (1562°F) and was observed both inter- and intradendritically, with much of the intradendritic precipitation occurring in areas near eutectic carbides. Some coarsening of eutectic carbides was also apparent. In addition, the microstructure also contained a blocky, globular or acicular precipitate quite typical of the sigma phase. The acicular phase was generally observed around the periphery of the larger dendrites; however, it was also seen completely across a number of the smaller dendrites.

At 850°C (1562°F), precipitated carbides generally appeared within interdendritic regions near eutectic carbides or at dendrite boundaries. This resulted in the apparent coarsening of eutectic carbides. Little in the way of intradendritic secondary carbide precipitation was observed. The dark etching, blocky particles in the interdendritic regions also suggest sigma phase precipitation in these areas.

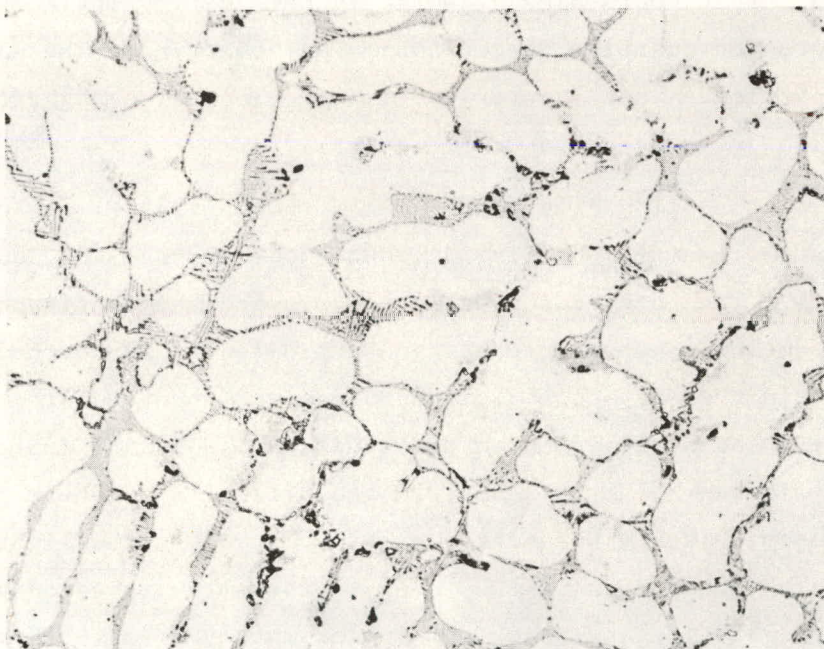
The surface of the alloy after exposure at 750°C (1382°F) was covered with a thin, approximately 1 μm (0.04 mils) discontinuous or spalled, double-layered scale, Figure 2.21. The inner layer was very thin and semicontinuous. The outer layer was continuous where present and exhibited a gray etched appearance. Little difference in the surface scale morphology was observed after 850°C (1562°F) exposure. The scale appeared to be discontinuous and duplex in nature, with an overall thickness of 1 to 3 μm (0.04 to 0.12 mils). Where present, the inner, dark-etching scale was nearly continuous, and a few light-etching particles were in evidence between the layers. The alloy revealed a depleted zone after 750°C (1382°F) aging which was approximately 3 μm (0.12 mils) deep below the scale. After 850°C (1562°F) aging, the depleted zone was approximately 14 μm (0.55 mils) deep.



(a)

Murakami's Etch

250X

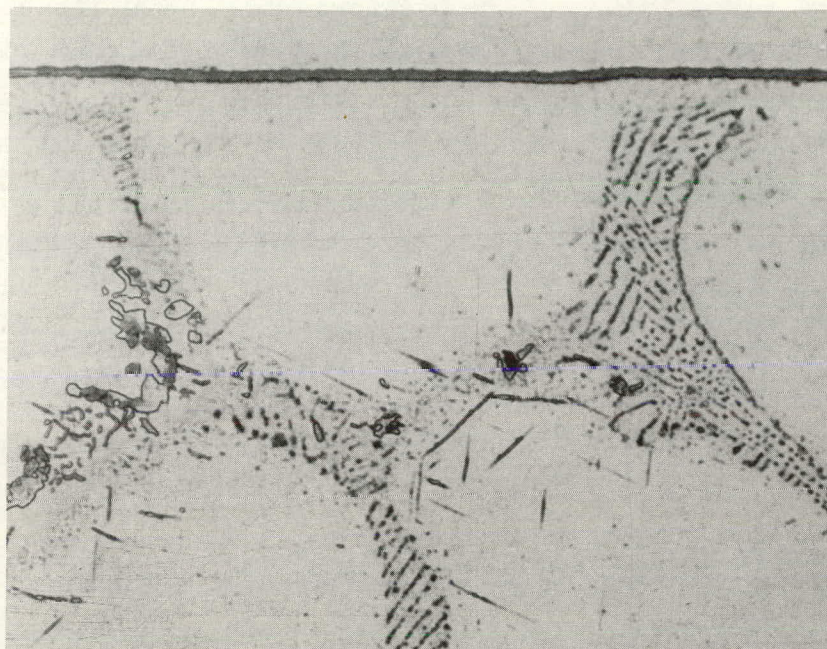


(b)

Murakami's Etch

250X

Figure 2.20 Optical Photomicrographs Showing Etched Internal Microstructure of IN-519 after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

Murakami's Etch

1000X



(b)

Unetched

1000X

Figure 2.21 Optical Photomicrographs Showing Surface Condition (a) and (b), and Etched Subsurface Microstructure, (a) only of IN-519, after ~ 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C .

Manaurite 36XS

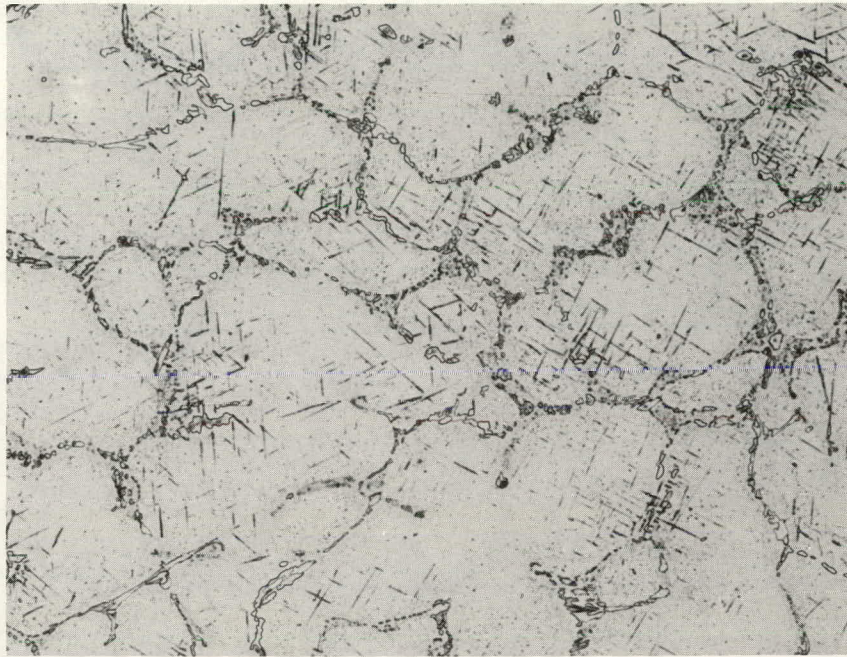
Representative optical photomicrographs showing the internal microstructure of Manaurite 36XS after approximately 1000-hr exposure in controlled purity He at 750°C and 850°C (1382 and 1562°F) are shown in Figure 2.22. After 750°C (1382°F) exposure, the microstructure revealed an abundant degree of secondary carbide precipitation both inter- and intradendritically. Coarsening of the eutectic carbide was also apparent. The intradendritic regions also contained acicular particles preferentially precipitated along certain crystallographic directions. These needle-like particles are typical of the sigma phase.

In contrast, at 850°C (1562°F) the acicular phase was no longer evident. However, copious amounts of secondary carbide precipitation were still in evidence even after 750°C (1382°F) exposure. Some coalescence of secondary carbides also was evident. Coarsening of the eutectic carbide structure was also apparent at both aging temperatures.

The surface condition after exposure at 750°C and 850°C (1382 and 1562°F) is shown in Figure 2.23. After 750°C (1382°F) exposure, the scale appeared to be composed of a continuous outer layer and a white, noncontinuous secondary layer. The overall thickness of the scale was about 2 μm (0.08 mils). Although no evidence of internal oxidation was apparent, a 7 μm (0.28 mils) deep alloy depleted region was observed below the scale and some fine, preferentially-oriented carbide precipitation (not shown) was evident to a depth of 12 μm (0.47 mils). The surface scale after 850°C (1562°F) exposure was discontinuous and/or partially spalled. Where observed, the scale appeared predominantly gray, contained a significant amount of discontinuous white layer and was about 3 to 5 μm (0.12 to 0.20 mils) thick. An internally oxidized zone also was observed to a depth of approximately 20 μm (0.79 mils) with interdendritic regions being most affected. Alloy depletion was evident to a depth of 6 to 7 μm (0.24 to 0.28 mils).

Manaurite 900

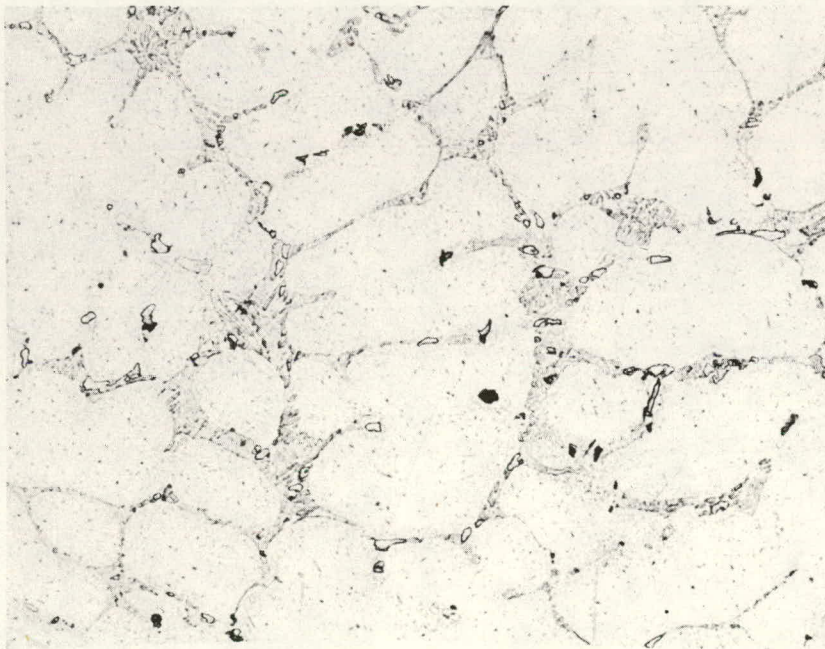
Optical photomicrographs showing the internal microstructure of Manaurite 900 after nominally 1000-hr exposure in controlled purity He at 750°C and 850°C (1382 and 1562°F) are presented in Figure 2.24. Aging at both temperatures resulted in the precipitation of fine carbide particles. Secondary carbide



(a)

Murakami's Etch

250X

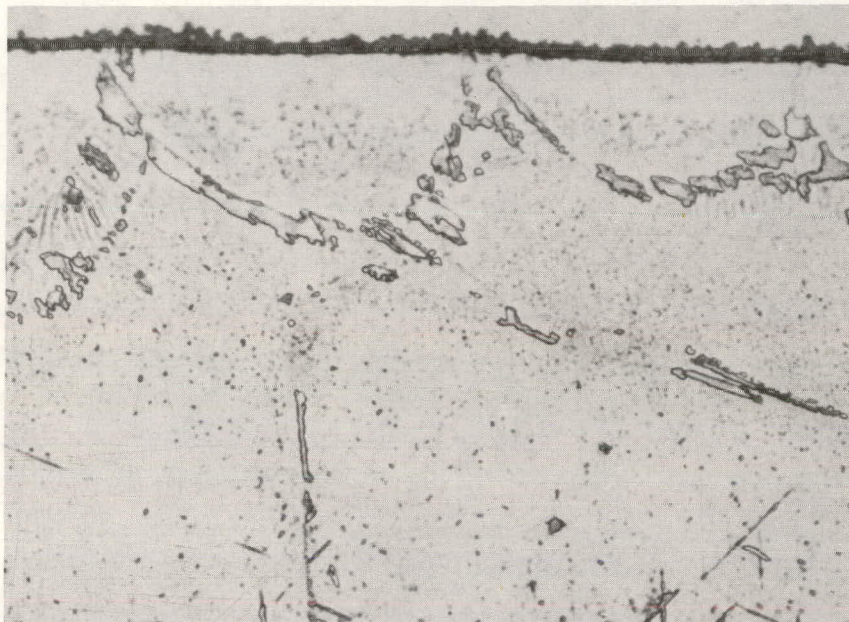


(b)

Murakami's Etch

250X

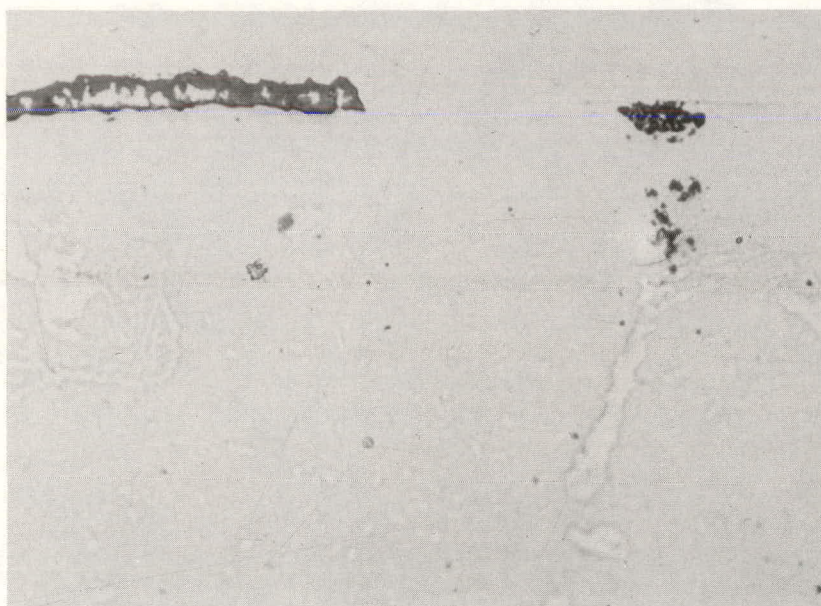
Figure 2.22 Optical Photomicrographs Showing Etched Internal Microstructure of Manaurite 36XS after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

Murakami's Etch

1000X

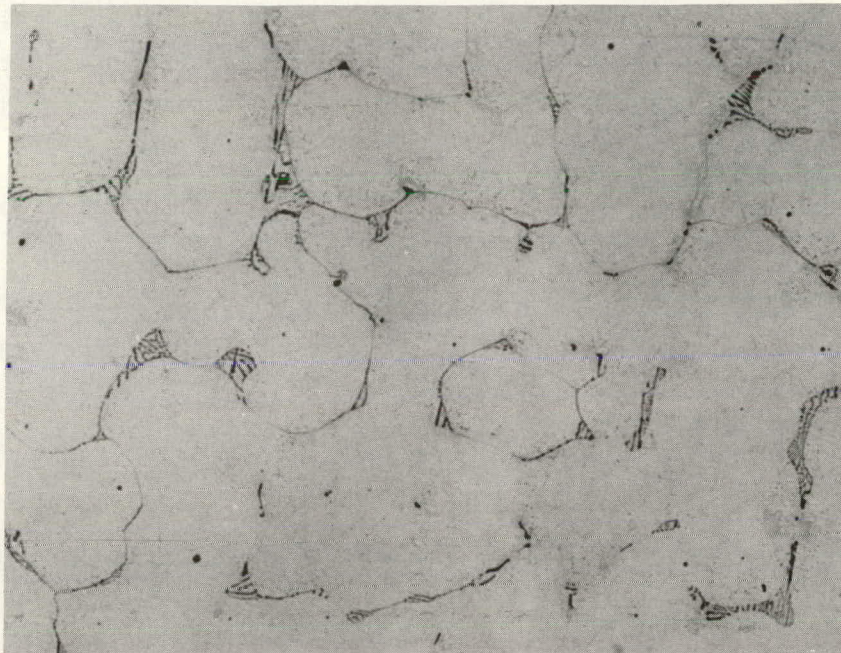


(b)

Unetched

1000X

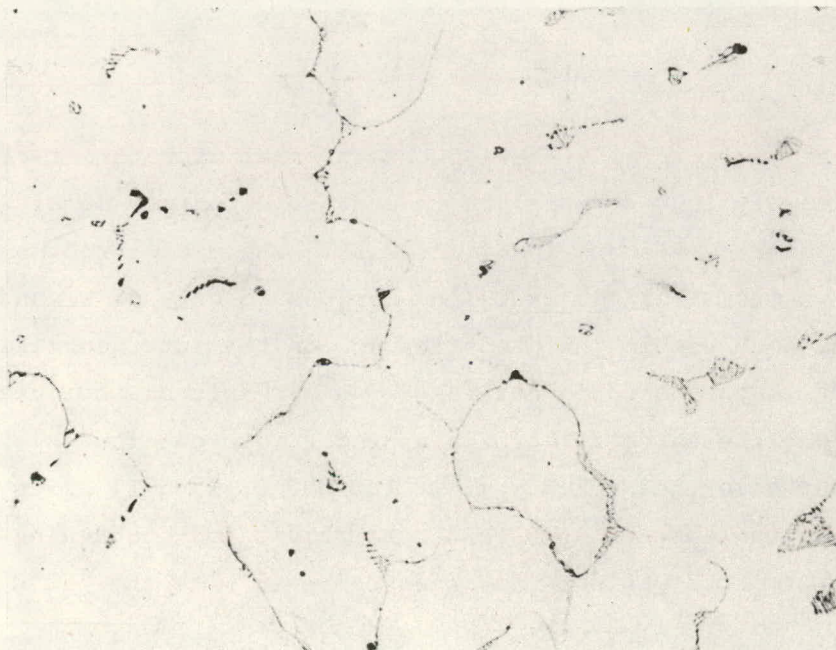
Figure 2.23 Optical Photomicrographs Showing Surface Condition (a) and (b), and Etched Subsurface Microstructure (a) only of Manaurite 36XS after ~ 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C .



(a)

Murakami's Etch

250X



(b)

Murakami's Etch

250X

Figure 2.24 Optical Photomicrographs Showing Etched Internal Microstructure of Manaurite 900 after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.

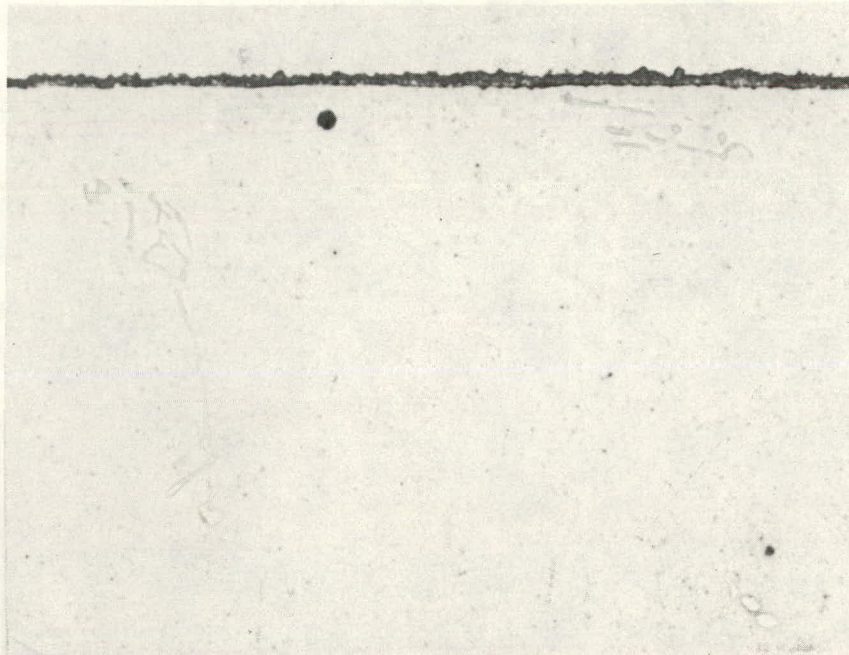
precipitation was observed both inter- and intradendritically after exposure at the two aging temperatures; however, after aging at 850°C (1562°F) precipitation in general was much reduced and fine intradendritic carbides were observed only in the vicinity of eutectic carbides. It is of interest to note that the degree of carbide precipitation is not as abundant in Manaurite 900 as was revealed by some of the other centrifugally cast alloys under study. This is probably attributable to the relatively low level of carbon present in this alloy compared to the other alloys in this class. Some coarsening of the lace-like eutectic carbide structure was also observed at both aging temperatures.

The surface microstructure after 750°C and 850°C (1382 and 1562°F) aging is shown in Figure 2.25. The scale after 750°C (1382°F) exposure was typically 1 μm (0.04 mils) thick, continuous, and duplex in nature. The scale was composed of a medium gray outer layer and a dark gray, very thin and discontinuous inner layer. A few areas of preferentially-oriented carbide precipitation were noted to a depth of approximately 18 μm (0.71 mils). After exposure at 850°C (1562°F) the duplex nature of the scale was much more pronounced, and a thickness of 2 to 3 μm (0.08 to 0.12 mils) was observed. Little, if any, internal oxidation or alloy depletion was observed.

Mo-Re 2

Typical optical photomicrographs showing the internal microstructure of Mo-Re 2 after approximately 1000-hr exposure in controlled purity He at 750°C and 850°C (1382 and 1562°F) are shown in Figure 2.26. The microstructures after aging were somewhat similar and exhibited copious amounts of secondary carbide precipitation, both within the dendrites and in the interdendritic regions on or adjacent to the eutectic carbides. As a result of the interdendritic precipitation, the eutectic carbide appeared more continuous relative to the as-cast alloy, especially after the 850°C (1562°F) aging. Additionally, at 850°C, coalescence of secondary carbides and coarsening of the eutectic carbide structure was more advanced compared with the 750°C (1382°F) alloy.

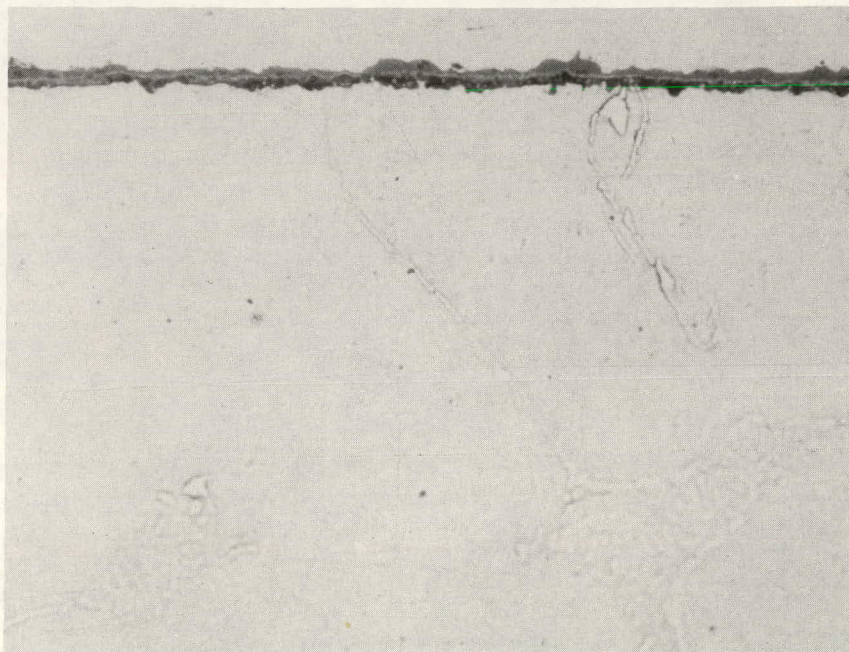
After the 750°C exposure, the surface of the alloy was characterized by a gray, fairly uniform and continuous scale about 1 μm (0.04 mils) deep, Figure 2.27. Below the scale small dark gray particles were evident in a 1 μm (0.04 mils) deep internally oxidized zone, and areas of alloy depletion were evident to a



(a)

Unetched

1000X

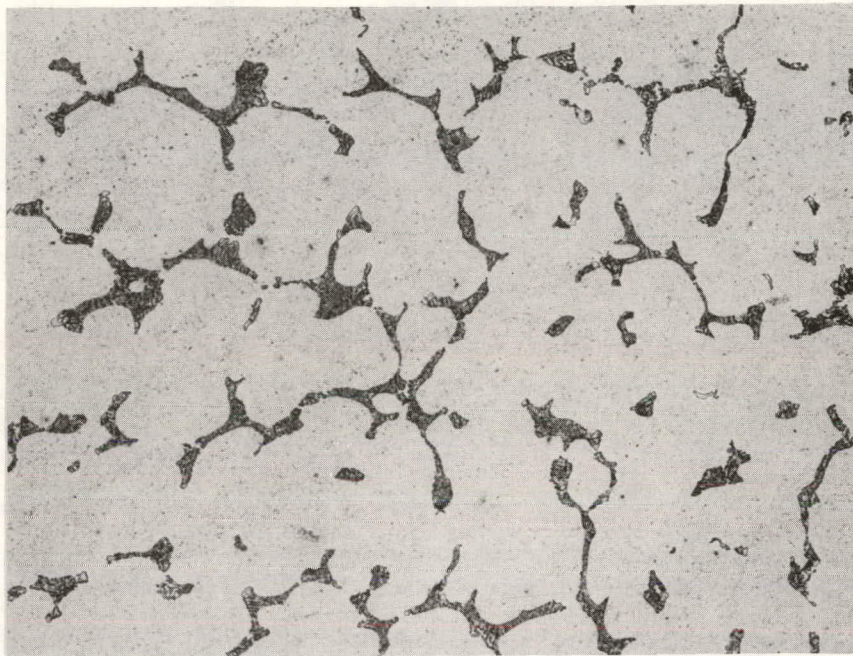


(b)

Unetched

1000X

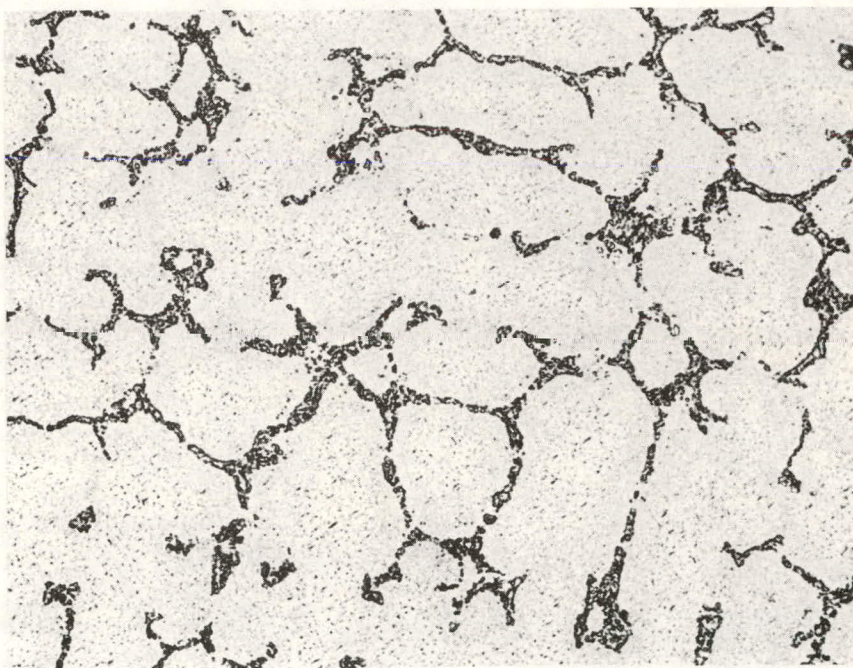
Figure 2.25 Optical Photomicrographs Showing Surface Condition of Manaurite 900 after ~ 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C .



(a)

Murakami's Etch

250X

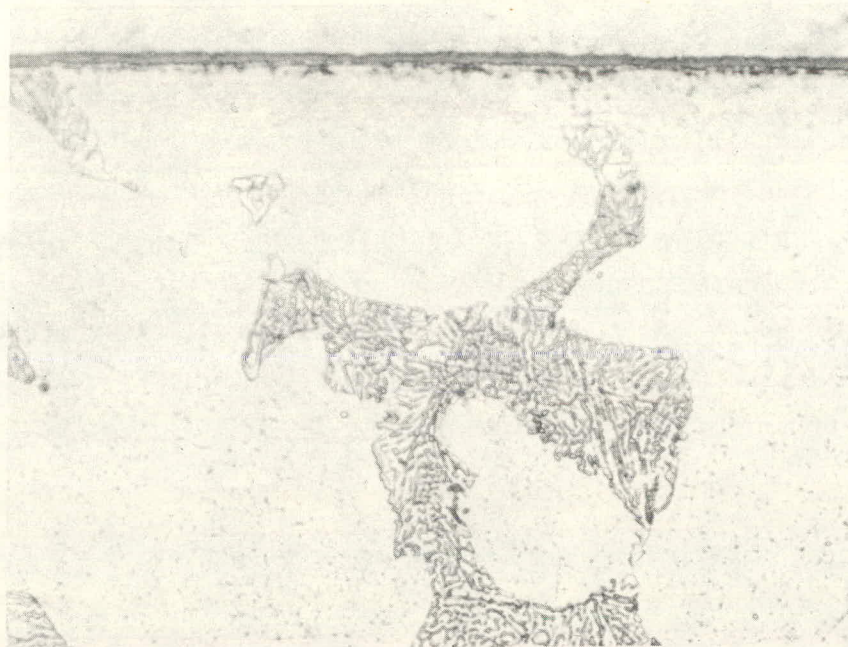


(b)

Murakami's Etch

250X

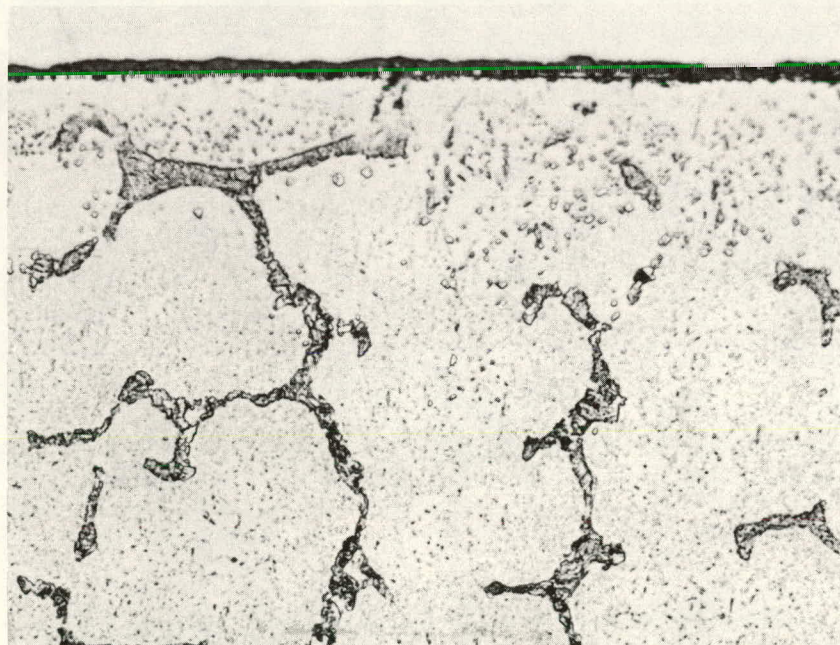
Figure 2.26 Optical Photomicrographs Showing Etched Internal Microstructure of Mo-Re 2 after Exposure in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

Murakami's Etch

1000X



(b)

Murakami's Etch

500X

Figure 2.27 Optical Photomicrographs Showing Surface Condition and Etched Surface Microstructure of Mo-Re 2 after ~ 1000 Hour Exposure in Controlled Purity He at (a) 750°C and (b) 850°C .

depth of up to 5 to 6 μm (0.20 to 0.24 mils). The scale on the 850 $^{\circ}\text{C}$ (1562 $^{\circ}\text{F}$) exposed alloy was thicker, 4 to 5 μm (0.16 to 0.20 mils), but continuous. It contained a noncontinuous white layer near the alloy surface. Below the scale, internal oxidation was evident to a depth of 10 to 20 μm (0.39 to 0.79 mils) with the most severe and deepest penetration taking place in the interdendritic regions. An alloy depleted zone 8 to 10 μm (0.31 to 0.39 mils) deep was also in evidence below the scale. Possible evidence of carburization was observed after both the 750 $^{\circ}\text{C}$ and 850 $^{\circ}\text{C}$ (1382 and 1562 $^{\circ}\text{F}$) exposures; the presence of coarse carbides was noted to depths of about 7 to 9 μm (0.28 to 0.34 mils) and approximately 60 μm (2.36 mils), respectively.

MA-956

A photomicrograph showing the microstructure representative of MA-956 both before and after exposure is shown in Figure 2.28. In the as-fabricated condition, the internal microstructure consists of a very fine dispersion of yttrium-aluminum oxide particles less than 50 nm in. (0.002 mils) in diameter. Additionally, fine dispersions of Cr_2O_3 and Ti (C,N) are reported to be present in this material. The size of the latter particles is indicated to be well above 100 nm (0.004 mils). After 1000-hr at 750 $^{\circ}\text{C}$ (1382 $^{\circ}\text{F}$) or 1104 hr at 850 $^{\circ}\text{C}$ (1562 $^{\circ}\text{F}$), no differences in the internal microstructure could be seen as compared to the as-received material, Figure 2.28. The fine dispersed particles seen in Figure 2.28 are presumably Ti (C,N).

The surface of the 750 $^{\circ}\text{C}$ (1382 $^{\circ}\text{F}$) aged specimen of alloy Ma-956 was characterized by a continuous, approximately 0.5 μm (0.02 mils) thick scale. After 850 $^{\circ}\text{C}$ (1562 $^{\circ}\text{F}$) aging, the scale was 1 to 2 μm (0.04 to 0.8 mils) thick and gray in color. No evidence of internal oxidation or an alloy depleted zone was observed in either case.

2.6.2.2 Direct Cycle Helium Turbine Alloys

TZM

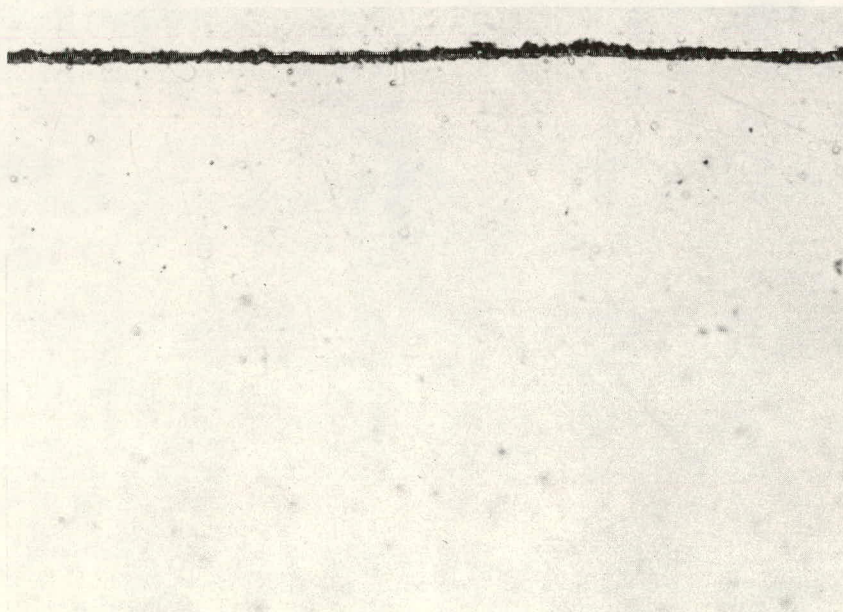
TZM was exposed in controlled purity He at 750 $^{\circ}\text{C}$ (1382 $^{\circ}\text{F}$) for 1000 hours and 850 $^{\circ}\text{C}$ (1562 $^{\circ}\text{F}$) for 1104 hours. In neither case, were substantial changes in internal structure or surface character found. Figure 2.29 (a) shows the typical post exposure microstructure found at 750 $^{\circ}\text{C}$ (1382 $^{\circ}\text{F}$). The structure found at 850 $^{\circ}\text{C}$ (1562 $^{\circ}\text{F}$) is very similar to that seen at 750 $^{\circ}\text{C}$ (1382 $^{\circ}\text{F}$).



(a)

Glyceregia Etch

1000X



(b)

Unetched

1000X

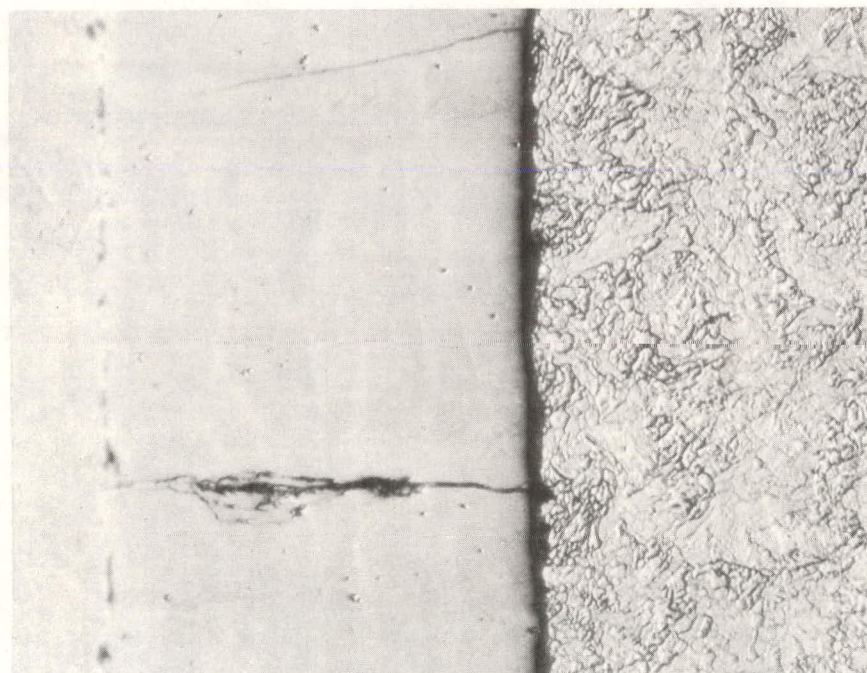
Figure 2.28 Optical Photomicrographs Showing (a) Etched Internal Microstructure and (b) Unetched Surface Condition of MA-956 after 1104 Hour Exposure in Controlled Purity He at 850°C.



(a) TZM

Murakami's Etch

1000X



(b) TZM-W3 Coated

Murakami's Etch

1000X

Figure 2.29 Optical Photomicrographs of TZM Exposed in Controlled Purity He at 750°C for 1000 Hours. (a) Uncoated, (b) W3 Coated.

Figure 2.29 (b) shows the post 750°C (1382°F) exposure W3 coating and TZM substrate structure. It is evident that through cracks can be found in the coating. These openings provide a path for corrosive attack, and minor subsurface attack was found at both 750°C and 850°C (1382 and 1562°F).

YD-Ni

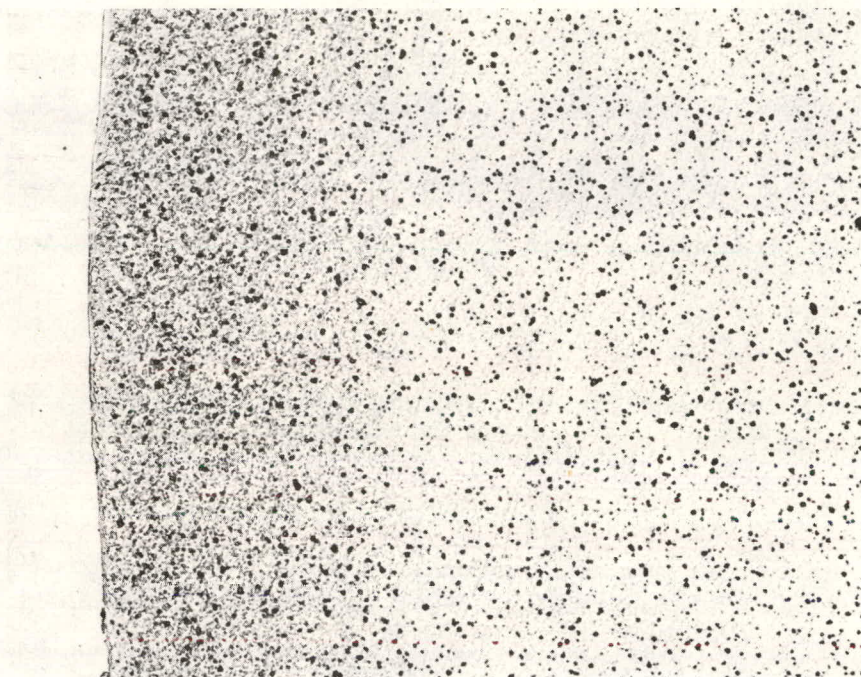
Optical photomicrographs of YD-Ni corrosion pins exposed at 750 and 850°C (1382 and 1562°F) for approximately 1000 hours are shown in Figure 2.30. Neither specimen developed a noticeable surface scale during exposure. A small weight change, 0.187 Mg/cm², was noted after exposure at 750°C (1382°F), but at 850°C (1562°F) the weight change was substantial, 1.332 Mg/cm². The appearance of large nodules compared to the dispersion, after exposure at 850°C (1562°F) may be the cause of the large increase in weight gain from 750 to 850°C (1382 to 1562°F). The exact nature of these nodules is presently under investigation.

YD-NiCrAl

Optical photomicrographs of YD-NiCrAl exposed in controlled purity He at 750°C (1382°F) for 1000 hours and at 800°C (1562°F) for 1104 hours are shown in Figure 2.31. The internal microstructures were unaffected by exposure in both cases. Surface and subsurface attack were apparent after exposure at 850°C (1562°F). The attack is internal oxidation of Al and/or Cr which are found alloyed at 4.5 and 16.1 weight percent respectively. MA-754, which contains similar Cr levels, but a much lower Al level of 0.29 weight percent, and, in addition, has 0.41 weight percent Ti, showed no similar subsurface attack at 850°C (1562°F). This would indicate that the higher Al content in YD-NiCrAl is a significant factor in the formation of internal oxide.

MA-754

Optical photomicrographs of MA-754 exposed in controlled purity He at 750°C (1382°F) for 1000 hours and at 850°C (1562°F) for 1104 hours are shown in Figure 2.32. At both temperatures, only a thin surface scale was formed, and the internal microstructure remains unaltered. There is an increase of small twins in the specimen volume just below the exposed surface. Twins of this nature are commonly found in MA-754 after the post processing heat



(a)

2-2-1 Etch

250X

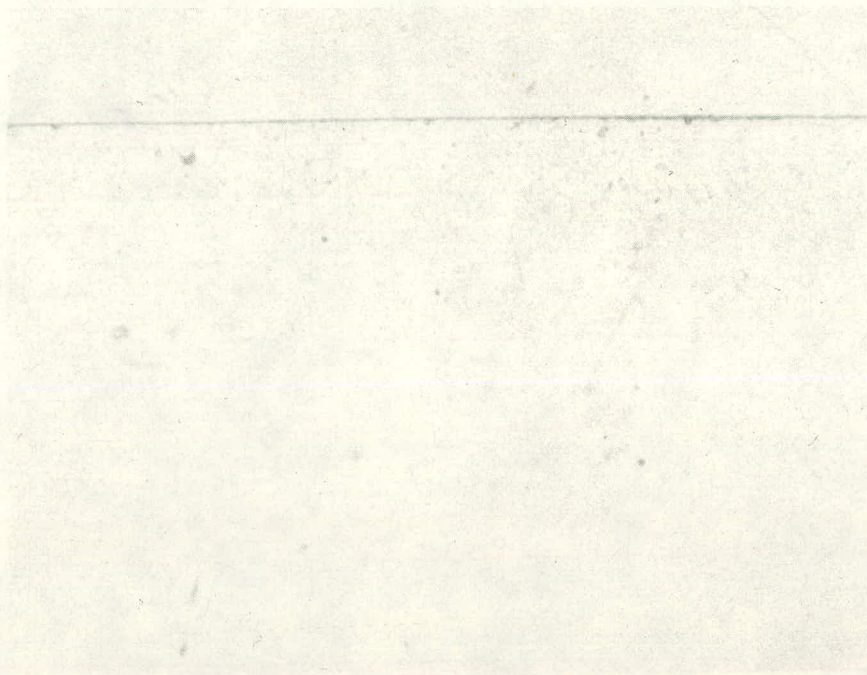


(b)

2-2-1 Etch

250X

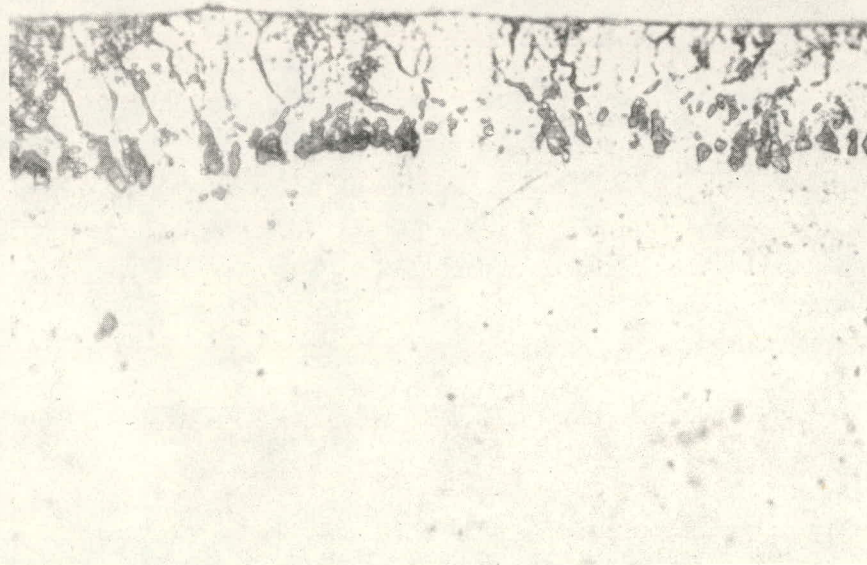
Figure 2.30 Optical Photomicrographs of Yd-Ni Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

As-Polished

1000X



(b)

As-Polished

1000X

Figure 2.31 Optical Photomicrographs of YD-NiCrAl Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

2-2-1 Etch

1000X



(b)

2-2-1 Etch

1000X

Figure 2.32 Optical Photomicrographs of MA-754 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.

treatment, typically 1 hour at 1316°C (2400°F). The occurrence of a higher number density of these twins at the specimen surface may be in part due to mechanical working imparted to the subsurface during specimen machining and subsequent low stress grinding.

MM-0011

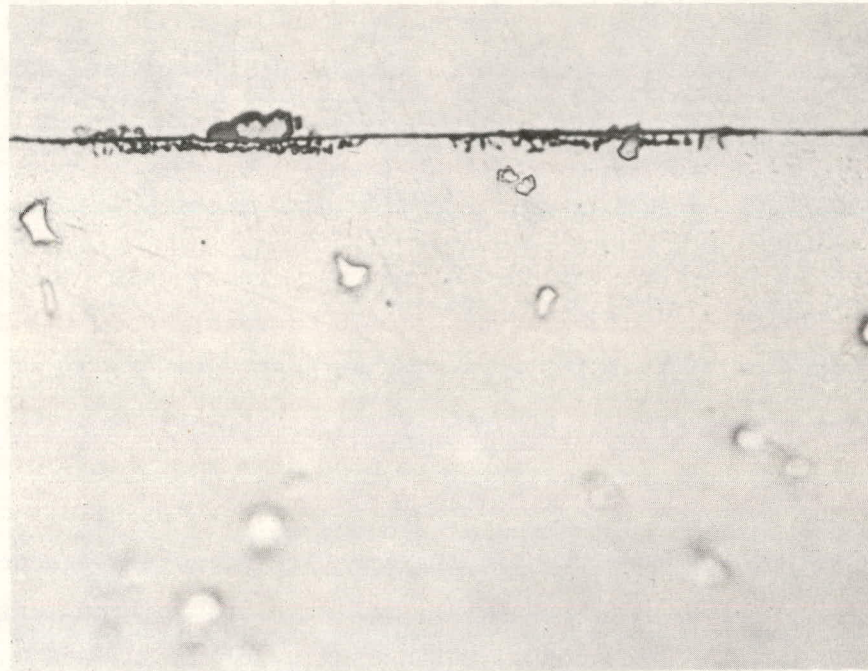
Optical photomicrographs of MM-0011 conventionally cast (CC) and MM-0011 directionally solidified (DS) exposed for 1000 hours at 750°C (1382°F) and for 1104 hours at 850°C (1562°F) are shown in Figures 2.33 and 2.34. The internal structures of both product forms were unaltered by exposure. The surface features were similar for both product forms. At 750°C (1382°F) a thin discontinuous scale formed and there is evidence of slight subsurface oxidation. Similar, but less drastic features are found after exposure at 850°C (1562°F). At both temperatures, the gamma prime structure near the surface has been altered by exposure. Dissolution of the Al and Ti contained in these precipitates probably contributes substantially to surface and subsurface scale formation.

Rene' 80

Optical photomicrographs of Rene' 80 exposed for 1000 hours at 750°C (1382°F) and for 1104 hours at 850°C (1562°F) are shown in Figures 2.35 and 2.36. Surface attack is more prevalent at 750°C (1382°F) than at the higher temperature. A substantial surface scale has formed, and evidence of internal oxidation also is seen. In Figure 2.36, it is apparent that internal grain boundary attack is more advanced at 750°C (1382°F) than at 850°C (1562°F). Again, a dissolution of gamma prime near the exposed surface is found at both exposure conditions.

Rene' 100

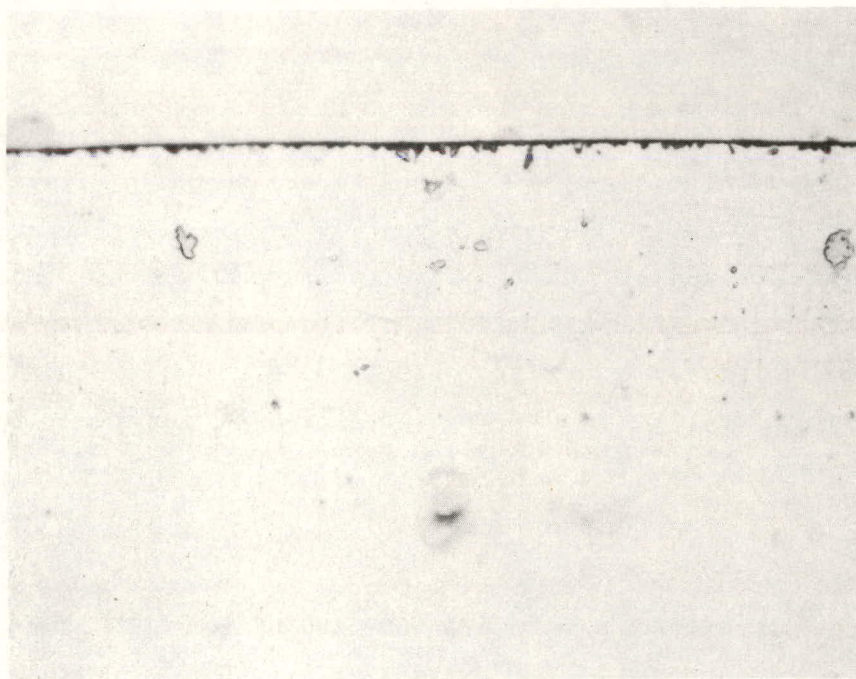
Optical photomicrographs of Rene' 100 exposed for 1000 hours at 750°C (1382°F) and for 1104 hours at 850°C (1562°F) are shown in Figure 2.37. No change in the internal phase structure during exposure at either temperature was detectable by optical metallography. Discontinuous surface and subsurface formation was found at both temperatures. The attack was slightly more severe at 750°C (1382°F).



(a)

As-Polished

1000X



(b)

As-Polished

1000X

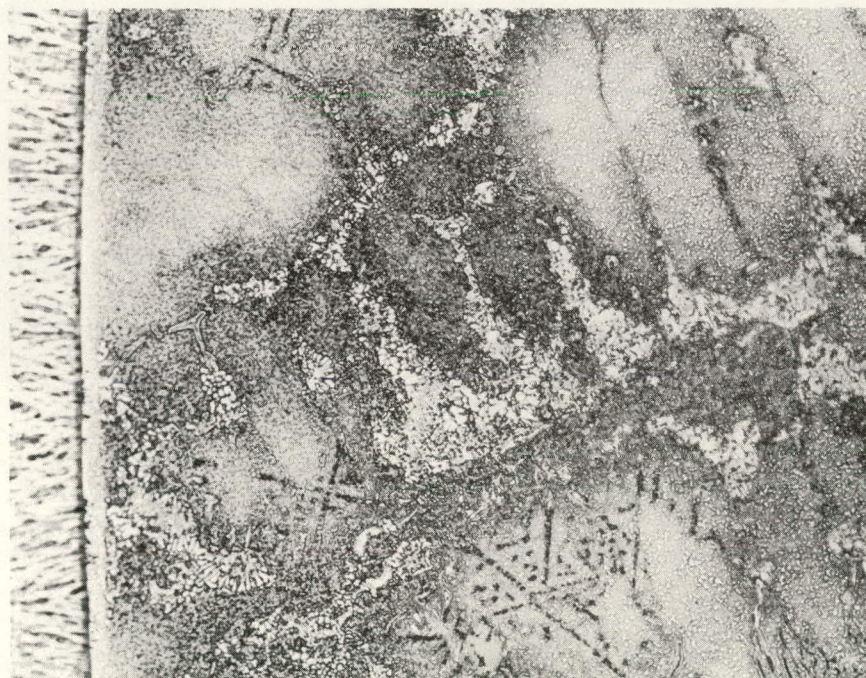
Figure 2.33 Optical Photomicrographs of MM-0011 Conventionally Cast Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

2-2-1 Etch

250X

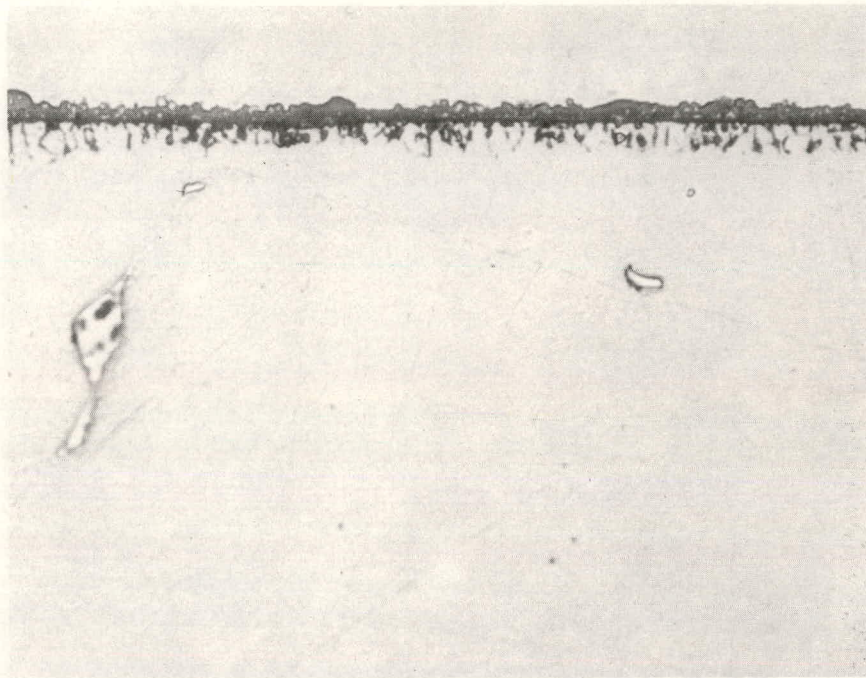


(b)

2-2-1 Etch

250X

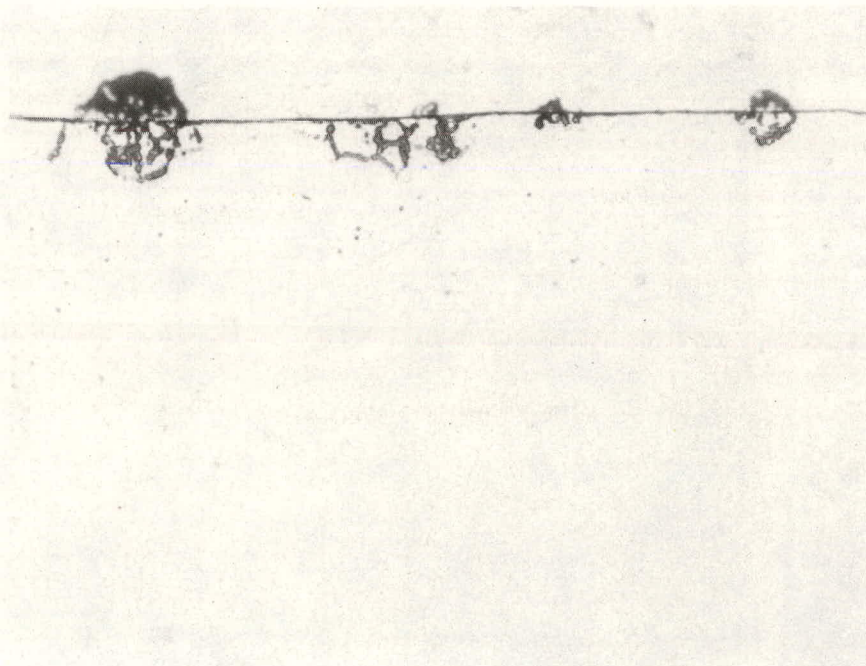
Figure 2.34 Optical Photomicrographs of MM-0011 Directionally Solidified Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

As-Polished

1000X

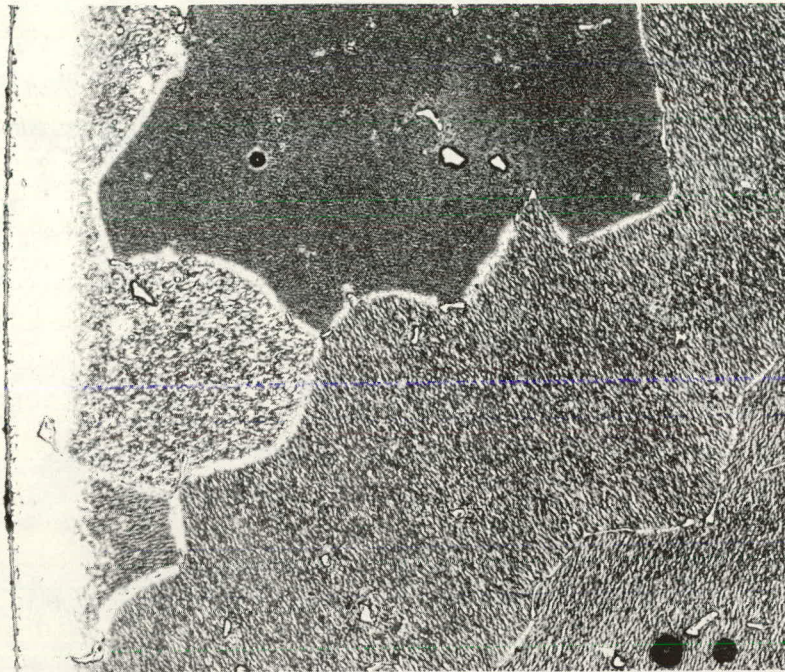


(b)

As-Polished

1000X

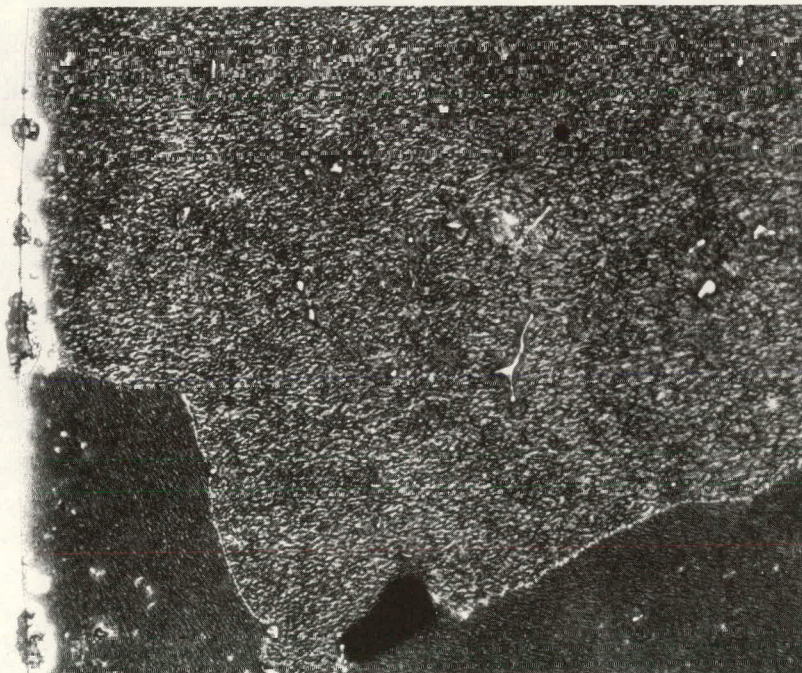
Figure 2.35 Optical Photomicrographs of Rene' 80 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

Phosphoric Etch

250X

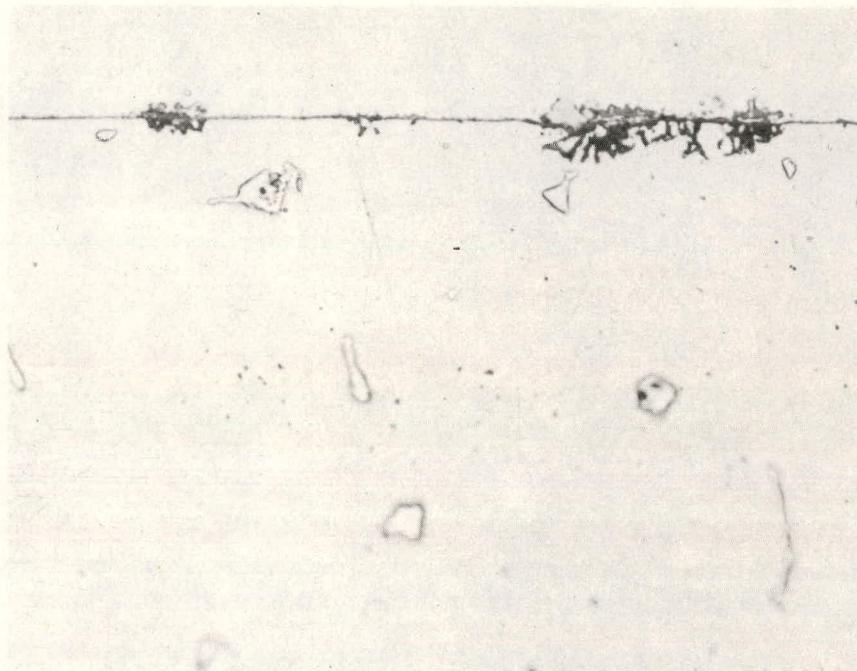


(b)

Phosphoric Etch

250X

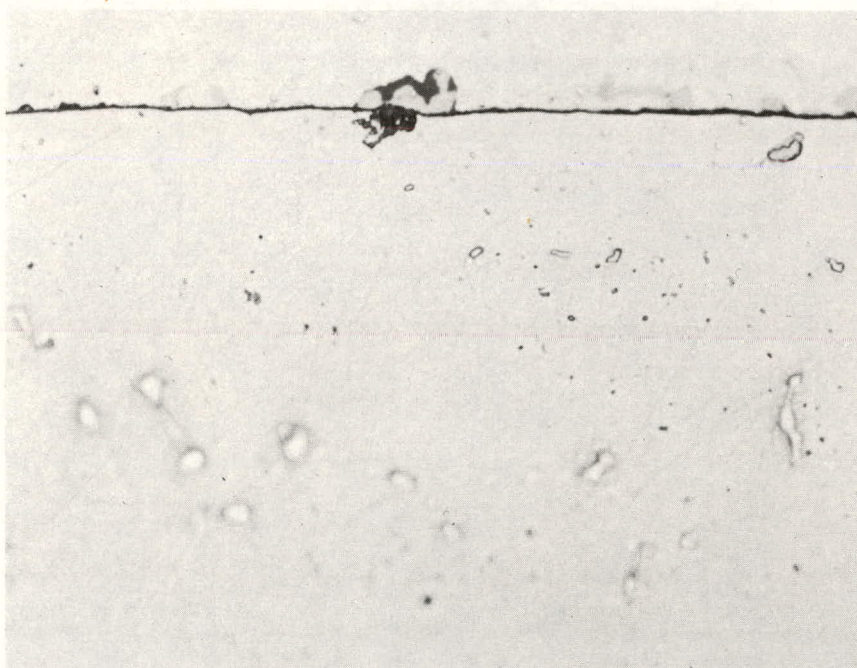
Figure 2.36 Optical Photomicrographs of Rene' 80 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

As-Polished

1000X



(b)

As-Polished

1000X

Figure 2.37 Optical Photomicrographs of Rene' 100 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.

Alloy 713LC

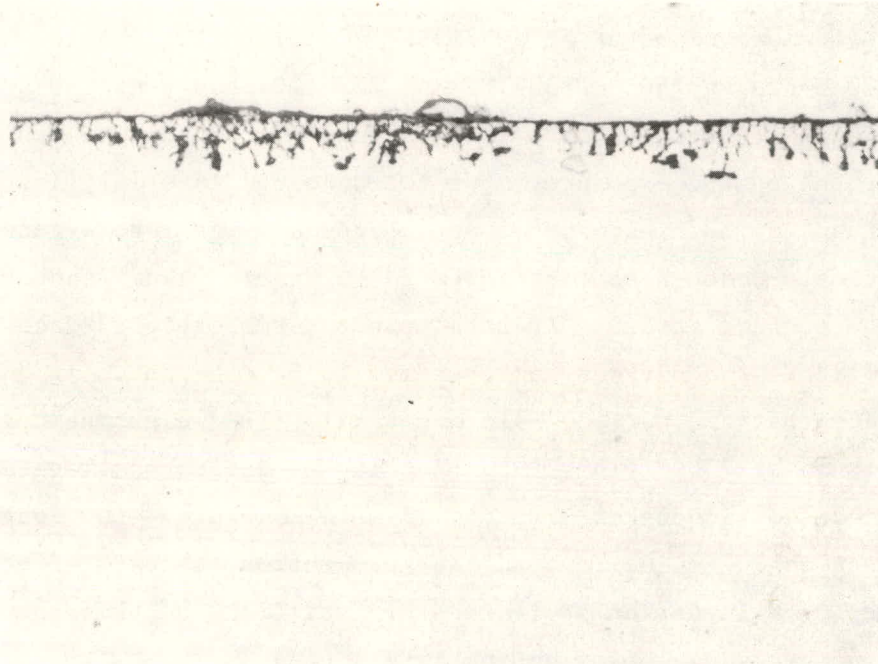
Optical photomicrographs of Alloy 713LC and Alloy 713LC coated with Codep B exposed for 1000 hours at 750°C (1382°F) and for 1104 hours at 850°C (1562°F) are shown in Figures 2.38 and 2.39. The internal microstructure was not noticeably affected by the exposure. For the uncoated material, internal oxidation was found at both exposure temperatures and was more severe at the higher temperature. Codep B coating appeared to have a significant beneficial influence on the surface attack. At both temperatures, thin surface scales formed and at 750°C (1382°F) the surface scale seemed to be coherent. At 850°C (1562°F), there is evidence that the surface film may spall. The weight gain is lower at 850°C (1562°F) than at 750°C (1382°F), which may be due to spalling and the surface film is discontinuous as observed in metallography. (Of course, this latter phenomenon could be due to specimen preparation, despite efforts to prevent such occurrences.) Evidence of spallation was not found on this specimen during routine post exposure visual examination.

It also is clear that the coating thickness has increased during exposure. This can be seen by comparing Figures 2.39 (a) and (b). The Codep B coating for Alloy 713LC was an 8 hour anneal at 1050°C (1922°F) in a 4% Al pack.

IN-738

Optical photomicrographs of IN-738 exposed in controlled-purity He for 1000 hours at 750°C (1382°F) and for 1104 hours at 850°C (1562°F) are shown in Figures 2.40 and 2.41. The internal morphology was not noticeably affected by aging at either temperature. A surface scale and internal oxide were observed at both exposure temperatures. The surface scale covered the specimen surface at 750°C (1382°F), but was found only intermittently along the surface at 850°C (1562°F). This observation is borne out by the relative weight gains, 0.647 Mg/cm² at 750°C (1382°F) versus 0.168 Mg/cm² at 850°C (1562°F).

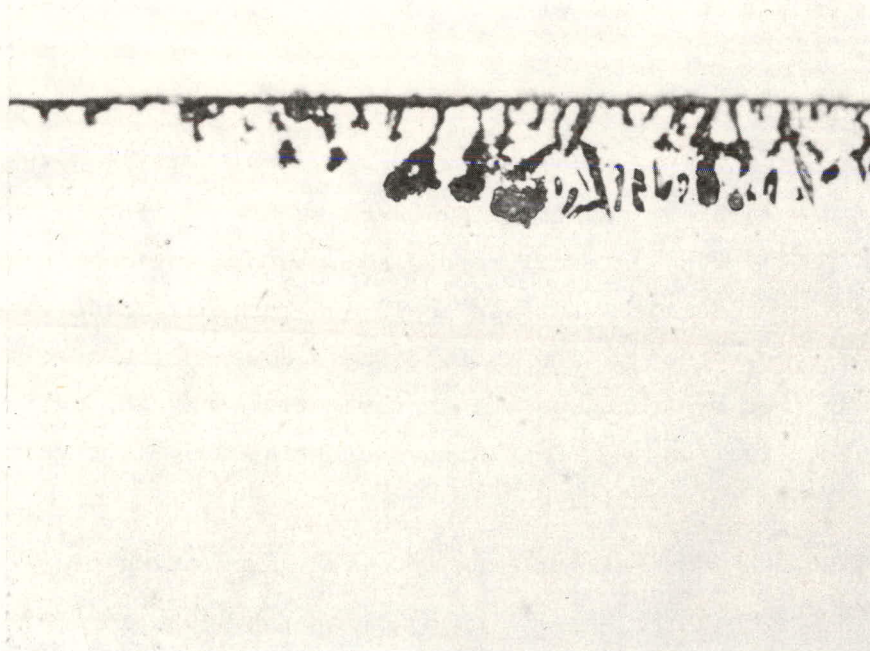
As with the other superalloys examined, the gamma prime near the exposed surface has been resolutioned and the Al and Ti probably contribute heavily to the scale formation and subsurface effects. There is some inconclusive evidence that the grain boundaries near the exposed surfaces have a higher carbide content than the bulk material. This phenomenon will have to be checked when longer exposure times are available.



(a)

As-Polished

1000X

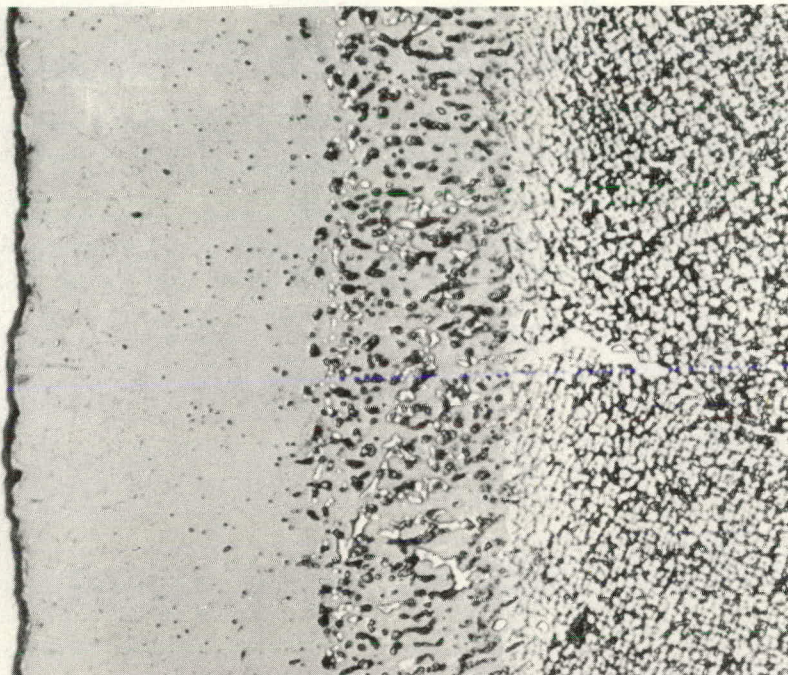


(b)

As-Polished

1000X

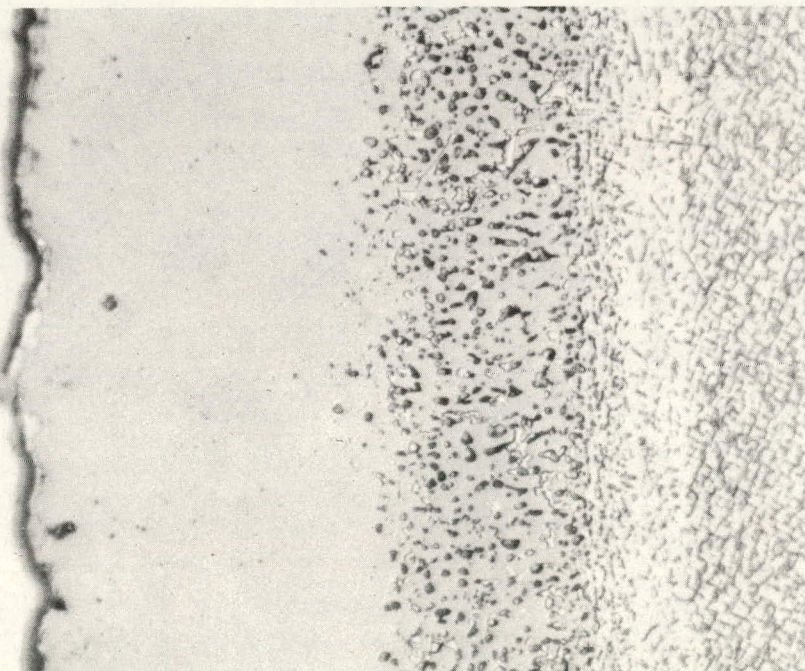
Figure 2.38 Optical Photomicrographs of Alloy 713LC Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

Phosphoric Etch

1000X

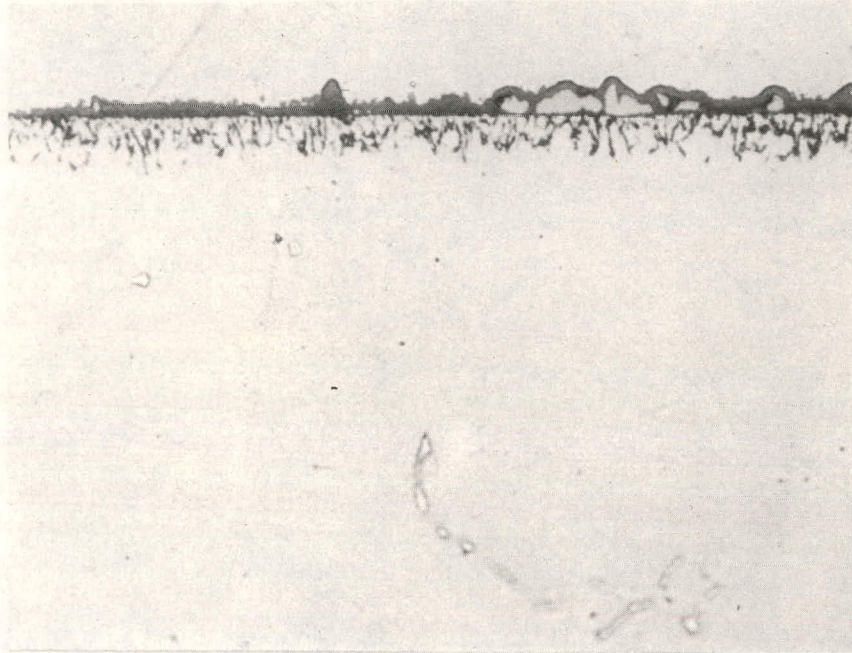


(b)

Phosphoric Etch

1000X

Figure 2.39 Optical Photomicrographs of Codep B-coated Alloy 713LC Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

As-Polished

1000X

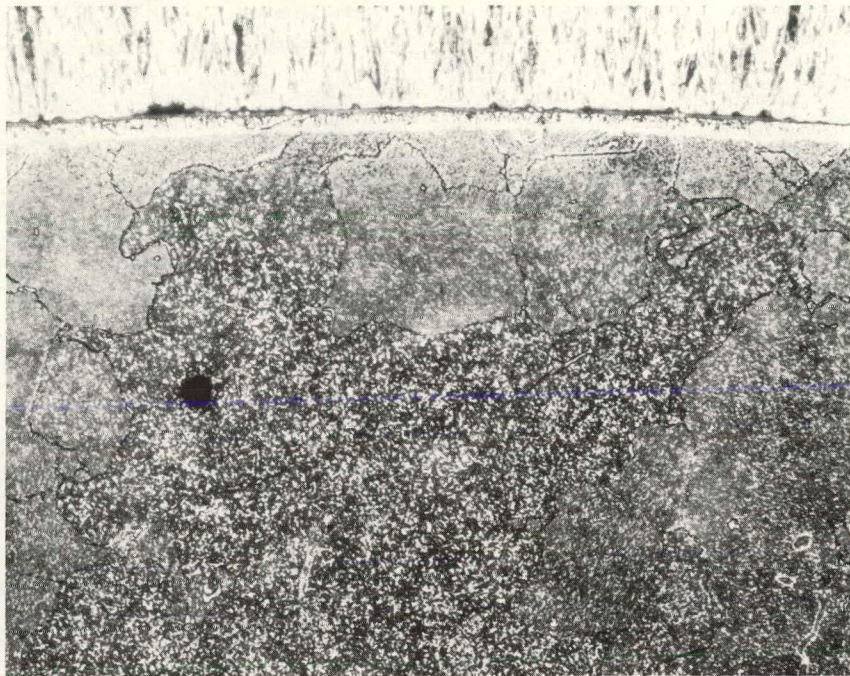


(b)

As-Polished

1000X

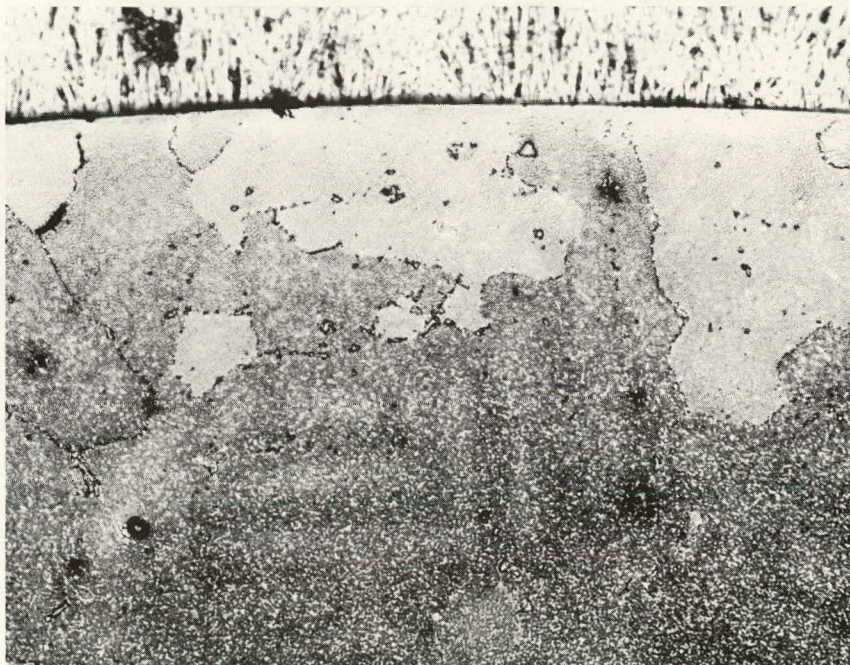
Figure 2.40 Optical Photomicrographs of IN-738 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

2-2-1 Etch

250X



(b)

2-2-1 Etch

250X

Figure 2.41 Optical Photomicrographs of IN-738 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.

M-21

Optical photomicrographs of M-21 exposed in controlled purity He for 1000 hours at 750°C (1382°F) and for 1104 hours at 850°C (1562°F) are shown in Figure 2.42. In both cases, severe internal oxidation was found. Again, this scale is formed largely at the expense of the Al in the gamma prime particles near the exposed surface, (M-21 contains no Ti.)

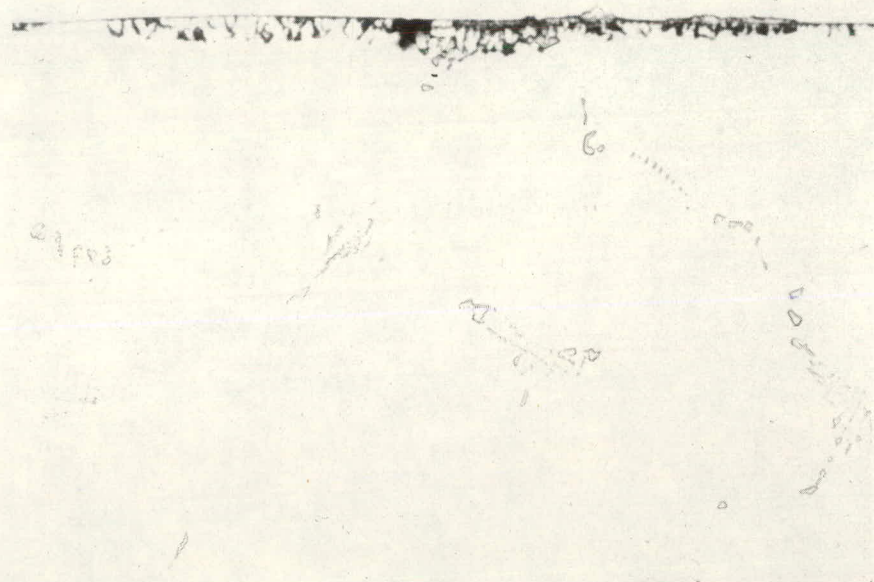
NX-188

Optical photomicrographs of NX-188 exposed in controlled-purity He for 1000 hours at 750°C (1382°F) and for 1104 hours at 850°C (1562°F) are shown in Figure 2.43. A large amount of subsurface attack has taken place, presumably internal oxidation of Al. This phenomenon is evident at both exposure temperatures. The role of the large amount of Mo in the alloy, 18.0 weight per cent, in the scale formation is presently uncertain. As with the other gamma prime strengthened alloys, that phase has redissolved in the subsurface region in NX-188. Of course, NX-188 has a gamma prime matrix with gamma second phase.

Udimet 720

Optical photomicrographs of Udimet 720 exposed for 1000 hours at 750°C (1382°F) and for 1104 hours at 850°C (1562°F) are shown in Figures 2.44 and 2.45. Udimet 720 is the only wrought Ni base superalloy under investigation in the program. After exposure at 750°C (1382°F), a continuous duplex surface scale was formed and internal oxidation was also evident. At 850°C (1562°F) a discontinuous surface scale was formed and some subsurface attack had taken place. Again, dissolution of gamma prime precipitate near the exposed surface was found.

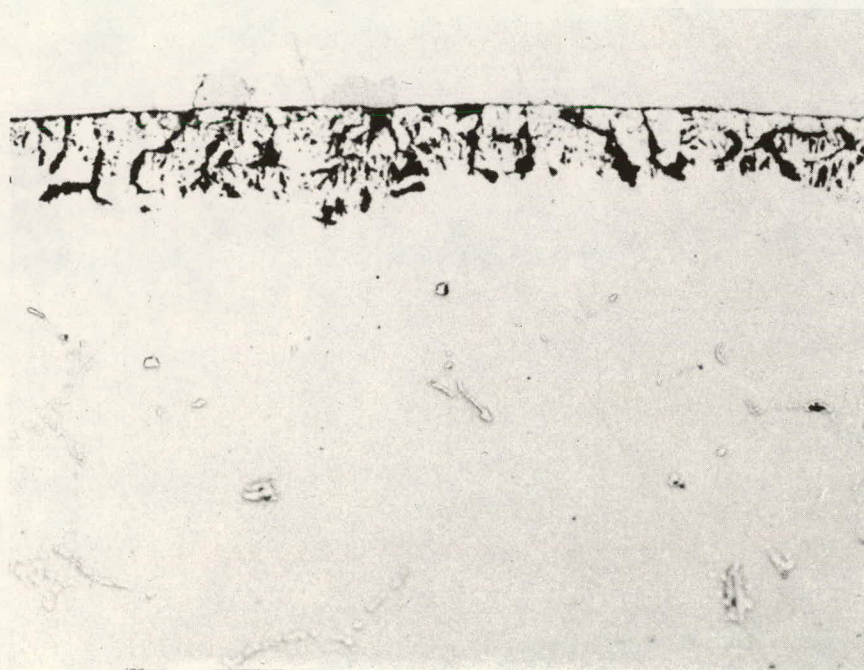
The bulk microstructure was altered by aging, primarily at 850°C (1562°F). The grains and twin boundaries were more heavily decorated with carbides after 850°C (1562°F) exposure. In addition, a new phase had precipitated in a Widmanstätten pattern, similar to some observations of the sigma phase or other TCP phases. These small needle-like precipitates are evident in Figure 2.45 (b).



(a)

As-Polished

500X

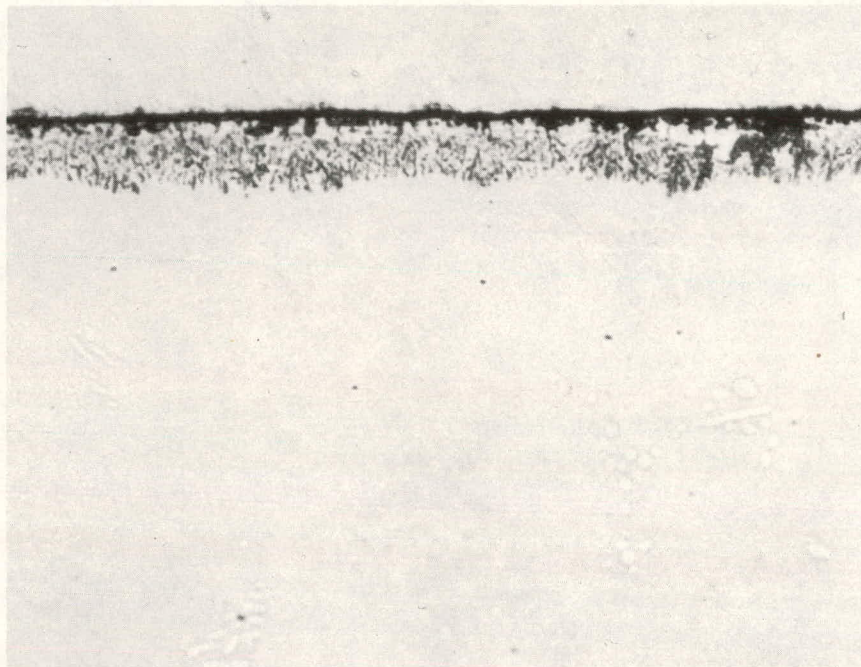


(b)

As-Polished

500X

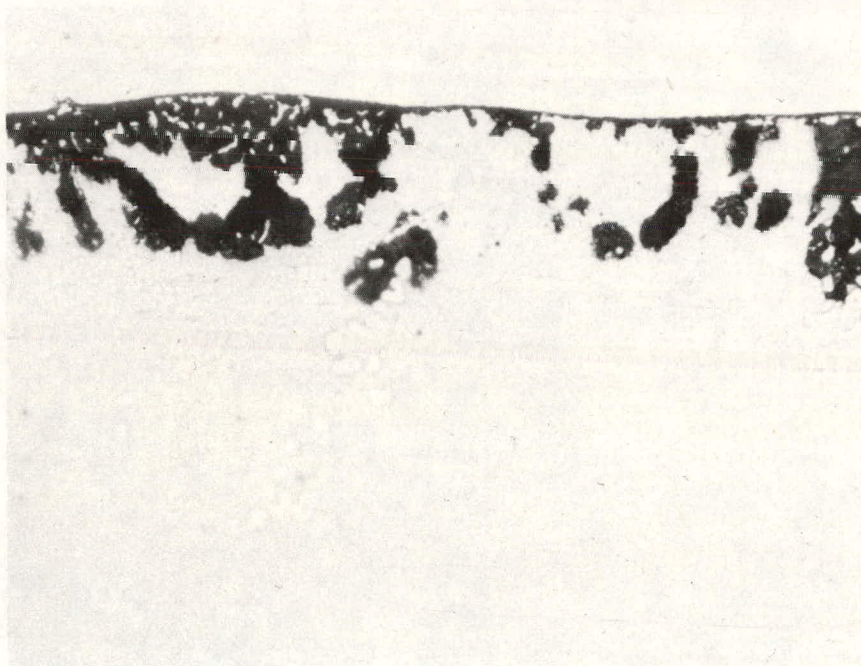
Figure 2.42 Optical Photomicrographs of M-21 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

As-Polished

1000X

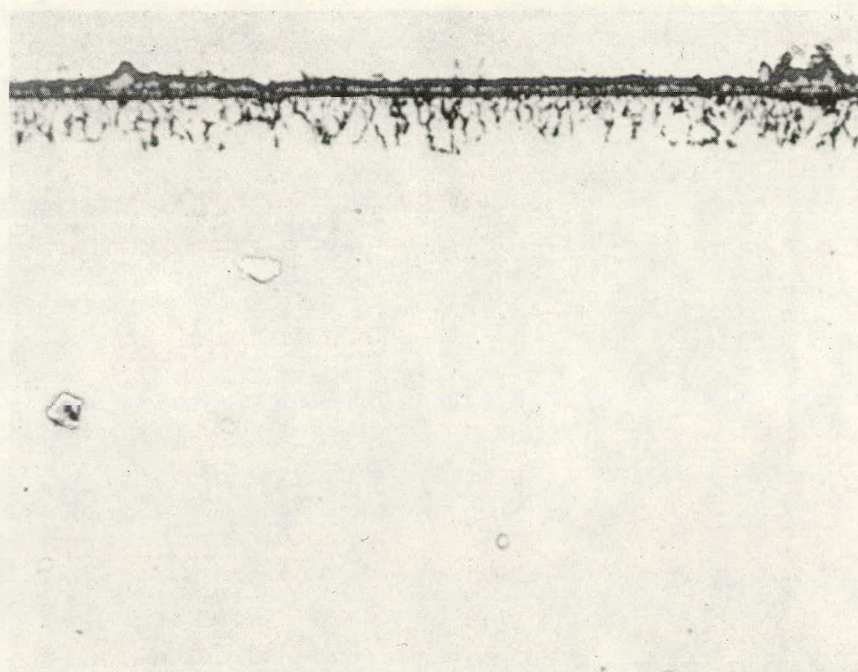


(b)

As-Polished

1000X

Figure 2.43 Optical Photomicrographs of NX-188 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

As-Polished

1000X

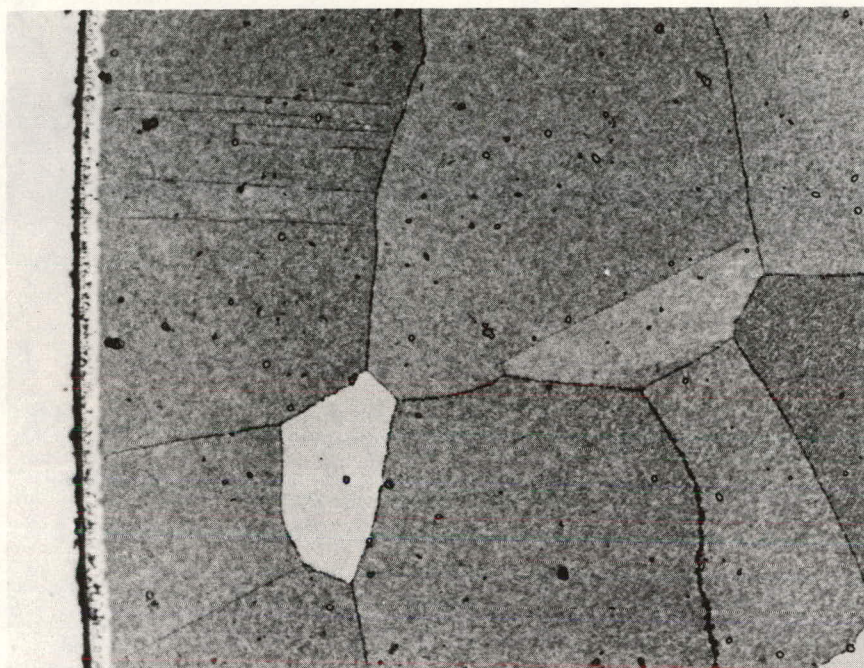


(b)

As-Polished

1000X

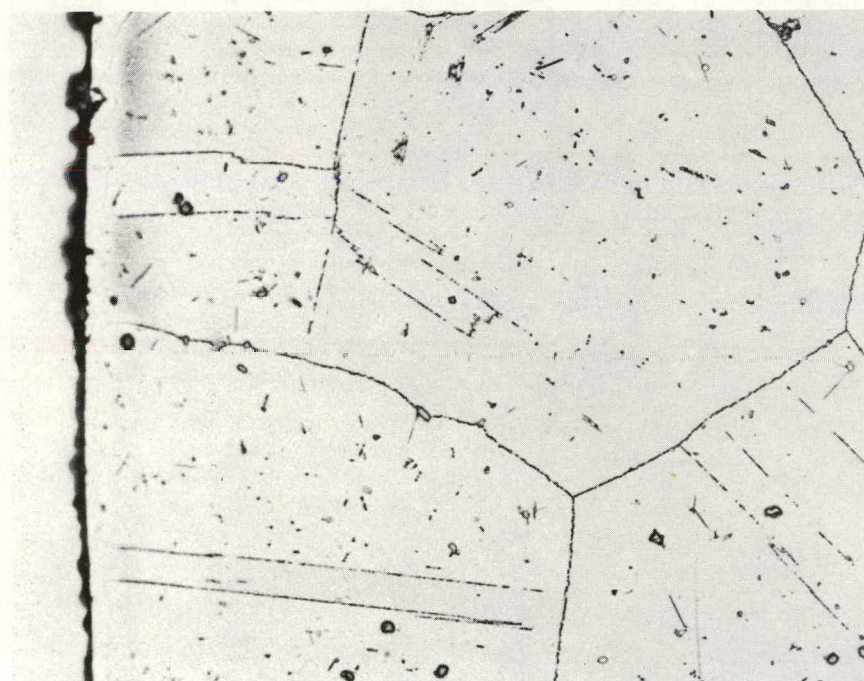
Figure 2.44 Optical Photomicrographs of Udimet 720 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.



(a)

75-25-10 Etch

250X



(b)

75-25-10 Etch

250X

Figure 2.45 Optical Photomicrographs of Udimet 720 Exposed in Controlled Purity He for (a) 1000 Hours at 750°C and (b) 1104 Hours at 850°C.

2.7 TASK 9 - DATA MANAGEMENT AND PROGRAM MANAGEMENT

2.7.1 Data Management

The Database Definition File was completed and successfully compiled on the MARK III computer system by mid-February. Work is continuing on the Preprocessor Program (PPP) which restructures the encoded data for delivery to the database.

2.7.2 Program Management

The following program meetings were attended or conducted by program staff during the report period.

- A technical review meeting was held at General Electric in Schenectady, N.Y. with Brookhaven National Laboratory personnel on January 9, 1980 and at BNL on January 28, 1980 to review gas chemistry control procedures.
- Two program review meetings were held for General Electric management during the reporting period: one (1) on February 8, 1980 and one (1) on February 29, 1980.
- An HTGR materials program coordination meeting was held in La Jolla, CA on February 20 and 21, 1980. The meeting was attended by program participants representing General Atomic Company (GAC), Oak Ridge National Laboratory (ORNL), Gas Cooled Reactor Associates (GCRA), and General Electric Company (GE).

In addition, weekly meetings were held with the General Electric program personnel in Schenectady, N.Y. to review technical results and program status.

The Program Master Schedule is presented in Figure 2.46.

Section 3.0.

WORK TO BE COMPLETED DURING THE NEXT QUARTER

3.0 WORK TO BE COMPLETED DURING THE NEXT QUARTER

- The 1,000- and 3,000-hour aging/corrosion tests at 950°C (1742°F) will be completed.
- The multispecimen creep tests at 750°C and 850°C (1382 and 1562°F) will reach 4,000 hours on test and the fourth 1,000-hour interval creep strain measurements will be completed on those specimens.
- The screening creep tests on TZM in the precision creep stands will reach 7,000 hours of 10,000 hours for the 850°C and 950°C (1562 and 1742°F) tests and 6,000 hours of 10,000 hours for the 1050°C (1922°F) test. All other tests are complete except for the 3,000-hour 1050°C (1922°F) test which will reach 1,000 hours on test.
- The fatigue test facility and gas supply system will be constructed and commissioned.
- The construction and checkout of Loop No. 2 and the tie-in of the remaining precision creep-rupture stands will be completed.
- Intensive screening creep-rupture testing in controlled purity He will be started.
- The direct cycle helium turbine (DCHT) alloys for intensive screening testing will be received from procurement, heat treated and machined.
- The preprocessor program for the data management system will be completed and the loading of test data into the data base file will be initiated.