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HIGH-TEMPERATURE GAS-COOLED REACTOR HELIUM COMPATIBILITY STUDIES

**RESULTS OF 10,000-HOUR EXPOSURE
OF SELECTED ALLOYS IN SIMULATED
REACTOR HELIUM**

by

T. A. LECHTENBERG, R. D. STEVENSON, and W. R. JOHNSON

**Prepared under
Contract DE-AT03-ET35300
for the San Francisco Operations Office
Department of Energy**

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ABSTRACT

The high-temperature gas-cooled reactor (HTGR) Helium Compatibility Task under the Department of Energy (DOE)-funded HTGR Generic Technology Program is being conducted to study the effects of long-term exposure in a controlled-impurity helium environment on the corrosion behavior and mechanical properties of candidate advanced-HTGR structural materials. This task is a continuation of the Gas-Turbine (GT) and Advanced-HTGR Materials Screening Test Program initiated in FY-75 and involves environmental creep and corrosion testing of candidate materials at the Central Institute for Industrial Research (CIIR) in Oslo, Norway, followed by detailed metallurgical analysis and evaluation of specimens at General Atomic Company (GA).

Work on the HTGR Helium Compatibility Task accomplished during the period March 31, 1977 through September 30, 1979 is documented in this report. Emphasis is on the results and analyses of creep data to 10,000 h and the detailed metallurgical evaluations performed on candidate alloy specimens tested for up to 10,000 h. Long-term creep and unstressed aging data in controlled-impurity helium and in air at 800°, 900°, and 1000°C are reported for alloys included in the program in FY-76, including the wrought solid-solution-strengthened alloys, Hastelloy X, Hastelloy S, RA 333, and HD 556, and the centrifugally cast austenitic alloys, HK 40, Supertherm, Manaurite 36X, Manaurite 36XS, and Manaurite 900.

The results of detailed metallurgical analysis on the nine alloys indicated that interactions with impurities in the helium affected both the surface and internal microstructure to varying degrees in all of the alloys during exposure at 800° to 1000°C. Both oxidation and internal

carburization occurred, the latter being much more severe. The extent of oxidation and carburization generally increased with temperature and time, with rates after 10,000 h generally being significantly lower than those after 3000 h. Environmentally independent changes in microstructure caused by thermal aging also occurred in increasing amounts with increasing temperature and time.

Surface scale formation, accompanied by surface alloy depletion, occurred in all nine alloys after exposure in controlled-impurity helium at 800°, 900°, and 1000°C. In addition, internal oxidation occurred in most of the wrought alloys. Corrosion scales varied from single-layered oxides to double- and triple-layered mixed oxides and carbides.

The alloys HK 40, Supertherm, and Manaurite 36X exhibited the best carburization resistance of all of the alloys analyzed with respect to relative or total carbon content increase. Their resistance is believed to be due, in part, to their relatively high initial bulk carbon content as compared to the other alloys. Alloys Hastelloy X, Hastelloy S, RA 333, HD 446, Manaurite 900, and, to a lesser extent, Manaurite 36XS, carburized severely at 900° and 1000°C in the dry helium environment investigated. Carburization was much less severe at 800°C for all of these alloys except Hastelloy S, which exhibited significant carbon pickup at this temperature.

Within experimental scatter and for test times to 10,000 h, all of the alloys except RA 333 and Hastelloy S exhibited no reduction in creep strength in controlled-impurity helium compared to tests in air at 800° and 900°C. Controlled-impurity helium appeared to increase creep rates for alloy RA 333 and decrease creep rates for alloy Hastelloy S at 800° and 900°C, and, in addition, decrease creep ductility for alloy RA 333 at 900°C. These results are attributed to carburization-induced microstructural changes in the alloys such as: (1) increased precipitation of internal carbides, which can strengthen or weaken a material

depending on their size and distribution, and (2) depletion of solid solution strengtheners adjacent to precipitated carbides.

Impact properties for all the alloys were reduced from as-received values after aging in controlled-impurity helium or air at 800° and 900°C. Values for all alloys after aging at 800°C were substantially the same for both air- and helium-aged specimens.

Yield and ultimate tensile strengths for 3000-h air- and helium-aged specimens at 800° and 900°C were comparable for all alloys, indicating no effect of the helium environment on these properties. Plastic strains to failure for the cast alloys at 800° and 900°C and for the wrought alloys at 800°C were comparable for both air- and helium-aged specimens, while ductilities of the wrought alloys were substantially reduced from air values by aging in helium at 900°C, the reductions being attributed to carburization.

Residual ultimate tensile strength and ductility for all of the alloys were reduced after high-temperature creep at 800° and 900°C compared to that obtained for unstressed and helium-aged specimens. The largest losses observed occurred for those alloys which contained significant amounts of creep strain prior to room-temperature testing.

CONTENTS

ABSTRACT	iii
1. INTRODUCTION	1-1
1.1. Program Description and Purpose	1-1
1.2. Background	1-4
References	1-7
2. MATERIALS AND EXPERIMENTAL PROCEDURES	2-1
2.1. Test Materials and Specimens	2-1
2.2. Test Equipment and Procedures	2-4
2.2.1. Creep-Corrosion Testing	2-4
2.2.2. Metallurgical Evaluation of Specimens	2-9
2.2.3. Property Testing	2-9
References	2-11
3. RESULTS	3-1
3.1. Creep Test Data	3-1
3.2. Weight Change Data	3-12
3.3. Bulk Carbon Analyses	3-24
3.4. Room Temperature Impact and Tensile Properties	3-28
3.4.1. Instrumented Impact Test Results	3-28
3.4.2. Tensile Test Results	3-33
3.5. Residual Tensile Properties	3-38
3.6. Metallurgical Evaluation Results	3-43
3.6.1. Centrifugally Cast Austenitic Alloys	3-43
3.6.2. Wrought Solid-Solution-Strengthened Alloys	3-71
3.6.3. Summary of Oxidation, Corrosion, and Carburization Depth Results	3-90
References	3-90
4. DISCUSSION OF RESULTS	4-1
4.1. Thermal Aging and Environmental Effects	4-1

4.1.1. Thermal Aging	4-1
4.1.2. Helium Impurity - Metal Interactions	4-2
4.2. Effect of Environmentally Affected Zone and Bulk Microstructures on the Mechanical Properties of Group II Alloys	4-14
References	4-19
5. CONCLUSIONS	5-1
5.1. General	5-1
5.2. Thermal Aging	5-1
5.3. Carburization	5-2
5.4. Surface Scale Formation	5-3
5.5. Mechanical Properties	5-3
5.5.1. Creep	5-3
5.5.2. Room-Temperature Impact and Tensile Properties . .	5-4
5.6. Residual Room-Temperature Tensile Properties After High- Temperature Creep	5-4
6. ACKNOWLEDGMENTS	6-1
APPENDIX A: COMPLETE CONTROLLED-IMPURITY HELIUM AND AIR CREEP DATA FOR GROUP II ALLOYS	A-1
APPENDIX B: COMPLETE WEIGHT CHANGE DATA FOR GROUP II ALLOYS	B-1
APPENDIX C: COMPLETE BULK CARBON ANALYSES FOR GROUP II ALLOYS . . .	C-1
APPENDIX D: SEM/EDX RESULTS FOR GROUP II ALLOYS EXPOSED IN CONTROLLED-IMPURITY HELIUM FOR 10,000 HOURS	D-1

FIGURES

2-1. Creep screening test sequence	2-8
3-1. Creep curves for alloys Hastelloy X and RA 333 in air and controlled-impurity helium at 800°C	3-6
3-2. Creep curves for alloys Hastelloy S and HD 556 in air and controlled-impurity helium at 800°C	3-7
3-3. Creep curves for alloys Hastelloy X and RA 333 in air and controlled-impurity helium at 900°C	3-8
3-4. Creep curves for alloys Hastelloy S and HD 556 in air and controlled-impurity helium at 900°C	3-9

FIGURES (Continued)

3-5.	Creep curves for alloys Hastelloy X and RA 333 in controlled-impurity helium at 1000°C	3-10
3-6.	Creep curves for alloys Hastelloy S and HD 556 in controlled-impurity helium at 1000°C	3-11
3-7.	Creep curves for alloys Manaurite 36X, Manaurite 36XS, and Manaurite 900 in air and controlled-impurity helium at 800°C	3-13
3-8.	Creep curves for alloys HK 40 and Supertherm in air and controlled-impurity helium at 800°C	3-14
3-9.	Creep curves for alloys Manaurite 36X, Manaurite 36XS, and Manaurite 900 in air and controlled-impurity helium at 900°C	3-15
3-10.	Creep curves for alloys HK 40 and Supertherm in air and controlled-impurity helium at 900°C	3-16
3-11.	Creep curves for alloys Manaurite 36X, Manaurite 36XS, and Manaurite 900 in controlled-impurity helium at 1000°C	3-17
3-12.	Creep curves for alloys HK 40 and Supertherm in controlled-impurity helium at 1000°C	3-18
3-13.	Weight change of Group II alloys exposed unstressed in controlled-impurity helium at 800°C	3-21
3-14.	Weight change of Group II alloys exposed unstressed in controlled-impurity helium at 900°C	3-22
3-15.	Weight change of Group II alloys exposed unstressed in controlled-impurity helium at 1000°C	3-23
3-16.	Plastic strain to failure versus aging temperature for Group II alloys exposed unstressed for 3000 h in controlled-impurity helium	3-36
3-17.	Typical surface appearance of HK 40 after creep testing and thermal aging in impure helium for ~10,000 hours	3-44
3-18.	Typical surface appearance of Supertherm after creep testing and thermal aging in impure helium for ~10,000 hours	3-45
3-19.	Typical surface appearance of Manaurite 36X after creep testing and thermal aging in impure helium for ~10,000 hours	3-46
3-20.	Typical surface appearance of Manaurite 36XS after creep testing and thermal aging in impure helium for ~10,000 hours	3-47
3-21.	Typical surface appearance of Manaurite 900 after creep testing and thermal aging in impure helium for ~10,000 hours	3-48
3-22.	Typical surface appearance of Hastelloy X after creep testing and thermal aging in impure helium for ~10,000 hours	3-49

FIGURES (Continued)

3-23.	Typical surface appearance of RA 333, Hastelloy S, and HD 556 after creep testing and thermal aging in impure helium for ~10,000 hours	3-50
3-24.	Optical photomicrograph showing etched internal microstructure of alloy HK 40 in the as-received condition	3-51
3-25.	Optical photomicrographs showing internal microstructure of HK 40 after 10,000-h exposure to impure helium at 900°C and 1000°C	3-54
3-26.	Optical photomicrographs showing internal microstructure of HK 40 after 3000-h and 10,000-h exposure in impure helium at 1000°C	3-55
3-27.	Optical photomicrographs showing internal microstructure of Supertherm in the as-cast condition and after 10,000-h exposure to impure helium at 800°C	3-58
3-28.	Optical photomicrographs showing internal microstructure of Supertherm after 10,000-h exposure to impure helium at 900°C and 1000°C	3-59
3-29.	Optical photomicrographs showing internal microstructure of Manaurite 36X after 7985-h exposure to impure helium at 1000°C	3-62
3-30.	Optical photomicrographs showing internal microstructure of Manaurite 36XS in the as-cast condition and after 10,000-h exposure to impure helium at 1000°C	3-66
3-31.	Optical photomicrograph (DIC) showing near surface region including layered scale, alloy depletion zone, carburization zone and internal microstructure after 10,000-h exposure in impure helium at 900°C	3-67
3-32.	Optical photomicrographs showing as-cast microstructure of Manaurite 900 and microstructure after 7075-h exposure in impure helium at 1000°C	3-70
3-33.	Optical photomicrograph of Manaurite 900 scale after 7075-h exposure in impure helium at 1000°C	3-72
3-34.	Optical photomicrographs showing internal microstructure of Hastelloy X in the as-received condition and after 10,000-h exposure in impure helium at 800°C	3-75
3-35.	Optical photomicrographs of internal microstructure of Hastelloy X after 10,000-h exposure in impure helium at 900°C and 1000°C	3-76
3-36.	Optical photomicrographs of Hastelloy S in the as-received condition and after 10,000-h exposure in impure helium at 800°C	3-79

FIGURES (Continued)

3-37.	Optical photomicrographs showing internal microstructure of Hastelloy S after 10,000-h exposure in impure helium at 900° and 1000°C	3-80
3-38.	Optical photomicrographs of the internal microstructure of RA 333 in the as-received condition and after 10,000-h exposure in impure helium at 800°C	3-83
3-39.	Optical photomicrographs of the internal microstructure of RA 333 after 10,000-h exposure in impure helium at 900° and 1000°C	3-84
3-40.	Optical DIC photomicrograph of RA 333 after 10,000-h exposure in impure helium at 1000°C	3-85
3-41.	Optical photomicrographs of HD 556 showing as-received microstructure and microstructure after 10,000-h exposure in impure helium at 800°C	3-88
3-42.	Optical photomicrographs of internal microstructure of HD 556 after 10,000-h exposure in impure helium at 900° and 1000°C	3-89
4-1.	Oxidation depth as a function of time for Group II alloys exposed in controlled-impurity helium at 900°C	4-4
4-2.	Oxidation depth as a function of time for Group II alloys aged unstressed in controlled-impurity helium at 1000°C	4-5
4-3.	Corrosion depth (alloy depletion) as a function of time for Group II alloys exposed in controlled-impurity helium at 900°C	4-6
4-4.	Corrosion depth (alloy depletion) as a function of time for Group II alloys aged unstressed in controlled-impurity helium at 1000°C	4-7
4-5.	Oxidation depth as a function of temperature for Group II alloys exposed to controlled impurity helium for 10,000 h	4-8
4-6.	Corrosion depth as a function of temperature for Group II alloys exposed to controlled-impurity helium for 10,000 h	4-9
4-7.	Change in bulk carbon content versus exposure temperature for Group II alloys aged 3000 h in controlled-impurity helium at GA	4-12
4-8.	Percent change in bulk carbon content versus exposure temperature aged for 10,000 h at CIIR	4-13
4-9.	Plastic creep strain versus time for Group II alloys tested in controlled-impurity helium at 800°C	4-16
4-10.	Plastic creep strain versus time for Group II alloys tested in controlled-impurity helium at 900°C	4-17
4-11.	Plastic creep strain versus time for Group II alloys tested in controlled-impurity helium at 1000°C	4-18

FIGURES (Continued)

D-1.	SEM photomicrograph showing the surface scales and subscale microstructure of alloy HK 40 after an exposure of 9675 h at 800°C or 9470 h at 900°C in a controlled-impurity helium environment	D-3
D-2.	SEM photomicrograph showing surface scales and subsurface microstructure of HK 40 after an exposure of 7985 h at 1000°C in a controlled-impurity helium environment	D-6
D-3.	SEM photomicrographs showing the surface scales and subsurface microstructures of Supertherm after an exposure of 9675 h at 800°C and 9470 h at 900°C in a controlled-impurity helium environment	D-8
D-4.	SEM photomicrograph showing scale and substructure of Supertherm after an exposure of 7985 h at 1000°C in a controlled-impurity helium environment	D-10
D-5.	SEM photomicrograph of Manaurite 36X after an exposure of 9675 h at 800°C in a controlled-impurity helium environment .	D-12
D-6.	SEM photomicrograph of Manaurite 36X after an exposure of 9470 h at 900°C in a controlled-impurity helium environment .	D-14
D-7.	SEM photomicrograph of Manaurite 36X after an exposure of 7985 h at 1000°C in a controlled impurity helium environment	D-16
D-8.	SEM photomicrographs of Manaurite 36XS after an exposure of 9470 h at 900°C or 9005 h at 1000°C in a controlled-impurity helium environment	D-18
D-9.	SEM photomicrograph of Manaurite 900 after an exposure of 9400 h at 900°C in a controlled-impurity helium environment	D-21
D-10.	SEM photomicrographs of Hastelloy X after an exposure of 10,000 h at 800°C and 10,000 h at 900°C in a controlled-impurity helium environment	D-23
D-11.	SEM photomicrograph of Hastelloy S after an exposure of 9935 h at 1000°C in a controlled-impurity helium environment	D-26
D-12.	SEM photomicrograph of RA 333 after an exposure of 9935 h at 1000°C in a controlled-impurity helium environment	D-28
D-13.	SEM photomicrograph of HD 556 after an exposure of 10,000 h at 800°C in a controlled-impurity helium environment	D-30

TABLES

1-1.	Screening test alloys and possible applications	1-3
1-2.	GT-HTGR materials screening test helium impurity levels compared to measured gas-cooled reactor environments	1-5
2-1.	Chemical composition of alloys tested in screening program .	2-2
2-2.	Test materials processing history	2-3
2-3.	Typical helium impurity levels in CIIR loop during operational period	2-6
3-1.	Summary of creep results for Group II alloys tested in air and controlled-impurity helium at CIIR	3-2
3-2.	Summary of weight change results for Group II alloys aged unstressed in controlled-impurity helium at CIIR	3-19
3-3.	Bulk carbon content of Group II alloy specimens aged unstressed in controlled-impurity helium at CIIR	3-25
3-4.	Summary of instrumented Charpy V-notch impact test results on specimens aged unstressed in air and controlled-impurity helium for 3000 hours	3-29
3-5.	Summary of room-temperature tensile properties for specimens aged unstressed in air and controlled-impurity helium	3-34
3-6.	Room-temperature tensile properties of Group II alloys after 3000-hour exposure (stressed and unstressed) in controlled- impurity helium	3-39
3-7.	Summary of surface corrosion measurements and internal microstructural analysis for alloy HK 40 aged unstressed in controlled-impurity helium for 10,000 hours	3-53
3-8.	Summary of surface corrosion measurements and internal microstructural analysis for alloy Supertherm aged unstressed in controlled-impurity helium for 10,000 hours	3-57
3-9.	Summary of surface corrosion measurements and internal microstructural analysis for alloy Manganese 36X aged unstressed in controlled-impurity helium for 10,000 hours . .	3-61
3-10.	Summary of surface corrosion measurements and internal microstructural analysis for alloy Manganese 36XS aged unstressed in controlled-impurity helium for 10,000 hours .	3-65
3-11.	Summary of surface corrosion measurements and internal microstructural analysis for alloy Manganese 900 unstressed in controlled-impurity helium for 10,000 hours	3-69
3-12.	Summary of surface corrosion measurements and internal microstructural analysis for alloy Hastelloy X aged unstressed in controlled-impurity helium for 10,000 hours . .	3-74

TABLES (Continued)

3-13.	Summary of surface corrosion measurements and internal microstructural analysis for alloy Hastelloy S aged unstressed in controlled-impurity helium for 10,000 hours . .	3-78
3-14.	Summary of surface corrosion measurements and internal microstructural analysis for alloy RA 333 aged unstressed in controlled-impurity helium for 10,000 hours	3-82
3-15.	Summary of surface corrosion measurements and internal microstructural analysis for alloy HD 556 aged unstressed in controlled-impurity helium for 10,000 hours	3-87
3-16.	Summary of oxidation, corrosion, and carburization depth measurements from controlled-impurity helium aging tests conducted at CIIR	3-91
D-1.	HK 40, 32146, 9675 hours at 800°C	D-2
D-2.	HK 40, 32142, 9470 hours at 900°C	D-4
D-3.	HK 40, 32143, 7985 hours at 1000°C	D-5
D-4.	Supertherm, 33145, 9675 hours at 800°C	D-7
D-5.	Supertherm, 33142, 9470 hours at 900°C	D-7
D-6.	Supertherm, 33143, 7985 hours at 1000°C	D-9
D-7.	Manaurite 36X, 28146, 9675 hours at 800°C	D-11
D-8.	Manaurite 36X, 28148, 9470 hours at 900°C	D-13
D-9.	Manaurite 36X, 28144, 7985 hours at 1000°C	D-15
D-10.	Manaurite 36XS, 29147, 9470 hours at 900°C	D-17
D-11.	Manaurite 36XS, 29149, 9005 hours at 1000°C	D-19
D-12.	Manaurite 900, 30143, 9470 hours at 900°C	D-20
D-13.	Hastelloy X, 08646, 10,000 hours at 800°C	D-22
D-14.	Hastelloy X, 08648, 10,000 hours at 900°C	D-24
D-15.	Hastelloy S, 34149, 9935 hours at 1000°C	D-25
D-16.	RA 333, 31150, 9935 hours at 1000°C	D-27
D-17.	HD 556, 39145, 10,000 hours at 800°C	D-29

1. INTRODUCTION

This report describes the progress of the high-temperature gas-cooled reactor (HTGR) Helium Compatibility Studies Task of the HTGR Generic Technology Program during the period March 31, 1977 through September 30, 1979. This task is a continuation of the former Gas-Turbine (GT) and Advanced-HTGR Materials Screening Test Program and is now funded under Department of Energy (DOE) Contract DE-AT03-76ET35300.

This document summarizes the status of the experimental studies at the end of the period and presents test data and analyses, with emphasis on the metallurgical evaluation results for Group II alloy specimens tested up to 10,000 h.

1.1. PROGRAM DESCRIPTION AND PURPOSE

The Materials Screening Test Program was initiated in July 1975 as part of the overall Advanced-HTGR design and development effort. The purpose of the program is to identify and evaluate candidate structural alloys for reactor power conversion loop components. During FY-76 the study was extended to include alloys and test conditions particularly pertinent to advanced process heat HTGRs (VHTRs).

The primary direction in the study is to define the performance and stability of alloys during high-temperature exposure to simulated reactor helium coolant containing controlled amounts of impurities. Emphasis has been placed on relating the observed corrosion mechanisms to changes in pertinent structural properties and to differences in alloy composition through the results of simulated reactor environmental screening tests, detailed metallurgical diagnostics, and data analyses.

The screening program originally included 26 alloys that are candidate materials for key components of the GT-HTGR and/or the VHTR power conversion loops. The alloys are listed in Table 1-1 along with their possible GT-HTGR and VHTR applications and generic type. The group number shown in the table indicates an alloy originally included in the program (Group I) for which results have been reported (Ref. 1-1), an alloy included during FY-76 (Group II), or an alloy included during FY-77 (Group III). The emphasis in the program has been on screening high-temperature alloys for components operating between 800° and 1000°C.

The experimental program included a coordinated series of preliminary screening tests being conducted at the Central Institute for Industrial Research (CIIR) in Oslo, Norway, and at General Atomic Company (GA), followed by residual property testing, detailed metallurgical evaluation, and data analyses at GA.

The testing at CIIR represented an international collaboration between GA/DOE and the High-Temperature Materials Program (HTMP) organization at Wimborne, England. The HTMP organization was formed after the termination of the OECD Dragon Project and was jointly supported by funds from the U.S. Federal Republic of Germany (FRG), Austria, and Switzerland through December 1978.

Screening tests were conducted at GA to complement the experimental effort at CIIR. The original work at GA involved the determination of the effects of simulated reactor environments on the strength, deformation, fracture, and corrosion behavior of candidate materials. These tests, which have now been completed, included unstressed environmental aging in vacuum to simulate the evaporative behavior of the materials in high-velocity helium, in controlled-impurity helium, and in air at 800° and 900°C, followed by tensile tests, instrumented impact tests, and metallurgical evaluations. The major portion of this work has been documented (Refs. 1-1, 1-2).

TABLE 1-1
SCREENING TEST ALLOYS AND POSSIBLE APPLICATIONS

Alloy	Group	Type/Form	Potential Applications	
			Gas Turbine HTGR	VHTR
IN 100	I	Vacuum shell cast - γ' strengthened superalloys ↓	Turbine blade/vanes, primary ducts, fasteners ↓	Primary and secondary ducts, IHX hardware ↓
Rene 100	I			
IN 713	I			
MM 004	I			
M 21	I			
IN 738	I			
MoTZM	I	Wrought molybdenum base	Turbine blade/vanes, primary ducts	Primary ducts, IHX tubes and hardware
Hastelloy X (081)	I	Wrought - solid solution strengthened ↓	Primary ducts, turbine hot ducts, casings ↓	Primary and secondary ducts, IHX tubes and hardware ↓
Hastelloy X (086)	II			
Inconel 617	I			
Hastelloy S	II			
RA 333	II			
HD 556	II			
MA 753	I	Mechanically alloyed - oxide dispersion strengthened	Primary ducts, casings	Primary ducts, IXH hardware
MA 754				
Inconel 706	I	Wrought - γ' strengthened ↓	Turbine discs, fasteners ↓	Fasteners ↓
Inconel 718	I			
A 286	I			
HK 40	II	Centrifugal cast - austenitic ↓	Primary ducts ↓	PHX tubes and components, primary ducts, IHX hardware ↓
IN 519	I			
Supertherm	II			
Manaurite 36X	II			
Manaurite 36XS	II			
Manaurite 900	II			
SS 316	I	Wrought - austenitic ↓	Recuperator, ducts, casings ↓	IHX tubes and hardware, secondary ducts ↓
Incoloy 800	I			
Incoloy 800H	III			

1.2. BACKGROUND

The alloys employed in GT- and Advanced-HTGR components will be exposed under stress to high-temperature, high-velocity helium containing trace amounts of impurities for times up to 280,000 h. The primary factors determining the applicability of these alloys will be their long-time strength, thermal (metallurgical) stability, and resistance to interactions with helium impurities.

The types and quantities of helium impurities in HTGRs are determined by the ingress of impurities through leaks, diffusion, and outgassing; the reaction of impurities with each other, the core, or other loop surfaces; and the characteristics of the purification system. The steady-state helium impurity levels are also dependent on the thermodynamic equilibria and kinetics of the chemical reactions involved. The typical impurity levels in the steam-generating, helium-cooled reactors (Peach Bottom, Dragon, and AVR) are shown in Table 1-2, along with the projected levels for the GT-HTGR. The estimated levels for the GT-HTGR, which are being utilized in the materials screening program, were derived by collaborative analyses at GA and at Dragon (Ref. 1-1). The only sources of H_2O in a GT-HTGR will be outgassing of core components and fibrous ceramic insulation, and perhaps the reduction of preexisting Fe_2O_3 (rust) on ferritic reactor surfaces. The primary sources of H_2 will be reaction of desorbed H_2O with the core and protonic diffusion through the precooler tubes from H_2 produced during H_2O -Fe corrosion reactions on the low-pressure side of the ferritic heat exchanger. The H_2O -graphite core reaction will produce CO, while the primary sources of CH_4 will be from the pyrolysis of trace organic lubricants or from the reaction of H_2 with the core.

The interaction of structural materials with impurities in helium has been under investigation for over 18 years (Refs. 1-3, 1-4). These studies, as well as the more recent investigations at GA (Refs. 1-5, 1-6), indicate that the oxygen potential and the carbon activity of the

TABLE 1-2
GT-HTGR MATERIALS SCREENING TEST HELIUM IMPURITY LEVELS COMPARED
TO MEASURED GAS-COOLED REACTOR ENVIRONMENTS

Species	Composition (μ atm) (a)			
	Peach Bottom	Dragon	AVR	GT-HTGR Screening Tests
Core outlet temperature	700°C	700°C	950°C	850°C
H ₂ O	10	1	1	0.5-3
CO ₂	<1	<0.4	5	--
H ₂	230	20	30-100	500
CO	10	12	500	50
CH ₄	23	3	1-2	50
N ₂	10-20	3	50-500	--
O ₂	<1	<0.4	--	--

(a) μ atm = vol ppm x He pressure; 23, 20, 10, and 70 atm primary coolant He for Peach Bottom, Dragon, AVR, and GT-HTGR, respectively.

impure helium are the two most important factors determining its compatibility with metallic circuit materials. For the oxygen potential of the helium in the GT-HTGR, thermodynamic data indicate that the oxides of Fe, Ni, and Co will not form. The protective nature of the high-temperature scales formed on many of the candidate alloys often depends on the presence of spinel oxides (AB_2O_4) containing Fe or Ni.

Some data concerning the effects of accurately simulated reactor helium on creep or stress-rupture properties of Fe- and Ni-base alloys have been reported in Refs. 1-7 through 1-9. These data, for temperatures up to 900°C and for stresses significantly higher than allowable design levels, show no apparent effect of controlled-impurity helium on creep or rupture properties, although the rupture ductility may have been somewhat lower in a few cases. However, these tests usually involved creep strains and creep rates far greater than would be obtained in much-lower-stressed reactor components. Subtle effects of gas-metal interactions on creep properties could be masked under these high-creep-rate conditions. In the current series of GT-HTGR materials screening creep tests, realistic design-level stresses are employed, and the presence of environmental effects that might accelerate creep rates or reduce rupture ductility should be discernible.

At the time this report was prepared, the creep-corrosion screening tests on Group I alloys had been completed (Ref. 1-1). Group II alloys had accumulated ~10,000 h of exposure in controlled-impurity helium at 800°, 900°, and 1000°C. The 3000-h exposure of unstressed tensile specimens had been completed as well as the detailed metallurgical evaluation of unstressed corrosion specimens. The results and analyses of tests on Group II alloys are presented in this report and include detailed creep data for up to 10,000 h, 3000-h tensile and impact properties, weight change data, and detailed metallurgical analysis of corrosion specimens.

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2. MATERIALS AND EXPERIMENTAL PROCEDURES

The test data from the collaborative creep-corrosion screening tests on Group II alloys being conducted at CIIR and the results of the detailed posttest metallurgical evaluation of unstressed corrosion specimens removed from CIIR after 10,000 h of exposure are reported and discussed in this section. Detailed creep, weight change, carbon analysis, and metallurgical evaluation results are presented and discussed for the Group II alloys including Hastelloy X, Hastelloy S, RA 333, HD 556, HK 40, Supertherm, Manaurite 36X, Manaurite 36XS, and Manaurite 900. The detailed tensile and fracture characteristics of 3000-h Group II alloy specimens removed from creep tests at CIIR and unstressed aging tests at GA are also presented.

2.1. TEST MATERIALS AND SPECIMENS

The chemical compositions of the Group II alloys used in the screening test program are shown in Table 2-1. The GA identification number is shown for each material. The compositions listed are the result of analyses conducted by the International Nickel Company (Inco) Research Laboratories with the exception of some producer-certified values as noted in the table. The concentrations of most heavy elements were determined by X-ray fluorescence analyses, while all carbon analyses of as-received alloys and environmentally exposed samples were performed using the LECO combustion method.

The producer, heat number, as-received form, and as-received heat treatment of each test material for which data are presented in this report are given in Table 2-2.

GA Code No. (a)	Element (wt %)																					S	Other
	Alloy	C	Ni	Fe	Cr	Mn	Si	Mo	Co	W	Nb	Ta	Al	Ti	Cu	V	Zr	Y	B	f			
281	Manaurite 36X	0.47	33.3	36.4 ^(b)	25.5	1.21	1.41	<0.10	<0.10	<0.1	0.75	<0.10	<0.1	<0.1	<0.05	<0.10	<0.10	<0.10	-	0.025 ^(c)	0.021 ^(c)	--	
291	Manaurite 36XS	0.39	36.0	32.1 ^(b)	24.8	1.30	1.92	<0.10	0.18	1.5	1.05	<0.10	<0.1	<0.05	<0.10	<0.10	<0.10	<0.10	--	--	0.018 ^(c)	--	
301	Manaurite 90U	0.12	34.8	40.1 ^(b)	22.4	0.99	0.74	<0.10	<0.10	<0.1	--	--	<0.1	<0.1	<0.05	<0.1	<0.1	<0.1	--	0.0.4 ^(c)	0.018 ^(c)	-	
321	HK 40	0.44	20.5	50.9 ^(b)	25.3	0.41	1.32	<0.10	0.15	<0.1	<0.10	<0.10	<0.1	<0.1	0.05	<0.10	<0.10	<0.10	--	-	-	--	
331	Supertherm	0.43	34.9 ^(b)	19.6	22.9	0.12	1.49	<0.10	14.8	4.9	<0.10	<0.10	<0.1	<0.1	0.05	<0.10	<0.10	<0.10	--	-	-	--	
086	Hastelloy X	0.080	45.3 ^(b)	20.9	20.5	0.48	0.49	8.60	2.1	0.5	0.25	<0.10	<0.1	<0.1	0.20	<0.10	<0.10	<0.10	<0.002 ^(c)	0.023 ^(c)	0.005 ^(c)	Ca 0.012 ^(c) , Mg 0.026 ^(c) , Pb <0.001 ^(c) , Ag 0.0008 ^(c)	
311	RA 333	0.034	45.2 ^(b)	19.4	23.0	1.59	1.11	3.10	2.9	2.8	0.12	<0.10	<0.1	<0.1	0.12	<0.10	<0.10	<0.10	--	0.012 ^(c)	0.004 ^(c)	Sn 0.014 ^(c) , Pb 0.002 ^(c)	
341	Manaurite S	0.007	68.6 ^(b)	1.0	15.7	0.30	0.37	13.0	<0.10	0.2	0.12	<0.10	<0.1	<0.1	0.15	<0.10	-	-	0.012 ^(c)	0.006 ^(c)	0.006 ^(c)	Ca 0.018 ^(c) , Mg <0.010 ^(c)	
391	HD 556	0.092	20.0	29.7	21.2	1.63	0.59	3.10	19.5	3.1	0.18	<0.10	0.35	<0.1	0.15	<0.10	<0.10	<0.10	-	--	<0.005 ^(c)	N 0.15 ^(c) , La 0.015 ^(c) , Mg 0.011 ^(c)	

(a) All test specimen numbers referenced to alloy identification numbers shown in this column
(b) Balance of alloy content obtained by difference
(c) Values from producer certifications

TABLE 2-2
TEST MATERIALS PROCESSING HISTORY

Alloy	GA Code No.	Producer	Heat No.	As-Received Form	As-Received Heat Treatment
Manaurite 36X	281	Acieries Manoir Pompey	1268.0401	15.6-cm o.d. x 11.9-cm i.d. cast tube	As-cast
Manaurite 36XS	291	Acieries Manoir Pompey	1268.0726	15.6-cm o.d. x 11.9-cm i.d. cast tube	As-cast
Manaurite 900	301	Acieries Manoir Pompey	1268.0402	15.6-cm o.d. x 11.9-cm i.d. cast tube	As-cast
HK 40	321	Abex	--	13.3-cm o.d. x 8.9-cm i.d. cast tube	As-cast
Supertherm	331	Abex	--	13.3-cm o.d. x 8.9-cm i.d. cast tube	As-cast
Hastelloy X	341	Cabot	5-2831	1.27-cm-diameter round	S.A. ^(a) 1177°C
Hastelloy S	341	Cabot	3-7316	1.27-cm-diameter round	S.A. 1066°C
RA 333	311	Rolled Alloys	7090-1	1.27-cm-diameter round	S.A. 1121°C
HD 556	391	Cabot	8556-9-7304	1.27-cm-diameter round	S.A. 1177°C

(a) S.A. = solution annealed.

The two types of test specimens utilized at CIIR, tensile creep and corrosion control, are described in Ref. 2-1. The threaded creep specimens were 76.2 mm long with 6.35-mm-diameter reduced sections, and the cylindrical corrosion specimens were 25.4 mm long with a 6.35-mm diameter.

Wrought alloy specimens and centrifugally cast alloy specimens were lathe machined from bar stock and longitudinally cut blanks, respectively, resulting in a uniform surface finish of 8 to 16 rms.

All specimens were X-ray radiographed, visually inspected, weighed, and characterized with respect to surface finish prior to testing. Cast specimens were dye-penetrant inspected at the foundry. The specimens were ultrasonically cleaned for 10 min in a solvent (35% methyl ethyl ketone, 10% acetone, 15% toluene, 15% isopropyl alcohol, 5% xylene, and 20% ethyl acetate) and then rinsed in ethyl alcohol before testing.

2.2. TEST EQUIPMENT AND PROCEDURES

2.2.1. Creep-Corrosion Testing

The controlled-impurity helium rig at CIIR is fully described in Ref. 2-2. The rig is divided into a gas loop, which provides the test helium at prescribed controlled-impurity levels, and a furnace loop, which receives the controlled-impurity helium and exposes specimens under controlled temperatures and loading conditions.

The gas circuit consists of two main parts, a purification loop and a helium loop with its large manifold that supplies gas to the furnaces. In the purification loop, the commercially pure helium is purified to very low partial pressures of the impurities CO , CO_2 , H_2 , H_2O , O_2 , and N_2 .

The instrument circuit monitors and controls the impurity content of the test gas. Hydrogen is monitored by converting it to H_2O in a CuO furnace and employing a P_2O_5 Meeco Model W electrolytic H_2O analyzer (to 10 μatm). Water is monitored with a Goldsmith P_2O_5 cell with extra-fine platinum electrodes. A detection limit of $0.2 \pm 0.04 \mu atm H_2O$ is claimed. The overall loop and the outlet from each set of furnaces are monitored. A Carle Model 8000 gas chromatograph monitors CH_4 and CO , although an infrared gas analyzer (IRGA) is sometimes employed for rougher estimates.

The helium impurity levels for the first 3000 h of Group I specimen tests are presented in Ref. 2-2. The levels of CH_4 and CO were initially lower than the target values of 50 μatm . By the end of 3000 h, CH_4 partial pressure was brought to 50 μatm , but the CO partial pressure could not be increased much above 30 μatm . The H_2 and H_2O partial pressures were at or near the target values of 500 μatm and 1.5 μatm , respectively, during the first 3000 h of testing. Throughout the Group II alloy testing period of this report, the apparatus operated under the conditions indicated in Table 2-3. A typical analysis over a period of 1 month is presented in Table 2-3.

Although it has been noted that at the 1 liter/min flow rate in each furnace some depletion of species, particularly CH_4 at above $850^\circ C$, occurs as the helium travels through the furnace, no significant effect of position on oxidation/carburization of specimens has been determined, based on metallographic evaluations at HTMP.

The uninstrumented, multiple-specimen string technique, developed by the OECD Dragon Project at CIIR, is being utilized for all creep testing in this program. The designs of the creep testing furnaces and retorts are described in Ref. 2-2. Each of the five-zone, independently controlled furnaces has a 75-cm hot zone and five monitoring thermocouples. The Inconel 600 retort, which has a 7.9-cm internal

TABLE 2-3
TYPICAL HELIUM IMPURITY LEVELS IN CIIR LOOP
DURING OPERATIONAL PERIOD

Impurity	Mean Partial Pressure (μ atm)	Deviation from Mean (μ atm)
H ₂	500	± 25
H ₂ O	1.5	± 1
CH ₄	50	± 10
CO	30	± 10
CO ₂	0	0

diameter, contains three specimen strings, each individually loaded using a deadweight lever system. Ten creep specimens of the GA design can be loaded in each string. Unstressed corrosion bars are suspended from the center post.

Temperatures are monitored and controlled automatically using a Solitron analogue scanner, a Data General logger unit, and a Nova 1210 computer.

At the end of 1976 and during the first quarter of 1977, rig upgrading was carried out by CIIR. In particular, temperature controllers (which were approaching 10 years of age) were replaced with solid-state devices, and improvements were made to gas sampling and gas analysis systems. New fail-safe systems were installed to prevent damage to equipment and specimens in the event of various failures in temperature and gas conditions in the furnaces.

The creep tests at CIIR are performed in accordance with established international standards (ISO). The test sequence is detailed in Fig. 2-1. The creep test is interrupted and creep strains are measured on unloaded specimens initially at 500 h and thereafter at 1000-h intervals. The creep strains of machined alloy specimens are measured from the sharply defined shoulder edges. The creep strain measurement is made at a magnification of 20X with a Leitz microscope in conjunction with a Sony magnetic coil G50 digital gage and an MSR digital counter. Using this technique, measurements on any given specimen were shown to be repeatable to within 0.04% plastic creep strain.

The stress level selected for each Group II alloy at each temperature was approximately two-thirds of the estimated stress for rupture in 100,000 to 200,000 h, which would be typical of a design-allowable stress.

Fig. 2-1. Creep screening test sequence

Each creep specimen is tested, along with an unloaded corrosion-control sample from which weight change data are obtained at each test interruption interval. Two creep specimens and two corrosion-control specimens of each alloy were loaded initially. One specimen of each type was removed after 3000 h for metallurgical evaluation and/or tensile testing. The 3000-h data on the Group II alloys were reported in Refs. 2-1, 2-4, and 2-5. Corrosion specimens of Group II alloys were removed after 10,000 h for metallurgical evaluation.

2.2.2. Metallurgical Evaluation of Specimens

After weighing, all specimens removed from test were visually inspected to characterize their surface appearance. All corrosion specimens were cut perpendicular to their longitudinal axis to obtain sections for metallography, bulk carbon analysis, and surface studies. Metallography of etched and unetched 10,000-h specimens of Group II alloys was performed to study surface corrosion scales, internal oxidation, carburization, and thermal aging effects. Comparisons were made with as-received alloy microstructures. Mean oxidation, corrosion, and carburization depths were measured when possible by taking the average of four metallographic measurements at 90-deg increments around the specimen cross section.

Alloy phases and surface scale composition were identified by scanning electron microscope (SEM) microprobe analysis, including selected area X-ray energy dispersive (EDX) and wavelength analysis, and by electron microprobe analysis supplemented with transmission electron microscopy (TEM).

2.2.3. Property Testing

Room-temperature tensile tests and a limited number of instrumented Charpy impact tests were performed on 3000-h test samples to assess the

effect of the environment on pertinent mechanical properties. The procedures employed are discussed below.

2.2.3.1. Tensile Tests

Room-temperature tensile tests were performed using an MTS closed-loop electrohydraulic system in the load-control mode on specimens machined prior to exposure. An electronic ramp-test program was employed in conjunction with a $6.3 \times 10^{-5} \text{ m}^3/\text{s}$ (1 gpm) servovalve, which provided a loading rate of 2268 kg/min. A 25.4-mm-gage-length mechanical extensometer (MTS 632.11B20) was used to monitor strain. Load-strain curves were recorded on a chart. Ultimate strength, 0.2% yield strength, Young's modulus, percent elongation (25.4-mm gage length), and percent reduction in area were determined. Tensile tests were performed on all CIIR 3000-h creep specimens, on 3000-h unstressed 800° and 900°C helium- and air-aged specimens, and on a minimum of two as-received tensile specimens of each alloy.

2.2.3.2. Instrumented Impact Tests

The changes in both resistance to dynamic loading and fracture toughness due to aging in controlled-impurity helium and air at 800° and 900°C were determined for a selected number of Group II alloys, using instrumented impact tests. These tests, performed at Effects Technology, Inc. (ETI), Santa Barbara, California, were conducted on a standard Satec pendulum-type impact machine, equipped with "dynatup" instrumentation. The specimens were completely machined prior to exposure in the controlled-impurity helium.

Three primary components of the instrumented impact system monitor the dynamic behavior and supply an analog output signal of the load-time history of the impacted test piece. These three components are the instrumented tup located in the pendulum, an amplifier-oscilloscope for signal display, and a fiber-optic trigger mechanism.

During impact testing of the aged test specimens, the output from the load cell was recorded on the oscilloscope, producing a trace that represented the load-time history of the specimen. Load-time traces provided data on the general yield load (plastic yielding across the entire cross section of the Charpy specimen), the maximum load, the brittle fracture load, and their times of occurrence. The area under the load-time curve corresponded to the total energy absorbed, which was integrated by the electronic package to develop the energy time trace. This system permitted separation of the absorbed energy into W_I , the energy required to initiate fracture (or pre-maximum load energy) and W_p , the energy required to extend fracture (or post-maximum load energy). The sum of the two parameters is the total fracture energy W_T .

In addition to the oscilloscope record, the load signal from each of the tests was also recorded by a computer system. This unit contained a plotting accessory to reproduce a hard copy of the load-time signal. The Dyna-1 computer program developed at ETI for analyzing the results of instrumented impact tests was used to obtain additional data from the tests, including several estimates of fracture toughness, K_d , and critical crack length, C_{cr} .

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3. RESULTS

The results obtained to date from the CIIR creep tests and corrosion tests on Group II alloys are presented in this section. These data include a detailed metallurgical evaluation of the oxidation-carburization scales observed for 10,000-h exposed specimens utilizing optical microscopy and EDX analysis. Where necessary for completeness, data for previously reported 3000-h exposed specimens are included.

3.1. CREEP TEST DATA

The controlled-impurity helium and air creep test results are given in Table 3-1, including the data for exposures up to 10,000 h at temperatures of 800°, 900°, and 1000°C. Table 3-1 specifies the environment, the exposure temperature and time, and the stress and creep strain for each specimen and also indicates whether the specimen ruptured (R) or terminated (T), with the time given in parentheses, or is continuing (C). The complete creep data measured at 500- and 1000-h intervals for all tests are given in Appendix A.

The stress applied to each Group II alloy test specimen was chosen from design curves to give a design life of 100,000 to 200,000 h. It is noted here that certain specimens were reported by CIIR to have been overstressed during testing. These are Manaurite 900 (30143), Manaurite 36XS (29147), Manaurite 36X (28148), and HK 40 (32142) at 900°C. Creep data for these specimens have not been included in any of the graphical presentations in this section.

It should also be noted that the inherent scatter in creep testing, particularly in comparing a limited number of specimens in constant load

TABLE 3-1
SUMMARY OF CREEP RESULTS FOR GROUP II ALLOYS TESTED IN AIR AND
CONTROLLED-IMPURITY HELIUM AT CIIR

Alloy	Temp (°C)	Stress (MPa)	Environment	Test Time (hours)	Creep Strain (%)	Status ^(a)
Hastelloy X	800	31.4	Air	16,000	0.40	C
	900	12.7	Air	14,000	2.57	C
	800	31.4	He	3,000	0.01	T
	800	31.3	He	12,000	0.49	C
	900	12.8	He	3,000	0.41	T
	900	12.7	He	12,000	1.51	C
	1000	4.8	He	2,000	0.57	T
	1000	4.7	He	10,000	4.73	T(10,935)
Manaurite 36X	800	31.4	Air	16,000	0.23	C
	900	12.8	Air	9,000	0.32	T(9910)
	800	31.3	He	3,000	0.09	T
	800	31.2	He	9,000	0.00	C
	900	12.8	He	3,000	0.04	T
	900	12.9	He	9,000	0.88	T(9470)
	1000	4.8	He	3,000	1.54	T
	1000	4.8	He	7,000	0.33	C
	1000	4.8	He	6,000	0.27	C
Manaurite 36XS	800	31.1	Air	11,000	0.44	C
	900	12.8	Air	9,000	1.00	T(9910)
	800	31.3	He	3,000	0.35	T
	800	31.3	He	12,000	0.95	C
	900	12.7	He	3,000	0.39	T
	900	12.8	He	9,000	0.92	T(9470)
	1000	4.7	He	3,000	0.21	T
	1000	4.8	He	9,000	0.63	C
	1000	4.8	He	8,000	1.37	T(8935)

TABLE 3-1 (continued)

Alloy	Temp (°C)	Stress (MPa)	Environment	Test Time (hours)	Creep Strain (%)	Status ^(a)
Manaurite 900	800	31.6	Air	10,000	0.07	T(11,030)
	900	12.9	Air	4,000	2.21	T
	800	31.3	He	300	0.37	T
	800	31.1	He	9,000	0.85	T(9675)
	900	12.7	He	3,000	3.63	T
	900	12.6	He	9,000	7.15	T(9470)
	1000	4.8	He	3,000	0.07	T
	1000	4.8	He	6,000	0.37	C
RA 333	800	31.2	Air	16,000	8.59	T(17,030)
	900	12.7	Air	10,000	72.66	T
	800	31.5	He	3,000	0.80	T
	800	31.5	He	12,000	11.43	R(12,010)
	900	12.7	He	3,000	11.33	T
	900	12.8	He	2,000	9.02	R(2745)
	1000	4.8	He	3,000	12.26	T
	1000	4.8	He	3,000	8.73	R(3890)
HK 40	800	31.6	Air	14,000	0.73	R(14,630)
	900	12.7	Air	13,000	0.27	T(13,935)
	800	31.9	He	3,000	0.00	T
	800	31.5	He	9,000	0.16	C
	900	12.9	He	3,000	0.23	T
	900	12.8	He	9,000	1.15	T(9470)
	1000	4.8	He	2,000	0.23	T
	1000	4.8	He	8,000	1.60	C
Supertherm	1000	4.8	He	6,000	0.12	C
	800	31.6	Air	10,000	0.12	C
	900	12.9	Air	9,000	0.24	C
	800	31.4	He	3,000	0.06	T

TABLE 3-1 (continued)

Alloy	Temp (°C)	Stress (MPa)	Environment	Test Time (hours)	Creep Strain (%)	Status(a)
Hastelloy S	800	31.1	He	9,000	0.11	C
	900	12.8	He	3,000	0.10	T
	900	12.9	He	9,000	0.05	C
	1000	4.8	He	3,000	0.81	T
	1000	4.8	He	7,000	0.26	C
	1000	4.8	He	6,000	0.05	C
	800	31.1	Air	16,000	17.04	C
	900	12.7	Air	9,000	69.60	R(9921)
	800	31.6	He	3,000	1.57	T
	800	31.9	He	12,000	7.93	C
	900	12.8	He	3,000	4.82	T
	900	12.7	He	5,000	18.66	T
	1000	4.8	He	3,000	13.41	T
	1000	4.8	He	4,000	21.04	T
HD 556	800	31.4	Air	16,000	0.73	C
	900	12.7	Air	14,000	1.40	C
	800	31.4	He	3,000	0.25	T
	800	31.6	He	12,000	0.42	C
	900	12.8	He	3,000	0.13	T
	900	13.0	He	12,000	0.55	C
	1000	4.8	He	3,000	1.71	T
	1000	4.8	He	10,000	3.13	T(10,935)

(a) C = continuing, T = terminated, and R = ruptured; rupture or termination time (h) in parentheses.

(variable stress) tests, has shown that differences in creep strains for wrought alloys, such as Hastelloy X, Hastelloy S, RA 333, and HD 556, on the order of several tenths of a percent are probably not significant. This is especially true at lower stresses and during primary or secondary creep. However, for cast alloys, such as HK 40, Supertherm, and Manaurites 36X, 36XS, and 900, the differences may be several percent. This is due to the inherently larger grain size of these specimens since no grain refining heat treatment was performed.

If there are only several grains in a gage length cross section, a grain alignment with slip planes at a favorable orientation may make deformation easier than for a specimen with the same number of grains aligned in a different manner. Finer grained material effectively "averages-out" the effect of orientation. Therefore, when observing the results, only similarly processed material should be compared and one should be aware that creep data inherently lead to some scatter, the amount depending on processing, history, chemistry, grain size, and any grain alignment.

Creep data for the wrought alloys Hastelloy X, RA 333, Hastelloy S, and HD 556 are presented in Figs. 3-1 through 3-6 for 800°, 900°, and 1000°C. For equivalent stresses at each temperature, alloys RA 333 and Hastelloy X exhibited the largest creep strains. Significant differences in creep strains in air and helium are indicated for alloys RA 333 and Hastelloy S at 800° and 900°C. For RA 333, helium appears to have a weakening effect, while for Hastelloy S, testing in helium appears to strengthen the alloy compared to its behavior in air. The low rupture ductility and rupture life of RA 333 in helium at 900°C should be noted. The RA 333 specimen tested in air at 900°C continues after more than 9000 h with a creep strain of ~72%, while a parallel helium-tested specimen ruptured at 2745 h with a rupture ductility of 13.75%. Alloys Hastelloy X and HD 556 exhibited very small creep strains (~0.5%) at 800° and 900°C out to their maximum test times. At 1000°C all four wrought alloys experienced significant creep strains in the helium environment.

Fig. 3-1 Creep curves for alloys Hastelloy X and RA 333 in air and controlled-impurity helium at 800°C

Fig. 3-2. Creep curves for alloys Hastelloy S and HD 556 in air and controlled-impurity helium at 800°C

Fig. 3-3. Creep curves for alloys Hastelloy X and RA 333 in air and controlled-impurity helium at 900°C

Fig. 3-4. Creep curves for alloys Hastelloy S and HD 556 in air and controlled-impurity helium at 900°C

Fig. 3-5. Creep curves for alloys Hastelloy X and RA 333 in controlled-impurity helium at 1000°C

Fig. 3-6. Creep curves for alloys Hastelloy S and HD 556 in controlled-impurity helium at 1000°C

Creep strains as a function of time for the centrifugally cast alloys HK 40, Supertherm, and Manaurites 36X, 36XS, and 900 are presented in Figs. 3-7 through 3-12 at 800°, 900°, and 1000°C. The data for these alloys tended to have significantly more scatter in each environment than data for the wrought alloys, probably associated with their inherently larger and more variable grain structure as indicated previously. The smallest degree of scatter (<1%) for these alloys was observed at 800°C. At this temperature and within expected experimental scatter, the behavior of the alloys in helium and in air is similar and no effect of environment can be discerned. Similar behavior in helium and air within ~1% strain was observed for alloys HK 40, Supertherm, Manaurite 36X, and Manaurite 36XS at 900°C, while substantially larger scatter was indicated for Manaurite 900 at this temperature. However, no environmental effect for Manaurite 900 at 900°C can be definitely established since the air data for this alloy appeared to be within the scatter band for the two helium tests at this temperature. At 1000°C, all of the alloys exhibited similar creep behavior in helium.

3.2. WEIGHT CHANGE DATA

The weight change data for Group II alloys aged unstressed in controlled-impurity helium at CIIR are summarized in Table 3-2 for 800°, 900°, and 1000°C. The total test time and the respective weight change for each specimen are given in the table. The complete weight change data listed for every 500- or 1000-h interval are contained in Appendix B. One specimen was removed after 3000 h for a complete microstructural evaluation; these results have been reported previously (Ref. 3-1). The metallurgical evaluation results for 10,000-h specimens are reported in Section 3.6.

Plots of weight change as a function of exposure time at 800°, 900°, and 1000°C are presented in Figs. 3-13 through 3-15. At 800°C, Hastelloy S experienced the largest weight change (approximately +2 mg/cm²) of all alloys over the 10,000-h aging time. In general,

Fig. 3-7. Creep curves for alloys Manaurite 36X, Manaurite 36XS, and Manaurite 900 in air and controlled-impurity helium at 800°C

Fig. 3-8. Creep curves for alloys HK 40 and Superttherm in air and controlled-impurity helium at 800°C

Fig. 3-9. Creep curves for alloys Manaurite 36X, Manaurite 36XS, and Manaurite 900 in air and controlled-impurity helium at 900°C

Fig. 3-10. Creep curves for alloys HK 40 and Supertherm in air and controlled-impurity helium at 900°C

Fig. 3-11. Creep curves for alloys Manaurite 36X, Manaurite 36XS, and Manaurite 900 in controlled-impurity helium at 1000°C

Fig. 3-12. Creep curves for alloys HK 40 and Superttherm in controlled-impurity helium at 1000°C

TABLE 3-2
SUMMARY OF WEIGHT CHANGE RESULTS FOR GROUP II
ALLOYS AGED UNSTRESSED IN CONTROLLED-IMPURITY HELIUM AT CIIR

Alloy	Specimen	Temp (°C)	Time (h)	Weight Change (mg/cm ²)
Hastelloy X	08645	800	3,000	0.69
	08646	800	10,000	1.36
	08647	900	3,000	1.84
	08648	900	10,000	1.11
	08649	1000	2,000	2.00
	08650	1000	10,000	3.62
Manaurite 36X	28145	800	3,000	0.49
	28146	800	9,000	0.71
	28147	900	3,000	0.07
	28148	900	9,000	0.96
	28149	1000	3,000	-0.53
	28144	1000	6,000	-1.42
Manaurite 36XS	29144	800	3,000	-0.80
	29145	800	10,000	-0.76
	29146	900	3,000	-1.00
	29147	900	9,000	2.02
	29148	1000	3,000	0.09
	29149	1000	8,000	-5.73
Supertherm	33144	800	3,000	0.62
	33145	800	9,000	-1.56
	22146	900	3,000	1.42
	33142	900	9,000	0.91
	33148	1000	3,000	0.69
	33143	1000	7,000	0.73
	33149	1000	6,000	0.73

TABLE 3-2 (continued)

Alloy	Specimen	Temp (°C)	Time (h)	Weight Change (mg/cm ²)
Hastelloy S	34144	800	3,000	1.13
	34145	800	10,000	1.96
	34146	900	3,000	1.80
	34147	900	10,000	3.24
	34148	1000	3,000	3.36
	34149	1000	9,000	4.89
HD 556	39144	800	3,000	0.49
	39145	800	10,000	0.27
	39146	900	3,000	0.60
	39147	900	10,000	-2.36
	39148	1000	3,000	-0.49
	39149	1000	10,000	-0.71
Manaurite 900	30145	800	3,000	0.36
	30146	800	9,000	0.42
	30148	900	3,000	0.22
	30143	900	9,000	1.02
	30153	1000	3,000	-0.82
	30156	1000	6,000	-2.47
RA 333	31145	800	3,000	0.42
	31146	800	10,000	1.11
	31147	900	3,000	1.82
	31148	900	10,000	-9.78
	31149	1000	3,000	0.58
	31150	1000	9,000	-0.78
HK 40	32144	800	3,000	0.49
	32146	800	9,000	0.13
	32147	900	3,000	0.71
	32142	900	9,000	0.58
	32149	1000	3,000	-0.64
	32150	1000	6,000	-0.40
	32143	1000	7,000	-0.49

Fig. 3-13. Weight change of Group II alloys exposed unstressed in controlled-impurity helium at 800°C

Fig. 3-14. Weight change of Group II alloys exposed unstressed in controlled-impurity helium at 900°C

Fig. 3-15. Weight change of Group II alloys exposed unstressed in controlled-impurity helium at 1000°C

all of the alloys experienced rapid increases in weight up to 500 h followed by a significant decrease in weight gain rate thereafter. Alloys Manaurite 36XS and Supertherm exhibited severe weight losses after several thousand hours, indicative of scale spallation.

At 900°C, all of the alloys except HD 556 and RA 333 exhibited fairly uniform weight gain behavior. Alloys HD 556 and RA 333 showed initial weight increases out to 4000 to 6000 h followed by weight losses due to spallation. In general, the weight gains observed at 900°C were significantly larger for each alloy than those at 800°C.

At 1000°C all of the alloys except Hastelloy X, Hastelloy S, and Supertherm exhibited losses in weight. Alloys RA 333 and Manaurite 900 increased in weight initially and then lost weight, apparently due to scale spallation, while alloys Manaurite 36X, Manaurite 36XS, HK 40, and HD 556 decreased in weight throughout their total exposure. The weight gains observed for Hastelloy X, Hastelloy S, and Supertherm at 1000°C were larger than those observed for the lower temperatures.

3.3. BULK CARBON ANALYSES

The results of bulk carbon analyses on Group II alloy specimens exposed unstressed at 800°, 900°, and 1000°C in controlled-impurity helium at CIIR for times up to 10,000 h are presented in Table 3-3. For comparative purposes, Table 3-3 also includes as-received bulk carbon values. The carbon values represent the average carbon content through the cross section of the 6.4-mm-diameter specimens.

The results show that most of the alloys, with the exception of Hastelloy S, experienced no, or only slight, carbon pickup at 800°C. Moderate increases in carbon content were observed at 900°C, and at 1000°C very large increases were observed for some alloys. The greatest increases in bulk carbon content relative to the as-received

TABLE 3-3
BULK CARBON CONTENT OF GROUP II ALLOY SPECIMENS AGED UNSTRESSED
IN CONTROLLED-IMPURITY HELIUM AT CIIR

Alloy	Temperature (°C)	Time (h)	Carbon Content (wt %)(a)
Hastelloy X	As-received		0.080
	800	3,000	0.095
	800	10,000	0.11
	900	3,000	0.141
	900	10,000	0.23
	1000	3,000	0.19
	1000	10,000	0.301
Manaurite 36X	As-received		0.47
	800	3,000	0.47
	800	9,675	0.47
	900	3,000	0.52
	900	9,470	0.56
	1000	3,000	0.55
	1000	7,075	0.60
Manaurite 36XS	As-received		0.39
	800	3,000	0.42
	800	10,000	0.43
	900	3,000	0.46
	900	9,470	0.63
	1000	3,000	0.54
	1000	9,005	0.62
Manaurite 900	As-received		0.12
	800	3,000	0.13
	800	9,675	0.12
	900	3,000	0.13
	900	9,470	0.28

TABLE 3-3 (continued)

Alloy	Temperature (°C)	Time (h)	Carbon Content (wt %)(a)
RA 333	1000	3,000	0.35
	1000	7,075	0.56
	As-received		0.034
	800	3,000	0.04
	800	10,000	0.057
	900	3,000	0.14
	900	10,000	0.17
	1000	3,000	0.14
	1000	9,935	0.32
HK 40	As-received		0.44
	800	3,000	0.45
	800	9,675	0.45
	900	3,000	0.51
	900	9,470	0.58
	1000	3,000	0.50
	1000	7,985	0.71
Supertherm	As-received		0.430
	800	3,000	0.45
	800	9,675	0.45
	900	3,000	0.51
	900	9,470	0.53
	1000	3,000	0.50
	1000	7,985	0.56
Hastelloy S	As-received		0.007
	800	3,000	0.050
	800	10,000	0.048
	900	3,000	0.08
	900	10,000	0.16

TABLE 3-3 (continued)

Alloy	Temperature (°C)	Time (h)	Carbon Content (wt %) (a)
HD 556	1000	3,000	0.11
	1000	9,935	0.25
	As-received		0.092
	800	3,000	0.10
	800	10,000	0.10
	900	3,000	0.16
	900	10,000	0.20
	1000	3,000	0.26
	1000	10,000	0.402

(a) Values are from single determinations on 0.4-g samples using LECO method.

values were observed for those alloys with very low initial carbon contents. Only small relative bulk carbon content increases were observed for the high carbon cast alloys. In general, the carbon content for each alloy at constant temperature increased with increased exposure time.

3.4. ROOM TEMPERATURE IMPACT AND TENSILE PROPERTIES (UNSTRESSED AGING)

Room-temperature mechanical property measurements were conducted on Group II alloys in the as-received condition and after unstressed exposure in controlled-impurity helium and/or air at 800° and 900°C. The measurements included instrumented Charpy V-notch impact tests and tensile tests. The results of these measurements are reported in the following sections.

3.4.1. Instrumented Impact Test Results

The results of room-temperature instrumented Charpy V-notch impact tests on a number of Group II alloys in the as-received condition and after aging in controlled-impurity helium and air at 800° and 900°C are given in Table 3-4. The total absorbed energy as determined from the instantaneous load-versus-time plots for each specimen is given in the table along with calculated values of stress concentration, K. The values of fracture toughness, K_Q in Table 3-4, were calculated from the impact data employing the assumption of the equivalence of the critical strain energy release rate to the ratio of the total impact energy to the fracture surface area. The equation (Ref. 3-2) used in the calculations is given by

$$K = \frac{W_T E}{5a(1 - \mu)} ,$$

where W_T is the total absorbed energy, E is the elastic modulus, μ is Poisson's ratio, and a is the fracture surface area. This method of

TABLE 3-4
SUMMARY OF INSTRUMENTED CHARPY V-NOTCH IMPACT TEST
RESULTS ON SPECIMENS AGED UNSTRESSED IN AIR AND CONTROLLED-
IMPURITY HELIUM FOR 3000 HOURS

Alloy	Specimen Number	Condition	Total Absorbed Energy [Joules (ft-lb)]		Fracture Toughness, K_Q [MPa $\sqrt{\text{mm}}$ (ksi $\sqrt{\text{in.}}$)]	
Manaurite 36X	28187	As-received	4.5	(3.3)	1637	(47.1)
	28188	As-received	4.9	(3.6)	1637	(47.1)
	<u>Average</u>	As-received	4.7	(3.5)	1637	(47.1)
	28185	800°C air aged	2.2	(1.6)	1206	(34.7)
	28184	800°C He aged	2.3	(1.7)	1269	(36.5)
	28183	800°C He aged	2.2	(1.6)	1237	(35.6)
	<u>Average</u>	800°C He aged	2.3	(1.7)	1253	(36.1)
	28181	900°C He aged	2.4	(1.8)	1269	(36.5)
	28186	900°C air aged	1.64	(1.1)	865	(24.9)
	29187	As-received	5.3	(3.9)	1731	(49.8)
	29188	As-received	5.4	(4.0)	1731	(49.8)
	<u>Average</u>	As-received	5.4	(4.0)	1731	(49.8)
	29185	800°C air aged	1.4	(1.0)	997	(28.7)
	29184	800°C He aged	1.1	(0.8)	876	(25.2)
Manaurite 36XS	29183	800°C He aged	1.4	(1.0)	959	(27.6)
	<u>Average</u>	800°C He aged	1.3	(0.9)	918	(26.4)
	29181	900°C He aged	1.6	(1.2)	997	(28.7)
	29186	900°C air aged	1.25	(0.9)	820	(23.6)
	30188	As-received	45.7	(33.7)	5067	(145.8)
	30187	As-received	56.5	(41.7)	5371	(153.0)
	<u>Average</u>	As-received	51.6	(38.1)	5192	(149.4)
	30185	800°C air aged	11.7	(8.6)	2436	(70.1)
	30184	800°C He aged	9.5	(7.0)	2346	(67.5)
	30183	800°C He aged	8.1	(6.0)	2179	(62.7)
	<u>Average</u>	800°C He aged	8.8	(6.5)	2263	(65.1)
	30186	900°C air aged	22.5	(16.6)	1442	(41.5)
Manaurite 900	30188	As-received	45.7	(33.7)	5067	(145.8)
	30187	As-received	56.5	(41.7)	5371	(153.0)
	<u>Average</u>	As-received	51.6	(38.1)	5192	(149.4)
	30185	800°C air aged	11.7	(8.6)	2436	(70.1)
	30184	800°C He aged	9.5	(7.0)	2346	(67.5)
	30183	800°C He aged	8.1	(6.0)	2179	(62.7)
	<u>Average</u>	800°C He aged	8.8	(6.5)	2263	(65.1)
	30186	900°C air aged	22.5	(16.6)	1442	(41.5)

TABLE 3-4 (continued)

Alloy	Specimen Number	Condition	Total Absorbed Energy [Joules (ft-lb)]		Fracture Toughness, K_Q [MPa√mm (ksiv√in.)]	
Hastelloy X	08687	As-received	>136	(>100) (a)	---	---
	08688	As-received	186.8	(137.8)	13662	(393.1)
	08685	800°C air aged	9.2	(6.8)	3253	(93.6)
	08684	800°C He aged	10.6	(7.8)	3503	(100.8)
	08683	800°C He aged	9.6	(7.1)	2641	(76.0)
	<u>Average</u>	800°C He aged	10.1	(7.5)	3072	(88.4)
	08681	900°C He aged	19.1	(14.1)	4480	(128.9)
Hastelloy S	34188	As-received	>240	(>176.5) (a)	---	---
	38187	As-received	276.6	(204.0)	17075	(491.3)
	34185	800°C air aged	117.4	(86.6)	11048	(317.9)
	34184	800°C He aged	92.2	(68.0)	9751	(280.6)
	34183	800°C He aged	115.4	(85.1)	10759	(309.6)
	<u>Average</u>	800°C He aged	103.8	(76.5)	10256	(295.1)
	34186	900°C air aged	126.0	(92.9)	3131	(90.1)
HD 556	39188	As-received	147.6	(108.9)	12296	(353.8)
	39187	As-received	144.5	(106.6)	12167	(350.1)
	<u>Average</u>	As-received	146.1	(107.8)	12231	(351.9)
	39185	800°C air aged	5.0	(3.7)	2315	(66.6)
	39184	800°C He aged	5.7	(4.2)	2627	(75.6)
	39183	800°C He aged	6.2	(4.6)	2568	(73.9)
	<u>Average</u>	800°C He aged	6.0	(4.4)	2598	(74.7)
	39186	900°C air aged	8.1	(6.0)	1922	(55.3)

TABLE 3-4 (continued)

Alloy	Specimen Number	Condition	Total Absorbed Energy [Joules (ft-lb)]		Fracture Toughness, K_Q [MPa/mm (ksiv/in.)]	
RA 333	31188	As-received	181.4	(133.8)	13787	(396.7)
	31187	As-received	174.4	(128.6)	13656	(392.9)
	<u>Average</u>	As-received	177.9	(131.2)	13721	(394.8)
	31185	800°C air aged	5.6	(4.1)	2506	(72.1)
	31184	800°C He aged	6.6	(4.9)	2770	(79.7)
	31183	800°C He aged	7.3	(5.4)	2892	(83.2)
	<u>Average</u>	800°C He aged	7.0	(5.2)	2831	(81.4)
	31186	900°C air aged	13.0	(9.6)	2117	(60.9)
HK 40	32188	As-received	9.1	(6.7)	2179	(62.7)
	32187	As-received	6.1	(4.5)	1839	(52.9)
	<u>Average</u>	As-received	7.6	(5.6)	2009	(57.8)
	32185	800°C air aged	3.7	(2.7)	1411	(40.6)
	32184	800°C He aged	3.1	(2.3)	1525	(43.6)
	32183	800°C He aged	3.5	(2.6)	1383	(39.8)
	<u>Average</u>	800°C He aged	3.3	(2.5)	1449	(41.7)
	32186	900°C air aged	2.3	(1.7)	893	(25.7)
Supertherm	33188	As-received	5.4	(4.0)	1706	(49.1)
	33187	As-received	5.8	(4.3)	1793	(51.6)
	<u>Average</u>	As-received	5.6	(4.2)	1749	(50.3)
	33185	800°C air aged	1.9	(1.4)	1175	(33.8)
	33184	800°C He aged	1.8	(1.3)	1070	(30.8)
	33183	800°C He aged	1.8	(1.3)	1119	(32.2)
	<u>Average</u>	800°C He aged	1.8	(1.3)	1095	(31.5)
	23186	900°C air aged	1.24	(0.9)	803	(23.1)

(a) No break - specimen stopped impact hammer.

calculation yields the most conservative (i.e., lowest) values of stress concentration to cause failure of the several approaches available.

The high carbon (0.3% to 0.5%) centrifugally cast alloys Manaurite 36X, Manaurite 36XS, HK 40, and Supertherm exhibited low as-received impact values [4.5 to 9.1 Joules (3.3 to 6.7 ft-lb)]. Helium or air aging at 800° and 900°C reduced these values by ~50% to 75%. Each alloy exhibited similar impact strengths after aging in air and in helium at 800°C. Helium aging of Manaurites 36X and 36XS at 900°C had a slight strengthening effect with respect to air aging on impact energy and fracture toughness. Manaurite 900, a lower carbon content centrifugally cast alloy, exhibited relatively high as-received impact energy and fracture toughness values of ~52 Joules (38 ft-lb) and ~5200 MPa $\sqrt{\text{mm}}$ (150 ksi $\sqrt{\text{in.}}$), respectively. Helium aging or air aging at 800°C decreased the impact energy for this alloy by ~80% and the fracture toughness by ~55%. Manaurite 900 exhibited a ~50% decrease in impact energy from that of the as-received material when aged in air at 900°C.

High values of impact energy [~135 to 270 Joules (100 to 200 ft-lb)] and fracture toughness [~7,000 to 17,000 MPa $\sqrt{\text{mm}}$ (200 to 500 ksi $\sqrt{\text{in.}}$)] were obtained in the as-received condition for the wrought alloys Hastelloy X, Hastelloy S, RA 333, and HD 556. The as-received values were substantially reduced (50% to 95%) by helium or air aging at 800° and 900°C. For example, Hastelloy X (086), which exhibited a fracture energy of 187 Joules (138 ft-lb) before aging, decreased to 10.1 Joules (7.5 ft-lb) after helium aging at 800°C and decreased to 19 Joules (14 ft-lb) after helium aging at 900°C. Comparison of values obtained from air-aged specimens with those obtained from specimens aged in controlled-impurity helium shows that at 800°C the air- and helium-aged specimens of all of the wrought alloys exhibited similar impact energies and/or fracture toughness values.

3.4.2. Tensile Test Results

The results of room-temperature tensile tests on Group II alloys in the as-received condition and after unstressed aging for 3000 h in controlled-impurity helium and/or air at 800° and 900°C are given in Table 3-5. The room-temperature properties presented are 0.2% yield strength, ultimate strength, and plastic strain to failure (percent elongation).

The centrifugally cast alloys, with the exception of Manaurite 900, exhibited relatively lower strengths and percent elongations (ductilities) than the wrought alloys in the as-received condition. As-received values for yield and ultimate strengths ranged from ~206 MPa (30 ksi) and ~515 MPa (75 ksi), respectively, for Manaurite 900 to ~245 MPa (50 ksi) and ~620 MPa (90 ksi), respectively, for Supertherm. As-received ductilities ranged from 41% for Manaurite 900 to ~10% to 20% for the other cast alloys. The greater as-received ductility of Manaurite 900 is due to its lower carbon content (0.1%) compared to the other cast alloys (0.3% to 0.4%).

The yield strength and ultimate strength of the unstressed centrifugally cast alloys increased from their as-received values or remained constant after aging in controlled-impurity helium at 800°C and were near as-received values after aging at 900°C. The strength increases after aging at 800°C generally did not exceed 10% of the as-received values except for Manaurite 36X and Supertherm, which exhibited >30% yield strength increases at this temperature. The plastic strain to failure for all the alloys was substantially decreased after aging in helium at 800° and 900°C (Fig. 3-16). The ductility of Manaurite 900, for example, decreased from 41% in the as-received condition to 15% and 25% after helium aging at 800° and 900°C, respectively. Ductilities of alloys Manaurite 36X, Manaurite 36XS, and Supertherm were decreased from ~10% to ~3% due to helium aging.

TABLE 3-5
SUMMARY OF ROOM-TEMPERATURE TENSILE PROPERTIES
FOR SPECIMENS AGED UNSTRESSED IN AIR AND CONTROLLED-IMPURITY HELIUM

Alloy	Temp. (°C)	Condition (a)	0.2% Yield Strength		Ultimate Strength		Percent Elongation [25.4 mm (1 in.) gauge length]
			MPa	ksi	MPa	ksi	
Manaurite 900	-	As-received (1)	197.0	28.7	516.4	74.9	41.7
	800	Air aged (1)	239.2	34.7	428.1	62.1	10.3
	900	Air aged (1)	186.2	27.0	480.6	69.7	32.9
	900	He aged (2)	184.4	26.8	487.4	70.7	24.9
RA 333	-	As-received (1)	392.3	56.9	762.5	110.6	46.7
	800	Air aged (1)	346.1	50.2	818.3	118.7	13.1
	800	He aged (1)	400.5	58.1	876.6	130.1	17.4
	900	Air aged (2)	298.5	43.3	699.1	101.4	29.6
	900	He aged (2)	320.9	46.6	672.9	97.6	15.3
HK 40	-	As-received (1)	324.0	47.0	575.6	83.5	18.7
	800	Air aged (1)	298.5	43.3	441.2	64.0	5.5
	800	He aged (2)	339.9	49.3	623.2	90.4	6.4
	900	Air aged (1)	299.2	43.4	499.9	72.5	5.3
	900	He aged (2)	313.4	45.5	571.9	83.0	15.8
Hastelloy X (086)	-	As-received (1)	329.5	47.8	776.3	112.6	46.8
	800	Air aged (1)	322.6	46.8	850.0	123.3	22.0
	800	He aged (1)	361.9	52.5	852.0	123.6	16.1
	900	Air aged (1)	275.8	40.0	727.6	105.4	23.1
	900	He aged (2)	272.0	39.5	626.0	90.8	12.8
Manaurite 36X	-	As-received (1)	280.6	40.7	531.5	77.1	11.7
	800	Air aged (1)	286.8	41.6	537.0	77.9	3.5
	800	He aged (2)	263.4	38.2	561.2	81.4	3.2
	900	Air aged (1)	264.1	38.3	468.2	67.9	3.1
	900	He aged (2)	287.9	41.8	502.9	73.0	4.1

TABLE 3-5 (continued)

Alloy	Temp. (°C)	Condition ^(a)	0.2% Yield Strength		Ultimate Strength		Percent Elongation [25.4 mm (1 in.) gauge length]
			MPa	ksi	MPa	ksi	
Manaurite 36XS	-	As-received (1)	273.0	39.6	560.5	81.3	10.8
	800	Air aged (1)	305.4	44.3	602.5	87.4	2.4
	800	He aged (2)	373.0	54.1	585.3	84.9	2.7
	900	Air aged (1)	274.4	39.8	506.1	73.4	4.0
	900	He aged (2)	258.2	37.5	508.1	73.7	3.5
Supertherm	-	As-received (1)	352.3	51.1	601.2	87.2	9.1
	800	Air aged (1)	388.8	56.4	634.2	92.0	2.3
	800	He aged (2)	455.7	66.1	732.8	106.3	2.6
	900	Air aged (2)	338.5	49.1	530.9	77.0	1.2
	900	He aged (2)	365.7	53.1	587.7	85.2	2.8
Hastelloy S	-	As-received (1)	373.0	54.1	864.5	125.4	52.9
	800	Air aged (1)	353.7	51.3	876.9	127.2	48.9
	800	He aged (1)	384.7	55.8	828.9	120.2	29.0
	900	Air aged	342.7	49.7	843.9	112.4	40.2
	900	He aged (2)	349.9	50.8	813.2	118.0	29.9
HD 556	-	As-received (1)	370.2	32.7	785.2	113.9	48.7
	800	Air aged (1)	307.5	44.6	707.3	102.6	6.8
	800	He aged (1)	398.5	57.8	859.5	124.7	10.9
	900	Air aged (2)	257.9	37.4	726.0	105.3	32.5
	900	He aged (2)	317.1	46.2	699.4	101.5	15.4

(a) Number in parentheses is the number of tests averaged for the value given.

Fig. 3-16. Plastic strain to failure versus aging temperature for Group II alloys exposed unstressed for 3000 h in controlled-impurity helium

Strength values obtained after aging in helium at 800°C were the same as or greater than values obtained after air aging. The increases were generally less than 15% except for alloys Manaurite 36X, HK 40, and Supertherm. Yield and/or ultimate strengths for these alloys after helium aging were 20% to 40% greater than comparable air-aged specimens. There was little difference in strength values for alloys aged in air or helium at 900°C. Plastic strains to failure for air- and helium-exposed specimens were comparable at 800° and 900°C for all alloys except HK 40, which had a significantly higher strain value for helium compared to air at 900°C.

The wrought solid-solution-strengthened alloys exhibited as-received yield and ultimate strength values that were generally somewhat higher than the centrifugally cast alloys. Plastic strains to failure were substantially greater than those for all of the cast alloys except Manaurite 900. Yield and ultimate strengths were increased slightly or remained essentially the same as as-received values after aging in helium at 800°C and decreased below as-received values after aging at 900°C. Plastic strains to failure for all alloys progressively decreased with increased aging temperature to values of 10% to 30% (Fig. 3-16). The lowest ductilities at 900°C were obtained for Hastelloy X, and the highest values were observed for Hastelloy S.

Strength values for air- and helium-aged specimens at 800° and 900°C were comparable for all wrought alloys except alloys RA 333 and HD 556, which exhibited slightly higher strengths in helium than in air. Yield strengths for Hastelloy X aged at 900°C were comparable in air and helium, while ultimate strength values for both alloys were lower after aging in helium than in air. Comparable values of plastic strain to failure after aging in air and helium at 800°C were observed for alloys RA 333, Hastelloy X, and HD 556. Ductilities of Hastelloy S after aging at 800°C and Hastelloy X after aging at 900°C were significantly lower in the helium-aged condition.

3.5. RESIDUAL TENSILE PROPERTIES (CREEP SPECIMENS)

The changes in room-temperature tensile properties of the Group II alloys after controlled-impurity helium creep testing for 3000 h at 800°, 900°, and 1000°C are presented in Table 3-6. The table includes values for 0.2% yield strength, ultimate strength, percent elongation, and percent reduction of area as well as final creep strains. For comparative purposes, the tensile properties of 3000-h unstressed and helium-aged (800° and 900°C) specimens have also been included in the table.

The residual tensile properties of stressed specimens of the Group II alloys exhibited changes with temperature that were similar to those observed for unstressed specimens at 800° and 900°C. Yield strengths for the cast alloys increased from their as-received values after creep at 800° and 900°C, whereas those of the wrought alloys were essentially unchanged at 800°C and had decreased at 900°C. Yield strength values of the cast alloys remained nearly the same or increased slightly from their as-received values after creep at 1000°C, whereas values for the wrought alloys generally decreased. The ultimate strengths of the cast alloys Manaurite 900 and Manaurite 36X and the wrought alloys Hastelloy X and Hastelloy S decreased from their as-received values after creep at 800°, 900°, and 1000°C, whereas values for Manaurite 36XS, Supertherm, HK 40, RA 333, and HD 556 increased after 800°C creep and decreased after 900° and 1000°C creep. Room-temperature tensile elongations and reductions of area for all alloys after creep at 800°, 900°, and 1000°C were markedly lower than corresponding as-received values.

A comparison of the tensile properties of unstressed versus stressed specimens shows that prior creep strain at elevated temperatures markedly affected the residual properties of the helium-exposed Group II alloys. Yield strength values for stressed specimens of the cast alloys were similar to or significantly greater than those values obtained

TABLE 3-6
ROOM-TEMPERATURE TENSILE PROPERTIES OF GROUP II ALLOYS
AFTER 3000-HOUR EXPOSURE (STRESSED AND UNSTRESSED) IN CONTROLLED-IMPURITY HELIUM^(a)

Alloy	Condition	0.2% Yield Strength		Ultimate Strength		Elongation (%)	Reduction of Area (%)	Creep Strain (%)
		MPa	ksi	MPa	ksi			
Manaurite 900	As-received	197.9	28.7	516.4	74.9	41.7	37.6	--
	800°C (b)	228.9	33.2	494.3	71.7	15.1	15.1	--
	800°C - stressed	224.7	32.6	488.1	70.8	16.1	13.5	0.37
	900°C (b)	184.4	26.8	487.4	70.7	24.9	24.0	--
	900°C - stressed	234.4	34.0	419.6	60.9	10.0	6.3	3.63
	1000°C - stressed	181.3	26.3	444.0	64.4	16.5	14.2	0.07
Manaurite 36X	As-received	280.6	40.7	531.5	77.1	11.7	6.7	--
	800°C (b)	263.4	38.2	561.2	81.4	3.2	3.0	--
	800°C - stressed	328.2	47.6	483.3	70.1	2.6	0.7	0.09
	900°C (b)	287.9	41.8	502.9	73.0	4.1	3.2	--
	900°C - stressed	289.5	42.0	514.2	74.6	3.7	1.5	0.04
	1000°C - stressed	339.9	49.3	501.3	72.7	3.8	3.1	1.54
Manaurite 36XS	As-received	273.0	39.6	560.5	81.3	10.8	9.7	--
	800°C (b)	373.0	54.1	585.3	84.9	2.7	2.7	--
	800°C - stressed	345.4	50.1	575.0	83.4	2.2	1.4	0.35
	900°C (b)	358.2	37.5	508.1	73.7	3.5	2.7	--
	900°C - stressed	321.9	46.7	496.6	72.0	2.5	0.3	0.39
	1000°C - stressed	297.8	43.2	519.0	75.3	4.0	2.8	0.21
Supertherm	As-received	352.3	51.1	601.2	87.2	9.1	7.3	--
	800°C (b)	455.7	66.1	732.8	106.3	2.6	1.5	--
	800°C - stressed	450.2	65.3	700.4	101.6	2.2	0.9	0.06
	900°C (b)	365.7	53.1	587.7	85.3	2.8	2.0	--
	900°C - stressed	399.9	58.0	553.9	80.3	2.4	0.8	0.10
	1000°C - stressed	381.9	55.4	561.4	81.4	2.0	0.5	0.81

TABLE 3-6 (continued)

Alloy	Condition	0.2% Yield Strength		Ultimate Strength		Elongation (%)	Reduction of Area (%)	Creep Strain (%)
		MPa	ksi	MPa	ksi			
HK-40	As-received	324.0	47.0	575.6	83.5	18.7	11.9	--
	800°C (b)	339.9	49.3	632.2	90.4	6.4	6.3	--
	800°C - stressed	351.6	51.0	587.4	85.2	5.2	3.8	0.00
	900°C (b)	313.4	45.5	571.9	83.0	15.8	7.6	--
	900°C - stressed	353.0	51.2	543.6	78.9	3.2	0.6	0.23
	1000°C - stressed	315.7	45.8	452.2	65.6	4.5	3.0	2.04
Hastelloy X	As-received	329.5	47.8	776.3	112.6	46.8	66.5	--
	800°C (b)	361.9	52.5	852.0	123.6	16.1	15.2	--
	800°C - stressed	369.5	53.6	756.3	109.7	8.0	6.0	0.01
	900°C (b)	272.0	39.5	626.0	90.8	12.8	14.8	--
	900°C - stressed	283.3	41.1	593.4	86.1	11.8	8.8	0.41
	1000°C - stressed	330.9	48.0	585.0	84.9	12.0	10.2	1.87
RA 333	As-received	392.3	56.9	762.5	110.6	46.7	67.1	--
	800°C (b)	400.5	58.1	896.6	130.1	17.4	16.6	--
	800°C - stressed	376.4	54.6	845.9	122.7	13.6	8.4	0.80
	900°C (b)	320.9	46.6	672.9	97.6	15.3	13.8	--
	900°C - stressed	288.9	41.9	397.9	57.7	5.0	3.2	11.33
	1000°C - stressed	282.0	40.9	620.9	90.1	27.5	31.5	12.26
Hastelloy S	As-received	373.0	54.1	864.5	125.4	52.9	70.1	--
	800°C (b)	384.7	55.8	828.9	120.2	29.0	20.2	--
	800°C - stressed	384.7	55.8	648.0	94.0	9.4	5.0	1.57
	900°C (b)	349.9	50.8	813.2	118.0	29.9	26.3	--
	900°C - stressed	350.2	50.8	683.4	99.1	12.7	12.4	4.82
	1000°C - stressed	324.7	47.1	750.1	108.8	40.0	34.3	13.41

TABLE 3-6 (continued)

Alloy	Condition	0.2% Yield Strength		Ultimate Strength		Elongation (%)	Reduction of Area (%)	Creep Strain (%)
		MPa	ksi	MPa	ksi			
HD 556	As-received	370.2	53.7	785.2	113.9	48.7	62.3	--
	800°C(b)	398.5	57.8	859.5	124.7	10.9	8.2	--
	800°C - stressed	371.6	53.9	823.8	119.5	15.6	16.5	0.25
	900°C(b)	317.1	46.0	699.4	101.5	15.4	12.4	--
	900°C - stressed	304.7	44.2	707.3	102.6	13.9	9.8	0.13
	1000°C - stressed	340.6	49.4	657.5	95.4	15.1	9.4	1.71

(a) All values are for single tests unless otherwise specified. Unless otherwise noted, specimens are unstressed.

(b) Average value for tests on two specimens.

for unstressed and aged specimens, whereas yield strength values for the wrought alloys were unchanged or markedly lowered by prestrain in creep. Ultimate strength values for all of the Group II alloys were generally lowered by prestrain in creep at 800° and 900°C compared to their unstressed counterparts. In general, the largest decreases in ultimate strength were observed for the wrought alloys, especially alloys RA 333 at 900°C and Hastelloy S at 800° and 900°C.

Prestrain in creep also resulted in lower values of tensile elongation and reduction of area for all of the alloys when compared to values for unstressed specimens. Only small differences were observed for Manaurite 36X, Manaurite 36XS, Supertherm, and HD 556 at 800° and 900°C; for Manaurite 900, HK 40, and RA 333 at 800°C; and for Hastelloy X at 900°C. Relatively large tensile elongation and reduction of area differences between stressed and unstressed specimens were observed for Hastelloy X and Hastelloy S at 800°C and for Manaurite 900, HK 40, RA 333, and Hastelloy S at 900°C. However, except for Hastelloy X and HK 40, at least a portion of the large differences observed may be related to the fact that these alloys contained significant amounts of creep strain prior to room-temperature testing, ranging from ~1.5% for Hastelloy S at 800°C to over 11% for RA 333 at 900°C. A significant portion of the plastic strain capability of these alloys may have been consumed during creep testing, thus reducing the remaining tensile ductility compared to that of an unstressed or zero prestrained specimen. In fact, those alloys that contained large amounts of creep strain prior to tensile testing (i.e., Manaurite 900, RA 333, and Hastelloy S) may have actually reached the tertiary stage of creep, which could account for the large differences also observed in ultimate strength for these alloys.

3.6. METALLURGICAL EVALUATION RESULTS

Analysis of the microstructure of all nine (Group II) alloys was performed on coupons after unstressed exposure in controlled-impurity helium for 10,000 h at temperatures of 800°, 900°, and 1000°C. Metallurgical analysis was performed using two analytical techniques. Optical microscopy was used to measure the depth of internal oxidation, alloy depletion, and carburization. Identification of corrosion products and internal microstructural constituents was conducted on selected specimens using SEM/EDX analysis.

The nine different alloys of Group II belong to two distinct microstructural classes. The first includes the austenitic, centrifugally cast alloys HK 40, Supertherm, Manaurite 36X, Manaurite 36XS, and Manaurite 900. The second group contains the wrought solid-solution-strengthened alloys, including Hastelloy X, Hastelloy S, RA 333, and HD 556.

Photomicrographs of many of the creep and corrosion specimens are shown in Figs. 3-17 through 3-23. Evidence of spalling is visible on many of the specimens.

3.6.1. Centrifugally Cast Austenitic Alloys

The five centrifugally cast alloys all exhibited a similar initial microstructure. This microstructure consisted of large dendritic grains containing primary eutectic carbides dispersed interdendritically within an austenitic matrix. A typical example, HK 40, is shown in Fig. 3-24. The eutectic carbides had a fine, lacy appearance and the austenite matrix contained no precipitated (secondary) carbides.

HK 40
(32105) 800°C

(32146)

HK 40
(32107) 900°C

(32142)

HK 40
(32103) 1000°C

(32143)

Fig. 3-17. Typical surface appearance of HK 40 after creep testing and thermal aging in impure helium for ~10,000 hours (1.5X)

SUPERTHERM
(33106)

800°C

(33145)

SUPERTHERM
(33108)

900°C

(33142)

SUPERTHERM
(33103)

1000°C

(33143)

Fig. 3-18. Typical surface appearance of Supertherm after creep testing and thermal aging in impure helium for ~10,000 hours (1.5X)

MANAURITE 36X
(28106)

800°C

(28146)

MANAURITE 36X
(28108)

900°C

(28148)

MANAURITE 36X
(28103)

1000°C

(28144)

Fig. 3-19. Typical surface appearance of Manaurite 36X after creep testing and thermal aging in impure helium for ~10,000 hours (1.5X)

MANAURITE 36XS
(29109)

800°C

MANAURITE 36XS
(29108)

900°C

(29147)

MANAURITE 36XS
(29111)

1000°C

(29149)

Fig. 3-20. Typical surface appearance of Manaurite 36XS after creep testing and thermal aging in impure helium for ~10,000 hours (1.5X)

MANAURITE 900
(30107)

800°C

(30146)

MANAURITE 900
(30109)

900°C

(30143)

MANAURITE 900
(30111)

1000°C

(30156)

Fig. 3-21. Typical surface appearance of Manaurite 900 after creep testing and thermal aging in impure helium for ~10,000 hours (1.5X)

HASTELLOY X 800°C
(08612)

HASTELLOY X 900°C
(08610)

HASTELLOY X 1000°C
(08610)

Fig. 3-22. Typical surface appearance of Hastelloy X after creep testing and thermal aging in impure helium for ~10,000 hours (1.5X)

RA 333 (31110)
800°C

RA 333 (31150)
1000°C

HASTELLOY S (34111)
800°C

HASTELLOY S (34149)
1000°C

HD 556 (39112)
800°C

HD 556 (39109)
900°C

HD 556 (39111)
1000°C

Fig. 3-23. Typical surface appearance of RA 333, Hastelloy S, and HD 556 after creep testing and thermal aging in impure helium for ~10,000 hours (1.5X)

HK 40 (321) MURAKAMI'S ETCH 500X
MICROSTRUCTURE SHOWS CASTING DEFECTS AND
PRIMARY EUTECTIC CARBIDES AT DENDRITE
BOUNDARIES.

Fig. 3-24. Optical photomicrograph showing etched internal microstructure of alloy HK 40 in the as-received condition

3.6.1.1. HK 40

A summary of the internal microstructural changes observed for alloy HK 40 after 10,000-h exposure in controlled-impurity helium is presented in Table 3-7 and shown in Figs. 3-24 and 3-25. Selected results of the SEM/EDX analyses for the three different exposure temperatures are presented in Tables D-1 through D-3 and Figs. D-1 and D-2 of Appendix D. Carbide and oxide identification was made using prior experience gained from analysis of Group I alloys (Ref. 3-1).

Internal Microstructure. Exposure of HK 40 at 800°, 900°, and 1000°C resulted in the precipitation, coalescence, and coarsening of secondary carbides. Primary eutectic carbides also coarsened and became blocky in morphology. At 900° and 1000°C, secondary carbides in the neighborhood of the primary carbides dissolved into the matrix. Figure 3-25 shows the differences in morphology of primary carbides and in the distribution of secondary carbides between exposures of 10,000 h in impure helium at 900° and 1000°C. Coarsening of primary eutectic carbides has taken place at 1000°C and a much wider precipitate-free zone has formed around the primary carbides. Figure 3-26 shows the effect of time on the microstructure of HK 40 at 1000°C.

The density of secondary carbide precipitation and coarsening was greater nearer the surface than at the center of the specimen. Primary eutectic carbides were also coarsened, thus outlining the grain boundaries.

The depth of heavy carbide precipitation (carburization) in HK 40 was not measurable at any temperature. This was because the large (0.44 wt %) carbon content of the alloy made differences in the density of carbide precipitation hard to discern. The alloy depletion zone depth increased with increasing exposure temperature. The oxidation depth was small ($\sim 8 \mu\text{m}$) at 800°C, increased at 900°C, and remained the same at 1000°C.

TABLE 3-7
SUMMARY OF SURFACE CORROSION MEASUREMENTS AND INTERNAL
MICROSTRUCTURAL ANALYSIS FOR ALLOY HK 40 AGED UNSTRESSED IN CONTROLLED-
IMPURITY HELIUM FOR 10,000 HOURS

Specimen No.	Condition (Exposure Temp. and Time)	Mean Corrosion Depth (μm)			Summary of Internal Structure
		Oxidation	Alloy Depletion	Heavy Carbide Precipitation (Carburization)	
HK 40	As-cast	--	--	--	Fe-rich γ -matrix (austenite) containing primary eutectic carbides in the dendrite boundaries. The eutectic carbide has a fine, lacy appearance and the austenitic matrix contains no precipitated (secondary) carbides. Numerous casting defects are evident throughout the structure.
HK 40 (32146)	800°C (9675 h)	8	14	N.D. ^(a)	Constituents same as for as-cast structure except that a dense array of fine carbides has precipitated inside the dendrite grains. Microstructure very much the same as at 3000 h except that the eutectic nature of the primary carbides is much less evident.
HK 40 (32142)	900°C (9470 h)	19	38	N.D.	Fe-rich γ -matrix (austenite) with primary and secondary precipitates. Primary eutectic carbides show little evidence of being eutectic in morphology. The 3000-h specimen did show traces of eutectic structure. Density of secondary intradendritic precipitates lower than in 800°C specimen. A narrow precipitate-free zone exists around the primary carbides. Not much coarsening of primary carbides evident in comparison with 800°C or 3000-h specimens. A small amount of interdendritic precipitation has taken place.
HK 40 (32143)	1000°C (7985 h)	19	127	720	Constituents the same as for 900°C condition except that primary eutectic carbides have coarsened and begun to link up to outline the grains. Fine, secondary carbides have redissolved in wider area around primary carbides than at 900°C. Density of secondary carbides greater in center of specimen than at near-surface regions. Only a small amount of coarsening of secondary precipitates has taken place.

^(a)N.D. = not determinable.

HK 40 (32142)

MURAKAMI'S ETCH

500X HK 40 (32143)

MURAKAMI'S ETCH

500X

- (a) MICROSTRUCTURE SHOWS LARGE COARSENEED
PRIMARY CARBIDES AT DENDRITE BOUNDARIES
AND SECONDARY, INTRADENDRITIC CARBIDES

- (b) MICROSTRUCTURE SHOWS VERY LARGE COARSENEED
PRIMARY CARBIDES AT DENDRITE BOUNDARIES
AND COARSENEED, SECONDARY INTRADENDRITIC
PRECIPITATES

Fig. 3-25. Optical photomicrographs showing internal microstructure of HK 40 after 10,000-h exposure to impure helium at 900°C (a) and 1000°C (b)

HK 40 (32149) MURAKAMI'S ETCH 1000X

(a) MICROSTRUCTURE SHOWS PRIMARY EUTECTIC CARBIDES AND SECONDARY INTRADENDRITIC CARBIDES

HK 40 (32143) MURAKAMI'S ETCH 1000X

(b) MICROSTRUCTURE SHOWS LARGE COARSENEED PRIMARY EUTECTIC CARBIDES AND COARSENEED SECONDARY CARBIDES

Fig. 3-26. Optical photomicrographs showing internal microstructure of HK 40 after 3000-h (a) and 10,000-h (b) exposure in impure helium at 1000°C

Surface Conditions. The surface condition of HK 40 specimens after 10,000-h exposure at 800°, 900°, and 1000°C is summarized below. Selected SEM/EDX results for specimens exposed at all three test temperatures are presented in Tables D-1 through D-3 and Figs. D-1 and D-2 of Appendix D.

The appearance and composition of the surface scale on HK 40 was dependent on the temperature at which it was tested. At 800°C it consisted of a continuous inner scale of Si-rich oxide and a highly discontinuous series of protrusions of Cr-rich carbides above the oxide. After testing at 900°C the inner Si-rich oxide had developed stringers into the bulk and the Cr-rich carbide outer scale had developed into a continuous layer on the surface. In addition, patches of mixed carbide-oxide had formed in the inner scale. At 1000°C spalling had occurred, breaking up the continuous layers of carbide and oxide. As shown in Fig. D-2, there are locations where the scale has reformed. The discontinuous outer scale shown is a Cr-rich carbide with significant amounts of Ni and Fe. The inner dark-etching scale is a Si-rich oxide with a large amount of magnesium, the origin of which is unknown.

3.6.1.2. Supertherm

Detailed metallurgical analysis for Supertherm exposed for 10,000 h to controlled-impurity helium at 800°, 900°, and 1000°C is presented in Table 3-8. Figures 3-27 and 3-28 show representative internal microstructure photomicrographs of Supertherm for the as-cast microstructure and all three exposure temperatures. Selected results of SEM/EDX analyses for all the exposure conditions are shown in Tables D-4 through D-6 and Figs. D-3 and D-4 of Appendix D.

Internal Microstructure. As indicated in Figs. 3-27 and 3-28 and Table 3-8, the secondary precipitates present in Supertherm after exposure were sparse and small at 800°C, denser but still small at 900°C, and less dense but larger at 1000°C. The primary eutectic carbides increased

TABLE 3-8
SUMMARY OF SURFACE CORROSION MEASUREMENTS AND INTERNAL
MICROSTRUCTURAL ANALYSIS FOR ALLOY SUPERTHERM AGED UNSTRESSED IN CONTROLLED-
IMPURITY HELIUM FOR 10,000 HOURS

Specimen No.	Condition (Exposure Temp. and Time)	Mean Corrosion Depth (μm)			Summary of Internal Structure
		Oxidation	Alloy Depletion	Heavy Carbide Precipitation (Carburization)	
Superttherm (331)	As-cast	--	--	--	Ni-rich γ -matrix (austenite) containing primary eutectic carbides at the dendrite boundaries. The eutectic carbides have a fine lacy appearance. The carbides occupy a larger volume of the as-cast alloy than they do in as-cast HK-40.
Superttherm (33145)	800°C (9675 h)	13	22	N.D. ^(a)	Primary carbide morphology is nearly the same as in as-cast structure with very little coarsening of the carbides having taken place. Secondary carbides have precipitated in the intradendritic regions. The secondary precipitates are more distinct than they were after 3000 h. The zone of no precipitation around the primary carbides is fairly wide. The eutectic structure of the primary carbides is still very visible.
Superttherm (33142)	900°C (9470 h)	20	35	N.D.	Primary eutectic carbides have changed in that they have become more blocky than at 800°C. Their eutectic origin is still visible at the grain boundary triple points. The secondary precipitates have become denser and more distinct (coarsened). The precipitate free zone appears to be narrower than at 800°C. Growth of the secondary carbides during aging from 3000 to 10,000 h is evident.
Superttherm (33143)	1000°C (7985 h)	5	22	N.D.	The extensive coarsening of the primary eutectic carbides at 1000°C is evident as shown in Fig. 3-28b. Some of the secondary carbides have undergone extensive coarsening but the overall density of secondary carbides has decreased from the density found at 900°C.

^(a) N.D. = not determinable.

SUPERTHERM (331) MURAKAMI'S ETCH 500X

(a) MICROSTRUCTURE SHOWS PRIMARY
EUTECTIC CARBIDES AT DENDRITE
BOUNDARIES

SUPERTHERM (33145) MURAKAMI'S ETCH 500X

(b) MICROSTRUCTURE SHOWS PRIMARY
EUTECTIC CARBIDES AT DENDRITE
BOUNDARIES AND SECONDARY
PRECIPITATED CARBIDES

Fig. 3-27. Optical photomicrographs showing internal microstructure of Supertherm in the as-cast condition (a) and after 10,000-h exposure to impure helium at 800°C

SUPERTHERM (33143) MURAKAMI'S ETCH 500X

(a) MICROSTRUCTURE SHOWS PRIMARY EUTECTIC CARBIDES AND SLIGHTLY COARSENEED SECONDARY CARBIDES

SUPERTHERM (33143) MURAKAMI'S TECH 500X

(b) MICROSTRUCTURE SHOWS COARSENEED PRIMARY EUTECTIC CARBIDES AND SECONDARY CARBIDES

Fig. 3-28. Optical photomicrographs showing internal microstructure of Supertherm after 10,000-h exposure to impure helium at 900°C (a) and 1000°C (b)

in size with increasing exposure temperature. At 1000°C it was difficult to discern their eutectic origin. The precipitate-free zone around the primary carbides was widest at 800° and 1000°C.

Because of the high carbon content of Supertherm (0.43%), differences in carbide density between the near-surface region and the central matrix were very hard to observe. Hence, no carburization depths were determinable. The alloy depletion depth increased from 22 to 35 μm as the exposure temperature was increased from 800° to 900°C, and then decreased to 22 μm at 1000°C. The oxidation depth also increased as the exposure temperature was increased from 800° to 900°C but then decreased after exposure at 1000°C.

Surface Condition. The SEM photomicrographs of Supertherm are shown and described in Figs. D-3 and D-4 and Tables D-4 through D-6 of Appendix D. The scales at 800° and 1000°C were different from the duplex scale formed at 900°C. The scale at 800°C appeared to be a mixture of Cr-rich and Si-rich oxides. Oxide stringers of this composition intruded into the bulk. At 900°C the duplex scale consisted of an outer layer of Cr-rich carbide and an inner layer of almost pure Si-rich oxide. After exposure at 1000°C the thin scale consisted of regions of Si-rich oxide and Cr-rich carbides. Magnesium was also present in large amounts in some particles.

3.6.1.3. Manurite 36X

Detailed metallurgical analysis for the alloy Manaurite 36X exposed for 10,000 h to controlled-impurity helium at 800°, 900°, and 1000°C is presented in Table 3-9. Figure 3-29 shows the microstructure of Manaurite 36X after exposure at 1000°C for 7985 h. Selected results of the SEM/EDX analysis for all three exposure conditions are shown in Tables D-7 through D-9 and Figs. D-5 through D-7 of Appendix D.

TABLE 3-9
SUMMARY OF SURFACE CORROSION MEASUREMENTS AND INTERNAL
MICROSTRUCTURAL ANALYSIS FOR ALLOY MANAURITE 36X AGED UNSTRESSED IN CONTROLLED-
IMPURITY HELIUM FOR 10,000 HOURS

Specimen No.	Condition (Exposure Temp. and Time)	Mean Corrosion Depth (μm)			Summary of Internal Structure
		Oxidation	Depletion	Heavy Carbide Precipitation (Carburization)	
Manaurite 36X (281)	As-cast	--	--	--	Fe-Ni-rich γ -matrix (austenite) containing primary eutectic carbides at the dendrite boundaries. Eutectic structure is fairly coarse and weblike.
Manaurite 36X (28146)	800°C (9675 h)	7.6	30	214	Secondary carbides have precipitated out in the intradendrite regions and also appear interdendritically in places. The primary eutectic carbides have coarsened and their initial weblike appearance has almost disappeared. No precipitate-free zone around the primary carbides is visible.
Manaurite 36X (28148)	900°C (9470 h)	18	32	254	Coarsening of the primary and secondary carbides has taken place. The density of secondary carbides remained about the same as at 800°C, but they coarsened. The primary carbides became more blocky in morphology.
Manaurite 36X (28144)	1000°C (7985 h)	6	41	275	As shown in Fig. 3-22 the primary eutectic carbides have coarsened a great deal. In some locations, especially closer to the surface scale, the grains have been completely outlined by the primary carbides. The secondary carbides have coarsened and coalesced, leading to a decrease in the number density from the 900°C specimen. In some places evidence of a second phase is visible at interdendritic sites.

(a) N.D. = not determinable.

MANAURITE 36X (28144) DIC 250X

- (a) MICROGRAPH SHOWS NEAR SURFACE REGION INCLUDING SCALE, DE-NUDED ZONE, CARBURIZED ZONE, AND INTERNAL MICROSTRUCTURE. LAYER ON TOP OF SCALE IS ELECTRODEPOSITED NICKEL

MANAURITE 36X (28144) MURAKAMI'S ETCH 500X

- (b) MICROSTRUCTURE SHOWS COARSENEED PRIMARY EUTECTIC CARBIDES AND COARSENEED SECONDARY CARBIDES. ALSO SHOWN IS A DARK ETCHING SECOND PHASE

Fig. 3-29. Optical photomicrographs showing internal microstructure of Manaurite 36X after 7985-h exposure to impure helium at 1000°C

Internal Microstructure. The as-cast microstructure of Manaurite 36X consisted of a Ni-rich γ -matrix (austenite) containing eutectic carbides. After thermal aging in impure helium at 800°C, secondary carbides precipitated at intradendritic sites. At 900°C the primary and secondary carbides had begun to coarsen, but the density of secondary carbides remained about the same as for the 800°C exposure. At 1000°C the primary carbides coarsened significantly and many of the secondary carbides coalesced, causing the density of secondary carbides to decrease.

The depth of carburization increased with increasing test temperature, reaching a maximum of 275 μm at 1000°C. The oxidation depth and alloy depletion depth also increased as the test temperature increased from 800° to 900°C. The mean oxidation depth was smaller at 1000°C than it was at 900°C. The mean corrosion depth was largest at 1000°C despite the spalling, as shown in Fig. D-7 of Appendix D.

Surface Conditions. The SEM photomicrographs of Manurite 36X are shown in Figs. D-5 through D-7 and described in Tables D-7 through D-9 of Appendix D. The scale of Manaurite 36X was composed of three layers at 800°C and two layers at 900°C and had spalled considerably at 1000°C.

At 800°C the outer scale was a mixture of finger-shaped Cr-rich carbide and Cr-Mn-rich oxide. The middle layer of scale was a Cr-rich carbide rich in Si, while the inner layer was a Si-rich oxide.

At 900°C the scale had two layers; the outer layer was thick and continuous and had occasional fingers of the inner scale growing into it. The outer scale was composed of a Cr-rich carbide, while the inner scale was a Si-rich oxide.

The scale at 1000°C is shown in Fig. D-7. The highly spalled outer layer was composed of a Cr-rich carbide, while the inner scale was a Si-rich oxide.

3.6.1.4. Manaurite 36XS

Detailed metallurgical analysis for the alloy Manaurite 36XS is presented in Table 3-10. Figures 3-30 and 3-31 show the morphology of the inner microstructure after exposure at different test temperatures. Selected results of the SEM/EDX analysis are shown in Tables D-10 and D-11 and Fig. D-8 of Appendix D.

Internal Microstructure. The as-cast microstructure of Manaurite 36XS consisted of a Fe-Ni-rich γ -matrix (austenite) containing primary eutectic carbides. The microstructure closely resembled that of Manaurite 36X except that the grain size was larger. After thermal aging in impure helium, secondary carbides appeared in the intradendritic regions at 800°C and also at the dendrite boundaries at 900°C. The secondary carbides consisted of two different morphologies, spherical and acicular. The acicular secondary carbides were most prevalent after 3000 h at 800°C. After 10,000 h at 800°C, they had nearly disappeared. Few acicular carbides remained after 3000 h at 900°C.

The primary eutectic carbides tended to coarsen and eventually covered the grain boundaries. After 10,000 h at 1000°C, they had almost completely encircled all of the grains. The spherical secondary carbides generally redissolved into the matrix. Near the center of the specimen at 1000°C, the grains were nearly carbide free.

A dark-etching second phase began to appear after 10,000 h at 900°C. It formed exclusively at dendrite grain boundaries. After 10,000 h at 1000°C these second phases became numerous and were located on almost every primary carbide.

The resulting measurements of oxidation depth, alloy depletion depth, and carburization depth were complicated by spallation at 1000°C. The oxidation depth increased from 16 to 36 μm as the test

TABLE 3-10
SUMMARY OF SURFACE CORROSION MEASUREMENTS AND INTERNAL
MICROSTRUCTURAL ANALYSIS FOR ALLOY MANAURITE 36XS AGED UNSTRESSED IN CONTROLLED-
IMPURITY HELIUM FOR 10,000 HOURS

Specimen No.	Condition (Exposure Temp. and Time)	Mean Corrosion Depth (μm)			Summary of Internal Structure
		Oxidation	Alloy Depletion	Heavy Carbide Precipitation (Carburization)	
Manaurite 36XS (291)	As-cast	--	--	--	Fe-Ni-rich γ -matrix (austenite) containing primary eutectic carbides at the dendrite boundaries. Eutectic structure is fairly coarse and weblike.
Manaurite 36XS (29145)	800°C (10,000 h)	16	42	N.D. ^(a)	Secondary carbides have precipitated out in the intradendritic regions in two forms, spherical and acicular. The acicular form, as shown in Fig. 3-28b, seems to lie preferentially on certain planes. Some of the spherical secondary carbides have already coarsened. The number density of secondary precipitates is lower than in Manaurite 36X. The acicular carbides were more dense after 3000 h at 800°C but by 10,000 h most of them had coarsened or dissolved.
Manaurite 36XS (29147)	900°C (9470 h)	36	57	275	Number density of secondary carbides lower than at 800°C. Acicular secondary carbides have all disappeared by 10,000 h. Primary carbides have coarsened lengthwise, nearly outlining the grain boundaries. Secondary carbides have precipitated out at the grain boundaries. Very little coarsening of the intradendritic secondary carbides has taken place. A dark etching second phase is visible at the grain boundaries in a few areas.
Manaurite 36XS (29149)	1000°C (9005 h)	14	35	191	Very few intradendritic carbides are left. The primary carbides have coarsened and interconnected in many places. The dark-etching second phase is very common and is always located in a primary carbide.

^(a) N.D. = not determinable.

MANAURITE 36XS (291)

500X

- (a) MICROSTRUCTURE SHOWS PRIMARY EUTECTIC CARBIDES AT DENDRITE BOUNDARIES

MANAURITE 36XS (29149) MURAKAMI'S ETCH 500X

- (b) MICROSTRUCTURE SHOWS COARSENEED PRIMARY EUTECTIC CARBIDES AT DENDRITE BOUNDARIES WITH PATCHES OF DARK ETCHING SECOND PHASE. ALSO INTRADENDRITIC SECONDARY CARBIDES ARE PRESENT

Fig. 3-30. Optical photomicrographs showing internal microstructure of Manaurite 36XS in the as-cast condition (a) and after 10,000-h exposure to impure helium at 1000°C

EXTERNAL
SCALE →

CARBURIZATION
DEPTH
~ 275 μm

MANAURITE 36XS (29147) DIC 250X

MICROSTRUCTURE SHOWS TWO LAYERED SCALE,
DEPLETED ZONE, CARBURIZATION ZONE, AND
INTERNAL MICROSTRUCTURE. GREY AREA
ABOVE SCALE IS ELECTRODEPOSITED NICKEL.

Fig. 3-31. Optical photomicrograph (DIC) showing near surface region including layered scale, alloy depletion zone, carburization zone and internal microstructure after 10,000-h exposure in impure helium at 900°C

temperature increased from 800° to 900°C. However, because of the spallation that occurred at 1000°C, the measured depth of oxidation was only 14 μm . The alloy depletion depth followed the same pattern, increasing from 42 to 57 μm and then decreasing to 35 μm at 1000°C. Because of the high carbon content of Manaurite 36XS, carburization depth measurements for 800°C were difficult. The measurements at 900° and 1000°C were 275 and 191 μm , respectively.

Surface Condition. SEM photomicrographs of Manaurite 36XS are shown in Fig. D-8 of Appendix D. As seen in Fig. D-8b, the scale that formed at 1000°C had mostly spalled off and then reformed in some locations. At 900°C the two-layer scale remained nearly intact up to 10,000 h.

The scale at 900°C was composed of an outer scale of Cr-rich carbide and an inner Si-rich oxide scale. The near-surface primary carbides were Cr rich. The scale at 1000°C had approximately the same composition as at 900°C, except that the outer scale contained more base alloy elements.

3.6.1.5. Manaurite 900

Detailed metallurgical analysis for alloy Manaurite 900 is presented in Table 3-11. Figure 3-32 shows the as-cast structure and the microstructure after 10,000-h exposure at 1000°C. Selected results of the SEM/EDX analysis are shown in Table D-12 and Fig. D-9 of Appendix D.

Internal Microstructure. The as-cast microstructure of Manaurite 900 consisted of a Fe-Ni-rich γ -matrix (austenite) containing coarse primary eutectic carbides. Numerous casting defects were observed throughout the structure.

After thermal aging in impure helium, the primary eutectic carbides coarsened. At 900°C a few secondary carbides precipitated at the grain

TABLE 3-11
SUMMARY OF SURFACE CORROSION MEASUREMENTS AND INTERNAL
MICROSTRUCTURAL ANALYSIS FOR ALLOY MANAURITE 900 UNSTRESSED IN CONTROLLED-
IMPURITY HELIUM FOR 10,000 HOURS

Specimen No.	Condition (Exposure Temp. and Time)	Mean Corrosion Depth (μm)			Summary of Internal Structure
		Oxidation	Alloy Depletion	Heavy Carbide Precipitation (Carburization)	
Manaurite 900 (301)	As-cast	--	--	--	Fe-Ni-rich γ -matrix (austenite) containing primary eutectic carbides at the dendrite boundaries. Eutectic structure is very coarse and weblike. Numerous casting defects are present.
Manaurite 900 (30146)	800°C (9675 h)	5.5	28	N.D. ^(a)	No secondary carbides visible. Some coarsening of primary carbides has taken place.
Manaurite 900 (30143)	900°C (9470 h)	24	66	286	Some secondary carbides have precipitated both intra- and interdendritically. Blocky precipitates have begun to grow at the grain boundaries.
Manaurite 900 (30156)	1000°C (7075 h)	2.0	95	N.D.	Intradendritic secondary carbides have condensed into small blocky particles. Large angular precipitates have grown at grain boundaries. A dark-etching second phase has developed at some of the grain boundaries.

^(a)N.D. = not determinable.

MANAURITE 900 (301) OXALIC ACID ETCH 500X

(a) MICROSTRUCTURE SHOWS PRIMARY
EUTECTIC CARBIDES AT DENDRITE
BOUNDARIES

MANAURITE 900 (30156) MURAKAMI'S ETCH 500X

(b) MICROSTRUCTURE HAS PRIMARY
EUTECTIC CARBIDES AND SECONDARY
CARBIDES

Fig. 3-32. Optical photomicrographs showing as-cast microstructure of Manaurite 900 (a) and microstructure after 7075-h exposure in impure helium at 1000°C (b)

boundaries and within the grains themselves. After 10,000 h at 1000°C the intradendritic carbides had coarsened into optically resolvable particles, and large blocky carbides had formed at the grain boundaries. A dark-etching second phase also formed at the grain boundaries at 1000°C.

Oxidation depth was small at 800°C (5.5 μm), increased at 900°C (24 μm), and decreased to ~ 2 μm at 1000°C. The alloy depletion depth increased at each test temperature to a maximum of 95 μm at 1000°C. As with the other cast alloys, carburization depths were difficult to measure. The only depth measurable was for 900°C (286 μm).

Surface Condition. A SEM photomicrograph of the Manaurite 900 scale at 900°C is shown in Fig. D-9, Appendix D. The scale at 800°C was very thin and even and was composed of a dark-etching oxide. At 900°C the scale had two layers; the outer layer was a Cr-rich carbide rich in Ni and Fe and the inner layer was a Si-rich oxide. After 10,000-h exposure at 1000°C, much of the scale had spalled. Figure 3-33 shows an optical photomicrograph of the scale at 1000°C.

3.6.2. Wrought Solid-Solution-Strengthened Alloys

The four wrought alloys all exhibited similar initial microstructures. The grains were large and heavily twinned. Hastelloy X contained large globular carbides and a low density of smaller carbides precipitated intragranularly. The other three alloys exhibited a lower number density of carbides. After thermal aging secondary precipitates appeared both intragranularly and intergranularly. At higher aging temperatures the carbides coarsened and many redissolved in the alloy matrix.

The scales on all four alloys were duplex in morphology. Hastelloy X and Hastelloy S both showed spallation at 1000°C, while RA 333 did not. Hastelloy X also showed spallation at 900°C.

MANAURITE 900 (30156)

MURAKAMI'S ETCH

1000X

SURFACE SCALE SHOWING SPALLED Cr-RICH
CARBIDE OUTER SCALE AND Si-RICH OXIDE
INNER SCALE (DARK ETCHING). WHITE AREA
ABOVE SCALE IS ELECTRODEPOSITED NICKEL.

Fig. 3-33. Optical photomicrograph of Manaurite 900 scale after 7075-h exposure in impure helium at 1000°C

3.6.2.1. Hastelloy X

Detailed metallurgical analysis for alloy Hastelloy X is presented in Table 3-12. Figures 3-34 and 3-35 show the microstructure as a function of aging temperature. Selected results of the SEM/EDX analyses are shown in Tables D-14 and D-15 and Fig. D-10 of Appendix D.

Internal Microstructure. The as-received microstructure of Hastelloy X consisted of large-grained, Ni-rich γ -matrix (austenite) containing coarse globular, intragranular and intergranular, primary M_6C -type carbides and very fine $M_{23}C_6$ carbides dispersed intragranularly and intergranularly and around primary carbides.

After aging in impure helium a dispersion of fine, discrete precipitates appeared both intragranularly and intergranularly. Many of the twin boundaries were also decorated with carbides. After 10,000 h at 800°C the intragranular carbides had become coarsened to an appreciable extent. At 900°C most of the intragranular precipitates had coalesced and/or coarsened. The initial large globular carbides remained relatively unaffected. Even greater coalescence and coarsening took place after exposure at 1000°C.

The depth of oxidation, alloy depletion, and carburization increased as the exposure temperature was increased from 800° to 1000°C. The carburization depth at 900°C was 930 μm , almost one-third of the distance to the center of the specimen. After exposure at 1000°C the carburization extended to the center of the specimen (3175 μm).

Surface Condition. SEM photomicrographs of the surface scale on Hastelloy X after exposure to impure helium at 800° and 900°C are shown in Fig. D-10 and detailed in Tables D-14 and D-15 of Appendix D.

TABLE 3-12
SUMMARY OF SURFACE CORROSION MEASUREMENTS AND INTERNAL
MICROSTRUCTURAL ANALYSIS FOR ALLOY HASTELLOY X AGED UNSTRESSED IN CONTROLLED-
IMPURITY HELIUM FOR 10,000 HOURS

Specimen No.	Condition (Exposure Temp. and Time)	Mean Corrosion Depth (μm)			Summary of Internal Structure
		Oxidation	Alloy Depletion	Heavy Carbide Precipitation (Carburization)	
Hastelloy X (086)	As-received	--	--	--	Ni-rich γ-matrix (austenite) containing large and small, globular primary carbides heterogeneously dispersed intra- and intergranularly, and finer, less extensive carbides dispersed intergranularly and surrounding the large globular carbides.
Hastelloy X (08646)	800°C (10,000 h)	19	44	375	Same morphology as the as-received specimen except that discrete, very fine, precipitated carbides are dispersed intra- and intergranularly and on twin boundaries. Some coarsening of intergranular precipitates has occurred.
Hastelloy X (08648)	900°C (10,000 h)	28	76	930	Extensive coalescence and coarsening of intra- and intergranular precipitates has taken place. Grain boundaries outlined by series of large blocky precipitates.
Hastelloy X (08649)	1000°C (Still in test)	51	89	To center of specimen	Coalescence and coarsening has resulted in a series of large blocky carbides at grain boundaries. Many of the intra-granular carbides have dissolved into matrix.

HASTELLOY X (086) OXALIC-HCl ACID ETCH 500X

- (a) MICROSTRUCTURE SHOWS LARGE TWINNED GRAINS WITH LARGE GLOBULAR CARBIDES AND SMALLER INTRAGRANULAR CARBIDES. SMALL NUMBER OF INTERGRANULAR CARBIDES.

HASTELLOY X (08646) MURAKAMI'S ETCH 500X

- (b) MICROSTRUCTURE SHOWS PRECIPITATED INTRA- AND INTERGRANULAR CARBIDES AND LARGE GLOBULAR PRIMARY CARBIDES

Fig. 3-34. Optical photomicrographs showing internal microstructure of Hastelloy X in the as-received condition (a) and after 10,000-h exposure in impure helium at 800°C (b)

HASTELLOY X (08648) MURAKAMI'S ETCH 500X

(a) MICROSTRUCTURE SHOWS COARSENED
INTRA- AND INTERGRANULAR
CARBIDES

HASTELLOY X (08650) MURAKAMI'S ETCH 500X

(b) MICROSTRUCTURE SHOWS HEAVY
COARSENED INTERGRANULAR
CARBIDES AND COALESCED AND
COARSENED INTRAGRANULAR
CARBIDES

Fig. 3-35. Optical photomicrographs of internal microstructure of Hastelloy X after 10,000-h exposure in impure helium at 900°C (a) and 1000°C (b)

The scales at both temperatures were composed of two layers. The outer layer was a Cr-rich carbide containing small amounts of Ni, Fe, Si, and Mo. The inner layer, often interconnected with the outer layer, was composed of a Cr-Mn-rich oxide. Large primary Mo-rich carbides were present in the near-surface region. The scale at 900°C showed that spallation had taken place. A Mn-rich oxide was also present throughout the scale.

3.6.2.2. Hastelloy S

Detailed metallurgical analysis for the alloy Hastelloy S is presented in Table 3-13. Figures 3-36 and 3-37 show the microstructure of Hastelloy S in the as-received condition and after thermal aging. Selected results of the SEM/EDX analysis are shown in Table D-16 and Fig. D-11 of Appendix D.

Internal Microstructure. The as-received microstructure of Hastelloy S consisted of a Ni-rich γ -matrix (austenite) containing virtually no optically visible carbides. The grains were large and heavily twinned.

After thermal aging at 800°C secondary carbides appeared both intragranularly and intergranularly. At 900° and 1000°C the intragranular carbides dissolved and the blocky carbides at the grain boundaries began to coarsen.

The depth of oxidation was about the same at 800° and 900°C (15 μm) and increased to a value of 33 μm at 1000°C. The alloy depletion depth, as well as the carburization depth, was less at 900°C than at 800°C. The values for alloy depletion depth and carburization depth at 1000°C were not determinable.

TABLE 3-13
SUMMARY OF SURFACE CORROSION MEASUREMENTS AND INTERNAL
MICROSTRUCTURAL ANALYSIS FOR ALLOY HASTELLOY S AGED UNSTRESSED IN CONTROLLED-
IMPURITY HELIUM FOR 10,000 HOURS

Specimen No.	Condition (Exposure Temp. and Time)	Mean Corrosion Depth (μm)			Summary of Internal Structure
		Oxidation	Depletion	Heavy Carbide Precipitation (Carburization)	
Hastelloy S (341)	As-received	--	--	--	Ni-rich γ -matrix (austenite) containing virtually no optically visible carbides. Grains are large and heavily twinned.
Hastelloy S (34145)	800°C (10,000 h)	16	25	762	Secondary carbides have precipitated out as blocky carbides at grain boundaries. Also some carbides have precipitated out intragranularly.
Hastelloy S (34147)	900°C (10,000 h)	15	15	381	Carbides have coarsened at grain boundaries and the number density of carbides is lower than at 800°C. Intragranular carbides have almost all gone into solution.
Hastelloy S (34149)	1000°C (9935 h)	33	N.D.	N.D. ^(a)	Grain boundary carbides have grown to very large size. Very few carbides in center of grains.

^(a)N.D. = not determinable.

HASTELLOY S (341) HCl-OXALIC ACID ETCH 500X

(a) MICROSTRUCTURE SHOWS LARGE,
HEAVILY TWINNED GRAINS
CONTAINING PRIMARY CARBIDES

HASTELLOY S (34145) MURAKAMI'S ETCH 500X

(b) MICROSTRUCTURE SHOWS PRECIPITATED
SECONDARY CARBIDES

Fig. 3-36. Optical photomicrographs of Hastelloy S in the as-received condition (a) and after 10,000-h exposure in impure helium at 800°C (b)

HASTELLOY S (34147) MURAKAMI'S ETCH 500X

(a) MICROSTRUCTURE SHOWS COARSENE
SECONDARY CARBIDES LOCATED
MOSTLY AT GRAIN BOUNDARIES

HASTELLOY S (34149) MURAKAMI'S ETCH 500X

(b) MICROSTRUCTURE SHOWS GREATLY
COARSENE SECONDARY CARBIDES

Fig. 3-37. Optical photomicrographs showing internal microstructure of Hastelloy S after 10,000-h exposure in impure helium at 900° (a) and 1000°C (b)

Surface Condition. The scale on Hastelloy S was relatively thin at all exposure temperatures making composition analysis impossible by standard techniques. A large number of subscale oxide particles were present. The outer scale at 1000°C was composed mostly of Mo-rich carbides.

3.6.2.3. RA 333

Detailed metallurgical analysis for alloy RA 333 is presented in Table 3-14. Figures 3-38 and 3-39 show the internal microstructure as a function of exposure temperature. Selected results of the SEM/EDX analysis are shown in Table D-16 and Fig. D-12 of Appendix D.

Internal Microstructure. The as-received microstructure of RA 333 consisted of large, heavily twinned grains, with relatively small amounts of primary carbides. After exposure for 10,000 h at 800°C, secondary carbides precipitated intragranularly and intergranularly. Many of the grain boundaries were completely outlined.

After exposure at 900°C most of the intragranular carbides had dissolved into the matrix and the few that remained had coarsened. Intergranular carbides had coarsened greatly. After exposure at 1000°C most of the intragranular carbides were dissolved into the matrix. The intergranular carbides were very large and blocky.

As the thermal aging temperature was increased, the oxidation depth, the alloy depletion depth, and the carburization depth all increased. The depth of carburization increased dramatically from 97 μm at the 800°C exposure temperature to 720 μm at the 900°C exposure temperature. The depth at 1000°C was not determinable. Figure 3-40 shows the alloy depletion depth and the surface scale after 10,000 h at 1000°C.

Surface Condition. The surface scale on RA 333 remained intact even after 10,000 h at 1000°C as shown in Fig. D-12, Appendix D. The thick outer

TABLE 3-14
SUMMARY OF SURFACE CORROSION MEASUREMENTS AND INTERNAL
MICROSTRUCTURAL ANALYSIS FOR ALLOY RA 333 AGED UNSTRESSED IN CONTROLLED-
IMPURITY HELIUM FOR 10,000 HOURS

Specimen No.	Condition (Exposure Temp. and Time)	Mean Corrosion Depth (μm)			Summary of Internal Structure
		Oxidation	Alloy Depletion	Heavy Carbide Precipitation (Carburization)	
RA 333 (311)	As-received	--	--	--	Ni-rich γ -matrix (austenite) consisting of large, heavily twinned grains and intra- and intergranular primary carbides.
RA 333 (31146)	800°C (10,000 h)	14	32	97	Very heavy secondary carbide precipitation has taken place both intra- and intergranularly. Carbides are relatively large and appear to have begun coarsening.
RA 333 (31148)	900°C (10,000 h)	84	127	720	Very few intragranular carbides left. Grain boundary carbides have coarsened into large particles.
RA 333 (31150)	1000°C (9935 h)	127	140	N.D. ^(a)	Great amounts of coarsening very evident. Very large blocky carbides are present at the grain boundaries.

^(a)N.D. = not determinable.

RA 333 (311) HCl-OXALIC ACID ETCH 500X

(a) MICROSTRUCTURE SHOWS LARGE, HEAVILY TWINNED GRAINS WITH LARGE HOMOGENEOUSLY DISPERSED PRIMARY CARBIDES

RA 333 (31146) MURAKAMI'S ETCH 500X

(b) MICROSTRUCTURE SHOWS SECONDARY CARBIDE LOCATED BOTH INTER- AND INTERGRANULARLY

Fig. 3-38. Optical photomicrographs of the internal microstructure of RA 333 in the as-received condition (a) and after 10,000-h exposure in impure helium at 800°C (b)

RA 333 (31148) MURAKAMI'S ETCH 500X

(a) MICROSTRUCTURE SHOWS LARGE
COARSENEED GRAIN BOUNDARY
CARBIDES AND INTRAGRANULAR
CARBIDES

RA 333 (31150) MURAKAMI'S ETCH 500X

(b) MICROSTRUCTURE SHOWS LARGE
COARSENEED GRAIN BOUNDARY
CARBIDES AND INTRAGRANULAR
CARBIDES

Fig. 3-39. Optical photomicrographs of the internal microstructure of RA 333 after 10,000-h exposure in impure helium at 900° (a) and 1000°C (b)

RA 333 (31150)

DIC

250X

MICROSTRUCTURE SHOWS TWO LAYERED EXTERNAL
SCALE, ALLOY DEPLETION ZONE, AND INTERNAL
CARBIDES

Fig. 3-40. Optical DIC photomicrograph of RA 333 after 10,000-h exposure in impure helium at 1000°C

scale was composed of a Cr-rich carbide, while the inner scale was mostly a Si-rich oxide. The near-surface secondary carbides were Cr-rich with large amounts of Mo.

3.6.2.4. HD 556

Detailed metallurgical analysis of alloy HD 556 is presented in Table 3-15. Figures 3-41 and 3-42 show the internal microstructure of HD 556 in the as-received condition and after 10,000 h in impure helium at 800° and 900°C. Selected results of the SEM/EDX analysis are shown in Table D-17 and Fig. D-13 of Appendix D.

Internal Microstructure. The as-received microstructure of HD 556 consisted of large, heavily twinned grains. Large primary carbides were present throughout the structure. Very few smaller primary carbides were visible. After exposure for 10,000 h at 800°C a continuous network of carbides had precipitated at the grain boundaries. Small intragranular precipitates, as well as the large primary carbides, were present.

After exposure at 900°C the intergranular carbides had coarsened into a series of discrete particles. Few intragranular carbides remained. The density of secondary intragranular carbides after exposure at 1000°C was greater than after exposure at 900°C. The intergranular carbides at 1000°C were more coarsened than after exposure at 900°C.

The oxidation depth increased with increased test temperature varying from 14 to 83 μm . The alloy depletion depth was 38 μm at 800°C, peaked at 98 μm at 900°C, and declined slightly to 83 μm at 1000°C. Only the 1000°C carburization depth was determinable, and the specimen was carburized all the way to the center (3175 μm).

Surface Condition. The surface scale on HD 556 was composed of two layers at both 800° and 900°C. The scale at 800°C is shown in Fig. D-13,

TABLE 3-15
SUMMARY OF SURFACE CORROSION MEASUREMENTS AND INTERNAL
MICROSTRUCTURAL ANALYSIS FOR ALLOY HD 556 AGED UNSTRESSED IN CONTROLLED-
IMPURITY HELIUM FOR 10,000 HOURS

Specimen No.	Condition (Exposure Temp. and Time)	Mean Corrosion Depth (μm)			Summary of Internal Structure
		Oxidation	Alloy Depletion	Heavy Carbide Precipitation (Carburization)	
HD 556 (391)	As-received	--	--	--	Ni-rich γ -matrix (austenite) containing large, heavily twinned grains. Large primary carbides are randomly distributed throughout the structure.
HD 556 (39145)	800°C (10,000 h)	14	38	N.D. ^(a)	Grain boundaries have become outlined by lines of carbides. Large primary carbides and smaller secondary carbides occupy intragranular sites. Some of the secondary carbides lie preferentially on twin boundaries.
HD 556 (39147)	900°C (10,000 h)	31	98	N.D.	Carbides at grain boundaries have coarsened resulting in discrete carbides at grain boundaries. Many of the intragranular carbides have dissolved into matrix.
HD 556 (39149)	1000°C (10,000 h)	83	83	To center of specimen	Intergranular carbides have coarsened into strings of large blocky carbides. Secondary precipitates appear to be more numerous than after exposure at 900°C.

^(a)N.D. = not determinable.

HD 556 (391) OXALIC-HCl ETCH 500X

(a) MICROSTRUCTURE SHOWS LARGE
TWINNED GRAINS WITH LARGE
PRIMARY CARBIDES AT ETCH PIT
SITES

HD 556 (39145) MURAKAMI'S ETCH 500X

(b) MICROSTRUCTURE SHOWS LARGE
INTRAGRANULAR PRIMARY CARBIDES
AND PRECIPITATED SECONDARY
CARBIDES AT GRAIN AND TWIN
BOUNDARIES

Fig. 3-41. Optical photomicrographs of HD 556 showing as-received microstructure (a) and microstructure after 10,000-h exposure in impure helium at 800°C (b)

HD 556 (39147) MURAKAMI'S ETCH 500X

(a) MICROSTRUCTURE SHOWS FEW INTRA-GRANULAR PRIMARY CARBIDES AND COARSENEED SECONDARY CARBIDES AT GRAIN BOUNDARIES

HD 556 (39149) MURAKAMI'S ETCH 500X

(b) MICROSTRUCTURE SHOWS VERY LARGE COARSENEED INTERGRANULAR CARBIDES AND SECONDARY INTRAGRANULAR CARBIDES

Fig. 3-42. Optical photomicrographs of internal microstructure of HD 556 after 10,000-h exposure in impure helium at 900° (a) and 1000°C (b)

Appendix D. At 800°C the outer portion of the scale was a Cr-rich carbide and the inner part of the scale was a Cr-rich oxide. The near-surface carbides were Mo-W-rich.

3.6.3. Summary of Oxidation, Corrosion, and Carburization Depth Results

The metallurgical data for Group II alloys after 3000 and 10,000 h exposures in impure helium are summarized in Table 3-16. In many cases carburization depths were not determinable by standard optical techniques.

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TABLE 3-16
SUMMARY OF OXIDATION, CORROSION, AND CARBURIZATION DEPTH MEASUREMENTS^(a)
FROM CONTROLLED-IMPURITY HELIUM AGING TESTS CONDUCTED AT CIIR

Alloy	Temp (°C)	3000 Hour			10,000 Hour					
		Mean Oxidation	Mean Corrosion Depth	Mean Carburization Depth	Oxidation		Corrosion		Carburization	
					Mean	Max	Mean	Max	Mean	Max
HK 40	800	6.4	16	N.D. ^(b)	8	--	14	19	N.D.	N.D.
	900	14	16	N.D.	19	32	38	--	N.D.	N.D.
	1000	3.2	14	N.D.	19	--	127	--	N.D.	N.D.
Supertherm	800	14	19	57	13	16	22	--	N.D.	N.D.
	900	16	25	N.D.	20	--	35	41	N.D.	N.D.
	1000	44	19	N.D.	5	--	22	--	N.D.	N.D.
Manaurite 36X	800	11.4	19	N.D.	7.6	19	30	38	214	305
	900	22.2	35	N.D.	18	22	32	45	254	--
	1000	12.7	12.7	N.D.	6	--	41	51	275	381
Manaurite 36XS	800	22.2	29	114	16	23	42	57	N.D.	N.D.
	900	25.4	51	178	36	41	57	76	275	316
	1000	12.7	19	N.D.	14	25	35	51	191	222
Manaurite 900	800	6.4	16	102	5.5	--	28	--	N.D.	N.D.
	900	9.5	19	N.D.	24	--	66	76	286	381
	1000	6.4	19	N.D.	2.0	--	95	127	N.D.	N.D.
Hastelloy X	800	9.5	16	114	19	--	44	--	375	508
	900	25.4	51	318	28	--	76	--	930	1016
	1000	25.4	32	1143	51	--	89	--	Center	--
Hastelloy S	800	9.5	19	254	16	--	25	--	889	--
	900	19	19	762	15	25	15	25	Center	381
	1000	29	29	To center	33	44	N.D.	N.D.	N.D.	N.D.
RA 333	800	6.4	16	N.D.	14	25	32	--	97	152
	900	38	89	254	84	127	127	140	720	--
	1000	16	114	1270	127	140	140	165	N.D.	N.D.
HD 556	800	9.5	29	N.D.	14	--	38	--	N.D.	N.D.
	900	41	83	To center	31	38	98	--	N.D.	N.D.
	1000	35	44	To center	83	--	83	--	Center	--

(a) All depth measurements are in microns.

(b) N.D. = not discernible.

SECRET

4. DISCUSSION OF RESULTS

The results of the microstructural examinations and mechanical tests performed on Group II alloys are discussed below.

4.1. THERMAL AGING AND ENVIRONMENTAL EFFECTS

The microstructural changes that occurred during the exposure of specimens to impure helium at elevated temperatures resulted from thermal aging and helium impurity - metal interactions.

4.1.1. Thermal Aging

Internal microstructural changes occurred in all nine alloys independently of the helium impurity - metal interactions that took place. These changes were the result of thermal aging.

The five cast alloys all exhibited similar initial microstructures and experienced similar internal microstructural changes during thermal aging at 800°, 900°, and 1000°C. The as-cast microstructures consisted of large dendrites containing lace-like or script-type eutectic primary carbides dispersed interdendritically within an austenitic matrix. Thermal aging resulted in the coalescence and coarsening of the primary carbides and precipitation of secondary carbides, which coalesced and coarsened at the higher aging temperatures (900° and 1000°C).

A dark-etching second phase, possibly σ , formed on the primary eutectic carbides of HK 40 and Manaurites 36X, 36XS, and 900.

The four wrought alloys, Hastelloy X, Hastelloy S, RA 333, and HD 556, also exhibited similar initial microstructures. The grains were large and heavily twinned. All four contained a bimodal distribution of large and small primary carbides. The effect of thermal aging in all four alloys was the same. At 800°C intragranular and intergranular carbides precipitated. At 900°C the carbides began to coalesce and coarsen, and their number density decreased. After exposure at 1000°C very few intragranular carbides were present and the intergranular carbides had coarsened greatly.

4.1.2. Helium Impurity - Metal Interactions

Interactions between the alloys and impurities in the helium environment occurred at all exposure temperatures. They resulted in surface oxide and carbide scale formation, internal oxidation, alloy depletion near the surface, and high-density carbide precipitation (carburization) extending into the matrix.

4.1.2.1. Oxidation and Scale Formation

The surface conditions of the alloys after 10,000-h exposure at 800°, 900°, and 1000°C were characterized by single or multilayered surface oxide or carbide/oxide scales, intruding internal oxides, and alloy-depleted regions. Zones of high-density carbide precipitation extending from the surface inward also occurred as discussed in the following section. The types and degrees of surface scale and internal phase formation varied with alloy type, temperature, and exposure time.

In general, all of the Group II alloys exhibited surface scales, which increased in thickness with increasing temperature from 800° to 900°C. Alloys HK 40; Manaurites 36X, 36XS, and 900; and Supertherm showed clear signs of spallation during the course of their 10,000-h exposure at 1000°C, resulting in smaller scale thicknesses for this temperature

in comparison with those observed at 900°C. Alloys Hastelloy X, Hastelloy S, HD 556, and RA 333 exhibited adherent surface scales at 1000°C, which were the same or significantly greater in thickness than those observed at lower temperature.

Figures 4-1 through 4-6 show typical results of oxidation and corrosion (alloy depletion) depth measurements for all Group II alloys as functions of exposure time and temperature. In general, all of the centrifugally cast alloys exhibited oxidation and corrosion depths that increased with increasing temperature between 800° and 900°C but decreased at 1000°C. Unlike the wrought alloys, oxidation of the cast alloys involved only the formation of surface oxide scales without significant internal oxidation and, therefore, values for these alloys were strongly affected by scale losses (spallation) that occurred during their exposure. With only few exceptions, the oxidation and corrosion depths for the wrought alloys increased between 800° and 900°C and increased or remained nearly the same as the 900° values after exposure at 1000°C. The behavior at the highest exposure temperature may be attributed to the formation of less permeable (to oxygen) surface scales at 1000°C.

In general, oxidation and corrosion depths for the wrought alloys increased with increasing exposure time for all exposure temperatures, while nonuniform or erratic behavior was exhibited by the cast alloys due to spallation. All nine of the Group II alloys formed surface scales of similar morphology at 800°, 900°, and 1000°C but with differing compositions. The centrifugally cast alloys exhibited multilayered scales consisting of outer Cr-rich carbide and inner Si-rich oxide at all exposure temperatures. Inner oxide scales on these alloys also contained significant Cr at 800°C which was not apparent at the higher exposure temperatures. Layered surface scales formed on the wrought alloys contained outer Cr-rich carbides and inner Cr-rich oxides. Inner oxide scales observed for alloy Hastelloy X additionally contained significant amounts of Mn (20 to 30 wt %).

Fig. 4-1. Oxidation depth as a function of time for Group II alloys exposed in controlled-impurity helium at 900°C

Fig. 4-2. Oxidation depth as a function of time for Group II alloys aged unstressed in controlled-impurity helium at 1000°C

Fig. 4-3. Corrosion depth (alloy depletion) as a function of time for Group II alloys exposed in controlled-impurity helium at 900°C

Fig. 4-4. Corrosion depth (alloy depletion) as a function of time for Group II alloys aged unstressed in controlled-impurity helium at 1000°C

Fig. 4-5. Oxidation depth as a function of temperature for Group II alloys exposed to controlled impurity helium for 10,000 h

Fig. 4-6. Corrosion depth as a function of temperature for Group II alloys exposed to controlled-impurity helium for 10,000 h

The alloys RA 333, HK 40, Supertherm, and Hastelloy S contained areas of large Mg content in their scales after exposure at 1000°C. None of the four alloys was expected to contain much Mg. Supertherm was analyzed spectrographically and found to contain less than 0.006 wt % Mg. The origin of the Mg in the scales of the Group II alloys is unknown.

4.1.2.2. Carburization. A major result of the interaction between the alloys and impurities in the helium environment and the most significant in terms of alloy degradation was carburization. This phenomenon occurred to some extent in all of the alloys, manifesting itself as a zone of high-density precipitated carbides near the surface and often extending deep into the alloy, and resulting in increases in bulk carbon content of each alloy specimen. The precipitated carbides appeared both intragranularly and intergranularly (or intradendritically and interdendritically in the cast alloys) and were accompanied by increased alloy depletion adjacent to grain boundaries in comparison with that produced by thermal aging. The precipitated carbides generally appeared coarser and more numerous at the higher temperatures, and in some cases (e.g., Manaurite 900) intergranular carbides near the surface became nearly continuous.

In general, all of the alloys exhibited carburization although for some alloys, especially HK 40 and Supertherm, carburization could not be determined by optical metallographic techniques, a difficulty primarily related to the high carbon content of these alloys. Except for these alloys and Manaurite 36XS, all of the alloys exhibited increasing depths of carburization and carbon content with increasing time and temperature (see Table 3-16). This behavior is related to the increased reaction of the alloys with the carbon-bearing impurities in the helium environment (CO , CH_4) and/or increased potential of the alloy to accept carbon. The latter is primarily related to the solubility and diffusion coefficient

of carbon that normally increases with increasing temperature. The presence and nature of the surface scales observed on the alloys may have also strongly influenced the carburization behavior. Manaurite 36XS appeared to show more carburization at 900°C than at 1000°C; however, the difference in the measured values for these two temperatures is quite small and may well be within the errors involved in measuring depths for such a high-carbon alloy.

Because of the difficulties in measuring carburization depths for many of the alloys, meaningful graphical presentations of carburization versus temperature or time for each alloy were not possible to summarize their carburization behavior. However, bulk carbon content measurements of the exposed alloys offer an excellent means of determining the relative behavior of the alloys. The alloys aged at CIIR ranged in carbon content from 0.007% to 0.47%. The change in bulk carbon content for any temperature and time is a measure of the carburization potential for a material. It was generally noted that those alloys with a low initial carbon content readily absorbed larger amounts of carbon from the HTGR environment than those with a higher initial concentration. This is shown graphically in Figs. 4-7 and 4-8 for 3000- and 10,000-h exposures, respectively. Hastelloy S contained 0.007% C in the as-received condition and after 10,000 h at 800°C the bulk carbon content had increased by 80%. Alloys Hastelloy X and HD 556 had comparable as-received carbon contents of 0.080% and 0.095%, respectively, and were increased by 60% and 53%, respectively, after 10,000-h exposure at 900°C. Those alloys with higher as-received bulk carbon contents picked up the least amount of carbon. Manaurite 36SX, Manaurite 900, Supertherm, and HK 40, all exhibited less than a 3% increase in bulk carbon content at 800°C. As the temperature increased, the amount of carbon content increased except in the case of Manaurite 36XS, which exhibited a 34% increase at 900°C and a 23% increase at 1000°C. This particular alloy registered a weight loss of 1.42 mg/cm^2 , and a macroscopic evaluation of the surface revealed spalling to have

Fig. 4-7. Change in bulk carbon content versus exposure temperature for Group II alloys aged 3000 h in controlled-impurity helium at GA

Fig. 4-8. Percent change in bulk carbon content versus exposure temperature aged for 10,000 h at CIIR

occurred. It is suggested the percent carbon concentration decrease from 900° to 1000°C is due to spalling of an outer carbide layer that would have added to the bulk carbon content.

The carburization behavior of the alloys and their relative susceptibility to carbon pickup can be related to differences in surface catalysis of the decomposition of carbon-bearing impurities, matrix carbon solubility, and carbon diffusivity, and the effect of surface corrosion scales on carbon transport.

For carburization to occur in a metal, a carbon activity gradient must exist within the material to provide a driving force for carbon diffusion. The establishment of such a gradient is dependent both on the deposition of carbon at the metal surface and on the dissolution of carbon in the surface matrix. Carbon deposition is dependent on the decomposition of carbon-bearing species in the environment (CO , CH_4) and is related to the catalytic effectiveness of the metal surface. Carbon dissolution is dependent on the solubility of carbon in the alloy. If the solubility of carbon in an alloy is very small, carbon cannot be continually introduced into the matrix unless concurrent processes such as carbide precipitation, which may be very slow, can occur to deplete matrix carbon levels. Once carbon has gone into solution, diffusion processes must occur to take the carbon further into the bulk of the alloy. The presence and nature of surface scales (oxides, carbides) can also have a significant influence, since they can limit both carbon deposition by virtue of their catalytic effects and carbon diffusion by providing an effective barrier to carbon transport (Refs. 4-1 through 4-4).

4.2. EFFECT OF ENVIRONMENTALLY AFFECTED ZONE AND BULK MICROSTRUCTURES ON THE MECHANICAL PROPERTIES OF GROUP II ALLOYS

The effects of thermally aged and environmentally induced microstructural changes on the mechanical properties, such as creep strain,

tensile properties, and fracture behavior, of the Group II alloys are discussed in this section.

Creep strains as a function of time can be compared for the alloys tested in Figs. 4-9 through 4-11. At all temperatures RA 333 and Hastelloy S exhibited creep strains significantly higher than those for all other alloys. These are wrought solid-solution-strengthened alloys. However, the other wrought solid-solution-strengthened alloys (Hastelloy X and HD 556) exhibited creep strains as low or lower than the centrifugally cast alloys. Thus, no effect of manufacturing process is discernible from this study.

The tensile properties are tabulated in Table 3-6. The decrease in ductility for Hastelloy X was previously attributed to carburization resulting from interaction of the alloy with impurities in the helium environment (Ref. 4-5). The loss in ductility observed for Hastelloy S at 800°C is also believed to be due to carburization based on the very large increases (>10 times as-received value) in bulk carbon content previously observed for this alloy at somewhat higher temperatures (Ref. 4-6).

It is believed that the changes in the room-temperature strength and ductility of the Group II alloys are related to a combination of the thermal aging that occurred during exposure and carburization resulting from interaction of the alloys with impurities in the controlled-impurity helium environment. The property changes observed for unstressed specimens of those alloys that exhibited small amounts of carbon uptake during exposure are believed to result primarily from thermally induced microstructural changes such as carbide precipitation. The property changes exhibited by the unstressed wrought alloys and the cast alloy Manaurite 900, which picked up relatively large amounts of carbon from the environment, resulted from both thermal aging and carburization. In the case of Hastelloy X and Hastelloy S, reductions

Fig. 4-9. Plastic creep strain versus time for Group II alloys tested in controlled-impurity helium at 800°C

Fig. 4-10. Plastic creep strain versus time for Group II alloys tested in controlled-impurity helium at 900°C

Fig. 4-11. Plastic creep strain versus time for Group II alloys tested in controlled-impurity helium at 1000°C

in tensile properties greater than those expected from thermal aging alone were observed. These alloys also exhibited significant increases in their bulk carbon contents and, therefore, it is believed that these reductions in properties can be attributed to carburization. Carburization may also be partially responsible for the reduced properties exhibited by alloys RA 333, HD 556, and Manaurite 900 since these alloys also showed significant amounts of carburization.

The similarity of impact values for air- and helium-aged specimens of the wrought and centrifugally cast alloys at 800° and 900°C indicates that the helium environment had no significant effect on the impact strengths of these alloys over the range of temperatures and times tested. The changes in impact properties from the as-received values can be attributed to well known thermal aging effects (carbide precipitation, coarsening, agglomeration, etc.).

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5. CONCLUSIONS

The conclusions discussed below were made based on the metallurgical and mechanical property evaluation results of specimens exposed for up to 10,000 h, creep test results for times up to 10,000 h, and detailed analyses of these data.

5.1. GENERAL

The most significant effects of exposure in controlled-impurity helium at 800° to 1000°C after 10,000 h are changes in microstructure caused by well known thermal aging mechanisms and by in-depth carburization of all alloys resulting from helium impurity - metal interactions. Oxidation is a less important effect in the helium environment in terms of penetration depth but may affect surface-sensitive properties. The extents of thermal aging, carburization, and oxidation are a function of temperature, time, and alloy composition.

5.2. THERMAL AGING

Microstructural changes independent of helium impurity - metal interactions occur at 800°, 900°, and 1000°C in all Group II alloys. Thermal aging of the centrifugally cast alloys results in precipitation of intradendritic and interdendritic secondary carbides, coarsening and coalescence of primary and secondary interdendritic secondary carbides, and formation of tertiary (probably TCP-type) phases. Thermal aging of the wrought solid-solution-strengthened alloys produces precipitation of fine intragranular carbides, which decrease in number density and grow larger with increasing temperature, and coarser intergranular carbides, which result in alloy-depleted regions near grain

boundaries. The extent of the microstructural changes in all of the alloys increases with increasing temperature, and these changes become more pronounced with increase in exposure time from 3000 to 10,000 h.

5.3. CARBURIZATION

Significant carburization occurs in all Group II alloys at 800°, 900°, and 1000°C after exposure in the controlled-impurity helium environment. The phenomenon manifests itself for most alloys as a zone of high-density, precipitated, intragranular and intergranular (or interdendritic) carbides extending inward from the surface and is accompanied by increases in bulk carbon content.

The alloys HK 40, Supertherm, and Manaurite 36X have the best carburization resistance of all of the alloys analyzed with respect to relative or total carbon content increase. The similar composition of these alloys, except for their higher initial carbon contents, and similar surface scale morphologies and compositions with respect to the other cast alloys, suggest that their carburization resistance is primarily related to their initially higher bulk carbon levels. The driving force for initial carbon solution in the matrix of an alloy is related to the difference between the carbon solubility limit, which is a function of alloy composition, and the initial carbon content; therefore, those alloys with a low initial carbon content may have a large driving force for carburization.

The relative ranking in carburization resistance of the wrought alloys, as well as the explanation, is more complicated. The carburization resistance of these alloys is a function of initial carbon content and alloy composition differences, which can profoundly affect carbon solubility, carbon diffusivity, and carbide precipitation rates.

In general, carburization depth and/or change in bulk carbon content (carbon pickup) increase with increasing temperature and exposure time for all Group II alloys.

5.4. SURFACE SCALE FORMATION

Surface scale formation, accompanied by surface alloy depletion, occurs in all Group II alloys after exposure in controlled-impurity helium at 800°, 900°, and 1000°C. In addition, internal oxidation occurs in many of the wrought alloys. The extent of surface scale formation and internal oxidation, and the subsequent effect on alloy degradation, is insignificant compared to carburization.

The centrifugally cast alloys formed layered Cr-rich carbide/Si-rich oxide surface scales at 800°, 900°, and 1000°C, while surface scales formed on the wrought alloys primarily consisted of duplex Cr-rich carbide/Cr- or Cr-Mn-rich oxide scales.

In general, the amount of surface scale formation and surface corrosion increases with increasing temperature and exposure time except for alloys that experience scale spallation during exposure.

5.5. MECHANICAL PROPERTIES

5.5.1. Creep

Within experimental scatter and for test times up to 10,000 h, all Group II alloys except RA 333 and Hastelloy S exhibit no reduction in creep strength in controlled-impurity helium compared to tests in air at 800° and 900°C. Controlled-impurity helium appears to increase creep rates for alloy RA 333 and decrease creep rates for alloy Hastelloy S at 800° and 900°C. In addition, the creep ductility of alloy RA 333 in controlled-impurity helium at 900°C is significantly reduced compared to its expected value in air. The reduction in creep strength for alloy RA 333 results from a combination of increased precipitation of coarse or semicontinuous intragranular and intergranular carbides due to carburization, resulting in a weakened, alloy-depleted matrix and

embrittled grain boundaries. Strengthening of alloy Hastelloy S is believed to result from carburization-induced precipitation of finer intragranular carbides.

5.5.2. Room-Temperature Impact and Tensile Properties

The impact strengths of all Group II alloys are reduced from as-received values after aging in controlled-impurity helium or air at 800° and 900°C. Values for all alloys after aging in helium and air at 800°C are substantially the same.

The yield and ultimate strengths of all Group II alloys increase from their as-received values or remain constant after aging in controlled-impurity helium or air at 800°C. Strength values for the cast Group II alloys remain the same as as-received values after aging at 900°C, while values for the wrought alloys are decreased by 900°C aging. Plastic strain to failure for all alloys is reduced from as-received values by controlled-impurity helium or air aging at 800° and 900°C.

Strength values for air- and helium-aged specimens at 800° and 900°C are comparable for all alloys. Plastic strains to failure for air- and helium-aged specimens are comparable for the cast alloys at 800° and 900°C and for the wrought alloys at 800°C. Ductilities of all wrought alloys are substantially reduced from air values by aging in helium at 900°C, the reductions being attributed to carburization.

5.6. RESIDUAL ROOM-TEMPERATURE TENSILE PROPERTIES AFTER HIGH-TEMPERATURE CREEP

The residual tensile properties of stressed specimens of the Group II alloys exhibit changes with temperature that are similar to those obtained for unstressed specimens after controlled-impurity helium aging at 800° and 900°C. Yield strength values are unchanged or

markedly lowered for the wrought alloys and unchanged for the cast alloys by prestrain in creep at 800° and 900°C, while ultimate strength and ductility (elongation and reduction in area) are reduced for all Group II alloys after creep at these temperatures. The largest strength and ductility losses observed due to prior creep at 800° and 900°C occur for those alloys that contain significant amounts of creep strain prior to room-temperature testing.

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APPENDIX A
COMPLETE CONTROLLED-IMPURITY HELIUM AND AIR CREEP DATA
FOR GROUP II ALLOYS

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture(a)
Hastelloy X	08601	Air	800	31.4	500	0.16	C
					1000	0.21	
					2000	0.22	
					3000	0.24	
					4000	0.24	
					5000	0.23	
					6000	0.26	
					7000	0.25	
					8000	0.36	
					9000	0.36	
					10000	0.36	
					11000	0.32	
					12000	0.33	
					13000	0.33	
					14000	0.37	
					15000	0.39	
					16000	0.40	
Hastelloy X	08603	Air	900	12.7	500	0.06	C
					1000	0.22	
					2000	0.24	
					3000	0.36	
					4000	0.41	
					5000	0.58	
					6000	0.61	
					8000	0.81	
					9000	1.16	
					10000	1.68	
					11000	1.90	
					12000	2.05	
					13000	2.28	
					14000	2.57	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
Hastelloy X	08605	Helium	800	31.4	500	0.00	T
					1000	0.00	
					2000	0.00	
					3000	0.01	
Hastelloy X	08614	Helium	800	31.3	500	0.16	C
					1000	0.17	
					2000	0.17	
					3000	0.15	
					4000	0.18	
					5000	0.23	
					6000	0.38	
					7000	0.39	
					8000	0.43	
					9000	0.42	
					10000	0.47	
					11000	0.48	
					12000	0.49	
Hastelloy X	08609	Helium	900	12.8	500	0.02	T
					1000	0.07	
					2000	0.22	
					3000	0.41	
Hastelloy X	08610	Helium	900	12.7	500	0.12	C
					1000	0.18	
					2000	0.26	
					3000	0.36	
					4000	0.41	
					5000	0.59	
					6000	0.70	
					7000	0.77	
					8000	0.90	
					9000	0.96	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
Hastelloy X	08611	Helium	1000	4.8	10000	1.13	T
					11000	1.27	
					12000	1.51	
					500	0.02	
					1000	0.18	
					2000	0.57	
Hastelloy X	08612	Helium	1000	4.7	500	0.10	T(10,935)
					1000	0.18	
					2000	0.33	
					3000	1.87	
					4000	1.93	
					5000	2.23	
					6000	2.37	
					7000	2.49	
					8000	3.72	
					9000	4.61	
					10000	4.73	
Manuarite 36X	28101	Air	800	31.4	500	0.00	C
					1000	0.04	
					2000	0.06	
					3000	0.09	
					4000	0.13	
					5000	0.11	
					6000	0.20	
					7000	0.20	
					8000	0.14	
					9000	0.23	
					10000	0.17	
					11000	0.15	
					12000	0.19	
					13000	0.18	
					14000	0.21	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
Manuarite 36X	28102	Air	900	12.8	15000	0.20	T(9,910)
					16000	0.23	
					500	-0.04	
					2000	0.03	
					3000	0.08	
					4000	0.09	
					5000	0.14	
					6000	0.16	
					7000	0.21	
					8000	0.22	
					9000	0.32	
Manuarite 36X	28105	Helium	800	31.3	500	0.06	T
					1000	0.07	
					2000	0.07	
					3000	0.09	
Manuarite X	28106	Helium	800	31.2	500	-0.03	C
					1000	-0.02	
					2000	-0.02	
					3000	-0.03	
					4000	-0.01	
					5000	0.01	
					6000	0.00	
					7000	0.01	
					8000	0.01	
					9000	0.00	
Manuarite 36X	28107	Helium	900	12.8	500	-0.03	T
					1000	0.10	
					2000	0.11	
					3000	0.04	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture(a)
Manuarite 36X	28108	Helium	900	12.9	500	-0.05	T(9,470)
					1000	-0.05	
					2000	-0.04	
					3000	-0.02	
					4000	-0.05	
					5000	-0.03	
					6000	-0.03	
					7000	0.86	
					8000	0.86	
					9000	0.88	
Manuarite 36X	28111	Helium	1000	4.8	500	0.00	T
					1000	0.03	
					2000	0.12	
					3000	1.54	
Manuarite 36X	28103	Helium	1000	4.8	500	-0.01	C
					1000	-0.08	
					2000	-0.02	
					3000	-0.03	
					4000	0.00	
					5000	0.07	
					6000	0.36	
					7000	0.33	
Manuarite 36X	28113	Helium	1000	4.8	500	-0.07	C
					1000	0.04	
					2000	0.01	
					3000	0.05	
					4000	0.28	
					5000	0.33	
					6000	0.27	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
Manuarite 36XS	29101	Air	800	31.1	500	0.05	C
					1000	0.09	
					1500	0.20	
					2000	0.19	
					3000	0.20	
					4000	0.23	
					5000	0.20	
					6000	0.23	
					7000	0.29	
					8000	0.30	
					9000	0.29	
					10000	0.41	
					11000	0.44	
Manuarite 36XS	29103	Air	900	12.8	500	-0.05	T(9,910)
					1000	0.05	
					2000	0.11	
					3000	0.17	
					4000	0.31	
					5000	0.34	
					6000	0.68	
					7000	0.87	
					8000	0.84	
					9000	1.00	
Manuarite 36XS	29105	Helium	800	31.3	500	0.18	T
					1000	0.28	
					2000	0.29	
					3000	0.35	
Manuarite 36XS	29106	Helium	800	31.3	500	0.20	C
					1000	0.25	
					2000	0.37	
					3000	0.42	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
Manuarite 36XS	29107	Helium	900	12.7	4000	0.49	T
					5000	0.49	
					6000	0.57	
					7000	0.68	
					8000	0.79	
					9000	0.82	
					10000	0.82	
					11000	0.88	
					12000	0.95	
	29108	Helium	900	12.8	500	0.10	T(9,470)
					1000	0.15	
					2000	0.44	
					3000	0.39	
					500	-0.05	
					1000	-0.08	
					2000	-0.01	
					3000	0.02	
					4000	0.05	
					5000	0.05	
Manuarite 36XS	29109	Helium	1000	4.7	6000	0.08	T
					7000	0.86	
					8000	0.85	
					9000	0.92	
					500	0.00	
Manuarite 36XS	29111	Helium	1000	4.8	1000	0.18	C
					2000	0.21	
					3000	0.21	
					500	0.00	
					1000	0.06	
					2000	-0.03	
					3000	-0.02	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
Manuarite 36XS	29104	Helium	1000	4.8	4000	0.03	T(8,935)
					5000	0.07	
					6000	0.33	
					7000	0.41	
					8000	0.57	
					9000	0.63	
					1000	0.03	
					2000	0.09	
					3000	0.24	
					4000	0.28	
					5000	0.53	
					6000	0.68	
					7000	1.11	
					8000	1.37	
					500	0.04	
					1000	0.02	
					2000	0.03	
					3000	0.04	
					4000	0.06	
					5000	0.04	
					6000	0.05	
					7000	0.05	
					8000	0.07	
					9000	0.06	
					10000	0.07	
Manuarite 900	30102	Air	900	12.9	500	0.57	T
					1000	1.56	
					2000	1.69	
					4000	2.21	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
Manuarite 900	30104	Helium	800	31.3	500	0.35	T
					1000	0.36	
					2000	0.36	
					3000	0.37	
	30107	Helium	800	31.1	500	0.06	T(9,657)
					1000	0.06	
					2000	0.07	
					3000	0.10	
					4000	0.10	
					5000	0.16	
					6000	0.15	
					7000	0.87	
					8000	0.87	
					9000	0.85	
	30108	Helium	900	12.7	500	-0.02	T
					1000	0.00	
					2000	3.55	
					3000	3.63	
	30109	Helium	900	12.6	500	-0.02	T(9,470)
					1000	-0.02	
					2000	0.09	
					3000	0.11	
					4000	0.11	
					5000	0.11	
					6000	0.11	
					7000	7.03	
					8000	7.08	
					9000	7.15	
Manuarite 900	30110	Helium	1000	4.8	500	0.00	T
					1000	0.05	
					2000	0.05	
					3000	0.07	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
Manuarite 900	30111	Helium	1000	4.8	500	-0.02	C
					1000	0.03	
					2000	0.09	
					3000	0.15	
					4000	0.21	
					5000	0.26	
					6000	0.37	
RA 333	31101	Air	800	31.2	500	0.17	T(17,030)
					1000	0.30	
					2000	0.47	
					3000	0.52	
					4000	0.64	
					5000	0.93	
					6000	1.21	
					7000	1.43	
					8000	2.16	
					9000	2.50	
					10000	2.70	
					11000	3.17	
					12000	3.65	
					13000	4.29	
					14000	5.44	
					15000	6.53	
					16000	8.59	
RA 333	31102	Air	900	12.7	500	0.40	T
					1000	0.50	
					2000	2.63	
					3000	5.33	
					4000	10.38	
					5000	25.54	
					6000	46.94	
					7000	59.23	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
RA 333	31103	Helium	800	31.5	8000	67.80	T
					9000	72.66	
					10000	72.66	
					500	0.24	
					1000	0.32	
					2000	0.49	
					3000	0.80	
RA 333	31110	Helium	800	31.5	500	0.24	R(12,010)
					1000	0.32	
					2000	0.57	
					3000	0.73	
					4000	0.90	
					5000	1.19	
					6000	1.69	
					7000	2.25	
					8000	3.30	
					9000	4.10	
					10000	5.25	
					11000	6.87	
					12000	11.43	
RA 333	31106	Helium	900	12.7	500	0.36	T
					1000	2.10	
					2000	4.62	
					3000	11.33	
RA 333	31107	Helium	900	12.8	500	0.52	R(2,745) 13.75%
					1000	3.64	
					2000	9.02	
RA 333	31108	Helium	1000	4.8	500	1.57	T
					1000	2.40	
					2000	6.91	
					3000	12.26	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
RA 333	31109	Helium	1000	4.8	500	0.95	R(3,890)
					1000	1.65	
					2000	5.21	
					3000	8.73	
HK 40	32101	Air	800	31.6	500	0.08	R(14,630) 1.90%
					1000	0.11	
					2000	0.12	
					4000	0.20	
					5000	0.25	
					7000	0.33	
					8000	0.33	
					9000	0.34	
					10000	0.35	
					11000	0.41	
					12000	0.48	
					13000	0.60	
					14000	0.73	
HK 40	32102	Air	900	12.7	500	0.04	T(13,935)
					1000	0.04	
					2000	0.13	
					3000	0.16	
					4000	0.14	
					5000	0.14	
					6000	0.17	
					7000	0.17	
					8000	0.19	
					9000	0.23	
					10000	0.23	
					11000	0.27	
					12000	0.28	
					13000	0.27	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
HK 40	32104	Helium	800	31.9	500	0.00	T
					1000	0.00	
					2000	0.00	
					3000	0.00	
HK 40	32105	Helium	800	31.5	500	0.00	C
					1000	0.00	
					2000	0.02	
					3000	0.05	
					4000	0.06	
					5000	0.07	
					6000	0.07	
					7000	0.15	
					8000	0.16	
					9000	0.16	
HK 40	32106	Helium	900	12.9	500	-0.11	T
					1000	0.00	
					2000	0.25	
					3000	0.23	
HK 40	32107	Helium	900	12.8	500	-0.09	T(9,470)
					1000	-0.05	
					2000	-0.03	
					3000	0.00	
					4000	0.02	
					5000	0.01	
					6000	0.02	
					7000	1.12	
					8000	1.13	
					9000	1.15	
HK 40	32108	Helium	1000	4.8	500	0.04	T
					1000	-0.07	
					2000	0.23	
					3000	0.34	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture(a)
HK 40	32103	Helium	1000	4.8	1000	0.52	C
					2000	0.57	
					3000	0.60	
					4000	0.58	
					5000	0.66	
					6000	0.73	
					7000	1.39	
					8000	1.60	
HK 40	32109	Helium	1000	4.8	500	-0.09	C
					1000	-0.10	
					2000	-0.10	
					3000	-0.11	
					4000	0.00	
					5000	0.02	
					6000	0.12	
Super-therm	33101	Air	800	31.6	500	0.07	C
					1000	0.08	
					2000	0.09	
					3000	0.09	
					4000	0.09	
					5000	0.08	
					6000	0.09	
					7000	0.08	
					8000	0.11	
					9000	0.11	
					10000	0.12	
Super-therm	33102	Air	900	12.9	500	0.03	C
					1000	0.08	
					2000	-0.01	
					3000	0.09	
					4000	0.12	
					5000	0.12	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture(a)
Super-therm	33111	Helium	800	31.4	6000	0.14	T
					7000	0.18	
					8000	0.23	
					9000	0.24	
					500	0.00	
					1000	0.00	
					2000	0.03	
					3000	0.06	
Super-therm	33106	Helium	800	31.1	500	0.03	C
					1000	0.10	
					2000	0.07	
					3000	0.11	
					4000	0.10	
					5000	0.11	
					6000	0.12	
					7000	0.09	
					8000	0.10	
					9000	0.11	
Super-therm	33107	Helium	900	12.8	500	0.00	T
					1000	0.04	
					2000	0.05	
					3000	0.10	
Super-therm	33108	Helium	900	12.9	500	0.01	C
					1000	0.01	
					2000	-0.02	
					3000	0.01	
					4000	0.02	
					5000	0.03	
					6000	0.00	
					7000	0.02	
					8000	0.04	
					9000	0.05	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
Super-therm	33109	Helium	1000	4.8	500	-0.05	T
					1000	0.00	
					2000	0.04	
					3000	0.81	
Super-therm	33103	Helium	1000	4.8	500	-0.02	C
					1000	-0.02	
					2000	0.04	
					3000	0.01	
					4000	0.04	
					5000	0.10	
					6000	0.28	
					7000	0.26	
Super-therm	33110	Helium	1000	4.8	500	-0.02	C
					1000	-0.04	
					2000	-0.06	
					3000	-0.05	
					4000	0.01	
					5000	-0.04	
					6000	0.05	
Hastelloy S	34102	Air	800	31.1	500	0.38	C
					1000	0.75	
					2000	1.28	
					3000	2.12	
					4000	3.10	
					5000	3.84	
					6000	4.97	
					7000	5.72	
					8000	6.98	
					9000	8.03	
					10000	8.89	
					11000	9.99	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture(a)
Hastelloy S	34103	Air	900	12.7	12000	10.89	R(9,921)
					13000	11.94	
					14000	13.64	
					15000	15.17	
					16000	17.04	
					500	1.82	
					1000	3.78	
					2000	9.12	
					3000	16.23	
					4000	47.57	
					5000	60.46	
					6000	64.17	
					7000	65.31	
					8000	68.46	
					9000	69.60	
					500	0.46	T
					1000	1.03	
					2000	1.18	
					3000	1.57	
Hastelloy S	34111	Helium	800	31.9	500	0.58	C
					1000	0.66	
					2000	1.18	
					3000	1.79	
					4000	2.53	
					5000	3.27	
					6000	3.83	
					7000	4.54	
					8000	5.04	
					9000	5.72	
					10000	6.51	
					11000	7.14	
					12000	7.93	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture(a)
Hastelloy S	34107	Helium	900	12.8	500	0.76	T
					1000	1.29	
					2000	3.17	
					3000	4.82	
Hastelloy S	34108	Helium	900	12.7	500	1.10	T
					1000	2.08	
					2000	4.97	
					3000	8.09	
					4000	10.48	
					5000	18.66	
Hastelloy S	34109	Helium	1000	4.8	500	0.69	T
					1000	1.11	
					2000	6.99	
					3000	13.41	
Hastelloy S	34110	Helium	1000	4.8	500	1.08	T
					1000	1.73	
					2000	4.64	
					3000	8.23	
					4000	21.04	
HD 556	39102	Air	800	31.4	500	0.09	C
					1000	0.17	
					2000	0.20	
					3000	0.34	
					4000	0.35	
					5000	0.36	
					6000	0.36	
					7000	0.37	
					8000	0.47	
					9000	0.47	
					10000	0.50	
					11000	0.56	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
HD 556	39104	Air	900	12.7	12000	0.57	C
					13000	0.59	
					14000	0.58	
					15000	0.68	
					16000	0.73	
					500	0.05	
					1000	0.41	
					2000	0.45	
					3000	0.53	
					4000	0.54	
					5000	0.57	
					6000	0.60	
					7000	0.60	
					8000	0.80	
					9000	0.76	
					10000	0.89	
					11000	1.07	
					12000	1.24	
					13000	1.40	
					14000	1.40	
HD 556	39105	Helium	800	31.4	500	0.08	T
					1000	0.12	
					2000	0.19	
					3000	0.25	
HD 556	39112	Helium	800	31.6	500	0.06	C
					1000	0.12	
					2000	0.18	
					3000	0.22	
					4000	0.25	
					5000	0.31	
					6000	0.33	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture(a)
HD 556	39108	Helium	900	12.8	7000	0.33	T
					8000	0.41	
					9000	0.39	
					10000	0.39	
					11000	0.38	
					12000	0.42	
					500	0.02	
HD 556	39109	Helium	900	13.0	1000	0.03	C
					2000	0.09	
					3000	0.13	
					500	0.04	
					1000	0.07	
					2000	0.15	
					3000	0.22	
HD 556	39110	Helium	1000	4.8	4000	0.22	T
					5000	0.26	
					6000	0.31	
					7000	0.38	
					8000	0.40	
					9000	0.43	
					10000	0.50	
HD 556	39111	Helium	1000	4.8	11000	0.52	T(10,935)
					12000	0.55	
					500	0.01	
					1000	0.06	
					2000	0.01	
					3000	1.71	
					500	0.00	
HD 556	39111	Helium	1000	4.8	1000	0.03	
					2000	0.18	

Alloy	Specimen Number	Environment	Temperature (°C)	Stress (MPa)	Time (hours)	Creep Strain (%)	Time and/or Strain at Rupture ^(a)
					3000	1.59	
					4000	1.60	
					5000	1.63	
					6000	1.70	
					7000	1.79	
					8000	2.45	
					9000	3.05	
					10000	3.13	

Note: ^(a)C = continuing, T = terminated, R = ruptured; rupture or termination time (h) indicated in parentheses.

APPENDIX B
COMPLETE WEIGHT CHANGE DATA FOR GROUP II ALLOYS

Alloy	Specimen Number	Temperature (°C)	Time (hours)	Weight Change (mg/cm ²)	Status ^(a)
Hastelloy X	08645	800	500	0.51	T
			1000	0.51	
			2000	0.62	
			3000	0.69	
Hastelloy X	08646	800	500	0.55	T
			1000	0.53	
			2000	0.60	
			3000	0.64	
			4000	0.73	
			5000	0.84	
			6000	1.02	
			7000	1.13	
			8000	1.27	
			9000	1.33	
			10000	1.36	
Hastelloy X	08647	900	500	0.69	T
			1000	1.13	
			2000	1.56	
			3000	1.84	
Hastelloy X	08648	900	500	0.84	T
			1000	1.22	
			2000	1.53	
			3000	1.33	
			4000	1.33	
			5000	1.18	
			6000	1.04	
			7000	1.20	
			8000	0.76	
			9000	0.47	
			10000	0.11	
Hastelloy X	08649	1000	500	0.51	T
			1000	0.69	
			2000	1.29	
			3000	2.00	
Hastelloy X	08650	1000	500	0.73	T
			1000	0.82	
			2000	1.15	
			3000	1.91	
			4000	2.13	
			5000	2.91	
			6000	3.16	
			7000	3.18	
			8000	3.42	
			9000	3.42	
			10000	3.62	

Alloy	Specimen Number	Temperature (°C)	Time (hours)	Weight Change (mg/cm ²)	Status ^(a)
Manuarite 36X	28145	800	500	0.42	T
			1000	0.26	
			2000	0.44	
			3000	0.49	
Manuarite 36X	28146	800	500	0.47	T (9,675)
			1000	0.56	
			2000	0.76	
			3000	0.78	
			4000	0.76	
			5000	0.76	
			6000	0.76	
			7000	0.71	
			8000	0.73	
			9000	0.71	
Manuarite 36X	28147	900	500	0.17	T
			1000	0.66	
			2000	0.89	
			3000	0.07	
Manuarite 36X	28148	900	1000	0.47	T (9,470)
			2000	0.82	
			3000	1.04	
			4000	1.07	
			5000	1.02	
			6000	0.91	
			7000	0.91	
			8000	0.96	
			9000	0.89	
Manuarite 36X	28149	1000	500	-0.15	T
			1000	-0.13	
			2000	-0.42	
			3000	-0.53	
Manuarite 36X	28144	1000	1000	-0.44	T (7,985)
			2000	0.38	
			3000	0.16	
			4000	-0.13	
			5000	-1.71	
			6000	-2.67	
			7000	-4.15	
Manuarite 36X	28150	1000	500	-0.53	T (7,075)
			1000	-0.53	
			2000	-0.84	
			3000	-0.91	
			4000	-0.98	
			5000	-1.24	
			6000	-1.42	

Alloy	Specimen Number	Temperature (°C)	Time (hours)	Weight Change (mg/cm ²)	Status ^(a)
Manuarite 36X	29144	800	500	0.42	T
			1000	-0.33	
			2000	-0.73	
			3000	-0.80	
Manuarite 36XS	29145	800	500	0.66	T
			1000	-0.15	
			2000	-0.44	
			3000	-0.67	
			4000	-0.80	
			5000	-0.82	
			6000	-0.78	
			7000	-0.76	
			8000	-0.78	
			9000	-0.76	
			10000	-0.76	
Manuarite 36XS	29146	900	500	-0.04	T
			1000	-0.62	
			2000	-0.67	
			3000	-1.00	
Manuarite 36XS	29147	900	1000	0.69	T (9,470)
			2000	1.07	
			3000	1.22	
			4000	1.16	
			5000	1.20	
			6000	1.33	
			7000	1.49	
			8000	1.87	
			9000	2.02	
Manuarite 36XS	29148	1000	1000	-0.26	T
			2000	0.04	
			3000	0.09	
Manuarite 36XS	29149	1000	1000	-0.42	T (9,005)
			2000	-0.33	
			3000	0.09	
			4000	-2.53	
			5000	-3.29	
			6000	-3.84	
			7000	-4.91	
			8000	-5.73	
Manuarite 900	30145	800	500	0.22	T
			1000	0.15	
			2000	0.31	
			3000	0.36	

Alloy	Specimen Number	Temperature (°C)	Time (hours)	Weight Change (mg/cm ²)	Status(a)
Manuarite 900	30146	800	500	0.40	T (9,675)
			1000	0.44	
			2000	0.67	
			3000	0.60	
			4000	0.53	
			5000	0.51	
			6000	0.53	
			7000	0.51	
			8000	0.51	
			9000	0.42	
Manuarite 900	30148	900	500	0.13	T
			1000	0.31	
			2000	0.33	
			3000	0.22	
Manuarite 900	30143	900	1000	0.27	T (9,470)
			2000	0.33	
			3000	0.56	
			4000	0.69	
			5000	0.84	
			6000	0.93	
			7000	1.02	
			8000	1.02	
Manuarite 900	30153	1000	1000	1.22	T
			2000	-0.13	
			3000	-0.82	
Manuarite 900	30156	1000	500	-0.16	T (7,075)
			1000	-0.02	
			2000	0.27	
			3000	0.58	
			4000	-1.00	
			5000	-2.09	
RA 333	31145	800	500	0.13	T
			1000	0.07	
			2000	0.31	
			3000	0.42	
RA 333	31146	800	500	0.06	T
			1000	0.02	
			2000	0.29	
			3000	0.40	
			4000	0.56	
			5000	0.76	

Alloy	Specimen Number	Temperature (°C)	Time (hours)	Weight Change (mg/cm ²)	Status(a)
RA 333	31147	900	6000	0.89	T
			7000	0.93	
			8000	1.00	
			9000	1.07	
			10000	1.11	
RA 333	31147	900	500	0.95	T
			1000	1.20	
			2000	1.44	
			3000	1.82	
RA 333	31148	900	500	0.84	T
			1000	1.13	
			2000	1.51	
			3000	1.84	
			4000	1.80	
			5000	-1.89	
			6000	-3.36	
			7000	-6.40	
			8000	-7.04	
			9000	-9.47	
			10000	-9.78	
RA 333	31149	1000	1000	0.46	T
			2000	0.76	
			3000	0.58	
RA 333	31150	1000	1000	0.04	T (9,935)
			2000	0.40	
			3000	1.11	
			4000	0.60	
			5000	0.33	
			6000	-0.02	
			7000	-0.16	
			8000	-0.62	
			9000	-0.78	
HK 40	32144	800	500	0.26	T
			1000	0.24	
			2000	0.42	
			3000	0.49	
HK 40	32146	800	500	0.53	T (9,675)
			1000	0.49	
			2000	0.62	
			3000	0.51	
			4000	0.42	
			5000	0.36	
			6000	0.31	

Alloy	Specimen Number	Temperature (°C)	Time (hours)	Weight Change (mg/cm ²)	Status ^(a)
HK 40	32147	900	7000	0.29	T
			8000	0.24	
			9000	0.13	
			500	0.58	
HK 40	32142	900	1000	0.86	T
			2000	0.87	
			3000	0.71	
			1000	0.42	
HK 40	32149	1000	2000	0.51	(9,470)
			3000	0.44	
			4000	0.24	
			5000	0.33	
HK 40	32150	1000	6000	0.38	T
			7000	0.40	
			8000	0.58	
			9000	0.64	
HK 40	32143	1000	500	-0.22	(7,075)
			1000	-0.49	
			2000	-0.60	
			3000	-0.64	
Super-therm	33144	800	500	-0.33	T
			1000	-0.07	
			2000	-0.29	
			3000	-0.29	
Super-therm	33145	800	4000	-0.47	(9,675)
			5000	-0.47	
			6000	-0.40	
			7000	-0.49	
Super-therm	33144	800	1000	-0.58	T
			2000	-0.27	
			3000	-0.33	
			4000	-0.16	
Super-therm	33144	800	5000	-0.40	T
			6000	-0.60	
			7000	-0.49	
			500	0.51	
Super-therm	33144	800	1000	0.31	T
			2000	0.55	
			3000	0.62	
			500	0.33	
Super-therm	33145	800	1000	0.36	(9,675)
			2000	0.53	
			500	0.33	

Alloy	Specimen Number	Temperature (°C)	Time (hours)	Weight/Change (mg/cm ²)	Status(a)
Super-therm	33146	900	3000	0.16	T
			4000	-0.96	
			5000	-1.20	
			6000	-1.24	
			7000	-1.33	
			8000	-1.40	
			9000	-1.56	
	33142	900	500	0.33	T (9,470)
			1000	0.75	
			2000	1.42	
			3000	1.42	
			1000	0.51	
			2000	0.78	
			3000	0.96	
	33148	1000	4000	0.96	T
			5000	1.09	
			6000	1.22	
			7000	1.36	
			8000	1.36	
			9000	0.91	
	33143	1000	500	0.37	T (7,985)
			1000	0.37	
			2000	0.33	
			3000	0.69	
Hastelloy S	33149	1000	500	0.04	T (7,075)
			1000	0.16	
			2000	0.27	
			3000	0.40	
			4000	0.58	
			5000	0.64	
			6000	0.73	
	34144	800	500	0.00	T
			1000	0.07	
			2000	0.29	
			3000	0.40	
			4000	0.56	
	34144	800	5000	0.60	T
			6000	0.73	
			500	0.71	
			1000	0.73	
			2000	0.91	
			3000	1.13	

Alloy	Specimen Number	Temperature (°C)	Time (hours)	Weight Change (mg/cm ²)	Status(a)
Hastelloy S	34145	800	500	0.51	T
			1000	0.46	
			2000	0.60	
			3000	0.73	
			4000	0.93	
			5000	1.22	
			6000	1.47	
			7000	1.67	
			8000	1.80	
			9000	1.91	
			10000	1.96	
Hastelloy S	34146	900	500	0.75	T
			1000	1.17	
			2000	1.58	
			3000	1.80	
Hastelloy S	34147	900	500	0.62	T
			1000	1.06	
			2000	1.53	
			3000	1.78	
			4000	2.07	
			5000	2.27	
			6000	2.49	
			7000	2.67	
			8000	2.91	
			9000	3.07	
			10000	3.24	
Hastelloy S	34148	1000	1000	2.28	T
			2000	2.98	
			3000	3.36	
Hastelloy S	34149	1000	1000	3.04	T (9,935)
			2000	3.40	
			3000	3.51	
			4000	3.80	
			5000	4.09	
			6000	4.40	
			7000	4.56	
			8000	4.73	
			9000	4.89	
HD 556	39144	800	500	0.35	T
			1000	0.46	
			2000	0.47	
			3000	0.49	

Alloy	Specimen Number	Temperature (°C)	Time (hours)	Weight Change (mg/cm ²)	Status ^(a)
HD 556	39145	800	500	0.31	T
			1000	0.20	
			2000	0.20	
			3000	0.20	
			4000	0.22	
			5000	0.29	
			6000	0.31	
			7000	0.27	
			8000	0.27	
			9000	0.27	
			10000	0.27	
HD 556	39146	900	500	0.15	T
			1000	0.24	
			2000	0.33	
			3000	0.60	
HD 556	39147	900	500	0.13	T
			1000	0.17	
			2000	0.27	
			3000	0.36	
			4000	0.47	
			5000	0.27	
			6000	0.22	
			7000	-1.29	
			8000	-1.69	
			9000	-2.27	
			10000	-2.36	
HD 556	39148	1000	500	-0.11	T
			1000	-0.15	
			2000	-0.40	
			3000	-0.49	
HD 556	39149	1000	500	0.09	T
			1000	-0.09	
			2000	-0.62	
			3000	-0.49	
			4000	-0.42	
			5000	-0.07	
			6000	-0.18	
			7000	-0.49	
			8000	-0.53	
			9000	-0.67	
			10000	-0.71	

Note: T = terminated; termination time (h) in parentheses.

APPENDIX C
COMPLETE BULK CARBON ANALYSES FOR GROUP II ALLOYS

Alloy	Specimen Number	Environment	Temperature (°C)	Time (hours)	Carbon Content (weight %)
Hastelloy X	08643	Helium	900	3000	0.141
Hastelloy X	08645	Helium	800	3000	0.095
Hastelloy X	08646	Helium	800	10000	0.11
Hastelloy X	08647	Helium	900	3000	0.16
Hastelloy X	08648	Helium	900	10000	0.23
Hastelloy X	08649	Helium	1000	3000	0.19
Hastelloy X	08653	Helium	800	3000	0.09
Hastelloy X	08650	Helium	1000	10000	0.301
Hastelloy X	08655	Air	800	3000	0.077
Hastelloy X	08656	Air	900	3000	0.078
Manuarite 36X	28142	Helium	900	3000	0.49
Manuarite 36X	28144	Helium	1000	7985	0.63
Manuarite 36X	28145	Helium	800	3000	0.47
Manuarite 36X	28146	Helium	800	9675	0.47
Manuarite 36X	28147	Helium	900	3000	0.52
Manuarite 36X	28148	Helium	900	9470	0.56
Manuarite 36X	28149	Helium	1000	3000	0.55
Manuarite 36X	28150	Helium	1000	7075	0.60
Manuarite 36X	28153	Helium	800	3000	0.48
Manuarite 36X	28155	Air	800	3000	0.46
Manuarite 36X	28156	Air	900	3000	0.46
Manuarite 36XS	29144	Helium	800	3000	0.42
Manuarite 36XS	29145	Helium	800	10000	0.43
Manuarite 36XS	29146	Helium	900	3000	0.46
Manuarite 36XS	29147	Helium	900	9470	0.63
Manuarite 36XS	29148	Helium	1000	3000	0.54
Manuarite 36XS	29149	Helium	1000	9005	0.62
Manuarite 36XS	29151	Helium	900	3000	0.43
Manuarite 36XS	29153	Helium	800	3000	0.42
Manuarite 36XS	29155	Air	800	3000	0.41
Manuarite 36XS	29156	Air	900	3000	0.41
Manuarite 900	30142	Helium	900	3000	0.13
Manuarite 900	30143	Helium	900	9470	0.28
Manuarite 900	30145	Helium	800	3000	0.13
Manuarite 900	30146	Helium	800	9675	0.12
Manuarite 900	30148	Helium	900	3000	0.17
Manuarite 900	30149	Helium	800	3000	0.12
Manuarite 900	30151	Air	800	3000	0.12
Manuarite 900	30153	Helium	1000	3000	0.35
Manuarite 900	30154	Air	900	3000	0.12
Manuarite 900	30155	Helium	1000	7075	0.50
RA 333	31142	Air	900	3000	0.032
RA 333	31145	Helium	800	3000	0.04
RA 333	31146	Helium	800	10000	0.057
RA 333	31147	Helium	900	3000	0.14

Alloy	Specimen Number	Environment	Temperature (°C)	Time (hours)	Carbon Content (weight %)
RA 333	31148	Helium	900	10000	0.17
RA 333	31149	Helium	1000	3000	0.14
RA 333	31150	Helium	1000	9935	0.32
RA 333	31152	Helium	900	3000	0.076
RA 333	31154	Helium	800	3000	0.052
RA 333	31156	Air	800	3000	0.032
HK 40	32142	Helium	900	9470	0.58
HK 40	32143	Helium	1000	7985	0.71
HK 40	32144	Helium	800	3000	0.45
HK 40	32146	Helium	800	9675	0.45
HK 40	32147	Helium	900	3000	0.51
HK 40	32149	Helium	1000	3000	0.50
HK 40	32150	Helium	1000	7075	0.59
HK 40	32152	Helium	900	3000	0.49
HK 40	32154	Helium	800	3000	0.44
HK 40	32156	Air	800	3000	0.45
HK 40	32157	Air	900	3000	0.45
Supertherm	33142	Helium	900	9470	0.53
Supertherm	33143	Helium	1000	7985	0.56
Supertherm	33144	Helium	800	3000	0.45
Supertherm	33145	Helium	800	9675	0.45
Supertherm	33146	Helium	900	3000	0.51
Supertherm	33148	Helium	1000	3000	0.50
Supertherm	33149	Helium	1000	7075	0.57
Supertherm	33151	Helium	900	3000	0.47
Supertherm	33153	Helium	800	3000	0.45
Supertherm	33155	Air	800	3000	0.43
Supertherm	33156	Air	900	3000	0.44
Hastelloy S	34144	Air	800	3000	0.003
Hastelloy S	34144	Helium	800	3000	0.05
Hastelloy S	34145	Helium	800	10000	0.040
Hastelloy S	34146	Helium	900	3000	0.08
Hastelloy S	34147	Helium	900	10000	0.16
Hastelloy S	34148	Helium	1000	3000	0.11
Hastelloy S	34149	Helium	1000	9935	0.25
Hastelloy S	34151	Helium	900	3000	0.07
Hastelloy S	34153	Helium	800	3000	0.018
Hastelloy S	34156	Air	900	3000	0.065
HD 556	39144	Helium	800	3000	0.10
HD 556	39145	Helium	800	10000	0.10
HD 556	39146	Helium	900	3000	0.16
HD 556	39147	Helium	900	10000	0.20
HD 556	39148	Helium	1000	3000	0.26
HD 556	39151	Helium	900	3000	0.15
HD 556	39153	Helium	800	3000	0.11
HD 556	39149	Helium	1000	10000	0.402
HD 556	39155	Air	800	3000	0.093
HD 556	39156	Air	900	3000	0.091

APPENDIX D
SEM/EDX RESULTS FOR
GROUP II ALLOYS EXPOSED IN
CONTROLLED-IMPURITY HELIUM FOR 10,000 HOURS

Note: Areas identified in the SEM photographs of this appendix and for which EDX analysis could not establish meaningful phase compositions and identities have not been included in the accompanying tables.

TABLE D-1
HK 40, 32146, 9675 HOURS AT 800°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-1(a)	Discontinuous light grey surface scale	87 Cr, 11 Fe, 1 Si	Cr-rich carbide containing Fe, Si
4	D-1(a)	Region below scale (denuded zone)	58 Fe, 22 Ni, 18 Cr, 1 Si	Cr-depleted subscale region

(a) HK 40 (32146) 1000X

(b) HK 40 (32142) 1000X

Fig. D-1. SEM photomicrograph showing the surface scales and subscale microstructure of alloy HK 40 after an exposure of (a) 9675 h at 800°C or (b) 9470 h at 900°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Tables D-1 and D-2.

TABLE D-2
HK 40, 32142, 9470 HOURS AT 900°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-1(b)	Continuous light grey outer scale	98 Cr, 2 Fe	Cr-rich carbide outer scale
2	D-1(b)	Continuous dark grey subsurface scale	98 Cr, 1 Fe	Cr-rich oxide inner scale
3	D-1(b)	Discontinuous mottled subsurface scale	84 Cr, 4 Fe, 4 Cu, 3 Si, 3 Ni	Cr-rich oxide mixed with γ -matrix
4	D-1(b)	Continuous dark grey inner scale	89 Cr, 2 Fe, 8 Ni	Cr-rich oxide inner scale
6	D-1(b)	Region below scale (denuded zone)	57 Fe, 26 Ni, 16 Cr	Cr-depleted subscale area

TABLE D-3
HK 40, 32143, 7985 HOURS AT 1000°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-2	Highly discontinuous light grey outer scale	70 Cr, 18 Fe, 10 Ni	Cr-Fe-Ni carbide outer scale
2	D-2	Light grey outer scale, adhered	77 Cr, 18 Fe, 4 Ni	Cr-rich carbide + Fe
3	D-2	Discontinuous black inner scale	52 Mg (?), 35 Si, 8 Ni, 2 Cr, 2 Mn, 1 Fe	Si oxide inner scale, origin of magnesium unknown
5	D-2	Region below scale (denuded zone)	57 Fe, 25 Ni, 17 Cr, 1 Si	Cr-depleted sub-scale region
6	D-2	Grey primary carbide in rear surface matrix	75 Cr, 21 Fe, 4 Ni	Cr-Fe carbide + adjacent γ -matrix
None	D-2	Center of specimen	50 Fe, 25 Cr, 22 Ni, 1.5 Si	Nominal alloy composition

HK 40 (32143) 1000 X

Fig. D-2. SEM photomicrograph showing surface scales and subsurface microstructure of HK 40 after an exposure of 7985 h at 1000°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Table D-3.

TABLE D-4
SUPERTHERM, 33145, 9675 HOURS AT 800°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-3(a)	Continuous mottled surface scale	48 Cr, 29 Si, 10 Ag, 9 Ni, 3 Fe, 1 Mn	Mixed Cr-Si-rich carbide with adjacent silver electrodeposit
2	D-3(a)	Continuous mottled surface scale	77 Cr, 14 Si, 6 Ni, 2 Fe	Mixed Cr-Si-oxide

TABLE D-5
SUPERTHERM, 33142, 9470 HOURS AT 900°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-3(b)	Light grey semicontinuous outer scale	98 Cr, 1 Fe	Cr-carbide outer scale
2	D-3(b)	Dark continuous inner scale	98 Si, 1 Cr	Si-oxide inner scale
4	D-3(b)	Subscale region	30 Ni, 23 Fe, 23 Cr, 16 Co, 8 Si	Near-surface matrix + adjacent Si-rich oxide

(a) SUPERTHERM (33145) 1000X

(b) SUPERTHERM (33142) 1000X

Fig. D-3. SEM photomicrographs showing the surface scales and subsurface microstructures of Supertherm after an exposure of (a) 9675 h at 800°C and (b) 9470 h at 900°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Tables D-4 and D-5.

TABLE D-6
 SUPERTHERM, 33143, 7985 HOURS AT 1000°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-4	Dark grey discontinuous protruding scale	47 Si, 44 Cr, 2 Ni	Mixed Si-oxide and Cr-carbide
2	D-4	Black subsurface discontinuous scale	17 Si, 27 Cr, 18 Fe, 13 Co, 25 Ni	Si-rich oxide adjacent to Cr-rich carbide and denuded matrix
3	D-4	Black discontinuous inner scale	52 Mg, 41 Si, 5 Cr, 2 Mn	Si-rich oxide + Mg (origin unknown)

SUPERTHERM (33143) 1000X

Fig. D-4. SEM photomicrograph showing scale and substructure of Supertherm after an exposure of 7985 h at 1000°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Table D-6.

TABLE D-7
MANAURITE 36X, 28146, 9675 HOURS AT 800°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-5(a)	Continuous outer dark, spotty grey scale	82 Cr, 10 Mn, 4 Ni, 3 Fe	Cr-carbide + Cr-Mn-oxide
2	D-5(a)	Continuous grey middle layer scale	82 Cr, 8 Si 6 Fe, 4 Ni	Cr-rich carbide
3	D-5(a)	Continuous black inner layer scale	36 Fe, 35 Ni, 21 Cr, 8 Si	Si-oxide + adjacent γ -matrix
4	D-5(a)	Continuous black inner layer scale	64 Si, 22 Cr 8 Ni, 7 Fe	Si-rich oxide + Cr-carbide + γ -matrix
7	D-5(b)	Primary carbide in near-surface region	89 Cr, 8 Fe, 3 Ni	Cr-rich carbide
None	D-5(b)	Center of specimen	37 Ni, 35 Fe, 25 Cr, 1.5 Si	Nominal alloy composition

(a) MANAURITE 36X (28146) 1000X

(b) MANAURITE 36X (28146) 1000X

Fig. D-5. SEM photomicrograph of Manaurite 36X after an exposure of 9675 h at 800°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Table D-7.

TABLE D-8
MANAURITE 36X, 28148, 9470 HOURS AT 900°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-6	Continuous outer light grey scale	92 Cr, 4 Fe 2 Ni, 1 Nb	Cr-rich carbide outer scale
2	D-6	Dark continuous inner scale	100 Si	Si-oxide inner scale
3	D-6	Lighter region at inner scale	100 Si	Si-oxide inner scale
4	D-6	Continuous dark inner scale	31 Cr, 23 Si, 29 Ni, 12 Fe, 3 Al	Si-oxide + γ-matrix
5	D-6	Region below scale (denuded zone)	43 Ni, 39 Fe 17 Cr, 1 Si	Cr-depleted near-surface matrix

MANAURITE 36X (28148) 1000X

Fig. D-6. SEM photomicrograph of Manaurite 36X after an exposure of 9470 h at 900°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Table D-8.

TABLE D-9
MANAURITE 36X, 28144, 7985 HOURS AT 1000°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-7(a)	Discontinuous grey outer scale	81 Cr, 14 Fe, 5 Ni	Cr-carbide outer scale + Fe and Ni
2	D-7(a)	Discontinuous light grey particle	47 Ni, 36 Fe, 15 Cr, 1 Si	Remnant of γ -matrix, Cr-depleted
3	D-7(b)	Light grey discontinuous region	99 Ni	Electrodeposited Ni
4	D-7(b)	Continuous dark grey inner scale	67 Si, 11 Ni, 9 Cr, 7 Fe, 4 Nb	Si-oxide + part of γ -matrix + Nb (probably in the form of a carbide)
5	D-7(b)	Continuous black inner scale	47 Si, 31 Ag, 9 Ni, 7 Cr, 3 Nb, 2 Mn	Si-rich oxide and adjacent silver electrodeposit
6	D-7(b)	Subscale matrix (denuded region)	40 Ni, 39 Fe, 19 Cr, 1 Si	Cr-depleted matrix area
7	D-7(b)	Precipitated carbide	91 Nb, 5 Fe, 3 Cr, 1 Ni	Nb-carbide precipitate (secondary)
8	D-7(b)	Center of specimen	35 Fe, 35 Ni, 27 Cr, 1.6 Si, 0.8 Nb	Nominal alloy composition
10	D-7(a)	Primary carbide	85 Cr, 12 Fe, 3 Ni	Cr-carbide (primary)

(a) MANAURITE 36X (28144) 1000X

(b) MANAURITE 36X (28144) 1000X

Fig. D-7. SEM photomicrograph of Manaurite 36X after an exposure of 7985 h at 1000°C in a controlled impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Table D-9.

TABLE D-10
MANAURITE 36XS, 29147, 9470 HOURS AT 900°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-8(a)	Continuous gray outer scale	97 Cr, 2 Fe	Cr-carbide + Fe outer scale
2	D-8(a)	Continuous light grey middle scale	24 Si, 47 Cr, 19 Ni, 10 Fe, 1 Nb	Cr-carbide + Si-oxide + Ni, Fe and Nb
3	D-8(a)	Continuous light grey middle scale closer to outer scale	84 Cr, 15 Fe	Cr-carbide + Fe
4	D-8(a)	Subscale matrix (denuded region)	46 Fe, 28 Ni, 21 Cr, 5 Si	Cr-depleted matrix region
5	D-8(a)	Near-surface primary carbide	84 Cr, 8 Fe, 6 Si, 2 Ni	Cr-carbide + Fe + matrix

(a) MANAURITE 36XS (29147) 1000X

(b) MANAURITE 36XS (29149) 1000X

Fig. D-8. SEM photomicrographs of Manaurite 36XS after an exposure of (a) 9470 h at 900°C or (b) 9005 h at 1000°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Tables D-10 and D-11.

TABLE D-11
MANAURITE 36XS, 29149, 9005 HOURS AT 1000°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-8(b)	Discontinuous outer grey scale	88 Cr, 8 Fe, 3 Ni, 1 Si	Cr-carbide outer scale + Fe + Ni + Si
3	D-8(b)	Discontinuous inner black scale	41 Si, 25 Fe, 18 Cr, 15 Ni	Si-oxide + matrix
5	D-8(b)	Near-surface carbide	86 Cr, 6 Fe, 6 Si, 1 Ni	Precipitated Cr- carbide + Fe + Si

TABLE D-12
MANAURITE 900, 30143, 9470 HOURS AT 900°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-9	Continuous grey outer scale	52 Cr, 24 Ni, 16 Fe, 6 Si	Cr-rich carbide with Ni and Fe
2	D-9	Mottled discontinuous area between inner and outer scales	32 Cr, 26 Si, 13 Mn, 12 Ag, 9 Ni, 6 Fe, 2 Nb	Mixed Cr-Si-oxide with Mn, Ni, Fe, Nb and adjacent electrodeposited silver
3	D-9	Continuous black inner scale layer	34 Nb, 20 Si, 20 Fe, 10 Ni	Si-rich oxide + Nb-rich carbide + matrix
4	D-9	Subscale matrix (denuded)	44 Ni, 42 Fe, 13 Cr	Cr-depleted near-surface matrix
5	D-9	Near-surface carbide	57 Cr, 26 Fe, 16 Ni	Cr-carbide (primary) + matrix
6	D-9	Center of specimen	39 Ni, 38 Fe, 20 Cr, 1 Si, 1 Nb	Nominal alloy composition

MANAURITE 900 (30143) 1000X

Fig. D-9. SEM photomicrograph of Manaurite 900 after an exposure of 9400 h at 900°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Table D-12.

TABLE D-13
HASTELLOY X, 08646, 10,000 HOURS AT 800°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-10(a)	Continuous grey outer scale	91 Cr, 4 Ni, 3 Fe, 1 Mn, 1 Mo, 1 Si	Cr-rich carbide outer scale + Ni, Fe
2	D-10(a)	Continuous darker grey inner scale	75 Cr, 18 Mn, 4 Ni, 1 Mo, 1 Si	Cr-rich carbide enriched with Mn-oxide
3	D-10(a)	Discontinuous light grey scale	84 Cr, 6 Ni, 5 Fe, 3 Mo, 1 Mn	Cr-rich carbide containing Ni, Fe, Mo, Mn
4	D-10(a)	Continuous darker grey inner scale	72 Cr, 22 Mn, 3 Ni, 2 Mo, 1 Si	Cr-rich carbide enriched with Mn-oxide
5	D-10(a)	Continuous dark grey inner scale	51 Cr, 21 Mn, 13 Ni, 10 Mo, 3 Fe, 2 Si	Cr-Mn-rich oxide + Ni, Mo, and Fe
6	D-10(a)	White near-surface precipitate	41 Mo, 30 Ni, 14 Cr, 7 Si, 3 Mn, 3 Fe, 2 W	Mo-carbide secondary pre- cipitate adja- cent to denuded γ-matrix
7	D-10(a)	Light grey subsur- face particle	53 Mo, 30 Ni, 8 Si, 3 W, 3 Cr, 3 Fe	Mo-rich carbide secondary pre- cipitate adja- cent to γ- matrix
None	None	Center of specimen	50 Ni, 21 Cr, 18 Fe, 7 Mo, 2 Co, 1 Si, 5 W	Nominal base alloy composi- tion

(a) HASTELLOY X (08646) 4000X

(b) HASTELLOY X (08648) 4000X

Fig. D-10. SEM photomicrographs of Hastelloy X after an exposure of (a) 10,000 h at 800°C and (b) 10,000 h at 900°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Tables D-13 and D-14.

TABLE D-14
HASTELLOY X, 08648, 10,000 HOURS AT 900°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-10(b)	Discontinuous light grey outer scale	72 Cr, 11 Ni, 10 Mo, 4 Fe, 3 Si	Cr-rich carbide containing Ni, Mo, Fe, and Si
2	D-10(b)	Discontinuous light grey outer scale	90 Cr, 5 Fe, 3 Ni, 1 Mo	Cr-rich carbide containing Fe, Ni, Mo
3	D-10(b)	Light grey near surface particle	49 Mo, 27 Ni, 11 Cr, 7 Si, 3 Fe, 2 W	Mo-rich carbide + adjacent γ -matrix
4	D-10(b)	Discontinuous light grey outer scale	85 Cr, 6 Fe, 5 Mo, 4 Ni	Cr-rich carbide containing Fe, Mo, Ni
5	D-10(b)	Light grey particle rear surface	49 Mo, 27 Ni, 10 Cr, 7 Si, 3 Fe, 2 W	Mo-rich carbide adjacent to denuded γ -matrix (austenite) and Si-oxide
6	D-10(b)	Continuous darker grey inner scale	35 Cr, 28 Mo, 18 Ni, 12 Mn, 4 Si, 1 Fe	Cr-Mn oxide + Mo-carbide + Ni
7	D-10(b)	Continuous darker grey inner scale	67 Cr, 29 Mn, 3 Ni, 5 Si	Cr-Mn rich oxide
8	D-10(b)	Continuous darker grey inner scale	54 Cr, 27 Mn, 10 Ni, 4 Mo, 2 Fe, 2 Si	Cr-Mn rich oxide
None	None	Center of specimen	48 Ni, 21 Cr, 18 Fe, 9 Mo, 1.5 Co, 1 Si, 5 W	Nominal base alloy composition

TABLE D-15
HASTELLOY S, 34149, 9935 HOURS AT 1000°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
2	D-11	Dark discontinuous subsurface particle	57 Ni, 25 Mo, 8 Cr, 6 Si, 1 Fe, 1 Mg	Mo-rich carbide adjacent to denuded γ -matrix
3	D-11	Near-surface matrix region (denuded)	80 Ni, 12 Cr, 7 Mo	Cr- and Mo-depleted γ -matrix
4	D-11	Dark subsurface particle	97 Al, 2 Mg	Al-oxide subscale particle
5	D-11	Near-surface matrix region (denuded)	79 Ni, 14 Cr, 7 Mo	Mo-depleted γ -matrix
7	D-11	Light subsurface particle	64 Mo, 24 Ni, 8 Cr, 4 Si	Mo-rich carbide + Cr + Si adjacent to matrix
None	None	Center of specimen	77 Ni, 12 Cr, 10 Mo	Nominal alloy composition

HASTELLOY S (34149) 1000X

Fig. D-11. SEM photomicrograph of Hastelloy S after an exposure of 9935 h at 1000°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Table D-15.

TABLE D-16
RA 333, 31150, 9935 HOURS AT 1000°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
1	D-12	Continuous light grey outer scale	86 Cr, 7 Mo, 3 Fe, 2 Ni, 2 Si	Cr-rich carbide + Mo, Fe, Ni, and Si
2	D-12	Continuous light grey outer scale	85 Cr, 7 Mo, 3 Fe, 2 Ni, 2 Si	Cr-rich carbide + Mo, Fe, Ni and Si
3	D-12	Continuous dark grey inner scale	50 Mg, 41 Si, 5 Cr, 3 Mn	Si-rich oxide + Cr and Mn + Mg (origin unknown)
4	D-12	Continuous dark grey inner scale	81 Cr, 5 Ni, 4 Fe, 4 Mo, 3 Mg, 1 Mn, 1 Si	Cr-rich oxide inner scale
5	D-12	Continuous dark grey inner scale	69 Cr, 19 Mg, 4 Mn, 2 Si, 2 Mo, 2 Fe 2 Ni	Cr-rich oxide inner scale + Mg (origin unknown)
6	D-12	Near-surface matrix (denuded)	35 Ni, 23 Cr, 18 Fe, 6 Si, 6 Mo, 1 Co	γ -matrix
7	D-12	Subsurface needle-like precipitate	77 Cr, 12 Mo, 7 Si, 2 Fe, 1 Ni	Cr- and Mo-rich carbide

RA 333 (31150) 1000X

Fig. D-12. SEM photomicrograph of RA 333 after an exposure of 9935 h at 1000°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Table D-16.

TABLE D-17
HD 556, 39145, 10,000 HOURS AT 800°C

Area ID	Fig. Ref.	Description	Composition	Probable ID of Phases
2	D-13	Black subsurface area	32 Fe, 24 Ni, 21 Co, 21 Cr, 1 Mo, 1 Si	γ-matrix, remnant of torn out carbide
3	D-13	Discontinuous light grey outer scale	93 Cr, 5 Fe, 1 Ni	Cr-rich carbide + Fe
4	D-13	Discontinuous dark grey inner scale	87 Cr, 5 Fe, 4 Si, 3 Ni	Cr-rich oxide + Si and Fe
5	D-13	Subsurface region (denuded)	32 Fe, 24 Cr, 23 Ni, 19 Co, 1 Si, 1 Mo	γ-matrix slightly depleted in Cr and Mo
6	D-13	White subsurface precipitate	24 Fe, 18 Cr, 17 Co, 15 Ni, 11 Mo, 7 Si, 8 W	Mo-W-carbide adjacent to γ-matrix base alloy
None	None	Center of specimen, base alloy	32 Fe, 23 Cr, 22 Ni, 20 Co, 2 Mo, 1 Si	Nominal alloy composition

HD 556 (39145) 1000 X

Fig. D-13. SEM photomicrograph of HD 556 after an exposure of 10,000 h at 800°C in a controlled-impurity helium environment. Numbers indicate areas analyzed by EDX and are listed in Table D-17.

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