REPORT

ON

HIGH TEMPERATURE PROPERTIES OF MANGANESE MODIFIED 16-25-6 ALLOY

By

R. Jackowski J. W. Freeman

PROJECT 842 REPORT 207

October 25, 1954

THE TIMKEN ROLLER BEARING COMPANY STEEL AND TUBE DIVISION CANTON, OHIO

INTRODUCTION

15-25-6 alloy as developed by the Timken Roller Bearing Company is widely used in the rotor disks of jet engines. In view of the possible use of the alloy for this purpose under high production conditions in case of war, it would be desirable to conserve the amount of strategic alloy if otherwise suitable properties could be developed. This investigation was undertaken to establish the influence, on properties at high temperature, of reducing the strategic nickel content by substituting manganese for part of the nickel. Manganese stabilizes the structure of such alloys much as does nickel and therefore appeared to be a logical substitute.

Specimens were furnished from a heat in which the manganese was raised from the usual 1.8 percent to about 7 percent and the nickel reduced from 25 to 15 percent. Thus about 5 percent of manganese was substituted for 10 percent of the nickel.

Creep and rupture tests were conducted at 1100°, 1200°, 1300°, and 1400°F to evaluate the influence of change in composition. The material was tested in two conditions of treatment, solution treated at 2150°F and hot rolled and stress relieved.

CONCLUSIONS

The creep and rupture data for the manganese modification of 16-25-6 alloy when compared to similar data for the standard alloy led to the following conclusions:

1. The substitution of 5 percent Mn for 10 percent nickel did not alter the rupture and creep strengths from the standard alloy when both were solution quenched from 2150°F.

2. The hot rolled and stress relieved material was considerably weaker than standard 16-25-6 alloy at similar high hardness values. This, however, could well have been due to differences in prior working conditions. Thus the probability is that the manganese modification probably does have similar properties to the standard alloy as indicated by the solution quenched condition. Such solution treatments would largely remove any effects from variations in prior working.

TEST MATERIALS

The material for this investigation was supplied in the form of 1-1/2 by 3/4 inch bars 7 inches long from a one-ton heat (Heat 02400) having the following reported analysis:

One set of bars was hot rolled and then tempered for 15 hours at 1300°F to a hardness of 280 Brinell. The other set, solution treated at 2150°F, had a hardness of 176/183 Brinell. The grain size of the hot rolled and of the solution treated material were 8 and 3/5 respectively.

Standard 0.505-inch diameter specimens were used for stresses less than 40,000 psi. For higher stresses, 0.400-inch diameter specimens were used to bring the load requirements within the capacity of the creep-rupture units.

RESULTS

Stress-rupture time curves were established at 1100° through 1400°F.

Testing times for these curves were extended to 10,000 hours at 1200° and

1300°F, while testing was terminated at about 1000 to 3000 hours at

1100° and 1400°F.

Rupture Test Data

The stress-rupture time curves of Figures 1 and 2 for the two conditions were plotted from the test data in Table I. It will be noted that tests of 7,000 to 10,000 hours duration were conducted at 1200° and 1300°F for the solution treated condition, Figure 1. The longest test at 1300°F for the hot rolled condition, Figure 2, was 2400 hours. Tests of 2000 to 3000 hours were conducted at 1400°F. Only two tests were run at 1100°F for both conditions to give an indication of the level of strengths at this temperature. The extrapolation of the stress-rupture time curves are based on the best indications of the data. All of the curves at 1200°, 1300°, and 1400°F underwent a change in slope between 300 and 1500 hours. The data are too incomplete to be certain whether a similar change in slope occurs at 1100°F. Prolonged extrapolations were not carried out at 1100°F, since it appears highly probable that a break might also occur at 1100°F. The stresses for rupture in time periods of 100, 1000, 10,000 and 100,000 hours derived from Figures 1 and 2 are given in Table II. Estimated elongations at fracture are included in this table where they could be obtained from the data.

The rupture strength - temperature curves of Figure 3 show that the solution quenched condition had the highest strength at the higher temperatures and longer time periods. The hot rolled condition was superior only to 1250°F for 100 hours and to 1200°F for 1000 hours.

Both conditions had high ductility in the rupture tests out to 1000 hours with the hot rolled being generally somewhat higher. The solution treated samples, however, decreased more in the longer time tests at 1200° and 1300°F so that as low values as 5 percent elongation resulted for fracture in 10,000 hours.

Creep Test Data

Time-elongation curves for the tests at 1200°, 1300°, and 1400°F are shown in Figures 4 through 7. The hot-rolled condition had considerably lower creep resistance than the solution treated condition. This is evident from the time-elongation curves, the tabulation of observed creep rates at indicated time periods in Table III, and the derived curves of time versus creep rate of Figures 8, 9, and 10. The solution treated condition underwent more primary creep than the hot rolled material, but had a lower and more prolonged second stage of creep. The duration of third stage creep tended to be equal to or greater than the total time in first and second stages of creep, particularly at 1300° and 1400°F.

The stress-second stage creep rate curves, Figures 11 and 12, at 1200°, 1300°, and 1400°F, for the modified 16-25-6 alloy, although not completely established, indicate that the stresses for 0.001 percent per hour and for 0.1 and 0.01 percent per thousand hour creep rates are as follows:

	${f Temp}$	Stres	s for Indicated Cr	eep Rates (psi)
Material	(°F)	0.001%/hr	0.1%/1000 hrs	0.01%/1000 hrs
Sol. Treated Hot Rolled	1200 1200	25,000 24,000	21,500 15,500	(18,000)
Sol. Treated Hot Rolled	1300 1300	17,500 (13,000)	12,500	(9,200)
Sol. Treated Hot Rolled	1400 1400	9,200 5,300	(6,000)	(2,400)

Metallographic Examination

Solution Treated Material

The original structure of the solution treated material is shown in Plate 1. The structures of the most prolonged creep-rupture specimens tested at 1100°, 1200°, 1300°, and 1400°F are shown in Plates 2, 3, 4, and 5.

The grain size was about 3 to 5, tending to be coarser at the surface

of the specimens than in the center. The original material had a typical austenitic structure for the solution-treated condition, except for the stringers of globules of an undissolved constituent. This structure is typical for 16-25-6 steel.

The specimen ruptured at 1100°F, Plate 2, had very fine precipitates in the grain boundaries and some evidence of the beginning of a precipitation reaction within the grain. Raising the testing temperature caused a progressive increase in the size and number of precipitates present, Plates 3, 4, and 5.

The rupture specimens fractured predominately through the grain boundaries. Those tested at 1100° and 1200°F had some intergranular cracks at both the interior and at the surface adjacent to the fracture. The 1300° and 1400°F testing temperatures resulted in severe cracking that was predominately intergranular.

Hot Rolled and Tempered Material

The original hot rolled and tempered material had a structure (Plate 6) consisting of fine grains (ASTM grain size of 8) with considerable precipitation in the grain boundaries. The stringers of an undissolved globular constitutent present in the solution-treated condition did not appear to be present in the hot rolled material.

As in the case of the solution treated material, a precipitation and agglomeration reaction occurred in the hot rolled material during the rupture test which became more pronounced as the temperature of testing was raised.

Comparison after testing of the hot rolled structure with that of the solution-treated structure under the same condition shows that precipitation and agglomeration was greater in the hot rolled condition despite the shorter time of testing. The fractures of the hot rolled material appeared to be primarily intergranular, particularly at the higher temperatures. In general,

the surfaces adjacent to the fractures did not show the severe cracking present at the surfaces of the solution-treated specimens.

Stability Data

Short time tensile data obtained at 1200° and 1300°F for both conditions are shown in Table IV. The strength of the solution treated material was on the order of 10,000 psi lower than that of the hot rolled material.

Hardness measurements were taken on all of the fractured specimens which were examined metallographically. These values are shown on the plates and summarized in the following tabulation:

Test Temp (°F)	Solution-Treated Material (BHN)	Hot-Rolled Material (BHN)
Original	176/183	280
1100	310	285/300
1200	270	256
1300	238	235/256
1400	220	185

The solution-treated material underwent a phenomenal increase in hardness to 310BHN at 1100°F. At 1200°F the hardness was still at 270 despite nearly 10,000 hours at the test temperature. The 1300° and 1400°F specimens still had higher hardness than the original material.

The hot-rolled material did not show as great an increase in hardness as the solution-treated at 1100°F. At temperatures of 1200°F and higher the hardness of the ruptured specimens dropped below that of the original hot-rolled material.

DISCUSSION OF RESULTS

Sufficient data were collected for this investigation of the manganese modification of the 16-25-6 alloy to compare the rupture strength and to some extent the creep strength of the material in the hot rolled and in the

solution treated condition and in addition to compare with those of the standard 16-25-6 alloy presented previously in Report No. 182 and its supplement, Report No. 189.

Rupture Strength of the Modified 16-25-6 Alloy

The rupture strength of the solution-treated material was equal to or greater than that of the hot-rolled material for time periods beyond 10,000 hours at 1100°F and beyond 1000 hours at 1200°F and considerably superior for all time periods at 1300° and 1400°F. The ductility of the hot-rolled material was greater than that of the solution-treated alloy for practically all time periods. It would be expected that the higher initial hardness of the hot-rolled material would be associated with higher strength at the shorter time periods and lower temperatures. Structural instability as evidenced by the microstructural changes and drop in hardness appears to be a major reason for the decrease in relative strength with increasing temperatures and time periods of testing. The fine grain size is also usually associated with the development of low strength at the higher temperatures and longer time periods.

No information is available concerning the conditions of hot-working. It is therefore only possible to speculate as to the cause for the relatively low strength in the hot-rolled condition. For the type of alloy it would be expected that a well solution treated material cold worked to the hardness observed would have had much higher strengths than those observed. Consequently the conditions of hot-working were such as to have prevented solution-treatment; or the conditions of working influenced precipitation so as to introduce instability or prevent maximum strengthening. The large increases in hardness after testing the solution-treated condition at 1100° and 1200°F suggests that precipitation is a major factor in the

strength of the alloy, possibly more so than has generally been found for standard 16-25-6 alloy.

The properties of the solution-treated material were consistent with those which would be expected for the alloy. The only exception to this is the high hardness after testing at 1100° and 1200°F and to a degree at the higher temperatures. It seems unlikely that the creep deformation could have introduced sufficient cold work to raise the hardness at 1100° to 310 BHN and very unlikely that the 5.5 percent elongation in 10,000 hours at 1200°F would have cold worked the alloy to 270 BHN. Consequently it appears as if rather pronounced precipitation hardening occurs in the alloy. While detailed comparative values are not available it seems likely that the precipitation hardening factor was greater than in standard 16-25-6 alloy.

Comparative Rupture Strengths of the Modified and Standard 16-25-6 Alloy

The manganese modification of the 16-25-6 alloy in the solutiontreated condition appeared to have 100-hour and 1000-hour rupture strengths
from 1100° to 1400°F higher or equal to those of the standard 16-25-6
alloy, Heat 11873, previously presented in Report No. 182 and 189.
However, for 10,000 hours or more only the extrapolated 1100°F strength
of the modified alloy was higher. The modification had somewhat higher
ductility. The rupture strength and elongation at rupture for the modified
and standard 16-25-6 heats are compared in the following tabulations:
Comparative Rupture Strengths of the Modified 16-25-6, Heat 02400, and
the Standard 16-25-6 Alloy, Heat 11873, Solution Treated at 2150°F

Heat No.	Temp (°F)	Ruptur 100-hr	re Strength	10,000-hr		re Elongat 1000-hr	ion (%) 10,000-hr
02400	1100	58,000	48,000	(39,000)	20	13	
11873	1100	53,000	44,000	36,000	12	7	
02400	1200	44,000	33,000	20,000	30	25	5
11873	1200	42,000	32,000	23,000	12	16	(10)

(contin	ued)							
Heat	Temp	Rupti	are Streng	th (psi)	Rupture Elongation (%)			
No.	(°F)	100-hr	1000-hr	10,000-hr	100-hr	1000-hr	10,000-hr	
02400	1300	32,500	22,500	11,000	40	** **	6	
11873	1300	29,000	21,000	13,500	20	10	2	
02400	1400	20,000	11,000	5,300	60	2.0	₩.=-	
11873	1400	20,000	12,000	(7,500)	16	2.0	2	

Comparison of the hot rolled modification with the standard 16-25-6 alloy, Heat 13242, hot-coldworked at 1500°F and tempered indicates that the modification is considerably weaker, although more ductile. This is illustrated by the following tabulation:

Comparative Rupture Strengths of the Hot Rolled Modification and the Hot-Cold Worked Standard 16-25-6 Alloy, Heat 13242.

Heat	Temp	Ruptu	re Strengt		Rupture Elongation (%)				
No.	(°F)	100-hr	1000-hr	10,000-hr	100-hr	1000-hr	10,000-hr		
02400	1200	46,000	34,000	19,000	35	3.5	13		
13242*	1200	50,000	36,000	27,000	29	20	Allen (digita		
02400	1300	27,500	17,000	(7,200)	50	35	* *		
13242*	1300	33,000	22,000	14,500	2.9	15	7		
02400	1400	15,500	7,400	(3,000)	37	58	⇔ .∞		
13242*	1400	17,000	11,000	7,200	29	15	days e, sint		

*Reduced 15-20% at 1500°F - Tempered 6 hours at 1275°F

Creep Strength of the Modified 16-25-6 Alloy

The resistance to creep of the solution treated material is somewhat lower than that of the standard 16-25-6 alloy in the same condition, as is evident from the following tabulation:

Comparative Creep Strengths for Solution Treated Modified 16-25-6, Heat 02400, and Standard 16-25-6, Heat 11873

Heat	Temp	Stress	for Observed Creep	Rates (psi)
No.	(°F)	0.001%/hr	0.1%/1000 hrs	0.01%/1000hrs
02400	1200	25,000	21,500	18,000
11873	1200	28,000	22,500	18,000

(contin	(continued)										
Heat	Temp		or Observed Cree	p Rates (psi)							
No.	(°F)	0.001%/hr	0.1%/1000 hrs	0.01%/1000hrs							
				_							
02400	1300	17,500	12,500	(9,200)							
11873	1300	***	(17,500)	11,500							
02400	1400	9,200	(6,000)	(2,400)							
11873	1400		9,400	6,000							
			•	•							

The general characteristics observed in the time-elongation curves of the solution treated modified alloy appear to be similar to those previously obtained for 16-25-6 alloy. The curves for the solution treated material are characterized by a long period of primary creep, a relatively short secondary creep, and a third stage at least equal to or greater than the combined time for first and second stage put together.

The hot-rolled material, run at the same stress levels as the solution-treated material, had considerably lower resistance to creep. The level of stresses used permitted the establishment of only the stresses for 0.001 percent per hour creep rate at all temperatures and for the 0.1 percent per 1000 hour rate at 1200°F.

The lower creep strength of the hot-rolled material might be attributed to the same factors previously discussed for the rupture tests. Comparison of the only data available for 0.1 percent per 1000 hour creep rate at 1200°F shows that the modified 16-25-6 creep strength is on the order of 10,000 psi lower than that of the hot-cold worked material from Heat 13242, 15,500 psi as compared to 25,500 psi.

The data indicate that, for this material as in the case of the standard 16-25-6, the rupture strength is a better criterion of strength than creep data. Comparison of the 10,000 and 100,000 hour rupture strength of the solution-treated material with the creep strengths for rates of 0.1 percent and 0.01 percent per 1000 hour creep respectively shows that the creep strengths were higher than that of the rupture strengths as is shown by the following

tabulation:

Temp (°F)	Stress for Inc Rates	-	Rupture S (psi	. •	Ratio of Creep Str(psi) to Rupture Str (psi)		
africa monași arcana	0.1%/1000hr	0.01%/1000hr	10,000hr	100,000hr	10,000hr	100,000hr	
1200	21,500	(18,000)	20,000	(12,000)	1.1	1.5	
1300	12,500	(9,200)	11,000	(7,000)	1.1	1.3	
1400	(6,000)	(2,400)	5,300	(2,500)	1.1	. 95	

Thus the creep strengths are not reliable. The rupture data show that fracture would occur in less time than 10,000 or 100,000 hours under the stresses developing minimum creep rates of 0.1 and 0.01 percent per 1000 hours.

Third stage creep must occur at relatively short time periods so that fracture would occur in less time with more than 1-percent deformation; rather than the deformation being limited to 1-percent in 10,000 or 100,000 hours implied in the usual extrapolation of such creep data.

A sufficient number of tests were not conducted to give sufficient data to plot complete stress-time for total deformation curves in the range from 0.1 to 1.0 percent deformation. The limited available data are shown in Figure 13 for the solution treated condition. Because even less data are available for the hot rolled condition, no attempt was made to plot the data. It was observed, however, that the hot-rolled material required more time to reach a deformation of 1.0 percent than the solution treated, except for longer times at 1300° and at 1400°F.

The graphical presentation of the data for total deformation of Figure 13 was used primarily to illustrate the following points about the creep characteristics of the solution-treated material.

- 1. Due to rapid and extensive primary creep, rather large deformation (in excess of 1.0 percent) occurred in relatively short time periods compared to the rupture time at 1200° and 1300°F.
- 2. The extensive primary creep results in deformation of 1.0 percent at 1200° and 1300°F in less than 10 percent of the rupture life.

- 3. The solution-quenched material did not develop high creep resistance until the total deformations were quite large. Likewise, third stage creep did not occur until the deformations were quite large.
- 4. At 1400°F, the total deformation strengths were closer to the rupture strengths. Third-stage creep, however, started at rather small deformations.

The substitution of manganese for nickel in the 16-25-6 alloy does not appear to alter the strength of the material in the solution treated condition. The rupture and creep strength values established for the modification fall well within the range to be expected for the standard 16-25-6 alloy, at least in the solution treated condition. The effect of the substitution on the strength of the hot-rolled material was obscured because the data obtained in this investigation tend to show that the hot working of the modified 16-25-6 material was probably sufficiently different from that given the hot-cold work standard 16-25-6 alloy to cause the difference observed in the creep and rupture strengths.

TABLE I Rupture Test Data for Modified 16-25-6 Alloy (Heat 02400 - .07 C - 7.10 Mn - 16.16Cr - 15.60 N - 6.10 Mo) $1-1/4" \times 3/4" \times 7" \text{ Bars}$

Temp (°F)	Stress (psi)	Rupture Time (hours)	Elongation (% in 2 in.)	Reduction of Area (%)
		Solution Treat	ed at 2150°F	
1100	60,000	64	21.5	26.5
	50,000	549	13.5	15.0
1200	69,500	S. T. T. T.	44.0	44.4
	50,000	25. 5	24.0	25.6
	40,000	251	34.0	34.5
	35,000	540	26.5	33.5
	25,000	3545	9.5	18.5
	20,000	9649	5.5	7.0
1300	57,750	S. T. T. T.	44.5	45.0
	40,000	21	40.0	46.0
	30,000	184	41.5	50.0
	25,000	377	81.5	68.3
	15,000	3724	7.0	10.0
	12,500	6856	6.5	10.5
1400	20,000 15,000 10,000 7,250 5,000		63.0 43.5 18.0 d after 2885 hourd after 2297 hourd	ırs
1100	60,000	195	24.5	55.0
	50,000	1394	38.5	42.0
1200	78,500	S.T.T.T.	36.0	56.5
	50,000	45	44.0	65.5
	40,000	286	30.0	64.1
	35,000	826	34.5	48.0
	25,000	3702	18.0	32.5
	20,000	6872	13.5	22.5

TABLE I, Continued

Temp (°F)	Stress (psi)	Rupture Time (hours)	Elongation (% in 2 in.)	Reduction of Area (%)
1300	66,400	S. T. T. T.	45.0	65.6
	40,000	19	58.5	59.4
	30,000	53	58.5	72.3
	25,000	150	49.5	67.2
	15,000	1441	33.0	48.0
	12,500	2312	29.5	45.0
1400	20,000	43	66.5	74.6
	15,000	115	37.0	61.2
	10,000	381	46.5	56.8
	7,250	1038	57.5	61.0
	5,000	2750	52.0	63.0

TABLE II

Rupture Strengths at 1100°, 1200°, 1300°, and 1400°F at Indicated Time Periods for the Modified 16-25-6 Alloy, Heat 02400, in the Solution Treated from 2150°F and Hot Rolled and Tempered Conditions

ation (%) 10,000-hr	t t	5 13	9 !	11
Rupture Elongation (%)	13 35	25 35	35	20 58
Ruj 100-hr	2.0	30 35	40 50	92
100,000-hr	11	(12;000) (9;800)	(5,400) (3,000)	(2,500) (1,200)
Rupture Strength (psi)	(39,000) (42,000)	20,000	11,000 (7,200)	(5,300) (3,000)
Rupture 1000-hr	48,000 52,000	33,000 34,000	22,500 17,000	11,000
100-hr	58,000 63,000	44,000	32,500 27,500	20,000 15,500
Temp (°F)	1100	1200	1300	1400 1400
Condition	Solution Treated Hot Rolled	Solution Treated Hot Rolled	Solution Treated Hot Rolled	Solution Treated Hot Rolled

TABLE III

Observed Creep Rates at Indicated Time Periods for Modified 16-25-6 Alloy, Heat 02400

9500 hr			1.47									
9000 hr			. 55									
8500 hr			. 33									
7500 8000 hr hr			. 22									
7500 hr			. 14									
7000 hr			.104									
6500 hr			.04.039.040.040.080.104					ட் I				
	Solution Treated from 2150°F		. 040		. 94			1300°F				
5000 5500 6000 hr hr hr	om 2		. 040		. 58			at		1.68		
5000 hr	ted fr	1200°F	. 039	1300°F	.38	1400°F		Hot Rolled + 15 Hours	1200°F	. 92 1.18	1300°F	1400°F
4500 hr	Tre	- 11 -		41	. 26	÷Ι		d + 1	71			-1
4000 hr	olution		. 052		. 17			Rolle		. 68		
3500 hr	S		90.		. 13			Hot		. 50		
3000 hr			2.60		1.14					. 38		
2500 hr			1.34		. 68		1.02			2, 12		
2000 hr			. 84		.49		. 58			1.70		1,06
1500 hr	.		. 21		. 43		2. 1 . 43 . 11			1.23		. 92
1000 hr			1.04		. 45		2.2.2.29.			1.21	2.48	2.16 1.54
500 hr			2.02		1.09		2.52 .16 .14			1.33	1.52 1.33	2, 35
Stress (psi)			25,000 20,000		15,000 12,500		10,000 7,250 5,000			25,000 20,000	15,000 12,500	7,250

TABLE IV

Short Time Tensile Properties of Modified 16-25-6 at 1200° and 1300°F

Material	Test Temp (*F)	Tensile Strength (psi)	Offset Yield S	Offset Yield Strength (psi)	Proportional Limit (psi)	Elongation (%)	Reduction Area (%)
Solution Treated	1200	69,500	t _i	e e	17,500	44.0	44.4
Hot Rolled	1200	78, 500	61,000	65,000	36,000	36.0	56.5
Solution Treated	1300	57,750	28,500	30,000	16,000	44.5	45.0
Hot Rolled	1300	66,400	49,700	56,500	22,000	45.0	65.6

13 Bruned Figure Pages

and

10 Pages of Mounted Pictures follow here.

See ERI File Copy