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METALLOGRAPHIC EXAMINATIONS OF TYPE 304 STAINLESS STEEL (HEAT 9T2796) TESTED IN HIGH-TEMPERATURE UNIAXIAL AND MULTIAXIAL EXPERIMENTS

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CONTENTS

ABSTRACT	1
INTRODUCTION	1
MATERIAL	2
METALLOGRAPHIC TECHNIQUES	2
OPTICAL MICROSCOPY OF AS-TESTED SPECIMEN SURFACES	4
OPTICAL MICROSCOPY OF METALLURGICALLY PREPARED SURFACES	4
SCANNING ELECTRON MICROSCOPY	13
TRANSMISSION ELECTRON MICROSCOPY	14
OTHER TECHNIQUES	14
DEFINITION OF FAILURE MODES	17
UNIAXIALLY STRESSED SPECIMENS	20
OPTICAL MICROSCOPY	21
Creep Tests from 760 to 980°C (1400–1800°F)	21
Creep Tests at 704°C (1300°F)	21
Creep Tests at 649°C (1200°F)	23
Creep Tests at 621°C (1150°F)	27
Creep Tests at 593°C (1100°F)	27
Creep Tests at 566°C (1050°F)	29
Creep Tests at 538°C (1000°F)	29
Creep Tests at 510°C (950°F)	32
Creep Tests at 482°C (900°F)	32
SCANNING ELECTRON MICROSCOPY OF FRACTURE SURFACES	35
QUANTITATIVE CRACKING DATA	38
VARIABLY STRESSED SPECIMENS	47
MULTIAXIALLY STRESSED SPECIMENS	50
PRESSURIZED TUBES	52
TENSION-TORSION TESTING	54
METALLURGICAL STUDIES OF A NOZZLE-TO-SPHERE TEST	58
METALLURGICAL STUDY OF TT-6	64
NOTCHED BAR TESTS	67
DISCUSSION	67
CONCLUSIONS	72
ACKNOWLEDGMENTS	73
REFERENCES	73

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(HEAT 9T2796) TESTED IN HIGH-TEMPERATURE UNIAXIAL
AND MULTIAXIAL EXPERIMENTS*

R. W. Swindeman and C. W. Houck

ABSTRACT

The results obtained from a number of metallographic examinations of type 304 stainless steel specimens were compiled. Samples were obtained from uniaxial and multiaxial tests covering a very broad span of temperatures and times. Special emphasis was on the identification of failure modes, cracking patterns, grain distortion, and grain-boundary microstructures. Uniaxial specimens exhibited the following sequence of failure modes with increasing temperature and time: ductile plastic tearing, ductile plastic shear, wedge cracking, and microvoid cracking. Over most of the temperature range examined (482-871°C), $M_{23}C_6$ precipitated on grain boundaries at long times. Sigma phase and possibly ferrite were often present in the stressed areas at temperatures as low as 482°C (900°F). These metallurgical features promoted a severe loss in creep ductility at long times and low temperatures.

Most multiaxial tests were performed under conditions that promoted wedge cracking. Stress gradients also favored surface crack initiation rather than bulk damage. Testing times for multiaxial tests were less than 10,000 h; hence, there was insufficient time for the development of embrittling features such as microvoids, sigma, and ferrite. Long-time multiaxial tests to failure are recommended.

INTRODUCTION

Over a ten-year period a variety of simple and complex mechanical tests have been performed on type 304 stainless steel as part of a program to develop high-temperature structural design methods for liquid metal fast breeder reactor applications. We report the results of metallurgical studies on samples prepared from specimens tested in the program. The

*Work performed under DOE/BTP AF 15 40 10 3, Task No. OR-1.1, High-Temperature Structural Design Technology.

objective of this report is to identify microstructural changes as a function of loading and thermal history. Special emphasis is on the identification of failure modes. Nearly all the information pertains to a well-characterized reference heat of type 304 stainless steel (heat 9T2796). However, in a few instances other heats were examined, and the data are reported here.

MATERIAL

The reference heat of type 304 stainless steel was produced in 19 different product forms, including plates, pipes, bars, and formed hemispherical heads. Extensive characterization was performed to determine chemistry, grain size, hardness, tensile properties, and creep-rupture behavior.¹⁻² Three products were examined in great detail: 25-mm plate, 50-mm plate, and 16-mm bar. The 25-mm plate was studied as part of the high-temperature structural design program,³⁻⁴ the 50-mm plate was used to develop long-time engineering design data,⁵⁻⁶ and the 16-mm bar was used for producing a creep-fatigue data base.⁷

Table 1 gives the ladle chemistry and check analyses for the products. Available grain sizes are also listed. In most materials and structures tests, the starting material was in the reannealed condition produced by solution treating at 1093°C (2000°F) for 0.5 h per 25 mm (1 in.) of thickness.⁸ Aging treatments were introduced in many instances, however,⁹⁻¹⁰ and microstructures produced in aged and tested material will be presented where applicable.

METALLOGRAPHIC TECHNIQUES

Several metallographic techniques were used to examine microstructures. These ranged from simple optical microscopy (OM) of surfaces at low magnification to transmission electron microscopy (TEM) of thin foils prepared from samples. We identify each technique, briefly describe the procedures used, and give an example of the results.

Table 1. Analyses of several products of type 304 stainless steel (heat 9T2796)

Item	Form	Size (mm)	Content (wt %)													Grain size, ASTM
			Vendor analysis							ORNL analysis			Hardness, R _B			
			C	Mn	P	S	Si	Cr	Ni	Mo	C	N				
Ladle			0.048	1.22	0.028	0.015	0.48	18.6	9.7	0.32						
1	Billet ^a	355	0.05	1.30	0.026	0.011	0.45	18.84	9.63	0.32	0.05	0.028	b			3
2	Plate	4.8	0.05	1.26	0.028	0.017	0.49	18.5	9.72	0.32	b	b	b			b
3	Plate	6.3	0.05	1.26	0.022	0.016	0.48	18.52	9.64	0.32	b	b	b			b
4	Plate	9.5	0.049	1.26	0.028	0.017	0.49	18.56	9.72	0.32	0.045	0.041	63			1-4
5	Plate	12.7	0.054	1.22	0.027	0.017	0.48	18.53	9.75	0.32	0.052	0.046	66			3
6	Plate	19	0.051	1.23	0.027	0.017	0.48	18.52	9.68	0.33	0.051	0.046	65			3
7	Plate	25	0.048	1.28	0.030	0.016	0.48	18.44	9.72	0.32	0.051	0.031	65			1-3
8	Plate	51	0.050	1.23	0.026	0.016	0.49	18.44	9.72	0.32	0.055	0.04	65			1-2
10	Pipe	102	0.046	1.23	0.026	0.020	0.50	18.57	9.58	b	0.052	0.051	63			4
11	Pipe	203	0.041	1.24	0.029	0.017	0.55	18.56	9.66	b	0.056	0.042	69			2
15	Pipe ^c	64	0.045	1.28	0.028	0.014	0.54	18.56	9.83	b	0.050	0.047	67			3
51	Gun-drilled bar ^d	64	b	b	b	b	b	b	b	b	0.051	0.050	65			3
18	Bar ^d	16	0.059	1.26	0.033	0.015	0.44	18.6	9.5	0.35	0.047	0.031	64			0-3
23	Bar ^c	16	0.044	1.26	0.033	0.016	0.45	18.64	9.5	0.34	b	b	68			3
19	Bar	25	0.045	1.28	0.033	0.016	0.46	18.6	9.54	0.35	0.048	0.030	64			0-3
20	Bar	44	0.049	1.26	0.033	0.015	0.45	18.6	9.5	0.35	0.052	0.048	65			3
21	Bar	48	0.045	1.26	0.033	0.015	0.47	18.6	9.58	0.35	0.053	0.048	65			3-5
22	Bar	114	b	b	b	b	b	b	b	b	0.051	0.049	67			3

^aORNL analysis.

^bNot determined.

^cCold drawn.

^dHot rolled.

Source: R. W. Swindeman, W. J. McAfee, and V. K. Sikka, "Product Form Variability in the Mechanical Behavior of Type 304 Stainless Steel at Room Temperature and 523°C (1100°F)," pp. 41-64 in *Reproducibility and Accuracy of Mechanical Tests*, ASTM STP 626, American Society for Testing and Materials, Philadelphia, 1977.

OPTICAL MICROSCOPY OF AS-TESTED SPECIMEN SURFACES

Optical microscopy was often performed at low magnification to show specimen distortion (Fig. 1) or macroscopic cracking patterns (Fig. 2). No special surface preparation was necessary here. For higher magnification work (e.g., 100 $\times$ or more), electropolished surfaces were needed. On such surfaces slip bands were observed [Fig. 3(a)] in addition to oxidation patterns [Fig. 3(b)] and cracking patterns. One drawback of electropolishing was the tendency to groove grain boundaries and produce pits at some types of inclusions. We generally reannealed the specimens after polishing. The grain boundaries often migrated from the grooves during the reannealing.

Electropolishing was also helpful in removing oxide layers for the examination of carbide-decorated slip bands [Fig. 4(a)] and microcracks [Fig. 4(b)].

OPTICAL MICROSCOPY OF METALLURGICALLY PREPARED SURFACES

High-magnification optical microscopy was used to study metallurgical features such as grain size, twin size, stringers, inclusions, grain-boundary and matrix precipitates, sigma and martensite formation, grain-boundary migration, recrystallization, slip bands, cracking behavior, and corrosion attack. At Oak Ridge National Laboratory (ORNL), small specimens were prepared by techniques described by Long and Gray.¹¹ Specimens were mounted in plastic and polished on vibratory platens that produced minimum smearing and rounding of edges. Large samples were either sectioned and prepared by the above method, or they were prepared in situ by field metallography procedures described by Snyder and Leslie.¹²

Inspection of the as-polished specimens was important in the metallographic studies and revealed such features as inclusion counts and ferrite stringers in as-received material (Fig. 5), wedge crack density (Fig. 6), and microvoid density (Fig. 7).

Fig. 1. Gross distortion of a tubular specimen of type 304 stainless steel tested in creep rupture at 593°C (1100°F). Photo courtesy of Y-12 Plant.

Fig. 2. Microcracking on the outside of a tubular specimen of type 304 stainless steel tested in creep rupture at 593°C (1100°F).

CYN-2615

Y-143608

(a) 100 μm

(b) 100 μm

Fig. 3. High magnification of the surface of type 304 stainless steel showing (a) slip bands on the surface of a sample strain cycled at 0.65% strain range and 482°C (900°F) and (b) oxide islands on the surface of a sample stress cycled between 138 and 0 MPa at 593°C (1100°F).

Y-141955

Y-142761

(a) 100 μm

(b) 100 μm

Fig. 4. Surface microstructure revealed by removing the oxide layers on specimens strain cycled at (a) 593°C (1100°F) and (b) 649°C (1200°F).

Y-11443

	Fer- rite	Austen- ite
Content, %		
Fe	67	67
Cr	27	19
Ni	5	11
Mn	1	1
Cr/Fe	0.40	0.28

Fig. 5. Inclusions and ferrite stringers on the surface of polished specimens.

The choice of etchants was also very important. Early in the program, we generally chose chemical etchants based on combinations of water, hydrochloric acid, and nitric acid, typically 50 mL HCL, 10 mL HNO₃, remainder water. This etchant was needed to reveal grain size, twins, and recrystallization [Fig. 8(a)]. The problem with the chemical etchant for examining tested samples, however, was that it often produced heavy attack of grain-boundary carbides and grain surfaces and made it difficult to interpret high-magnification photomicrographs. Figure 8(b) is an example of heavy attack. Murakami's reagent* was especially useful in the study of creep-tested type 304 stainless steel. This etchant stained sigma

*Murakami's reagent: 10 g K₃Fe(CN)₆, 10 g KOH, and 100 mL water; immerse in boiling solution.

(a)

1 mm

(b)

Fig. 6. Wedge-cracking patterns introduced into the walls of tubular specimens tested at 593°C (1100°F) under (a) 172 MPa (25 ksi) constant stress, as polished (1000 h, monotonic), and (b) +172 MPa (25 ksi)/-155 MPa (-22.5 ksi) reversal stresses, as polished (3600 h, cyclic).

Y-177506

Y-188279

Fig. 7. Microvoid cracking patterns produced by (a) long time (45,000 h) creep testing at 593°C (1100°F) and 110 MPa (16 ksi), as polished, and (b) long hold time (24 h) creep-fatigue tests at 593°C (1100°F) and 0.65% strain range, as polished.

phase, ferrite, and carbides without heavily attacking the grain boundaries or grain surfaces. The light attack permitted us to examine microstructures at magnifications up to 1500 $\times$. Figure 9(a) shows the typical structure at conditions under which sigma phase formed.

Another useful etchant was Beraha's reagent.[†] Although originally developed to reveal martensite, the etchant stained grain surfaces [Fig. 10(a)], feathery martensite produced by polishing [Fig. 10(a)], deformation martensite [Fig. 10(b)], and thermal martensite [Fig. 10(c)] very nicely. The carbides on the grain boundaries and in the matrix were darkly etched, as was δ -ferrite. Sigma phase and sulfide inclusions, however, were not stained and could be clearly identified [Fig. 10(d)]. The Beraha's etch permitted good resolution at magnifications up to 1500 $\times$.

[†]Beraha's reagent: 2 g ammonium biftouride, 2 g potassium metabisulfite, 10 mL HCl, and 50 mL water.

Y-131999

Y-168687

(a)

200 μm

(b)

15 μm

Fig. 8. Typical microstructure revealed by chemical etching. Etchant: HCL and HNO_3 . (a) Low magnification showing grain size and other coarse features. (b) High magnification showing poor resolution in grain-boundary areas and pitting at matrix precipitates.

Y-190958

Y-192777

(a)

100 μm

(b)

10 μm

Fig. 9. Typical microstructures revealed by Murakami's reagent. (a) Solution-treated specimen showing no response. (b) Creep-tested specimen [10,000 h at 649°C (1200°F)] showing sigma phase, ferrite phase, and matrix carbides.

Y-191711

Y-175201

(a) 100 μm

(b) 50 μm

Y-174940

Y-191732

(c) 40 μm

(d) 40 μm

Fig. 10. Typical microstructures revealed by Beraha's reagent. (a) Solution-treated specimens showing grain structure, compositional banding, and feathery martensite from polishing. (b) Tensile-tested specimens showing grain distortion and deformation martensite. (c) Creep-tested specimen revealing grain structure, feathery martensite, and thermal martensite on grain boundaries. (d) Creep-tested specimen showing grain structure, sigma phase, and matrix carbide structure.

Y-175429

Y-175428

Fig. 11. Microstructure revealed by magnetic etching. (a) Magnet on; colloidal particles are collected on grain boundaries. (b) Magnet off; colloidal particles diffuse onto grain surfaces.

The magnetic etchant developed by Gray was used somewhat sparingly.¹³ This etchant consisted of colloidal magnetic particles suspended in water. In the presence of a magnetic field, the particles collected at ferromagnetic phases such as ferrite, martensite, and magnetic $M_{23}C_6$ [Fig. 11(a) and (b)].

SCANNING ELECTRON MICROSCOPY

The usefulness of the scanning electron microscope (SEM) was limited because the testing was performed in air. Thus, oxidation obscured most of the fine detail on fracture surfaces. The scanning work therefore served only to confirm the conclusions from OM studies.

Typical SEM results (Figs. 12 and 13) include examination of surface fatigue crack initiation [Fig. 12(a)], fatigue crack growth by ductile striations [Fig. 12(b)], and microvoid growth patterns on the fracture surface of specimens that failed predominantly by intergranular fracture [Fig. 12(c)]. In a few instances, unoxidized fracture surfaces were studied by (1) breaking the surfaces apart at liquid nitrogen temperatures [Fig. 13(a)], (2) fatigue breaking a sample exposed to high-temperature creep [Fig. 13(b)], or (3) metallographically preparing sliced sections. In this last technique, samples were studied in both the as-polished and etched conditions as illustrated in Fig. 13(c) and (d). The SEM studies of unoxidized fracture surfaces were helpful in examining the shapes and sizes of carbide precipitates and sigma particles.¹⁴

TRANSMISSION ELECTRON MICROSCOPY

Much of the TEM on the reference heat of type 304 stainless steel was performed by Moteff and coworkers, and a number of available reports and papers describe their techniques and results.¹⁵⁻¹⁹ Information obtained from these studies includes carbide particle characterization and precipitation kinetics, cell and subgrain size and orientation measurements, dislocation density studies, stacking fault energy, examination of precipitate-dislocation interactions, and grain-boundary migration. We do not cover details of TEM studies here.

OTHER TECHNIQUES

We do not discuss in any detail a number of specialized metallographic techniques used to study the microstructure of the reference heat of type 304 stainless steel. However, the reader should note that a great deal of work has been supported by the materials science programs. Typical examination methods include TEM (refs. 19-22), microprobe examinations,⁸ quantitative metallography,²³ and small-angle neutron scattering.²⁴⁻²⁵

M-12470

(a)

0.2 mm

(b)

100 μ m

M-16829

(c)

100 μ m

Fig. 12. Scanning electron micrographs of specimens tested in fatigue.
(a) Intergranular surface cracking.
(b) Ductile transgranular fatigue striations on the fracture surface.
(c) Intergranular fracture surface.

M-18812

M-0396

(a) 50 μm

(b) 10 μm

M-18682

M-19096

(c) 10 μm

(d) 50 μm

Fig. 13. Scanning electron micrographs of creep-tested specimens prepared by (a) breaking open at liquid nitrogen; (b) fatiguing apart at room temperature; (c) cutting and polishing, as polished; and (d) cutting, polishing, and etching. Etchant: HCl and HNO₃.

DEFINITION OF FAILURE MODES

The different types of failure that occur in structural materials as a result of uniaxial mechanical stress and monotonic strains have been brought together by Ashby and Tomkins in terms of a failure map.²⁶ They placed a temperature scale on the abscissa and a stress or modulus-compensated stress scale on the ordinate. On the grid are plotted points representing tests that produced failures. Fields are constructed in which all the test data exhibit a certain characteristic failure mode. These modes include cleavage, ductile transgranular fracture, transgranular creep, intergranular creep, and dynamic recrystallization. Ashby and Tomkins collected and interpreted predictive models that correlated failure in each of these regions and placed special emphasis on the stress and temperature dependence of each failure mode. The failure map is a rather simple way of estimating failure modes and the limit of extrapolation of creep rupture data with respect to either time or stress. However, the studies performed on austenitic steels suggest that the field positions are not unique to a grade of material (e.g., type 304 stainless steel) but change significantly with minor compositional variation, grain size, and heat treatment.

In our examinations of failure modes in the reference heat of type 304 stainless steel, we did not use the categories identified by Ashby and Tomkins. Furthermore, we did not attempt to associate any specific failure mode with a failure model. We did this partly because the failures were often of a compound type in which several damaging phenomena were clearly active. We identified one of four failure modes as the most prominent. These are schematically shown in Fig. 14. First, at low temperatures [e.g., from room temperature to 427°C (800°F)], the short-time tensile failures were initiated by a ductile tearing mode (the cup part of the cup-and-cone failure). The high stresses and multiaxial stress state in the center of a necked sample produced nucleation and growth of voids by a plastic deformation mechanism. Viewed on the fracture surface, the

ORNL-DWG 83-19809

Fig. 14. Failure modes produced by (a) low-temperature tensile tests [cup-and-cone (ductile tearing)], (b) intermediate temperature tensile tests (shear-off), (c) short-time creep tests (wedge), and (d) long-time creep tests (microvoid).

voids appeared as the plastic dimples. Viewed from the cross section, these voids had a rounded appearance when isolated and a jagged transgranular pattern after total separation. Severe grain distortion always accompanied this failure mode.

Over a narrow range of temperatures [e.g., 427 to 538°C (800 to 1000°F)] and short testing times, sometimes a ductile shearing failure mode occurred. Again, necking occurred before failure, but, instead of the classical cup-and-cone appearance, the failure occurred entirely by shear on one plane inclined to the specimen axis. Local grain distortion was always severe, although the elongated shear dimples could be observed at high magnification. Although we distinguished between ductile tearing and ductile shearing, they were manifestations of the same basic failure mode. We suspect that the ductile shear mode dominated under conditions in which strain aging mechanisms were most active.

Wedge cracking was a fairly ductile failure mode, in which sharp-pointed cracks generally nucleated at grain-boundary triple points and grain-boundary-twin intersections. Often, these wedge cracks grew by the nucleation and coalescence of microvoids at the crack tip or in front of the crack tip. Moderate grain distortion and grain-boundary sliding were characteristics of wedge cracks. Furthermore, wedge cracks were sometimes

blunted by plastic strains toward the end of life. Wedge cracking always occurred between the extreme modes — plastic tearing (or shear) and microvoid cracking — and usually were at shallow angles to the applied stress.

Microvoid cracking represented the long-time failure mode at temperatures in the creep regime. These voids, rarely more than a few micrometers in diameter, nucleated and coalesced on grain boundaries normal to the applied stress. Microcracking was a low-ductility failure mode; however, after complete grain-boundary facets had cracked, subsequent sliding of boundaries oriented for shear often opened the boundaries and gave the cracked boundaries an appearance of the wedge-cracking mode. The failure mode map for the reference heat is shown in Fig. 15.

Fig. 15. Failure mode map for type 304 stainless steel heat 9T2796.

UNIAXIALLY STRESSED SPECIMENS

Hundreds of test samples of the reference heat were metallographically examined. Tensile and creep specimens cover temperatures from 25 to 980°C, with times from a few seconds to more than 50,000 h. The broadest range of testing variables was imposed on the 25-mm plate, and the longest test times at the lowest stresses were for the 50-mm plate. In Fig. 16, we plot points corresponding to metallurgically examined specimens. The stress corresponds to the engineering ultimate strength for tensile tests and the constant load or engineering stress applied in the creep tests. In the subsequent discussions we begin with the highest temperature and shortest time data and work toward lower temperatures.

Fig. 16. Stress-versus-temperature chart showing the range of test conditions in metallurgically examined specimens.

Annealing studies of 25- and 50-mm plate revealed virtually no change in grain size or other microstructural features as a result of solution treatment at temperatures from 1065 to 1100°C (1950 to 2000°F). Typical microstructures are shown in Fig. 17 for the 25-mm plate. On the other hand, nearly all the bar products exhibited some degree of duplex grain size after such annealing. An example for the 16-mm bar is shown in Fig. 18. This duplex grain size added to the variability in the yield strength and creep-rupture behavior.

OPTICAL MICROSCOPY

Creep Tests from 760 to 980°C (1400–1800°F)

Testing times from 760 to 980°C (1400–1800°F) were relatively short (1000 h or less). The microstructures revealed a wedge cracking mode (Fig. 19). Longer time test samples contained globular grain-boundary and twin-boundary carbides. Considerable grain distortion occurred, as would be implied by high elongation values, which typically exceeded 40%. Recrystallization occurred near the fracture surfaces, where grain distortion was the greatest. Grain-boundary migration was also observed. No high-resolution microscopy was performed, nor was any effort made to identify microvoid formation patterns.

Creep Tests at 704°C (1300°F)

Only a few creep tests on the 25-mm plate were performed at 704°C (1300°F), and these covered times to 3100 h. Specimens from the 50-mm plate, however, were tested for longer than 30,000 h; metallographic data were described in some detail by Sikka.⁶ Specimens that failed in short times exhibited wedge cracking [Fig. 20(a)] and considerable grain distortion; with increasing testing time, microvoids appeared on uncracked grain boundaries [Fig. 20(b)], and grain distortion decreased considerably. Heavy carbide precipitation was observed on grain boundaries and twin boundaries at long times. High-magnification studies by Sikka revealed needle-shape matrix precipitates and massive precipitates that appeared to be sigma phase. Carbides of the form Cr_{23}C_6 were also identified by microprobe methods.^{6,14}

Fig. 17. Microstructure of type 304 stainless steel (25-mm plate of heat 9T2796) annealed at (a) 980°C (1800°F), (b) 1023°C (1900°F), (c) 1065°C (1950°F), and (d) 1093°C (2000°F). Etchant: HCl and HNO₃.

Fig. 18. Typical variation in the grain size within a bar product (16-mm diam), etched with HCl and HNO_3 . (a) Fine-grained region. (b) Coarse-grained region.

One of the specimens from the 25-mm plate exhibited a coarser grain size than typical (200 μm instead of 120 μm). The rupture life of the specimen was shorter than expected and more closely approached the life typical of the 50-mm plate at the same stress and temperature.

Creep Tests at 649°C (1200°F)

Samples from the 25-mm plate were tested at 649°C (1200°F) for 0.1 to 10,000 h or more. Most of the metallurgical examinations were performed at the University of Cincinnati. The 50-mm plate was tested by McCoy⁵ and Sikka^{6,27} for longer than 50,000 h and examined metallurgically by Sikka.^{6,27} Metallurgical examinations of creep-tested specimens have also been reported by Chopra and Natesan,²⁸ Ajaja and Ardell,²⁹ and Sikka and coworkers.³⁰ Typical microstructures for short and long testing times are shown in Fig. 21 for tests on the 25-mm plate at 649°C (1200°F). The

Y-132030

Y-132028

(a)

(b)

Y-132017

Y-132024

(c)

(d)

200 μm

Fig. 19. Typical microstructures developed in specimens tested at high temperatures: (a) 137 h at 871°C (1600°F) and 34 MPa (5 ksi), (b) 731 h at 816°C (1500°F) and 34 MPa (5 ksi), (c) 0.75 h at 760°C (1400°F) and 138 MPa (20 ksi), and (d) 698 h at 760°C and 52 MPa (7.5 ksi). Etchant: HCl and HNO_3 .

Y-132015

(a)

Y-130858

(b)

Y-132022

(c)

Fig. 20. Microstructures developed in specimens tested at 704°C (1300°F): (a) 8.7 h at 138 MPa (20 ksi) and (b) and (c) 2296 h at 52 MPa (7.5 ksi); (a) and (c) etched with HCl and HNO₃, (b) as polished.

Fig. 21. Microstructures developed in specimens tested at 649°C (1200°F): (a, 2.8 h at 207 MPa (30 ksi) and (b), (c), and (d) 9668 h at variable stresses up to 86 MPa (12.5 ksi); (a) etched with HCl and HNO₃, (b) as polished, (c) etched with Murakami's reagent, and (d) etched with Beraha's reagent. Note grain-boundary migration in (c) and (d). The sigma phase is also present.

microstructure at short time revealed little or no evidence of matrix precipitation, but high-resolution metallography showed some grain-boundary precipitation of carbides. The grain distortion was moderate, producing elongation values of about 30%. Failures were generally of a wedge cracking mode, although fairly large microvoids often occurred on uncracked grain boundaries. Twin-boundary cracking was also observed under some conditions, as reported by Michel, Nahm, and Moteff¹⁷ and Sikka and coworkers.³⁰ Long-time microstructure indicated the presence of grain-boundary and matrix carbides. Sigma phase was observed in the stressed regions. Microvoids were present on boundaries normal to the applied stress, and grain-boundary migration was also present [Fig. 21(c)].

Creep Tests at 621°C (1150°F)

Specimens of 25-mm plate were tested from 10 to 33,000 h at 621°C (1150°F). Again, the samples from short-time tests revealed fairly simple microstructures at low magnifications. For example, Fig. 22(a) shows the microstructure for a specimen tested for 21 h at 207 MPa (30 ksi). Moderate grain distortion (29% elongation) and wedge cracking are seen. Fine carbides were present on some boundaries. Photomicrographs of the long-time test at 621°C (1150°F) are shown in Fig. 22(b) and (c). These reveal the microvoid formation patterns in the as-polished surface [Fig. 22(b)] and the matrix carbides, grain-boundary carbides, microvoids, and sigma phase after Murakami's and Beraha's stain etches. An unidentified gray-etching phase appeared near the sigma particles [Fig. 22(d)]. This is possibly a ferrite phase, which has been observed in type 304 stainless steel tested at 650°C (1202°F) by Muddle.³¹ Regions of matrix carbide denuding often occurred in regions of sigma formation.

Creep Tests at 593°C (1100°F)

A large number of creep-tested specimens were accumulated on all product forms of the reference heat at 593°C (1100°F). Testing times ranged from shorter than 1 h to longer than 40,000 h for a variety of loading patterns. Nearly all the structure test data were obtained at 593°C (1100°F). Again, short-time creep-rupture tests exhibited a wedge

Fig. 22. Microstructures developed in specimens tested at 621°C (1150°F): (a) 21 h at 207 MPa (30 ksi) and (b), (c), and (d) 31,527 h at 86 MPa (12.5 ksi); (a) etched with HCl and HNO₃, (b) as polished, (c) etched with Murakami's reagent, and (d) etched with Beraha's reagent. The sigma phase is present in (c) and (d). Note the gray etching phase in (d), which may be ferrite.

cracking mode [Fig. 23(a)]. Wedge cracks were often blunted near the failure surface, and some grain matrix tearing was observed in scanning micrographs. Moderate grain distortion was also observed. For testing times beyond a few hours, grain-boundary carbides occurred.

Long-time creep-rupture tests revealed microstructures containing matrix and carbide precipitates. Sometimes the matrix precipitate structure varied from the surface to the midsection [Fig. 23(b)], where the carbides were aligned in planar arrays in the surface grains and in a cellular pattern in the internal grains. Precipitation seemed to be heavier in the internal grains. Similar microstructures were observed by Sikka for specimens tested at higher temperatures.⁶

The microvoid density was quite high in the long-time tests, and the grain distortion was relatively low [Fig. 23(c) and (d)]. Indeed, nearly all the tertiary creep behavior could be attributed to microvoid formation.⁴

Sigma phase was prevalent. Often the sigma seemed to develop as plates or rods moving in the direction of grain-boundary migration. The sigma phase on grain boundaries normal to the applied stress was often linked with microvoids (Fig. 24).

Creep Tests at 566°C (1050°F)

Creep tests on the 25-mm plate ranged from 10 to 2000 h at 566°C (1050°F). Microstructures revealed wedge cracking at short times, with grain boundary carbide precipitates. The 2000-h test revealed wedge cracking and fine carbide precipitates within the matrix and on grain boundaries. Microvoids were also present. No examinations for other phases were undertaken.

Creep Tests at 538°C (1000°F)

Creep tests at 538°C (1000°F) on 25-mm plate ranged from 10 to 30,000 h. Again, short-time tests revealed wedge cracking, with some crack blunting by plasticity near the failure surface. The failure surface was sometimes a mixture of transgranular ductile tearing and blunted wedge cracks [Fig. 25(c)]. Fine carbides were observed on grain boundaries of stressed regions.

Y-132005

R-39575

Y-164618

Y-193115

Fig. 23. Microstructures developed in specimens tested at 593°C (1100°F): (a) 98 h at 207 MPa (30 ksi), (b) 9584 h at 138 MPa (20 ksi), and (c) and (d) 44,296 h at 110 MPa (16 ksi). Etchants: (a) and (b), HCl and HNO₃; (c), Murakami's reagent; and (d) Beraha's reagent.

Fig. 24. High-magnification of specimens tested for long times at 593°C (1100°F): (a) and (b) 12,755 h at 138 and 69 MPa (20 and 10 ksi), etched with Murakami's and Beraha's reagents, respectively, (c) and (d) 44,296 h at 110 MPa (16 ksi), etched with Murakami's and Beraha's reagents, respectively.

Fig. 25. Microstructures developed in specimens tested at 538°C (1000°F): (a) 60 h at 311 MPa (45 ksi) and (b), (c), and (d) 28,000 h at variable stresses between 207 and 10 MPa (30 and 1.5 ksi); (a) etched with HCl and HNO₃, (b) as polished, (c) etched with Murakami's reagent, and (d) etched with Beraha's reagent.

Long-time creep tests revealed microvoids and grain-boundary and very fine matrix carbides [Fig. 25(c)]. Small particles of sigma phase were in the stressed regions. Again, regions of carbide and sigma formation were observed in the wake of migrating grain boundaries. Similar microstructures were observed in specimens of 50-mm plate creep tested longer than 30,000 h (ref. 6). Similar to those in long-time tests at 593°C (1100°F), failures occurred by the coalescence of microvoids on grain-boundary facets normal to the applied stress. Grain distortions were small, and total elongations were 10% or less.

Creep Tests at 510°C (950°F)

Short-time creep failures at 510°C (950°F) exhibited microstructural features similar to those from tests at 538°C (1000°F); compare Figs. 25(a) and 26(a). Failures were initiated by wedge cracking, but plasticity dominated the final separation. Fine carbides were observed on grain boundaries.

The longest test at 510°C (950°F) was about 55,000 h at 172 MPa (25 ksi). The test was discontinued in what appeared to be the end of the primary creep stage. The microstructure revealed a relatively high density of fine microvoids [Fig. 26(b)] and sigma formation [Fig. 26(c)]. Again, grain-boundary migration produced regions of what appeared to be high carbide densities.

Creep Tests at 482°C (900°F)

Short-time tests at 482°C (900°F) failed primarily by plastic instability, assisted by loss of load-bearing area because of wedge cracking. This failure mode actually extended to fairly long times [Fig. 27(a)]. The microstructure for a test that lasted 3621 h at 411 MPa (45 ksi) reveals wedge cracks blunted by plasticity. Fine grain-boundary carbides were also present on some grain boundaries in the stressed regions. Longer time test samples were carefully examined in grain-boundary regions. An example of the observed microstructure is typified in Fig. 27(b), which shows the grain-boundary region for a 15,000-h test. Here microvoid formation is clearly evident. Carbide precipitation

Y-131996

Y-191806

Y-191819

Y-192061

Fig. 26. Microstructures developed in specimens tested at 510°C (950°F): (a) 270 h at 311 MPa (45 ksi) and (b), (c) and (d) 55,450 h at 172 MPa (25 ksi); (a) etched with HCl and HNO₃, (b) and (c) etched with Murakami's reagent, and (d) etched with Beraha's reagent.

Y-131994

Y-190484

Y-190790

Y-191755

Fig. 27. Microstructures developed in specimens tested at 482°C (900°F): (a) 3620 h at 311 MPa (45 ksi) and (b), (c), and (d) 14,490 h at 276 MPa (40 ksi). Note in (a) that the wedge cracks have been blunted by plasticity; (a) etched with HCl and HNO₃, (b) as polished, (c) etched with Murakami's reagent, and (d) etched with Beraha's reagent.

sometimes occurred in clusters, as reported by Sikka after the examination of a specimen from a 50-mm plate that failed near 35,000 h (ref. 6). Another precipitate that we have not identified also appeared on the grain boundaries [Fig. 27(c)]. Possibly this could be a ferrite phase, but confirmation is necessary. Studies of matrix microstructures have not been performed in sufficient detail to provide a full characterization. We expect that some Cr_{23}C_6 precipitates will form, possibly in needlelike morphologies.

Grain distortion at 482°C (900°F) was relatively small and largely produced by the initial loading strains. Creep strains were generally less than a few percent.

SCANNING ELECTRON MICROSCOPY OF FRACTURE SURFACES

Typical SEM photographs are shown in Fig. 28. These were taken from specimens tested at the lower temperatures, where fracture surfaces were not greatly obliterated by oxidation or recrystallization effects. Figure 28(a) and (b) shows the highly intergranular failure that occurred in 3510 h at 241 MPa (35 ksi) and 510°C (950°F). Details on the fracture surface are too fine to be resolved at the magnification chosen for SEM in Fig. 28(a). Higher magnification studies revealed that the dimples and particles lying on the boundaries were about 1 μm in size [Fig. 28(b)]. Similar photos are shown in Fig. 28(c) and (d) for a specimen that failed after 2054 h at 172 MPa (25 ksi) and 566°C (1050°F). Again, the failure surfaces were almost entirely intergranular. Some grains exhibited rather smooth textures, with particles (probably oxidized precipitates) covering large regions. These particles ranged from 1 to 5 μm . Other grain surfaces were covered with dimples ranging from 2 to 10 μm [Fig. 28(d)]. Scanning electron microscopy of sectioned and polished surfaces was undertaken on a limited basis. The work at the University of Cincinnati was hampered by a rounding off of polished edges.¹⁶ Chemically etched samples often showed a great deal of pitting, and samples exposed to Murakami's reagent did not have enough attack to permit meaningful examination. Scanning work on surfaces exposed by breaking open the samples produced

Fig. 28. Scanning electron micrographs of specimens creep tested at lower temperatures: (a) and (b) 3510 h at 241 MPa (35 ksi) and 510°C (950°F); (c) and (d) 2054 h at 172 MPa (25 ksi) and 566°C (1050°F). University of Cincinnati micrographs.

the most interesting results. Typical SEM photos produced by this procedure are in Fig. 29, showing smooth and dimpled grain surfaces exposed on a sample tested for 55,000 h at 172 MPa (25 ksi) and 510°C (950°F). The carbides, sigma, and possible ferrite phases can be observed on the fracture surface. The chromium-to-iron ratio is indicated for selected areas and approached values expected for sigma phase at some locations. One foil was examined by electron transmission microscopy. No sigma phase or ferrite was found on the grain boundaries, but this is not unexpected because of the very limited grain-boundary volume included in the foil.

Scanning electron microscopy in conjunction with Auger studies has also produced interesting results. For example, White, Padgett, and Swindeman used this method to establish the presence of sulfur on creep-induced microvoids (Fig. 30) (ref. 32).

QUANTITATIVE CRACKING DATA

The microcracking characteristics of the 25-mm plate were studied in considerable detail by Moteff and coworkers.^{16-18,33-34} Generally, the metallographic specimens were sectioned and polished parallel to the tensile axis; etched with a mixture of nitric acid, hydrochloric acid, and water (1:3:4 parts, respectively); and examined at magnifications of about 200×.

Each crack length was measured on the basis of the fraction of grain-boundary facet covered and the angle of the crack to the stress axis. The locations of the cracks with respect to triple points and twin-boundary intersections were also determined. No data on microvoids (e.g., cracks less than 2 μm long) were collected. Crack density was measured over an area of about 75 mm^2 . Supplementary data were produced from measurements at ORNL. In the latter case, a smaller area was sampled (e.g., around 15 mm^2). Crack density was measured from the failure edge toward the shoulder of the specimens. Often, there was a significant change in crack density with the distance from the fracture zone (Fig. 31) for test data processed by Nahm and Moteff for tests at 649°C (1200°F) (ref. 18). The University of Cincinnati work sampled almost all the gage section, but ORNL studies covered only the first 6 to 8 mm. Several factors

H-18855

(a)

20 μm

H-18860

(b)

5 μm

Fig. 29. Scanning electron micrographs of the fracture surfaces of a specimen broken in liquid nitrogen after 55,450 h at 510°C (950°F) and 172 MPa (25 ksi). Particle 1 is approximately 8 μm in diameter and has a Cr:Fe ratio near 0.77, which is close to that expected for sigma phase. Area 2 has a Cr:Fe ratio near 0.45, which also indicates chromium enrichment relative to type 304 stainless steel base composition (see Fig. 5).

Y-176610

Y-176611

(a)

5 μ m

(b)

10 μ m

Fig. 30. Sulfur traces from Auger analysis on the fracture surface of a specimen broken in liquid nitrogen after creeping into the tertiary stage for 700 h at 593°C (1100°F) and 207 MPa (30 ksi). (a) Secondary electron image. (b) Sulfur map (150-eV peak) showing sulfur enrichment on creep cavity surface.

ORNL-DWG 83-19808

Fig. 31. Wedge-crack density versus distance toward fracture for specimens tested at 649°C (1200°F) at three tensile strain rates.

bear on the validity of comparing and interpreting the crack density data. For example, the cracking uniformity depended on the testing temperatures and times. Short-time high-stress tests tended to show greater crack density gradients than did long-time low-stress tests. Second, not all data gathered were from ruptured test specimens. In fact, some tests were discontinued in the primary and secondary creep stages. We would expect such samples to contain lower crack densities and shorter cracks, although data did not show this trend. Oddly, Bhargava and Moteff reported creep crack densities in some samples that could not possibly contain cracks because of the short testing times and low stresses.¹⁶ Specimen R125, discontinued after 1580 h at 172 MPa (25 ksi) and 482°C (900°F), is an example of this problem. The cracks in such samples were probably etching effects on sensitized grain boundaries. All ORNL measurements of cracks were performed on polished but unetched specimens to avoid this problem. Finally, the grain size varied significantly from specimen to specimen, affecting crack density. The ORNL crack density measurements were made with and without adjustments for grain size variations. Deformation itself affected the grain density projected on the plane of measurement, but this rather minor adjustment was not used.

In Fig. 32, we plotted the crack densities on a grid of stress versus temperature. Several features emerge. The first is the apparent difference between crack densities measured at ORNL and those measured at the University of Cincinnati.¹⁶ Densities from ORNL measurements were roughly higher by a factor of 2. The second is a trend toward higher crack densities at higher temperatures, roughly doubling from 482 to 871°C (900 to 1600°F). Third, no clear trend of crack density occurred with stress. One exception to this was the study by Nahm and Moteff which indicated an increase in wedge crack density with decreasing stress at 649°C (1200°F) (ref. 18). Sikka observed a similar trend for the 50-mm plate.⁶

Figure 33 shows results from the ORNL crack density studies. The crack density is plotted against temperature in Fig. 33(a). The trend of increasing crack density with increasing temperature may be visualized despite the large variation in crack density at any one temperature. We believe that some of this variation may be due to grain size variation; in

Fig. 32. Wedge crack density variation with stress and temperature.

Fig. 33(b) we plotted the number of grains per square millimeter against temperature. This plot reveals a factor of 2 or more variation in the grain density. Finally, in Fig. 33(c), we plotted the ratio of grain density to crack density. With the exception of two ratios that were quite high, the data mostly fall in the range of 2 to 4. This suggests about one wedge crack per three grains. There seems to be a trend toward lower ratios with increasing temperature, which implies higher crack density with increasing temperature.

In the examination of crack density data, one must be mindful that the crack lengths may range from a fraction of a facet to several facets in length. Data from the measurements of Bhargava showing the general

Fig. 33. Data from measurements on creep-tested specimens. (a) Wedge crack density. (b) Grain density. (c) Grains per crack.

trend for crack lengths are plotted as histograms in Fig. 34. The length-selected increments increase as the bars move to the right, with 1 representing one grain-boundary facet and 2 representing two grain-boundary facets. Data clearly reveal a trend toward a decreasing number of cracks as the length increases. This would be expected. Most cracks were one facet or less. The high number that consistently occurred at unity may be due to an etching effect mentioned previously.

Fig. 34. Frequency of wedge cracks as a function of crack length.

Histograms for crack angles relative to the stress axis are shown in Fig. 35 for those cracks of about one facet in length. These histograms reveal that most cracks were oriented in the 80 to 90° range.

Fig. 35. Frequency of wedge cracks as a function of crack angle.

VARIABLY STRESSED SPECIMENS

From the beginning of the high-temperature structural design methods program, variable-stress and variable-temperature tests have played a major role in developing constitutive equations and failure criteria. Strain cycling and fatigue testing have been emphasized, of course, but a few variable-stress creep tests and variable-temperature creep tests have also been performed. Substructures, precipitates, dislocation cell sizes, and subgrain misorientation angles produced by fatigue and creep-fatigue have been fully characterized by Moteff and coworkers for the reference heat of type 304 stainless steel.^{16,19-20} These features have been correlated with stress history. More recently, surface crack characterizations have also been performed.³⁵ The key features of the fatigue or the creep-fatigue failure mode is the tendency for surface crack initiation. This seems to be true for both transgranular and intergranular crack initiation modes. Part of the reason for this surface crack initiation is that most of the fatigue testing has been under short-time failure conditions, where the surface fatigue damage component dominates the bulk damage characteristic of long-time creep-rupture conditions. On the other hand, evidence of internal wedge cracking and microvoid formation under fatigue conditions has been reported by Maiya.³⁶ This occurred in the reference heat of type 304 stainless steel under slow-fast and tensile relaxation hold cycling conditions. At ORNL microvoids were also found to develop in another heat of type 304 stainless steel under short-time testing conditions³⁷ (Fig. 36). Other heats may show no tendency to form wedge cracks or microvoids under similar testing conditions. Those heats that produce microvoids under short-time creep conditions tend to exhibit creep cavities under creep-fatigue conditions.

Creep-type cavitation has also been produced under two-bar thermal ratchetting conditions.³⁷ Typical results are shown in Fig. 37. Bar 1 experienced long hold periods at high temperature but at stresses near zero, and bar 2 experienced similar time-temperature profiles but at high stresses during the hold period. Bar 1 contained only a few plasticity-induced surface cracks, but bar 2 was heavily cavitated throughout the cross section.

Fig. 36. Microvoids that developed in type 304 stainless steel (heat 55365) under short-time creep-fatigue conditions: 0.65% strain range, 24-h hold time, 593°C; as polished.

In addition to fatigue, creep-fatigue, and two-bar ratchetting tests, a few specimens were tested under reversal creep conditions. These reversal creep tests were performed at 538 and 593°C (1000 and 1100°F) under a variety of hold times and ratios of minimum to maximum stress. When stresses were biased toward tension (R ratio greater than -1), the specimens elongated like a monotonic creep test and produced failures by wedge cracking or microcracking. See Fig. 6(b) for a typical failure at 593°C (1100°F). The longest time-reversal stress test was discontinued after 20,000 h at ± 138 MPa (± 20 ksi) and 538°C (1000°F). The sample, specimen CRP32, was cycled one week in tension and one week in compression (62 total cycles). Also, the sample was of a tubular configuration and contained four 0.9-mm holes in the test section. Wedge cracks filled with

Y-177486

(a)

Y-177483

(b)

20 μ m

Fig. 37. Microstructures developed in specimens subjected to a two-bar thermal ratchetting experiment. Maximum temperature, 593°C (1100°F); minimum temperature, 240°C (464°F); mean stress, 103 Mpa (15 ksi). Etched with Murakami's reagent. (a) Bar 1 experienced low stresses during the hold at 593° (1100°F) and exhibited only a few surface cracks. (b) Bar 2 experienced high tensile stresses during the hold and exhibited extensive grain-boundary microcracking.

oxide were found at the edges of the holes (Fig. 38). Some recrystallized finer grain regions were also around the holes. Away from the holes we observed a few grain-boundary facets that contained microvoids. Possibly, some microvoids formed on boundaries at higher than expected angles to the applied stress. Murakami's reagent revealed fine carbides, often in "nodules" and usually near microvoids. Some boundary migration also occurred, but no evidence of sigma phase was found.

A number of variable-stress creep tests under both uniaxial and multiaxial stresses have been performed to study the validity of linear damage summation in creep-rupture conditions. These samples have not yet been examined metallurgically, but this is a very important area of work that needs to be pursued.

MULTIAXIALLY STRESSED SPECIMENS

A variety of multiaxially stressed specimens have been tested throughout the course of the high-temperature structural design methods program. Simple specimen shapes such as thin-wall pressurized tubes and tension-torsion specimens experienced generally uniform stress states throughout most of the stress histories. One would expect to see failure modes that would best correspond to expectations based on uniaxial constant load tests. Studies of pressurized tubes in creep began with the work of Schultz and Leyda.³⁸

Parallel to this effort were tests by Gowda, Daniels, and Thomas³⁹ and by McAfee, Richardson, and Sartory.⁴⁰ Later studies were undertaken by Huddleston.⁴¹ Discontinuity stress effects were examined by Mallett, Dhalla, and Yocolano in a cylinder with a welded end cap⁴² and were later examined by Clinard and coworkers in a closed-end cylinder machined from a bar.⁴³ In several of these experiments variable stresses or temperatures were introduced. In only a few instances, however, were detailed metallographic studies undertaken. We present some representative samples.

Relatively uniform stress states were also produced by tension-torsion experiments on tubular geometries. Early work on creep testing was undertaken by Findley and coworkers.⁴⁴⁻⁴⁵ This work was followed by

Fig. 38. Microstructures developed in a tubular specimen subjected to ± 138 MPa (± 20 ksi) at 538°C (1000°F) for 20,000 h. (a) Wedge cracking near a drilled hole in the tube wall, etched with Murakami's reagent. (b) Grain size variation near a drilled hole etched with HCl and HNO_3 . (c) Microvoids, as polished. (d) Carbides and microvoids, etched with Murakami's reagent.

experiments by Ellis and coworkers.⁴⁶⁻⁴⁷ Cyclic testing by tension-torsion was undertaken by Blass and Zamrik,⁴⁸ Liu,⁴⁹ and Miller and coworkers.⁵⁰ Again, only short times were studied, and metallurgical examinations were somewhat limited. We present some examples of microstructures.

A number of more complicated tests have been performed that involved beams and plates in bending,⁵¹⁻⁵² nozzle-to-shell configurations,⁵³ and a series of thermal pipe ratchetting experiments.⁵⁴ Only a few of these experiments were carried to failure, and we briefly discuss the metallography of some representative samples.

Notch testing was undertaken by Manjoine and Konish, who included metallography studies in their work,⁵⁵⁻⁵⁶ and also by Raske and Maiya, who did not.⁵⁷ Some notched bar testing was also performed at ORNL on a tube with small holes under reversal creep (discussed earlier) and on circumferentially grooved bars. Metallographic studies on the grooved bars under steady and reversal stresses are briefly discussed.

PRESSURIZED TUBES

The creep rupture behavior of pressurized tubular specimens was analyzed recently by Huddleston,⁴¹ who examined data from several of the sources mentioned previously. One factor considered in the analysis of the data was the cracking patterns in the tube walls. Tests by Schultz and Leyda were performed on tubes with a 38-mm-diam (1.5-in.) outer surface and a 1.9-mm (0.075-in.) wall.³⁸ Samples stressed with circumferential-to-axial stress ratios of unity or greater exhibited cracking perpendicular to the circumferential stress,² and samples with ratios less than unity contained cracks orientated perpendicular to the axial stress. Samples failed by wedge cracking, with the larger cracks forming on the outside surfaces. Tube thicknesses were enough to contain about 20 grains across the tube wall. Samples tested by Huddleston were about 1 mm (0.04 in.) in wall thickness and contained as few as two or three grains across the wall. These also failed by wedge cracking, but we could not determine whether the inside or outside was favored as initiation sites. A typical cracked area is shown in Fig. 39 for a 627-h test at 593°C (1100°F) and 159 MPa (23 ksi). A midwall wedge crack can be seen as well as the coarse grain size relative to the wall thickness.

Fig. 39. Cracking patterns in the 1.0-mm (0.040-in) wall of pressurized tube specimens that failed in 627 h at 593°C (1100°F); etched with HCl and HNO₃.

Thicker wall samples were tested by McAfee and coworkers under tube-burst conditions.⁴⁰ The pipe diameter was about 100 mm (4 in.), and the thickness, about 4.5 mm (0.18 in.). The ruptured samples showed crack initiation from the inner surface.

Clinard and coworkers described the mechanical response of a capped cylinder subjected to varying stresses and to temperatures of 538 and 593°C (1000 and 1100°F) (ref. 43). The sample failed after 3300 h, when a crack that had developed in the fillet region of the head-to-cylinder joint propagated from the inside to the outside of the section [Fig. 40(a)]. Failure was predominantly by wedge cracking in the plane perpendicular to the axial direction in the cylinder. We observed significant

grain-boundary oxidation on the inner surface of the cylinder and evidence of surface cold work due to machining [Fig. 40(b)]. This cold work was typical of several large specimens.

TENSION-TORSION TESTING

Ellis has performed a series of tension-torsion creep tests on the reference heat at 593°C (1100°F). Several wall thicknesses were examined to develop a specimen form that would be geometrically stable for large rotations. The specimen design selected for rupture testing was 25 mm (1 in.) in diameter with a 5-mm (0.2-in.) wall. Tests were performed from 200 to 2000 h. The microstructures produced in the torsion test series differed greatly from those observed in uniaxial tests and in other multiaxial tests. This difference was largely caused by the high strains. Some typical photomicrographs are shown in Figs. 41 and 42, which compare the microstructures for three different loading conditions: tension-torsion, pure torsion, and compression-torsion. The cracking patterns through the thickness of the tubes shown in Fig. 41 reveal some internal cracking but largely show surface crack penetration along grain boundaries and heavy oxide formation. At least two oxides plus a metallic phase are present. In Fig. 42, we show sections corresponding to the outside surface of the tubes. These have been etched to reveal the grain deformation, microcracks, and carbide structure. The microstructures differ in several ways from the uniaxial microstructures. First, of course, is the severe grain distortion and rotation. The grains become roughly five times longer in one direction than in another, and the grain boundaries have rotated to shallow angles relative to the circumferential direction of the tubes. The microcavities have formed on boundaries that were normal to the tensile stress component; with continued shear deformation, however, the cracks have distorted, stretched, and rotated. Eventually, material between these cracks has sheared to produce failures. The bands of carbides are indicative of considerable grain-boundary migration. Grain distortion was least severe in the specimen under tension-torsion and most severe in the specimen under compression-torsion.

Y-165777

(a)

2 mm

Y-165779

(b)

Y-165772

(c)

200 μ m

Fig. 40. Wedge-cracking patterns produced on the inner surface of a capped-end cylinder tested in creep rupture for 3300 h at 593°C (1100°F). (a) As polished. (b) Inside surface, as polished. (c) Inside surface etched with HCl and HNO₃.

Y-191149

Y-191140

Y-191162

100 μ m

Fig. 41. Wedge-cracking patterns produced in the plane perpendicular to the circumferential direction of tubular specimens creep tested at 593°C (1100°F) in (a) tension-torsion for 1807 h, (b) pure torsion for 1698 h, and (c) compression-torsion for 932 h. As polished.

Y-192700

Y-192699

Y-192701

200 μm

Fig. 42. Cracking patterns produced in the plane perpendicular to the radial direction of tubular specimens tested at 593°C (1100°F) in (a) tension-torsion for 1807 h, (b) pure torsion for 1698 h, and (c) compression-torsion for 932 h. Etched with HNO_3 .

A few biaxial fatigue specimens from tests performed by Blass and Zamrik⁴⁸ were also examined metallurgically. These specimens were tubes tested in cyclic torsion and tension-compression-torsion at 538°C (1000°F) and 649°C (1200°F). Microstructures produced in specimens tested with an estimated octahedral shear strain of 3% are compared in Figs. 43 and 44 to show the effect of 1-h tensile hold time. Both samples experienced about the same torsion-to-axial strain ratios, but the specimen without a hold time lasted 428 cycles, and the specimen with 1-h hold time lasted 47 cycles. Cracking was transgranular without hold time, with no evidence of wedge cracking [Fig. 43(a)]. Cracking was intergranular when the 1-h hold time was introduced, and microvoids were observed in several areas. Regions of the tubes revealed differing densities and sizes of precipitates. This could indicate either temperature differences or strain gradients.

METALLURGICAL STUDIES OF A NOZZLE-TO-SPHERE TEST

A nozzle-to-sphere experiment at 593°C (1100°F) has been described by Clinard and coworkers.⁵³ The experiment involved a period of deformation testing under creep and reversed loadings, followed by a high-stress creep test to produce failure in a few thousand hours. The test was interrupted after 1000 and 2000 h to perform field metallography at the locations identified in Fig. 45. Failure (complete separation) occurred in another area of the sphere after 2800 h.

A typical microstructure obtained after the 1000-h test period is shown in Fig. 46. A number of fine wedge-type cracks extend along grain boundaries in the meridional direction. Often the cracks extend one or more grain-boundary facets [Fig. 46(a)]. Many grains contain what appear to be slip lines decorated with $M_{23}C_6$ precipitates. This same microstructure extended to the weld region, as shown in Fig. 46(b). Metallography after 2000 h revealed no significant changes; the cracks were longer, but the crack frequency was about the same.

Locations for laboratory metallographic examinations after failure included (1) the nozzle-to-sphere joint area, (2) a region 75 mm (3 in.) below the nozzle, (3) a region 280 mm (11 in.) below the nozzle, and (4) a

M-156278

M-156282

200 μ m

Fig. 43. Cracking patterns in the plane perpendicular to the circumferential direction of tubular specimens fatigue tested at 538°C (1000°F) in equal tension-torsion at 3% octahedral shear strain. As polished. (a) No hold time and 428 cycles. (b) One-hour hold time and 47 cycles.

Y-156645

Y-156653

Fig. 44. High-magnification of torsion-fatigue specimens tested at 538°C (1000°F) at 3% octahedral shear strain, etched with HCl and HNO₃. (a) No hold time and 428 cycles. (b) One-hour hold time and 47 cycles.

Fig. 45. Locations on nozzle-to-sphere experiment NS-2 structure where field and laboratory metallography were performed.

region 406 mm (16 in.) below the nozzle. A region through the wall near the nozzle-to-sphere joint is shown in Fig. 47(a). Again, there is evidence of grain-boundary cracking and carbide-decorated slip bands near the outside surface. The cracks appear to be wedge-type and heavily oxidized. Many cracks penetrated to only a depth of one grain-boundary facet. On the inside surface of the same region [Fig. 47(b)], a heavy corrosion layer and cracking to several grain facets were observed.

Y-163792

Y-163873

Fig. 46. Typical microstructure in the nozzle-to-sphere area of NS-2. Etched with HCl and HNO_3 . (a) Wedge cracks running in the meridional direction near the nozzle joint. (b) Wedge cracks at weld fusion line.

Y-167512

(a)

Y-167514

(b)

100 μm

Fig. 47. Surface cracks in nozzle-to-sphere experiment NS-2 near the nozzle. (a) Outside surface had cracks that penetrated about one grain boundary facet; etched with HCl and HNO_3 . (b) Inside surface contained deeper cracks and more corrosion, as polished.

The microstructures at other regions were similar to those observed near the joint. The exception was perhaps the region about 280 mm (11 in.) below the nozzle; few, if any, wedge cracks were observed on the outside surface. Figure 48(a) and (b) shows typical microstructures on the inside surface. Only surface cracks were noted. No cracks were observed in the sphere midwall.

All cracks were surface nucleated. The outside cracks developed as a result of creep strain superimposed on cold work produced by final machining. The cracks on the inside probably developed as a result of corrosive attack of the molten carbonate salt mixture used as a pressurizing medium.

METALLURGICAL STUDY OF TT-6

The thermal ratchetting test TT-6 involved a pipe specimen fabricated from a 208-mm-diam (8-in.) hot-extruded pipe of type 304 stainless steel. The sample was axially stressed to 130 MPa (19 ksi) at 593°C (1100°F) and exposed to a down shock to 260°F (500°C) once each day. Failure occurred in about three weeks, but extensive cracking was observed in less than two weeks.

A section of TT-6 pipe was examined metallurgically to compare the microstructures developed during the ratchetting test with those observed in specimens obtained from laboratory creep and fatigue tests at comparable temperatures and testing times. Intergranular cracks typical of creep-rupture failures were observed on both the inside and outside surfaces of the pipe (Fig. 49). Cracking was perpendicular to the axial direction of the pipe and was more severe on the outside surface. Heavy oxidation was present in the surface-connected cracks.

In addition to the relatively long wedge-type cracks, microvoid formation was observed on many grain boundaries throughout the cross section of the pipe, usually on boundaries that were perpendicular to the axial direction of the pipe. Some of these microvoids are evident in Fig. 49(c) and (d). These microstructural features are typical of specimens that fail by creep rupture. An etchant revealed the presence of carbide-decorated slip bands penetrating one or two grain diameters on the outside

Y-167494

Y-167506

Fig. 48. Cracking pattern in nozzle to sphere experiment about 280 mm (11 in.) from nozzle. Etched with HCl and HNO₃. (a) Cracking on the inside sphere surface. (b) Crack penetration through the thickness from the inside surface.

Y-181296

Y-181294

(a)

(b)

Y-181407

Y-181406

(c)

(d)

40 μ m

Fig. 49. Cracks produced through the thickness of the TT-6 pipe that experienced 19 cycles between 593°C (1100°F) and 260°C (500°F). (a) Outside edge that experienced high tensile residual stress after cycling, as polished. (b) Inside surface that experienced low residual stress after cycling, as polished. (c) Same as (a) but etched with HCl and HNO₃. (d) Same as (b) but etched with HCl and HNO₃.

surface and less than one grain diameter on the inside surface. This kind of microstructure is indicative of cold working, which may have been introduced during the machining operations on the pipe. Our opinion is that the pipe failed as a result of the penetration of wedge cracks that originated at the outside surface by exhaustion of creep ductility. The existence of microvoids on the grain boundaries, the presence of cold work at the surface, and the high-stress conditions during the ratchetting transients contributed to the failure.

NOTCHED BAR TESTS

Creep-rupture tests of notched bars on the reference heat of type 304 stainless steel were performed at 482, 538, and 593°C (900, 1000, and 1100°F) for as long as 15,000 h. At ORNL a shallow circumferential groove of the type designed by Raske and Maiya was used.⁵⁷ The theoretical stress concentration factor was 1.45. In constant-stress creep rupture, all specimens were notch strengthened. Typically, wedge cracks developed in testing times of a few thousand hours, and the major crack followed the maximum effective stress. This failure pattern is shown in Fig. 50(a). The fracture surface and a mirrored crack surface are evidently following the conical shape characteristic of the maximum effective stress profile in a notched bar or grooved cylinder. Under reversing stress conditions, however, notch strengthening was not observed, and wedge-type cracks initiated at the notch root and moved across the minimum section as if controlled by the maximum principal stress. This behavior pattern is illustrated in Fig. 50(b).

DISCUSSION

We have tried to cover a broad spectrum of testing conditions to demonstrate the range of microstructures that can be produced in a relatively simple alloy such as type 304 stainless steel. Detailed discussions and analyses of the microstructural features of type 304 stainless steel are available in several of the referenced reports and papers. We summarize in a qualitative way much of this information.

Y-162476

Y-161457

(a)

(b)

400 μm

Fig. 50. Wedge-cracking pattern in a notched bar creep tested at 593°C (1100°F): (a) 172 MPa (25 ksi) for 1000 h; and (b) ± 172 MPa (± 25 ksi) for 1000 h. As polished.

In the laboratory-reannealed condition, type 304 stainless steel is a solid-solution alloy. The yield strength is very low relative to the ultimate strength, and dislocation structure consists of a low density of dislocations. These are partial dislocations and may be pinned to some degree by the Suzuki mechanism. Short-range stress fields result in a significant thermally activated glide component at low temperatures [below 250°C (482°F)]. Slip is planar at first and controlled by a Taylor mechanism for hardening, which produces grain-size effects on the yield strength and hardening behavior. With increasing monotonic and cyclic strain, however, cross slip becomes an important factor. Dynamic recovery processes become significant with cross slip, and the dislocation structure eventually takes on a cellular form. At high strains, the dynamic recovery processes become so significant that the material loses its work-hardening capacity. Strain localization occurs along with the nucleation of microvoids at initiation sites such as manganese sulfide particles, slag inclusions, and other stress concentration points. Failure occurs by the classic cup-and-cone mode.

At intermediate temperatures [250 to 450°C (482 to 842°F)], thermal energy is sufficient to overcome short-range obstacles to plastic flow. As a consequence, little or no temperature or strain rate dependence is seen in the small-strain deformation behavior. With increased monotonic and cyclic strain, however, planar slip gives rise to hardening, and subsequently cross slip processes become significant. With cross slip comes temperature and strain rate sensitivity as the thermal component assists the cross slip process. Dynamic recovery processes also become important; again, dislocation cells develop. Concurrent with the increase in dislocation density is an increasing influence of strain-aging processes, probably due to carbon or nitrogen drag forces on the extended dislocations. This may produce inverse strain rate effects and may also be responsible for the occurrence of the shearing-off process instead of the cup-and-cone failure process, although we have not developed a convincing argument to support this. The important aspect of this deformation behavior relative to structural response, however, is that dislocation structures may be produced at intermediate temperatures that are different from

lower temperature substructures. This greatly affects the hardening response under nonisothermal plasticity conditions. Furthermore, we speculate that the instability and failure conditions at low temperatures could be affected by deformation at intermediate temperatures. The isothermal failure mode data at low temperatures may not be a reliable data base if the structure is subjected to significant strain accumulation at intermediate temperatures.

Between 450°C (842°F) and 650°C (1204°F), it is clear from all the microstructural data presented in this report that the deformation response will be quite complicated. On initial strain, the material will again exhibit planar slip, with little or no temperature and strain rate response. As cross slip develops, jerky flow becomes evident; along with this process is an inverse strain rate dependence. At very slow strain rates, however, true creep-dependent processes become important. The ultimate strength decreases with decreasing strain rate, and grain-boundary deformation and fracture become important factors. Contributing to the problem, however, is a rather significant matrix-hardening phenomenon produced by dynamic precipitation. The initial plastic loading strain sweeps carbon atoms to precipitation sites, which are often dislocation bundles produced at the planar slip intersection sites or at grain-boundary-dislocation concentration sites produced by accommodation strains. The fine precipitates tend to stabilize the dislocation network, requiring the production of more dislocations to maintain the creep rate or cyclic strain rate. Thus, even after the initial loading, the total dislocation density continues to increase, the creep rate decreases, and the development of dislocation cells or subgrains is retarded or suppressed. At higher stresses, matrix deformation and recovery are sufficient to permit grain-boundary sliding, especially for the short-time conditions before grain-boundary carbides develop to a large size. This sliding builds up the stresses at grain-boundary triple points or twin-boundary-grain-boundary intersections and initiates microvoids. Once formed, the stress concentration accelerates crack growth, and wedge-crack microstructures develop. At low stresses, grain-boundary sliding rates are too low to develop these stresses before the matrix hardening and the

grain-boundary carbide precipitation block further long-range sliding. Wedge cracking is suppressed. The microvoids produced in long-time low-stress situations are probably produced very early in the creep-rupture process. Possibly, the impingement of slip bands produced during the initial plastic-loading process could produce the conditions needed for microvoid initiation. Such initiation sites could be left behind by grain-boundary migration, of course, or could remain inactive for a long time until conditions favor growth. We know from TEM that microvoids often exist on the initial grain boundaries and are also present in the starting matrix. Migrating boundaries could sweep in the matrix microvoids. Once the microvoid develops to a stable size, it could collect surface-active elements, such as sulfur, which may enhance growth rates by any of several mechanisms. Our studies of long-time test specimens suggest that the microvoids are often associated with sigma phase, carbides, and perhaps ferrite. All these phases require material transport, which in turn requires diffusion of vacancies by lattice, dislocation pipe, grain-boundary, or surface mechanisms. All phases also involve dimensional changes, which may be promoted by hydrostatic stresses. The fact that sigma formed only in the stressed condition attests to the importance of the hydrostatic stress component. For more information on microstructural changes during grain-boundary migration, see the work of Morris and Harries⁵⁸ and Rosa.⁵⁹

It is important to note that most of the multiaxial testing was performed at 593°C (1100°F) under conditions where wedge cracking was the dominant failure mode under uniaxial stressing. Most of the multiaxial tests also exhibited wedge cracking, usually with higher crack densities near the surface. This surface wedge cracking may be due either to the stress gradient that produced higher stresses at the surface of the structures or to the cold work that was often present at the surface. But some evidence suggests that the slip characteristics could be different in the near-surface grains because of the lack of restraint from neighboring grains. Oxidation effects would be most significant at the surface and would promote wedge-type cracks rather than microvoids as the elements important to the oxidation process diffuse along the grain boundaries. Finally, note that sigma phase was not observed in any of the multiaxial

tests. The reason for this is not known but could be the short testing times. Work is continuing in this area.⁵⁹ It is extremely important that longer time, biaxially stressed tests be performed.

The microstructures produced in the tension-torsion and compression-torsion tests differed so greatly from those produced in other tests that one must question the significance of these tests for determining failure criteria. We think that the difference was largely due to the severe distortion of microstructure, and it is clear that the axis of the principal stresses in the torsion test must rotate significantly relative to the microstructural features involved in the actual failure processes. Long-time torsion tests are needed for conditions under which microvoids are known to form on grain boundaries.

We have presented a great deal of information regarding microstructures that were developed from tests at 649°C (1200°F) and above. Evidence suggests that these microstructures differ in several ways from those developed in lower temperature tests. At temperatures of 649°C (1200°F) and above, carbon diffuses very rapidly, and strain aging, if it occurs, is due to substitutional element locking effects. This actually causes not only rapid hardening relative to lower temperature deformation but also the rapid attainment of recovery-controlled microstructures. Subgrains form readily, and carbide precipitates on grain boundaries and in the matrix are too large to provide significant strengthening. The failure modes still appear to be wedge cracking at short time and microvoid development at long time, but it appears that grain-boundary migration, recrystallization, and other diffusion-controlled processes can produce significant evolution in the microstructures. We believe that data produced at 649°C (1200°F) or above would not be proper for developing failure criteria for lower temperature applications.

CONCLUSIONS

1. Metallurgical studies of uniaxially stressed specimens revealed very large effects of temperature and time on microstructures. Low-temperature and short-time failures were generally by plastic tearing or

shear brought on by large plastic deformation. At intermediate stresses in the creep regime, wedge cracking was the dominant failure mode. At long times, failures were produced by the coalescence of microvoids uniformly nucleated on grain boundaries normal to the applied stress.

2. At temperatures above 649°C (1200°F), the carbides were fairly large and blocky. Grain-boundary migration and recrystallization were prevalent. At temperatures below 649°C (1200°F), grain-boundary and matrix carbides were very fine. At long times, sigma phase developed on grain boundaries of all stressed samples at all temperatures. Often sigma phase, voids, and carbides were present in the wake of migrating boundaries. No sigma was found in either cyclic or multiaxial tests.

3. Most of the multiaxial tests were under conditions where wedge cracks formed. Often these cracks were surface related. Surface cold work and oxide penetration seemed to be more prominent in the multiaxial test specimens.

4. Long-time multiaxial tests are needed to examine the relationship between the microvoid cracking mode and stress state.

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