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**EFFECT OF TITANIUM ADDITIONS ON THE STRESS-RUPTURE
PROPERTIES OF TYPE 304 STAINLESS STEEL**

E. E. Bloom

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EFFECT OF TITANIUM ADDITIONS ON THE STRESS-RUPTURE
PROPERTIES OF TYPE 304 STAINLESS STEEL

E. E. Bloom

ABSTRACT

The effect of small additions of titanium on the stress-rupture properties of types 304 and 304L stainless steel has been determined. Stress-rupture tests were performed on rod specimens at 704 and 815°C. For tests in the solution-treated condition, titanium increased the rupture life and decreased the minimum creep rate by approximately an order of magnitude. This increase in strength was attributed to the locking of dislocations during creep and was demonstrated by the appearance of small precipitates on dislocations. When carbon was removed from solution by prior precipitation, dislocation locking did not occur and the minimum creep rate was increased.

The titanium-bearing steels failed by a mixture of transgranular and intergranular fracture whereas type 304 stainless steel fractured intergranularly.

INTRODUCTION

Martin and Weir¹ have reported that AISI type 304 stainless steel can be made more resistant to elevated-temperature irradiation embrittlement by the addition of small amounts of titanium. The titanium level at which maximum postirradiation ductility was observed, approximately 0.2 wt %, is below the level required to meet the chemical specifications for type 321 stainless steel. The primary purpose of this investigation was to determine the manner in which small titanium additions influence the stress-rupture properties of the alloy.

¹W. R. Martin and J. R. Weir, "Solutions to the Problems of High-Temperature Irradiation Embrittlement," ORNL-TM-1544 (June 1966).

Freeman and White² reported that rupture strengths of type 321 stainless steel increased with temperature of heat treatment up to a temperature governed by the carbon content and then remained nearly constant. Their data indicated that the stress-rupture strength at 650°C was governed mainly by the amount of titanium and carbon in solution before testing. Miyoshi³ has investigated an 18-8 titanium stainless steel containing 0.4 wt % Ti. Samples were solution treated at temperatures between 900 and 1300°C and then aged at 750°C. Creep rates were increased as a result of carbide precipitation during aging; however, it was found that for higher solution annealing temperatures this aging process was slower. Barnby⁴ has shown that the creep strength of type 316 stainless steel can be attributed to strain aging during creep. The creep rate of type 316 stainless steel in which $(\text{FeCr})_{23}\text{C}_6$ was removed from solution by prior precipitation was 20 to 30 times greater than that of solution-treated material. The increased strength of solution-treated material was attributed to precipitates formed on dislocations thus preventing their movement.

Garofalo et al.⁵ reported that prestraining a type 316 stainless steel at room temperature followed by treating at 482 and 704°C resulted in the precipitation of M_{23}C_6 particles at dislocation sites. At 704°C and 20,000 psi the minimum creep rate decreased and rupture life increased with increasing prestrain up to 30 to 35%, beyond which the effects were reversed.

²J. W. Freeman and J. E. White, "Metallurgical Principles Governing the Creep-Rupture Strength of Type 321 Austenitic Steel Superheating Tubing with Limited Extension to Type 304 and Type 316 Austenitic Steels," J. Eng. Power 85, 119 (1963).

³E. Miyoshi, "Creep Characteristics as Related to Heat-treatment and Microstructures in Stainless Steel Stabilized by Titanium," J. Japan Inst. Metals 28, 604 (1964).

⁴J. T. Barnby, "Effect of Strain Aging on Creep of an AISI 316 Austenitic Stainless Steel," J. Iron Steel Inst. (London) 204, 23 (1966).

⁵F. Garofalo, F. Von Gemmingen, and W. F. Domis, "The Creep Behavior of an Austenitic Stainless Steel as Effected by Carbides Precipitated on Dislocations," Trans. Am. Soc. Metals 54, 430 (1961).

EXPERIMENTAL PROCEDURE

The chemical composition of the three heats of stainless steel used in this investigation is given in Table 1. Heat 33107 is AISI type 304 stainless steel; heat D-6 is type 304 stainless steel with 0.15 wt % Ti; and heat D-9 is type 304L stainless steel with 0.14 wt % Ti. All material was received as 0.50-in.-diam hot-rolled rod, which was swaged to 0.375 in. in diameter, annealed 1 hr at 1038°C, and swaged to 0.25 in. in diameter. Creep specimens of the design shown in Fig. 1 were machined from the

Table 1. Chemical Composition of Experimental Materials

Material and Heat Number	Chemical Content, wt %							
	C	Mn	Si	Cr	Ni	P	S	Ti
304 stainless steel, Heat 33107	0.057	0.76	0.48	18.0	9.0	0.020	0.016	
304 stainless steel plus titanium, Heat D-6	0.053	1.53	0.70	19.0	10.3	0.006	0.004	0.15
304L stainless steel plus titanium, Heat D-9	0.014	1.54	0.72	18.3	10.0	0.005	0.005	0.14

ORNL-DWG 67-3013

Fig. 1. Creep Specimen Design.

0.25-in.-diam rod. All heat treatments were carried out in an argon atmosphere. Dead-load creep machines within which an argon atmosphere was maintained were used for all stress-rupture tests. Test temperatures were measured by thermocouples attached to the gage length of the test specimen. Creep strain, as a function of time, was measured by means of a dial gage in contact with the pull rod.

RESULTS

Properties of Solution-Treated Material

Specimens of each of the three heats were solution treated for 1 hr at 1038°C and tested at 704 and 815°C at stresses sufficient to produce rupture in 1 to 2000 hr. Grain sizes, measured by the linear intercept method, are given in Table 2.

Table 2. Grain Size Developed as a Result of Solution Treatment

Heat Number	ASTM Grain Size	Average Grain Diameter (in.)
33107	5.5	0.0021
D-6	5.0	0.0025
D-9	3.5	0.0042

Each of the titanium-bearing alloys contained precipitates that were pink to orange in color and were not dissolved at temperatures up to 1300°C. Such precipitates have been identified by Comstock⁶ as being titanium carbonitrides.

Experimental data are listed in Table 3. Figures 2 and 3 show the stress-rupture life relationships for each test temperature. The effect

⁶G. F. Comstock, Titanium in Iron and Steel, pp. 49-55, Wiley, Inc. New York, 1955.

Table 3. Stress-Rupture Properties of Type 304 Stainless Steel Alloys

Stress (psi)	Temperature (°C)	Minimum Creep Rate (%/hr)	Rupture Life (hr)	Elongation (%)	Reduction in Area (%)
AISI Type 304 Stainless Steel (Heat 33107)					
30,000	704	15.7	1.7	44.9	35.8
27,000	704	8.8	3.0	51.9	33.8
20,000	704	0.9	11.7	18.7	19.9
17,000	704	0.35	52.2	28.1	24.8
15,000	704	0.116	105	26.6	19.9
12,000	704	0.0185	619	26.3	33.1
10,000	704	0.0041	2217	15.0	13.0
8,000	704	0.0035			
14,000	815	20.4	1.5	49.0	30.7
12,000	815	5.75	4.6	42.7	32.7
10,000	815	1.20	16.9	32.1	11.2
8,000	815	0.84	25.9	28.5	22.5
7,500	815	0.28	95.0	29.7	20.3
6,000	815	0.047	348	28.1	22.8
5,000	815	0.0145	735	18.1	13.8
4,500	815	0.0057			
Type 304 Stainless Steel Containing 0.15 wt % Ti (Heat D-6)					
30,000	704	18.4	1.7	66.8	78.3
25,000	704	0.124	19.7	23.9	65.9
20,000	704	0.043	302	36.3	44.5
20,000	704	0.082	197	38.4	43.8
15,000	704	0.0063	2150	22.7	31.3
10,000	704	0.00089	9880	25.5	39.9
18,000	815	138	0.3	80.3	80.6
15,000	815	36.5	1.4	93.7	82.1
12,000	815	5.75	8.9	72.0	72.4
9,000	815	0.575	60.0	81.3	50.1
7,500	815	0.072	248	51.3	42.6
6,000	815	0.0042	2298	22.0	24.4
5,000	815	0.0037			
3,500	815	0.0002			
Type 304 Low-Carbon Stainless Steel Containing 0.14 wt % Ti (Heat D-9)					
30,000	704	43.5	1.1	65.4	43.1
25,000	704	19.4	1.5	52.2	72.3
20,000	704	2.3	19.0	69.9	69.4
17,500	704	0.43	53.2	45.6	33.0
15,000	704	0.042	362	39.1	44.3
15,000	704	0.079	145	31.5	32.3
12,000	704	0.0057	1272	21.7	39.3
15,000	815	70.0	0.5	58.9	93.6
13,500	815	25.0	2.0	83.4	76.7
12,000	815	77.0	7.1	77.0	81.0
9,000	815	0.31	82.0	45.6	42.1
7,000	815	0.092	320.0	53.5	45.4
6,000	815	0.051	495	47.6	38.0
6,000	815	0.011	1206	23.2	13.6
4,000	815	0.0017			

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Fig. 2. Stress-Rupture Properties at 704°C after Solution Treatment of 1 hr at 1038°C.

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Fig. 3. Stress-Rupture Properties at 815°C after Solution Treatment of 1 hr at 1038°C.

of titanium upon rupture life is best illustrated by comparing heats D-6 and 33107 since each had essentially the same carbon content and grain size. The addition of 0.15 wt % Ti increased the rupture life by a factor of 5 to 10.

Graphs of stress versus minimum creep rate are shown in Figs. 4 and 5. The addition of titanium to type 304 stainless steel decreased the minimum

Fig. 4. Effect of Stress upon Minimum Creep Rate at 704°C after Solution Treatment at 1038°C.

Fig. 5. Effect of Stress on Minimum Creep Rate at 815°C after Solution Treatment at 1038°C.

creep rate by approximately an order of magnitude when tested at 704°C. The low-carbon, titanium-bearing alloy had a strength comparable to type 304 stainless steel at high applied stresses; however, at lower stresses the minimum creep rate approached that of the high-carbon, titanium-bearing alloy. At 815°C the strengths of the three alloys as measured by minimum creep rate were approximately equal.

No significant differences in ductility were observed at 704°C between the titanium-modified alloys and type 304 stainless steel. At

815°C both the low- and high-carbon titanium-bearing alloys exhibited ductilities that were a factor of 1.5 to 2.0 greater than type 304 stainless steel.

Metallography

Metallography of fractured specimens indicates that titanium additions affect the deformation and fracture characteristics of type 304 stainless steel. Photomicrographs of type 304 stainless steel tested at 704 and 815°C are shown in Fig. 6. At both test temperatures wedge-type cracks were formed on grain boundaries normal to the applied load; the resulting fracture was intergranular with little deformation occurring within the matrix. Carbides precipitated primarily on grain boundaries at 704°C; while at 815°C, precipitation occurred both on grain boundaries and within the matrix.

Failures of both the high- and low-carbon, titanium-bearing steels, shown in Figs. 7 and 8, were a mixture of transgranular and intergranular fracture. The number of wedge-type cracks in the titanium-bearing alloys was significantly lower than that present in type 304 stainless steel. It was also noted that the two alloys containing titanium recrystallized in the region near the fracture under suitable conditions of temperature and stress. Heat D-6 did not recrystallize when tested at 704°C; at 815°C the fraction of material that recrystallized depended upon the applied stress. Figure 9 shows three specimens tested at 815°C and three different stress levels. More recrystallization occurred in the specimen tested at 12,000 psi than at either 9,000 or 15,000 psi.

In the low-carbon, titanium-bearing alloy (heat D-9) a small amount of recrystallization had occurred in specimens tested at 704°C. The fracture area of a specimen tested at 17,500 psi is shown in Fig. 10. At the same temperature and a lower stress, little recrystallization occurred (see Fig. 11); however, grain boundaries appear to have migrated during the test and small voids or cavities have formed on many of the grain boundaries. When tested at 815°C, little recrystallization occurred and grain boundary migration was quite evident. An example was shown previously in Fig. 8.

Fig. 6. Fractures in Type 304 Stainless Steel (Heat 33107).
(a) Tested at 704°C and 12,000 psi; rupture life 620 hr. (b) Tested at 815°C and 5,000 psi; rupture life 735 hr.

Fig. 7. Fractures in Type 304 Stainless Steel Containing 0.15 wt % Ti (Heat D-6). (a) Tested at 704°C and 20,000 psi; rupture life 320 hr. (b) Tested at 815°C and 7,500 psi; rupture life 95 hr.

Fig. 8. Fractures in Type 304L Stainless Steel Containing 0.14 wt % Ti (Heat D-9). (a) Tested at 704°C and 15,000 psi; rupture life 362 hr. (b) Tested at 815°C and 7,000 psi; rupture life 320 hr.

Fig. 9. Type 304 Stainless Steel Containing 0.15 wt % Ti (Heat D-6) Tested at 815°C and Different Stress Levels. (a) Tested at 9,000 psi. (b) Tested at 12,000 psi. (c) Tested at 15,000 psi.

Fig. 10. Type 304L Stainless Steel Containing 0.14 wt % Ti (Heat D-9) Tested at 704°C and 17,500 psi. Note recrystallization that has occurred adjacent to the fracture

The dislocation structures formed in solution-treated specimens during creep at 704°C and 15,000 psi are shown in Figs. 12 and 13. In type 304 stainless steel the cell structure is well defined with individual dislocations and dislocation tangles remaining within the cells; precipitation occurred mainly at grain boundaries with an occasional massive precipitate within the matrix.

In the titanium-bearing alloys the cell structure is also well established. The significant difference between type 304 stainless steel and the titanium-bearing stainless steel lies in the precipitate formed during creep deformation. As can be seen in Fig. 13, a general dispersion of precipitate particles has formed throughout the matrix of the titanium-bearing alloy. Nearly all dislocation lines have precipitate particles attached to them.

Fig. 11. Type 304 Stainless Steel Containing 0.14 wt % Ti (Heat D-9) Tested at 704°C and 12,000 psi. Both wedge-type voids and small rounded cavities are present.

Fig. 12. Transmission Micrographs of Type 304 Stainless Steel Solution Treated 1 hr at 1038°C and Tested at 704°C and 15,000 psi. 25,000X.

Fig. 13. Transmission Micrograph of Type 304L Stainless Steel Containing 0.14 wt % Ti (Heat D-9) Solution Treated 1 hr at 1038°C and Tested at 704°C and 15,000 psi. 25,000X.

Properties of Aged Materials

Specimens of each of the three alloys were annealed for 1 hr at 1038°C and then aged for various times at 600 and 700°C. The effect of such aging treatments upon stress-rupture properties was investigated at 704°C and stresses of 15,000 and 20,000 psi. Data for the tests are given in Table 4. The effect of aging at 600 and 700°C upon the minimum creep rate is shown graphically in Figs. 14 and 15.

The minimum creep rate of type 304 stainless steel was increased slightly by the aging treatment. Ductilities were improved as a result of aging at either temperature; however, the greatest improvement occurred after aging 1000 hr at 700°C.

The response of the two titanium-bearing steels to aging was dependent upon carbon content. The low-carbon alloy was not as sensitive to aging as was the high-carbon alloy. Aging the low-carbon alloy 1000 hr at 700°C

Table 4. Effect of Aging Upon the Stress-Rupture Properties of Stainless Steel Alloys at 704°C

Annealing History	Stress (psi)	Heat 33107 ^a			Heat D-6 ^b			Heat D-9 ^c		
		Minimum Creep Rate (%/hr)	Rupture Life (hr)	Total Elonga- tion (%)	Minimum Creep Rate (%/hr)	Rupture Life (hr)	Total Elonga- tion (%)	Minimum Creep Rate (%/hr)	Rupture Life (hr)	Total Elonga- tion (%)
1 hr at 1038°C	15,000	0.116	105	26.6	0.0063	2150	22.7	0.043	362	39.1
	20,000	0.9	11.7	18.7	0.043	302	36.3	2.3	19.0	69.9
1 hr at 1038°C plus 100 hr at 600°C	15,000	0.108	117	20.2	0.0064	2064	26.3	0.079	145	31.5
	20,000	1.06	14.4	25.4	0.117	104	37.4	2.34	16.8	56.0
1 hr at 1038°C plus 1000 hr at 600°C	15,000	0.161	110	32.3	0.291	95.2	63.1	0.143	252	54.6
	20,000	0.52	36.2	29.1	0.452	60.1	60.3	3.40	14.1	75.1
1 hr at 1038°C plus 100 hr at 700°C	15,000	0.228	94.9	32.4	0.014	678	33.7	0.114	185	46.8
	20,000	2.94	8.95	54.9	1.21	21.5	53.3	3.65	12.4	58.6
1 hr at 1038°C plus 1000 hr at 700°C	15,000	0.165	199	59.4	0.226	127	69.6	0.257	152	66.2
	20,000	1.08	42.5	51.4	2.82	13.2	58.9	3.46	11.3	59.8

^aType 304 stainless steel.

^bType 304 stainless steel with addition of 0.15 wt % Ti.

^cType 304L stainless steel with 0.14 wt % Ti.

Fig. 14. Effect of Aging at 600°C Upon the Minimum Creep Rate at 704°C for Type 304 Stainless Steel Alloys.

Fig. 15. Effect of Aging at 700°C Upon the Minimum Creep Rate at 704°C for Type 304 Stainless Steel Alloys.

increased the minimum creep rate by a factor of from 2 to 4 while a similar aging treatment increased the minimum creep rate of the high-carbon alloy by a factor of 15 to 100.

Ductilities of the titanium-bearing alloys were improved by the aging treatment. In general the increase in ductility was greater for either the longer aging times (at a given temperature) or higher temperatures (for a given aging time).

The significant microstructural change that occurred as a result of aging was the precipitation of carbides at grain boundaries. After 1000 hr at 600 or 700°C, type 304 stainless steel and the high-carbon, titanium-bearing alloy contained precipitates such as those shown in Fig. 16a. The low-carbon, titanium-bearing alloy contained precipitates that were similar in size but fewer in number with a larger interparticle spacing as shown in Fig. 16b.

Fractures of the three alloys given a pretest aging were essentially unchanged from those of specimens tested at the same stress and temperature, but in the solution-treated condition. Fractures in the type 304 stainless steel were intergranular while those in the titanium-modified alloys were a mixture of transgranular and intergranular.

The dislocation structure formed during creep of a type 304 stainless steel specimen that was given a pretest aging of 1000 hr at 700°C is shown in Fig. 17. The number of dislocations remaining within the cells is considerably less than that present in the same material tested in the solution-treated condition (Fig. 12).

Aging the titanium-bearing steels for 1000 hr at 700°C and then creep testing at 704°C result in a structure shown in Fig. 18. The pretest aging treatment has removed carbon and titanium from solution thus preventing the formation of precipitates throughout the matrix as was observed in the specimens tested in the solution-treated condition.

Fig. 16. Grain-Boundary Precipitates Formed During Aging 1000 hr at 600°C; (a) Heat D-6 and (b) Heat D-9.

Fig. 17. Type 304 Stainless Steel Aged 1000 hr at 700°C and Tested at 704°C and 15,000 psi.

Fig. 18. Type 304L Stainless Steel Containing 0.14 wt % Ti Aged 1000 hr at 700°C and Tested at 704°C and 15,000 psi. 50,000X.

DISCUSSION

The addition of titanium to type 304 stainless steel decreased the minimum creep rate and increased the rupture life by approximately an order of magnitude when tested at 704°C in the solution-treated condition. This behavior is believed to be due to a strengthening mechanism similar to that observed by Barnby⁷ in AISI type 316 stainless steel.

In the titanium-bearing steels small precipitates were observed throughout the matrix after creep testing at 704°C (Fig. 13). In most cases the precipitates were associated with dislocations. This precipitation phenomenon was not observed in regular type 304 stainless steel. Aging the titanium-bearing alloys increased the minimum creep rate by a factor of 2 to 4 for the low-carbon alloy and 15 to 100 for the high-carbon alloy. The dependence of the creep rate upon the amount of carbon in solution was shown qualitatively in Figs. 14 and 15, and this is in agreement with Barnby's conclusion that strengthening occurs as a result of the locking of dislocations by the small precipitate particles. This was verified by transmission microscopy of specimens of titanium-modified steel creep tested in the aged condition. In this case no precipitates other than those at grain boundaries were observed.

When tested in the solution-treated condition the ductility of the titanium-bearing steels was not significantly different from that of type 304 stainless steel. It was observed, however, that the titanium-bearing steels are not as subject to grain-boundary crack formation as type 304 stainless steel and that the titanium-bearing steels fracture by a mixture of transgranular and intergranular paths whereas type 304 stainless steel fractures intergranularly. When the titanium-bearing steels were given a pretest aging treatment to remove carbon from solution the fracture ductilities were significantly increased. This observation is also in agreement with the precipitate strengthening mechanism. As a result of the pretest aging treatment, precipitates

⁷J. T. Barnby, "Effect of Strain Aging on Creep of an AISI 316 Austenitic Stainless Steel," J. Iron Steel Inst. (London) 204, 23 (1966).

that act as barriers to dislocation motion are not formed during creep deformation; and the amount of strain due to dislocation motion within the matrix is increased.

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