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CREEP PROPERTIES OF MODIFIED 9 Cr-1 Mo STEEL*

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Creep properties of modified 9 Cr-1 Mo steel, an alloy significantly improved in elevated-temperature strength over 2 1/4 Cr-1 Mo and other similar alloys, are presented here. Data are primarily on material in the normalized and tempered condition. Effects of variables such as isothermal annealing treatment, cold work, normalizing temperature, tempering temperature, notch, and biaxial stress state have also been examined. Data analysis and comparisons have shown that modified 9 Cr-1 Mo alloy is very insensitive in response to several material variables, heat treatments, and specimen design variables.

Introduction

Modified 9 Cr-1 Mo steel has recently been developed jointly by the Oak Ridge National Laboratory (ORNL) and Combustion Engineering, Inc. (CE), Chattanooga. This alloy — currently in the approval process for inclusion in ASTM Specifications A 387 for plate, A 213 for tube, and for other product forms as grade 91 (see Table I) — offers significantly improved elevated-temperature strength properties over those of 2 1/4 Cr-1 Mo (grade T22) and the standard 9 Cr-1 Mo (grade T9) (1,2). The alloy also has higher creep strength than does type 304 stainless steel for temperatures up to 600°C (1,2). The alloy is recommended for use in the normalized and tempered condition. The nominal normalizing treatment consists of holding at 1040°C for 1 h per 25 mm of thickness followed by air cooling to room temperature. This step produces a transformation of austenite into martensite with a hardness of about Rockwell C 40. The tempering treatment consists of holding at a minimum of 732°C for 1 h per 25 mm of thickness followed by air cooling to room temperature. This treatment produces a hardness of about Rockwell B 94.

The commercial use of this alloy requires the approval of its design allowable stresses by various sections of the ASME Code. To derive the design allowable stresses, ASME requires tensile and creep data on at least three commercial heats. Tensile test data, including both ultimate tensile strength and yield strength, are required at room temperature and

Table I. Recommended specifications for modified 9 Cr-1 Mo steel
(For inclusion in ASTM specifications, the alloy is being designated as grade 91 by the plate and tube subcommittee of ASTM)

Element	Range (wt %)	Element	Range (wt %)
C	0.08-0.12	Mo	0.85-1.05
Mn	0.30-0.60	Ni	<0.40
P	<0.020	V	0.18-0.25
S	<0.010	Nb	0.06-0.10
Si	0.20-0.50	N	0.030-0.070
Cr	8.00-9.50	Al	<0.04

at 50°C intervals to a temperature at least 50°C above the intended use of the material. When creep properties may be expected to limit the allowable stresses, creep and creep-rupture data at temperature intervals of 50°C are also required. Such data should be for four or more time intervals, one of which should be longer than 2000 h but shorter than 6000 h, and one of which should be longer than 6000 h. In addition, creep data are also required to establish the effects of variables such as heat treatment, cold work, notch sensitivity, and aging.

Tensile properties for several heats of modified 9 Cr-1 Mo steel have been reported previously (1). The purpose of this paper is to present the creep data on commercial heats of modified 9 Cr-1 Mo, including the following variables:

1. nominal normalized and tempered condition,
2. effect of isothermal annealing,
3. effect of normalizing temperature,
4. effect of tempering temperature,
5. effect of cold work in the normalized and tempered condition,
6. effect of a notch in the specimen, and
7. effect of biaxiality (tube burst data).

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Materials and Test Methods

Creep tests were conducted on six commercial heats of this alloy. Heat F5349 was melted by Quaker Alloy Casting Company; heats 30176, 30182, 30383, and 30394 by Carpenter Technology; 10148 by Electralloy Corporation; and XA3602 and XA3618 by Combustion Engineering, Inc. Heats F5349, 30383, and 10148 were processed by argon-oxygen deoxidation (AOD). Heats 30176, 30182, and 30394 were processed by AOD and subsequently electroslag remelted (ESR). Heats 30176 and 30182 are the same heat with two different ingot numbers. Heat 30394 is the same heat as 30383 except for the processing differences. Heats XA3602 and XA3618 were air-induction melted at CE. Heat F5349 was a 4-ton heat; heats 30176, 30182, 30383, 30394, and 10148 were 15 tons each; and heats XA3602 and XA3618 were 0.5-ton heats. All heats were tested in the plate product form except for heats XA3602 and XA3618, which were tested in the tubular product form. All heats were tested in the nominal normalized and tempered condition (1040°C for 1 h and 760°C for 1 h). Heat 30394 was used to study the effect of variables stated above. Each of the variables will be described later. Creep tests on the normalized and tempered material were conducted in the temperature range from 427 to 704°C. Creep testing of this alloy is conducted at ORNL and through subcontracts at Combustion Engineering, Battelle Columbus Laboratories (BCL) and Westinghouse Advanced Energy Systems Division (W-AESD). The conventional creep testing was performed at all four laboratories; the effects of other variables were investigated only at ORNL.

The chemical analysis of various heats used in this test program is shown in Table II. Almost all specimens were taken transverse to the rolling direction and 6.35 mm in gage diameter (Figure 1). All creep tests were conducted in air except for tube burst tests (to be described later), which were conducted in argon. The creep test methods used conformed to the requirements of ASTM E 139-70 (3). Lever-arm creep machines were calibrated to a load accuracy of $\pm 0.5\%$. The temperature was measured by three Chromel vs Alumel thermocouples ($\pm 0.4\%$ accuracy) wired to the gage section. The temperature variation among these was less than $\pm 1.0^\circ\text{C}$, and the highest temperature was taken as the nominal test temperature. Proportioning temperature controllers were used to obtain control to $\pm 1.0^\circ\text{C}$, with the highest temperature being the nominal test temperature.

Figure 1 - Creep specimen used in the testing of modified 9 Cr-1 Mo steel.

Length changes were measured by an averaging extensometer attached by set screws to a small groove in the specimen shoulder. The extension readings were converted to strains by dividing by the reduced section length. The dial gage used in measuring the extension had a resolution of $2.5\ \mu\text{m}$ ($0.0001\ \text{in.}$).

Results and Discussion

Creep Data in Normalized and Tempered Condition

The creep-rupture and minimum creep rate data on commercial heats, tested in the normalized and tempered condition, were analyzed by lot-centered regression (4,5) with a generalized model selection procedure (6,7). Using this procedure, we found that rupture data could be described quite well by a model of the form

$$\log t_r = C_h + a_1 \sigma + a_2 \log \sigma + a_3/T, \quad (1)$$

where

- t_r = time to rupture (h),
- σ = applied stress (MPa),
- T = test temperature (K),
- a_1 - a_3 = regression constants.

The term C_h is a lot constant that describes variations in creep-rupture strength among the different lots. The equation with actual constants, including C_h averaged for all heats studied, can be written as

$$\log t_r = -24.244 - 0.02374\sigma - 2.4871 \log \sigma + 31,876/T. \quad (2)$$

The coefficient of determination (R^2) for the rupture data was 94.6%.

The minimum creep rate data were fitted to the following equation:

$$\log \dot{\epsilon}_m = 27.300 + 0.025265\sigma + 3.2172 \log \sigma - 35,594/T. \quad (3)$$

The minimum creep rate $\dot{\epsilon}_m$ was in percent per hour. The coefficient of determination (R^2) for minimum creep rate data was 90.1%. Equations (2) and (3) are only slightly different from similar equations reported in reference 2.

Isothermal stress-rupture data on commercial heats tested in the normalized and tempered condition are plotted in Figure 2. Average curves predicted from Equation (2) are also included in this figure. It can be noted that the average curves describe the stress-rupture data of commercial heats extremely well. Figure 3 shows the isothermal plots of stress versus minimum creep rate data on commercial heats tested in the normalized and tempered condition. Average curves from Equation (3) are included in Figure 3. Once again, the average curves describe the experimental data quite well.

Table II. Summary of chemical analyses of various commercial heats of modified 9 Cr-1 Mo steel used in the creep testing program

Element	Content, ^a wt %, for each heat									
	F5349	30383		30394		30176	30182	10148	XA3602	XA3618
		Plate	Tube ^b	Plate	Tube ^c	Plate	Tube ^c			
C	0.10	0.083	0.075	0.084	0.094	0.081	0.081	0.089	0.089	0.093
Mn	0.39	0.46	0.46	0.46	0.45	0.37	0.36	0.47	0.40	0.35
P	0.007	0.010	0.009	0.010	0.014	0.010	0.013	0.021	0.005	0.006
S	0.015	0.004	0.004	0.003	0.004	0.003	0.003	0.006	0.009	0.004
Si	0.35	0.41	0.40	0.40	0.37	0.11	0.11	0.28	0.19	0.14
Ni	0.10	0.09	0.08	0.09	0.09	0.09	0.09	0.16	0.05	0.10
Cr	8.80	8.46	8.17	8.57	8.11	8.61	8.32	9.24	9.21	8.59
Mo	0.94	1.02	0.99	1.02	1.03	0.89	0.90	0.96	0.93	0.98
V	0.205	0.198	0.195	0.198	0.200	0.209	0.208	0.21	0.197	0.202
Nb	0.060	0.072	0.064	0.073	0.072	0.072	0.076	0.054	0.059	0.123
Ti	0.006	0.005	0.005	0.005	0.002	0.004	0.002	0.002	0.005	0.002
Co	0.018	0.055	0.055	0.055	0.056	0.010	0.011	0.019	0.019	0.024
Cu	0.09	0.03	0.03	0.04	0.04	0.04	0.04	0.08	0.02	0.01
Al	<0.001	0.002	0.003	0.014	0.024	0.007	0.004	0.002	0.003	0.008
B	0.001	<0.001		<0.001		<0.09		<0.001	<0.001	
W	<0.01	0.05		0.05		<0.01		0.01	<0.01	
As	0.002	<0.001		<0.001		0.001		0.002	<0.001	
Sn	0.004	<0.001		<0.001		<0.001		0.004	0.001	
Zr	<0.001	<0.001		<0.001		<0.001		<0.001	<0.001	
N	0.011	0.051	0.050	0.053	0.053	0.055	0.053	0.035	0.065	0.060
O	0.012							0.008		

^aAll chemical analysis was carried out at Combustion Engineering, Inc., Chattanooga, Tenn.

^bHot extruded.

^cHot rotary pierced.

Figure 2 - Stress-rupture data for commercial heats of modified 9 Cr-1 Mo steel tested in the normalized and tempered condition. Average curves from the time-temperature parameter are also included in these plots.

Figure 3 — Stress-minimum creep rate data for commercial heats of modified 9 Cr-1 Mo steel tested in the normalized and tempered condition. Average curves from the time-temperature parameter are also included in these plots.

The total elongation and reduction of area of specimens tested in the normalized and tempered condition are plotted as a function of time to rupture in Figures 4 and 5. In each case we have drawn the lines representing the minimum value. Figure 4 shows that the total elongation values for all test temperatures exceed 20%. The reduction-of-area values exceed 60% for all test temperatures. Neither total elongation nor reduction of area shows a systematic decrease with increasing rupture time for times up to 20,000 h.

Creep Data in the Isothermally Annealed Condition

Specimens of heat 30394 were also creep tested in the isothermally annealed condition. This treatment consists of holding the specimens at 1038°C for 1 h and dropping the temperature to 704°C and holding for 24 h. Such a treatment prevents the transformation of austenite to martensite. It produces ferrite and coarse carbides instead. Stress-rupture data from material in the isothermally annealed condition are compared with the average curves for the material tested in the normalized and tempered condition in Figure 6. Similar comparisons for minimum creep rate are presented in Figure 7. These two figures show that the rupture and creep strengths of the isothermally annealed material are somewhat lower than the average curves for the normalized and tempered material at 538°C. However, the strengths are the same in the two heat-treated conditions at 593, 649, and 677°C. The total elongation and reduction of area values for the isothermally annealed condition exceed

the minimum values for the normalized and tempered material at all four test temperatures — 538, 593, 649, and 677°C (Figures 8 and 9).

Creep Data in the Cold-Worked Condition

Several creep tests were conducted on material that had been 20% cold rolled or 20% compressively cold worked in the normalized and tempered condition. Stress-rupture data on cold-worked specimens are compared with the average curves for the normalized and tempered material in Figure 10. This figure shows that at 593°C cold work has no effect on creep-rupture properties of modified 9 Cr-1 Mo steel for test times up to about 6000 h. At 649°C rupture lives for the 20% cold-worked material are somewhat lower than those of the normalized and tempered material for test times near 100 h; however, long-term rupture lives approach those of the normalized and tempered material.

Total elongation and reduction of area data for the cold-worked material are compared with the minimum values for the normalized and tempered material in Figure 11. The total elongation values of the cold-worked specimens were nearly the same as or exceeded those of the minimum properties of the normalized and tempered material. However, the reduction of area values of cold-worked material fell below the minimum values for the normalized and tempered material at 593°C. It should be noted that the lowest value observed is still 40%, which is significantly above the value for cold-worked stainless steels (8).

Figure 4 — Total elongation as a function of time to rupture for commercial heats of modified 9 Cr-1 Mo steel tested in the normalized and tempered condition. Lines in these plots represent the estimated minimum value of total elongation for each test temperature.

Figure 5 — Reduction of area as a function of time to rupture for commercial heats of modified 9 Cr-1 Mo steel tested in the normalized and tempered condition. Lines in these plots represent the estimated minimum value of reduction of area for each test temperature.

Figure 6 - Comparison of stress-rupture data for modified 9 Cr-1 Mo steel tested in the isothermally annealed condition with average curves for commercial heats tested in the normalized and tempered condition.

Figure 7 - Comparison of stress-minimum creep rate data for modified 9 Cr-1 Mo steel tested in the isothermally annealed condition with average curves for commercial heats tested in the normalized and tempered condition.

Figure 8 - Comparison of total elongation data for modified 9 Cr-1 Mo steel tested in the isothermally annealed condition with minimum curves for commercial heats tested in the normalized and tempered condition.

Figure 9 - Comparison of reduction-of-area data for modified 9 Cr-1 Mo steel tested in the isothermally annealed condition with minimum curves for commercial heats tested in the normalized and tempered condition.

Figure 10 - Comparison of stress-rupture data for modified 9 Cr-1 Mo steel tested in the normalized and tempered condition after 20% cold rolling or 20% compressive cold work with average curves for commercial heats tested in the normalized and tempered condition.

Figure 11 - Comparison of total elongation and reduction of area for modified 9 Cr-1 Mo steel tested in the normalized and tempered condition after 20% cold rolling or 20% compressive cold work with minimum curves for commercial heats tested in the normalized and tempered condition.

The Effect of Normalizing Temperature and Time on Creep Properties

Modified 9 Cr-1 Mo steel contains carbides of niobium and vanadium in addition to carbides of chromium and molybdenum unlike standard 9 Cr-1 Mo steel, which only contains carbides of chromium and molybdenum. The carbides of both niobium and vanadium require higher dissolution temperatures than do carbides of chromium and molybdenum. To determine optimum properties, we conducted creep tests on specimens subjected to various combinations of normalizing temperatures and times. The normalizing temperatures varied from 927 to 1121°C. Time to rupture for specimens tested after various normalizing temperatures and times are plotted as a function of the Hollomon-Jaffee parameter (HJ) in Figure 12. The HJ parameter allows combining normalizing time and temperature (9). Figure 12 also includes a normalizing temperature scale for a time of 1 h. This figure shows that time to rupture increases with increasing normalizing temperature and time. This figure also shows that a normalizing temperature in the range from 1038 to 1065°C provides the most desirable creep properties. We believe that this is the temperature range in which most of the vanadium and niobium carbides dissolve. Figure 13 shows total elongation and reduction of area as functions of time to rupture for specimens tested after various normalizing temperatures. This figure illustrates that both total elongation and reduction of area exceed the minimum values observed for several heats tested after a normalizing temperature of 1040°C and tempering temperature of 760°C. This figure also shows that, if the normalizing temperature is 1093°C, there may be some tendency for lower reduction of area. The lower reduction of area is believed to be the result of grain coarsening after a normalizing treatment at 1093°C.

Figure 12 - Time of time to rupture as a function of the Hollomon-Jaffee parameter for modified 9 Cr-1 Mo tested after various normalizing temperatures and time. All tests are at 649°C.

The Effect of Tempering Temperature on Creep Properties

Creep tests were conducted on specimens normalized at 1038°C followed by tempering at 732, 760, and 774°C. Creep curves for these specimens are compared in Figure 14. This figure shows that a slightly higher tempering temperature than the nominal 760°C has a minimal effect on creep-rupture properties. However, tempering at 732°C produces significantly greater creep strength. This type of data is useful when the tempering temperature deviates from the nominal.

Creep Properties of Notched Specimens

Four creep tests were conducted on specimens containing a notch (Figure 15). The notch geometry is such that it produced an elastic stress concentration factor of 3.9. Figure 16 compares the data on these specimens. This figure shows that for modified 9 Cr-1 Mo, the notch produces somewhat of a strengthening rather than a weakening. Such a behavior of an alloy is quite desirable for engineering applications.

Creep Properties of Tubular Specimens

Two tests were conducted on tubular specimens by internal pressurization to check the effect of biaxial stress state on the creep strength. The data are compared with the average curve for uniaxially tested specimens in Figure 17. This figure shows that the use of the maximum principal stress criterion will be conservative for thin-wall pressurized tube loading.

Figure 13 - Total elongation and reduction of area as functions of time to rupture for modified 9 Cr-1 Mo steel tested after various normalizing temperatures and times. Minimum curves for material tested in the nominal normalized & tempered condition are included for comparison.

Figure 14 - Effect of tempering temperature on creep curves of modified 9 Cr-1 Mo steel. All specimens were subjected to the same normalizing temperature before tempering. Tests were conducted at 593°C and 159 MPa.

Figure 15 - Specimen used to investigate the notch effect on creep properties of modified 9 Cr-1 Mo steel. The stress concentration factor for this notch is 3.9.

Figure 16 - Comparison of stress-rupture data on notched specimens with average curves for unnotched specimens of modified 9 Cr-1 Mo steel tested in the normalized and tempered condition.

Figure 17 - Comparison of stress-rupture data for tube-burst specimens with average curves of uniaxial specimens of modified 9 Cr-1 Mo steel tested in the normalized and tempered condition. The maximum principal stress was plotted for the tubular specimens.

Microstructure

Optical metallography was performed on the creep specimen that failed after 21,028 h at 649°C and 62 MPa. The microstructure at the fracture for this specimen is presented in Figure 18. Higher magnification micrographs comparing the microstructure of the unstressed shoulder, where the gage section away from the fracture, and the near-fracture region are shown in Figure 19. This figure also includes the appropriate microhardness numbers for various regions. Figures 18 and 19 show the absence of the classical grain boundary cavitation observed in stainless steels. The voids observed at and near the fracture are believed to be caused by ductile tearing due to the overload in the necked region.

Figures 18 and 19 also show that the microstructure and hardness at fracture differ significantly from those of the unstressed region or the gage region away from the fracture. The tempered martensitic structure in the unstressed region consists of a high dislocation density and carbides. The stress and temperature cause the annihilation of dislocations and growth of carbide particles. Such a process causes the cleaning of matrix microstructure, as shown in Figure 19. This figure also shows that the microstructural rearrangement is far more significant near the fracture than away from it. The cleaned microstructure has lower hardness and thus provides a site for initiation of fracture. The cleaned microstructure of the matrix also contributes to the absence of grain boundary cavitation of modified 9 Cr-1 Mo and other similar alloys.

Summary and Conclusions

Creep data are presented on commercial heats of modified 9 Cr-1 Mo steel. Data are primarily on material tested in the normalized and tempered

Figure 18 — Optical microstructure at the fracture for a specimen of modified 9 Cr-1 Mo ruptured after 21,028 h at 649°C and 62 MPa. The specimen was tested in the normalized and tempered condition.

condition. The nominal normalizing treatment consisted of holding at 1040°C for 1 h followed by air cooling to room temperature. The tempering treatment consists of holding at 760°C for 1 h followed by air cooling. Data are also presented to cover the effects of (1) isothermal annealing treatment, (2) cold work, (3) normalizing temperature and time, (4) tempering temperature, (5) notches, and (6) biaxiality.

Data for the normalized and tempered condition were fitted to a time-temperature parameter by the lot-centering procedure. The average curves predicted from this model for the normalized and tempered material were compared with the data for other heat treatments. The longest term ruptured specimen was examined by optical metallography. The following observations and conclusions were drawn from this work.

1. Creep rupture and minimum creep rate data for the normalized and tempered condition are well behaved and can be fitted by a time-temperature parameter for the temperature range from 427 to 704°C. The average predictions describe both time to rupture and minimum creep rate data quite well.

2. The total elongation and reduction of area plots as functions of time to rupture showed no indication of any systematic drop with increasing rupture time. These data were used to establish the minimum value curves for each test temperature. The minimum values were found to be independent of rupture time reached thus far.

3. Creep and rupture strengths of isothermally annealed material were slightly lower than the average for normalized and tempered material at 538°C; however, the strength values were the same for two heat-treated conditions at 593, 649, and 677°C. The total elongation and reduction of area data isothermally annealed material exceeded the minimum value curves for the normalized and tempered material.

4. Cold work (compressive or cold rolled) to 20% has an unimportant effect on the creep rupture strength at 593 and 649°C. The reduction of area for the long-term test at 593°C fell below the minimum for the normalized and tempered material; however, the lowest value still exceeded 40%.

5. Increasing the normalizing temperature and/or time increases the rupture time. The 1040°C normalizing and 760°C tempering treatment produced the optimum strength and ductility properties.

6. Tempering above the nominal temperature of 760°C has insignificant effect on creep strength. However, tempering below 760°C (namely, 732°C) significantly improves creep strength.

7. A notch with a stress concentration factor of 3.9 slightly strengthens this alloy.

8. Tubular specimens showed similar creep strength to that observed for uniaxial specimens.

9. The modified 9 Cr-1 Mo alloy is insensitive to several material, heat treatment, and specimen design variables. The creep process occurs by rearrangement of dislocation structure leading to cleaning the grain matrix and causing the final fracture to occur by ductile void tearing.

Figure 19 — Optical microstructure of (a) unstressed shoulder, (b) gage section away from fracture, and (c) near-fracture location of a specimen of modified 9 Cr-1 Mo ruptured after 21,028 h at 649°C and 62 MPa. The specimen was tested in the normalized and tempered condition.

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