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INFLUENCE OF TITANIUM, ZIRCONIUM, AND HAFNIUM ADDITIONS ON THE RESISTANCE OF MODIFIED HASTELLOY N TO IRRADIATION DAMAGE AT HIGH TEMPERATURE - PHASE I

H. E. McCoy, Jr.

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METALS AND CERAMICS DIVISION

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H. E. McCoy, Jr.

ABSTRACT

The influence of small additions of Ti, Zr, and Hf on the mechanical properties of a modified Hastelloy N with the nominal composition Ni–12% Mo–7% Cr–0.2% Mn–0.05% C is described in this report. It deals specifically with test results from numerous, small, laboratory melts and several 100-lb melts from commercial vendors. Additions of Ti, Zr, and Hf had beneficial effects on the properties of the alloy both unirradiated and after irradiation. Irradiation temperature had a marked effect upon the properties of all alloys investigated. Generally, good properties were observed when the irradiation temperature was 650°C or less and poor when the temperature was 700°C or higher. We attributed this large effect of irradiation temperature to coarsening of the carbide structure at the higher temperature.

INTRODUCTION

Previous studies[1–4] showed Hastelloy N (Ni–16% Mo–7% Cr–4% Fe–0.05% C) susceptible to a type of damage produced by irradiation at high temperatures that results in reduced stress-rupture properties and fracture ductility. One approach to solving this problem is that of making slight changes in the chemistry of the alloy. We initially modified the base composition to Ni–12% Mo–7% Cr–0.2% Mn–0.05% C to obtain an alloy that

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[1] H. E. McCoy and J. R. Weir, "Stress-Rupture Properties of Irradiated and Unirradiated Hastelloy N Tubes," Nucl. Appl. 4(2), 96–104 (1968).

[2] H. E. McCoy, "Variation of Mechanical Properties of Irradiated Hastelloy N with Strain Rate," J. Nucl. Mater. 31(1), 67–85 (1969).

[3] H. E. McCoy, An Evaluation of the Molten-Salt Reactor Experiment Hastelloy N Surveillance Specimens — First Group, ORNL-TM-1997 (1967).

[4] H. E. McCoy, An Evaluation of the Molten-Salt Reactor Experiment Hastelloy N Surveillance Specimens — Second Group, ORNL-TM-2359 (1969).

was free of the stringers of the M_6C type of carbide (where M is the metallic component) characteristically found in standard Hastelloy N. We then made a number of laboratory melts with various additions of Ti, Zr, and Hf, since they form very strong, stable diborides[5] and should tie up the boron as compounds rather than allow it to segregate to the grain boundaries. The more random distribution of the boron should reduce the radiation damage by reducing the concentration at grain boundaries of helium produced by the $^{10}B(n,\alpha)$ reaction.

The results of tensile and creep tests on these experimental alloys show that the resistance to irradiation damage is improved by the addition of Ti, Zr, or Hf. Results of tests on the first small (100-lb) heats of commercial alloys that contained these same alloy additions also indicate improved properties.

EXPERIMENTAL DETAILS

Test Materials

Our alloys were nonconsumably arc melted in an argon atmosphere from melting stock of commercial purity. A starting charge of about 2 lb was consolidated and melted several times. The charge was then placed in another arc-melting furnace with a hearth for drop casting. The alloy was again melted and drop cast into a 1-in.-diam × 6-in.-long ingot. The ingot was swaged to 1/4-in.-diam rod by the following schedule: from 1 in. to 3/4 in. at 1177°C, from 3/4 in. to 7/16 in. at 871°C, and from 7/16 in. to 1/4 in. at ambient temperature.

The commercial melts were melted by vacuum induction and cast into six 4-in.-diam ingots that weighed about 16 lb each. They were initially forged at 1177°C; the final working was done at room temperature. The final product was small 1/2-in.-thick plates with 40% cold work.

The chemical analysis of each alloy used in this study is given in Table 1.

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[5]B. Post, "Refractory Binary Borides," p. 340 in Boron, Metallo-Boron Compounds and Boranes, ed. by R. M. Adams, Interscience, New York, 1964.

Table 1. Chemical Compositions of Alloys

Alloy Number	Composition (wt %) Mo	Cr	Mn	Fe	Si	C	Ti	Zr	Other	(ppm) B	Weight of Melt (lb)
100	12.16	6.96	0.20	0.071	< 0.005	0.052	< 0.01	< 0.03		0.02	2
101	12.16	6.97	0.20	0.056	< 0.005	0.057	0.11	< 0.03		0.02	2
102	12.10	7.03	0.20	0.089	< 0.005	0.045	0.39	< 0.03		0.02	2
103	12.37	6.98	0.20	0.077	< 0.005	0.055	0.31	< 0.03		0.02	2
104	11.98	6.97	0.20	0.10	< 0.005	0.050	0.55	< 0.03		0.02	2
105	11.10	6.84	0.20	0.089	< 0.005	0.049	0.66	< 0.03		0.01	2
106	11.82	7.02	0.20	0.061	< 0.005	0.050	0.81	< 0.03		0.01	2
107	11.95	7.00	0.20	0.082	< 0.005	0.056	1.04	< 0.03		0.01	2
75	11.8	7.94	0.20	< 0.05	< 0.05	0.062	0.99				2
76	11.8	7.88	0.20	< 0.05	< 0.05	0.117	1.01				2
21545	12.0	7.18	0.29	0.034	0.015	0.050	0.49	0.01		0.7	100
108	11.9	7.2	0.21	0.065	< 0.005	0.056	< 0.001	0.10		0.2	2
109	12.5	7.2	0.21	0.081	< 0.005	0.060	< 0.01	0.28		0.01	2
110	12.6	6.7	0.19	0.12	< 0.005	0.065	< 0.01	0.53		0.01	2
111	12.7	6.6	0.21	0.069	< 0.005	0.065	< 0.01	0.75		0.02	2
112	11.3	7.5	0.23	0.069	< 0.005	0.065	< 0.01	1.18		0.01	2
146	12.4	6.7	0.23		0.005	0.060	0.01	0.90	Re=0.02	0.4	2
147	12.0	6.7	0.2	0.1	0.01	0.069	0.01	0.94	Re=0.3		2
21554	12.4	7.4	0.16	0.097	0.01	0.065	0.003	0.35		2	100
21555	12.4	7.2	0.16	0.065	0.008	0.052	0.003	0.05		2	100
152	12.2	6.2	0.21	0.03	0.03	0.054	< 0.01	0.02	Hf=0.16	0.2	2
153	12.4	7.0	0.16	0.02	0.019	0.062	< 0.01	< 0.01	Hf=0.06		2
154	12.4	6.9	0.14	0.01	0.01	0.058	< 0.01	< 0.01	Hf=0.10		2
155	12.5	6.9	0.12	0.03	< 0.01	0.052	< 0.01	0.01	Hf=0.13		2
156	12.9	7.0	0.12	0.03	< 0.01	0.056	< 0.01	0.03	Hf=0.43	0.2	2
67-503	12.5	7.8	0.08	0.15	< 0.01	0.06	0.06	< 0.01	Hf=0.08	1	100
67-504	12.7	7.5	0.12	0.12	0.02	0.07	< 0.01	0.01	Hf=0.50	0.3	100
21546	12.3	7.2	0.16	0.05	0.009	0.05	0.1	0.005		2	100

The rods were fabricated into the test specimens shown in Fig. 1. Our work with this specimen has shown that the rest results are reproducible and agree quite well with those obtained for more massive specimens. However, there is sufficient stress concentration to cause some of the more brittle specimens to break in the radius at the end of the gage length.

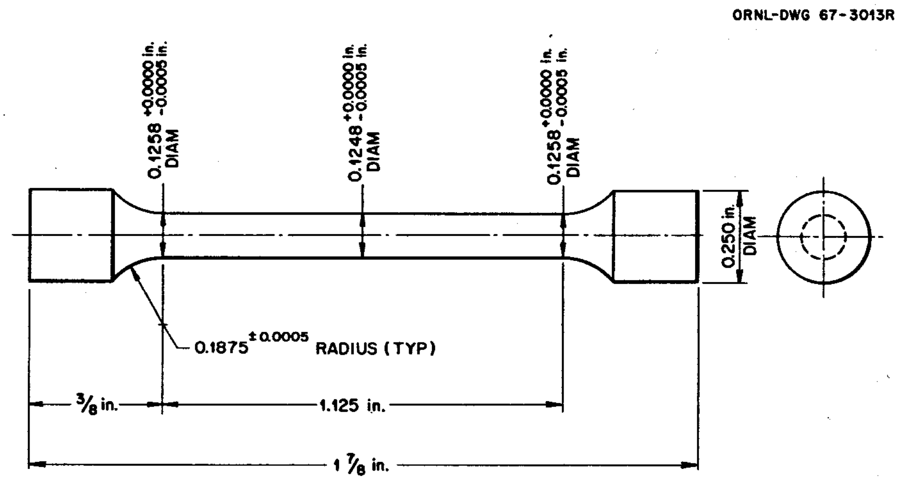

Fig. 1. Specimen Used for Tests of Mechanical Properties.

Irradiation Conditions

The results presented here were obtained from specimens irradiated in several experiments. These experiments were carried out in three reactors: the Engineering Test Reactor (ETR) at Idaho Falls, Idaho, and the Oak Ridge Research Reactor (ORR) and Molten Salt Reactor Experiment (MSRE) at Oak Ridge, Tennessee. A core facility was used in the ETR where the thermal and fast (> 1 Mev) fluxes were each 3.2×10^{14} neutrons cm^{-2} sec^{-1} and the fluence was 6×10^{20} neutrons/cm^2. The ETR experiments were uninstrumented, and the design temperatures were either below 150°C or 600 ± 100°C. Melt wires included in these experiments indicated that the operating temperatures were within the desired range. The experiments in the ORR were performed in a poolside facility where the peak fluxes were 6×10^{13} neutrons cm^{-2} sec^{-1} (thermal) and 5×10^{12} neutrons cm^{-2} sec^{-1} (> 2.9 Mev). The experiments were run for

either one or two cycles so that the thermal fluence was 2 to 5 $\times 10^{20}$ neutrons/cm^2 and the fast fluence was 2 to 4 $\times 10^{19}$ neutrons/cm^2. These experiments were instrumented, and the temperatures were controlled at 650 to 871°C. The experiments in the MSRE were run in the center of the core, where the thermal flux was 4.1×10^{12} neutrons cm^{-2} sec^{-1} and the fast (> 1.22 Mev) flux was 1.0×10^{12} neutrons cm^{-2} sec^{-1}. The samples were exposed to a noncorrosive fluoride salt environment for 4800 hr at 645 ± 10°C and received a thermal fluence of 1.3×10^{20} neutrons/cm^2 and a fast fluence of 3.1×10^{19} neutrons/cm^2. Since we could not see any consistent effects of thermal-neutron fluence over the small range encountered in these experiments, we present our data in this paper as having been obtained at a common fluence.

Testing Procedure

The creep-rupture tests after irradiation were run in lever-arm creep machines in the hot cells at the Oak Ridge National Laboratory. The strain was measured by an extensometer with rods attached to the upper and lower specimen grips. The relative movement of these two rods was measured by a linear differential transformer, and the transformer signal was recorded. The accuracy of the strain measurements is about ±0.1%, considering the effects of ambient temperature variations in the hot cell, mechanical vibrations, etc. This accuracy is considerably better than the specimen-to-specimen reproducibility that one would expect for this alloy. The systems for measuring and controlling temperature combine to give a temperature uncertainty of about ±1%.

The tensile tests were run on Instron Universal Testing Machines. The strain measurements were taken from the crosshead travel.

EXPERIMENTAL RESULTS

Alloys Containing Titanium

The alloys with additions of titanium fell into three groups. The first group, alloys 100 through 107, contained from less than 0.01 to

1.04% Ti. These alloys were of the nominal base composition of Ni–12% Mo–7% Cr–0.2% Mn–0.05% C. Typical microstructures of these alloys after a 1-hr anneal at 1177°C are shown in Fig. 2. The alloys were all free of intermetallic precipitates and contained only a small amount of carbide precipitates. Generally, the amount of carbide increased and the grain size decreased with increasing titanium concentration. The second group, alloys 75 and 76, contained 1% Ti and were made to explore the influence of varying carbon content. Typical microstructures of these alloys are shown in Fig. 3. The third material, a 100-lb commercial alloy from Special Metals Corporation, designated heat 21545, contained 0.49% Ti. Typical microstructures of this alloy are shown in Fig. 4. This alloy had a nominal composition quite close to that of alloy 104, and the microstructures of the two alloys after a 1-hr anneal at 1177°C were quite similar except that the commercial alloy contained patches of precipitate. Close examination showed these regions to be quite high in molybdenum. Some of the precipitates were carbides of the MC type and others were almost pure molybdenum. The grain size after annealing for 100 hr at 871°C was quite fine, and copious quantities of carbide precipitates were present.

Alloys 100 through 107 were tested under a variety of conditions. Tensile tests were run at 650°C on samples that had been annealed 1 hr at 1177°C and aged 1000 hr at 650°C to duplicate the thermal history of irradiated samples. The results of these tests are summarized in Table 2. The variation of the tensile and yield strengths with titanium content is shown in Fig. 5. The yield strength increased with increasing titanium. The tensile strength varied in a poorly defined manner: at a strain rate of 0.05 min^{-1}, it increased systematically with increasing titanium, and at a rate of 0.002 min^{-1} it reached a maximum at 0.55% Ti. The variation of the fracture strain with titanium content is shown in Fig. 6. At a strain rate of 0.05 min^{-1}, the addition of 0.11% Ti greatly increased the fracture strain; further additions decreased the fracture strain. At a strain rate of 0.002 min^{-1}, the fracture strain improved greatly with the addition of 0.11% Ti, and further additions caused a gradual improvement.

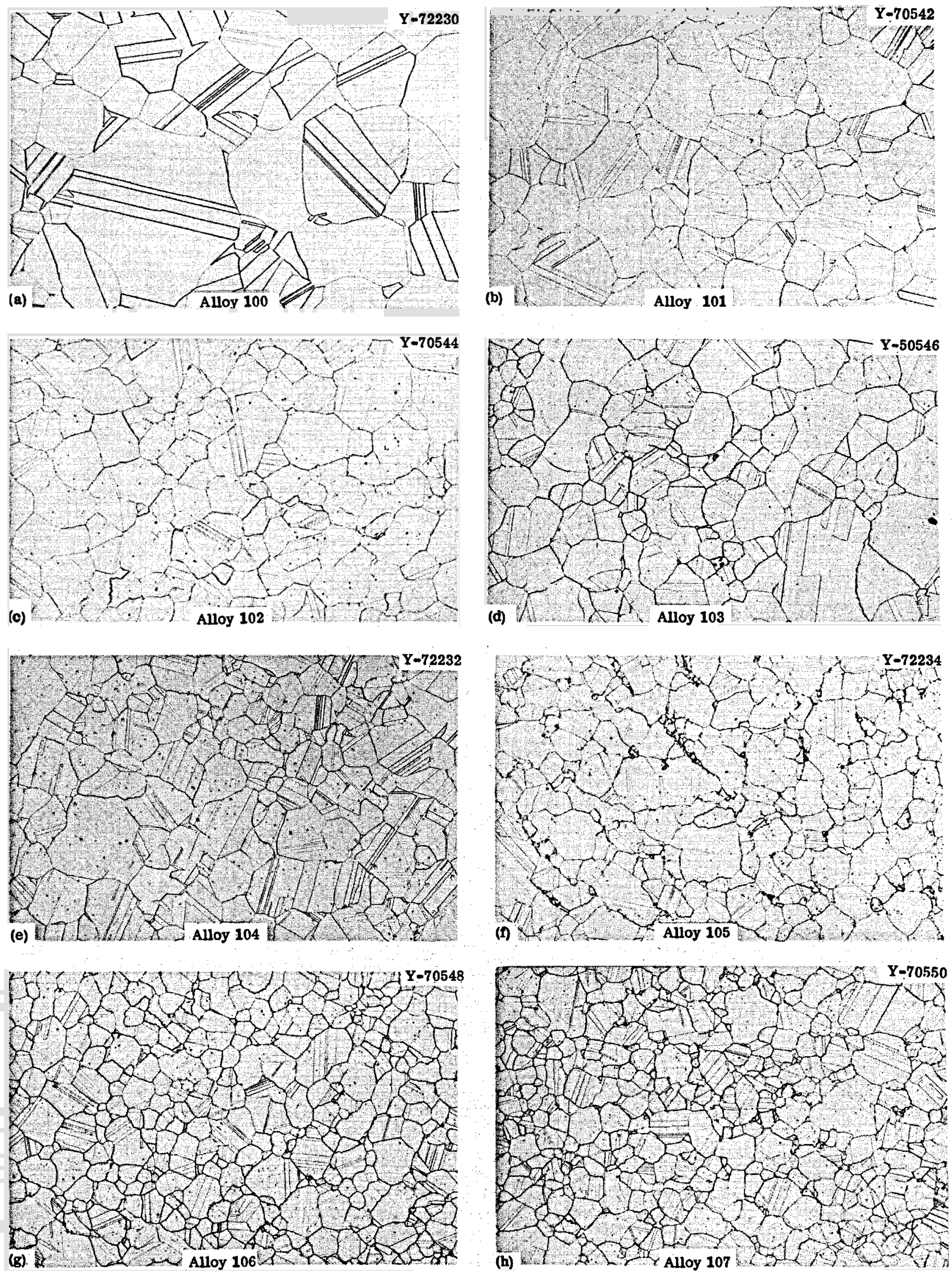

Fig. 2. Photomicrographs of Alloys That Contain Various Amounts of Titanium. The basic composition is Ni–12% Mo–7% Cr–0.2% Mn–0.05% C. Etchant: glyceria regia. 100×. Reduced 38%.

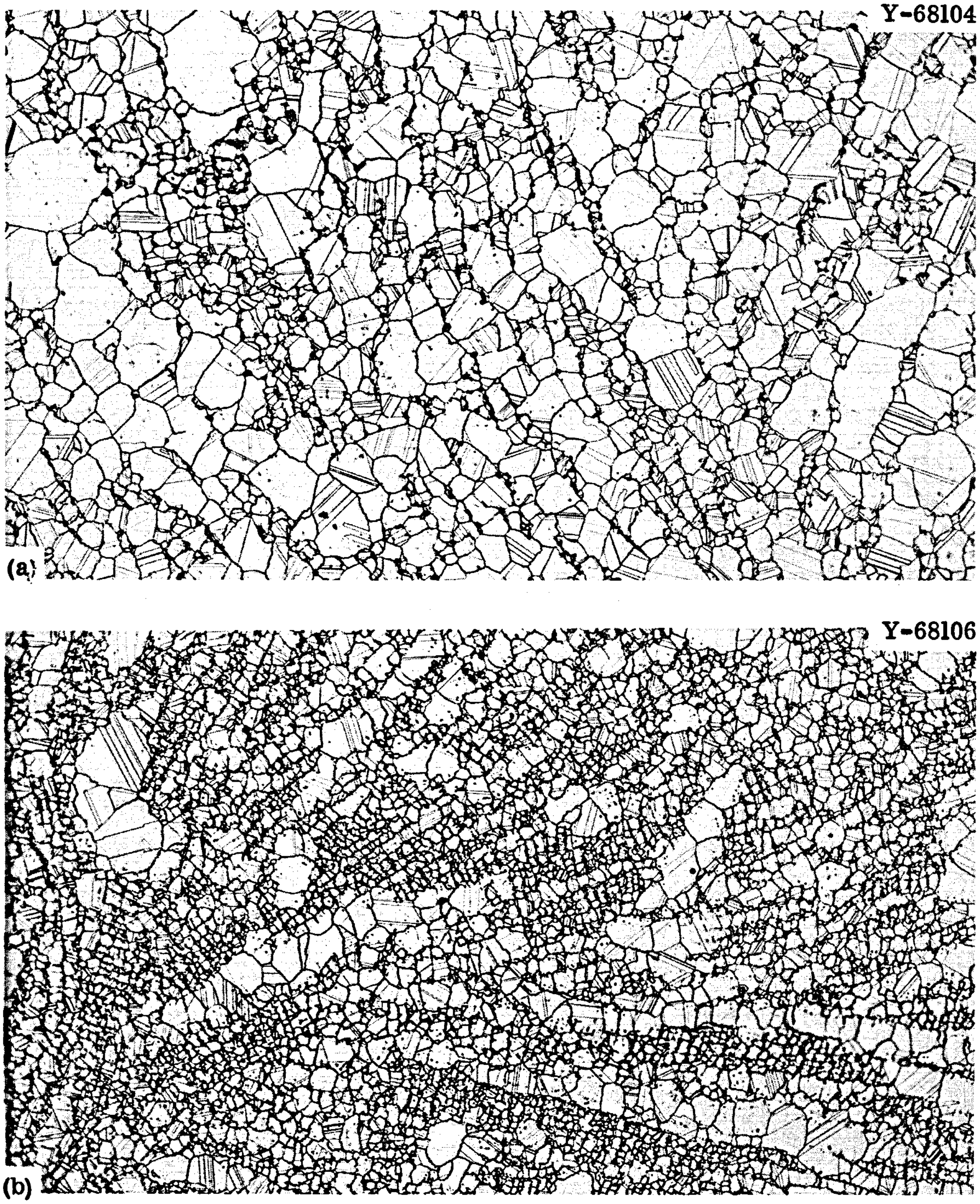

Fig. 3. Photomicrographs of Alloys (a) 75 and (b) 76. The nominal chemical compositions are Ni–12% Mo–7% Cr–0.2% Mn–1% Ti–0.05% C and Ni–12% Mo–7% Cr–0.2% Mn–1.0% Ti–0.10% C, respectively. Etchant: glyceria regia. 100×.

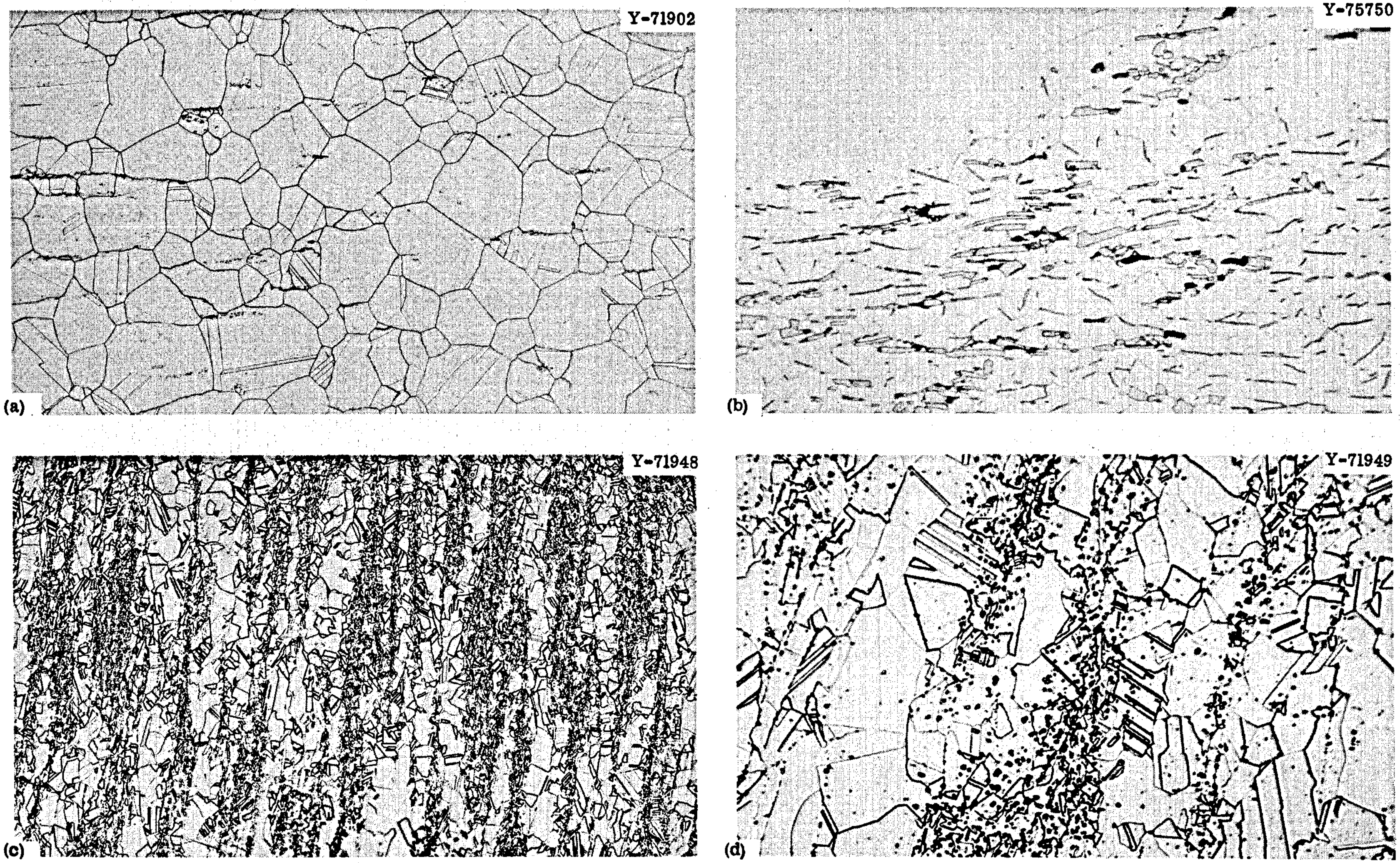

Fig. 4. Photomicrographs of Heat 21545, which Contained 0.49% Ti. (a) Annealed 1 hr at 1177°C, 100×; (b) precipitate after anneal of 1 hr at 1177°C, 1000×; (c) and (d) annealed 100 hr at 871°C, 100 and 500×. Samples (a), (c), and (d) etched with glyceria regia. Sample (b) etched with hydrochloric, nitric, and lactic acids. Reduced 28%.

Table 2. Tensile Properties of Several Unirradiated[a] Titanium-Modified Alloys at 650°C

Alloy Number	Specimen Number	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
			Yield	Ultimate Tensile	Uniform	Total	
			$\times 10^3$	$\times 10^3$			
100	1906	0.05	27.1	77.4	43.6	44.2	34.2
100	1910	0.002	30	73.7	27.8	29.7	25.6
101	1933	0.05	27.9	82.6	62.1	64.8	44.2
101	1926	0.002	27.4	75.2	33.0	37.3	29.2
102	1943	0.05	31.1	87.1	51.2	53.2	42.7
102	1952	0.002	30.9	78.4	31.4	37.5	32.8
103	1962	0.05	31.5	87.7	51.4	54.0	47.3
103	1968	0.002	28.9	69.4	24.5	37.5	33.8
104	1987	0.05	31.1	87.5	51.0	53.0	42.4
104	1984	0.002	35.4	87.9	28.2	36.7	28.8
105	2007	0.05	34.2	93.6	51.0	52.8	45.0
105	2004	0.002	34.3	84.1	31.9	40.3	32.4
106	2023	0.05	34.8	100.7	45.4	47.3	40.3
106	2026	0.002	33.9	79.8	26.0	37.8	35.1
107	2039	0.05	36.4	99.2	43.8	46.1	36.4
107	2041	0.002	33.7	75.1	22.3	38.5	32.5

[a]Annealed 1 hr at 1177°C and aged 1000 hr at 650°C.

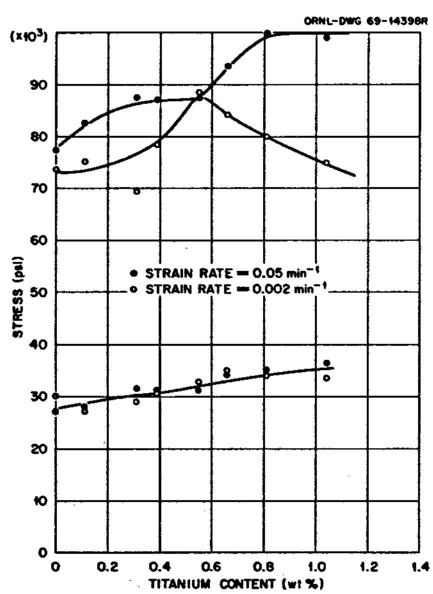

Fig. 5. Influence of Titanium Content on the Yield and Ultimate Tensile Strengths at 650°C after Annealing 1 hr at 1177°C and Aging 1000 hr at 650°C.

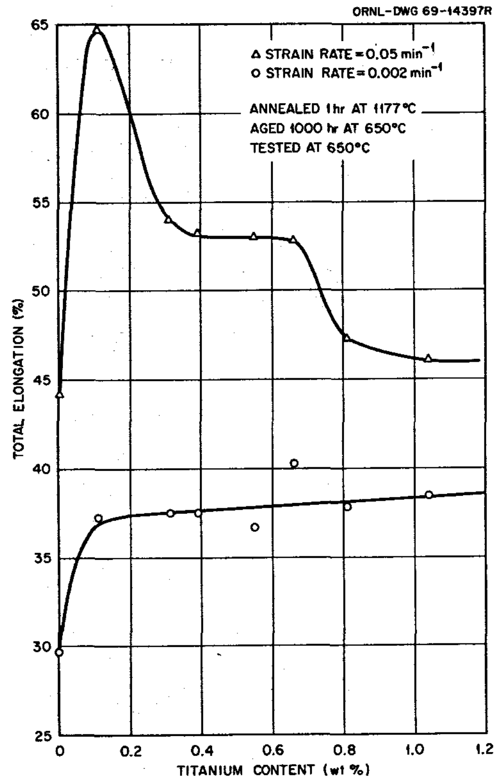

Fig. 6. Influence of Titanium Content on the Tensile Fracture Strain at 650°C of Ni–12% Mo–7% Cr–0.2% Mn–0.05% C Alloys.

Alloys 100 through 107 were irradiated at 650°C to a thermal fluence of 2.5×10^{20} neutrons/cm^2. The results of tensile tests at 650°C after irradiation are summarized in Table 3. The fracture strains measured at 650°C are plotted as a function of titanium content in Fig. 7. The data exhibit considerable variation, but show no systematic variation with titanium concentration. The results of these tests are analyzed further in Fig. 8, where the ratios of the various tensile properties of the irradiated and unirradiated alloys are compared. The yield stress is increased by irradiation, and the tensile stress and fracture strain are decreased. However, the ratios are only moderately dependent upon the titanium level.

Another set of samples of alloys 100 through 107 was irradiated at 50°C to a thermal fluence of 9×10^{19} neutrons/cm^2. The results of tensile tests of these samples at 650 and 760°C are given in Table 4. Comparison of these results with those in Table 3 for the samples irradiated to a higher fluence at a higher temperature reveals that the fracture strains are higher for the samples irradiated at 50°C. The fracture

Table 3. Tensile Properties of Several Titanium-Modified Alloys Tested at 650°C after Irradiation[a]

Alloy Number	Specimen Number	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
			Yield	Ultimate Tensile	Uniform	Total	
			$\times 10^3$	$\times 10^3$			
100	1908	0.05	31.5	66.3	26.3	26.6	23.3
100	1912	0.002	26.3	48.8	13.4	14.1	13.1
101	1924	0.05	35.5	68.9	23.2	23.7	19.1
101	1925	0.002	36.9	57.2	10.7	10.9	12.5
102	1949	0.05	35.1	74.8	33.7	35.1	28.8
102	1951	0.002	37.7	67.8	18.5	19.2	14.9
103	1978	0.002	35.3	65.9	21.9	22.8	18.8
104	2006	0.002	39.0	65.2	14.5	15.4	15.6
105	1989	0.002	35.5	69.7	21.9	22.7	31.6
106	2021	0.002	39.2	65.1	12.0	12.6	14.4
107	2051	0.002	39.3	71.6	15.9	16.3	13.0

[a]Annealed 1 hr at 1177°C before irradiation at 650°C to a thermal fluence of 2.5×10^{20} neutrons/cm^2.

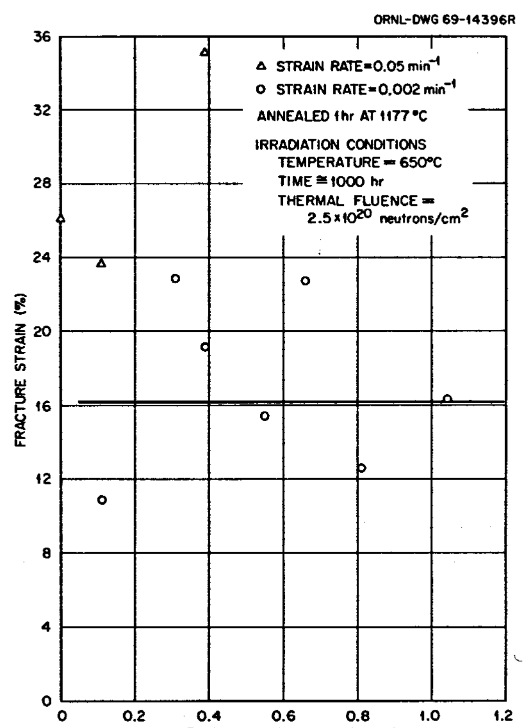

Fig. 7. Influence of Titanium Content on the Tensile Fracture Strain at 650°C of Ni–12% Mo–7% Cr–0.05% C Alloys after Irradiation.

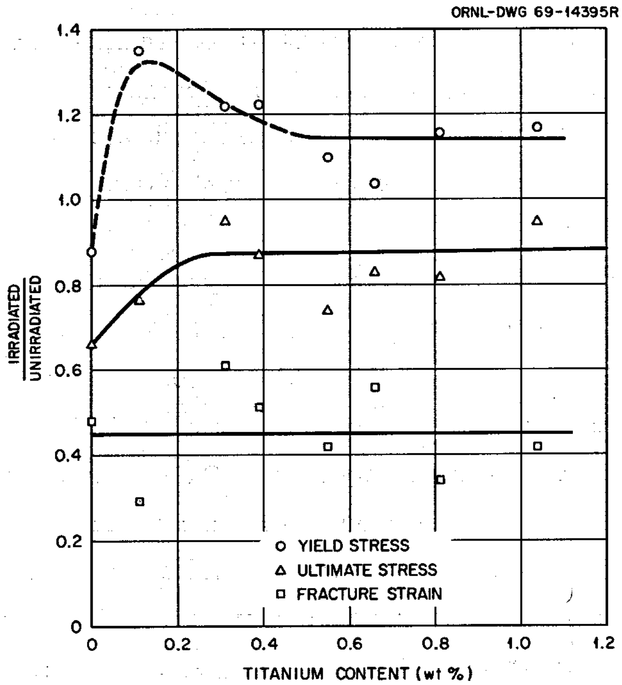

Fig. 8. Ratios of the Unirradiated and Irradiated Tensile Properties of Alloys Modified with Titanium. Tested at 650°C at a Strain Rate of 0.002 min^{-1}.

Table 4. Tensile Properties[a] of Several Titanium-Modified Alloys Tested at 650 and 760°C after Irradiation[b]

Alloy Number	Specimen Number	Stress, psi: Yield	Stress, psi: Ultimate Tensile	Elongation, %: Uniform	Elongation, %: Total	Reduction in Area (%)
		$\times 10^3$	$\times 10^3$			
		Test Temperature 650°C				
100	1907	26.4	60.3	21.3	21.9	24.0
101	1941	27.6	62.0	21.6	22.1	18.3
102	1944	31.3	71.4	22.8	23.4	19.4
103	1975	30.7	74.9	23.8	24.4	27.2
104	1992	29.8	71.8	25.1	25.7	33.2
105	2003	34.4	76.1	23.5	23.8	22.8
106	2025	33.4	80.8	26.0	26.4	34.5
107	2043	39.6	89.3	23.4	23.8	21.1
		Test Temperature 760°C				
100	1905	27.4	48.8	8.4	9.5	9.3
101	1940	27.6	51.0	8.1	9.2	5.6
102	1948	30.6	53.9	8.6	8.9	9.4
103	1977	26.8	53.8	9.3	10.3	10.4
104	1995	30.0	54.4	8.9	10.0	8.1
105	2016	31.8	56.7	8.8	11.2	8.6
106	2024	30.6	57.0	9.4	12.8	11.6
107	2056	31.5	58.9	9.3	13.2	12.1

[a]Strain rate = 0.002 min^{-1}.

[b]Annealed 1 hr at 1177°C before irradiation at 50°C to a thermal fluence of 9×10^{19} $neutrons/cm^2$.

strains measured at 650°C after irradiation ranged from 22 to 26% but did not vary systematically with titanium content. However, at a test temperature of 760°C, the fracture strain did show a slight dependence on titanium level; it varied from 9.5% with no titanium present to 13.2% with 1.04% Ti present.

Several unirradiated samples of alloys 100 through 107 were creep tested at 650°C. The results of these tests are summarized in Table 5 and plotted in Figs. 9 through 11. The results of stress-rupture tests in Fig. 9 show a marked improvement in rupture life at a given stress level with increasing titanium content. The results are somewhat scattered, and there are some questions about the exact location of the line for each heat. The measurements of minimum creep rate in Fig. 10 show that titanium decreased the creep rate. All of these tests were run without an extensometer, and the precision of the measurements was not very good. However, the data indicate that the creep rate did not vary appreciably with titanium content from 0.11 to 1.04% Ti (alloys 101 and 107). The data also indicate that titanium concentration may have no influence on the creep rate at low stress levels. The fracture strains are shown in Fig. 11 as a function of the minimum creep rate. Titanium additions definitely improve the fracture strain. The fracture strains of alloys 106 and 107, which contained 0.81 and 1.04% Ti, respectively, were almost independent of creep rate; the other alloys exhibited decreasing fracture strain with decreasing creep rate.

Several of these alloys were heat treated to simulate irradiated samples by anneals of 1 hr at 1177°C and 1000 hr at 650°C. The results of tests on these samples are summarized in Table 6. A comparison of these results with those in Figs. 9 through 11 for samples that were annealed 1 hr at 1177°C indicates how these alloys respond to aging. The rupture life, minimum creep rate, and fracture strain were all increased by this aging treatment.

Alloys 100 through 107 were irradiated at 650°C and creep tested at the same temperature after irradiation. The results of these tests are given in Table 7. All of these samples were tested at a stress level of 32,400 psi. The rupture lives and fracture strains are shown as functions of titanium content in Fig. 12. The rupture life was increased

Table 5. Creep-Rupture Properties of Unirradiated[a] Titanium-Modified Alloys at 650°C

Alloy Number	Specimen Number	Test Number	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
			$\times 10^3$				
100	1915	5725	55	3.4	0.43	22.5	20.8
100	1917	6099	47	20.8	0.55	14.5	12.5
100	1909	5554	40	47.4	0.088	12.5	9.5
100	1914	6108	32.4	185.1	0.0077	6.3	6.2
100	1921	6172	27	827.8	0.0022	4.9	4.0
101	1930	5753	70	0.6	2.9	34.5	28.3
101	1937	5692	55	23.4	0.064	21.0	21.3
101	1929	6203	47	52.1	0.050	16.0	14.0
101	1934	6203	40	292.1	0.0060	14.1	13.2
101	1932	6104	32.4	792.8	0.0037	10.5	12.8
102	1958	5751	70	3.3	0.56	28.5	22.8
102	1950	5726	55	32.4	0.047	20.0	15.4
102	1961	6100	47	162.4	0.016	15.8	16.9
102	1956	5669	40	506.5	0.0076	13.7	14.7
103	1979	5747	70	3.2	0.45	28.3	22.8
103	1976	5687	55	69.9	0.029	18.5	13.9
103	1966	6087	47	157.4	0.010	14.0	11.6
103	1980	5663	40	541.5	0.0082	15.6	18.4
104	1998	6101	70	3.5	0.88	32.3	20.8
104	1991	5566	55	41.6	0.060	18.8	14.0
104	1982	5959	36.5	2718.9	0.0033	13.9	21.4
104	1990	5638	40	1001.2	0.0066		19.9
105	2018	5754	70	7.7	0.25	23.4	18.5
105	2001	5698	55	94.0	0.019	15.9	13.7
105	2017	6089	47	179.7	0.015	15.5	19.6
105	2000	5586	40	476.6	0.0078	16.6	13.8
106	2031	5750	70	11.6	0.20	25.0	19.9
106	2029	5691	55	95.4	0.042	21.3	20.0
106	2022	5658	51	158.0	0.038	21.9	17.6
106	2032	5988	47	372.9	0.015	22.1	18.5
106	2034	6107	40	832.4	0.014	21.4	21.2
107	2054	5760	70	5.5	1.22	22.5	19.1
107	2046	5644	55	322.6	0.028	20.3	20.3
107	2055	6106	47	375.6	0.022	23.1	23.1
107	2040	5633	40	1490.5	0.010	26.5	27.3

[a]Annealed 1 hr at 1177°C before testing.

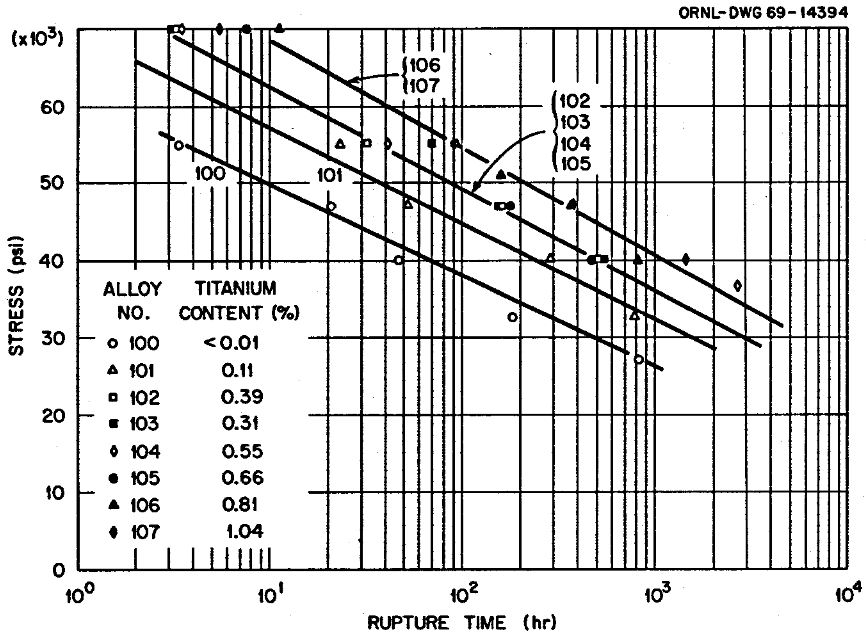

Fig. 9. Stress-Rupture Properties at 650°C of Several Alloys Containing Titanium. All samples annealed 1 hr at 1177°C before testing.

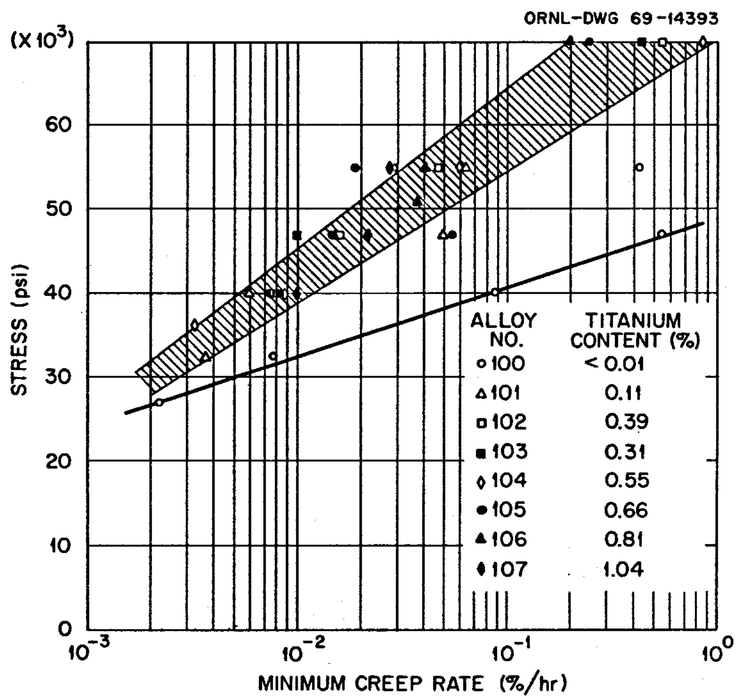

Fig. 10. Creep Properties at 650°C of Several Alloys Containing Titanium. All samples annealed 1 hr at 1177°C before testing.

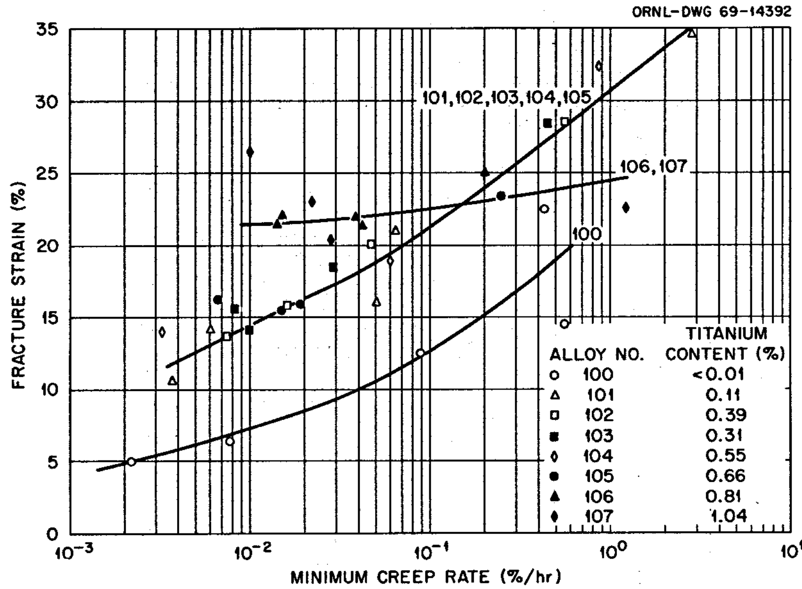

Fig. 11. Fracture Strains at 650°C of Several Alloys Containing Titanium. All samples annealed at 1177°C before testing.

Table 6. Creep Properties of Several Aged[a] Titanium-Modified Alloys at 650°C

Alloy Number	Specimen Number	Test Number	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
			$\times 10^3$				
100	1922	5723	55	5.9	0.61	23.0	19.2
100	1919	5630[b]	17.8	1007	0.0007	1.0	0
101	1936	5724	55	48.6	0.12	24.5	21.9
101	1935	5783[b]	32.4	1250.7	0.021	19.2	12.5
102	1960	5701	55	88.0	0.140	37.5	29.7
102	1959	5780[b]	32.4	1659.1	0.0051	10.3	7.2
103	1973	5729	55	54.0	0.21	36.4	25.6
103	1972	5757[b]	32.4	1006.9	0.0046	6.2	3.3
104	1999	5730	55	101.2	0.095	36.0	26.9
104	1994	5758[b]	32.4	1006.9	0.0054	5.8	3.3
105	2013	5728	55	66.6	0.119	38.0	32.4
105	2011	5785[b]	32.4	1704.5	0.0033	5.9	4.5
106	2035	5731	55	107.5	0.14	45.0	35.0
106	2027	5779[b]	32.4	1655.8	0.0039	7.5	4.8
107	2052	5654	42.7	902.2	0.027	42.2	35.0
107	2047	5784[b]	52.4	1704.5	0.004	6.0	4.8

[a]Samples annealed 1 hr at 1177°C and aged 1000 hr at 650°C.

[b]Test discontinued before failure.

Table 7. Creep Properties of Irradiated[a] Alloys That Contain Various Amounts of Titanium

Alloy Number	Specimen Number	Test Number	Titanium Content (wt %)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)	True Fracture Strain (%)
100	1916	R-146	< 0.01	105.4	0.0099	1.1	5.70	5.90
101	1928	R-147	0.11	169.9	0.0084	1.6	1.44	1.50
103	1971	R-150	0.31	570.1	0.0077	5.2	2.57	2.60
102	1954	R-148	0.39	1327.3	0.0034	5.8	4.63	4.70
104	2005	R-149	0.55	1446.3	0.0048	8.7	5.86	6.00
105	1983	R-151	0.66	1540.3	0.0046	9.2	b	b
107	2042	R-153	1.04	2928.8	0.0026	12.8	b	b

[a]Irradiated at 650°C to a thermal fluence of 2.5×10^{20} neutrons/cm^2. Tested at 650°C and 32,400 psi.

[b]Diameter not measured after test.

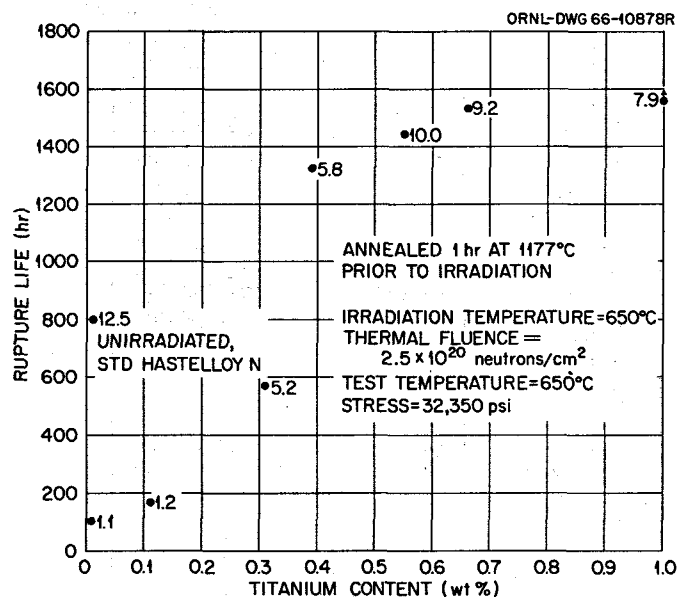

Fig. 12. Influence of Titanium on the Creep Properties of Ni–12% Mo–7% Cr–0.05% C after Irradiation. Tested at 650°C at a stress level of 32,400 psi.

by a factor of 25 and the fracture strain by a factor of 10 by the addition of 1.04% Ti. These results are compared with those of the unirradiated control samples in Table 8. This comparison is fraught with the uncertainties footnoted in Table 8, but it indicates that irradiated samples containing 0.55% Ti or more have rupture lives that are about 0.7 times the unirradiated value, minimum creep rates that are about equal to that of the unirradiated material, and fracture strains that are about 0.7 times the unirradiated value.

Alloys 100 through 107 were also irradiated at 50°C to a thermal fluence of 9×10^{19} neutrons/cm^2 and creep-rupture tested. The results of these tests are given in Table 9. These samples were tested at different stress levels than those used in the experiment described previously (Table 7). However, a rough comparison shows that the different irradiation temperatures (650 vs 50°C) and the different fluences (25 vs 9×10^{19} neutrons/cm^2) had little effect on the test results. Both experiments show clearly the increasing rupture life and fracture strain with increasing titanium level.

The combined results of the tensile and creep tests allow a determination of the variation of the fracture strain with strain rate at 650°C.

Table 8. Comparison of the Creep Properties at 32,400 psi and 650°C of Unirradiated and Irradiated Titanium-Modified Alloys

Titanium Content (%)	Unirradiated			Irradiated			Ratio (Irradiated:Unirradiated)		
	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Fracture Strain (%)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Fracture Strain (%)	Rupture Life	Minimum Creep Rate	Fracture Strain
< 0.01	185.1[a]	0.0077[a]	6.3[a]	105.4	0.0099	1.1	0.57	1.3	0.17
0.11	1250.7	0.021	19.2	169.9	0.0084	1.6	0.14	0.40	0.08
0.31	1900[b]	0.0046	12.5[a,b]	570.4	0.0077	5.2	0.30	1.7	0.42
0.39	1900[a,b]	0.0051	12.5[a,b]	1327.3	0.0034	5.8	0.70	0.62	0.47
0.55	1900[a,b]	0.0054	12.5[a,b]	1446.3	0.0048	8.7	0.76	0.89	0.70
0.66	1900[a,b]	0.0033	12.5[a,b]	1540.3	0.0046	9.2	0.81	1.4	0.74
1.04	3900[a,b]	0.0040	21[a,b]	2928.8	0.0026	12.8	0.75	0.65	0.61

[a]Values from unaged samples.

[b]Extrapolated.

Table 9. Creep Properties of Irradiated[a] Alloys That Contain Various Amounts of Titanium

Alloy Number	Specimen Number	Test Number	Titanium Content (wt %)	Test Temperature (°C)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)
					$\times 10^3$			
100	1911	R-257	< 0.01	650	40	68.9	0.019	1.79
101	1927	R-264	0.11	650	40	226.1	0.0037	1.55
103	1974	R-286	0.31	650	40	114.0	0.013	3.07
102	1957	R-282	0.39	650	40	255.4	0.010	5.04
104	1981	R-287	0.55	650	47	114.8	0.025	5.25
105	2008	R-290	0.66	650	47	200.9	0.0078	3.89
106	2030	R-288	0.81	650	47	318.9	0.0072	8.25
107	2044	R-289	1.04	650	47	374.0	0.0073	7.55

[a]Annealed 1 hr at 1177°C before irradiation at 50°C to a thermal fluence of 9×10^{19} neutrons/cm^2.

The data for samples irradiated at 650°C to a fluence of 2.5 × 10^{2C} neutrons/cm^2 were used to construct Fig. 13. The strain rate is a controlled parameter in a tensile test and was used directly in this figure. The stress is controlled in a creep test, and the resulting minimum creep rate was used in Fig. 13. The lines on this graph simply join the data points, and their slopes have little significance. The most important observation is that the addition of titanium causes a greater improvement in the fracture strain at low strain rates than at high strain rates.

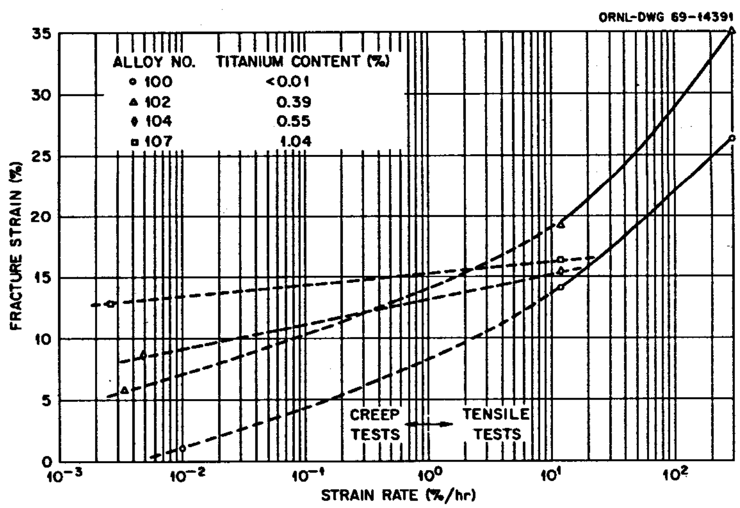

Fig. 13. Variation of Fracture Strain with Strain Rate at 650°C for Several Titanium-Bearing Alloys Irradiated at 650°C to a Thermal Fluence of 2.5 × 10^{20} neutrons/cm^2.

The results presented thus far for alloys 100 through 107 were obtained at a test temperature of 650°C for samples that were irradiated at 650°C or lower. The results in Table 10 illustrate the effects of varying the temperatures at which specimens were irradiated and tested. One group of samples was irradiated and tested at 760°C. These results show a general improvement in properties with increasing titanium level. The stress-rupture results for this series of tests are compared in Fig. 14 with those for tests on samples irradiated and tested at 650°C. Several samples were irradiated at 50°C and tested at 760°C. These were tested under different conditions, but the trend of increasing rupture

Table 10. Creep Properties of Alloys That Contain Various Amounts of Titanium After Irradiation[a] at Various Temperatures

Alloy Number	Specimen Number	Test Number	Titanium Content (wt %)	Temperature, °C Irra-diation	Temperature, °C Test	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)
						$\times 10^3$			
				2.5×10^{20} neutrons/cm^2 (thermal)					
100	3791	R-455	< 0.01	657	760	12.5	1.0	0.10	0.21
101	3801	R-454	0.11	760	760	12.5	0.8	0.10	0.20
102	3814	R-453	0.39	760	760	12.5	18.1	0.0252	0.71
103	3821	R-452	0.31	760	760	12.5	12.4	0.0333	0.66
104	3608	R-451	0.55	760	760	12.5	340.8	0.0086	5.72
104	3616	R-763		760	650	25.5	203.3	0.0042	1.36
105	3831	R-450	0.66	774	760	12.5	271.6	0.0066	3.05
106	3841	R-458	0.81	774	760	12.5	159.0	0.0196	5.40
107	3855	R-784	1.04	760	650	30	49.4	0.0238	1.58
107	3851	R-449		760	760	12.5	1000[b]	0.0039	5.19
						15	242[b]	0.0185	4.88
						17.5	38	0.0638	3.92
				9×10^{19} neutrons/cm^2 (thermal)					
100	1913	R-254	< 0.01	50	760	15	15.1	0.037	0.83
100	1918	R-308[c]	< 0.01	50	760	15	50.8	0.020	1.67
104	1986	R-255	0.55	50	760	15	358.8	0.016	11.9
104	1985	R-309[c]	0.55	50	760	15	316.3	0.011	11.3
107	2045	R-261	1.04	50	760	15	914.2	0.015	30.2
107	2035	R-310[c]	1.04	50	760	15	734.5[b]	0.016	25.1
						20	4.8	0.19	2.7

[a] Annealed 1 hr at 1177°C before irradiation.

[b] Annealed 1000 hr at 650°C after irradiation.

[c] Load increased at the intervals indicated.

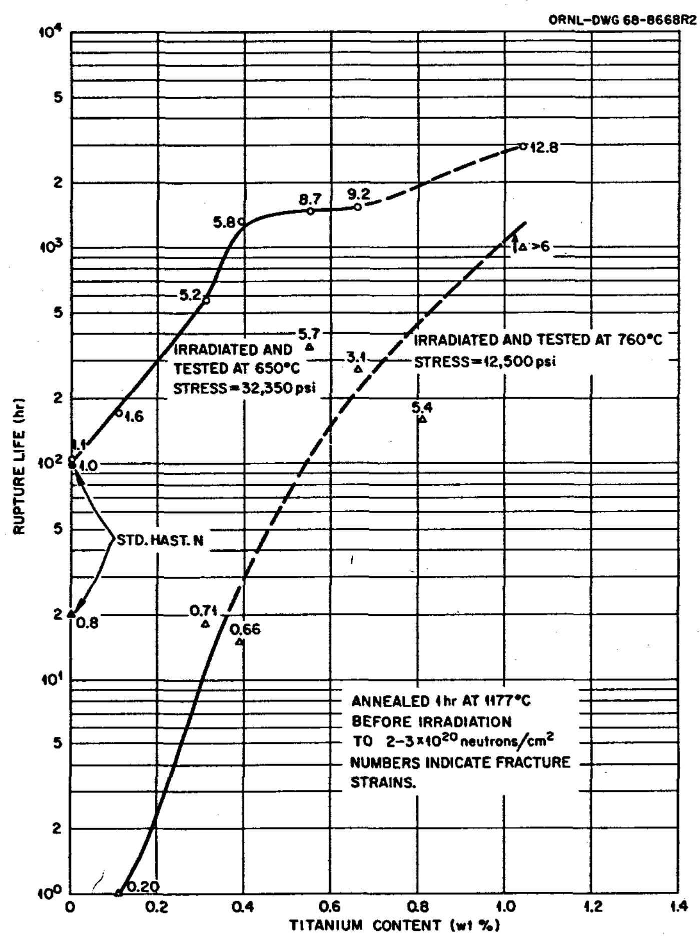

Fig. 14. Effect of Titanium Content on Variation of Creep Properties after Irradiation.

life and elongation is the same as for samples irradiated at 760°C. The only property that seems appreciably different is the fracture strain: higher values were obtained for samples irradiated at 50°C. The most dramatic effect of irradiation temperature is demonstrated by the combination of irradiation at 760°C and testing at 650°C (compare the results in Tables 7 and 10). The general effect is that the higher irradiation temperature results in a shorter rupture life, a higher minimum creep rate, and a lower fracture strain. For example, alloy 107 was irradiated at 650°C and tested at 32,400 psi and 650°C. The rupture life was 2928.8 hr, the minimum creep rate was 0.0026%/hr, and the fracture strain was 12.8%. The same alloy irradiated at 760°C and tested at 30,000 psi and 650°C had a rupture life of only 49.4 hr, a minimum creep rate of 0.024%/hr, and a fracture strain of 1.6%.

Alloys 75 and 76 were similar in composition to alloy 107, and only limited testing has been carried out. The results of tensile tests are summarized in Table 11. The results for the unirradiated samples agree well with those for alloy 107 (Table 2). Alloy 75 contains 0.062% C, and alloy 76 contains 0.117% C; therefore, the trends of higher strength and lower fracture strain for alloy 76 are reasonable. Irradiation increased strength and decreased fracture strain (Table 11). The fracture strains agree reasonably well with those for alloy 107 irradiated at 650°C (Table 3) and 50°C (Table 4). The samples annealed for 100 hr at 871°C had a very small grain size, and the fracture strains were lower for these samples. The higher carbon content of alloy 76 resulted in slightly lower fracture strains in tensile tests.

Table 11. Tensile Properties of Irradiated and Unirradiated Alloys 75 (0.99% Ti, 0.062% C) and 76 (1.01% Ti, 0.117% C) at 650°C

Alloy Number	Specimen Number	Anneal[a] Before Irradiation	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
				Yield	Ultimate Tensile	Uniform	Total	
				$\times 10^3$	$\times 10^3$			
			Unirradiated					
75	1197	121	0.05	31	89.3	45.0	45.8	36.3
75	1183	121	0.002	31.4	86.4	32.2	33.2	25.9
76	1300	121	0.05	39.3	100.3	34.6	34.8	26.9
76	1184	121	0.002	40.5	92.6	25.1	28.7	24.2
			Irradiated[b]					
75	1010	121	0.05	89.5	99.4	16.0	17.2	20.5
75	1011	121	0.002	90	90.4	5.1	6.1	11.7
75	1118	16	0.05	61.6	78	8.9	8.9	13.2
75	1119	16	0.002	60.8	66.5	6.4	6.4	10.2
76	1012	121	0.05	89.5	102.3	13.0	13.2	19.1
76	1013	121	0.002	91.8	95.3	5.0	5.3	11.7
76	1121	16	0.05	62.6	76.8	7.1	7.1	8.6
76	1122	16	0.002	61.4	65.7	5.2	5.2	8.6

[a]Annealing designations: 121 = annealed 1 hr at 1177°C.
16 = annealed 100 hr at 871°C.

[b]Irradiated at 150°C to a thermal fluence of 5.8×10^{20} neutrons/cm^2.

Several samples of alloys 75 and 76 were creep tested at 650°C; the results of these tests are summarized in Table 12. The properties of the unirradiated alloy are quite comparable with those for alloy 107 (1.04% Ti) that are given in Table 5. Same samples were irradiated at 760°C and tested at 650°C; the test results are given in Table 12. Both of these alloys had extremely good properties under these conditions. A comparison with the results for heat 107 (1.04% Ti) in Table 10 reveals the superiority of alloys 75 and 76. We have no explanation for this observation.

Table 12. Creep Properties of Irradiated and Unirradiated Alloys 75 (0.99% Ti, 0.062% C) and 76 (1.01% Ti, 0.117% C) at 650°C

Alloy Number	Specimen Number	Test Number	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
			$\times 10^3$				
			Unirradiated[a]				
75	8293	5276	55	128.5	0.0325	21.9	18.4
75	8341	5458	40	1919.9	0.0075	33.3	26.3
76	8292	5275	55	92.1	0.0880	15.6	22.7
			Irradiated[a,b]				
75	10266	R-888	47	248.1	0.0442	13.1	
75	10265	R-855	35	2669.2	0.0061	23.6	
76	10269	R-889	47	276.0	0.0540	19.6	
76	10268	R-852	35	2688.4	0.0059	22.0	

[a]Annealed 1 hr at 1177°C.

[b]Irradiated at 760°C to a thermal fluence of 3×10^{20} neutrons/cm^2.

The first commercial heat of a titanium-modified alloy was a 100-lb vacuum-melted alloy from Special Metals Corporation, designated 21545. This alloy was tested as received (40% cold work), after annealing 100 hr at 871°C (fine grain size, Fig. 4), and after annealing 1 hr at 1177°C (coarse grain size, Fig. 4). The results of tensile tests on these samples are summarized in Table 13. The fracture strains at a strain rate of 0.05 min^{-1} are shown in Fig. 15 as a function of test temperature. The fracture strain of the material as received was about 10% at 25°C and

Table 13. Tensile Properties of Unirradiated Alloy 21545 (0.49% Ti)

Specimen Number	Anneal[a] Before Test	Strain Rate (min^{-1})	Test Temperature (°C)	Stress, psi		Elongation, %		Reduction in Area (%)
				Yield	Ultimate Tensile	Uniform	Total	
				$\times 10^3$	$\times 10^3$			
3432	121	0.05	25	39.4	109.4	68.9	72.5	63.7
3451	121	0.05	200	31.8	99.2	70.9	74.9	58.1
3447	121	0.05	427	29.4	95.6	73.1	78.8	58.6
3435	121	0.05	500	28.1	91.1	78.9	81.9	57.5
2531	121	0.05	550	29.1	93.4	72.5	75.2	53.3
2521	121	0.002	550	30.5	94.1	65.5	67.9	43.9
3436	121	0.05	600	26.4	85.9	70.9	75.3	52.0
3444	121	0.002	600	27	74.8	41.8	43.0	37.5
3446	121	0.05	650	24.9	76	41.6	43.5	38.2
2530	121	0.002	650	22.1	67.4	21.2	22.0	30.7
3425	121	0.05	760	25.1	67.6	31.0	33.2	21.8
3429	121	0.002	760	24.7	50.8	12.0	19.2	20.4
2523	121	0.002	760	23.1	45.4	10.1	25.2	26.5
3448	121	0.05	800	23.7	59	20.5	27.7	22.6
3445	121	0.002	800	23.2	41.3	8.5	18.6	22.7
1565	000	0.05	25	159.7	170.4	8.5	11.9	17.5
2519	000	0.05	25	164.2	171.6	6.3	10.7	37.8
1566	000	0.05	427	131.5	147.1	11.4	15.2	28.8
1544	000	0.05	650	119.4	131.6	8.0	11.8	20.9
1545	000	0.002	650	113.4	116.8	3.4	8.9	13.2
1567	000	0.05	760	92.7	93.4	3.0	24.9	41.9
1549	16	0.05	25	54.2	122.9	48.7	52.0	56.6
1550	16	0.05	427	37	102.2	49.8	54.0	42.3
2544	16	0.05	500	43.3	103.6	42.1	48.1	42.2
2543	16	0.05	550	42.5	99.8	43.9	45.9	35.0
2520	16	0.05	600	40.4	89	30.0	31.5	27.3
1765	16	0.05	650	37.9	87.6	39.9	46.1	37.2
1766	16	0.002	650	42.2	73	19.4	48.3	53.2
2540	16	0.05	700	40.9	70.7	21.2	41.8	42.3
2542	16	0.002	700	41.4	59.6	12.3	46.0	28.3
2546	16	0.05	760	41.6	56	11.7	53.5	58.3
2454	16	0.05	760	38	50.4	13.0	56.2	74.4
2547	16	0.002	760	37	37.3	1.5	53.3	68.1
2522	16	0.05	800	36.9	44.9	11.0	57.8	70.2
2541	16	0.002	800	30	30	1.1	47.7	29.8
2455	16	0.05	871	31.8	32.1	1.3	57.4	78.1
2456	16	0.002	871	17.6	17.6	1.0	47.7	41.6
2457	16	0.05	982	17.9	17.9	0.8	59.8	64.6
2536	46	0.002	600	26.4	76.9	45.8	47.6	32.6
2535	46	0.002	650	26.5	74.6	35.5	38.5	31.0
2537	46	0.002	700	26.5	56.2	15.6	34.3	31.0

[a]Annealing designations: 121 = annealed 1 hr at 1177°C; 000 = as received (40% cold work); 16 = annealed 100 hr at 871°C; 46 = annealed 1 hr at 1177°C plus 1000 hr at 650°C.

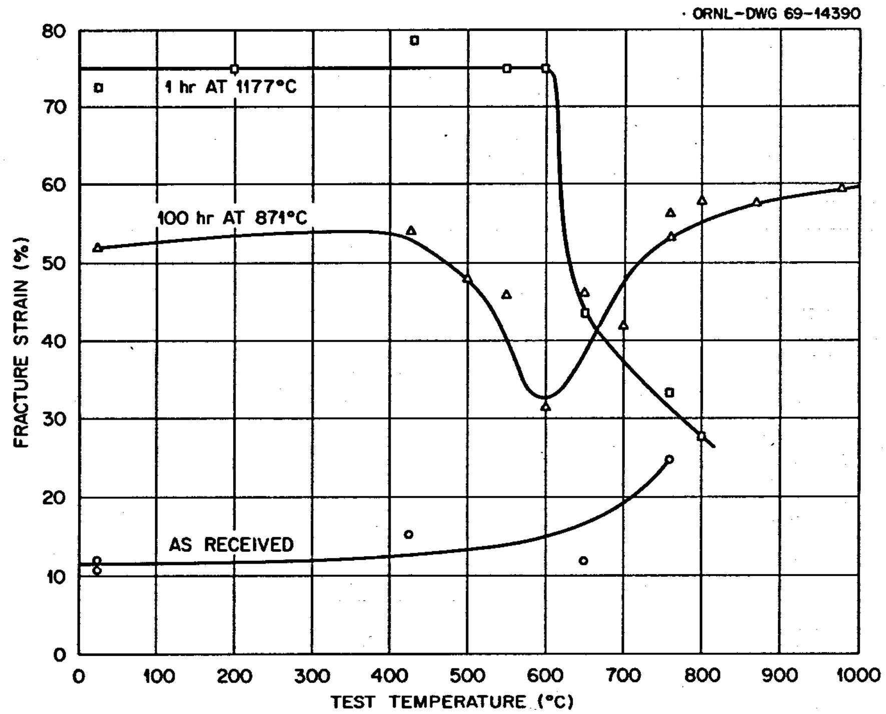

Fig. 15. Fracture Strains of Alloy 21545 (0.49% Ti) in Tensile Tests at a Strain Rate of 0.05 min^{-1}.

increased gradually with increasing temperature. The material annealed for 100 hr at 871°C had a fracture strain of 52% at 25°C with a minimum ductility of 32% at 600°C. After an anneal of 1 hr at 1177°C, the fracture strain was 75% at temperatures between 25 and 600°C and then dropped precipitously with increasing temperature. Three samples were annealed for 1 hr at 1177°C and aged for 1000 hr at 650°C. This anneal did not change the strength properties appreciably, but did increase the fracture strain. Alloy 104 (0.55% Ti) was given the same anneal before testing, and a comparison between the results for this alloy in Table 2 and those for heat 21545 (0.49% Ti) in Table 13 shows that the commercial alloy is slightly weaker and that both alloys have comparable fracture strains.

The results of tensile tests on irradiated samples of heat 21545 are given in Table 14. The results for samples irradiated at 650°C or below were used in constructing Fig. 16 for a strain rate of 0.002 min^{-1}. The samples that were irradiated as received and those irradiated after annealing 100 hr at 871°C had comparable fracture strains. The samples annealed for 1 hr at 1177°C were more ductile. A comparison with the

Table 14. Tensile Properties of Alloy 21545 (0.49% Ti) after Irradiation[a]

Specimen Number	Anneal[b] Before Test	Temperature, °C		Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
		Irradiation	Test		Yield	Ultimate Tensile	Uniform	Total	
					$\times 10^3$	$\times 10^3$			
1554	000	600	550	0.002	111.6	134.9	11.5	15.1	14.7
1553	000	600	600	0.002	110.1	127.9	11.2	13.4	11.7
1551	000	650	650	0.05	107.0	124.9	12.5	15.1	16.2
1552	000	650	650	0.002	107.7	113.3	5.1	9.5	11.7
2524	121	590	650	0.05	29.0	64.4	36.0	37.0	32.0
2525	121	590	650	0.002	28.9	58.8	21.6	21.9	17.1
2548	121	600	650	0.002	30.0	61.3	21.1	21.4	18.3
2550	121	600	760	0.002	29.2	51.7	10.3	13.6	19.7
5953	121	760	650	0.002	22.4	22.4	1.0	1.4	3.2
5954	121	760	760	0.002	15.8	15.8	0.7	0.7	0.81
1560	16	600	550	0.002	42.9	85.3	12.1	12.4	20.5
1557	16	600	600	0.002	39.3	69.1	13.3	13.4	8.6
1558	16	600	650	0.05	38.2	71.5	14.7	15.1	16.2
1559	16	600	650	0.002	38.8	62.4	9.7	9.7	13.2
1561	16	600	760	0.002	37.8	37.8	1.4	4.1	7.1

[a]Irradiated to a thermal fluence of 2 to 5 $\times 10^{20}$ neutrons/cm^2.

[b]Annealing designations: 121 = annealed 1 hr at 1177°C,
000 = as received (40% cold work),
16 = annealed 100 hr at 871°C.

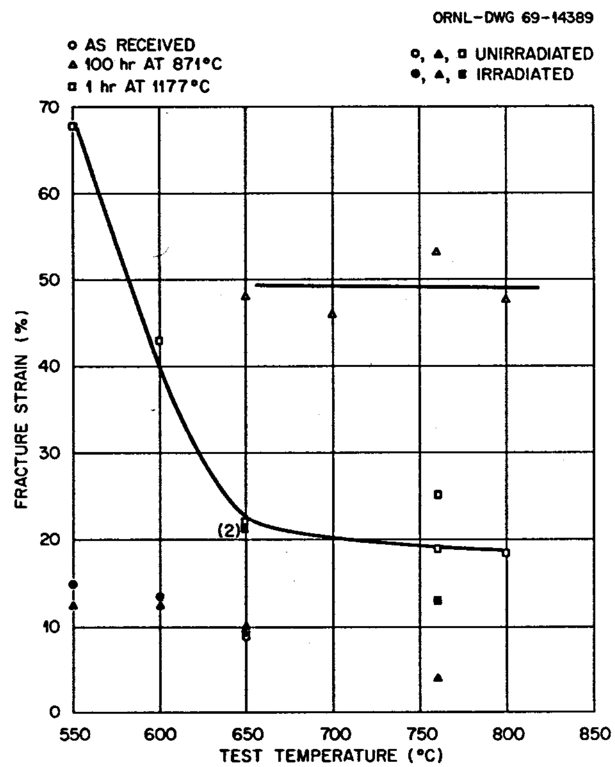

Fig. 16. Fracture Strains of Alloy 21545 (0.49% Ti) in Tensile Tests at a Strain Rate of 0.002 min^{-1}.

results for alloy 104 (0.55% Ti) in Tables 3 and 4 shows that the commercial alloy was more ductile and slightly weaker. Two samples in Table 14 were irradiated at 760°C. The samples had much lower fracture strains than those irradiated at 650°C or below, indicating again a very strong effect of irradiation temperature.

Samples of heat 21545 were subjected to creep tests in several annealed conditions; the results of these tests are presented in Table 15 for unirradiated samples and in Table 16 for irradiated samples. The stress-rupture properties at 650°C in the unirradiated condition are shown in Fig. 17. The as-received material had the longest rupture life at a given stress level and the lowest fracture strain. Annealing at 871°C produced a material with fine grain size that failed in much shorter times with fracture strains of about 50%. Annealing for 1 hr at 1177°C gave longer rupture lives with intermediate fracture strains of about 30%. Aging this material at 650 or 760°C before testing had only minor effects on the rupture life or the fracture strain. However, close

Table 15. Creep Properties of Unirradiated Alloy 21545 (0.49% Ti)

Specimen Number	Test Number	Anneal[a] Before Test	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
			$\times 10^3$				
			Test Temperature 650°C				
1564	5535	000	70	174.1	0.0093	14.1	20.5
1546	5578	000	55	1317.8	0.0009	18.8	31.2
3441	6294	121	70	0		41.1	32.6
3404	6295	121	62	17.7	0.288	27.2	26.7
5250	6417	121	60	26.8	0.062	27.0	23.5
3406	6296	121	55	39.9	0.148	22.6	17.0
5251	6418	121	52	170.9	0.015	24.8	27.2
3416	6297	121	47	197.6	0.037	26.3	27.7
5244	6419	121	44	466.1	0.011	26.3	22.6
3418	6298	121	40	564.2	0.015	31.3	29.7
5246	6420	121	37	1478.6	0.0035	34.6	31.7
3413	6299	121	35	1720.9	0.0063	30.1	30.0
5247	6421	121	30	4383.2	0.0013	22.2	26.7
1822	5564	816	40	248.1	0.0688	34.4	20.5
1771	5409	16	55	20.1	1.37	52.5	47.3
1773	5580	16	40	242.6	0.120	59.4	56.6
1768	5449	16	30	1171.5	0.017	56.7	51.5
2539	6167	46	40	893.7	0.0129	38.7	32.1
2538	6161	46	32.4	2077.6	0.0041	30.8	24.7
2528	7716	148	40	708.9	0.0318	33.3	
5534	7715	148	55	26.7	0.625	34.4	34.2
3400	7718	156	40	527.4	0.0334	36.1	41.4
2533	7717	156	55	33.7	0.570	32.1	31.8
5508	6384[b]	103	70	1.9		39.2	30.6
5509	6385[b]	103	63	5.5	4.53	46.1	33.1
5510	6386[b]	103	55	15.7	2.50	47.4	34.3
5511	6387[b]	103	47	50.3	0.50	43.9	65.9
5512	6388[b]	103	40	177.2	0.165	55.3	45.1
5513	6428[b]	103	32.4	438.8	0.050	40.1	50.3
			Test Temperature 760°C				
3424	7041	121	30	28.7	0.519	37.8	32.6
3431	7042	121	25	71.1	0.213	39.4	35.0
3422	6358	121	20	280.0	0.060	36.2	35.1

[a]Annealing designations: 121 = annealed 1 hr at 1177°C; 000 = as received (40% cold work); 816 = annealed 8 hr at 871°C; 16 = annealed 100 hr at 871°C; 46 = annealed 1 hr at 1177°C plus 1000 hr at 650°C; 148 = annealed 1 hr at 1177°C plus 1000 hr at 760°C; 156 = annealed 1 hr at 1177°C, 510 hr at 550°C, 1500 hr at 760°C, and 1 hr at 1177°C; 103 = annealed 100 hr at 871°C, 5600 hr in fluoride salt at 650°C.

[b]Molten Salt Reactor Experiment surveillance controls.

Table 16. Creep Properties of Alloy 21545 (0.49% Ti) after Irradiation

Specimen Number	Test Number	Anneal[a] Before Irradiation	Irradiation Temperature (°C)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
				$\times 10^3$				
			Test Temperature 650°C					
5235	R-676	121	650	60	12.7	0.445	7.0	28.6
2558	R-415	121	600	63	8.6	0.237	3.1	19.6
2554	R-328	121	600	55	24.2	0.114	4.0	23.2
2549	R-325	121	600	47	264.8	0.022	10.4	18.3
5237	R-653	121	650	50	65.0	0.023	16.8	23.8
5242	R-657	121	650	50	178.0	0.011	8.0	19.5
2527	R-258	121	590	47	87.4	0.071	10.0	
3402	R-909	121	654	47	33.5	0.258	13.2	31.1
5236	R-644	121	650	44	285.0	0.0040	18.9	26.3
2553	R-249	121	600	40	780.6	0.0091	16.5	
2526	R-232	121	590	40	453.8	0.010	11.3	
2529	R-348	121	590	40	261.2	0.017	8.2	17.3
5238	R-654	121	650	40	574.6	0.016	13.4	22.8
3426	R-771	121	760	27	11.6	0.019	0.33	5.4
3414	R-913	121	760	27	6.1	0.056	0.42	11.6
5240	R-841[b]	121	650	27	355.5	0.0081	4.3	9.1
5239	R-817[b]	121	650	27	109.4	0.0070	1.3	5.2
3409		121	704	27	1531.8		8.77	
3397		121	760	21.5	223.4			
1767	R-352	16	650	40	4.9	0.373	2.0	4.8
1563	R-125	16	600	40	72.6	0.077	6.0	
1769	R-353	16	650	35	43.4	0.039	3.4	6.4
1562	R-127	16	600	32.4	356.9	0.019	7.3	
1770	R-354	16	650	30	111.7	0.019	3.3	4.0
1774	R-416	16	650	27	430.8	0.0053	3.9	4.8
1772	R-421	16	650	25	1056.5	0.0056	6.5	10.7
2448	R-346	16	150	55	3.5	1.59	6.1	10.6
2449	R-378	16	150	47	16.1	0.40	7.8	11.1
2450	R-347	16	150	40	66.4	0.13	9.9	12.9
2451	R-362	16	150	35	193.9	0.037	16.2	9.9
2452	R-419	16	150	30	596.0	0.0075	9.9	11.4
2453	R-420	16	150	27	721.8	0.0059	6.9	10.9
9100	R-317[c]	16	650	47	2.8	0.46	1.6	
9099	R-314[c]	16	650	40	13.1	0.085	2.7	
9102	R-319[c]	16	650	32.4	51.1	0.013	3.4	
9103	R-323[c]	16	650	27	124.1	0.0089	3.7	
3430	R-829	113	760	27	7.8	0.024	0.32	13.5
3433	R-906	113	760	21.5	108.4	0.0052	1.3	6.3
			Test Temperature 760°C					
2556	R-394	121	650	25	351.0	0.0058	18.1	7.3
2555	R-329	121	600	20	127.4	0.0328	13.3	12.9
5234	R-652	121	650	20	200.2	0.037	13.8	13.3
d		121	760	25	0.50	0.50	0.66	
d		121	760	20	1.5	0.42	1.0	
d		121	760	11	94.0	0.034	4.7	
d		22	760	12	101.0	0.040	5.2	
d		22	760	11	296.0	0.020	7.0	

[a]Annealing designations: 121 = annealed 1 hr at 1177°C; 16 = annealed 100 hr at 871°C; 113 = annealed 1 hr at 1177°C plus 100 hr at 650°C; 22 = annealed 1 hr at 1177°C plus 100 hr at 871°C.

[b]Annealed 5000 hr at 760°C after irradiation.

[c]Molten Salt Reactor Experiment surveillance specimens exposed to MSRE core for 5600 hr at 650°C.

[d]D. G. Harman, personal communication, 1970.

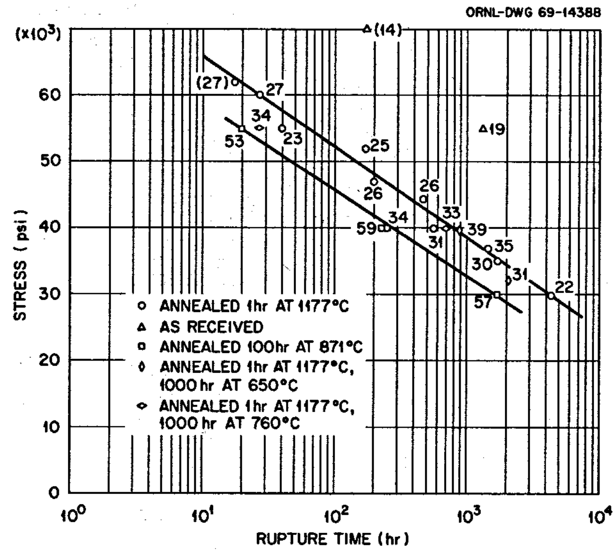

Fig. 17. Creep-Rupture Properties of Unirradiated Alloy 21545 (0.49% Ti) at 650°C. The number by each datum point designates the fracture strain.

examination of the data in Table 15 reveals that the aging treatment of 1000 hr at 760°C increased the minimum creep rate.

The results of creep-rupture tests at 650°C after irradiation are shown in Figs. 18 through 20 for material annealed 1 hr at 1177°C. The irradiation temperatures are indicated by each datum point. At irradiation temperatures of 650°C or below, the rupture life was reduced only a small amount, and the minimum creep rate was unchanged. At an irradiation temperature of 760°C and test temperature of 650°C, the rupture life decreased at least one order of magnitude, and the minimum creep rate increased one order of magnitude. The corresponding fracture strains shown in Fig. 20 are 3% and higher for the samples irradiated at 650°C or below and about 0.5% for samples irradiated at 760°C. Tests R-841 and R-817 (Table 16) demonstrate the ability of annealing at 760°C after irradiation to degrade the properties at 650°C. This observation supports the premise that a structural change during annealing at 760°C is responsible for the poor properties after irradiation. A comparison of the results for heat 21545 in Figs. 18 through 20 with those in Fig. 14 for alloy 104 (0.55% Ti) shows good qualitative agreement.

The results of postirradiation creep tests on material having a fine grain size (annealed at 871°C) are also given in Table 16. The fracture

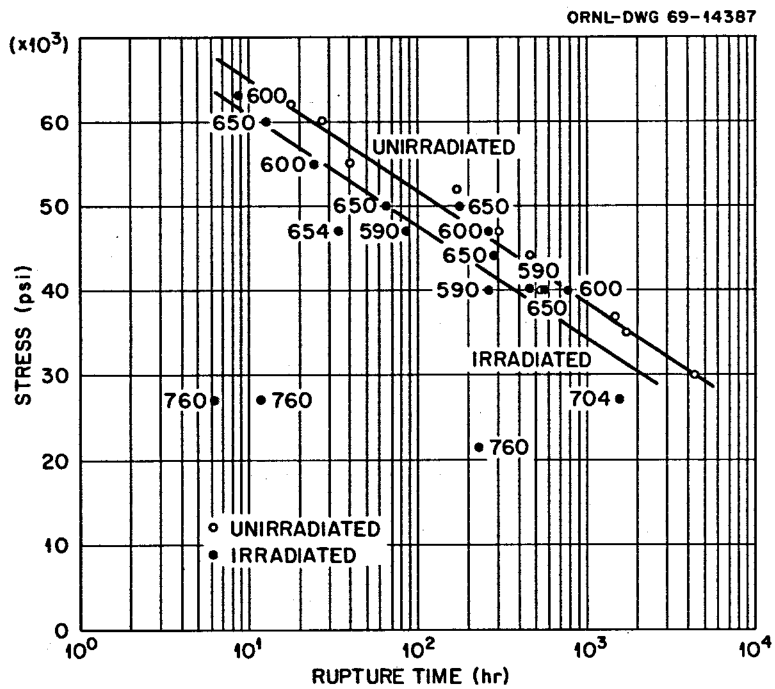

Fig. 18. Stress-Rupture Properties of Alloy 21545 (0.49% Ti) at 650°C. All material annealed 1 hr at 1177°C. Number by each datum point designates the irradiation temperature in degrees Celsius.

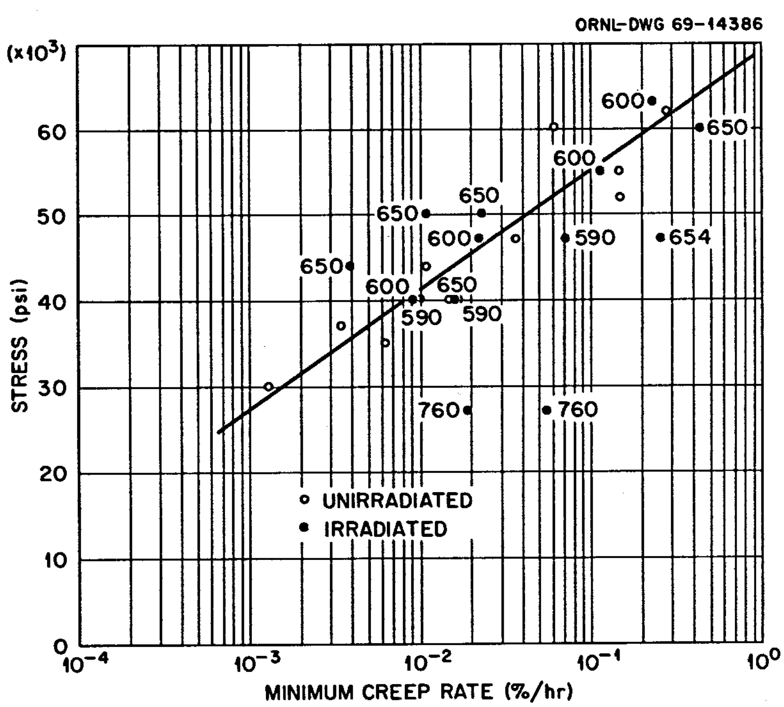

Fig. 19. Creep Properties at 650°C of Alloy 21545 (0.49% Ti). All material annealed 1 hr at 1177°C. Number by each datum point designates irradiation temperature in degrees Celsius.

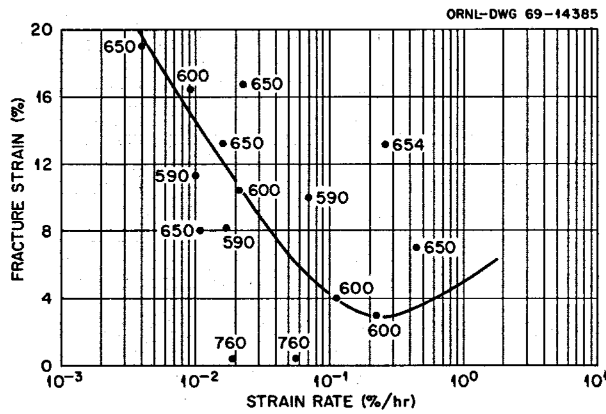

Fig. 20. Fracture Strain of Alloy 21545 (0.49% Ti) at 650°C after Irradiation. All material annealed 1 hr at 1177°C. Number by each datum point designates the irradiation temperature in degrees Celsius.

strains for these samples were generally lower than those for the material annealed 1 hr at 1177°C. The stress-rupture properties of this material are shown in Fig. 21. In addition to the rupture life being low for the fine-grained, unirradiated material, the rupture life was reduced at least 50% by irradiation.

Some of the fine-grained material of heat 21545 was included in the surveillance program for the MSRE. The detailed results of these tests

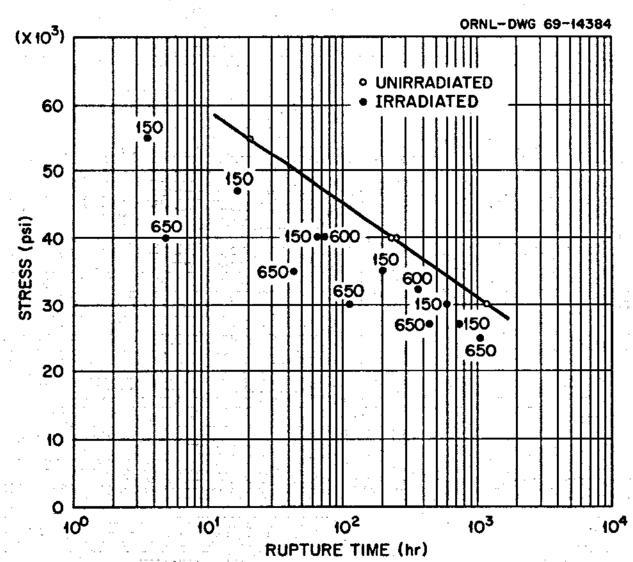

Fig. 21. Stress-Rupture Properties of Alloy 21545 (0.49% Ti) at 650°C. Annealed 100 hr at 871°C to produce a fine grain size. Number by each datum point indicates the irradiation temperature in degrees Celsius.

were reported previously[6] and will be discussed only briefly in this report. These samples were exposed to molten fluoride salt for 5600 hr at 650°C while receiving a thermal-neutron fluence of 4.1×10^{20} neutrons/cm^2. The stress-rupture properties of these samples are shown in Fig. 22. The unirradiated control samples were exposed to barren fuel salt for an equivalent length of time and show a slight reduction in rupture life (compare Figs. 17 and 22). Irradiation decreased the rupture life about an order of magnitude, and the fracture strains ranged from 1.6 to 3.7%. Since the metal was quite compatible with the salt, we attribute the changes to the long thermal anneal and the neutron exposure.

Several creep tests were run at 760°C on both the unirradiated and irradiated materials. The results of these tests are given in Tables 15 and 16, and the stress-rupture properties are shown graphically in Fig. 23. The rupture life at 760°C was not reduced by irradiation at 650°C or below, but was reduced drastically by irradiation at 760°C. The data in Table 16 also show that the higher irradiation temperature increased the minimum creep rate and decreased the fracture strain.

The microstructures of several samples were examined after testing. Typical photomicrographs of alloys 100, 102 (0.39% Ti), 104 (0.55% Ti), and 107 (1.04% Ti) after extended aging at 650°C are shown in Fig. 24. The carbide precipitation along grain boundaries and within the matrix was light in alloy 100, heavier in alloy 102, and still heavier for alloys 104 and 107. The fracture of alloy 100 after creep testing at 40,000 psi and 650°C is shown in Fig. 25. Numerous intergranular cracks were present, and the fracture was intergranular. The short time of about 50 hr at temperature was not sufficient to cause carbide precipitation. The fracture of alloy 104 (0.55% Ti) after testing at 40,000 psi and 650°C is shown in Fig. 26. Details of importance are the carbide stringers, general carbide precipitation, intergranular cracking, and a predominantly intergranular fracture. This sample was at temperature for 1000 hr, and copious amounts of carbide precipitate formed. Photomicrographs of the fracture of alloy 107 (1.04% Ti) are shown in Fig. 27.

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[6]H. E. McCoy, Jr., An Evaluation of the Molten-Salt Reactor Experiment Hastelloy N Surveillance Specimens — Second Group, ORNL-TM 2359 (1969).

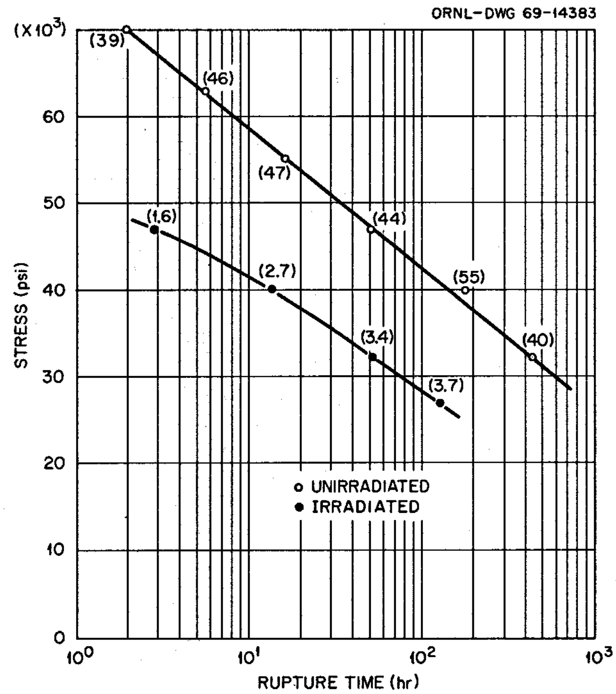


Fig. 22. Stress-Rupture Properties of Alloy 21545 (0.49% Ti) at 650°C. All material annealed 100 hr at 871°C and exposed to fluoride salt for 5600 hr at 650°C. Number by each datum point indicates fracture strain.

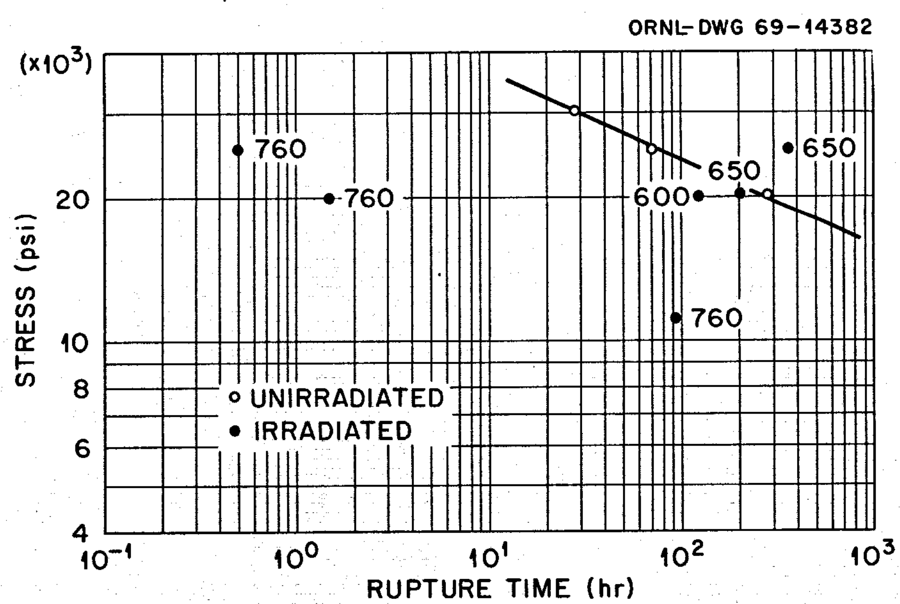


Fig. 23. Stress-Rupture Properties of Alloy 21545 (0.49% Ti) at 760°C. All material annealed 1 hr at 1177°C. Number by each datum point indicates the irradiation temperature in degrees Celsius.

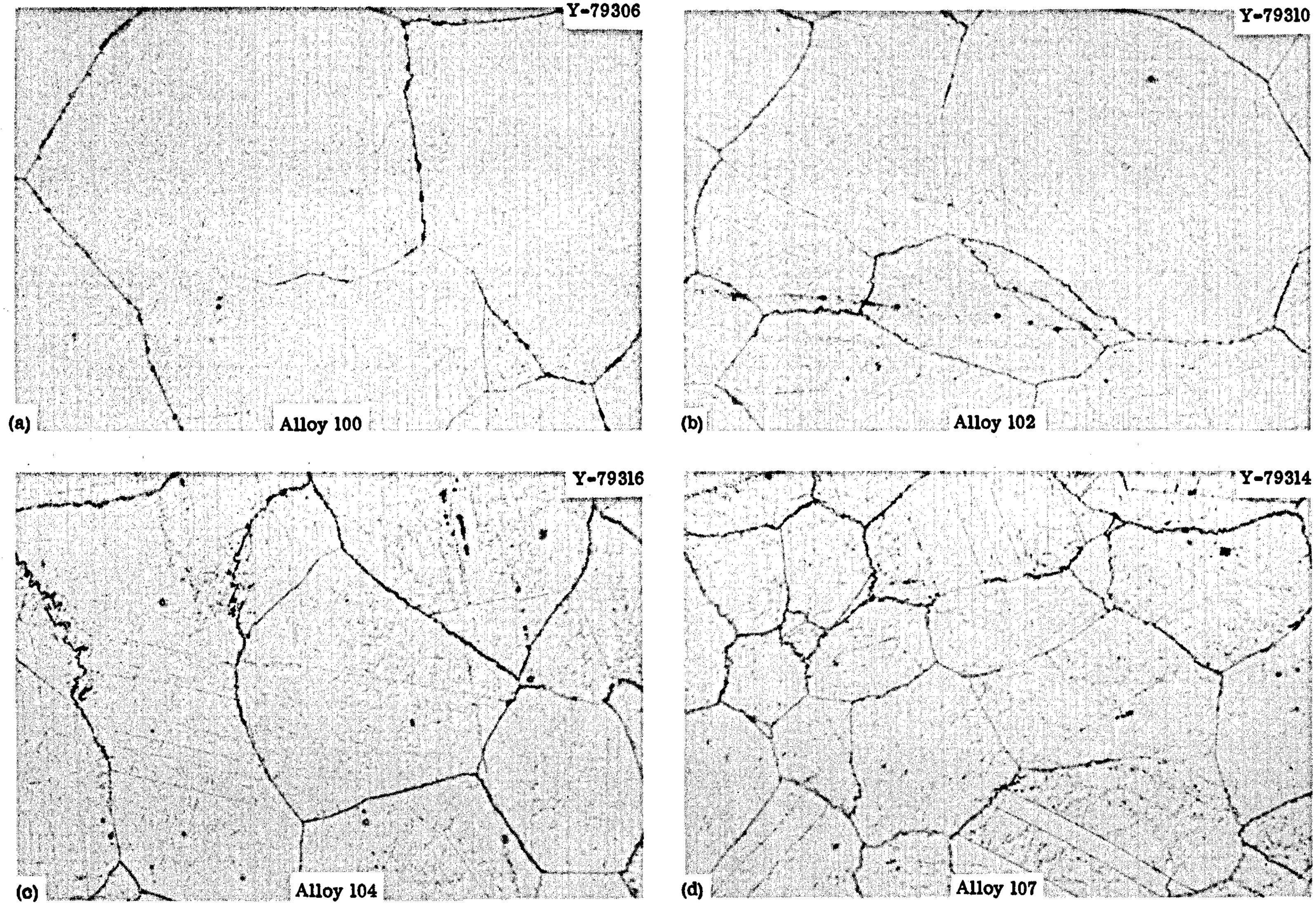


Fig. 24. Microstructures of Several Titanium-Modified Alloys Annealed 1 hr at 1177°C, Aged 1000 hr at 650°C, and Stressed 1000 hr at 650°C at 32,400 psi. Etchant: glyceria regia, 500X. Reduced 22.5%.

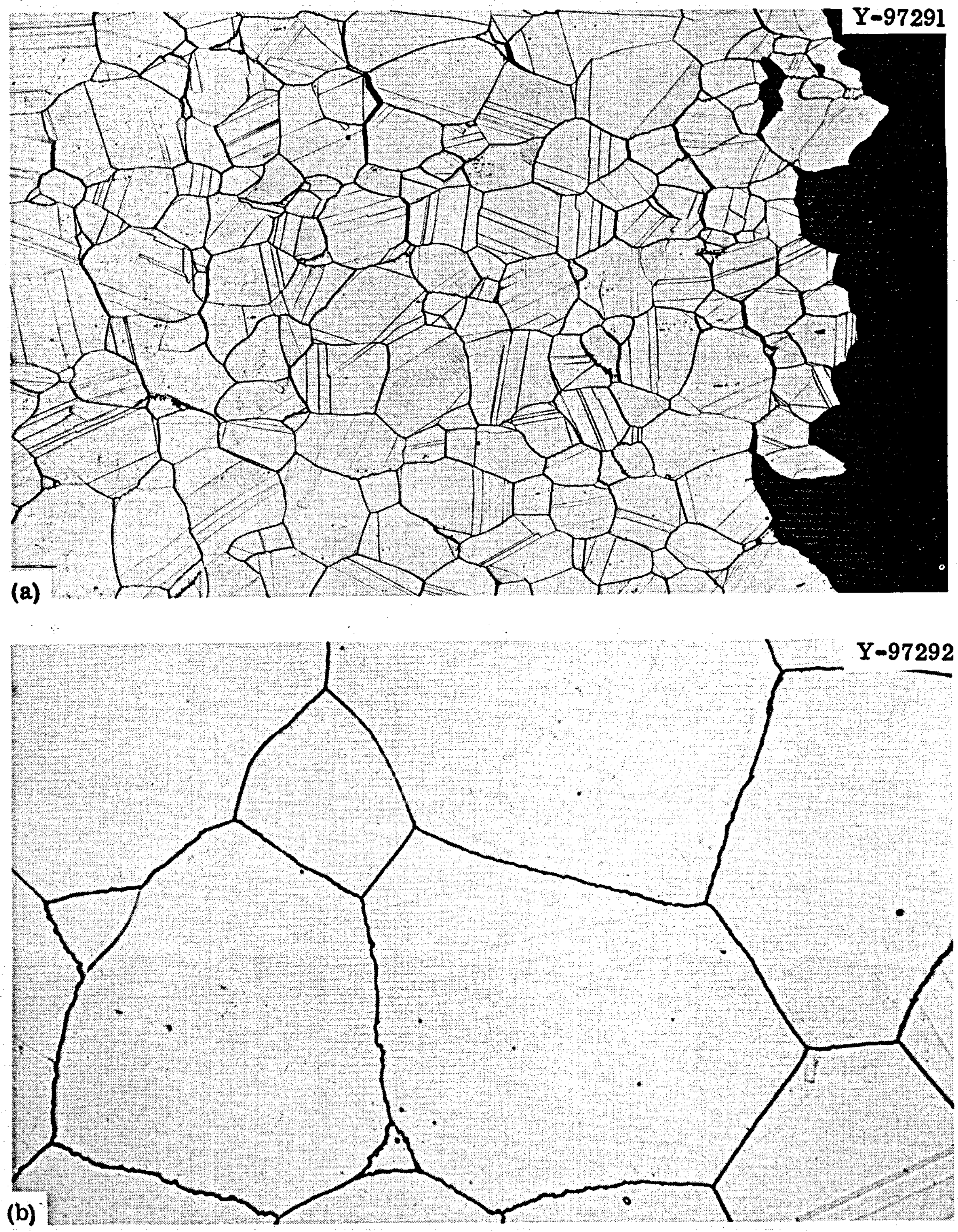


Fig. 25. Microstructures of Alloy 100 after Annealing 1 hr at 1177°C and Testing at 40,000 psi and 650°C. (a) Fracture, 100×; (b) typical, 500×. Etchant, glyceria regia.

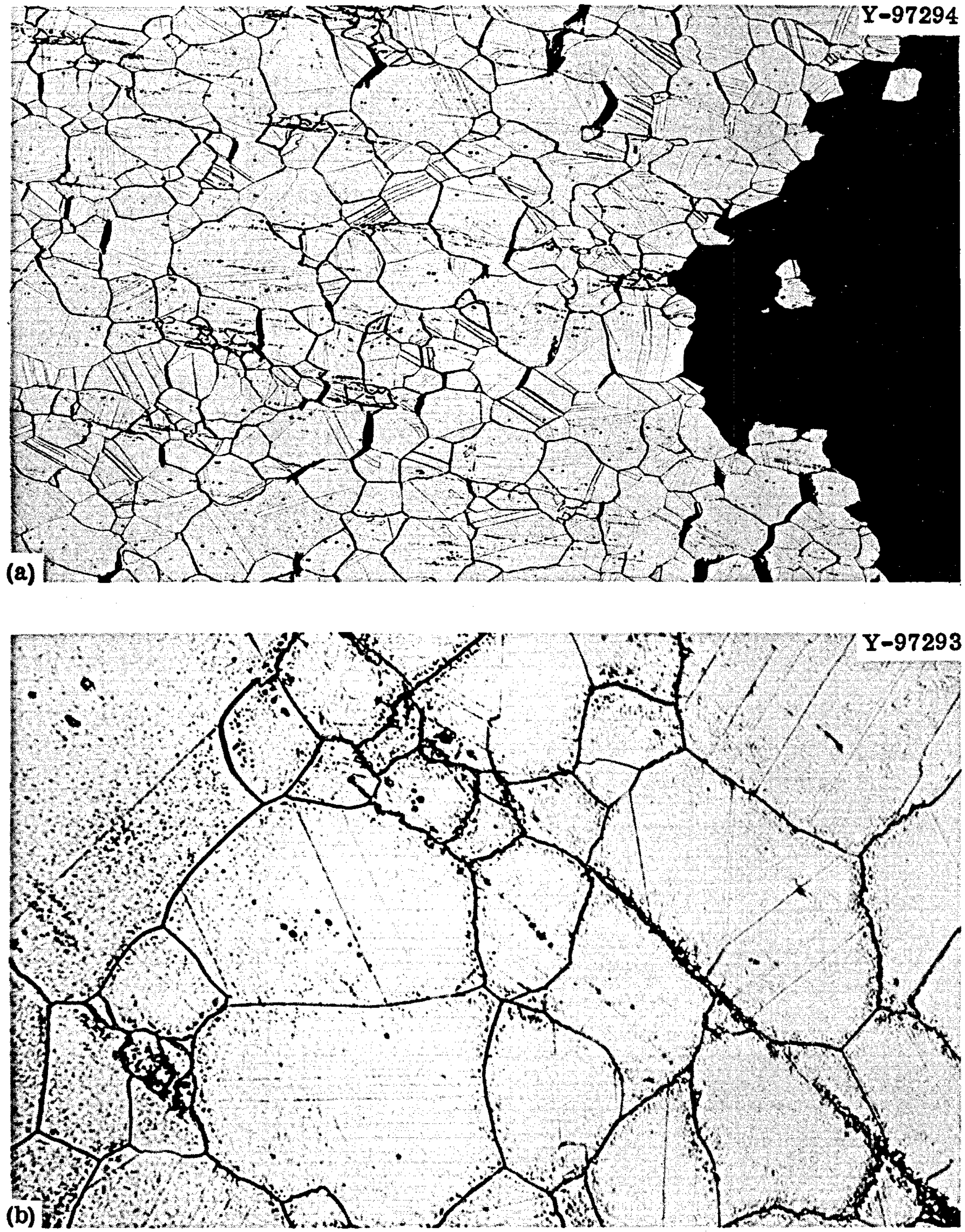


Fig. 26. Photomicrographs of Alloy 104 (0.55% Ti) after Annealing 1 hr at 1177°C and Stressing at 40,000 psi at 650°C. (a) Fracture, 100X; (b) typical, 500X. Etchant, glyceria regia.

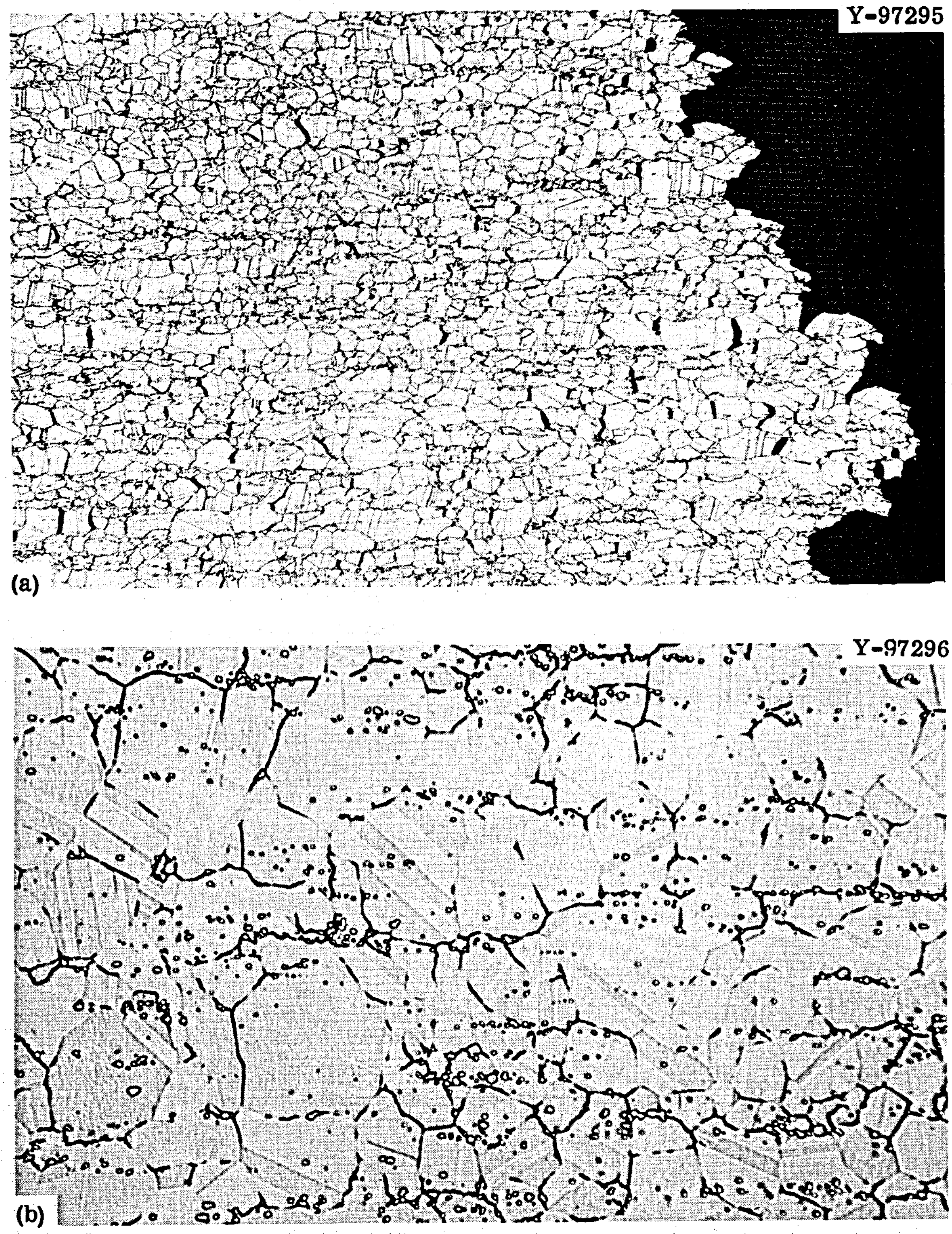


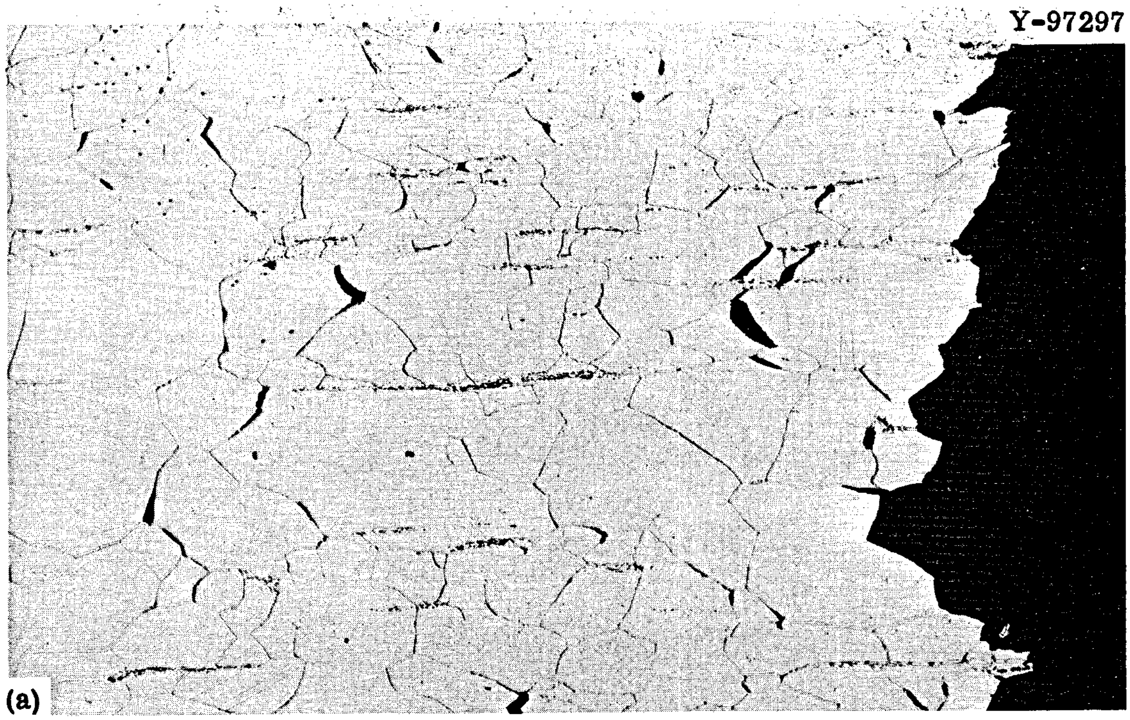
Fig. 27. Photomicrographs of Alloy 107 (1.04% Ti) after Annealing 1 hr at 1177°C and Testing at 650°C and 40,000 psi. (a) Fracture, 100X; (b) typical, 500X. Etchant, glyceria regia.

The sample was stressed at 40,000 psi at 650°C and fractured after 1490 hr. The grain size was quite small, and there were large quantities of precipitate present. The fracture was predominantly intergranular.

Alloy 21545 (0.49% Ti) was examined metallographically after being stressed at 40,000 psi at 650°C. The fracture was predominantly intergranular (Fig. 28). The sample was at temperature 564 hr, and some very fine precipitation occurred in the matrix and at grain boundaries. The stringers of precipitate were present in the annealed material before testing (Fig. 4).

Alloy 100 (< 0.01% Ti) was examined after irradiation and creep testing at 650°C. Typical microstructures are shown in Fig. 29. This sample failed after only 1.1% strain (Table 7). There were a few intergranular cracks, but the deformation seemed restricted largely to the zone near the fracture. Some fine carbides were formed at the grain boundaries and in the matrix. The microstructure of alloy 104 (0.55% Ti) is shown in Fig. 30 after irradiation and creep testing at 650°C. This alloy failed after 10.0% strain; the fracture was largely intergranular. Alloy 107 (1.04% Ti) failed after 11.1% strain. The microstructure, shown in Fig. 31, consists of an intergranular fracture with some carbide precipitation.

Alloy 21545 (0.49% Ti) was examined after exposure to the MSRE core and control facility. The material was annealed at 871°C before exposure to give a fine grain size (Fig. 4). The samples were then exposed to fluoride salt at 650°C for 5600 hr. Some of the samples were exposed to barren fuel salt for controls, and others were irradiated in the MSRE core. Microstructures of an irradiated sample that was tested at 25°C are shown in Fig. 32. The fracture was transgranular. There was some edge cracking due partially to slight changes in composition near the surface. The comparative control sample is shown in Fig. 33. The fracture was transgranular, and there was less edge cracking. Another irradiated sample was tested at 650°C; typical photomicrographs are shown in Fig. 34. The fracture was intergranular, and there were a few intergranular cracks near the fracture. There were some edge cracks. The control sample shown in Fig. 35 was more ductile, with 40.8% strain compared with only 6.2% for the irradiated sample shown in Fig. 34. The higher strain led to extensive intergranular cracking.



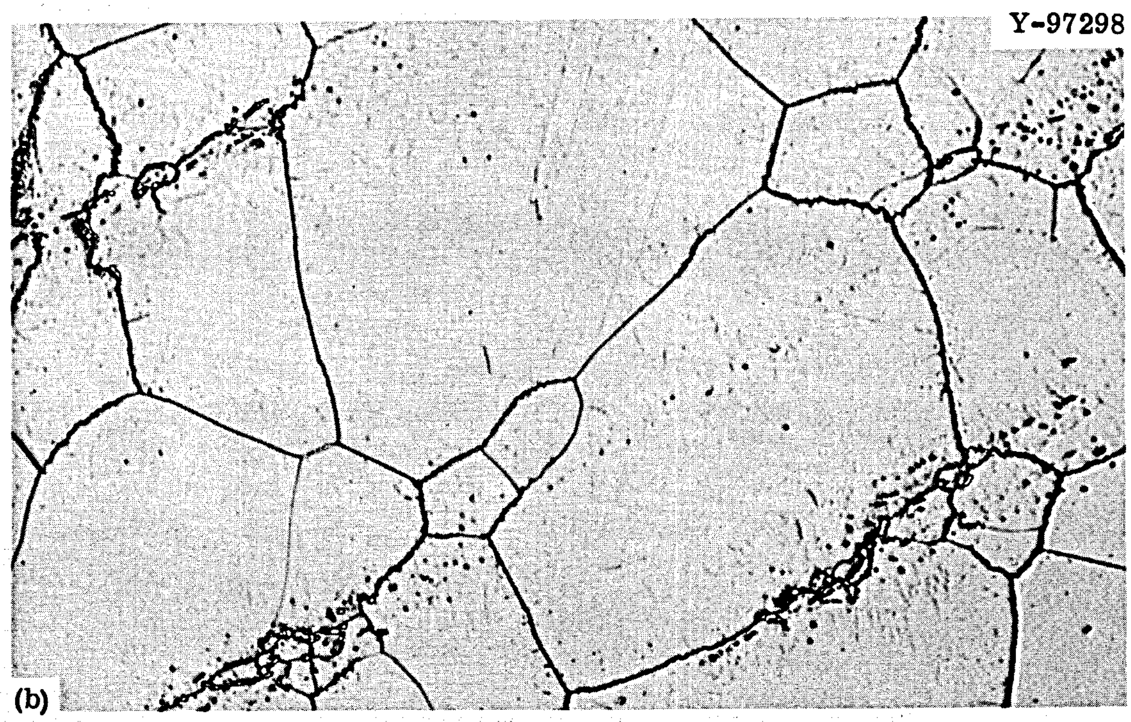
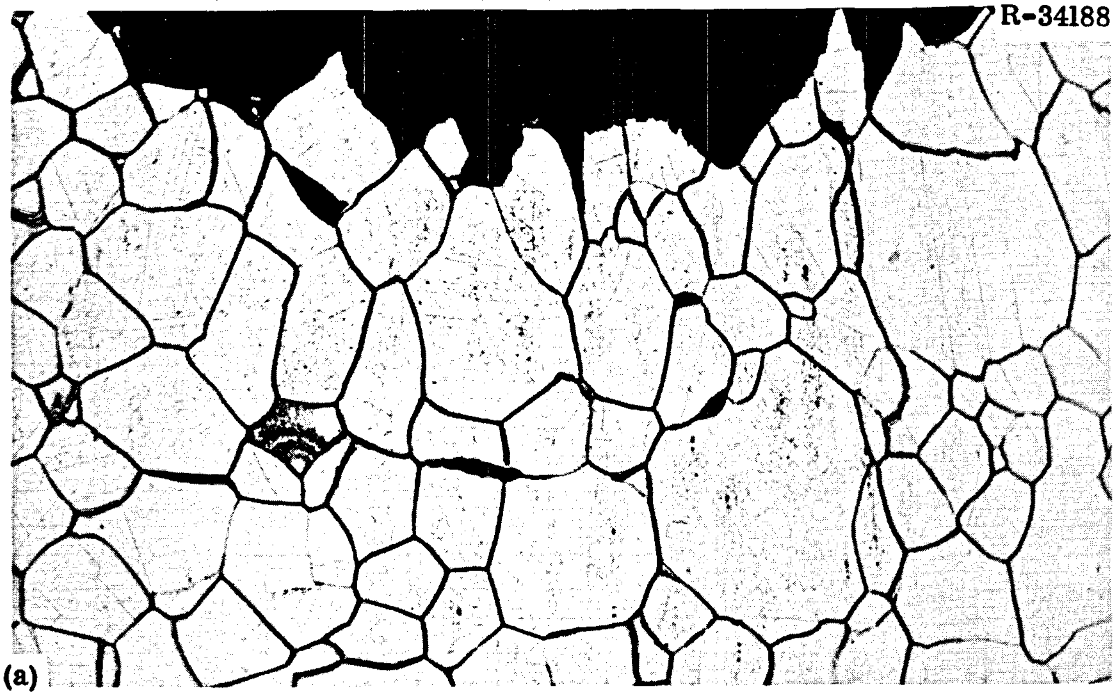


Fig. 28. Photomicrographs of Alloy 21545 (0.49% Ti) after Annealing 1 hr at 1177°C and Testing at 650°C and 40,000 psi. (a) Fracture, 100×; (b) typical, 500×. Etchant: glyceria regia.

R-34188



(a)

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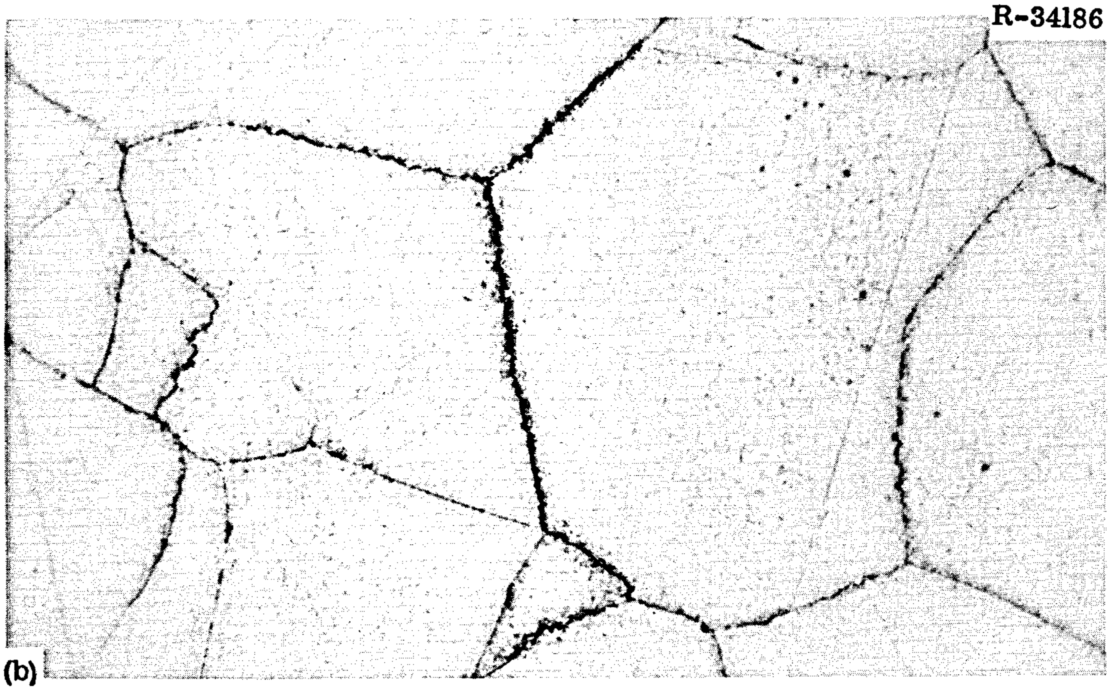


(b)

Fig. 29. Photomicrographs of Alloy 100 (< 0.01% Ti) after Annealing 1 hr at 1177°C, Irradiation for 1000 hr at 650°C to a Thermal Fluence of 2.5×10^{20} neutrons/cm^2, and Testing at a Stress of 32,400 psi at 650°C. Fractured in 105.4 hr with 1.1% strain. (a) Fracture, 100×; (b) typical, 500×. Etchant: aqua regia.

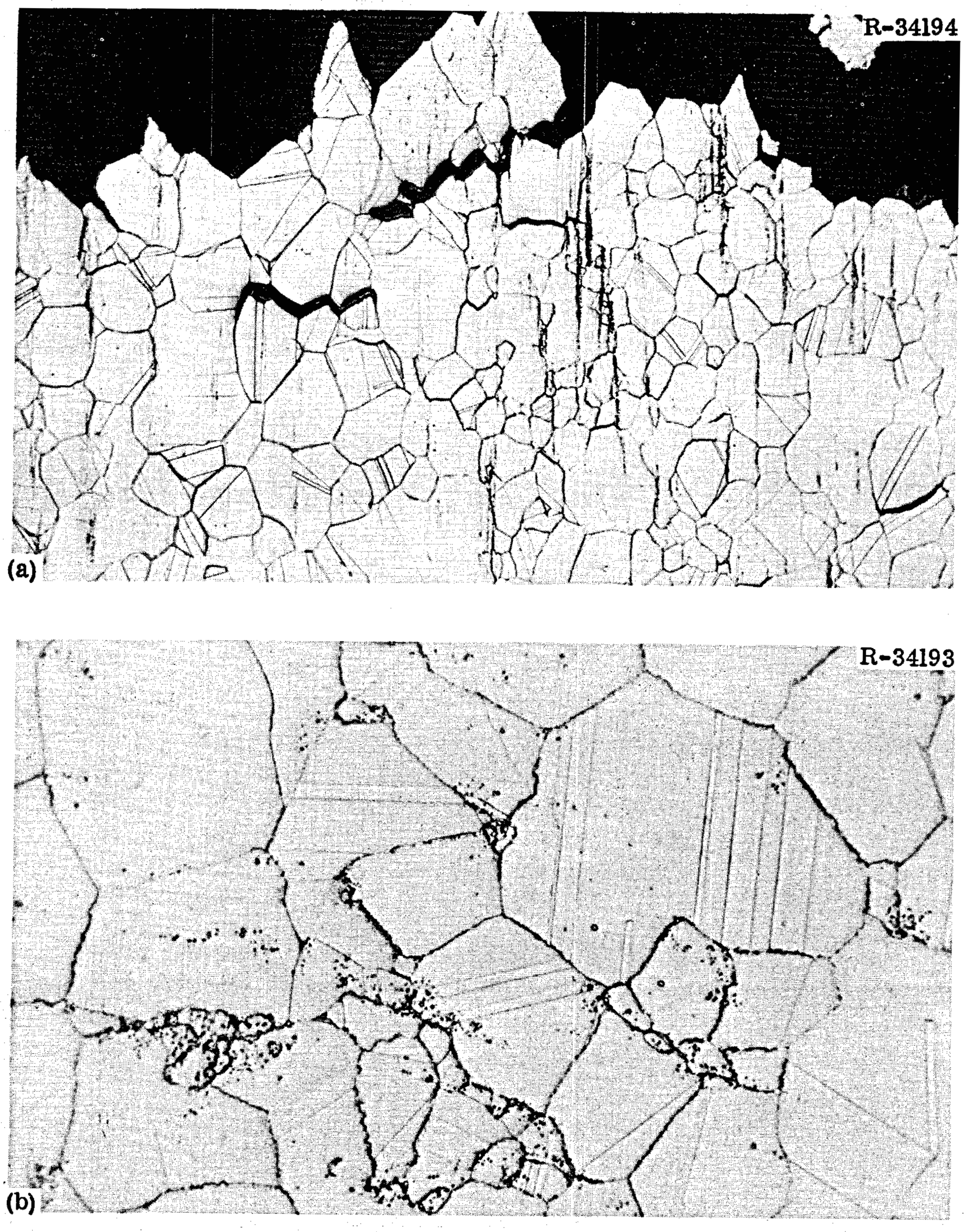
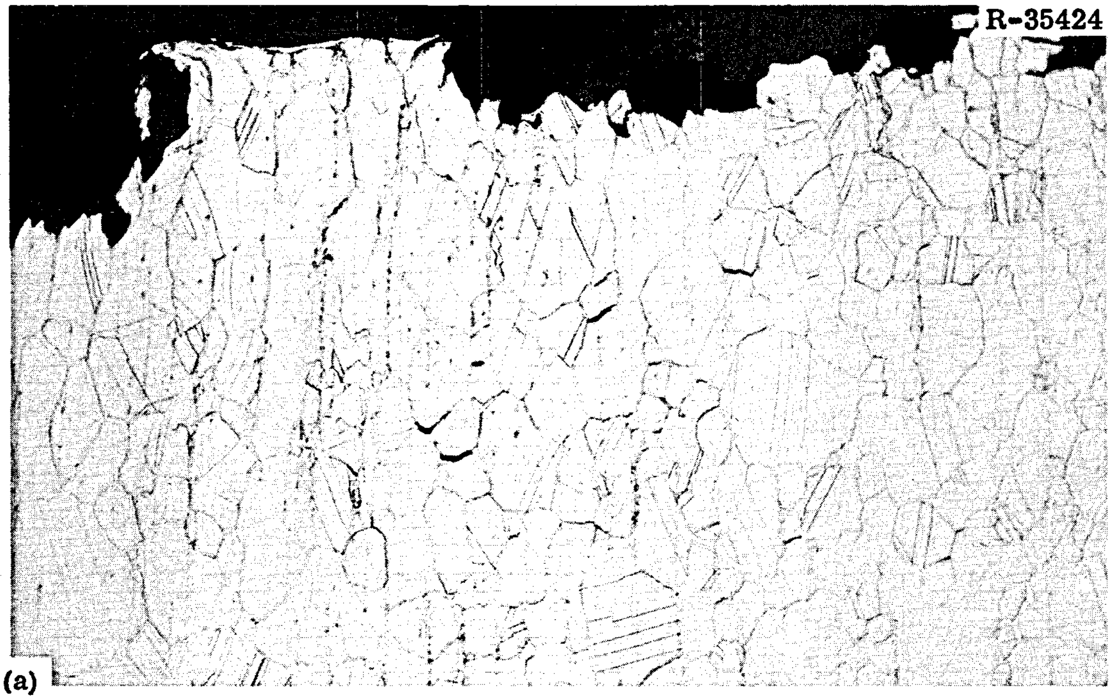
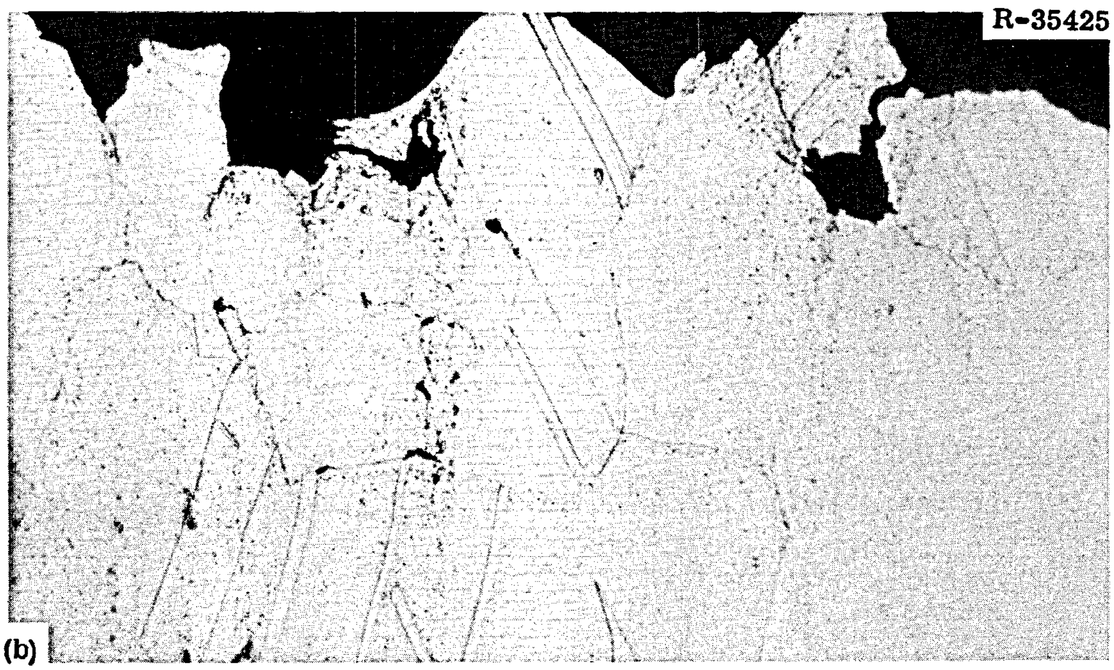


Fig. 30. Photomicrographs of Alloy 104 (0.55% Ti) Annealed 1 hr at 1177°C, Irradiated for 1000 hr at 650°C to a Thermal Fluence of 2.5×10^{20} neutrons/cm^2, and Stressed at 32,400 psi at 650°C. Fractured in 1446.3 hr with 10.0% strain. (a) Fracture, 100×; (b) typical, 500×. Etchant: aqua regia.



(a)



(b)

Fig. 31. Photomicrographs of Alloy 107 (1.04% Ti) Annealed 1 hr at 1177°C, Irradiated for 1000 hr at 650°C to a Thermal Fluence of 2.5×10^{20} neutrons/cm^2, and Stressed at 32,400 psi at 650°C. Fractured in 2497.2 hr with 11.1% strain. (a) Fracture (flattened end caused by handling), 100×; (b) fracture (blunted tip caused by handling), 500×.

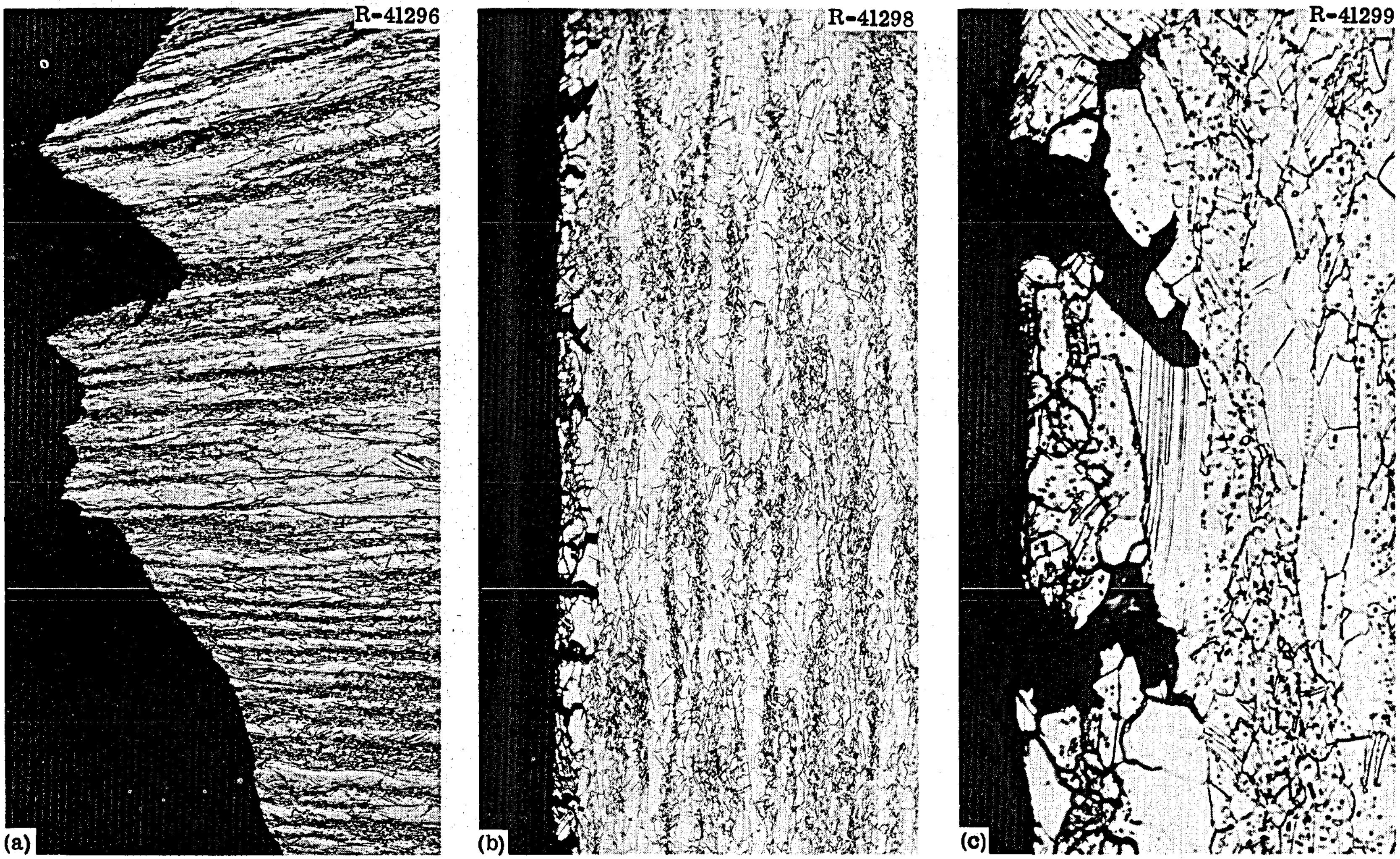


Fig. 32. Photomicrographs of a Titanium-Modified Hastelloy N Surveillance Sample (Heat 21545) Tested at 25°C at a Strain Rate of 0.05 min^{-1}. Exposed in the MSRE core for 5500 hr at 650°C to a thermal fluence of 4.1×10^{20} neutrons/cm^2. (a) Fracture, 100×; (b) edge of sample about 1/2 in. from fracture, 100×; (c) edge of sample showing edge cracking, 500×. Etchant: aqua regia. Reduced 8%.

Fig. 33. Photomicrograph of the Fracture of a Titanium-Modified Hastelloy N Surveillance Sample (Heat 21545) Tested at 25°C at a Strain Rate of 0.05 min^{-1}. Exposed to a static fluoride salt for 5500 hr at 650°C before testing. Note the shear fracture and the absence of edge cracking. 100×. Etchant: glyceria regia.

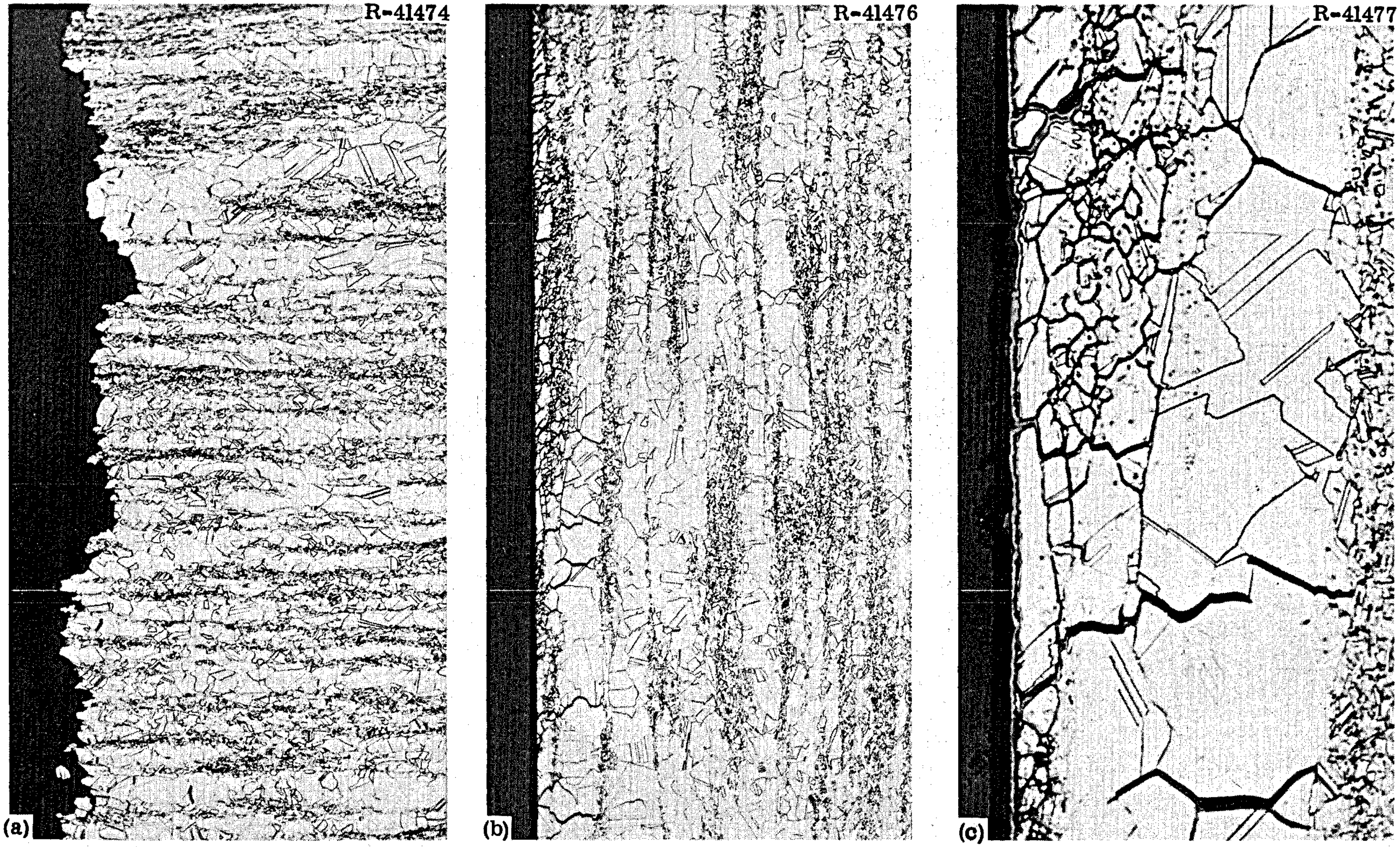


Fig. 34. Photomicrographs of a Titanium-Modified Hastelloy N Surveillance Sample (Heat 21545) Tested at 650°C at a Strain Rate of 0.002 min^{-1}. Exposed in the MSRE core for 5500 hr at 650°C to a thermal fluence of 4.1×10^{20} neutrons/cm^2. Failed after 6.2% strain. (a) Fracture, 100×; (b) edge of sample about 1/2 in. from fracture, 100×; (c) edge of sample showing edge cracking, 500×. Oxide formed during the tensile test. Etchant: aqua regia. Reduced 10%.

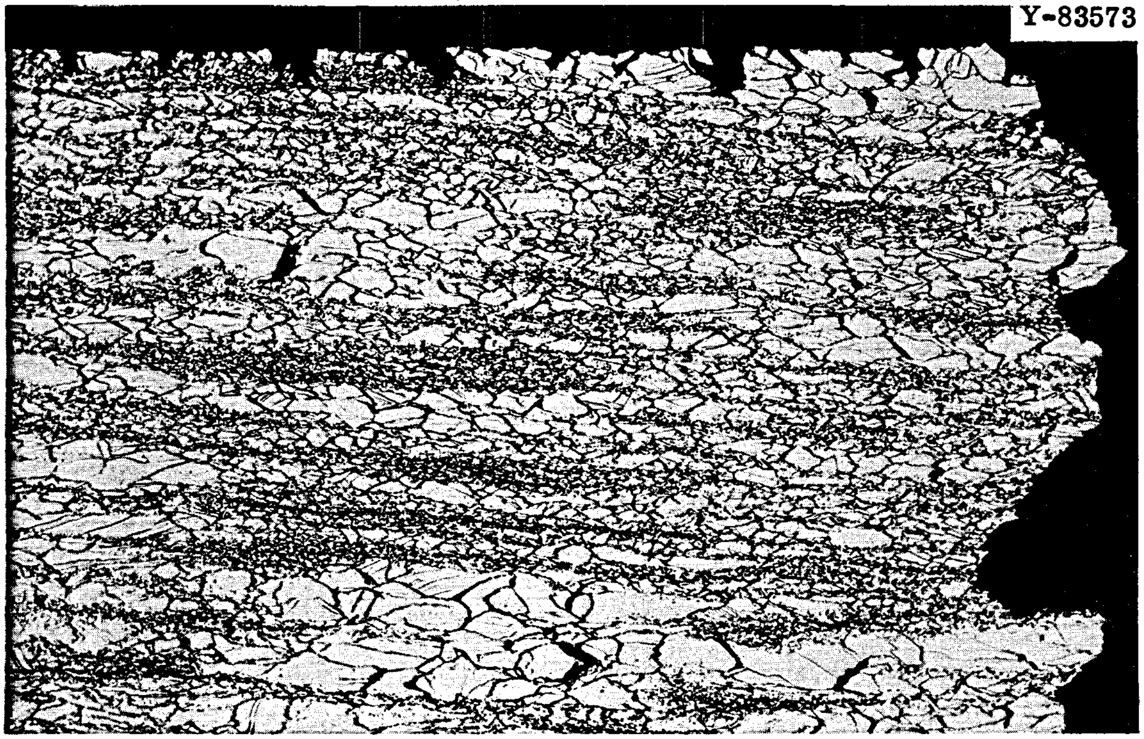


Fig. 35. Photomicrograph Showing a Portion of the Fracture of a Titanium-Modified Hastelloy N Surveillance Sample (Heat 21545) Tested at 650°C at a Strain Rate of 0.002 min^{-1}. Failed after 40.8% strain. Exposed to static fluoride salt for 5500 hr at 650°C before testing. 100×. Etchant: glyceria regia.

Alloys Containing Zirconium

Three groups of alloys were studied that contained various amounts of zirconium. A series of 2-lb laboratory melts containing from less than 0.03 to 1.18% Zr was made from virgin melting stock. These alloys are designated 100 and 108 through 112; their chemical compositions are given in Table 1. Typical microstructures of these alloys after a 1-hr anneal at 1177°C are shown in Fig. 36. The addition of the first 0.1% Zr (alloy 108) markedly decreased the grain size. Further additions up to the maximum 1.18% Zr caused only slight additional refinement in grain size. The second group consisted of two alloys that contained 1% Zr with additions of 0.1 and 1.0% Re. The third series of alloys consisted of two 100-lb heats from Special Metals Corporation that contained 0.05 and 0.35% Zr, designated heats 21555 and 21554, respectively. The alloy

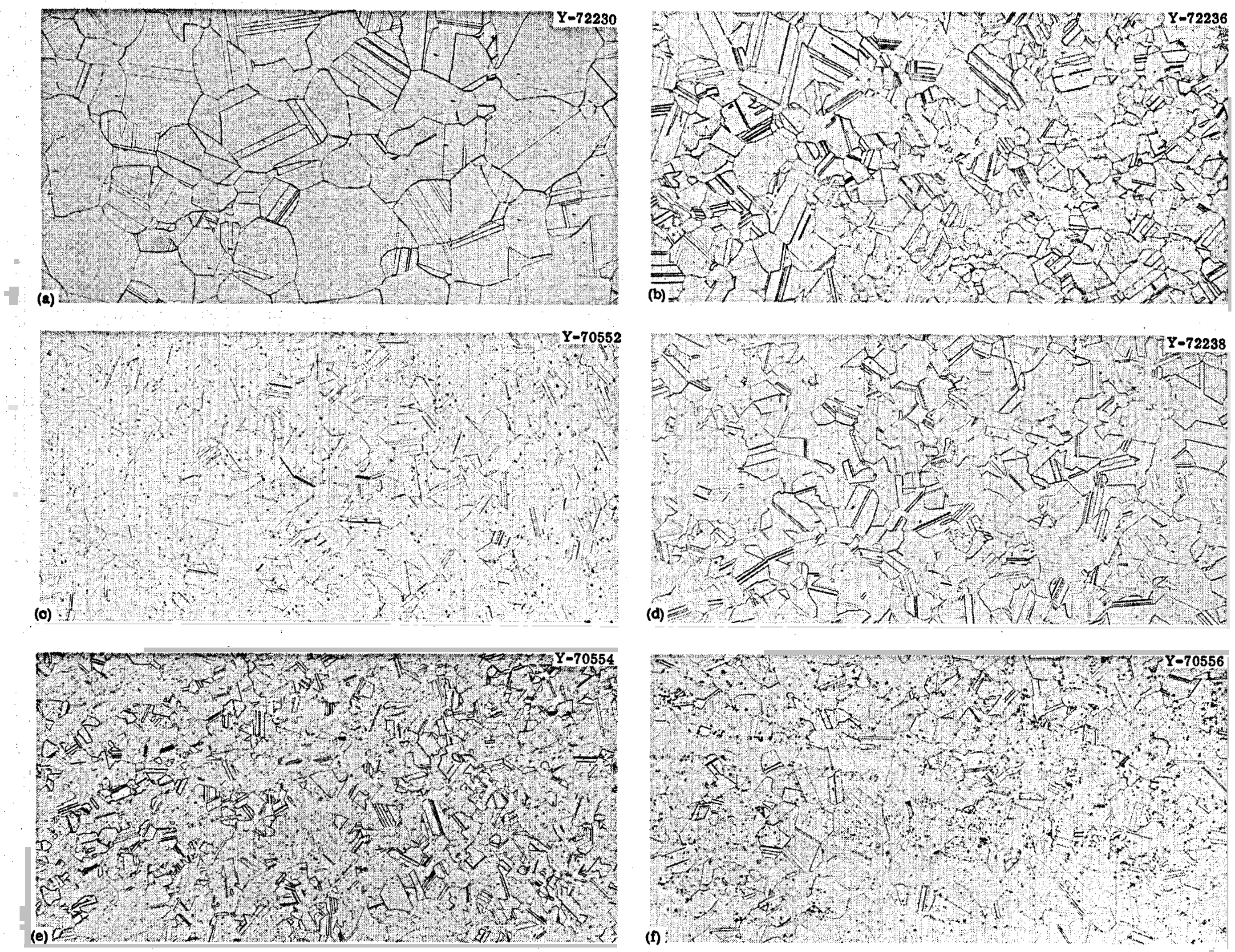


Fig. 36. Typical Photomicrographs of Alloys 100 and 108 Through 112. Annealed 1 hr at 1177°C. (a) Alloy 100 (< 0.03% Zr), (b) alloy 108 (0.10% Zr), (c) alloy 109 (0.28% Zr), (d) alloy 110 (0.53% Zr), (e) alloy 111 (0.75% Zr), and (f) alloy 112 (1.18% Zr). 100×. Etchant: glyceria regia. Reduced 42.5%.

with the higher zirconium content from Special Metals Corporation was initially forged at 1177°C and cracked profusely. A second alloy was melted and forged at 1066°C, and a good product was obtained. This experience gave the first indication that zirconium caused the alloy to be hot-short and that it would likely have poor weldability. Typical photomicrographs of these two alloys are shown in Figs. 37 and 38. As received, the material contained 40% cold work; annealing at 871°C produced a banded structure with a fine grain size and numerous carbides. The banding is likely due to the working pattern, but there was also some variation in the density of the carbides.

Alloys 100 and 108 through 112 were tested to determine their tensile properties. Samples of each alloy were annealed 1 hr at 1177°C and aged for 1000 hr at 650°C to match the thermal history of samples that were irradiated. The results of tensile tests on the unirradiated samples are given in Table 17. The variation of the strength parameters at 650°C with zirconium content is shown in Fig. 39. The yield strength was not affected appreciably until the zirconium level exceeded 0.3%, but it increased markedly above this level. The ultimate tensile strength increased about 10% with the addition of 0.1% Zr, but very little further strengthening occurred as the zirconium content increased. In the absence of aging, the strength was not affected by zirconium content. The fracture strains are shown in Fig. 40 as a function of zirconium content. A very distinct maximum for aged material occurred at a concentration of about 0.3% Zr. The fracture strain was almost doubled by the addition of even 0.1% Zr. The fracture strain at the lower strain rate was lower, a trend that is usually observed. In unaged material, a maximum occurred in the fracture strain for zirconium concentrations of 0.5 to 0.7%.

The same experimental alloys were irradiated at 650°C to a thermal fluence of 2.5×10^{20} neutrons/cm^2. The results of tensile tests on these samples after irradiation are summarized in Table 18, and the variations of the fracture strains with zirconium content are shown in Fig. 41. The postirradiation results also show a ductility maximum at about 0.3% Zr. The results for the irradiated and unirradiated samples are compared in Fig. 42, where the ratios of the various tensile properties

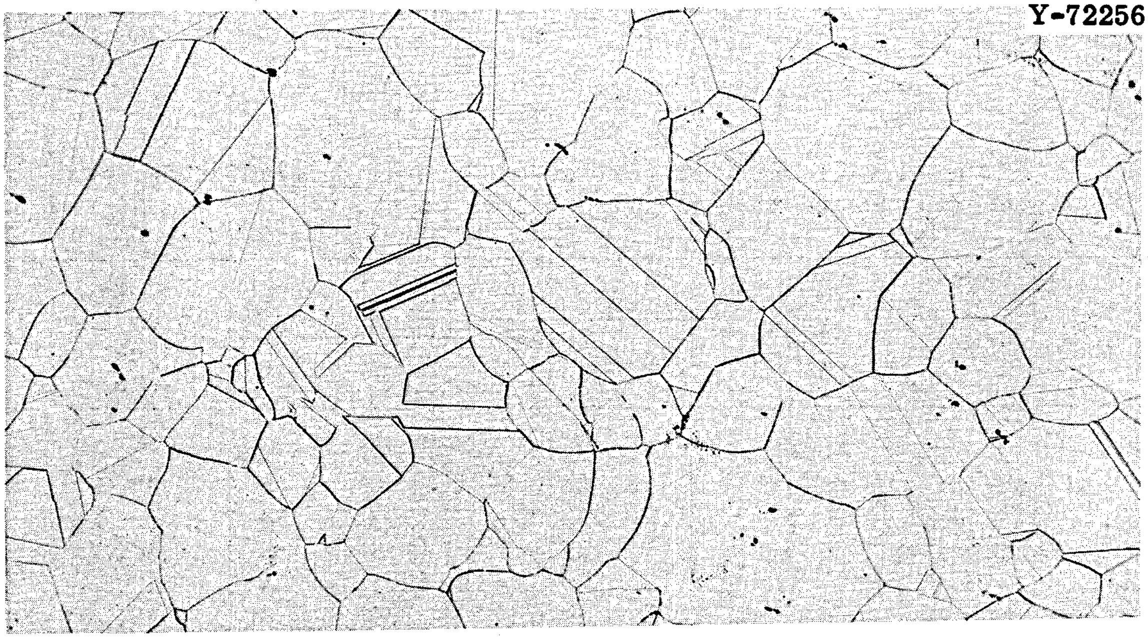


Fig. 37. Typical Photomicrograph of Alloy 21555 (0.05% Zr). Annealed 1 hr at 1177°C. 100×. Etchant: glyceria regia.

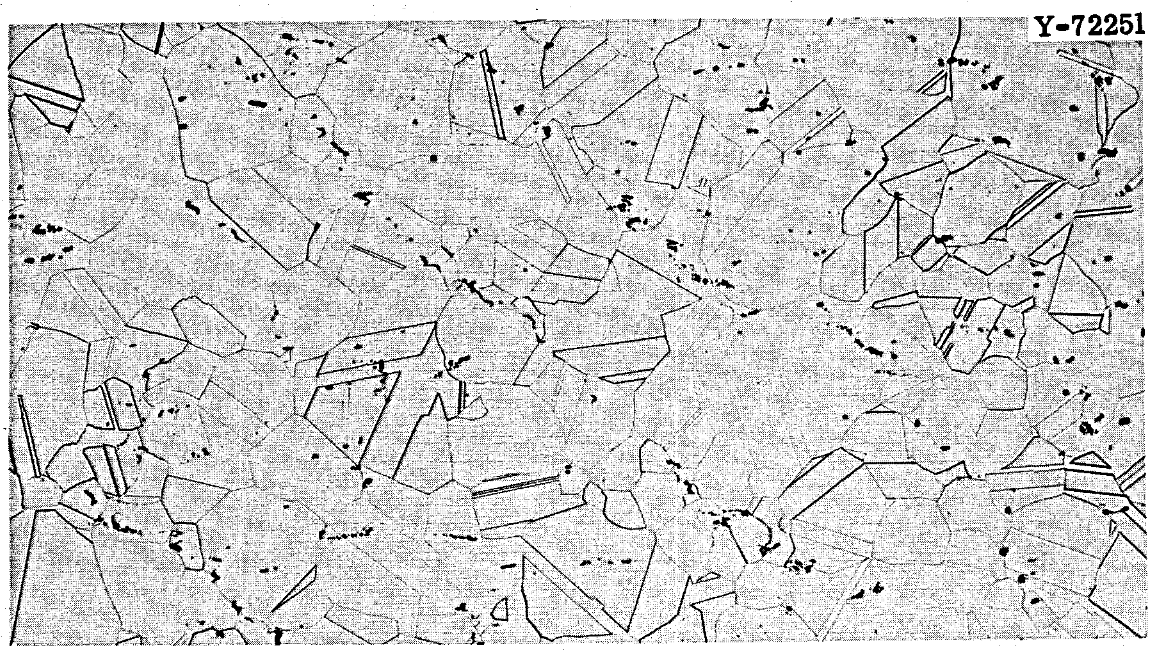


Fig. 38. Typical Photomicrograph of Alloy 21554 (0.35% Zr). Annealed 1 hr at 1177°C. 100×. Etchant: glyceria regia.

Table 17. Tensile Properties of Several Unirradiated Zirconium-Modified Alloys at 650°C

Alloy Number	Specimen Number	Strain Rate (min^{-1})	Stress, psi Yield	Stress, psi Ultimate Tensile	Elongation, % Uniform	Elongation, % Total	Reduction in Area (%)	Heat Treatment
			$\times 10^3$	$\times 10^3$				
108	3870	0.05	25.7	68.3	28.6	29.8	23.7	a
109	3880	0.05	29.4	78.6	33.7	34.7	26.8	a
110	3890	0.05	25.9	75.6	42.8	44.8	30.6	a
111	3900	0.05	26.7	75.5	42.3	44.6	39.9	a
112	3909	0.05	26.5	69.6	32.4	34.4	33.3	a
108	2084	0.05	28.9	86.4	46.1	48.9	42.6	b
108	2078	0.002	29.2	86.2	47.3	50.0	47.2	b
109	2061	0.05	29.1	85.9	54.8	57.7	50.9	b
109	2062	0.002	29.4	84.7	52.5	56.2	47.3	b
110	2105	0.05	43.8	85.8	49.0	56.8	53.1	b
110	2114	0.002	44.6	87	48.9	51.9	48.7	b
111	2132	0.05	50.5	92.2	45.6	50.3	53.2	b
111	2134	0.002	52.9	80.9	35.6	44.4	47.5	b
112	2146	0.05	51.2	89.1	26.9	28.8	28.1	b
112	2148	0.002	55.2	89.3	22.6	23.3	25.8	b

[a]Annealed 1 hr at 1177°C.

[b]Annealed 1 hr at 1177°C plus 1000 hr at 650°C.

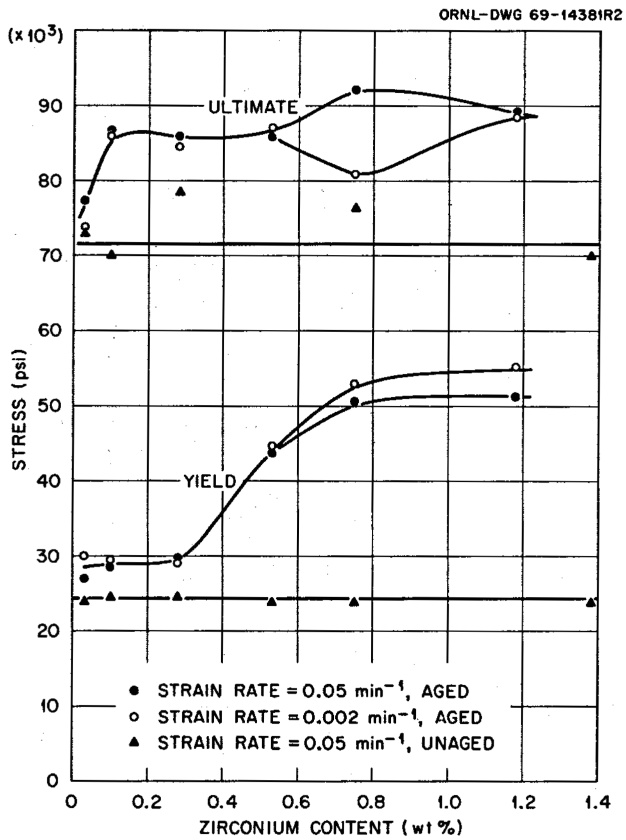


Fig. 39. Influence of Zirconium Content on Tensile Properties after Annealing 1 hr at 1177°C and Aging 1000 hr at 650°C.

Table 18. Tensile Properties of Several Zirconium-Modified Alloys at 650°C after Irradiation[a]

Alloy Number	Specimen Number	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
			Yield	Ultimate Tensile	Uniform	Total	
			$\times 10^3$	$\times 10^3$			
108	2075	0.05	33.9	72.6	28.0	29.0	18.7
108	2087	0.002	35.3	67.4	22.1	23.5	18.9
109	2074	0.002	37.2	65.2	24.5	29.8	28.5
110	2109	0.05	41.2	63.4	16.5	18.5	17.9
110	2116	0.002	43.4	58.1	13.4	17.1	13.6
111	2141	0.002	49.8	60.8	8.5	10.0	12.1
112	2147	0.05	53.9	74.6	14.9	17.2	13.4
112	2152	0.002	56.5	68.7	7.1	7.7	11.9

[a]Annealed 1 hr at 1177°C before irradiation at 650°C to a thermal fluence of 2.5×10^{20} neutrons/cm^2.

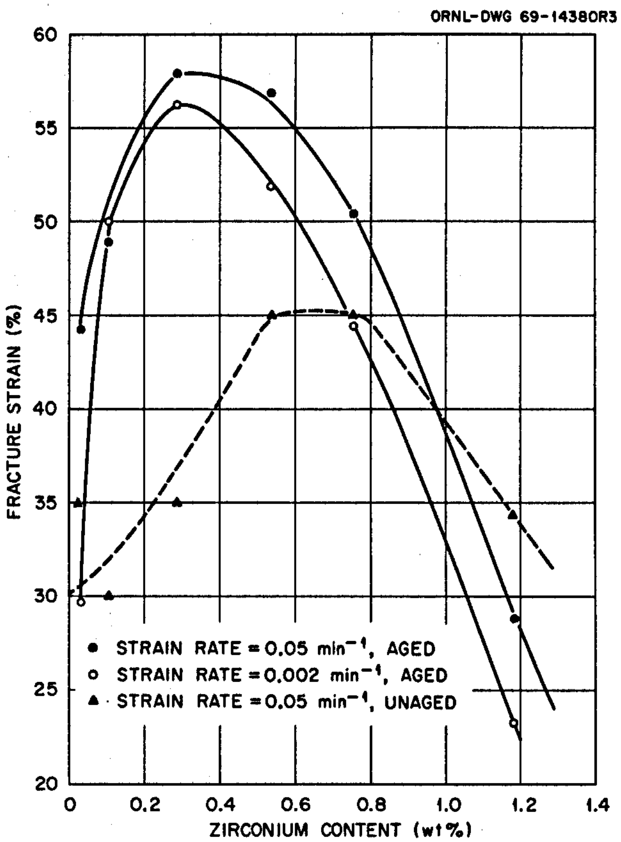


Fig. 40. Influence of Zirconium Content on Fracture Strain. The unaged samples were annealed 1 hr at 1177°C and the aged samples were annealed 1 hr at 1177°C and aged 1000 hr at 650°C before testing.

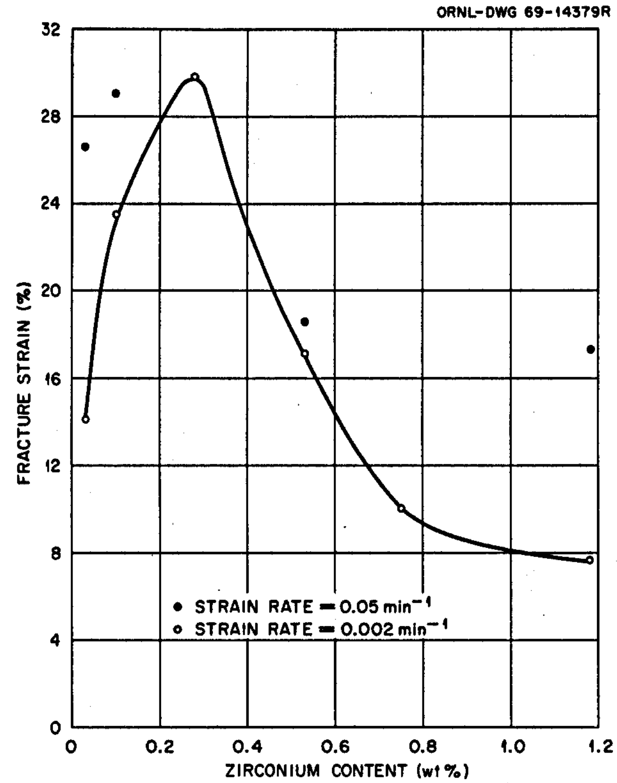


Fig. 41. Influence of Zirconium Content on Fracture Strain at 650°C after Irradiation. Annealed 1 hr at 1177°C before irradiation.

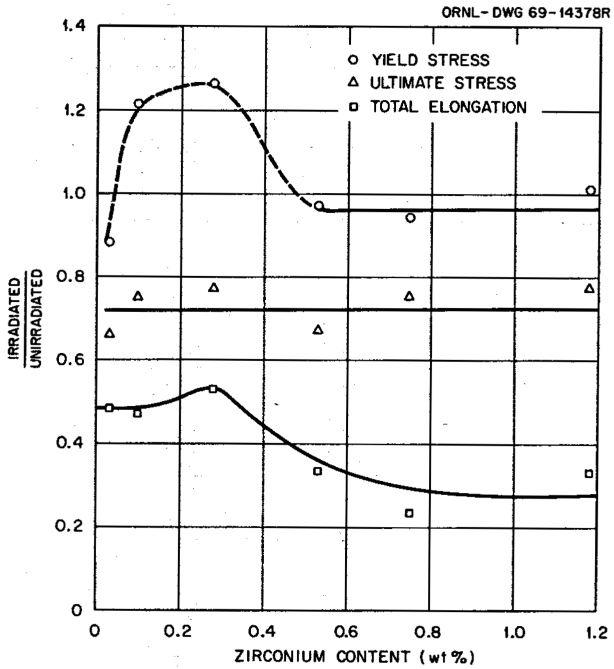


Fig. 42. Effects of Zirconium on the Unirradiated and Irradiated Tensile Properties at 650°C and a Strain Rate of 0.002 min^{-1}.

are also shown. The yield strength was increased slightly by irradiation in alloys that contained 0.10 and 0.28% Zr (alloys 108 and 109) and decreased slightly in the other alloys. The ultimate tensile strength was decreased about 30% in each alloy. The fracture strain was reduced about 50% by irradiation in alloys that contained 0.3% Zr or less and reduced about 70% for alloys that contained more zirconium.

Samples of alloys 100 and 108 through 112 were also irradiated at 43°C to a thermal fluence of 9×10^{19} neutrons/cm^2. These samples were tested at 650 and 760°C; the results are given in Table 19. The fracture strains are shown in Fig. 43 as a function of zirconium concentration. The peak in fracture strain at 650°C with zirconium concentration still occurs, but less sharply and at a different concentration than observed previously (Fig. 41). The fracture strains are also higher in the experiment at lower temperature. We do not know whether to attribute these differences to the variation in irradiation temperature or in fluence. However, the circumstantial evidence indicates that the irradiation temperature is the most important factor. Recall that the yield strengths of the aged control samples varied from 28,000 to 55,000 psi

Table 19. Tensile Properties of Several Zirconium-Modified Alloys after Irradiation[a]

Alloy Number	Specimen Number	Stress, psi		Elongation, %		Reduction in Area (%)
		Yield	Ultimate Tensile	Uniform	Total	
		$\times 10^3$	$\times 10^3$			
		Test Temperature 650°C				
108	2086	31.5	62.9	18.9	19.2	17.5
109	2064	34.7	88.8	37.8	38.9	24.0
110	2113	33.1	79.3	38.6	40.5	34.9
111	2139	33.6	80.8	33.3	37.4	33.8
112	2156	33.8	80	29.9	31.5	21.8
		Test Temperature 760°C				
108	2088	27.4	52.3	10.0	12.3	9.8
109	2058	29.8	58.3	10.2	25.6	28.3
110	2111	27.7	56.7	11.6	25.6	27.3
111	2128	32.2	56.2	12.4	30.7	34.2
112	2153	29	61.8	15.5	18.0	17.5

[a]Annealed 1 hr at 1177°C, irradiated at 50°C to a thermal fluence of 9×10^{19} neutrons/cm^2 and tested at a strain rate of 0.002 min^{-1}.

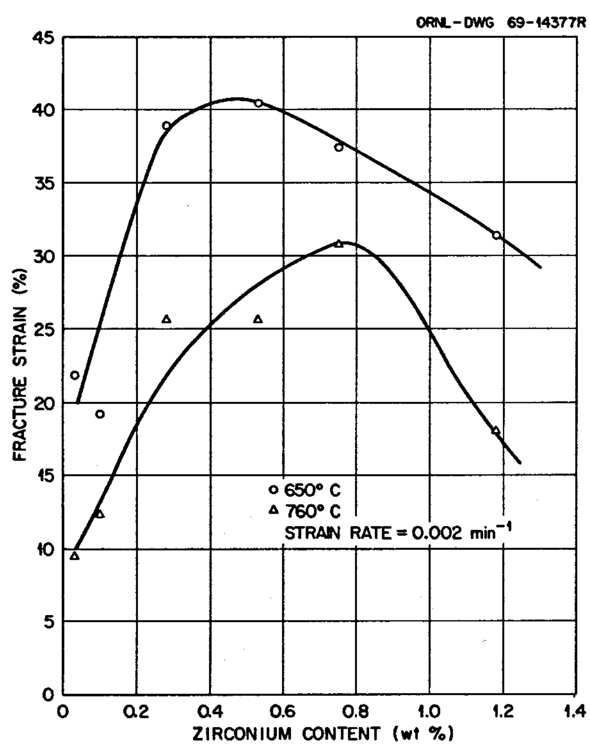


Fig. 43. Fracture Strains of Several Zirconium-Bearing Alloys after Irradiation at 43°C to a Thermal Fluence of 9×10^{19} neutrons/cm^2.

and varied systematically with zirconium content (Fig. 39). Irradiation at 650°C did not change these values markedly (Table 18). The yield strengths of the alloys irradiated at 43°C are all about 33,000 psi and do not vary detectably with zirconium content (Table 19). Thus, the thermal treatment seems necessary to bring about the changes characteristic of alloys with various concentrations of zirconium.

Some of the samples irradiated at 43°C were tested at 760°C. The fracture strain varied markedly with zirconium content (Fig. 43). However, the peak was shifted to a higher zirconium level than was noted at a test temperature of 650°C.

Alloys 100 and 108 through 112 were also creep-rupture tested. The results of several tests on material annealed for 1 hr at 1177°C are shown in Table 20 and in Figs. 44 through 46. The stress-rupture properties, shown in Fig. 44, illustrate the potent effect of zirconium in increasing the rupture life at a given level of stress. The rupture life of alloy 109 (0.28% Zr) seems anomalously high, but the other alloys followed the trend of increasing rupture life with increasing zirconium content. The creep-rupture strength, shown in Fig. 45, was also affected by the zirconium concentration. Again, alloy 109 was the only exception to the trend of increasing strength with increasing zirconium content. Zirconium improved the fracture strain (Fig. 46). The addition of only 0.1% Zr (alloy 108) improved the fracture strain, compared to that of alloy 100, several hundred percent at low strain rates. Higher concentrations of zirconium improved the fracture strain at high strain rates, but brought about little added improvement at low strain rates.

Some samples were aged for 1000 hr at 650°C to correspond to the thermal treatment of irradiated samples. The results of creep-rupture tests on these samples are given in Table 21. A comparison with the properties of the unaged samples in Table 20 shows that this aging treatment did not affect the properties appreciably.

Alloys 100 and 108 through 112 were irradiated to a thermal fluence of 2.5×10^{20} neutrons/cm^2 at 650°C and subjected to creep-rupture tests at 650°C after irradiation. The results of these tests are given in Table 22. These samples were stressed at 32,400 psi for about 675 hr

Table 20. Creep Properties at 650°C of Several Alloys That Contain Various Concentrations of Zirconium[a]

Alloy Number	Specimen Number	Test Number	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
			$\times 10^3$				
100	1915	5725	55	3.4	0.43	22.5	20.8
100	1917	6099	47	20.8	0.55	14.5	12.5
100	1909	5554	40	47.4	0.088	12.5	9.5
100	1914	6108	32.4	185.1	0.0077	6.3	6.2
100	1921	6172	27	827.8	0.0022	4.9	4.0
108	2077	5689	55	14.1	0.16	17.5	14.8
108	2082	5991	47	178.7	0.025	18.8	15.9
108	2091	5721	40	348.5	0.018	25.0	21.4
108	2094	6162	32.4	2137.3	0.0059	31.3	28.4
109	2068	5778	70	30.7	0.128	32.4	28.3
109	2066	5712	55	178.1	0.015	32.0	31.0
109	2057	5740	51	358.5	0.013	31.0	36.0
109	2072	5987	47	851.2	0.0075	34.1	34.8
109	2065	5909	40	1474.3	0.0042	46.9	35.3
110	2115	5746	70	12.5	0.138	39.3	26.9
110	2104	5673	55	200.7	0.028	35.9	31.1
110	2117	6096	47	283.0	0.030	38.2	38.6
110	2112	5665	40	709.0	0.0080	18.8	25.6
111	2130	5771	70	26.9	0.174	35.0	26.9
111	2135	5688	55	192.8	0.027	30.9	29.7
111	2126	6091	47	589.4	0.014	36.6	33.7
112	2159	5752	70	39.2	0.181	38.2	31.6
112	2151	5908	62	169.5	0.038	34.3	27.2
112	2145	5666	55	537.4	0.011	30.5	26.9
112	2163	5641	51	673.1	0.010	37.5	28.8
112	2158	6166	47	1254.8	0.0056	32.0	29.9

[a]Annealed 1 hr at 1177°C before testing.

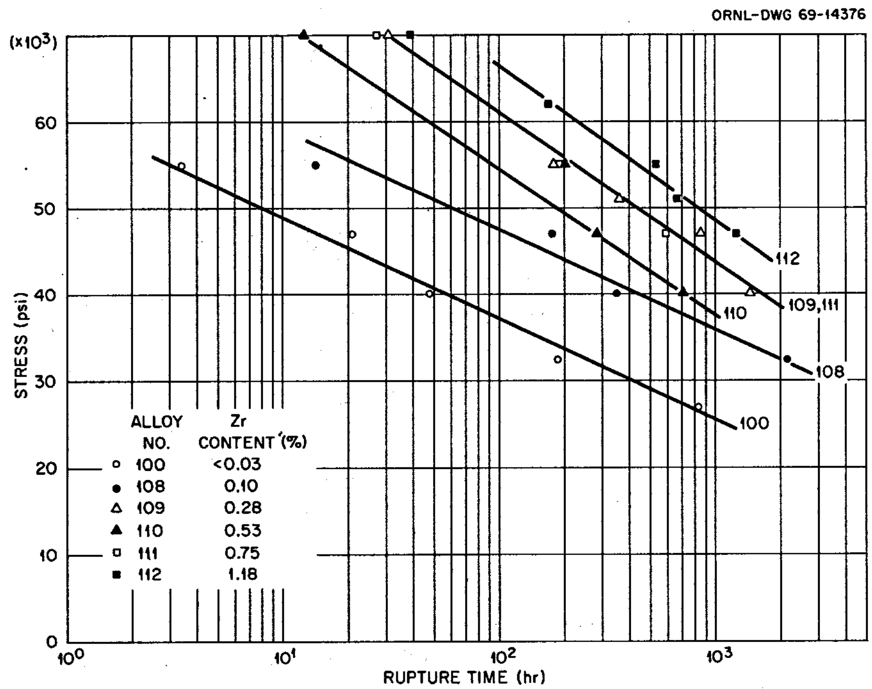


Fig. 44. Stress-Rupture Properties at 650°C of Several Alloys That Contain Various Concentrations of Zirconium. All material was annealed 1 hr at 1177°C before testing.

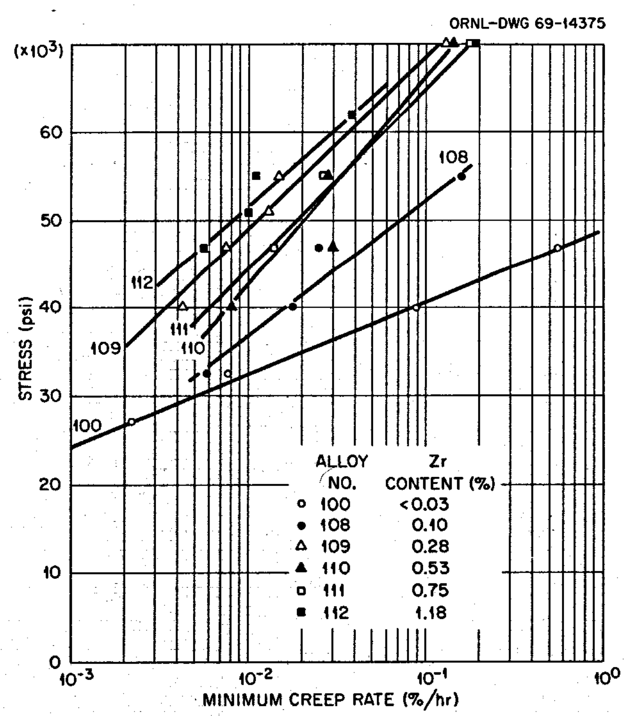


Fig. 45. Influence of Zirconium on Creep Rates at 650°C. All alloys annealed 1 hr at 1177°C before testing.

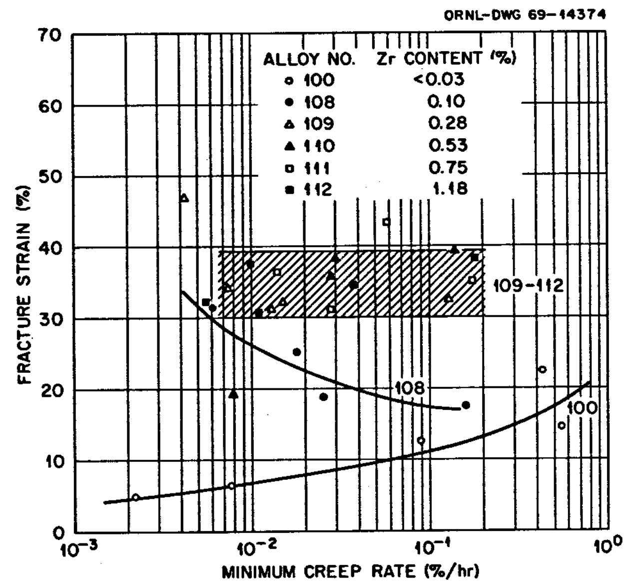


Fig. 46. Fracture Strains During Creep Tests at 650°C for Alloys That Contain Various Amounts of Zirconium. All samples annealed 1 hr at 1177°C before testing.

Table 21. Stress Properties at 650°C of Several Aged[a] Alloys That Contain Zirconium

Alloy Number	Specimen Number	Test Number	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
			$\times 10^3$				
100	1922	5723	55	5.9	0.61	23.0	19.2
100	1919	5630[b]	17.8	1007	0.0007	1.0	0
108	2090	5680	55	280.9	0.042	40.6	38.8
108	2085	5911[b]	32.4	1000	0.0096	15.0	9.8
109	2071	5727	55	152.7	0.068	38.4	44.7
109	2063	6092[b]	32.4	1000	0.0044	3.5	
110	2123	5682	55	197.7	0.098	37.5	35.0
110	2120	6086[b]	32.4	1002.2	0.0012		
111	2144	5674	55	305.5	0.052	29.7	28.3
111	2137	6144[b]	32.4	1002.2	0.0011	3.1	0.03
112	2154	5637	55	530.1	0.030	34.4	29.6
112	2149	5629[b]	32.4	1000.6	0.0016	3.1	0.01

[a]Annealed 1 hr at 1177°C and aged 1000 hr at 650°C before testing.

[b]Discontinued before failure.

Table 22. Creep Properties at 650°C of Irradiated[a] Alloys that Contain Various Amounts of Zirconium

Alloy Number	Specimen Number	Test Number	Zirconium Content (wt %)	Stress,[b] psi: 32,400		Stress,[b] psi: 40,000				
				Minimum Creep Rate (%/hr)	Elongation (%)	Minimum Creep Rate (%/hr)	Rupture Life (hr)	Elongation (%)	Reduction in Area (%)	True Fracture Strain (%)
100	1916	R-146	< 0.03	0.0099	1.1					
108	2076	R-163	0.10	0.0094	8.2	0.147	53.1	7.3	19.0	21.1
109	2070	R-171	0.28	0.0034	2.3	0.025	301.3	10.8	17.2	18.9
110	2121	R-172	0.53	0.0013	0.97	0.0094	429.5	5.9	11.1	11.7
111	2140	R-195	0.75	0.0037	3.3	0.035	127.1	5.7	13.9	15.0
112	2155	R-199	1.18	0.0033	5.1	0.080	46.5	3.9	6.8	7.0

[a]All samples annealed 1 hr at 1177°C before irradiation at 650°C to a thermal fluence of 2.5×10^{20} neutrons/cm^2.

[b]All tests except R-146 were initially stressed at 32,400 psi for about 675 hr and then stressed at 40,000 psi. Test R-146 failed while loaded at 32,400 psi after 105.4 hr.

and then stressed at 40,000 psi until failure. (This increase in stress level was necessary to cause failure in a reasonable time.) The rupture life at the higher stress is shown in Fig. 47 as a function of zirconium content. The strain during the first 675 hr at 32,400 psi is given in parentheses, and the strain at the higher stress level is shown by each

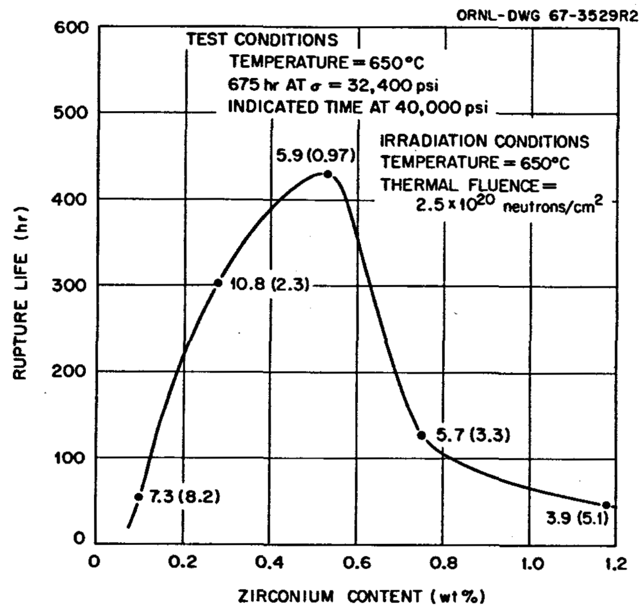


Fig. 47. Influence of Zirconium Content on the Creep-Rupture Properties at 650°C after Irradiation. All samples annealed at 1177°C before irradiation.

point. The rupture life increased rapidly with zirconium content up to a maximum for an alloy containing 0.53% Zr. The alloys containing less zirconium had better fracture strains under these test conditions; however, all alloys containing 0.1% Zr or more had excellent fracture strains after irradiation. The minimum creep rates for these same samples are given in Fig. 48. The creep rates at both stress levels exhibited a definite minimum for the alloy containing 0.53% Zr. The creep rates for the unirradiated control samples are also shown in Fig. 48 at the 32,400 psi stress level. The minimum creep rate was not affected detectably by irradiation except for the alloys containing 0.75 and 1.18% Zr.

A second irradiation experiment exposed samples to a thermal-neutron fluence of 9×10^{19} neutrons/cm^2 at 50°C. These results are summarized

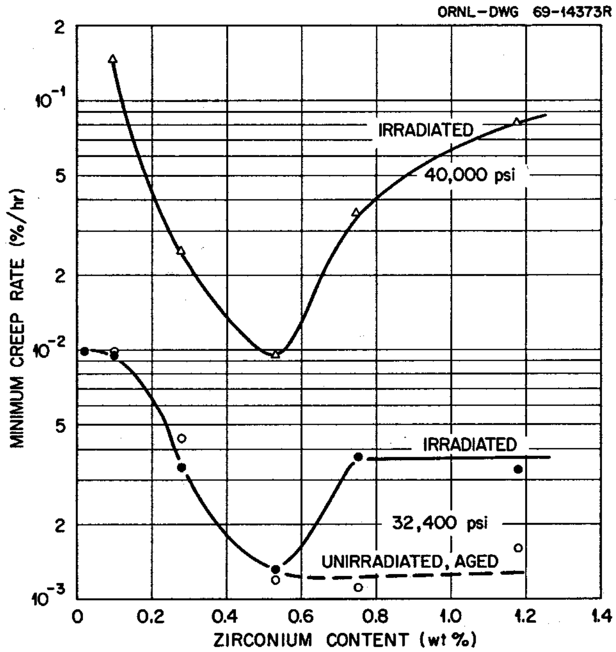


Fig. 48. Influence of Zirconium Content on the Creep Rates at 650°C. All samples annealed 1 hr at 1177°C before irradiation at 650°C.

in Table 23. Because of the different stress levels involved, the results are difficult to compare with those presented previously. However, the following points can be established by cross-plotting results:

Table 23. Creep Properties at 650°C of Irradiated[a] Alloys That Contain Various Amounts of Zirconium

Alloy Number	Specimen Number	Test Number	Zirconium Content (wt %)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)
				$\times 10^3$			
100	1911	R-257	< 0.03	40	68.9	0.019	1.79
108	2080	R-283	0.10	40	314.9	0.014	8.03
108				55[b]	5.1	0.96	5.05
109	2059	R-285	0.28	47	158.6	0.010	2.42
109				55[b]	43.0	0.116	5.26
110	2110	R-292	0.53	47	453.3	0.0063	7.52
111	2136	R-293	0.75	47	714.6	0.0076	15.5
112	2161	R-307	1.18	47	905.7	0.0028	5.32
112				55[b]	74.5	0.059	5.24

[a]Annealed 1 hr at 1177°C before irradiation at 50°C to a thermal fluence of 9×10^{19} neutrons/cm^2.

[b]Load increased during test.

(1) the rupture life increased and the creep rate decreased continuously with increasing zirconium (Table 23); (2) the rupture lives of these samples were greater than those of samples irradiated at 650°C (Table 22); (3) the minimum creep rates were lower than those observed before irradiation (Fig. 45); and (4) the fracture strains were all quite good.

Tests were run at 760°C on several samples; the results are shown in Table 24. For samples irradiated and tested at 760°C, the minimum creep rate decreased with increasing zirconium (Fig. 49). The fracture strain seemed to go through a maximum in alloys that contained 0.1 to 0.28% Zr. Test samples of alloy 108 (0.1% Zr) indicated that the irradiation temperature had very little effect when the test temperature was 760°C. A test sample of this same composition also indicated that annealing at 650°C after irradiation at 43°C had very little effect on the properties. A single sample of alloy 112 (1.18% Zr) was irradiated at 760°C and tested at 650°C. The rupture life at 30,000 psi was only 444.9 hr compared with more than 675 hr for a sample stressed at 32,400 psi after irradiation at 650°C (Table 22). However, the fracture strain was quite good for both samples.

Two other alloys were made that contained a nominal 1% Zr. These two alloys, designated 146 and 147, also contained 0.1 and 1% Re, respectively, an addition that was made in an attempt to improve the weldability. These alloys were creep tested in the irradiated and unirradiated conditions; the results are given in Tables 25 and 26, and the stress-rupture properties are shown graphically in Fig. 50. The stress-rupture properties of the unirradiated samples are about the same for both heats and quite similar to those shown in Fig. 44 for alloy 108 (1.18% Zr). Thus, rhenium does not appear to be a very influential addition; the properties are basically those of an alloy containing 1% Zr. The single test of an unirradiated sample of each heat that had been aged for 1000 hr at 760°C indicates an instability in this alloy. This anneal decreased the rupture life and increased the minimum creep rate. Irradiation at 650°C reduced the rupture life and fracture strain when samples were tested at 650°C (Fig. 50). Irradiation at a higher temperature further reduced rupture life, but the fracture strain remained reasonably high.

Table 24. Creep Properties of Irradiated[a] Alloys That Contain Various Amounts of Zirconium

Alloy Number	Specimen Number	Test Number	Zirconium Content (wt %)	Temperature, °C Irradiation	Temperature, °C Test	Neutron Fluence (neutrons/cm²)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)
						× 10^{20}	× 10^{3}			
100	3791	R-455	< 0.03	657	760	2.5	12.5	1.0	0.10	0.21
108	2089	R-284	0.10	43	760	0.9	15	468.1	0.037	36.1
108	2093	R-291[b]	0.10	43	760	0.9	15	380.9	0.049	28.3
108	3861	R-448	0.10	750	760	2.5	12.5	665.2	0.026	35.3
109	3871	R-456[c]	0.28	750	760	2.5	12.5	1009.6	0.0094	14.4
							15	310.4	0.062	23.8
							17.5	7.9	0.37	3.9
110	3881	R-447[c]	0.53	760	760	2.5	12.5	1007.4	0.0084	11.1
							15	262.4	0.014	3.8
							17.5	167.6	0.038	16.6
111	3891	R-459[c]	0.75	740	760	2.5	12.5	1296.3	0.0047	10.4
							17.5	308.6	0.043	19.2
112	3901	R-457[c]	1.18	740	760	2.5	12.5	1008.8	0.0013	2.5
							15	310.4	0.013	4.2
							17.5	218.4	0.041	12.4
112	3910	R-814	1.18	760	650	2.6	30	444.9	0.017	10.1

[a]Annealed 1 hr at 1177°C before irradiation.

[b]Annealed 1000 hr at 650°C after irradiation.

[c]Stress increased during test as indicated.

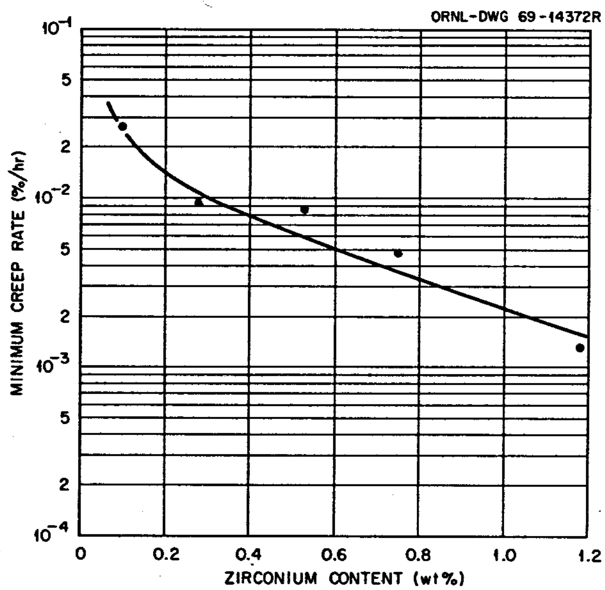


Fig. 49. Variation of the Minimum Creep Rate at 760°C and 12,500 psi with Zirconium Concentration. Samples annealed 1 hr at 1177°C and irradiated at 760°C to a thermal fluence of 2.5×10^{20} neutrons/cm^2.

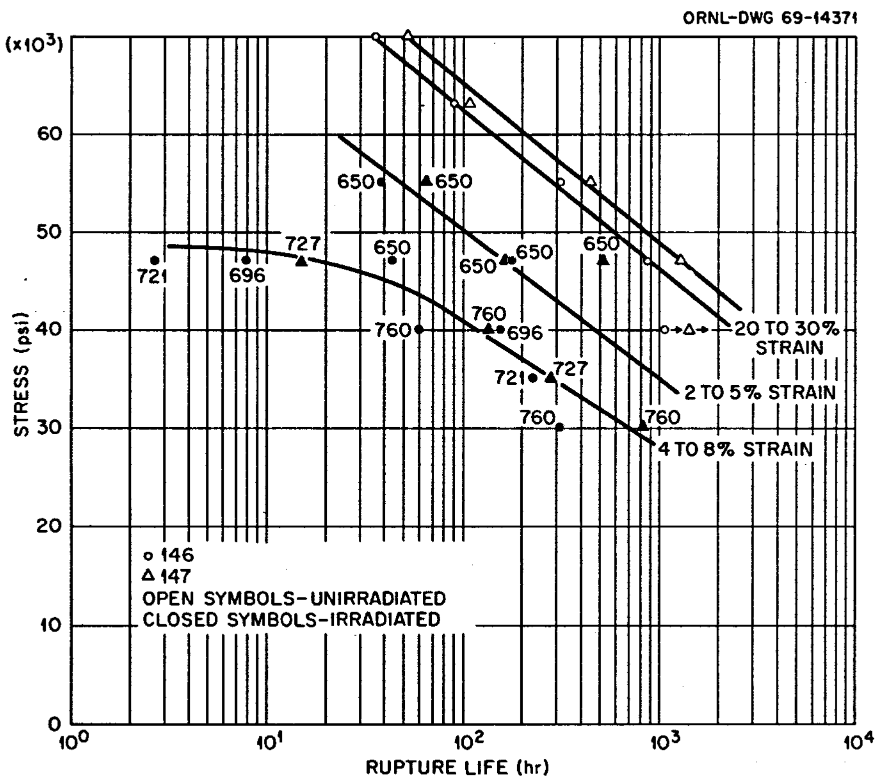


Fig. 50. Stress-Rupture Properties at 650°C of Alloys 146 and 147. All samples annealed 1 hr at 1177°C before testing. Numbers by each point designate the irradiation temperature.

Table 25. Creep Properties of Irradiated and Unirradiated Alloy 146

Specimen Number	Test Number	Anneal[a] Before Irradiation	Temperature, °C Irradiation	Temperature, °C Test	Thermal Neutron Fluence (neutrons/cm^2)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
					$\times 10^{20}$	$\times 10^3$				
3515	6647	121		650		70	36.1	0.113	29.6	24.7
3516	6648	121		650		63	90.1	0.037	24.9	22.2
3517	6649	121		650		55	311.3	0.012	20.1	20.6
3504	6136	121		650		47	865.5	0.0049	22.3	17.2
3503	6135	121		650		40	1003.2[b]	0.0023	5.4	2.4
3511	7769	148		650		47	110.5	0.16	20.9	15.8
3518	6650	121		760		25	352.2	0.016	32.2	32.8
3519	6651	121		760		20	984.1	0.0039	29.1	51.9
3509	R-678	121	650	650	2.6	55	38.4	0.043	2.8	
3510	R-656	121	650	650	2.6	47	177.4	0.021	4.9	
3508	R-648	121	650	650	2.6	47	42.7	0.046	2.3	
3522	R-915	121	696	650	2.6	47	7.9	0.20	1.8	
3523	R-916	121	721	650	2.6	47	2.7	1.0	3.0	
3526	R-928	121	696	650	2.6	40	153.5	0.024	4.9	
3512	R-801	121	760	650	2.6	40	59.0	0.071	4.8	
3528	R-935	121	721	650	2.6	35	226.3	0.022	7.2	
3513	R-830	121	760	650	2.6	30	306.3	0.016	5.4	
3506	R-552	121	760	760	2.4	15	380.1	0.0211	10.6	

[a]Annealing designations: 121 = annealed 1 hr at 1177°C,
148 = annealed 1 hr at 1177°C plus 1000 hr at 760°C.

[b]Test discontinued before failure.

Table 26. Creep Properties of Irradiated and Unirradiated Alloy 147

Specimen Number	Test Number	Anneal[a] Before Irradiation	Temperature, °C		Thermal Neutron Fluence (neutrons/cm^2)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
			Irradiation	Test						
					$\times 10^{20}$	$\times 10^{3}$				
4031	6652	121		650		70	51.8	0.069	26.2	20.0
4032	6653	121		650		63	106.2	0.038	23.3	16.6
4033	6654	121		650		55	461.1	0.10	22.1	17.8
4045	6116	121		650		47	1289.5	0.0054	25.3	21.5
4044	6117	121		650		40	1147.8[b]	0.0014	4.2	2.6
4038	7770	148		650		47	53.8	0.19	12.7	14.5
4034	6655	121		760		25	453.3	0.018	55.4	37.7
4035	6656	121		760		20	1240.1	0.0039	29.2	34.1
4028	R-679	121	650	650	2.6	55	65.2	0.0089	1.4	
4030	R-687	121	650	650	2.6	47	508.1	0.0068	4.8	
4027	R-647	121	650	650	2.6	47	164.2	0.0095	3.2	
4037	R-917	121	727	650	2.6	47	15.0	0.29	3.5	
4051	R-802	121	760	650	2.6	40	136.4	0.027	4.3	
4041	R-934	121	727	650	2.6	35	274.1	0.017	5.8	
4052	R-832	121	760	650	2.6	30	804.4	0.0078	8.2	
4049	R-553	121	685	760	2.4	15	1082.1	0.011	15.6	

[a]Annealing designations: 121 = annealed 1 hr at 1177°C,
148 = annealed 1 hr at 1177°C plus 1000 hr at 760°C.

[b]Test discontinued before failure.

The two 100-lb commercial melts, heat 21555 (0.05% Zr) and 21554 (0.35% Zr) were evaluated in both tensile and creep tests. The tensile results are shown in Tables 27 and 28. The tests on heat 21555 after irradiation showed that the coarse-grained material had superior ductility and that the fine-grained material had higher yield strength. The tensile results for heat 21554 (0.35% Zr) are summarized in Table 28, and the fracture strain for the fine-grained material is shown in Fig. 51. The behavior is similar to that observed for standard Hastelloy N, but at 760°C the ductility of the zirconium-modified alloy was much lower. The strong dependence of fracture strain on strain rate at 760°C was also quite unusual where the fracture strain was higher at the lower strain rate. After irradiation, the fracture strain of the fine-grained material was generally reduced, and the reduction was larger at higher test temperatures (Fig. 52). The ratios of the various properties in the irradiated and unirradiated alloys are shown in Fig. 53. The yield and ultimate tensile strengths were not affected appreciably by irradiation. The elongation remained high with increasing temperature and dropped at test temperatures above 600°C. The high ratio at 760°C is due to the very low fracture strain of the unirradiated sample. The reduction in area follows the normal pattern for standard Hastelloy N.

Further analysis of the results in Table 28 shows the effects of various annealing treatments. Several samples were annealed for 100 hr at 871°C followed by 1000 hr at 650°C. The strengths were lower and the fracture strains higher than those of the samples held at 650°C for a much longer time. Two samples were annealed for 1 hr at 1177°C and then for 1000 hr at 760°C. This treatment gave good strength and ductility characteristics. Some samples were irradiated that had heat treatments other than those already discussed. The strains after irradiation were higher for samples annealed for 1 hr at 1177°C than for those annealed at 871°C.

Irradiated and unirradiated samples of heats 21555 (0.05% Zr) and 21554 (0.35% Zr) were tested in creep. The creep results at 650°C for heat 21555 are given in Tables 29 and 30, and the stress-rupture properties are shown in Fig. 54. In the unirradiated conditions, the optimum rupture life was obtained after a 1-hr anneal at 1177°C. Further aging

Table 27. Tensile Properties of Irradiated and Unirradiated Alloy 21555 (0.05% Zr)

Specimen Number	Anneal[a] Before Irradiation	Temperature, °C		Thermal Neutron Fluence (neutrons/cm^2)	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
		Irradiation	Test			Yield	Ultimate Tensile	Uniform	Total	
				$\times 10^{20}$		$\times 10^3$	$\times 10^3$			
2488	36		600		0.002	24.4	4.1	50.2	52.5	37.6
2493	36		650		0.05	24.4	74.2	55.0	59.0	40.1
2494	36		650		0.002	25.2	66.8	33.0	42.6	35.2
2496	36		700		0.002	22.8	54.6	20.2	40.8	41.3
2478	148		25		0.05	43.9	116.1	54.3	56.9	44.2
2479	148		650		0.002	27.3	66.9	30.7	33.7	31.5
2480	121	540	650	2.5	0.05	26.6	63.6	43.3	45.0	33.8
2481	121	540	650	2.5	0.002	27.1	60	27.5	28.3	20.5
2464	16	540	650	2.5	0.05	36.8	60.2	11.3	11.9	12.6
2465	16	540	650	2.5	0.002	37.7	48.2	5.8	6.0	7.9

[a]Annealing designations: 36 = 100 hr at 871°C plus 1000 hr at 650°C,
148 = 1 hr at 1177°C plus 1000 hr at 760°C,
121 = 1 hr at 1177°C,
16 = 100 hr at 871°C.

Table 28. Tensile Properties of Irradiated and Unirradiated Alloy 21554 (0.35% Zr)

Specimen Number	Anneal[a] Before Irra-diation	Temperature, °C Irradiation	Test	Thermal-Neutron Fluence (neutrons/cm²)	Strain Rate (min⁻¹)	Stress, psi Yield	Ultimate Tensile	Elongation, % Uniform	Total	Reduction in Area (%)
				× 10^{20}		× 10^3	× 10^3			
5543	104		25		0.05	67.7	128.7	42.3	47.0	56.33
5544	104		200		0.05	60	115.9	39.9	42.9	59.80
5545	104		400		0.05	54.7	109.2	40.7	44.4	46.82
5553	104		400		0.002	54.2	111.8	45.8	48.3	53.59
5546	104		450		0.05	53	107.2	41.3	44.3	54.56
5554	104		450		0.002	55.9	110.5	42.7	48.1	48.26
5547	104		500		0.05	51.4	106.9	42.5	44.8	49.79
5555	104		500		0.002	58.6	111.4	39.9	45.3	43.69
5548	104		550		0.05	58.7	106.9	38.7	43.2	42.73
5556	104		550		0.002	59.8	101.3	26.4	28.0	24.70
5549	104		600		0.05	53.4	102	37.0	30.3	31.44
5557	104		600		0.002	56.8	91	22.7	25.0	24.18
5550	104		650		0.05	46.4	87.5	27.6	30.4	31.88
5558	104		650		0.002	55.8	75.5	13.3	46.6	63.15
5551	104		760		0.05	43.7	59	4.0	8.1	64.27
5559	104		760		0.002	37.9	37.9	1.6	47.4	69.45
5552	104		850		0.05	36.9	37.7	7.5	56.5	69.70
5560	104		850		0.002	22	22	100.0	46.9	60.94
10094	121		25		0.05	37.5	102.2	72.6	76.3	62.6
10095	121		200		0.05	33.2	100.1	64.0	65.3	46.3
10096	121		400		0.05	29.8	96.8	71.8	77.8	61.8
10097	121		500		0.05	25.6	84.4	79.9	83.7	50.7
10098	121		550		0.05	26.9	89.8	75.8	78.3	54.5
10099	121		600		0.05	27.4	89.1	74.3	78.0	49.6
10103	121		600		0.002	26.2	81	55.7	57.1	47.2
10100	121		650		0.05	25.7	81.2	65.8	68.5	44.1
10104	121		650		0.002	25.9	72.2	45.5	46.5	32.6
10101	121		700		0.05	25.8	72.5	47.0	48.7	38.9
10102	121		760		0.05	24.7	66	38.0	40.2	23.6
10105	121		760		0.002	24	45.5	11.5	33.5	29.9
2431	36		600		0.002	39.8	81	29.2	46.8	48.5
2436	36		650		0.05	26.5	71.3	51.6	60.6	42.5
2534	36		650		0.05	26	78.3	50.8	54.4	35.1
2437	36		650		0.002	27.2	69.1	43.5	49.6	31.4
2438	36		650		0.002	25.5	65.5	41.0	45.7	39.2
2439	36		700		0.002	25.2	56.7	18.7	34.4	31.2
10092	148		25		0.05	47.3	114.5	53.5	55.1	41.7
10093	148		650		0.002	30.1	68.9	37.3	47.3	42.9
9110	16	650	25	4.1	0.05	64.2	122.1	44.1	47.5	39.08
9111	16	650	200	4.1	0.05	61	116.1	43.7	46.0	51.90
9112	16	650	400	4.1	0.05	52.7	109.1	42.5	46.2	34.96
9131	16	650	400	4.1	0.002	64.6	112.6	39.5	45.2	36.26
9114	16	650	450	4.1	0.05	56	110.2	39.8	42.3	33.24
9132	16	650	450	4.1	0.002	57.4	107.9	41.8	45.7	33.94
9115	16	650	500	4.1	0.05	56.9	109.9	40.4	43.4	33.48
9113	16	650	500	4.1	0.002	50.5	119.7	43.4	45.6	25.54
9116	16	650	550	4.1	0.05	60.2	107.7	44.5	47.5	36.52
9121	16	650	550	4.1	0.002	43.6	91.7	22.5	23.0	21.15
9117	16	650	600	4.1	0.05	52.7	94.1	31.0	32.0	28.09
9122	16	650	600	4.1	0.002	50.3	80.3	15.7	16.8	18.42
9118	16	650	650	4.1	0.05					25.54
9123	16	650	650	4.1	0.002	48.1	65.6	10.9	11.2	12.39
9119	16	650	760	4.1	0.05	42.4	55.4	6.6	6.8	10.89
9124	16	650	760	4.1	0.002	37.6	37.9	1.7	5.4	4.74
9120	16	650	850	4.1	0.05	34.8	38.6	3.1	6.4	6.27
9125	16	650	850	4.1	0.002	3.8	12.1	5.1	10.4	13.88
2405	121	570	650	2.5	0.05	28.6	63.1	43.0	48.8	31.9
2406	121	575	650	2.5	0.002	30.5	56.6	28.4	32.6	34.0
2395	16	550	650	2.5	0.05	36.3	60.2	12.0	12.4	14.3
2396	16	550	650	2.5	0.002	38.7	50.9	6.3	6.5	8.8

[a]Annealing designations: 16 = 100 hr at 871°C,
36 = 100 hr at 871°C plus 1000 hr at 650°C,
148 = 1 hr at 1177°C plus 1000 hr at 760°C,
121 = 1 hr at 1177°C,
104 = 100 hr at 871°C plus 5600 hr at 650°C.

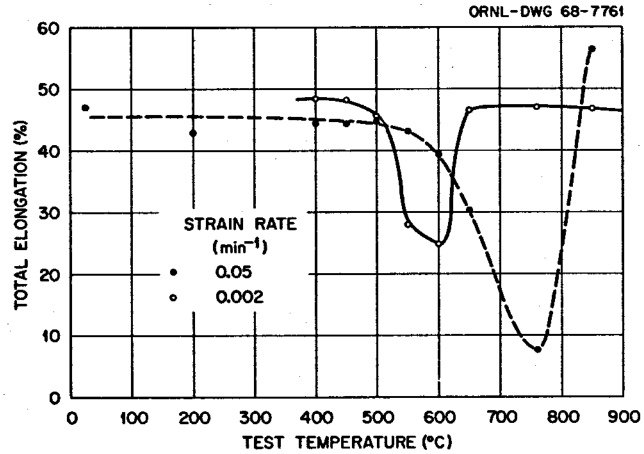


Fig. 51. Variation of Ductility with Temperature for Control Specimens of Zirconium-Modified Hastelloy N (Heat 21554). Annealed 100 hr at 871°C and aged 5600 hr at 650°C in barren salt.

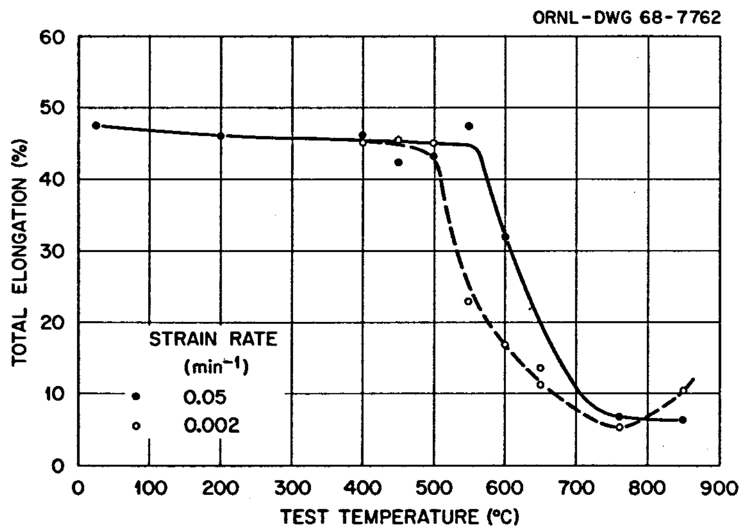


Fig. 52. Variation of Ductility with Temperature for Molten Salt Reactor Experiment Surveillance Specimens of Zirconium-Modified Hastelloy N (Heat 21554). Thermal fluence was 4.1×10^{20} neutrons/cm^2. Samples annealed 100 hr at 871°C before irradiation over a period of 5600 hr at 650°C.

Table 29. Creep Properties of Unirradiated Alloy 21555 (0.05% Zr)

Specimen Number	Test Number	Anneal[a] Before Test	Test Temperature (°C)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
				$\times 10^3$				
2500	6312	121	650	70	3.5	1.49	37.3	36.1
2506	6313	121	650	62	17.0	0.39	32.5	35.3
2506	6314	121	650	55	62.5	0.12	28.1	26.1
2507	6315	121	650	47	231.3	0.026	39.7	33.2
2501	6316	121	650	40	451.5	0.0085	34.7	32.0
2509	6317	121	650	35	1115.6	0.0065	31.8	32.8
2498	5863	37	650	40	511.2	0.0153	41.5	37.7
2497	6163	37	650	32.4	1673.8	0.0050	34.1	23.1
2461	7669	148	650	62	3.4	3.59	31.4	38.3
2459	7492	148	650	47	77.1	0.24	33.8	37.8
2460	7668	148	650	40	272.9	0.062	29.8	37.5
2492	5859	36	650	47	77.9	0.34	60.0	56.3
2490	6164	36	650	32.4	1028.9	0.028	65.9	51.0
2505	7282	121	760	30	14.4	0.64	38.4	39.1
2499	6357	121	760	20	183.8	0.087	34.6	27.1
2512	7281	121	760	15	527.3	0.023	26.8	26.9

[a]Annealing designations: 121 = annealed 1 hr at 1177°C,
37 = annealed 1 hr at 1177°C plus 1000 hr at 650°C,
148 = annealed 1 hr at 1177°C plus 1000 hr at 760°C,
36 = annealed 100 hr at 871°C plus 1000 hr at 650°C.

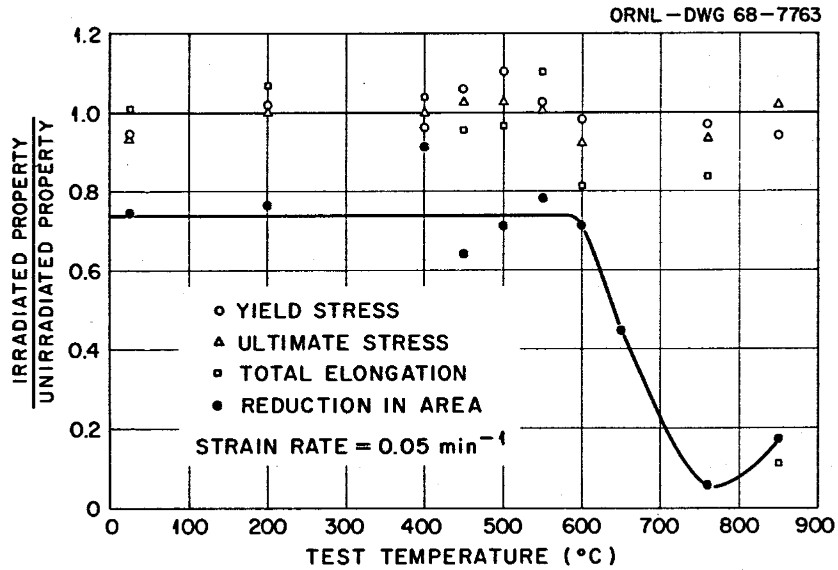


Fig. 53. Comparison of the Tensile Properties of Irradiated and Unirradiated Zirconium-Modified Hastelloy N (Heat 21554). Thermal fluence was 4.1×10^{20} neutrons/cm^2. Annealed 100 hr at 871°C and aged or irradiated over a period of 5600 hr at 650°C. Tested at a strain rate of 0.05 min^{-1}.

Table 30. Creep Properties of Irradiated[a] Alloy 21555 (0.05% Zr)

Specimen Number	Test Number	Anneal[b] Before Irradiation	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)
			$\times 10^3$			
			Test Temperature 650°C			
2483	R-381	121	47	106.4	0.032	6.7
2482	R-259	121	40	410.9	0.0089	10.0
2467	R-349	16	47	0		
2466	R-268	16	40	20.0	0.11	2.6
2469	R-350	16	35	45.7	0.032	3.0
			Test Temperature 760°C			
2485	R-227	121	15	110.1	0.036	7.2
2484	R-355	121	12.5	349.5	0.0095	7.7
2472	R-237	16	15	34.2	0.0096	5.0
2471	R-351	16	12	123.1	0.027	6.8

[a]Irradiated at 540°C to a thermal neutron fluence of 2.5×10^{20} neutrons/cm^2.

[b]Annealing designations: 121 = annealed 1 hr at 1177°C,
16 = annealed 100 hr at 871°C.

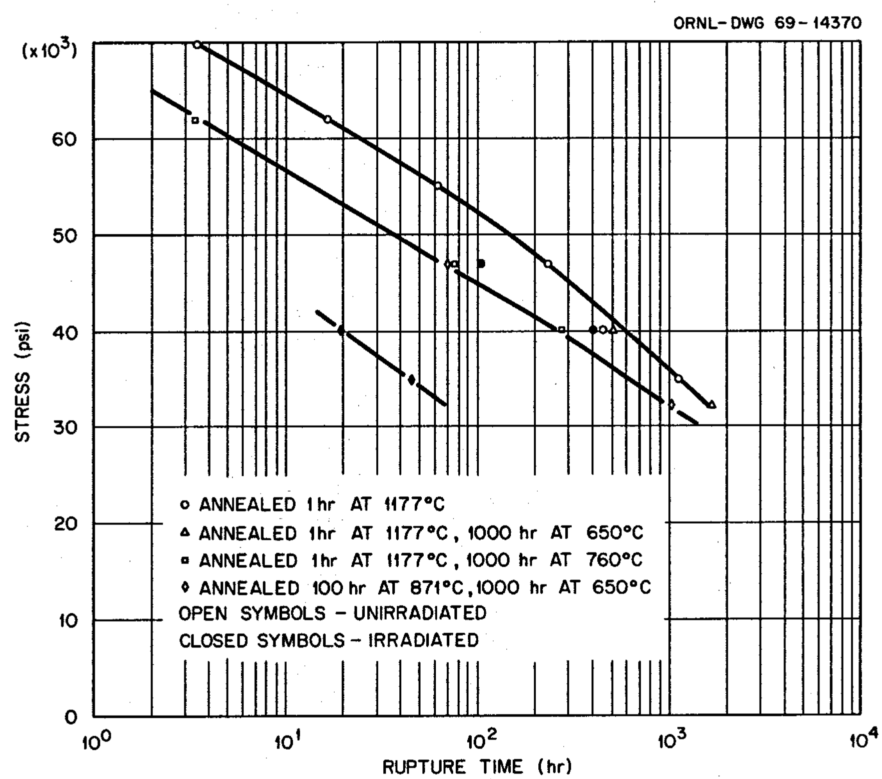


Fig. 54. Stress-Rupture Properties at 650°C of Alloy 21555 (0.05% Zr).

for 1000 hr at 650°C produced no detectable effect, but aging for 1000 hr at 760°C decreased the time to failure. Annealing at 871°C to produce a fine grain size resulted in shorter rupture lives. Irradiated samples had been annealed at 1177 and 871°C before irradiation. As shown in Fig. 54, irradiation reduced the rupture life of the larger grained material by a factor of about 2 and that of the finer grained material by a factor of about 10.

The minimum creep rate of heat 21555, shown in Fig. 55, was not affected appreciably by irradiation. The lowest minimum creep rate was obtained by annealing at 1177°C; aging at 650°C had no effect; aging at 760°C increased the creep rate; and annealing at 871°C to give a small grain size produced a high creep rate.

The results of creep tests on heat 21554 (0.35% Zr) are given in Tables 31 and 32. The stress-rupture properties at 650°C are shown in Fig. 56. The same general trends with regard to heat treatment were observed for this heat and for the heat with lower zirconium content (heat 21555). The optimum rupture life occurred after a 1-hr anneal at

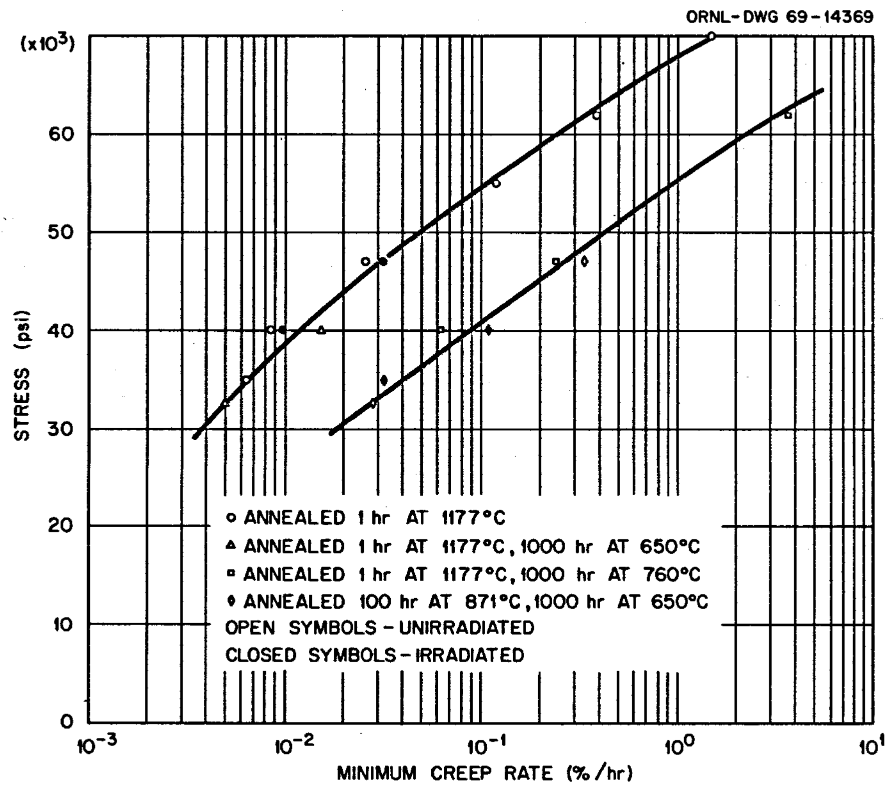


Fig. 55. Creep Rates at 650°C of Alloy 21555 (0.05% Zr).

Table 31. Creep Properties of Unirradiated Alloy 21554 (0.35% Zr)

Specimen Number	Test Number	Anneal[a] Before Test	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
			$\times 10^3$				
			Test Temperature 650°C				
2442	6306	121	70	7.3	1.38	49.4	43.8
2443	6307	121	62	32.0	0.20	38.1	43.1
2444	6308	121	55	94.4	0.062	31.6	26.7
2445	6309	121	47	395.2	0.018	26.8	27.7
2446	6310	121	40	803.8	0.0071	19.7	28.0
2441	5913	37	40	627.7	1.38	20.5	12.8
10086	7667	148	62	5.5	2.45	54.9	41.8
10084	7491	148	47	157.5	0.14	46.0	44.0
10085	7666	148	40	597.4	0.0313	36.5	33.4
2435	5862	36	47	128.3	0.20	63.0	50.6
2491	5858	36	40	226.3	0.13	60.8	50.9
2434	5857	36	40	324.2	0.056	35.5	51.9
2433	6168	36	32.4	1390.9	0.018	55.1	42.4
5535	6399	104	70	3.3	8.38	60.6	48.8
5536	6398	104	63	8.3	3.20	50.0	50.1
5537	6397	104	55	28.2	1.01	55.8	45.0
5538	6389	104	47	58.1	0.37	38.3	58.3
5539	6390	104	40	228.7	0.12	69.5	56.6
5540	6425	104	47	84.1	0.36	75.1	56.0
5541	6426	104	40	244.0	0.11	63.1	50.0
5542	6427	104	32.4	709.8	0.032	56.1	50.8
			Test Temperature 760°C				
2421	7286	121	30	35.6	0.33	30.3	25.9
2415	6356	121	20	354.1	0.047	32.9	30.7
2427	7285	121	15	1073.0	0.0123	28.7	31.3
2404	6056	16	20	85.0	0.35	61.7	45.0

[a]Annealing designations: 121 = annealed 1 hr at 1177°C,
37 = annealed 1 hr at 1177°C plus 1000 hr at 650°C,
148 = annealed 1 hr at 1177°C plus 1000 hr at 760°C,
36 = annealed 100 hr at 871°C plus 1000 hr at 650°C,
104 = annealed 100 hr at 871°C plus 5600 hr at 650°C,
16 = annealed 100 hr at 871°C.

Table 32. Creep Properties of Irradiated Alloy 21554 (0.35% Zr)

Specimen Number	Test Number	Anneal[a] Before Irradiation	Irradiation Temperature (°C)	Thermal-Neutron Fluence (neutrons/cm²)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)
				$\times 10^{20}$	$\times 10^{3}$			
			Test Temperature 650°C					
2408	R-382	121	590	2.5	47	140.2	0.024	4.75
2424	R-479	121	650	2.5	47	111.3	0.034	5.05
2407	R-260	121	590	2.5	40	336.3	0.0036	2.50
2420	R-480	121	650	2.5	40	279.0	0.0032	12.6
2398	R-265	16	550	2.5	47	3.9	0.35	1.51
2397	R-269	16	550	2.5	40	76.7	0.051	4.74
2402	R-356	16	550	2.5	35	163.7	0.0068	3.43
9127	R-316	61	650	4.1	47	11.1	0.34	4.57
9126	R-311	61	650	4.1	40	19.3	0.14	3.99
9128	R-318	61	650	4.1	32.4	65.4	0.013	2.36
9129	R-322	61	650	4.1	27	204.9	0.0088	3.45
			Test Temperature 760°C					
2409	R-364	121	590	2.5	20	71.5	0.014	1.95
2410	R-239	121	590	2.5	15	264.1	0.0094	6.52
2412	R-470	121	760	2.5	15	9.1	0.092	1.24
2403	R-174	16	760	2.3	20	5.5	0.36	2.95
2401	R-236	16	566	2.5	15	13.3	0.10	2.62
2400	R-357	16	560	2.5	12.5	222.8	0.0092	5.62

[a]Annealing designations: 121 = annealed 1 hr at 1177°C; 16 = annealed 100 hr at 871°C; 61 = annealed 200 hr at 871°C.

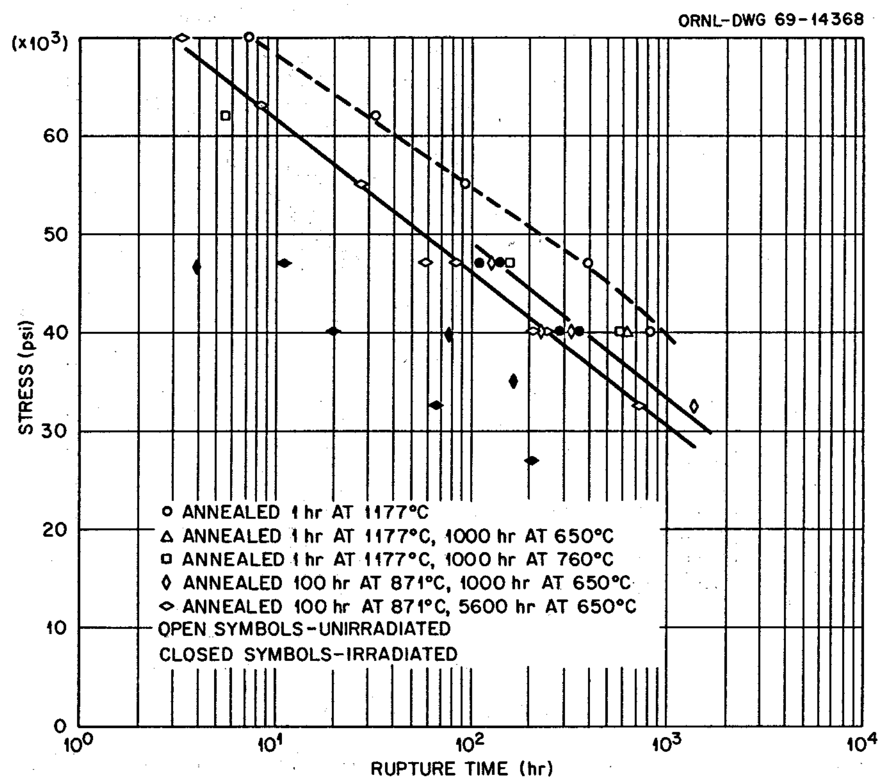


Fig. 56. Stress-Rupture Properties at 650°C of Alloy 21554 (0.35% Zr).

1177°C. Irradiation reduced the rupture life of the material annealed at 1177°C by a factor of 2 to 3 and that of the fine-grained material by a factor of about 10. The variation of the minimum creep rate with heat treatment and irradiation is shown in Fig. 57. The lowest minimum creep rate was observed for the material annealed at 1177°C. No effect of irradiation on the minimum creep rate was evident.

The fracture strains are shown as a function of minimum creep rate in Fig. 58 for material annealed at 1177°C and tested in the unirradiated condition at 650°C. The results for heat 21554 (0.35% Zr) show a clear trend of decreasing fracture strain with decreasing creep rate. The results for heat 21555 (0.05% Zr) show a similar trend, but the data seem to fall on two separate line segments. All of the fracture strains are quite high and fall largely in the range of 20 to 40%. The results in Tables 29 and 31 show that the samples of both alloys that were annealed at 871°C to give a fine grain size had fracture strains of 40 to 60%.

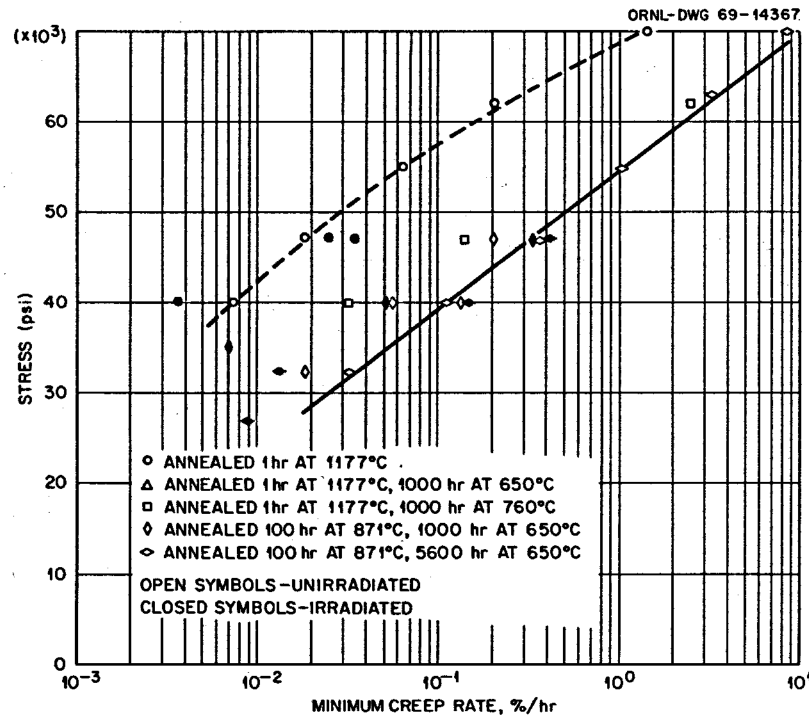


Fig. 57. Creep Properties at 650°C of Alloy 21554 (0.35% Zr).

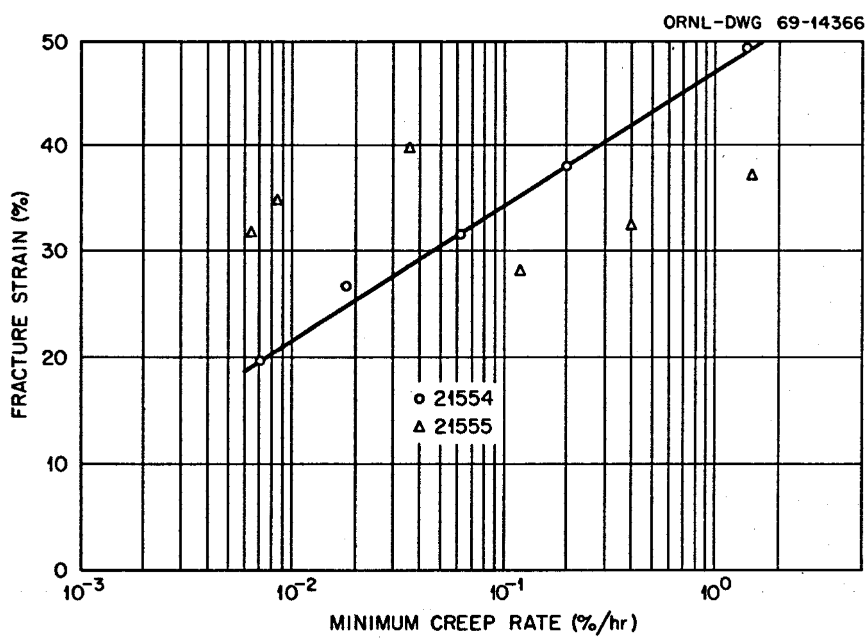


Fig. 58. Fracture Strains at 650°C of Alloys 21554 (0.35% Zr) and 21555 (0.05% Zr). All samples annealed 1 hr at 1177°C before testing.

The fracture strains of irradiated samples of heats 21554 and 21555 are shown in Fig. 59 as a function of strain rate. The results generally follow a trend of increasing fracture strain with decreasing creep rate, although one point definitely deviates from this trend.

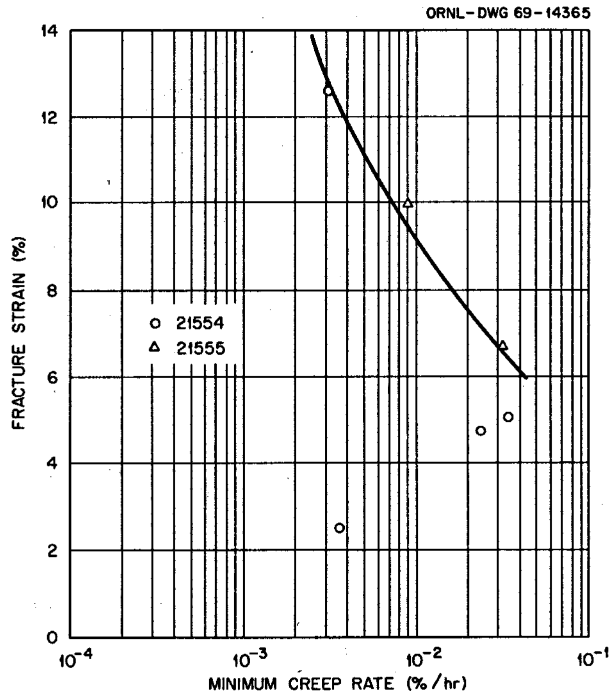


Fig. 59. Fracture Strains at 650°C of Alloys 21554 (0.35% Zr) and 21555 (0.05% Zr) after Irradiation. All samples annealed 1 hr at 1177°C before irradiation.

The results for a test temperature of 760°C are fragmentary (Tables 29 through 32). The stress-rupture characteristics are shown in Figs. 60 and 61 for heats 21555 (0.05% Zr) and 21554 (0.05% Zr). Rupture life was reduced by irradiation, but the samples annealed at 1177°C before irradiation had longer rupture lives than those annealed at 871°C. The fracture strains were reduced by irradiation, and the results for heat 21554 (Table 32) indicate that the reduction was greater as the irradiation temperature increased.

Several tested samples were examined metallographically. Typical photomicrographs of several alloys that contained zirconium are shown in Fig. 62. These samples were annealed 1 hr at 1177°C, aged 1000 hr

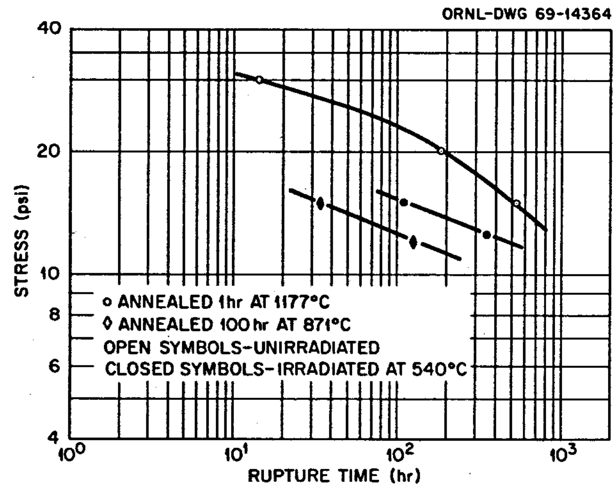


Fig. 60. Stress-Rupture Properties of Alloy 21555 (0.05% Zr) at 760°C.

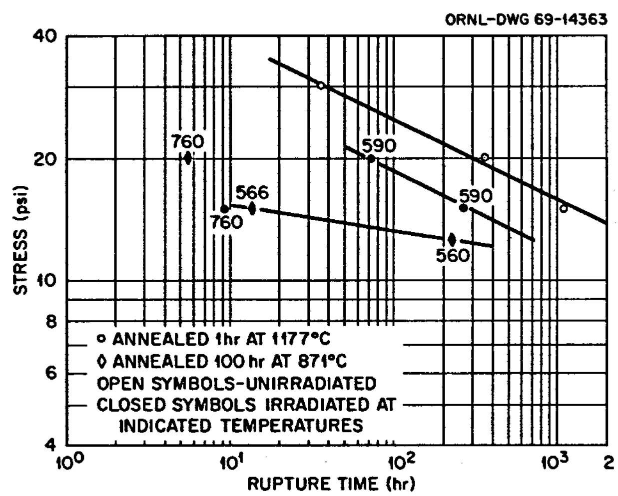


Fig. 61. Stress-Rupture Properties of Alloy 21554 (0.35% Zr) at 760°C.

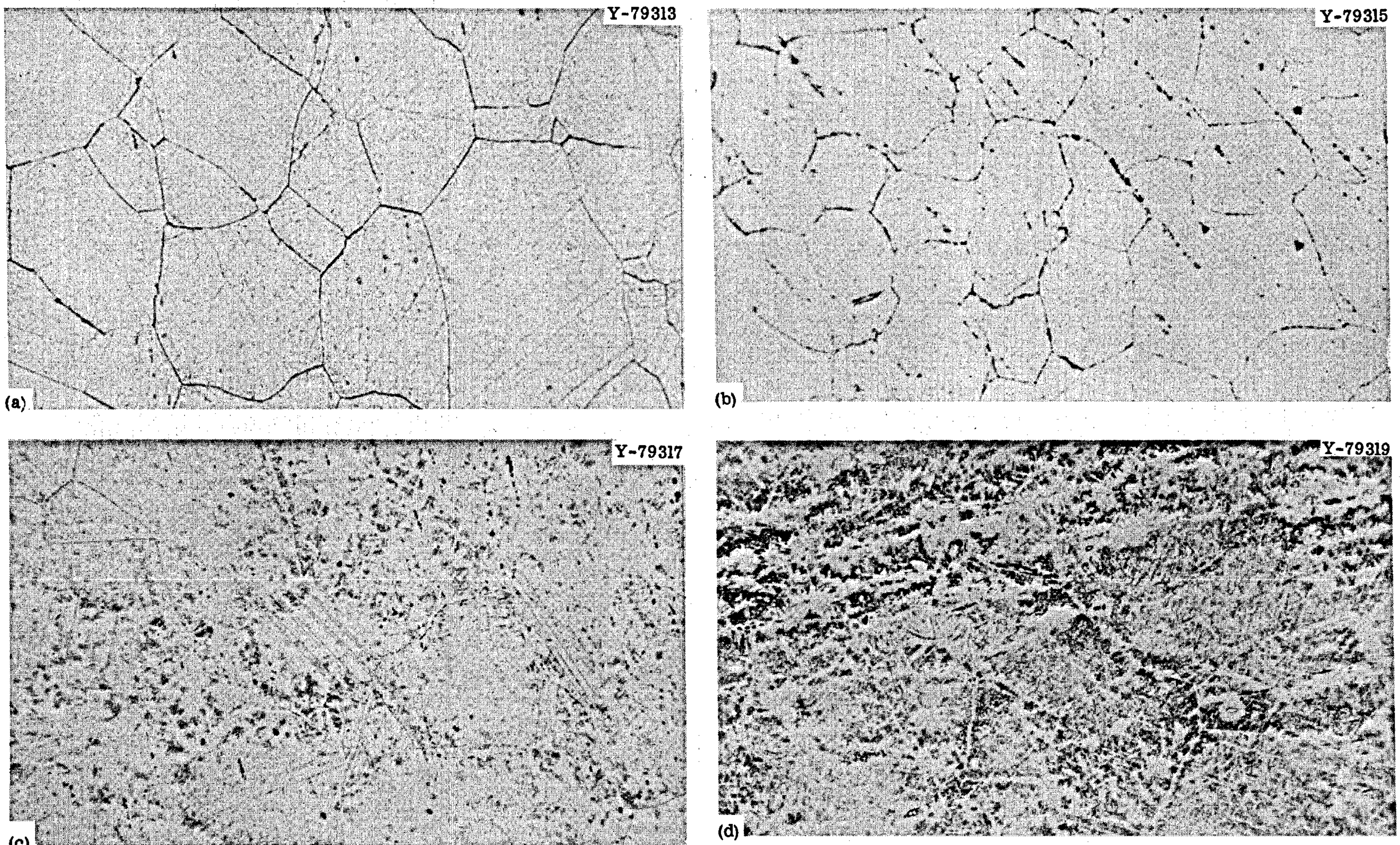


Fig. 62. Photomicrographs of Zirconium-Modified Alloys Annealed for 1 hr at 1177°C, Aged for 1000 hr at 650°C, and Stressed at 32,400 psi for 1000 hr. 500×. (a) Alloy 108 (0.10% Zr), (b) alloy 109 (0.28% Zr), (c) alloy 110 (0.53% Zr), and (d) alloy 111 (0.75% Zr). Etchant: glyceria regia. Reduced 26%.

at 650°C, and stressed for 1000 hr at 650°C. Alloy 108 (0.10% Zr) had a very fine carbide precipitate typical of that observed in alloy 100 (Fig. 24), which did not contain zirconium. In alloy 109 (0.28% Zr) the precipitate was coarser and located predominantly along the grain boundaries. Alloys 110 (0.53% Zr) and 111 (0.75% Zr) contained a very fine precipitate that could not be resolved at a magnification of 500×. Alloy 112 (1.18% Zr) had an even higher concentration of precipitate (Fig. 63). Figure 63(c) shows that the precipitate was acicular. The grain boundaries had a denuded zone, and there was a denuded region associated with the oxide at the surface. The precipitate was extracted and found by x-ray diffraction to have a lattice parameter of 4.68 A, equal to that for zirconium carbide. We also strained some samples of alloy 112 at 25°C after they had been aged at 650°C to produce the precipitate. The fracture strains were in excess of 50%, thus indicating that this microstructure was not brittle.

Some of the aged samples were tensile tested at 650°C (Table 17). The fracture of alloy 112 after testing at 650°C at a strain rate of 0.002 min^{-1} is shown in Fig. 64. The fracture strain was 23.3%, and the fracture was transgranular.

The fracture of alloy 110 (0.53% Zr), which was tested in creep at 650°C, is shown in Fig. 65. This alloy was at temperature 709 hr, and small quantities of precipitate are present in the microstructure. This precipitate cannot be resolved at low magnification and appears as dark spots. There are numerous intergranular cracks, and the fracture is intergranular. Typical photomicrographs of alloy 112 (1.18% Zr) after creep testing at 650°C are shown in Fig. 66. This sample was at temperature 1255 hr, and large quantities of precipitate are present in the microstructure. There are numerous intergranular cracks, and the fracture has both intergranular and transgranular components.

Typical fractures of alloy 21555 (0.05% Zr) after testing at 650°C and 32,400 psi are shown in Fig. 67. When this alloy was annealed 100 hr at 650°C before testing, the grain size was quite small. This sample was strained 65.9% before it fractured (Table 29), predominantly transgranularly [Fig. 67(a)]. Annealing for 1 hr at 1177°C and testing in creep resulted in the microstructure shown in Fig. 67(b). The

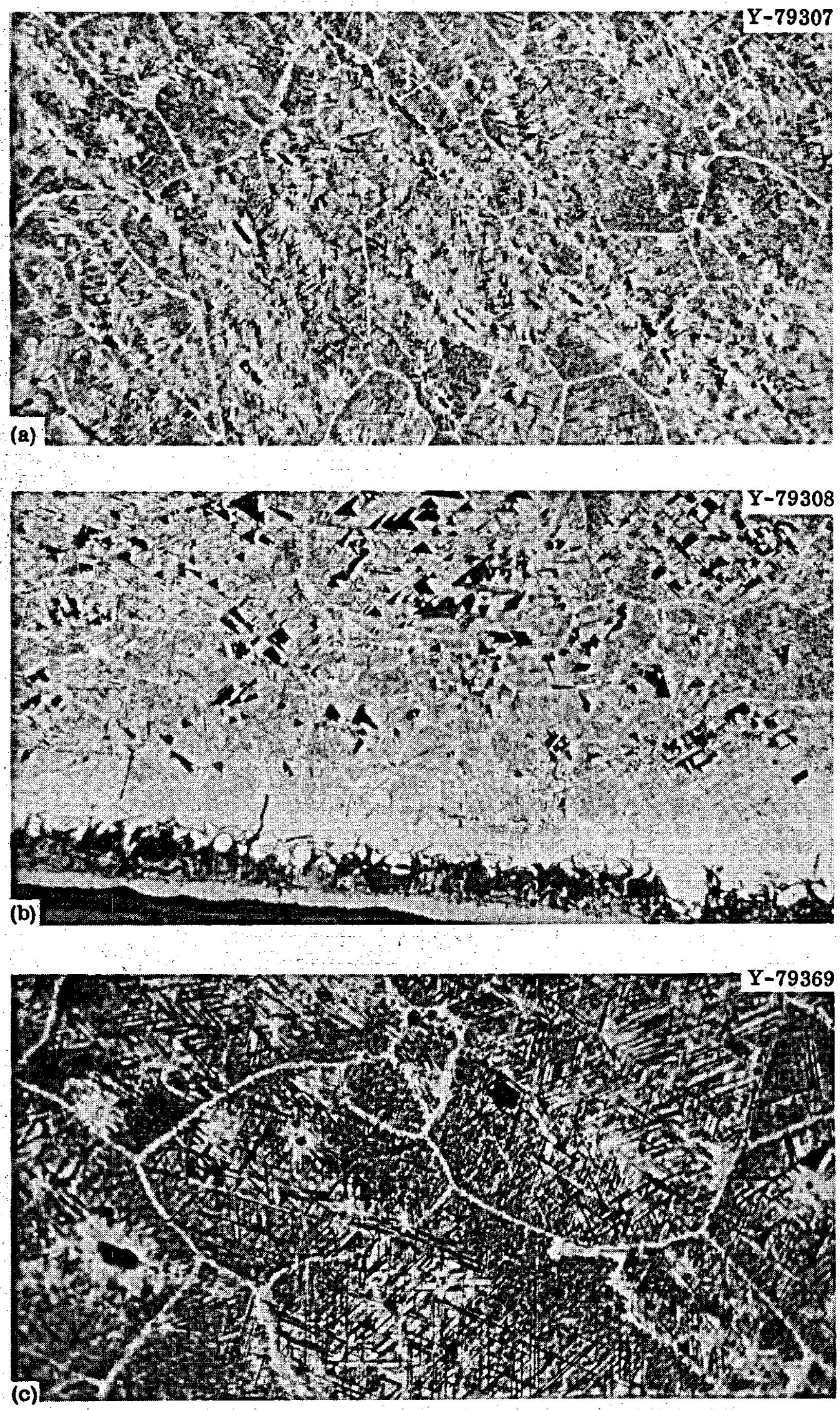
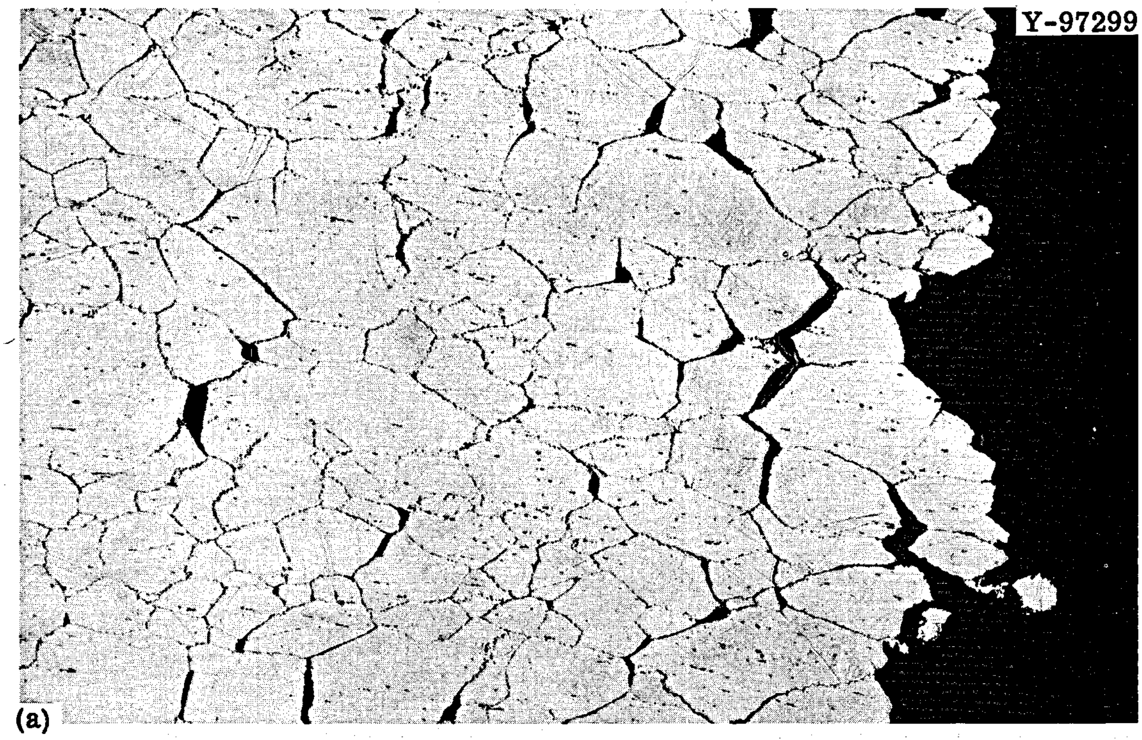


Fig. 63. Photomicrographs of Zirconium-Modified Alloy 112 (1.18% Zr) Annealed for 1 hr at 1177°C, Aged for 1000 hr at 650°C, and Stressed at 32,400 psi for 1000 hr. (a) Typical, 500×; (b) edge of sample, 500×; and (c) typical, 1000×. Etchant: glyceria regia. Reduced 23%.

Fig. 64. Photomicrographs of Alloy 112 (1.18% Zr) Annealed for 1 hr at 1177°C, Aged for 1000 hr at 650°C, and Tensile Tested at 650°C at a Strain Rate of 0.002 min^{-1}. (a) Fracture, 100×; (b) typical, 500×.



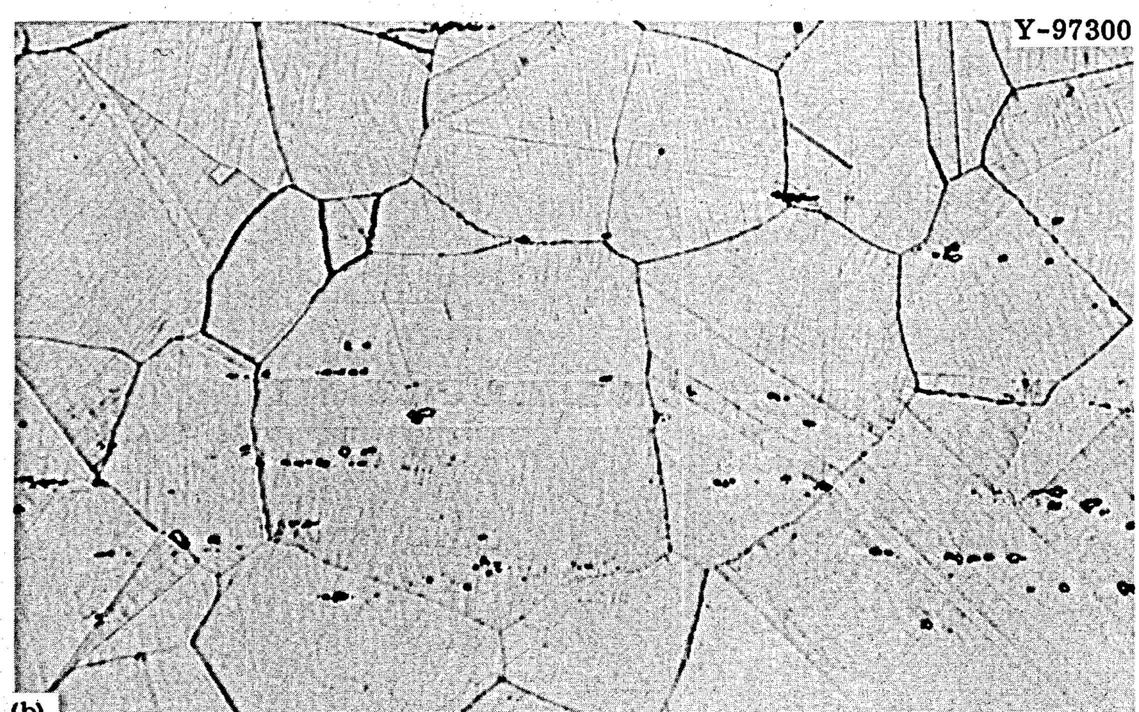


Fig. 65. Photomicrographs of Alloy 110 (0.53% Zr) Annealed 1 hr at 1177°C and Tested at 650°C and 40,000 psi. (a) Fracture, 100X; (b) typical, 500X. Etchant: glyceria regia.

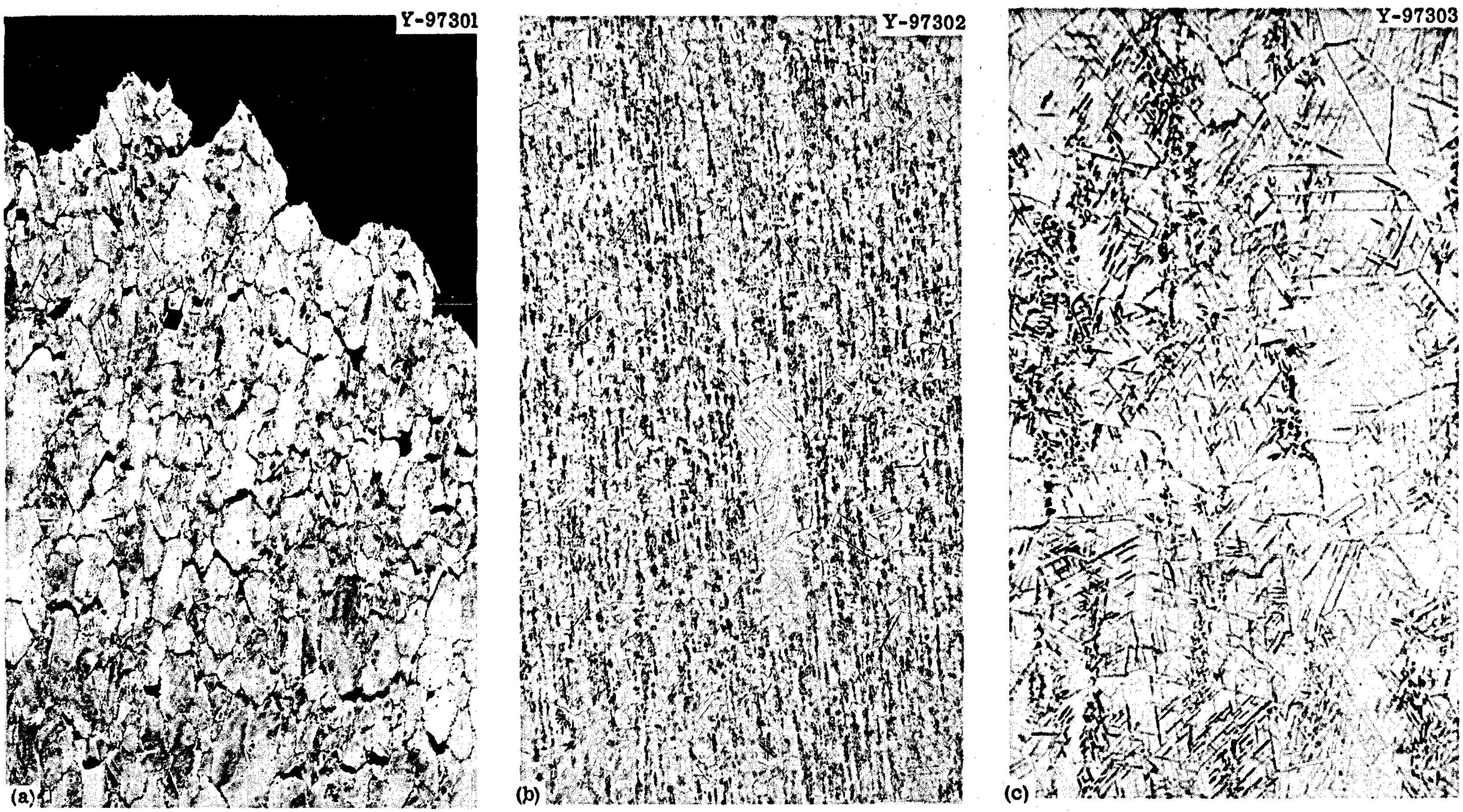


Fig. 66. Photomicrographs of Alloy 112 (1.18% Zr) Annealed 1 hr at 1177°C and Tested at 650°C and 47,000 psi. (a) Fracture, 100×; (b) typical, 100×, and (c) typical, 500×. Etchant: glyceria regia. Reduced 20%.

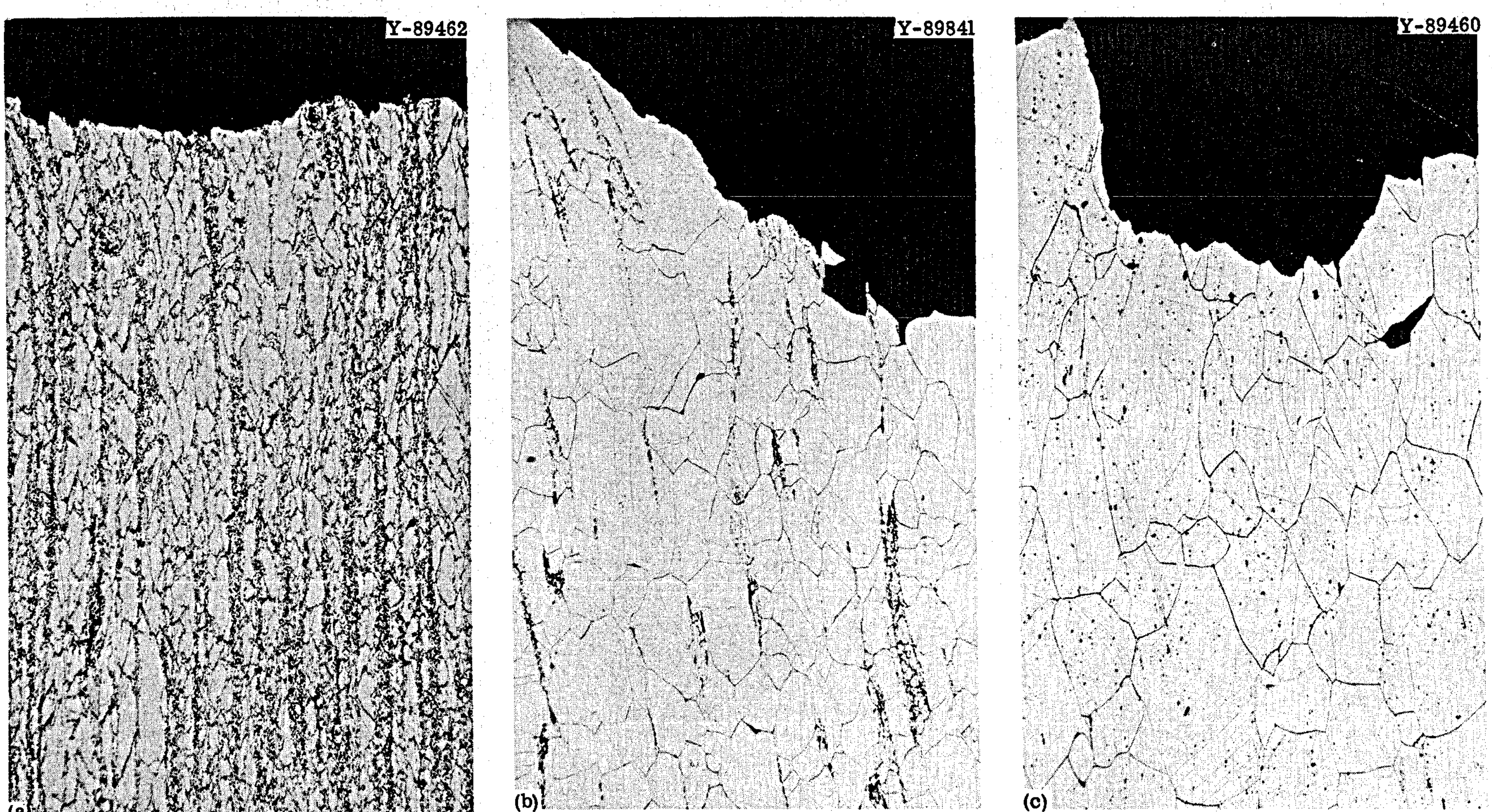


Fig. 67. Photomicrographs of Alloy 21555 (0.05% Zr) Creep Tested at 32,400 psi and 650°C. (a) Fracture of sample annealed for 100 hr at 871°C and 1000 hr at 650°C before test; (b) fracture of sample annealed 1 hr at 1177°C before test; and (c) fracture of sample annealed 1 hr at 1177°C and 1000 hr at 650°C. 100X. Etchant: glyceria regia. Reduced 21%.

fracture was still largely intergranular, and the fracture strain was 31.8%. Annealing at 1177°C and aging for 1000 hr at 650°C resulted in a fracture strain of 34.1% (Table 29). The microstructure contained only a few small precipitates, and the fracture was still largely transgranular. Alloy 21554 (0.35% Zr) was subjected to a similar annealing sequence. After annealing for 100 hr at 871°C and 1000 hr at 650°C and testing at 650°C the fracture shown in Fig. 68(a) was obtained. This alloy originally had stringers present (Fig. 38), and numerous cracks formed around these stringers during testing. The fracture was transgranular, and the fracture strain was 55.1% (Table 31). Annealing for 1 hr at 1177°C produced a coarser grain size. The fracture shown in Fig. 68(b) was mixed intergranular and transgranular, and the fracture strain was about 20%. The approximately 1000 hr at temperature during the creep test was sufficient to produce some precipitation. Another sample was annealed 1 hr at 1177°C and aged for 1000 hr before testing. This sample failed with about 20% strain. The fracture was mixed transgranular and intergranular [Fig. 68(a)]. Copious quantities of very fine precipitate formed.

Alloy 108 (0.10% Zr) was also examined metallographically after irradiation and creep testing. As shown in Fig. 69, a very fine precipitate was formed that was quite similar to that shown in Fig. 62 for an unirradiated sample. Alloy 110 (0.53% Zr) had more precipitate after irradiation than the control sample (compare Figs. 70 and 62). As shown in Fig. 70 there was a denuded zone along the boundaries and some banding of the precipitate, likely due to inhomogeneous distribution of zirconium in the alloy. The fracture was largely intergranular. Alloy 112 (1.18% Zr) had profuse quantities of the precipitate; the microstructure, however, was quite similar to that for the unirradiated sample (compare Figs. 71 and 63). The sample could not be etched heavily enough to show the grain boundaries without obliterating the precipitate. The fracture was mixed intergranular and transgranular.

Typical photomicrographs of alloy 21554 (0.35% Zr) are shown in Figs. 72 through 75. The sample shown in Fig. 72 was irradiated and tested at 25°C and failed transgranularly after straining 47.5%. The comparable control sample is shown in Fig. 73. This sample failed

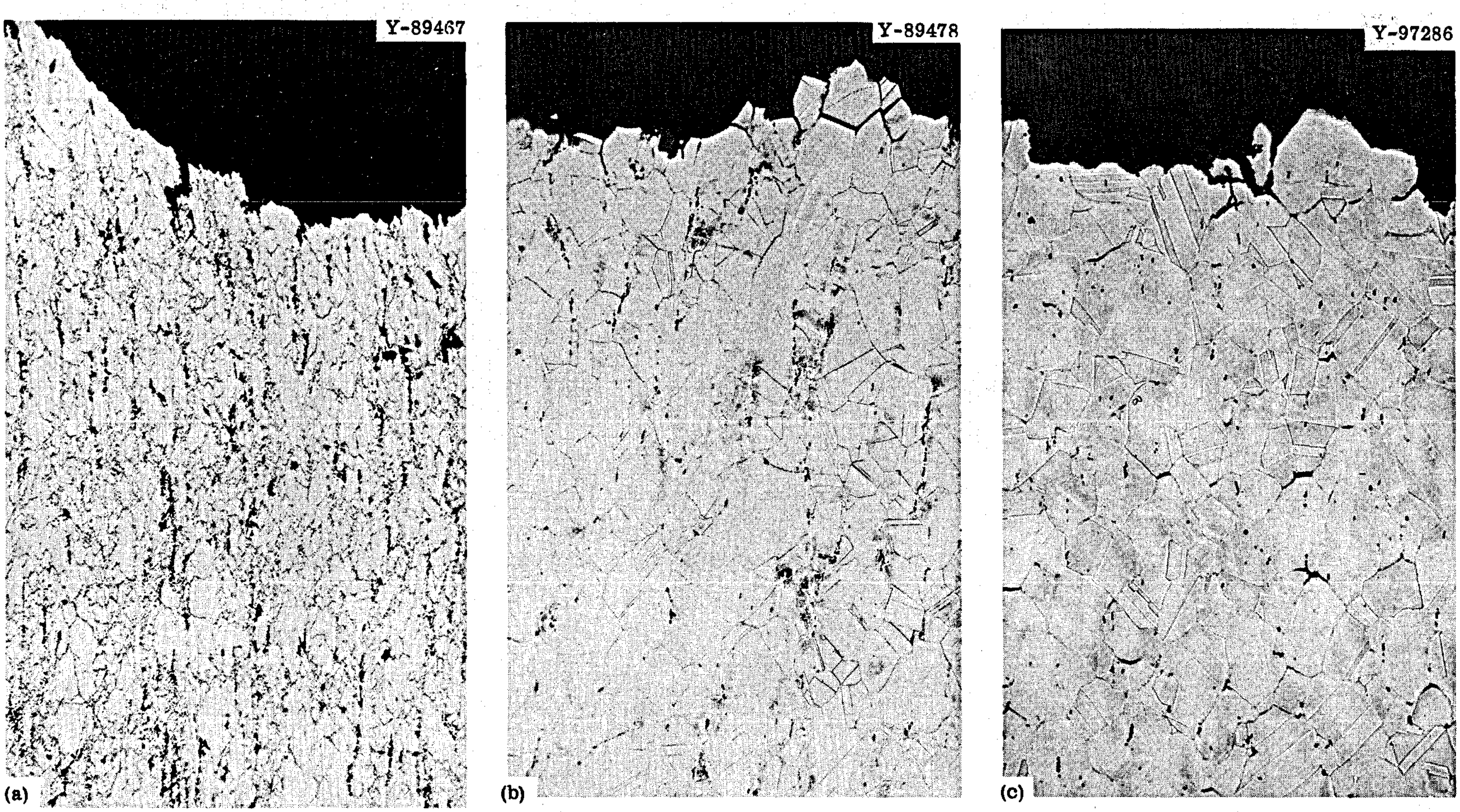


Fig. 68. Photomicrographs of Alloy 21554 (0.35% Zr) Creep Tested at 650°C. (a) Fracture of sample annealed 100 hr at 871°C and 1000 hr at 650°C before test; (b) fracture of a sample annealed 1 hr at 1177°C before test; and (c) fracture of a sample annealed 1 hr at 1177°C and 1000 hr at 650°C before test. 100×. Etchant: glyceria regia. Reduced 21%.

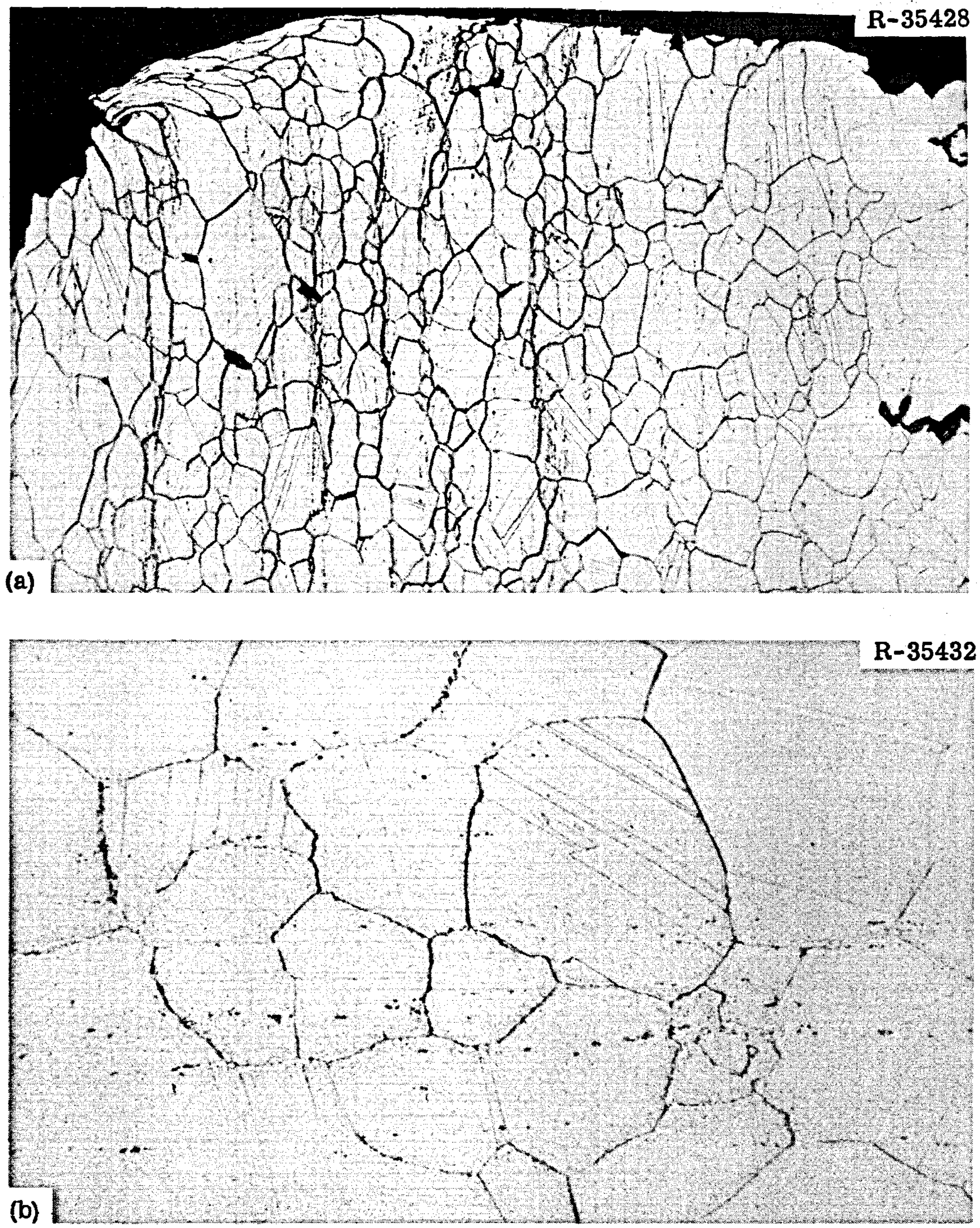


Fig. 69. Photomicrographs of Alloy 108 (0.10% Zr) Annealed 1 hr at 1177°C, Irradiated at 650°C for 1000 hr to a Thermal-Neutron Fluence of 2.5×10^{20} neutrons/cm^2, and Creep Tested at 650°C. Stressed at 32,400 psi for 675 hr (8.2% strain) and 40,000 psi for 53 hr (8.3% strain). (a) Fracture, blunted end caused in handling, 100×; (b) typical, 500×. Etchant: glyceria regia.

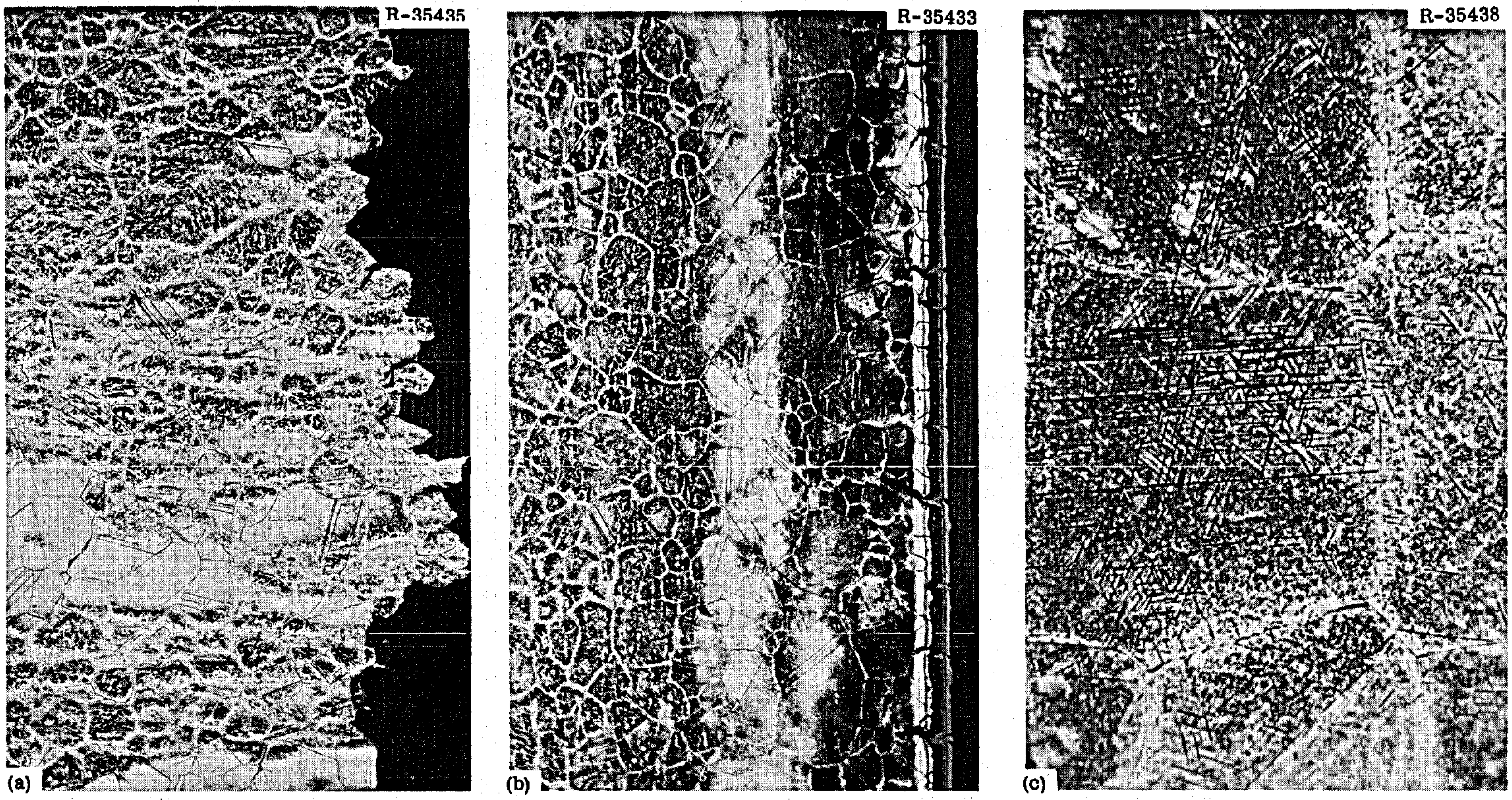


Fig. 70. Photomicrographs of Alloy 110 (0.53% Zr) Annealed 1 hr at 1177°C, Irradiated at 650°C for 1000 hr to a Thermal-Neutron Fluence of 2.5×10^{20} neutrons/cm^2, and Creep Tested at 650°C. Stressed at 32,400 psi for 675 hr (0.97% Strain) and 40,000 psi for 430 hr (5.9% Strain). (a) Fracture, 100×; (b) edge of gage length, 100×; and (c) typical, 1000×. Etchant: glyceria regia. Reduced 24%.

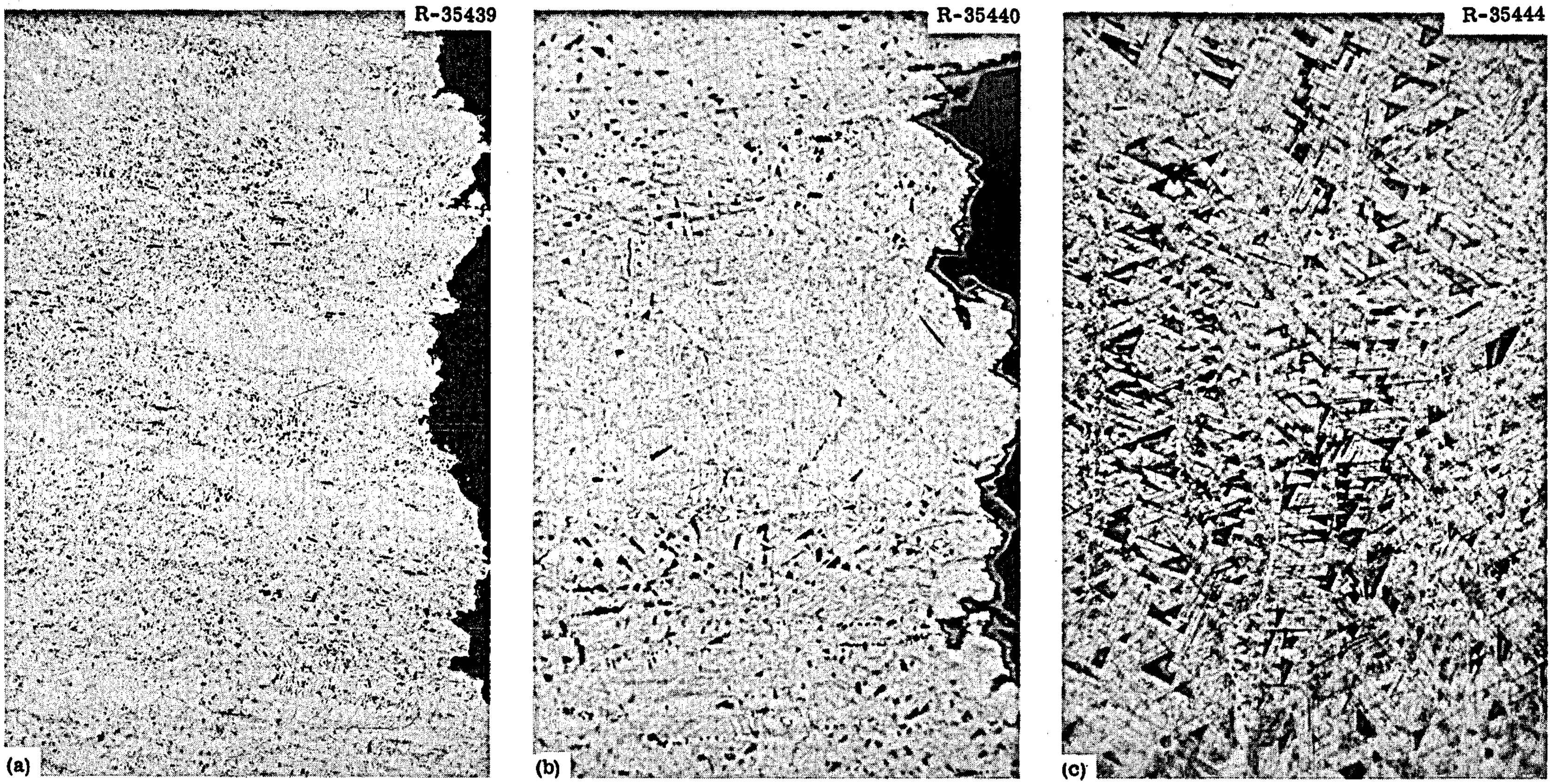


Fig. 71. Photomicrographs of Alloy 112 (1.18% Zr) Annealed 1 hr at 1177°C, Irradiated at 650°C for 1000 hr to a Thermal-Neutron Fluence of 2.5×10^{20} neutrons/cm^2, and Creep Tested at 650°C. Stressed at 32,400 psi for 677 hr (5.8% Strain) and 40,000 psi for 47 hr (3.9% Strain). (a) Fracture, 100×; (b) fracture, 500×; and (c) typical, 1000×. Etchant: glyceria regia. Reduced 24%.

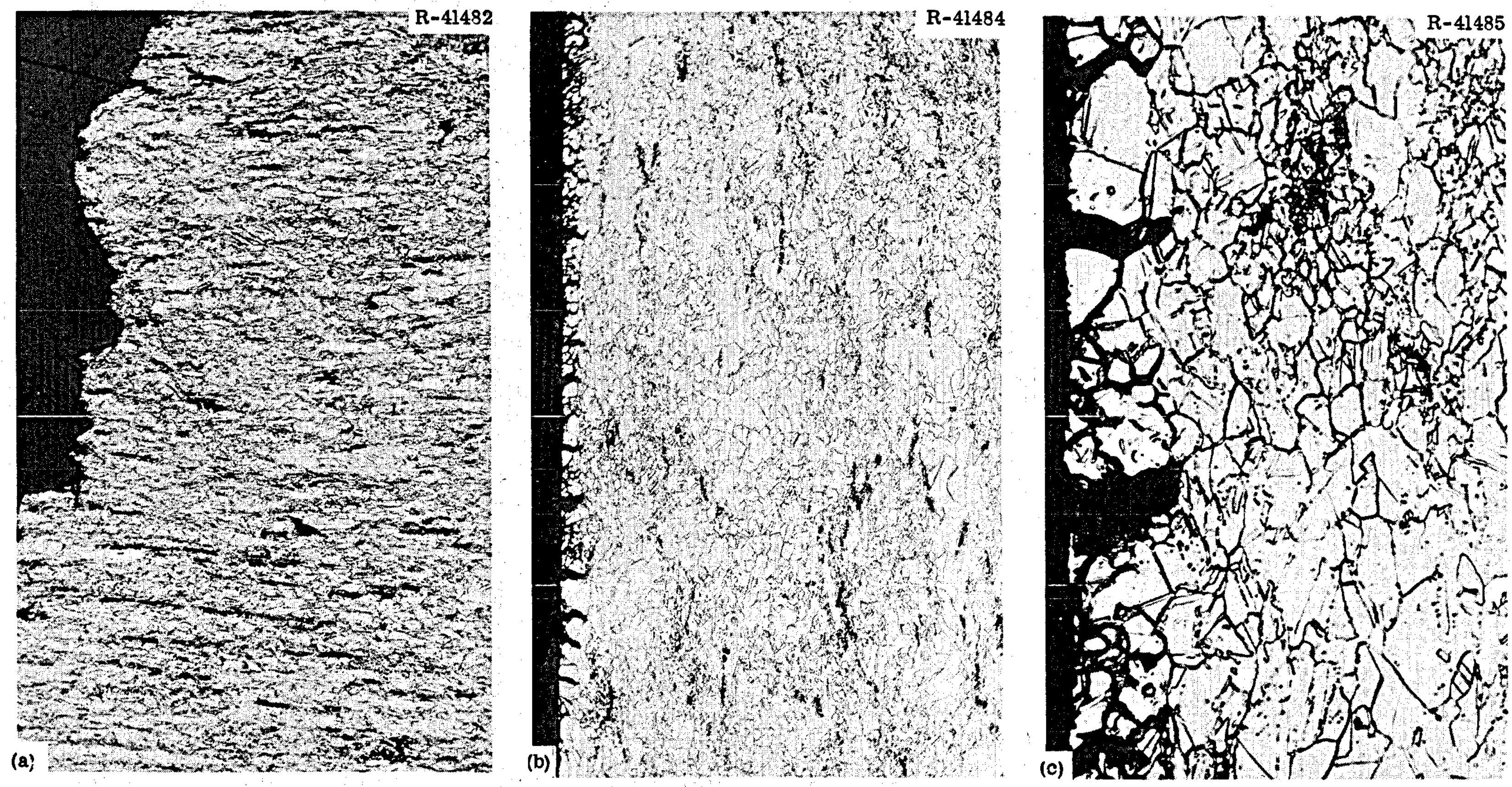


Fig. 72. Photomicrographs of a Zirconium-Modified Hastelloy N Surveillance Sample (Heat 21554) Tested at 25°C at a Strain Rate of 0.05 min^{-1}. Exposed in the Molten Salt Reactor Experiment core for 5500 hr at 650°C to a thermal-neutron fluence of 4.1×10^{20} neutrons/cm^2. (a) Fracture, 100×; (b) edge of sample about 1/2 in. from fracture, 100×; and (c) edge of sample showing edge cracking, 500×. Etchant: aqua regia. Reduced 24%.

Fig. 73. Photomicrograph of the Fracture of a Zirconium-Modified Hastelloy N Sample (Heat 21554) Tested at 25°C at a Strain Rate of 0.05 min^{-1}. Exposed to a static fluoride salt for 5500 hr at 650°C before testing. Note the shear fracture and the absence of edge cracking. 100×. Etchant: glyceria regia.

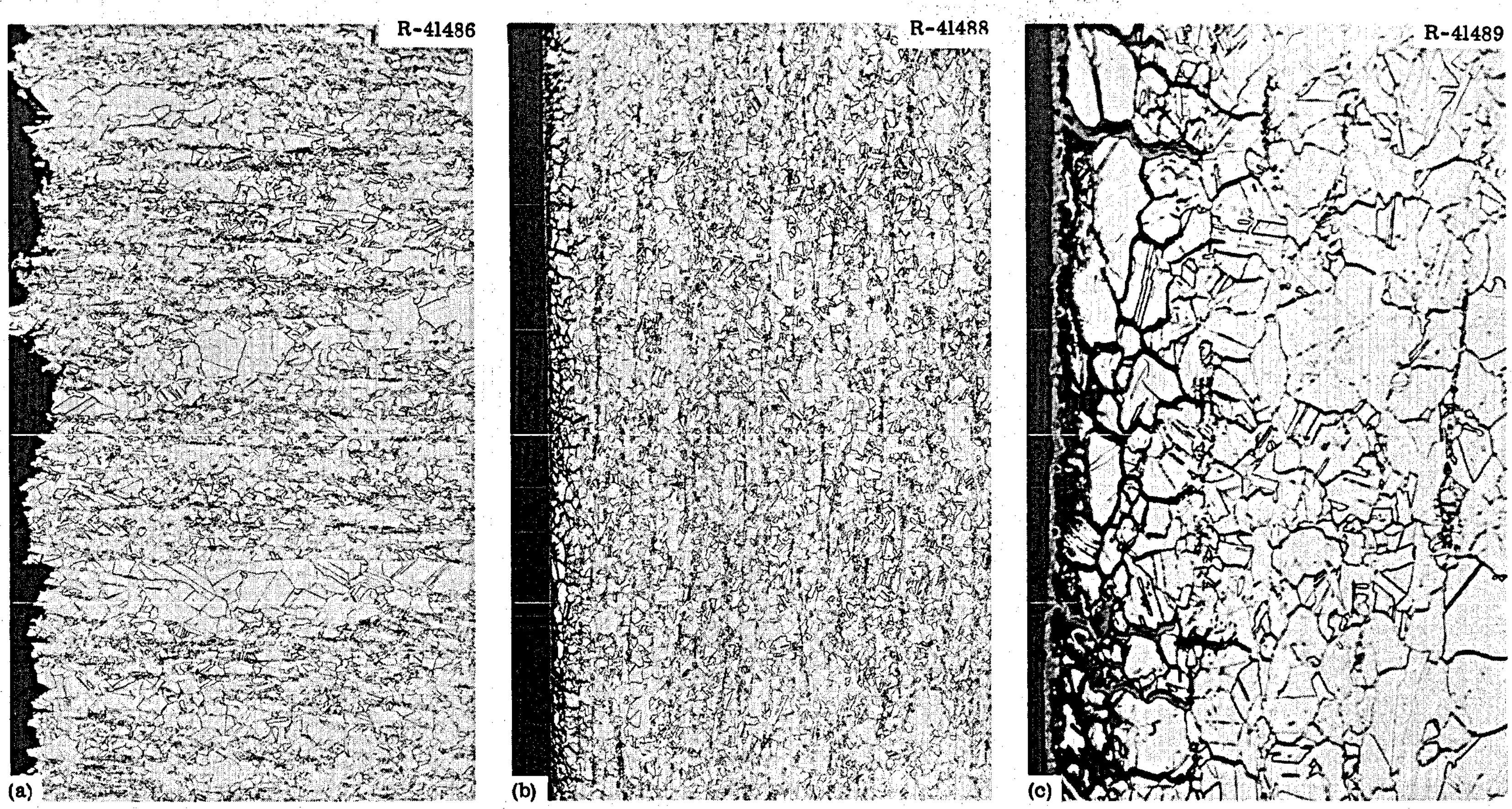


Fig. 74. Photomicrographs of a Zirconium-Modified Hastelloy N Surveillance Sample (Heat 21554) Tested at 650°C at a Strain Rate of 0.002 min^{-1}. Exposed in the Molten Salt Reactor Experiment core for 5500 hr at 650°C to a thermal fluence of 4.1×10^{20} neutrons/cm^2. (a) Fracture, 100×; (b) edge of sample about 1/2 in. from fracture, 100×; and (c) edge of sample showing edge cracking. Oxide formed during the tensile test. 500×. Etchant: aqua regia. Reduced 22%.

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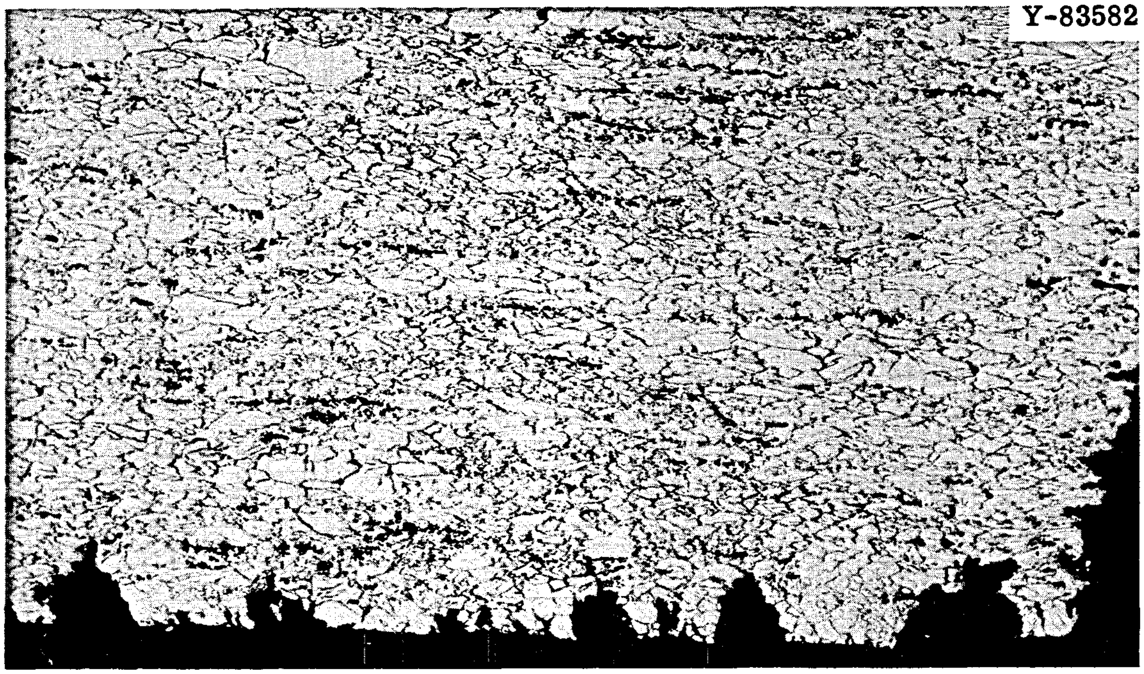


Fig. 75. Photomicrograph Showing a Portion of the Fracture of a Zirconium-Modified Hastelloy N Sample (Heat 21554) Tested at 650°C at a Strain Rate of 0.002 min^{-1}. Exposed to static fluoride salt for 5500 hr at 650°C before testing. 100×. Etchant: glyceria regia.

transgranularly after straining 47.0%. An irradiated sample tested at 650°C is shown in Fig. 74; it fractured intergranularly after 11.2% strain. The comparable control sample, shown in Fig. 75, strained 46.6% before failing. There are numerous intergranular separations, and the fracture is largely intergranular.

Alloys Containing Hafnium

Two groups of alloys were used to study the effects of hafnium content on the mechanical properties. The first group was composed of five laboratory melts that contained from 0.06 to 0.43% Hf (designated alloys 152 through 156), and the second group consisted of two 100-lb commercial melts that contained 0.08 and 0.50% Hf (designated alloys 67-503 and 67-504, respectively). The chemical compositions of these alloys are given in Table 1, and typical photomicrographs are shown in Figs. 76 through 80. Alloy 153 (Fig. 76) contained only 0.06% Hf and had the largest grain size of the group. Alloys 154 (0.10% Hf) and

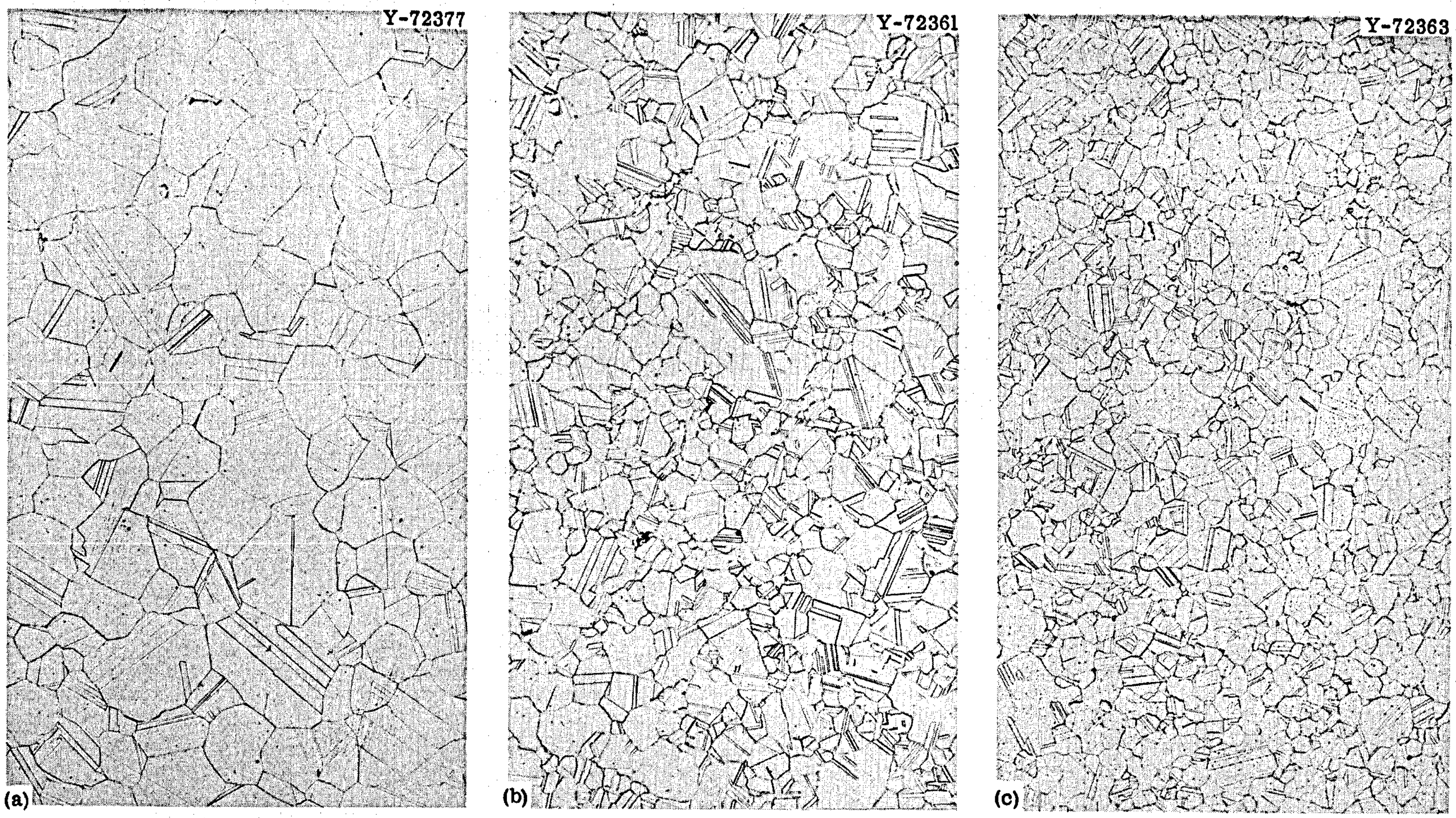


Fig. 76. Photomicrographs of Alloys Modified with Hafnium Annealed 1 hr at 1177°C. (a) Alloy 153 (0.06% Hf); (b) alloy 154 (0.10% Hf) and (c) alloy 155 (0.13% Hf). 100×. Etchant: glyceria regia. Reduced 18%.

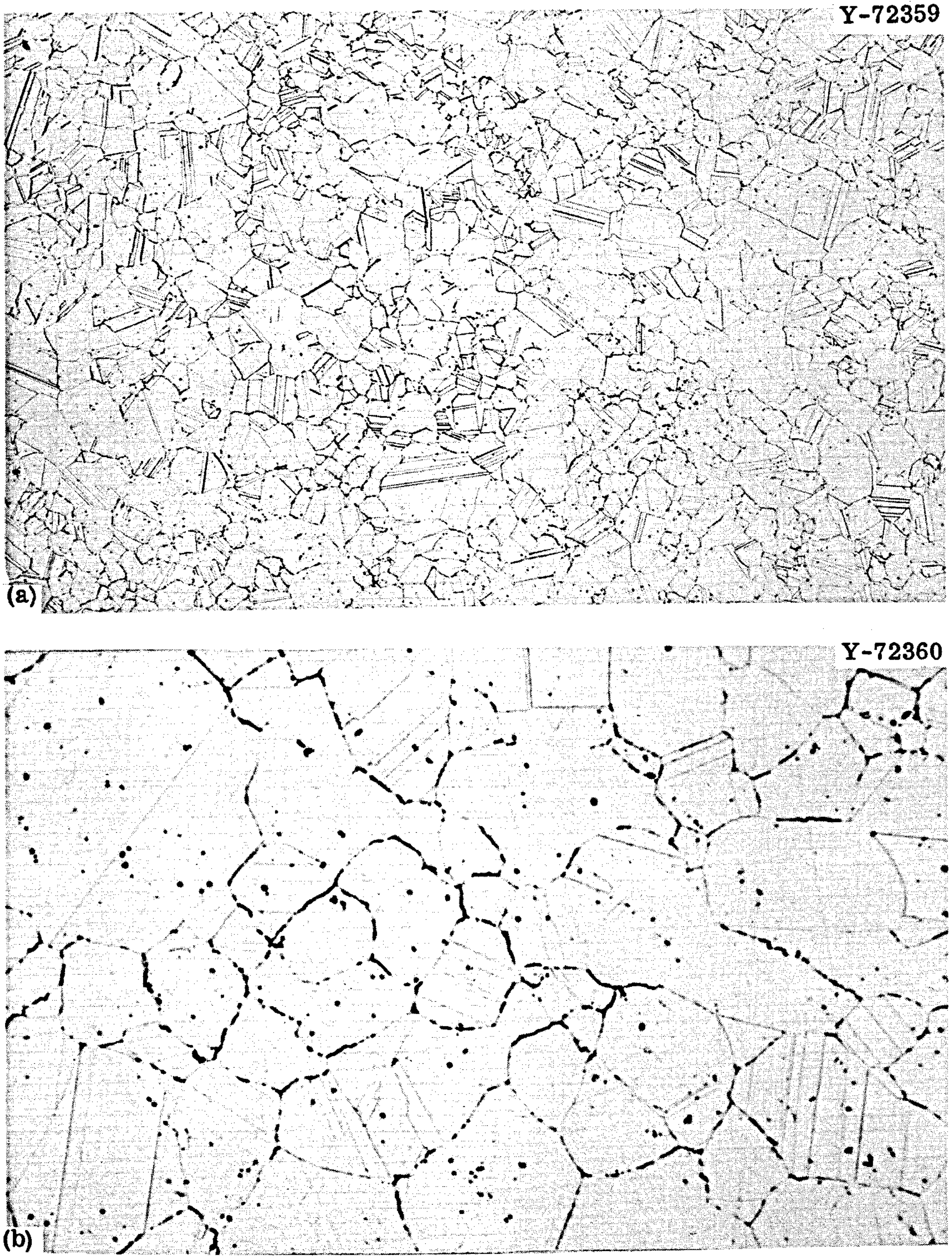


Fig. 77. Photomicrographs of Alloy 152 Modified with 0.16% Hf. Annealed 1 hr at 1177°C. (a) 100× and (b) 500×. Etchant: glyceria regia.

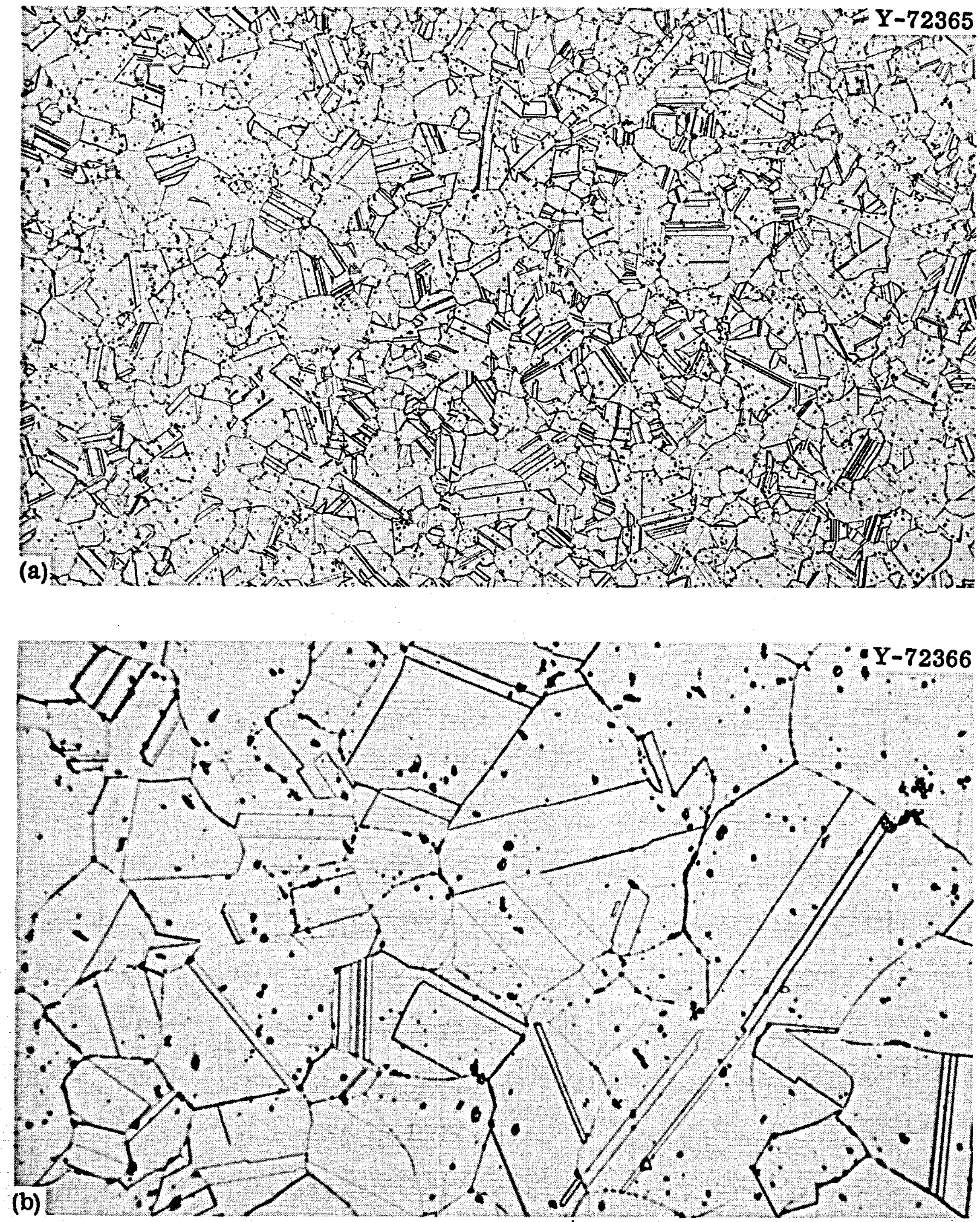


Fig. 78. Photomicrographs of Alloy 156 Modified with 0.43% Hf. Annealed 1 hr at 1177°C. (a) 100X and (b) 500X. Etchant: glyceria regia.

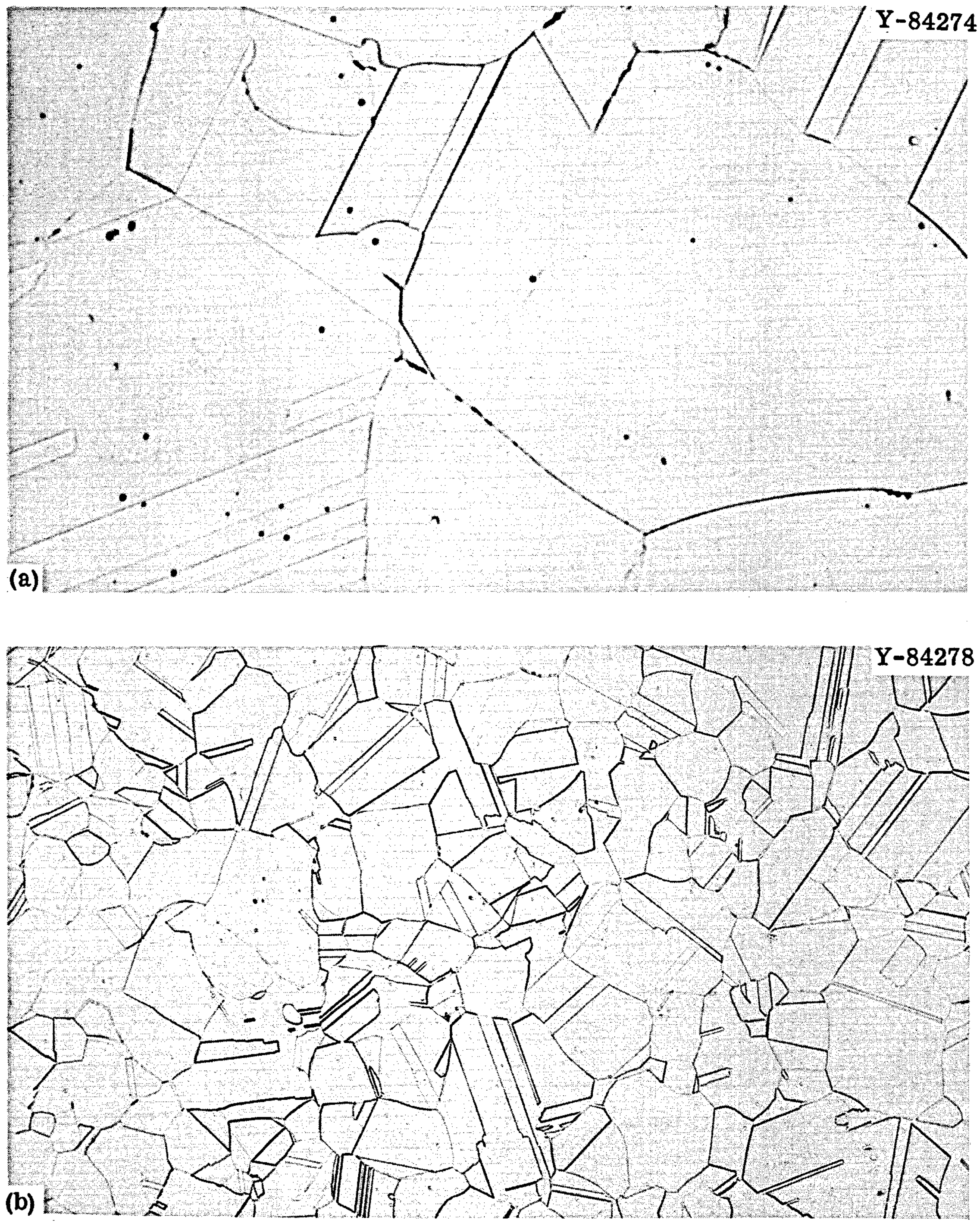
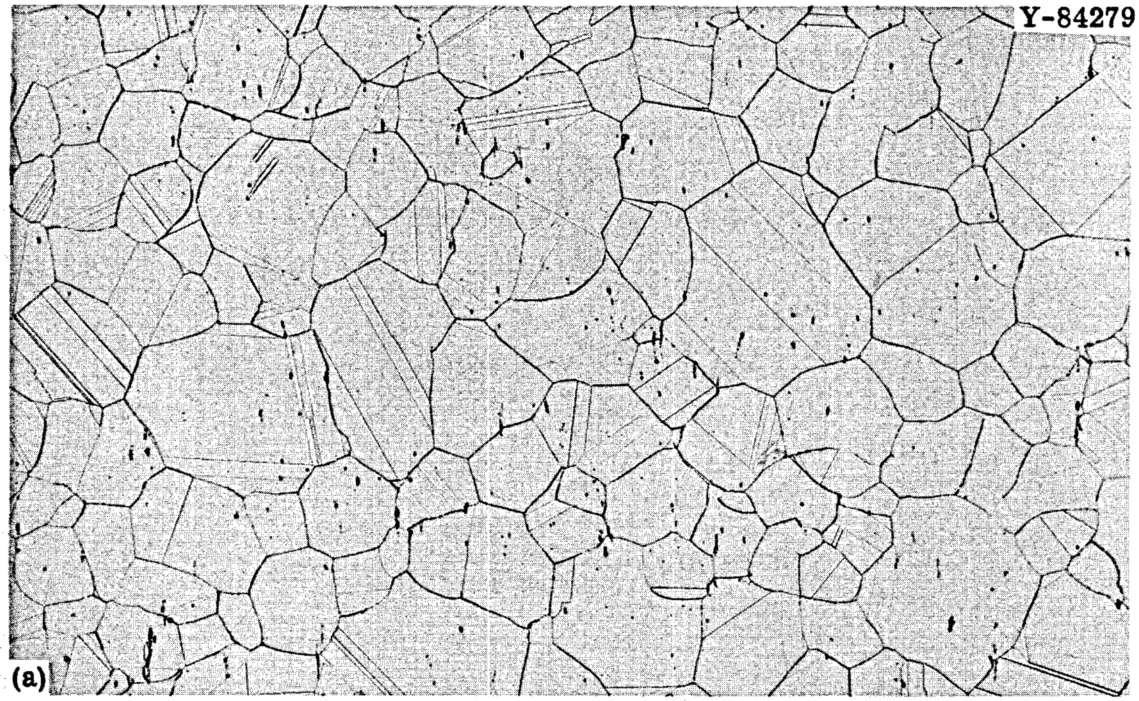


Fig. 79. Typical Photomicrographs of Heat 67-503 (0.08% Hf) Annealed 1 hr at 1177°C. Longitudinal section. (a) 100× and (b) 500×. Etchant: glyceria regia.



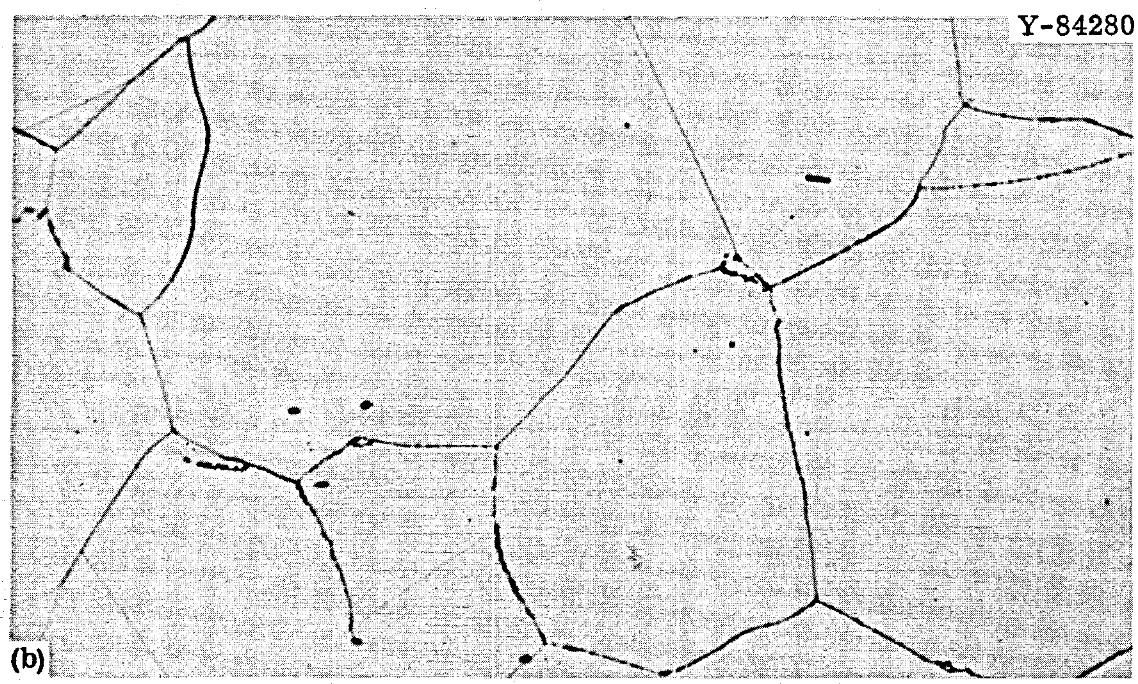


Fig. 80. Typical Photomicrographs of Heat 67-504 (0.50% Hf) Annealed 1 hr at 1177°C. Longitudinal section. (a) 100× and (b) 500×. Etchant: glyceria regia.

155 (0.13% Hf) had much smaller grain sizes. Alloy 152 contained 0.16% Hf, and the grain size was refined by copious quantities of carbide precipitates at grain boundaries. When the hafnium content was increased to 0.43%, as in alloy 156, the precipitation became more general with precipitation at grain boundaries and in the matrix. The two commercial alloys had larger grain sizes than their counterparts in the series of laboratory melts (Figs. 79 and 80).

The results of creep-rupture tests at 650°C on unirradiated and irradiated samples of alloys 152 through 156 are given in Tables 33 and 34, respectively. The stress-rupture properties for the unirradiated

Table 33. Stress Properties at 650°C of Several Hafnium-Modified Alloys[a]

Alloy Number	Specimen Number	Test Number	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)	Reduction in Area (%)
			$\times 10^3$				
152	2979	7506	35	1906.3	0.0015	15.0	20.5
152	2964	6537	70	3.4	1.23	23.4	22.3
152	2967	6538	55	21.1	0.146	14.1	15.6
152	2965	6539	47	67.4	0.051	12.2	13.3
152	2966	6540	40	234.4	0.018	11.7	11.4
153	6554	7507	35	216.5	0.0050	9.3	11.3
153	6453	6658	70	0.3	7.69	41.8	29.3
153	2864	6535	55	6.6	0.194	20.4	17.4
153	2863	6536	47	76.7	0.023	16.2	18.2
153	6545	6687	40	114.2	0.013	11.9	11.8
153	6544	6686	55	7.4	0.223	22.6	18.0
154	6559	6771	70	0.2	3.5	32.0	26.5
154	6556	6689	40	86.0	0.023	7.8	5.5
154	6567	7509	55	5.0	0.188	19.7	24.8
154	6566	7508	47	41.0	0.0352	13.1	18.7
155	8495	7554	70	0		48.4	37.3
155	2873	6531	55	33.6	0.112	14.0	17.9
155	2874	6532	47	90.2	0.043	13.2	14.0
156	6642	6692	70	1.9	1.25	22.4	22.0
156	6643	6693	55	19.0	0.152	14.7	11.9
156	2878	6694	47	257.5	0.0214	18.5	15.6
156	6650	7510	35	1210.5	0.011	22.2	20.3

[a]All materials annealed 1 hr at 1177°C before testing.

Table 34. Creep Properties at 650°C of Irradiated[a] Alloys Containing Various Amounts of Hafnium

Alloy Number	Specimen Number	Test Number	Hafnium Content (wt %)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Elongation (%)
				$\times 10^3$			
100	1916	R-146		32.4	105.4	0.0099	1.1
152	2961	R-373	0.16	47	10.7	0.240	3.14
152	2963	R-208	0.16	40	87.3	0.044	4.28
152	2962	R-371	0.16	32.4	459.5	0.0068	5.14
153	2860	R-212	0.06	40	35.6	0.053	2.38
153	2861	R-222	0.06	35	202.2	0.0065	3.07
153	2862	R-383	0.06	32.4	224.8	0.010	3.32
154	2865	R-374	0.10	47	7.1	0.253	2.25
154	2867	R-188	0.10	40	41.5	0.0548	2.70
154	2866	R-372	0.10	32.4	138.3	0.0115	3.09
155	2872	R-200	0.13	40	63.1	0.0385	2.78
155	2871	R-221	0.13	35	221.9	0.0080	1.93
155	2870	R-238	0.13	35	150.9	0.0084	2.85
156	2877	R-251	0.43	47	80.8	0.101	10.45
156	2875	R-190	0.43	40	395.1	0.0277	15.75
156	2876	R-213	0.43	35	1707.2	0.0064	14.20

[a]All materials annealed 1 hr at 1177°C before irradiation at 650°C to a thermal fluence of 2.3×10^{20} neutrons/cm^2.

alloys are shown in Fig. 81. The results for alloy 100, which contained no detectable hafnium, are used for comparative purposes. The general observation is that the rupture life increased with increasing hafnium content up to about 0.16%. The stress-rupture properties of the irradiated alloys shown in Fig. 82 were even more dependent upon the hafnium content than those of the unirradiated material. In this condition, alloy 156 (0.43% Hf) had far superior stress-rupture properties.

The minimum creep rates of alloys 100 and 152 through 156 are shown in Fig. 83. The alloys that contained hanfium had lower creep rates, but the magnitudes showed no clear dependence upon the concentration between 0.06 and 0.43% Hf. Irradiation caused a slight increase in the creep rate of most alloys; alloy 156 (0.43% Hf) seemed unaffected.

The fracture strains of the alloys in the unirradiated condition are shown in Fig. 84. Generally, the alloys followed the trend of decreasing

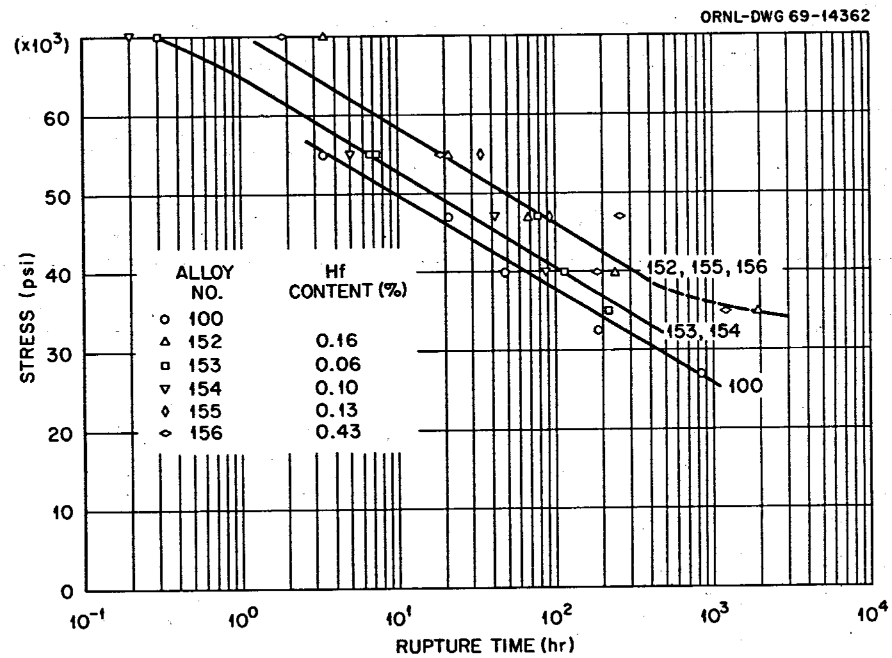


Fig. 81. Stress-Rupture Properties at 650°C of Several Alloys That Contain Hafnium. All samples annealed 1 hr at 1177°C before testing.

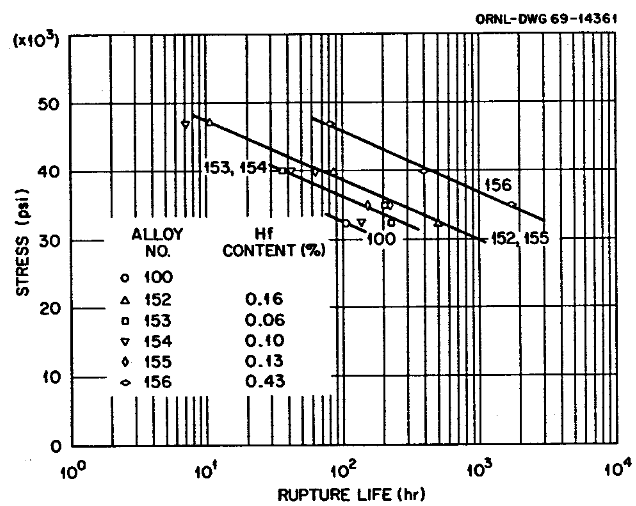


Fig. 82. Creep-Rupture Properties at 650°C of Irradiated Alloys That Contain Various Concentrations of Hafnium. All samples annealed 1 hr at 1177°C before irradiation at 650°C.

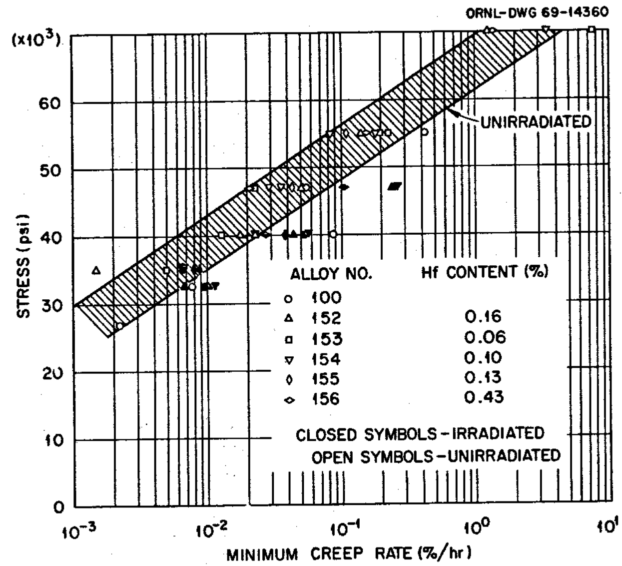


Fig. 83. Creep Rates at 650°C of Alloys That Contain Various Concentrations of Hafnium. All samples annealed 1 hr at 1177°C.

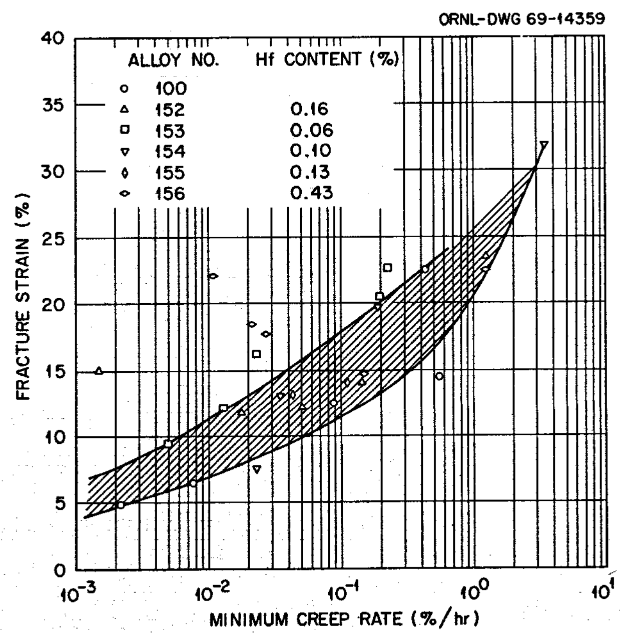


Fig. 84. Fracture Strains at 650°C of Alloys That Contain Various Amounts of Hafnium. All samples annealed 1 hr at 1177°C before testing.

fracture strain with decreasing creep rate. Alloy 156 (0.43% Hf) had slightly superior ductility, particularly at the lower strain rates. The fracture strains after irradiation are shown in Fig. 85. Alloy 156 (0.43% Hf) had high fracture strains, and alloy 152 (0.16% Hf) had fracture strains somewhat improved over those of the other alloys.

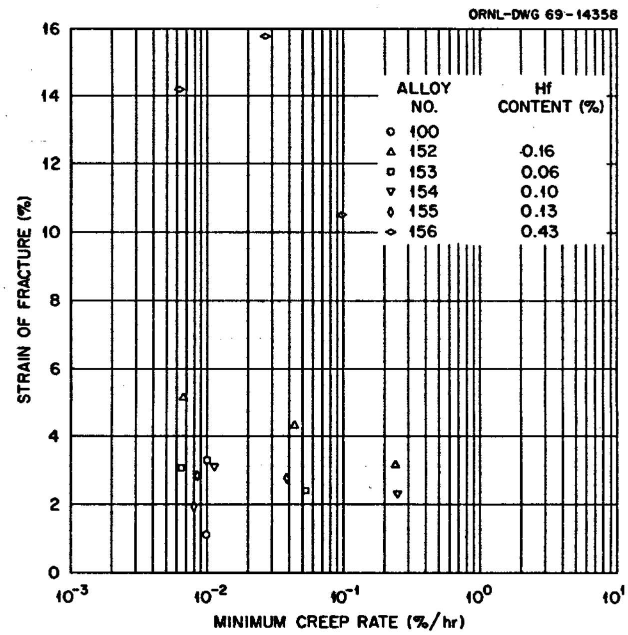


Fig. 85. Fracture Strains at 650°C of Irradiated Alloys That Contain Various Amounts of Hafnium. All samples annealed 1 hr at 1177°C before irradiation at 650°C.

The commercial alloys, 67-503 (0.08% Hf) and 67-504 (0.50% Hf) were subjected to various tests. The results of tensile tests on alloy 67-503 are given in Table 35. The results on irradiated tests showed clearly that the anneal of 1 hr at 1177°C before test gave superior fracture strains at 650°C. More detailed testing was carried out on heat 67-504 (0.50% Hf). This alloy was included in the surveillance program for the MSRE. Samples were exposed to a molten fluoride fuel salt in the core for 9800 hr at 650°C and received a thermal-neutron fluence of 5.3×10^{20} neutrons/cm^2. Control samples were exposed to static barren salt for 9800 hr at 650°C. Some test results on these samples are presented in

Table 35. Tensile Properties of Irradiated and Unirradiated Alloy 67-503 (0.08% Hf)

Specimen Number	Anneal[a] Before Irradiation	Temperature, °C		Thermal-Neutron Fluence (neutrons/cm^2)	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
		Irradiation	Test			Yield	Ultimate Tensile	Uniform	Total	
				$\times 10^{20}$		$\times 10^{3}$	$\times 10^{3}$			
6195	816		650		0.05	48	92.4	28.1	33.7	27.1
6196	816		650		0.002	50.6	74.5	14.6	33.8	31.5
6197	816		760		0.002	37.1	37.8	1.5	24.9	22.7
6193	121		650		0.002	24.3	55.8	21.7	22.9	17.4
6194	121		760		0.002	31.2	45.3	6.3	9.5	12.9
4999	123		760		0.002	27.7	41.2	5.5	6.7	4.7
6229	816	650	650	2.0	0.002	33.4	38.9	3.7	4.4	6.7
6234	816	650	650	2.0	0.05	35.1	46.2	6.0	7.0	14.1
4941	816	760	760	2.4	0.002	8.6	8.7	1.7	1.9	6.9
4974	121	760	760	2.4	0.002	14.3	14.7	1.6	1.7	3.2
4949	121	650	650	2.4	0.002	26.9	40.2	8.0	8.6	10.0
4966	123	760	760	2.4	0.002	7.8	7.8	0.7	0.8	2.1

[a]Annealing designations: 816 = annealed 8 hr at 871°C; 121 = annealed 1 hr at 1177°C; 123 = annealed 1 hr at 1260°C.

Table 36; details of these tests were discussed previously.[7] The fracture strains at a strain rate of 0.05 min^{-1} are shown in Fig. 86 as a function of test temperature. The material in the unirradiated condition was characterized by a high fracture strain of about 50% that decreased gradually with increasing temperature above 650°C. Irradiation caused a general decrease in the fracture strain; the decrease was largest at test temperatures of 25°C and above 600°C. The decrease at 25°C was likely associated with carbide precipitation and has been observed for many other modified and standard heats.[7] The decrease at high temperatures is due to helium produced during irradiation. Similar results for a lower strain rate of 0.002 min^{-1} are shown in Fig. 87. The fracture strains were lower than those observed at the higher strain rate. Additional results given in Table 36 show that the higher anneal of 1 hr at 1177°C resulted in better fracture strains after irradiation than the anneal of 8 hr at 871°C. The samples irradiated in the MSRE for 9800 hr had slightly higher fracture strains than those irradiated in the ORR for about 1100 hr. This may have resulted from the different thermal histories or from the drastically different flux spectra.

[7] H. E. McCoy, An Evaluation of the Molten-Salt Reactor Experiment Hastelloy N Surveillance Specimens — Third Group, ORNL-TM-2647 (1970).

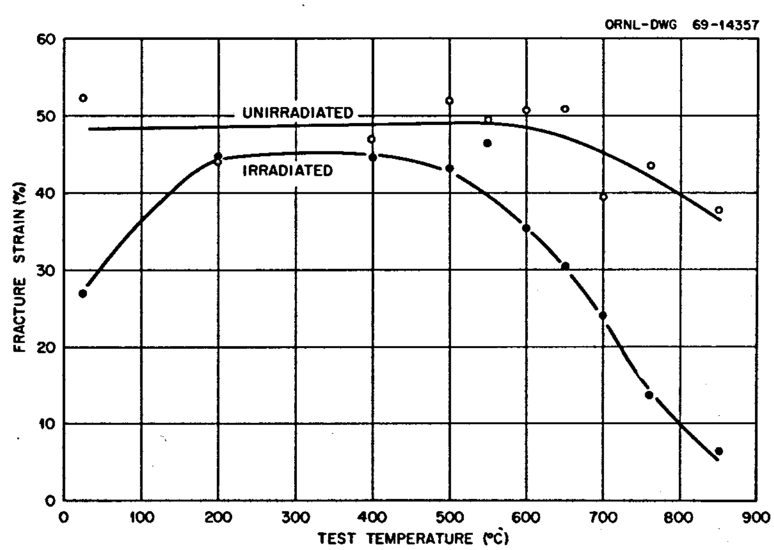


Fig. 86. Variation of Fracture Strains of Alloy 67-504 with Test Temperature at a Strain Rate of 0.05 min^{-1}. Annealed 1 hr at 1177°C. Irradiated at 650°C.

Table 36. Tensile Properties of Irradiated and Unirradiated Alloy 67-504 (0.50% Hf)

Specimen Number	Anneal[a] Before Irradiation	Temperature, °C		Thermal-Neutron Fluence (neutrons/cm²)	Strain Rate (min^{-1})	Stress, psi		Elongation, %		Reduction in Area (%)
		Irradiation	Test			Yield	Ultimate Tensile	Uniform	Total	
				$\times 10^{20}$		$\times 10^3$	$\times 10^3$			
5058	151		25		0.05	67.5	133	50.2	52.3	43.7
5046	151		200		0.05	59.7	119	43.5	44.0	41.6
5057	151		400		0.05	45.5	105	46.2	47.2	36.4
5064	151		400		0.002	53.4	123	46.2	47.9	34.2
5061	151		500		0.05	43.1	99.9	50.6	52.1	41.3
5041	151		500		0.002	61.3	116	44.5	46.1	33.5
5045	151		550		0.05	45.3	102	47.9	49.4	36.9
5053	151		550		0.002	49.2	99.7	47.2	48.5	39.6
5055	151		600		0.05	45.4	97.7	49.1	50.9	36.7
5044	151		600		0.002	45.8	91.3	41.9	43.7	37.3
5065	151		650		0.05	43.3	94.3	48.8	50.9	37.4
5042	151		650		0.002	44.9	80.2	30.2	41.2	38.1
5067	151		700		0.05	53.3	94.6	30.8	39.5	36.0
5048	151		700		0.002	42.1	68.1	16.2	45.8	40.6
5066	151		760		0.05	44.2	75.4	20.3	43.5	38.4
5047	151		760		0.002	42.7	50.8	6.0	38.9	41.8
5060	151		850		0.05	34.9	47.8	7.7	38.7	35.5
5056	151		850		0.002	31.5	32.4	2.9	34.8	34.3
6255	121		25		0.05	37.4	104.7	66.6	73.2	73.7
6256	121		200		0.05	33.1	104.2	68.7	71.3	52.2
6257	121		400		0.05	27.1	88	77.1	80.1	60.6
6258	121		500		0.05	30	96.6	67.3	73.9	50.0
6259	121		550		0.05	27.8	91.2	79.0	82.3	62.5
6260	121		600		0.05	25.5	87.4	73.6	79.6	56.3
6265	121		600		0.002	24.4	71.4	45.7	47.1	34.7
6262	121		650		0.05	25.6	83	63.9	66.9	45.1
6266	121		650		0.002	23.9	63.6	33.1	34.2	26.9
6263	121		700		0.05	25.7	76.2	46.7	49.3	32.4
6264	121		760		0.05	24	65.8	33.5	35.5	25.7
6267	121		760		0.002	35.8	42	6.3	11.3	16.4
5086	121	650	25	5.3	0.05	102	119	26.0	27.0	33.5
5087	121	650	200	5.3	0.05	46.4	106	43.7	44.4	31.9
5088	121	650	400	5.3	0.05	41	97.6	44.0	44.5	37.2
5089	121	659	400	5.3	0.002	41.8	99	41.8	42.9	36.4
5090	121	650	500	5.3	0.05	40.2	93	42.8	43.3	37.9
5091	121	650	500	5.3	0.002	41.6	91.2	42.2	43.2	28.2
5092	121	650	550	5.3	0.05	39.6	92	46.0	46.7	36.5
5093	121	650	550	5.3	0.002	38	78.2	28.2	29.9	21.2
5079	121	650	600	5.3	0.05	39.7	82.6	35.3	35.7	33.5
5078	121	650	600	5.3	0.002	40.2	69.6	23.7	24.8	17.2
5085	121	650	650	5.3	0.05	32.9	70	30.0	30.7	31.5
5084	121	650	650	5.3	0.002	34.7	63.9	21.9	23.1	17.0
5077	121	650	700	5.3	0.05	35	65.9	23.2	24.2	24.5
5076	121	650	700	5.3	0.002	35.8	56.2	11.9	14.6	13.1
5075	121	650	760	5.3	0.05	34.9	57.6	12.8	13.8	18.0
5074	121	650	760	5.3	0.002	37	42.3	4.3	6.7	11.9
5073	121	650	850	5.3	0.05	32.6	40.8	4.8	6.6	9.4
5072	121	650	850	5.3	0.002	25.4	26	2.1	4.4	11.5
6238	816	650	650	2.0	0.002	34.8	50.4	9.2	9.9	13.2
6239	816	650	650	2.0	0.05	34.5	54.5	10.3	12.0	19.4
4945	816	760	760	2.0	0.002	18.5	18.6	2.4	2.8	4.9
4927	121	650	650	2.4	0.002	30.4	50.4	12.8	16.8	7.0
4986	121	760	760	2.4	0.002	13.5	13.5	1.1	1.1	6.0
4970	123	760	760	2.4	0.002	9.3	9.3	1.2	1.3	5.1

[a]Annealing designations: 151 = annealed 1 hr at 1177°C plus 9800 hr at 650°C; 121 = annealed 1 hr at 1177°C; 816 = annealed 8 hr at 871°C; 123 = annealed 1 hr at 1260°C.

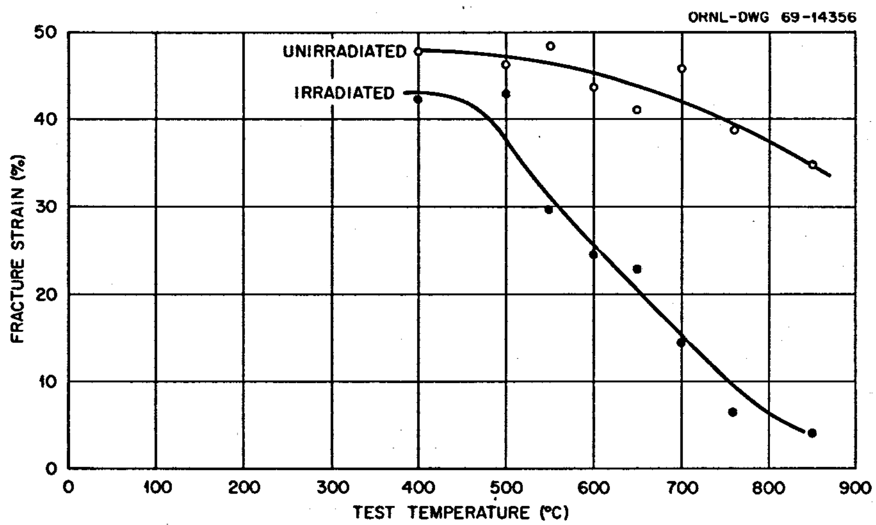


Fig. 87. Variation of Fracture Strain of Alloy 67-504 with Test Temperature at a Strain Rate of 0.002 min^{-1}. Annealed 1 hr at 1177°C. Irradiated at 650°C.

The results of creep-rupture tests on heat 67-503 are given in Table 37. The stress-rupture properties are shown graphically in Fig. 88, and the creep characteristics are shown in Fig. 89. Irradiation reduced the rupture life more at 760°C than at 650°C. Irradiation at 650°C had no effect on the minimum creep rate, but irradiation at 760°C increased the minimum creep rate (Fig. 89).

Results of creep-rupture tests for heat 67-504 (0.50% Hf) are summarized in Table 38. Data are included from the MSRE surveillance program. The stress-rupture characteristics are shown in Fig. 90. This heat had a rupture life eight to ten times higher than that of heat 67-503 (0.08% Hf). The samples aged in fluoride salt for 9800 hr had slightly shorter rupture lives than those annealed 1 hr at 1177°C before test. Irradiation at 650°C reduced the rupture life, but less for the samples irradiated in the MSRE than for those irradiated in the ORR. Irradiation at 760°C in the ORR drastically decreased the rupture life. The minimum creep rates for these samples are shown graphically in Fig. 91. The creep strength of heat 67-504 is about ten times that of heat 67-503. The minimum creep rate was unaffected by aging at 650°C for 9800 hr in static salt or by short-term (1000-hr) irradiation at 650°C. Irradiation at 650°C in the MSRE increased the creep rate, and irradiation at 760°C drastically increased the creep rate.

Table 37. Creep Properties of Irradiated[a] and Unirradiated Alloy 67-503 (0.08% Hf)

Specimen Number	Test Number	Anneal[b] Before Irradiation	Temperature, °C Irradiation	Temperature, °C Test	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Fracture Strain (%)	Reduction in Area (%)
					$\times 10^3$				
6179	7514	121		650	55	5.6	0.26	24.6	26.0
4957	6257	121		650	47	68.3	0.046	14.3	18.0
4958	6256	121		650	40	150.1	0.019	9.9	11.6
6178	7513	121		650	30	498.1	0.0045	8.3	17.5
6177	7512	121		760	20	41.1	0.15	9.2	5.1
6176	7511	121		760	15	128.1	0.039	8.4	16.8
4950	R-493	121	650	650	47	4.1	0.45	2.8	
4952	R-505	121	650	650	40	32.6	0.071	3.2	
4955	R-537	121	650	650	35	45.6	0.073	3.7	
4975	R-500	121	760	650	47	0			
4978	R-515	121	760	650	35	0.4	0.35	0.77	
4979	R-547	121	760	650	30	2.2	0.036	0.11	
4967	R-499	123	760	650	47	0			
4969	R-541	123	760	650	40	0			
4942	R-510	816	760	650	40	2.6	0.22	0.87	
4944	R-539	816	760	650	35	3.3	0.096	0.51	
4954	R-513	121	650	760	15	36.8	0.029	1.7	
4977	R-508	121	760	760	12.5	7.6	0.082	0.41	
4968	R-533	123	760	760	12.5	0.5	0.053	0.07	
4943	R-527	816	760	760	12.5	8.1	0.021	0.68	

[a]Irradiated at indicated temperatures to a thermal fluence of 2.4×10^{20} neutrons/cm^2.

[b]Annealing designations: 121 = annealed 1 hr at 1177°C; 123 = annealed 1 hr at 1260°C; 816 = annealed 8 hr at 871°C.

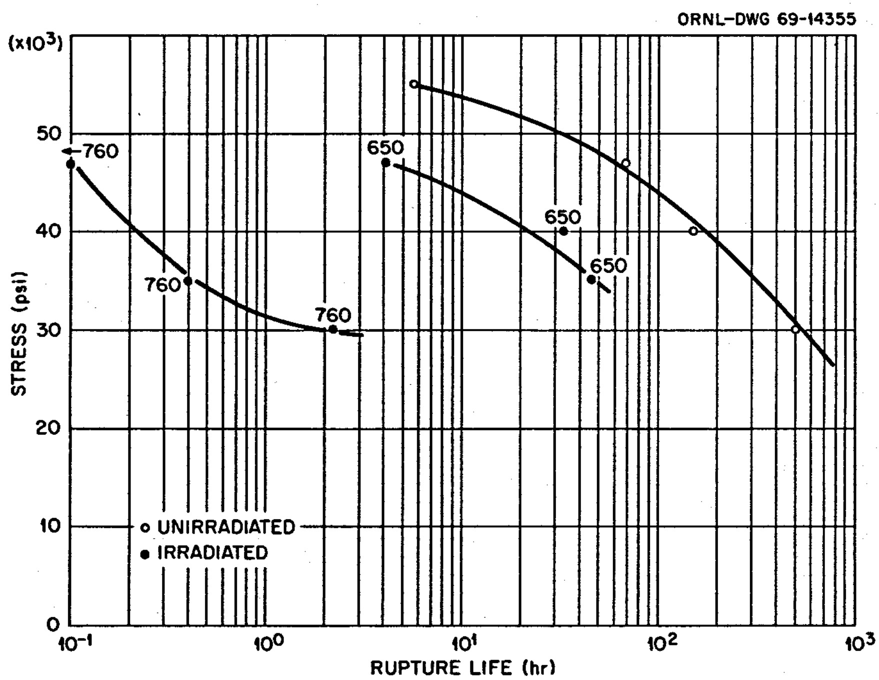


Fig. 88. Stress-Rupture Properties at 650°C of Alloy 67-503. All samples annealed 1 hr at 1177°C before test or irradiation. Number by each datum point indicates irradiation temperature.

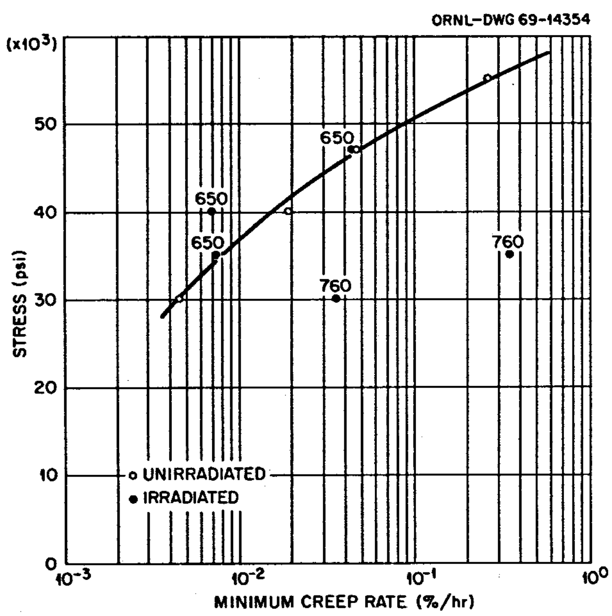


Fig. 89. Creep Rates at 650°C of Alloy 67-503. All samples annealed 1 hr at 1177°C. Numbers by each datum point indicate irradiation temperature.

Table 38. Creep Properties of Irradiated and Unirradiated Alloy 67-504 (0.50% Hf)

Specimen Number	Test Number	Anneal[a] Before Irradiation	Temperature, °C		Thermal-Neutron Fluence (neutrons/cm²)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Fracture Strain (%)	Reduction in Area (%)
			Irradiation	Test						
					$\times 10^{20}$	$\times 10^{3}$				
6247	7432	121		650		70	1.3	3.74	32.9	33.4
6245	7431	121		650		63	17.7	0.16	31.2	27.7
4991	6255	121		650		55	127.9	0.029	27.2	37.8
4936	6254	121		650		47	425.5	0.0068	28.7	19.7
4933	6253	121		650		40	876.9	0.0030	16.3	20.1
6243	7430	121		650		40	1236.5	0.0034	26.5	27.3
5050	7228	151		650		70	5.5	3.80	43.7	43.9
5043	7227	151		650		55	108.3	0.18	41.7	39.6
5049	7226	151		650		47	329.2	0.049	39.4	43.3
5062	7225	151		650		40	1014.6	0.020	40.8	48.0
6249	7516	121		760		20	213.4	0.088	34.8	27.2
4929	R-518	121	650	650	2.4	55	17.0	0.36	13.1	
4928	R-507	121	650	650	2.4	47	87.9	0.060	7.6	
4932	R-548	121	650	650	2.4	40	261.2	0.017	5.9	
4987	R-509	121	760	650	2.4	47	0.3	7.38	2.2	
4989	R-543	121	760	650	2.4	40	1.3	0.43	0.60	
5083	R-733	121	650	650	5.3	55	59.6	0.091	7.0	
5080	R-715	121	650	650	5.3	47	181.7	0.027	6.8	
5081	R-708	121	650	650	5.3	40	467.2	0.011	6.9	
5082	R-732	121	650	650	5.3	32.4	1643.8	0.0025	5.9	
4971	R-495	123	760	650	2.4	47	0			
4973	R-544	123	760	650	2.4	40	0.1		0.68	
4946	R-511	816	760	650	2.4	47	4.6	0.17	1.8	
4948	R-540	816	760	650	2.4	40	19.9	0.020	0.93	
5112	R-952	121	650	650	0.25	55	0.6		8.6	
5148	R-953	121	650	650	0.25	55	1.0		10.6	
4990	R-542	121	760	760	2.4	17.5	8.1	0.089	0.94	
4930	R-519	121	650	760	2.4	15	287.7	0.015	8.0	
4988	R-517	121	760	760	2.4	15	74.7	0.042	3.9	
4947	R-492	121	760	760	2.4	15	9.3	0.076	1.6	
4972	R-504	123	760	760	2.4	12.5	0.3	0.35	0.33	

[a]Annealing designations: 121 = annealed 1 hr at 1177°C; 151 = annealed 1 hr at 1177°C plus 9800 hr at 650°C; 123 = annealed 1 hr at 1260°C; 816 = annealed 8 hr at 871°C.

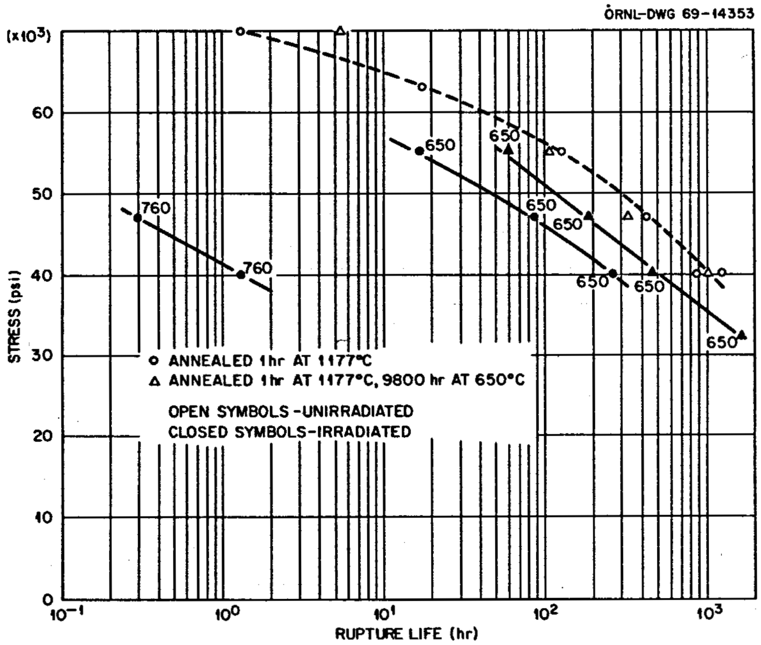


Fig. 90. Stress Rupture at 650°C of Alloy 67-504. Number by each datum point indicates irradiation temperature.

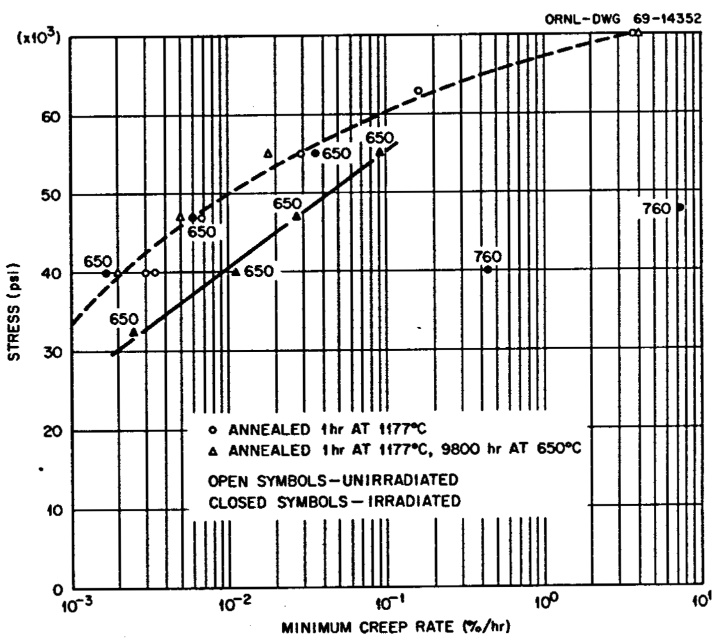


Fig. 91. Creep Rates at 650°C of Alloy 67-504. Number by each datum point indicates irradiation temperature.

The fracture strains of heats 67-503 and 67-504 in the unirradiated condition at 650°C are shown in Fig. 92. Heat 67-504 (0.50% Hf) had much higher fracture strains than heat 67-503 (0.08% Hf), particularly at the lower creep rate.

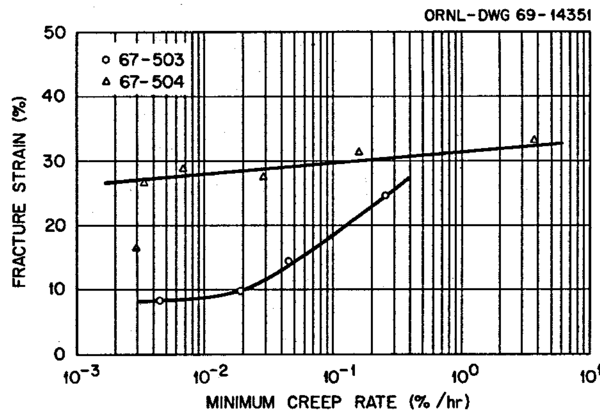


Fig. 92. Fracture Strains at 650°C of Unirradiated Alloys 67-503 and 67-504. All samples annealed 1 hr at 1177°C.

The rather sparse data for heats 67-503 and 67-504 at 760°C are presented in Tables 37 and 38, and the stress-rupture properties are plotted in Fig. 93. In the unirradiated condition, alloy 67-504 had a rupture life about five times greater than that of alloy 67-503. Irradiation at

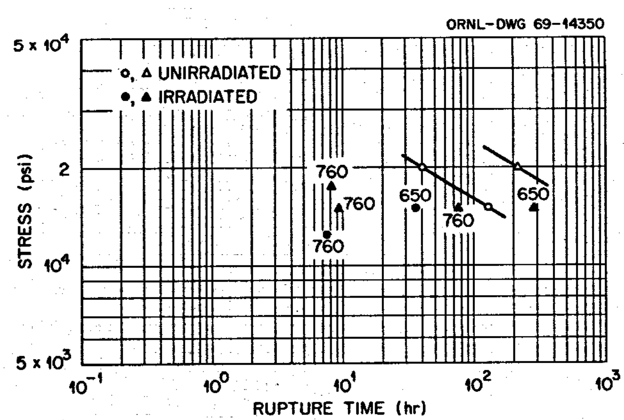


Fig. 93. Stress-Rupture Properties at 760°C of Alloys 67-503 and 67-504. All samples annealed 1 hr at 1177°C. Number by each datum point indicates the irradiation temperature.

650°C reduced the rupture life, but irradiation at 760°C reduced it much more. The fracture strains in the irradiated condition are shown in Fig. 94. The fracture strains of heat 67-503 were 3 to 4% when irradiated and tested at 760°C. Irradiation at 760°C and testing at either 650 or 760°C resulted in lower fracture strains. Heat 67-504 generally had fracture strains of 6 to 8% when irradiated at 650°C and tested at 650 or 760°C. Irradiation at 760°C resulted in fracture strains as low as 0.6%.

Several of the samples were examined metallographically after testing. Typical photomicrographs of a sample of heat 67-504 after exposure to a noncorrosive fluoride salt for 9800 hr at 650°C and testing at 25°C are shown in Fig. 95. The fracture was partially intergranular, although the bulk grains elongated greatly. Also more carbides formed than in the annealed material shown in Fig. 80. At 650°C, the grains still showed

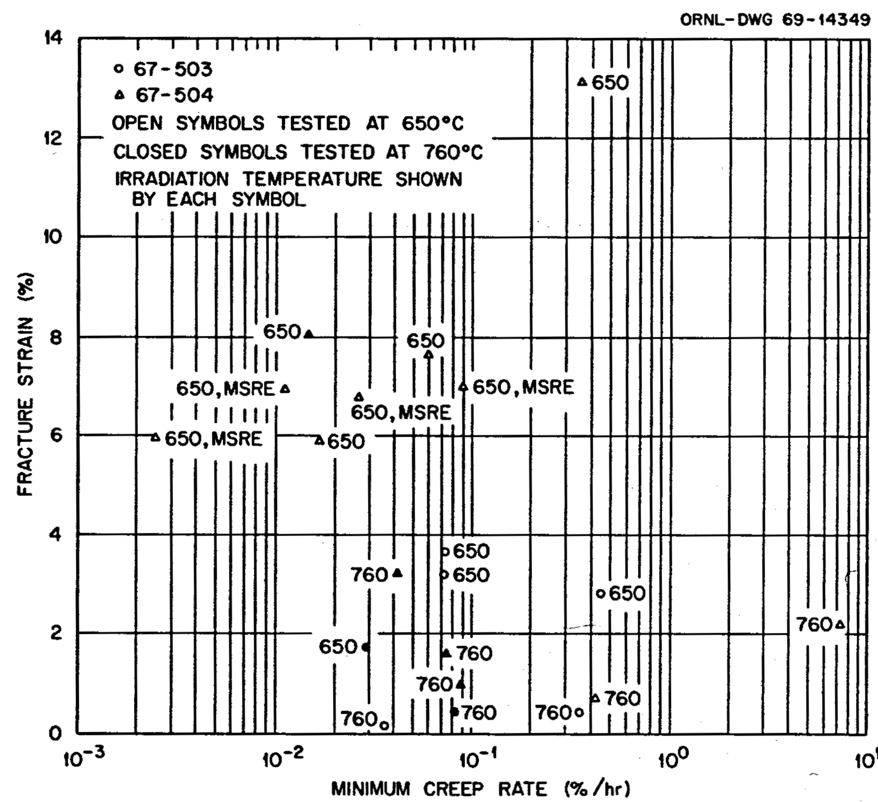


Fig. 94. Postirradiation Fracture Strains of Heats 67-503 and 67-504.

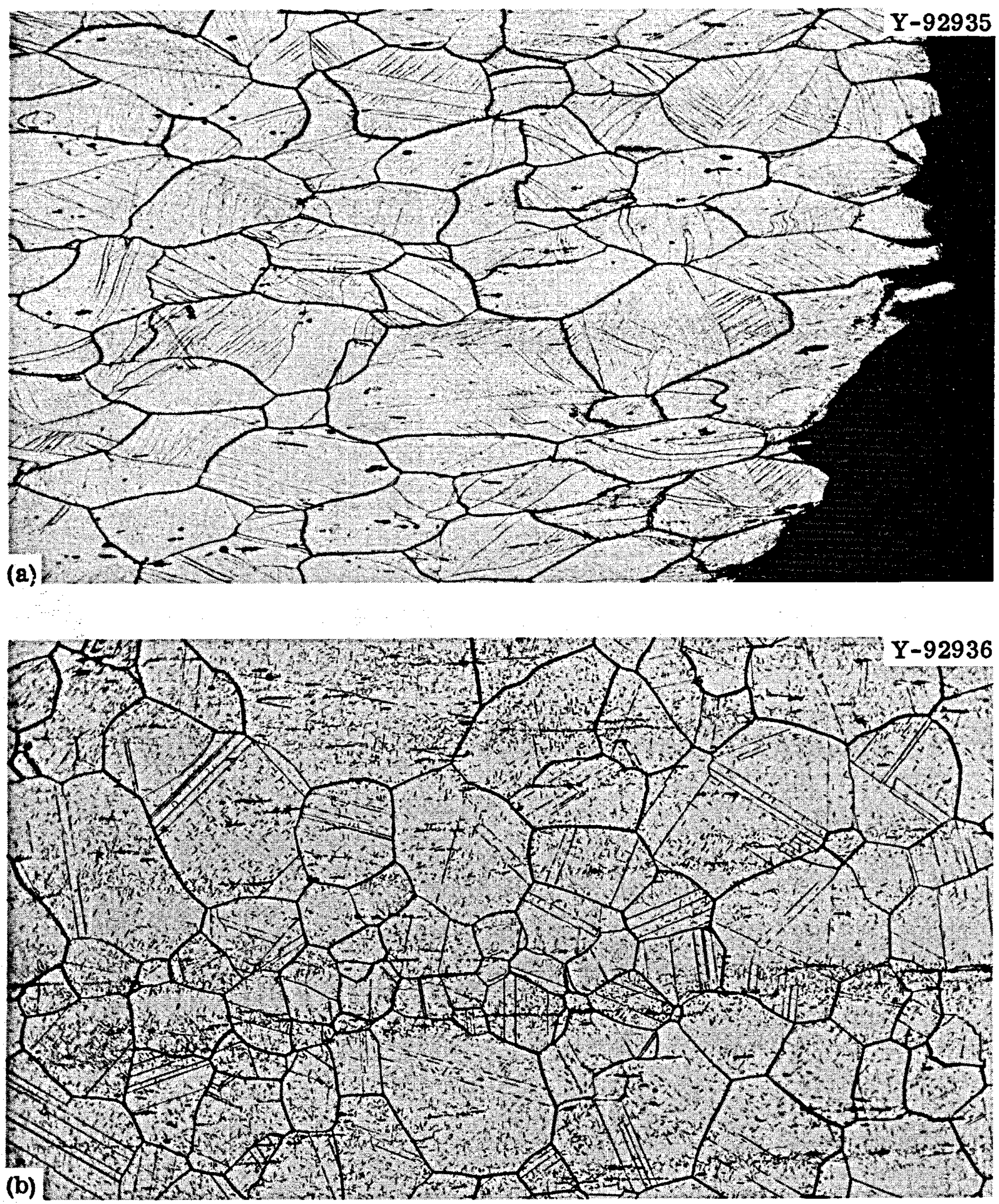


Fig. 95. Photomicrographs of Sample of Heat 67-504 (0.50% Hf) Annealed 1 hr at 1177°C, Exposed 9800 hr to Fluoride Salt at 650°C, and Tested in Tension at 25°C. (a) Fracture, 100×; (b) typical unstressed, 100×. Etchant: glyceria regia.

extensive elongation, and there are numerous intergranular cracks, although the final fracture was mixed intergranular and transgranular (Fig. 96). After irradiation, the fracture of heat 67-504 at 25°C (Fig. 97) was more intergranular than that of the unirradiated material (Fig. 95). A typical fracture of heat 67-503 after creep testing at 650°C is shown in Fig. 98. There are numerous intergranular cracks, and the fracture is predominantly intergranular. Heat 67-504 (Fig. 99) exhibited fewer intergranular cracks and a fracture that was mixed intergranular and transgranular.

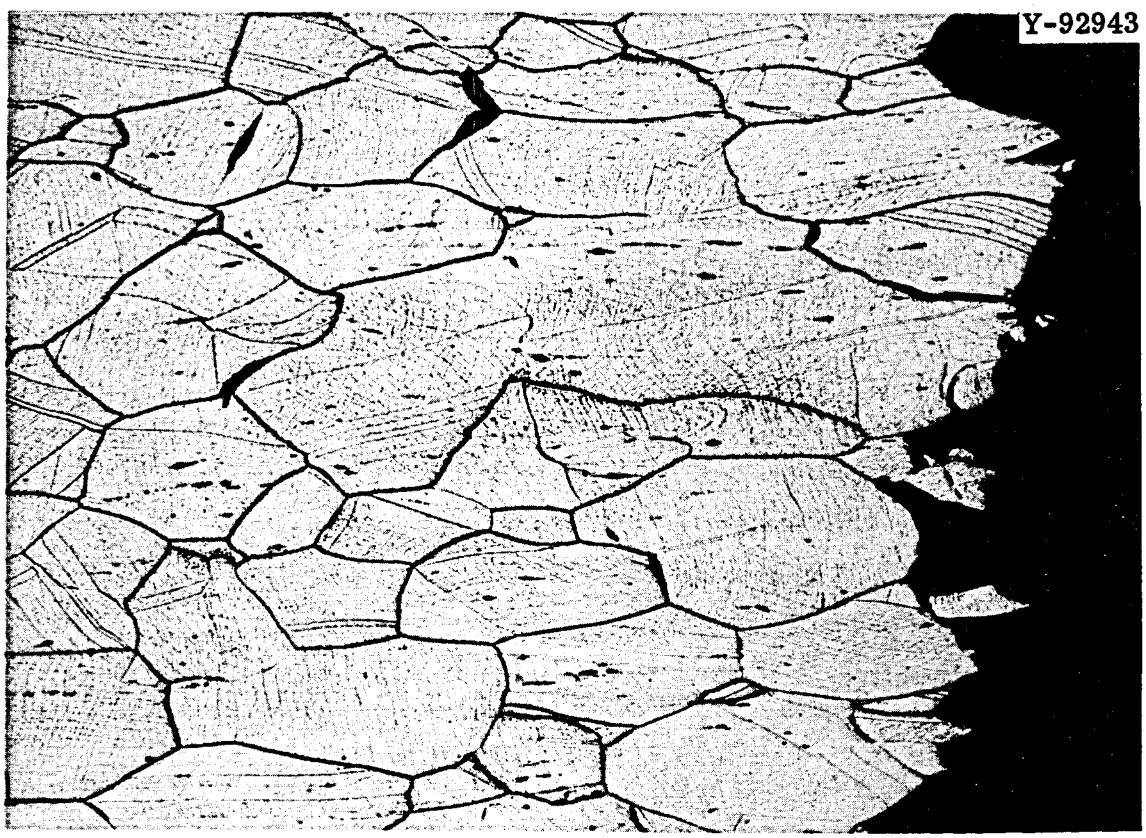


Fig. 96. Photomicrograph of the Fracture of a Sample of Heat 67-504 (0.50% Hf) Annealed 1 hr at 1177°C, Exposed 9800 hr to Fluoride Salt at 650°C, and Tested in Tension at 650°C and a Strain Rate of 0.002 min^{-1}. 100x.

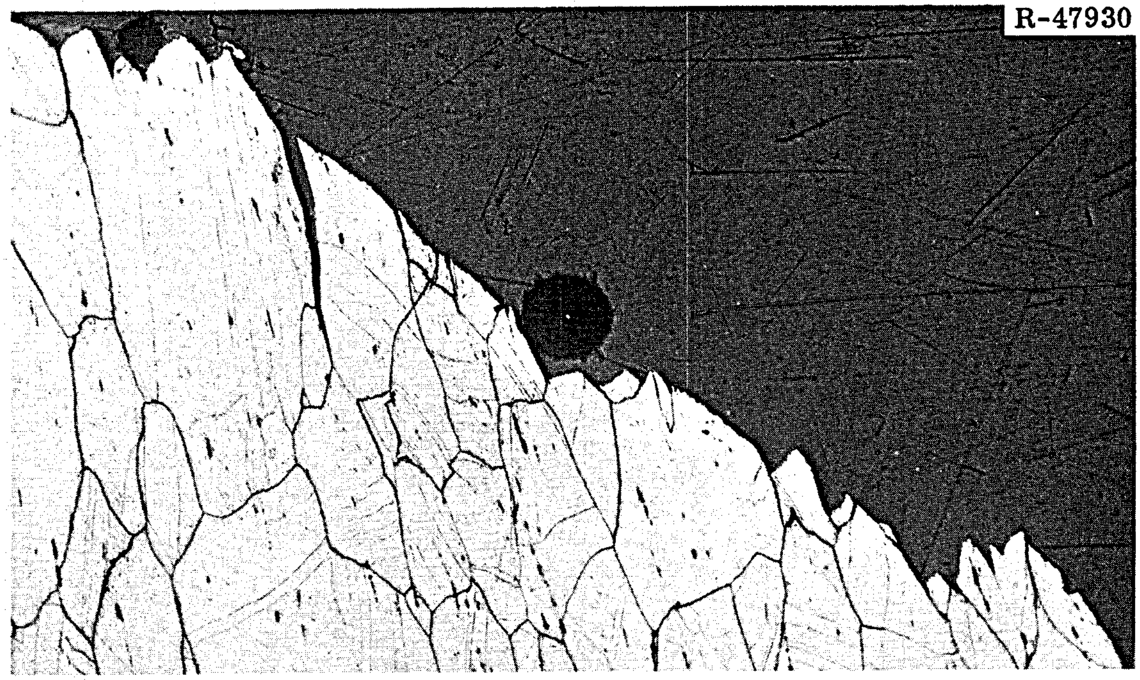


Fig. 97. Fracture of a Sample of Heat 67-504 (0.43% Hf) Annealed 1 hr at 1177°C, Irradiated in the Molten Salt Reactor Experiment to a Thermal Fluence of 5.3×10^{20} neutrons/cm^2 over a period of 9800 hr at 650°C, and Tested in Tension at 25°C. Etchant: glyceria regia. 100×.

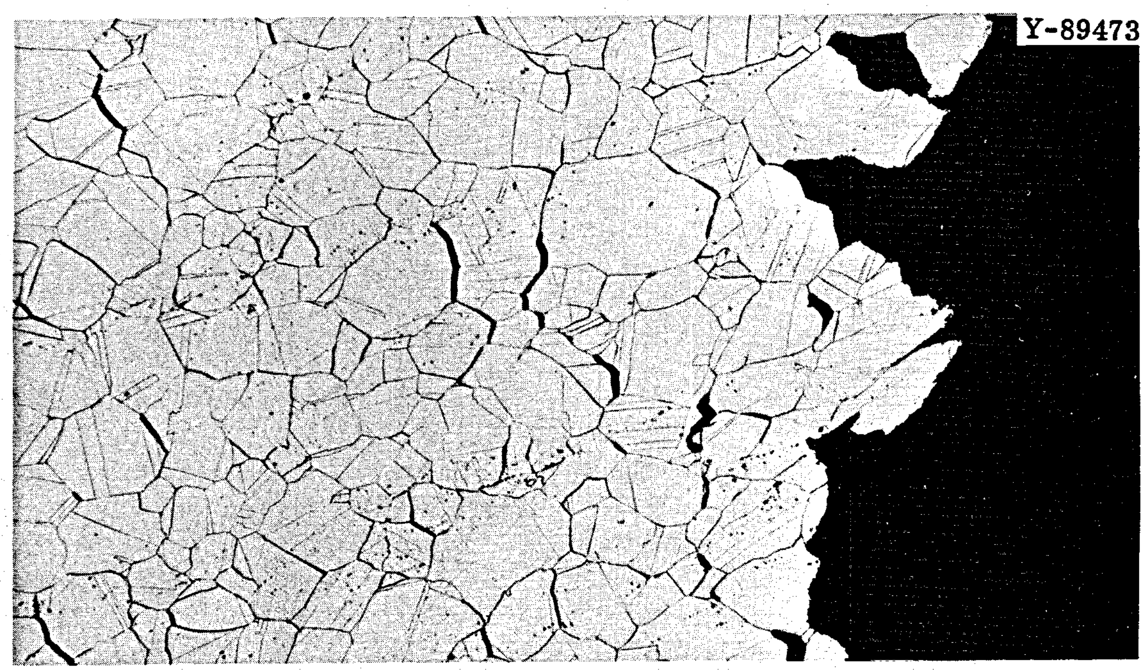


Fig. 98. Fracture of a Sample of Heat 67-503 (0.08% Hf) Annealed 1 hr at 1177°C and Creep Tested at 650°C and 40,000 psi. Etchant: glyceria regia. 100×.

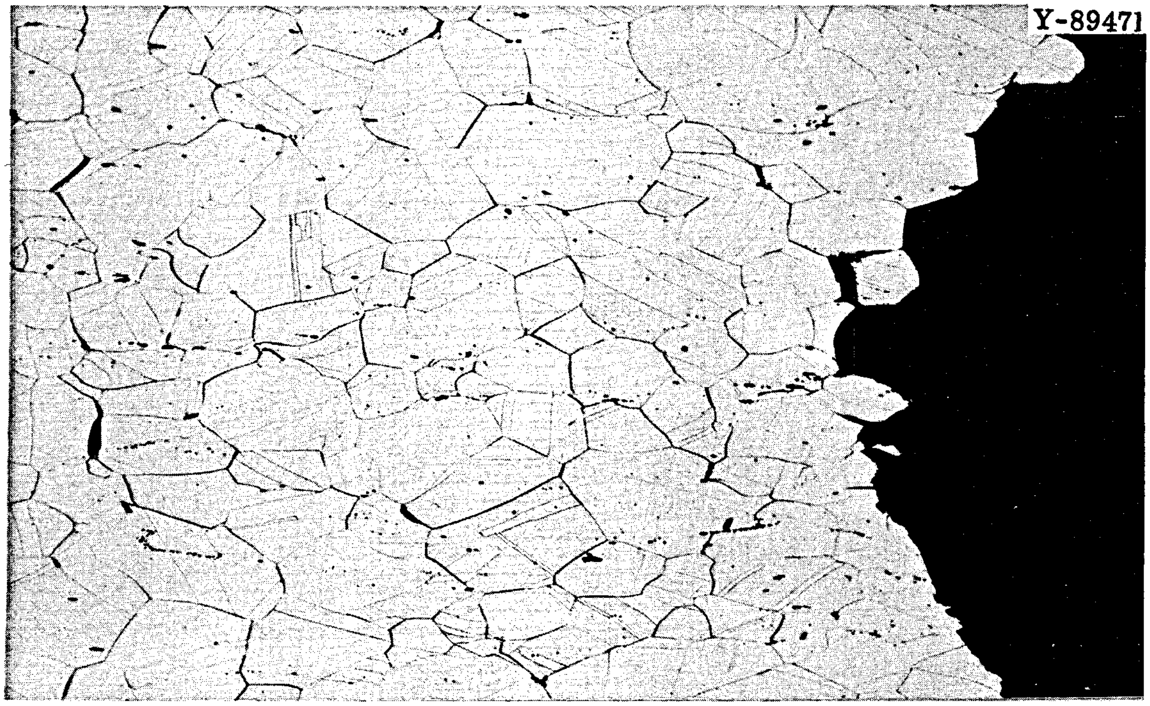


Fig. 99. Fracture of a Sample of Heat 67-504 (0.50% Hf) Annealed 1 hr at 1177°C and Creep Tested at 650°C and 40,000 psi. Etchant: glyceria regia. 100×.

Alloy Containing No Additions

The results for a small, 2-lb laboratory melt (alloy 100) that contained no additions of Ti, Zr, or Hf were presented above. One small, 100-lb commercial alloy (heat 21546) was also procured. This alloy was received in the cold-worked condition. Samples were tested in this condition and after annealing at 871 and 1177°C. Typical photomicrographs of the material are shown in Figs. 100 and 101. The microstructure after annealing at 871°C was characterized by bands of precipitate and fine grains. The bands were caused by carbide precipitation and retarded recrystallization in these areas. The material outside these bands was recrystallized. Annealing for 1 hr at 1177°C (Fig. 101) completed recrystallization. Some stringers were still present, although none are visible in Fig. 101. These stringers were quite similar to those found in heat 21545 and appeared to be unmelted molybdenum.

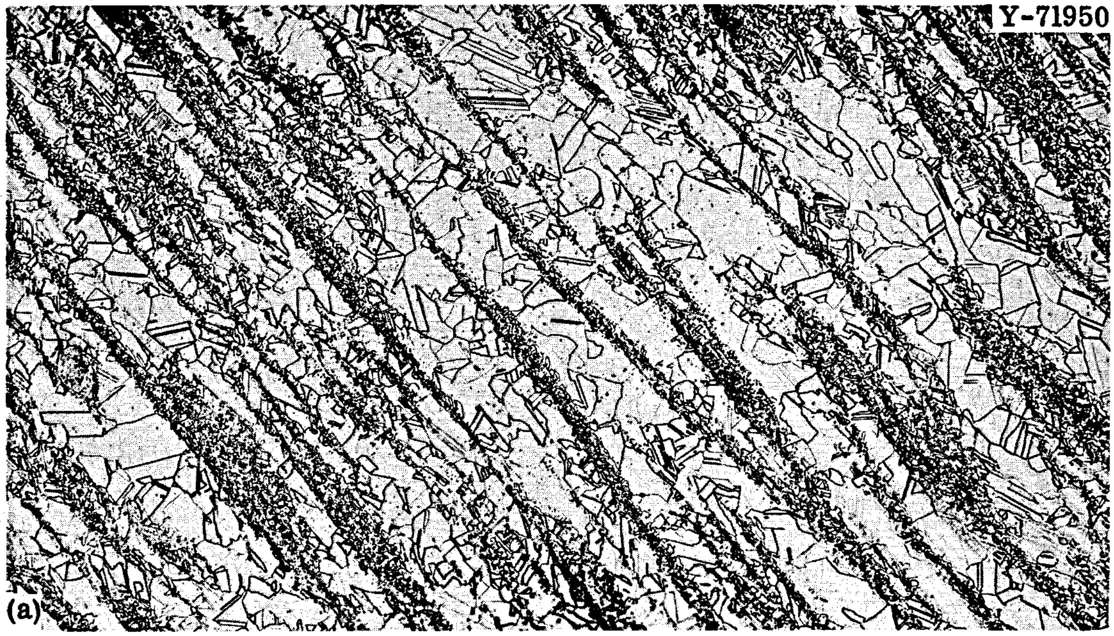
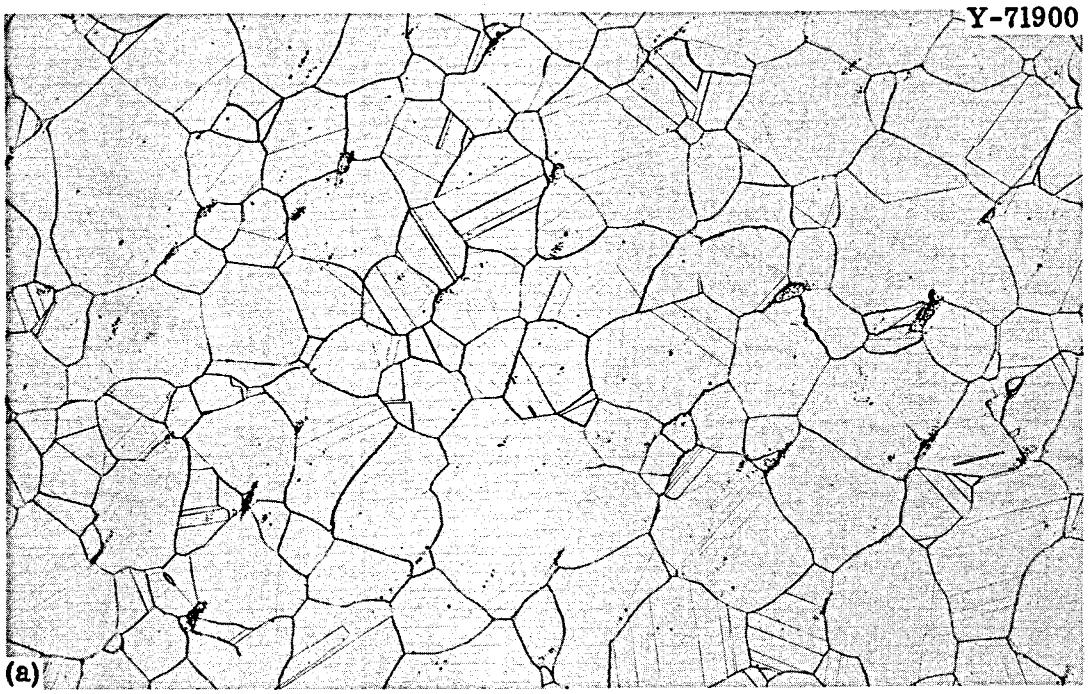


Fig. 100. Photomicrographs of Alloy 21546 Annealed 100 hr at 871°C. The material was cold worked 40% before annealing. (a) 100× and (b) 500×. Etchant: glyceria regia.



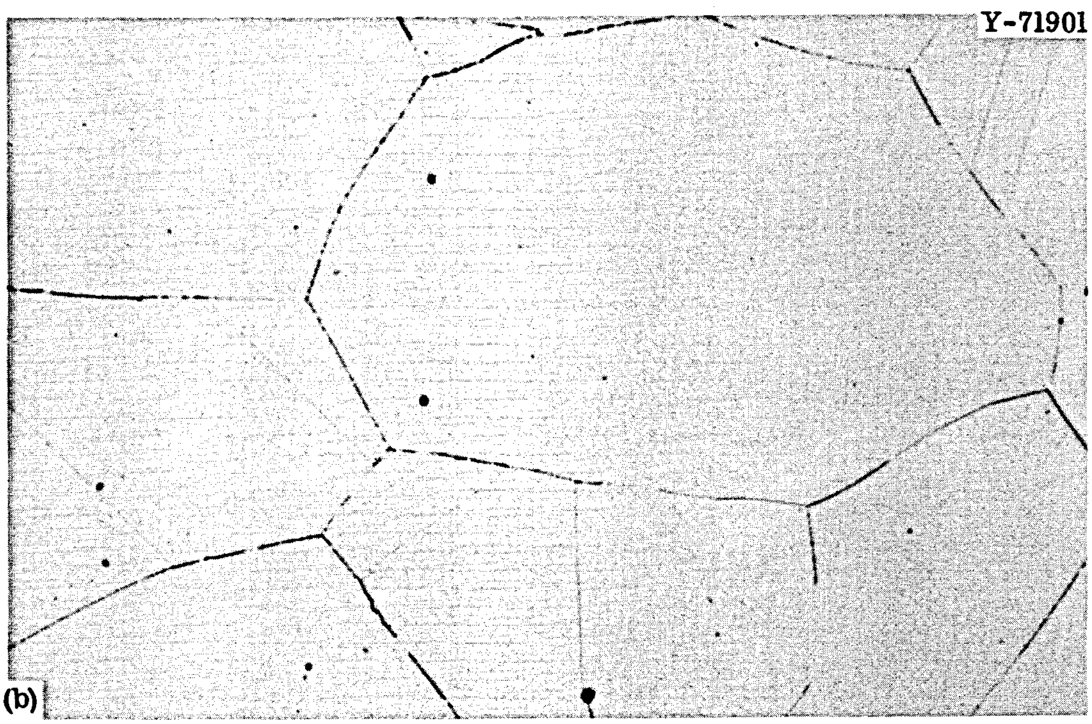


Fig. 101. Photomicrographs of Alloy 21546 Annealed 1 hr at 1177°C. The material was cold worked 40% before annealing. (a) 100× and (b) 500×. Etchant: glyceria regia.

The results of tensile tests on this material are summarized in Table 39. The fracture strains are shown as a function of temperature in Fig. 102. Up to about 700°C, the material annealed 1 hr at 1177°C had the highest fracture strain and the cold-worked material had the lowest fracture strain. Above 700°C, the ductility of the cold-worked material increased markedly. Only samples as received and as annealed 100 hr at 871°C were irradiated, and these were all tested at 600°C and above. Irradiation decreased the fracture strain of all samples.

The results of creep tests on this material are given in Table 40. In the unirradiated condition, the material as received had the highest strength and the material annealed at 871°C had the lowest strength. The strength of material annealed 1 hr at 1177°C fell intermediate, and several illustrations follow to compare the properties of irradiated and unirradiated material annealed 1 hr at 1177°C. The stress-rupture properties at 650°C are shown in Fig. 103. The irradiation temperature was an important factor; the higher the irradiation temperature, the shorter was the rupture life. The two sets of data seem to converge, indicating that at very low levels of stress there would be no effect of irradiation on the rupture life. The creep rates are shown in Fig. 104 for 650°C. As long as the irradiation temperature was about 650°C, there was no difference between the creep rates of irradiated and unirradiated material. At irradiation temperatures of 700°C and above, the postirradiation creep rate was increased. There was at least one datum point that disagreed with this general trend. These curves also converged, again indicating common behavior at low stresses.

The fracture strains of irradiated samples tested at 650°C are shown as a function of minimum creep rate in Fig. 105. All of the samples irradiated above 700°C had fracture strains of 1.1 to 0.2%. Irradiation at about 650°C resulted in fracture strains of 1.1 to 5.1%. The exact dependence of the fracture strain on strain rate cannot be determined from the available data, and the indicated curve is influenced by results on standard Hastelloy N (ref. 8).

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[8] H. E. McCoy, "Variation of Mechanical Properties of Irradiated Hastelloy N with Strain Rate," J. Nucl. Mater. 31(1), 67–85 (1969).

Table 39. Tensile Properties of Irradiated and Unirradiated Alloy 21546

Specimen Number	Anneal[a] Before Irradiation	Temperature, °C Irradiation	Temperature, °C Test	Thermal-Neutron Fluence (neutrons/cm²)	Strain Rate (min⁻¹)	Stress, psi Yield	Stress, psi Ultimate Tensile	Elongation, % Uniform	Elongation, % Total	Reduction in Area (%)
				× 10²⁰		× 10³	× 10³			
10220	121		25		0.05	38.7	107.5	69.6	73.7	75.1
10221	121		200		0.05	32.6	99.7	69.8	73.8	63.3
10223	121		260		0.05	31.4	100.9	71.5	76.4	68.0
10222	121		400		0.05	30.2	97.8	73.0	79.3	64.8
10224	121		550		0.05	27.1	90.5	73.4	78.3	59.5
10225	121		600		0.05	27.5	87.1	66.0	69.5	47.2
10226	121		650		0.05	23.7	69.8	46.3	48.3	32.1
10227	121		700		0.05	24.4	64.7	35.1	36.1	26.9
10228	121		760		0.05	24	60.4	25.0	25.9	20.0
1440	000		25		0.05	151.9	164.2	6.8	13.4	53.3
1441	000		427		0.05	127.3	144.2	12.1	15.5	32.7
1452	000		650		0.05	119.5	132.7	7.7	13.6	18.8
1453	000		650		0.002	109.1	114.8	3.4	8.2	16.3
1451	000		760		0.05	76.4	78.4	2.6	32.3	51.1
1457	000		871		0.05	31.5	31.7	1.4	46.7	58.4
1458	000		871		0.002	19	19	1.0	48.1	44.0
1459	000		982		0.05	17.9	17.9	0.9	60.8	60.6
1749	16		25		0.05	51.7	119.4	49.2	53.5	72.4
1750	16		427		0.05	40.9	105.2	47.9	52.4	46.2
1746	16		650		0.05	44.3	95.5	27.8	30.0	31.2
1747	16		650		0.002	38.8	69	19.0	35.1	64.7
1752	16		760		0.05	37.8	51.5	3.6	7.5	68.2
1753	16		871		0.05	27.7	28.8	10.0	53.5	69.1
1754	16		871		0.002	18.2	18.2	0.9	45.3	51.2
1438	000	600	550	3.5	0.002	115.2	127.9	6.0	6.4	7.1
1439	000	600	600	3.5	0.002	105.3	115	4.4	4.5	4.0
1436	000	600	650	3.5	0.05	106.8	116.6	5.7	6.2	8.6
1437	000	600	650	3.5	0.002	103.1	106.8	3.5	3.7	4.0
1447	16	600	550	3.5	0.002	41.9	74.8	14.8	15.1	16.2
1446	16	600	600	3.5	0.002	43.7	65.6	8.2	8.4	10.2
1444	16	600	650	3.5	0.05	38.8	64.2	10.3	10.5	11.7
1443	16	650	650	1.0	0.002	38.6	55	7.3	7.4	12.9
1442	16	650	650	1.0	0.05	38.4	67.5	13.5	13.7	14.7
1445	16	600	650	3.5	0.002	45	56.7	5.6	5.7	8.6
1448	16	600	760	3.5	0.002	33.5	33.8	1.8	4.3	7.1

[a]Annealing designations: 121 = annealed 1 hr at 1177°C; 000 = as received (40% cold work); 16 = annealed 100 hr at 871°C.

Table 40. Creep Properties of Irradiated and Unirradiated Alloy 21546

Specimen Number	Test Number	Anneal[a] Before Irradiation	Temperature, °C: Irradiation	Temperature, °C: Test	Thermal-Neutron Fluence (neutrons/cm²)	Stress (psi)	Rupture Life (hr)	Minimum Creep Rate (%/hr)	Fracture Strain (%)	Reduction in Area (%)
					$\times 10^{20}$	$\times 10^{3}$				
4056	6300	121		650		62	5.3	0.56	25.6	24.9
4057	6301	121		650		55	18.5	0.22	21.3	21.3
4058	6302	121		650		47	63.0	0.074	18.1	16.6
4059	6303	121		650		40	187.5	0.026	19.4	15.6
10219	7288	121		650		35	256.2	0.013	13.0	16.2
10218	7289	121		650		30	561.6	0.0080	11.1	18.7
1455	5528	000		650		70	11.7	0.31	12.5	15.5
1454	5527	000		650		55	92.7	0.025	12.5	14.0
1456	5408	000		650		47	223.6	0.014	7.5	6.3
1751	5531	16		650		70	1.1	18.0	34.4	31.0
1748	5536	16		650		55	6.9	2.87	46.9	33.7
1745	5532	16		650		47	31.7	0.83	46.9	39.6
1826	5435	816		650		40	28.8	0.20	8.5	9.4
10231	7521	121		760		15	221.3	0.08	16.3	15.8
10229	7517	121		760		12.5	414.7	0.0142	13.4	12.7
5988	R-598	121	235	650	2.4	47	84.5	0.011	2.3	
4060	R-294	121	650	650	2.5	47	10.1	0.41	5.1	
4061	R-482	121	657	650	2.5	40	0			
4065	R-481	121	650	650	2.5	40	9.2	0.019	2.4	
5981	R-635	121	666	650	2.4	40	62.0	0.033	2.8	
5980	R-604	121	666	650	2.4	35	20.1	0.042	1.1	
5979	R-642	121	766	650	2.4	35	0.4	2.1	0.9	
5974	R-602	121	754	650	2.4	35	0			
5965	R-605	121	732	650	2.4	35	0.4	1.3	0.6	
5975	R-607	121	760	650	2.4	30	1.2	0.44	0.8	
5966	R-609	121	704	650	2.4	30	1.1	0.15	0.7	
5982	R-643	121	666	650	2.4	27	727.8	0.0030	3.1	
5967	R-638	121	732	650	2.4	25	46.6	0.0065	0.4	
5969	R-639	121	816	650	2.4	25	11.1	0.015	0.4	
5985	R-600	121	499	650	2.4	25	9.4	0.26	3.2	
5976	R-631	121	760	650	2.4	21.5	68.3	0.0039	0.53	
5968	R-632	121	832	650	2.4	21.5	69.6	0.0028	0.24	
5971	R-637	121	871	650	2.4	17	1157.1	0.0006	1.1	
5985	R-600	121	499	760	2.4	25	9.4	0.26	3.1	
5972	R-640	121	799	760	2.4	15	14.3	0.061	1.3	
4066	R-296	121	746	760	2.5	15	0.5	0.12	0.7	
4067	R-468	121	746	760	2.5	12.5	3.6	0.060	0.4	
5970	R-634	121	832	760	2.4	12.5	45.7	0.014	0.8	
5973	R-641	121	799	760	2.4	10	110.9	0.0098	1.5	
4068	R-469	121	746	760	2.5	10	13.8	0.036	1.1	
1450	R-120	16	600	650	3.5	40	3.3	0.40	1.3	
1449	R-113	16	600	650	3.5	32.4	97.7	0.038	4.0	

[a]Annealing designations: 121 = annealed 1 hr at 1177°C; 000 = as received (40% cold work) 16 = annealed 100 hr at 871°C; 816 = annealed 8 hr at 871°C.

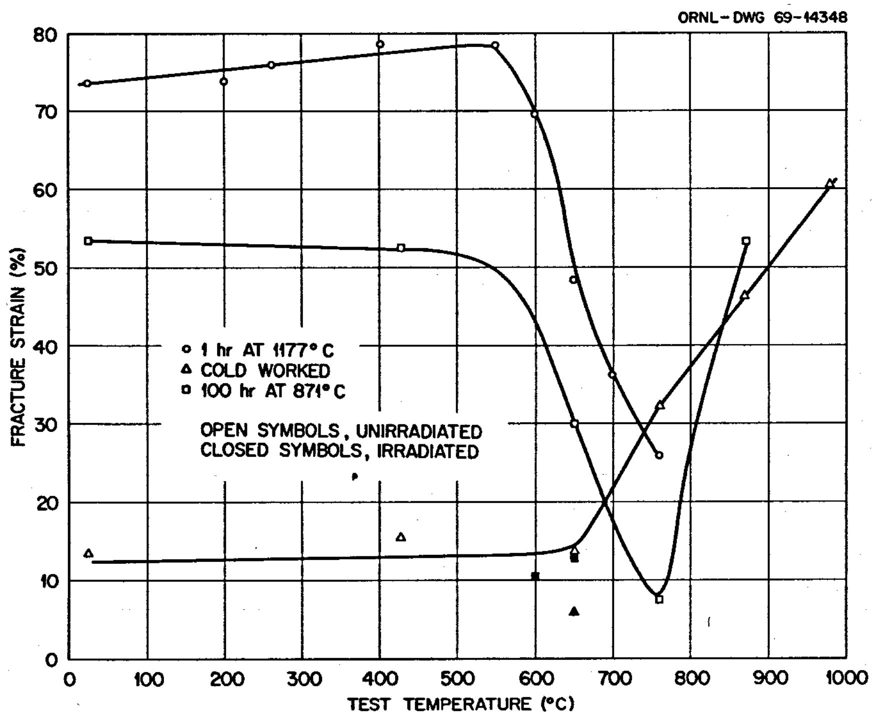

Fig. 102. Variation of the Fracture Strain with Test Temperature for Alloy 21546 at a Strain Rate of 0.05 min^{-1}.

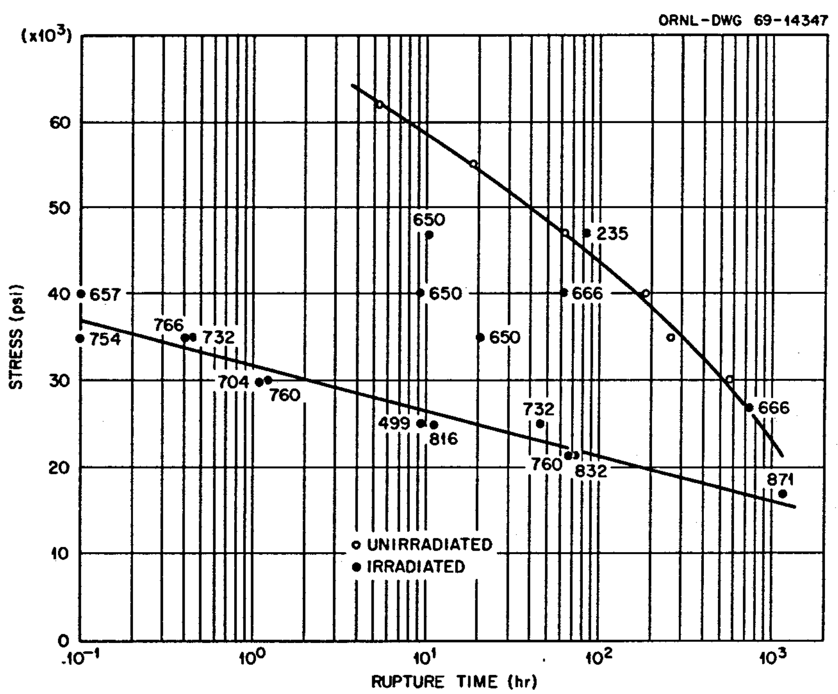

Fig. 103. Stress-Rupture Properties at 650°C of Alloy 21546. All samples annealed 1 hr at 1177°C before testing. Number by each datum point indicates irradiation temperature.

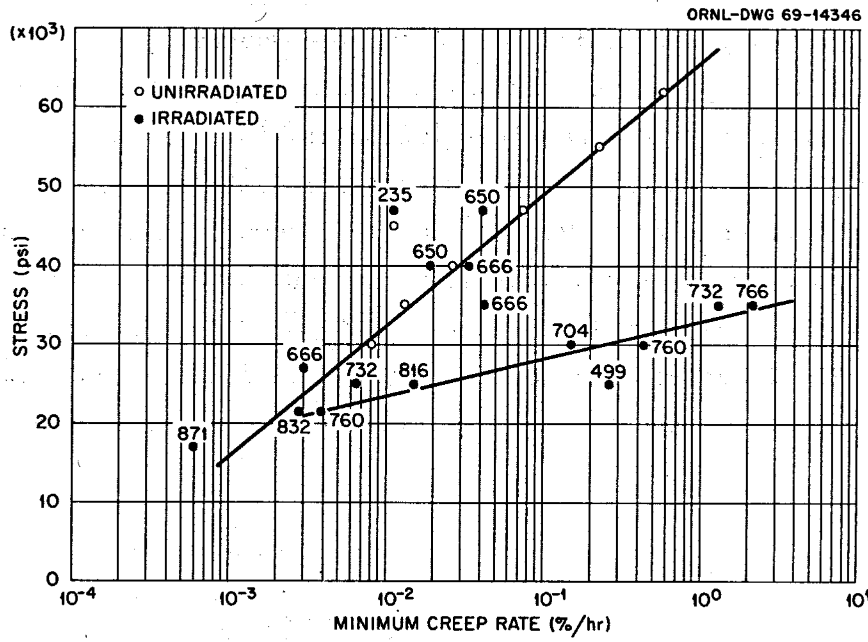

Fig. 104. Creep Rate at 650°C of Alloy 21546. All samples annealed 1 hr at 1177°C before testing. Number by each datum point indicates irradiation temperature.

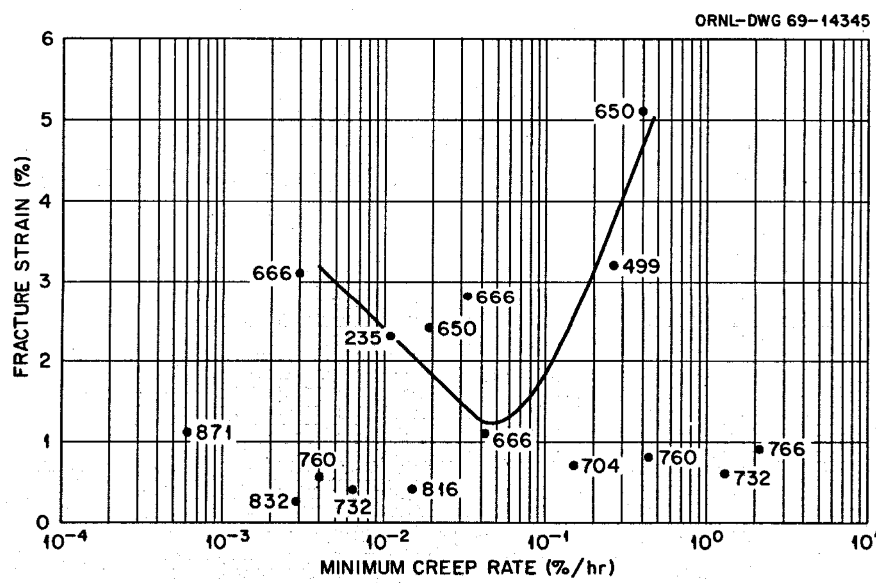

Fig. 105. Fracture Strains at 650°C of Irradiated Alloy 21546. All samples annealed 1 hr at 1177°C before testing. Number by each datum point indicates irradiation temperature.

Some samples were tested at 760°C; the rather sparse results are given in Table 40, and the stress-rupture data are shown in Fig. 106. There was an indication that irradiation at 746°C may result in poorer stress-rupture properties at 760°C than irradiation at 799 to 832°C. The minimum creep rate at 760°C (Fig. 107) showed that irradiation did not alter the creep rate drastically. However, there was again the tendency for the three samples irradiated at 746°C to show the greater effect. The fracture strains at 760°C were all quite low (0.4 to 1.5% strain) except for the sample irradiated at 499°C (3.1% strain).

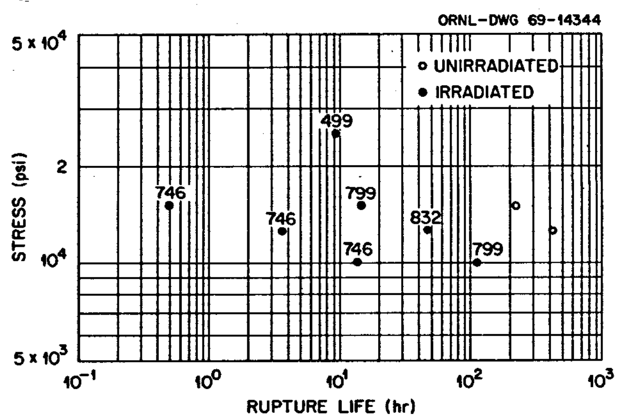

Fig. 106. Stress-Rupture Properties at 760°C of Alloy 21546. All samples annealed 1 hr at 1177°C. Number by each datum point indicates irradiation temperature.

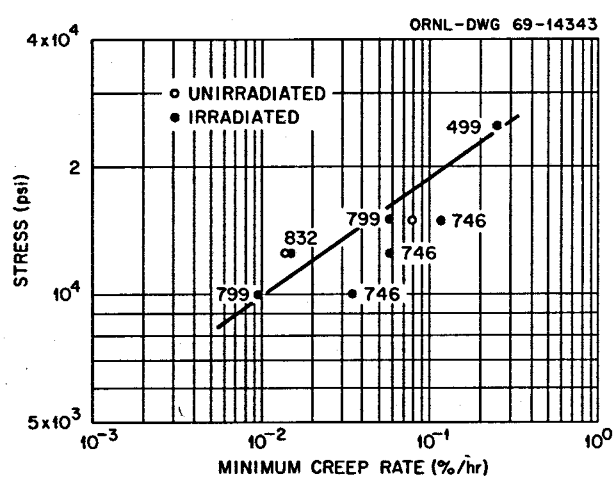

Fig. 107. Creep Rates at 760°C of Alloy 21546. Material annealed 1 hr at 1177°C. Number by each datum point indicates irradiation temperature.

Some insight into the effect of irradiation temperature was gained by transmission electron microscopy. Typical microstructures for two samples irradiated at 666 and 766°C are shown in Figs. 108 and 109.

Fig. 108. Transmission Electron Photomicrograph of Alloy 21546 after Irradiation at 666°C for about 1100 hr to a Thermal-Neutron Fluence of 2.4×10^{20} neutrons/cm^2. 50,000×.

Fig. 109. Transmission Electron Photomicrograph of Alloy 21546 after Irradiation at 766°C for about 1100 hr to a Thermal-Neutron Fluence of 2.4×10^{20} neutrons/cm^2. 50,000X.

After irradiation at 666°C, the grain boundaries were completely lined with fine precipitate particles, and there were also numerous precipitates within the grains. After irradiation at 766°C (Fig. 109), there were fewer relatively coarse precipitates along the grain boundaries, and only random coarse precipitates were present within the grains. The precipitates in both samples were identified as carbides of the M_2C type in which the metal component was primarily molybdenum. Thus, the poorer postirradiation properties are associated with the very coarse precipitate.

A typical fracture of heat 21546 at 650°C is shown in Fig. 110. There was extensive intergranular cracking, and the fracture was entirely intergranular. The random stringers are obvious in this field and may have been instrumental in causing the fracture at this particular location.

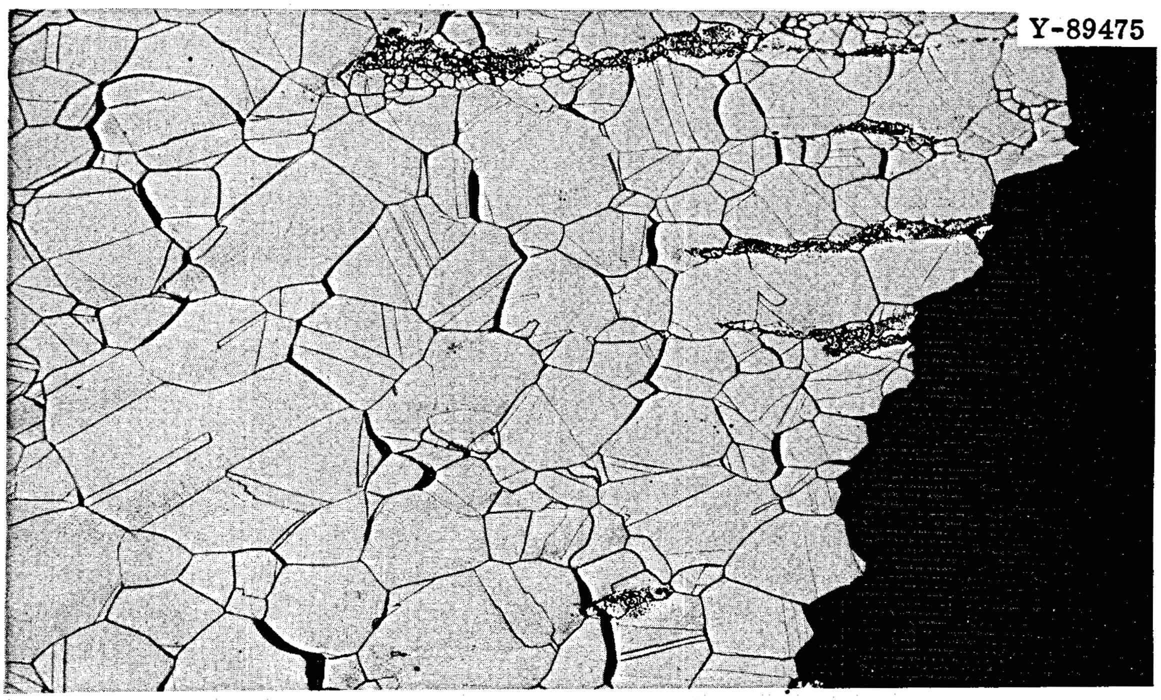

Fig. 110. Fracture of a Sample of Heat 21546 Annealed 1 hr at 1177°C and Tested at 40,000 psi and 650°C. Etchant: glyceria regia.

DISCUSSION OF RESULTS

A large amount of data has been presented, and we should recapitulate some of the most important observations.

1. A modified base composition of Hastelloy N was developed: Ni–12% Mo–7% Cr–0.2% Mn–0.05% C. This alloy is free of the carbide stringers present in standard Hastelloy N, but is still very susceptible to embrittlement by irradiation.

2. Additions of Ti, Zr, or Hf to this base composition improved resistance to irradiation embrittlement. Small heats showed a general improvement with increasing additions of titanium and hafnium. Similar work with additions of zirconium showed that an alloy containing 0.53% Zr had the minimum creep rate and maximum rupture life. Other concentrations of zirconium resulted in higher fracture strains.

3. Commercial melts (100 lb) were obtained with nominal additions of 0.5% each of Ti, Zr, and Hf. The properties of these alloys were comparable with those of the small, laboratory melts (2 lb).

4. Properties were very dependent upon the irradiation temperature. Irradiation at about 650°C resulted in good properties, but irradiation at 700°C and above gave relatively poor properties.

5. Transmission electron microscopy revealed that the effect of irradiation temperature was associated with the precipitation of coarser carbides at the higher temperature. During irradiation at 650°C, fine carbides precipitated, whereas above 700°C relatively coarse carbides formed.

6. The optimum properties after irradiation result from annealing for 1 hr at 1177°C before irradiation. The fine-grained microstructure obtained by annealing at low temperatures such as 871°C was not as strong or as ductile, and annealing above 1177°C decreased the fracture strain of irradiated and unirradiated samples.

7. The general trend revealed by metallographic examination after testing was that additions of Ti, Zr, and Hf reduced the frequency of cracking at grain boundaries. Fractures in these alloys in creep at 650°C are predominantly intergranular but usually have some transgranular components.

Some comparisons are in order to show the relative properties of these alloys and standard Hastelloy N. For this comparison, we used only the commercial alloys with 0.5% nominal additions of Ti, Zr, and Hf. All results are for samples annealed 1 hr at 1177°C. The tensile

and yield strengths are shown in Fig. 111. Alloys 21546 and 21545 (0.49% Ti) had very similar properties; the observed strengths were slightly less than those measured for standard Hastelloy N. The strength of heat 67-504 (0.50% Hf) was the highest of all alloys studied. The fracture strains of these samples are shown in Fig. 112. Alloys 21546, 21545 (0.49% Ti), 21554 (0.35% Zr), and 67-504 (0.50% Hf) have similar properties. Heat 67-504 (0.50% Hf) after aging for 9800 hr at 650°C had fracture strains lower than those of the other modified alloys when tested below about 650°C and higher above this temperature. Standard Hastelloy N had lower fracture strains than any of the modified alloys.

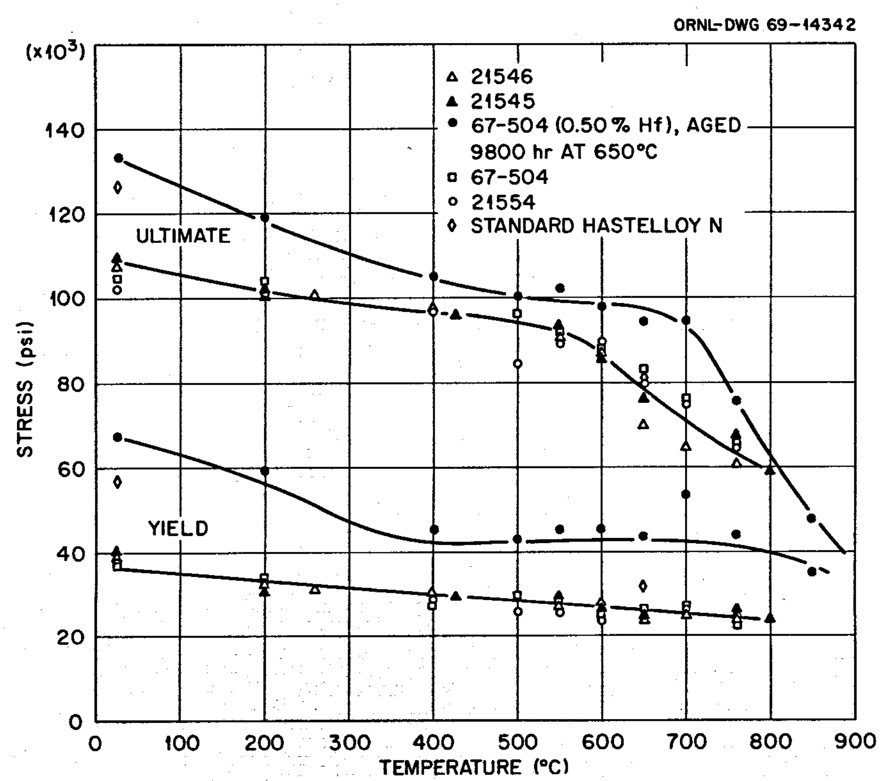

Fig. 111. Tensile Properties of Several Commercial Melts of Modified Alloys. All material annealed 1 hr at 1177°C.

The relative stress-rupture properties of these alloys at 650°C are shown in Fig. 113. The rupture lives at 40,000 psi range from 170 to 1100 hr; alloy 21546 had the shortest rupture life, and alloy 67-504 (0.50% Hf) had the longest. Minimum creep rate is a better measure of

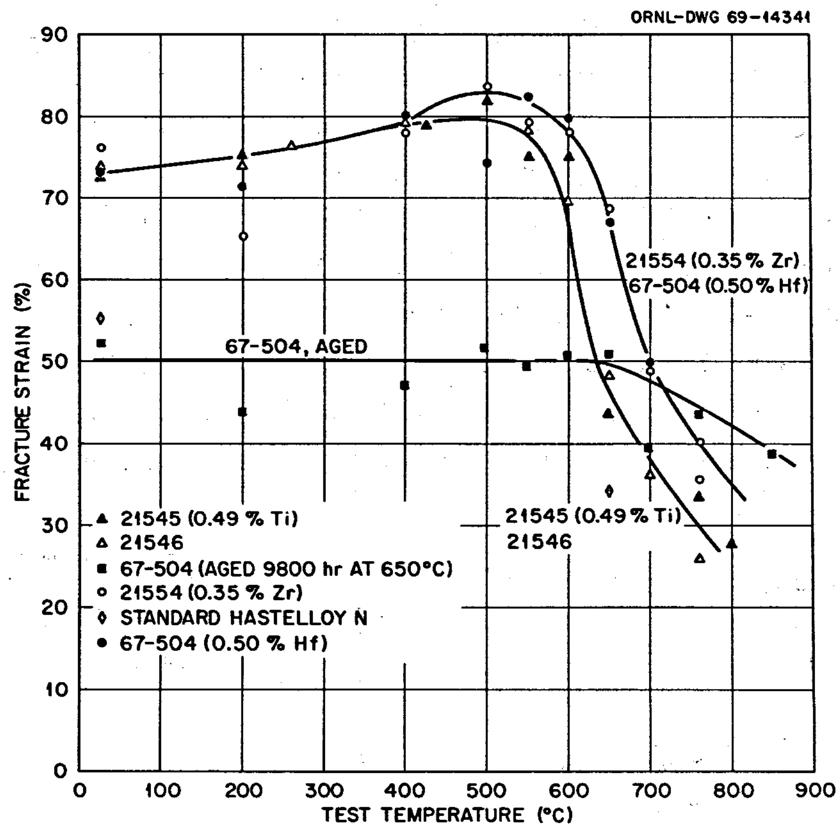

Fig. 112. Variation of Tensile Fracture Strain with Test Temperature for Several Commercial Melts of Modified Alloys at a Strain Rate of 0.05 min^{-1}. All material annealed 1 hr at 1177°C.

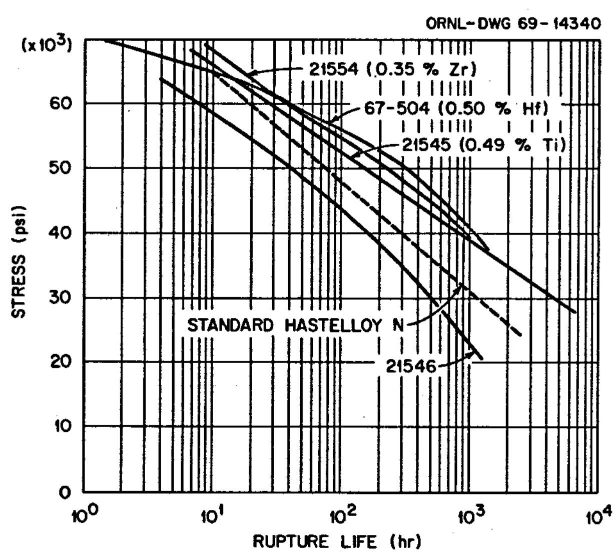

Fig. 113. Stress-Rupture Properties at 650°C of Several Commercial Melts of Modified Alloys. All material annealed 1 hr at 1177°C before testing.

strength than rupture life; these data are shown in Fig. 114. At 40,000 psi, the minimum creep rate varied from 0.03%/hr for alloy 21546 to 0.002%/hr for alloy 67-504 (0.50% Hf). The addition of hafnium increased strength the most, by far, but the three alloys modified with Hf, Zr, and Ti were all stronger than standard Hastelloy N. There was not much difference between the creep strengths of standard Hastelloy N and the modified base alloy 21546. The fracture strains of the unirradiated modified alloys are shown in Fig. 115. All of the alloys except alloy 21545 (0.49% Ti) followed the trend of decreasing fracture strain with decreasing creep rate. At high strain rates, alloy 21554 (0.35% Zr) had the highest fracture strain. At low strain rates, alloy 21545 (0.49% Ti) had superior ductility. Alloy 21546 and standard Hastelloy N had the lowest fracture strains and did not differ appreciably from each other.

The stress-rupture properties determined at 650°C after irradiation are compared in Fig. 116. After irradiation at 650°C, alloys 21554

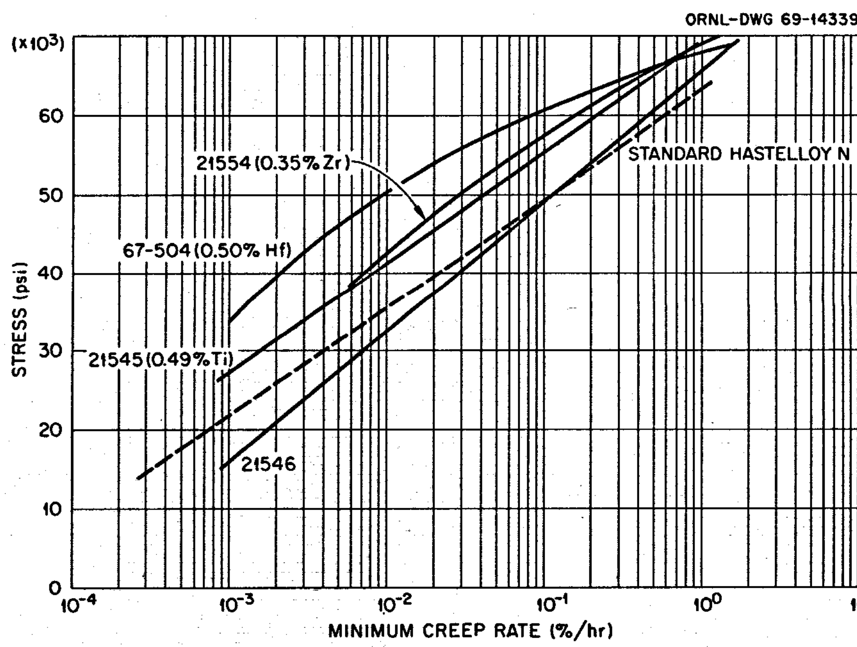

Fig. 114. Creep Rates at 650°C of Several Commercial Melts of Modified Alloys. All material annealed 1 hr at 1177°C before testing.

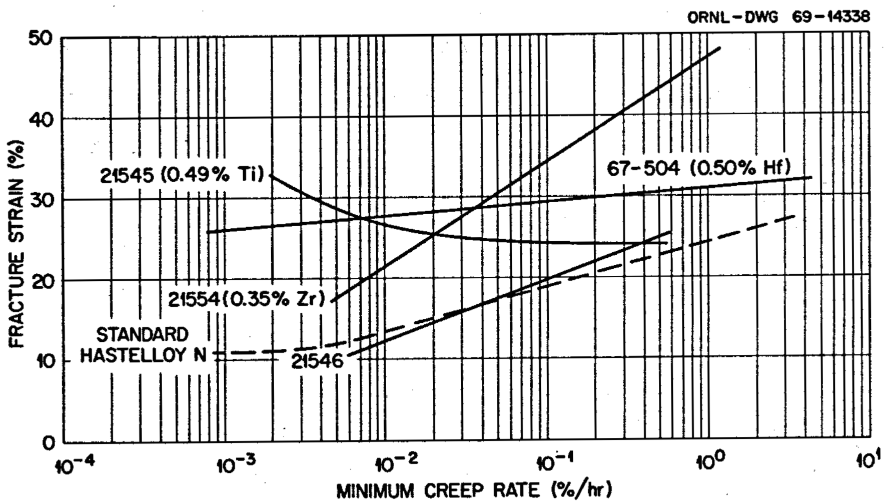

Fig. 115. Comparison of the Fracture Strains at 650°C of Several Commercial Melts of Modified Alloys. All material annealed 1 hr at 1177°C.

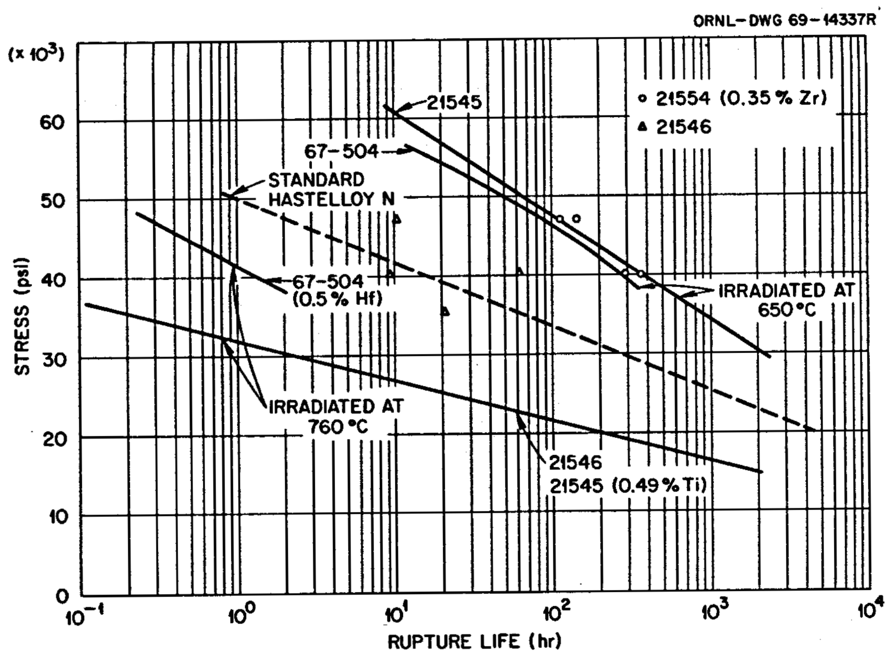

Fig. 116. Postirradiation Stress-Rupture Properties at 650°C of Several Commercial Melts of Modified Alloys. All material annealed 1 hr at 1177°C before irradiation.

(0.35% Zr), 21545 (0.49% Ti), and 67-504 (0.50% Hf) had equally good properties. All three of the modified alloys showed improvement over the standard Hastelloy N shown for reference. The base modified alloy 21546 had properties quite similar to those of standard Hastelloy N.

Standard Hastelloy N that contained about 0.5% Si is not sensitive to irradiation temperature, and the indicated line holds for irradiation at 760°C (ref. 9). Heats 21546 and 21545 (0.49% Ti) both had poor stress-rupture properties after irradiation at 760°C. The stress-rupture properties of alloy 67-504 (0.50% Hf) fell intermediate between those of these two alloys and those of standard Hastelloy N.

The postirradiation fracture strains for irradiation and testing at 650°C are summarized in Fig. 117. These curves are based on rather sparse data and should be considered as schematics. Alloy 21546 shows some improvement over standard Hastelloy N. Alloy 21545 (0.49% Ti) is generally better than standard Hastelloy N, but still has a distinct ductility minimum of only about 3%. The sparse data for heats 21554 (0.35% Zr) and 67-504 (0.50% Hf) indicate that these heats had improved fracture strains. The presence of a ductility minimum has been established for standard Hastelloy N and for heat 21545, but there are too few data to establish whether the other alloys conform to this pattern.

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[9] H. E. McCoy, An Evaluation of the Molten-Salt Reactor Experiment Hastelloy N Surveillance Specimens — Third Group, ORNL-TM-2647 (1970).

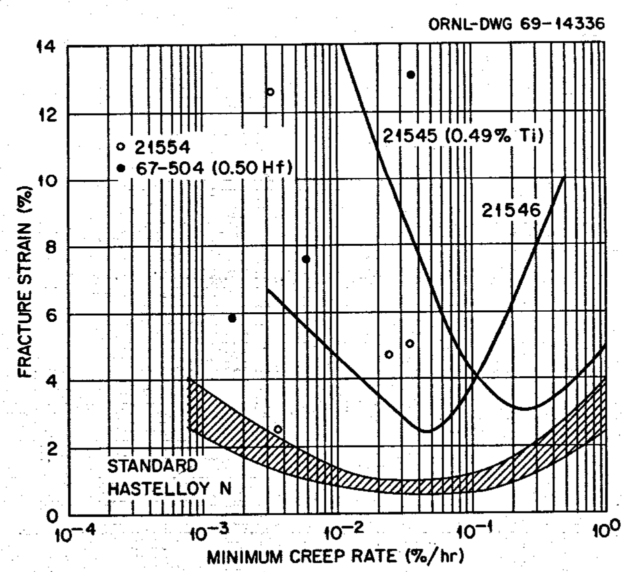

Fig. 117. Postirradiation Fracture Strains of Several Commercial Melts of Modified Alloys Irradiated and Tested at 650°C. All material annealed 1 hr at 1177°C before irradiation.

Many of the properties of these alloys are likely determined by the carbide precipitate. As already shown, alloy 21546 exhibited a change in properties after irradiation that correlated well with changes in the carbide structure (Figs. 108 and 109). Further work by Gehlbach and Cook[10] showed that Ti, Hf, and Zr promoted the formation of the MC type of carbides. The normal type of carbide for Hastelloy N is M_2C when the silicon content is low and M_6C when the silicon content is high.[11] The work of Gehlbach and Cook[10] also showed that low levels (about 0.5%) of Ti, Hf, and Zr can result in an MC type of carbide at 650°C and a relatively coarse M_2C type at 760°C. Thus, the strong effect of irradiation temperature on these alloys is associated with this transition in carbide types.

The mechanism whereby these precipitates improve the unirradiated creep properties also likely has to do with the carbide precipitates.[12] As shown in Fig. 114, the creep strength is increased with the addition of Ti, Zr, and Hf. These effects are larger than anticipated from solid-solution strengthening, particularly when one considers that most of the alloying additions are likely tied up as carbides. Thus, the strengthening observed is likely due to the finely dispersed carbide phase. These precipitates also likely impede crack propagation and account for the improved fracture strains (Fig. 115).

In irradiated material, the further complication of helium transmuted for the ^{10}B exists. Our reasoning in adding Ti, Hf, and Zr was to tie the boron up in dispersed precipitates. The only measure that we have of the location of boron is to assume that it behaves like the carbon. Thus, each carbide precipitate contains some boron. The finely

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[10] R. E. Gehlbach and S. W. Cook, Metals and Ceramics Div. Ann. Progr. Rept. June 30, 1969, ORNL-4470, p. 187.

[11] R. E. Gehlbach and H. E. McCoy, Jr., "Phase Instability in Hastelloy N," pp. 346–366 in International Symposium on Structural Stability in Superalloys, Seven Springs, Pennsylvania, September 4–6, 1968, Vol. II. Available from Dr. John Radavich, AIME High-Temperature Alloys Committee, Micromet Laboratories, West Lafayette, Indiana.

[12] C. E. Sessions, Influence of Titanium on the High-Temperature Deformation and Fracture Behavior of Some Nickel Based Alloys, ORNL-4561 (July 1970).

divided MC type of carbide gives a better dispersion and would be expected to give better properties. Thus, the qualitative observation that the fine MC type of precipitates are associated with good properties and the coarse M_2C type with poor porperties seems quite reasonable. The further role of these precipitates in inhibiting propagation of cracks may also be important.

All of the modified alloys are slightly susceptible to aging during long-term annealing. These changes are quite small and are associated with precipitation of carbides. The general effects of aging are that the minimum creep rate and fracture strain increase and the rupture life decreases. Because of the difference in the type of carbide precipitated at 650 and 760°C in many of these alloys, aging at 760°C seems to produce larger changes in properties. The precipitation of copious quantities of ZrC in alloys that contain less than 0.5% Zr is quite dramatic, but the changes in properties are minimal.

SUMMARY

This first phase of this study of the effects of additions of Ti, Zr, and Hf to a base alloy of Ni–12% Mo–7% Cr–0.2% Mn–0.05% C dealt with small, laboratory melts and the first 100-lb commercial melts. The results show that these three elements improve the creep strength and fracture strain in unirradiated alloys. Tests on samples irradiated at 650°C or less show that the rupture lives at a given stress level and the fracture strains are improved markedly. Irradiation at 760°C results in very poor properties. The good properties are associated with the formation of a very fine MC type of carbide and the poor properties with a very coarse M_2C type of carbide.

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