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CHARACTERIZATION OF MODIFIED 9 Cr-1 Mo STEEL EXTRUDED PIPE

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CHARACTERIZATION OF MODIFIED 9 Cr-1 Mo STEEL EXTRUDED PIPE*

V. K. Sikka and M. D. Hart[†]

ABSTRACT

The fabrication of hot-extruded pipe of modified 9 Cr-1 Mo steel at Cameron Iron Works is described. The report also deals with the tempering response; tensile, Charpy impact, and creep properties; and microstructure of the hot-extruded pipe. The tensile properties of the pipe are compared with the average and average ± 1.65 standard error of estimate curves for various product forms of several commercial heats of this alloy. The creep-rupture properties are compared with the average curve for various product forms of the commercial heats.

INTRODUCTION

As part of a continuing effort in fabrication experience on modified 9 Cr-1 Mo steel, a 15-ton heat of this alloy was melted at Electralloy Corporation, Oil City, Pennsylvania, during the last year. The heat was made by the argon-oxygen deoxidation process and bottom-poured into two 533-mm-diam and one 7-mm-diam ingots. The 533-mm-diam ingots were electroslag remelted (ESR) into 610-mm-diam ingots at National Forge Company, Irvine, Pennsylvania. One of the 610-mm-diam ingots was hot extruded to a 406-mm-OD by 25-mm-wall pipe. The pipe was heat treated and tested at Cameron Iron Works and Oak Ridge National Laboratory. This report presents the characterization of the extruded pipe and compares its properties with the results obtained previously on plate, bar, and tubes of several commercial heats.

*Work performed under DOE/BTP AF 15 40 10 3, Task OR-1.7, Advanced Alloy Technology.

[†]Cameron Iron Works, Houston, Tex.

PIPE FABRICATION AND CERTIFICATION

The ESR ingot was soaked at 1260°C (Fig. 1) and extruded in a 35,000-ton press at Cameron Iron Works (Figs. 2 and 3). Following the extrusion, the pipe was subjected to a hot dimensional inspection (Fig. 4) and serial number assignment for traceability (Fig. 5). The extruded pipe was stress relieved at 732°C for 2 h; a ring was then cut off to study the effect of tempering treatment on properties.

Y-196319

Fig. 1. Ingot weighing 3970 kg (8750 lb) being removed from furnace.
Source: Reprinted with permission from Cameron Iron Works, Houston, Tex.

Fig. 2. Blocker going into 35,000-ton extrusion press. Source: Reprinted with permission from Cameron Iron Works, Houston, Tex.

Fig. 3. Extrusion of pipe in 35,000-ton press. *Source:* Reprinted with permission from Cameron Iron Works, Houston, Tex.

Fig. 4. Hot dimensional inspection. *Source:* Reprinted with permission from Cameron Iron Works, Houston, Tex.

The pipe was subsequently normalized at 1040°C for 1 h, tempered at 760°C for 1 h, and straightened. Next, a ring was cut for a flattening test. Material was also removed from both ends (designated A and B) for chemical analysis and tensile tests at room temperature (listed below and given in Table 1). The pipe was then ground on both the inner and outer surfaces. The outer surface was buffed after grinding to prepare it for ultrasonic examination. The pipe was hydrostatically tested at 19.3 MPa for 5 s and

Y-196320

Fig. 5. Serial assignment to pipe for traceability. *Source:*
Reprinted with permission from Cameron Iron Works, Houston, Tex.

Table 1. Chemical analysis of modified 9 Cr-1 Mo steel
extruded pipe (406-mm OD by 25-mm wall) (heat 14361)

Elements	Content (wt %)			
	Specification	Ladle analysis ^a	Extruded pipe ^b	
			A-end	B-end
Carbon	0.08-0.12	0.12	0.11	0.12
Manganese	0.30-0.60	0.48	0.54	0.54
Phosphorus	0.020 Max	0.014	0.009	0.011
Sulfur	0.010 Max	0.009	0.004	0.002
Silicon	0.20-0.30	0.33	0.26	0.24
Nickel	0.40 Max	0.12	0.16	0.14
Chromium	8.00-9.50	8.62	8.85	8.90
Molybdenum	0.85-1.05	0.96	0.97	0.97
Vanadium	0.18-0.25	0.22	0.23	0.23
Niobium	0.06-0.10	0.09	0.07	0.08
Cobalt		0.02		
Copper				0.08
Aluminum	0.04 Max	0.02	0.010	0.014
Tin		0.004		
Nitrogen	0.03-0.07	0.038	0.037	0.038

^aArgon-oxygen-deoxidized (AOD) material.

^bElectroslag-remelted (ESR) material; pipe analysis
conducted by Cameron Iron Works.

ultrasonically inspected before shipping. Vendor certification of properties for the extruded pipe (406-mm OD by 25-mm wall) is listed below.

1. Chemical analysis: see Table 1
2. Tensile properties at room temperature:

	0.2% Yield strength (MPa)	Ultimate tensile strength (MPa)	Elongation (%)	Reduction of area (%)
Specified minimum	414	585	20.0	55.0
Actual	541	708	26.1	70.8

3. Flattening test: acceptable
4. Hydrostatic test: acceptable
5. Heat treatment: normalized at 1040°C for 1 h, air cooled to room temperature; tempered at 760°C for 1 h, air cooled to room temperature

No exceptions were found to the specification. A section of the pipe was cut and saved at Cameron Iron Works for detailed testing, and results are presented in subsequent sections.

TEMPERING STUDY

The pipe piece saved after postextrusion stress relief was normalized at 1040°C for 1 h and sectioned into tensile and hardness coupons. These coupons were tempered for 1 h at 538, 565, 593, 621, 649, 677, 704, 732, and 760°C. After tempering, coupons were machined into 12.83-mm-diam standard tensile specimens of transverse orientation. Tensile and hardness data after various tempering treatments are listed in Table 2 and plotted in Figs. 6 and 7. The hardness data on the extruded pipe are compared with data on two other commercial heats in Fig. 8. One of the heats was cold rolled before testing, and the other was hot rolled before testing. The following observations are made from examination of Figs. 6 through 8.

1. The tempering response of the hot-extruded pipe is very similar to cold-rolled or hot-rolled material.
2. Hardness and tensile strength properties (yield and ultimate) showed a large drop as a result of increasing the tempering temperature.
3. Elongation and reduction of area showed a small increase as a result of increasing the tempering temperature.
4. Even with very high yield and ultimate tensile strength, specimens showed about 18% elongation and over 60% reduction of area.

TENSILE DATA

Tensile tests were conducted from room temperature to 760°C on longitudinal and transverse specimens from both A- and B-ends of the

Table 2. Effect of tempering temperature on room-temperature tensile properties and hardness of hot-extruded pipe of modified 9 Cr-1 Mo steel

(The pipe was normalized at 1040°C for 1 h before tempering at various temperatures for 1 h. All tests were conducted at Cameron Iron Works on transverse specimens)

Tempering temperature (°C)	Strength (MPa)		Total elongation (%)	Reduction of area (%)	Brinell hardness (BHN)	Rockwell hardness (R _C)
	0.2% Yield	Ultimate				
538	1056	1457	18.1	62.8	418	41
538	1027	1458	17.9	62.3	418	41
565	1052	1376	17.7	66.3	388	39
565	1123	1382	17.7	66.8	388	39
593	1085	1257	18.2	65.9	387	37
593	1018	1247	17.5	67.7	387	37
621	934	1074	20.6	67.8	363	32
621	936	1071	20.2	67.4	363	32
649	894	1031	18.7	62.3	321	29
649	878	1031	20.0	67.0	321	29
677	843	971	19.8	67.0	293	29
677	845	947	20.0	64.7	293	29
704	803	927	19.6	66.1	286	27
704	809	928	20.7	68.4	236	27
732	713	844	21.7	69.3	269	20
732	714	847	22.0	70.8	269	20
760	696	757	23.7	71.4	228	18
760	611	757	24.4	72.7	228	18

hot-extruded pipe (Table 3). Tensile strength and ductility properties of the pipe are compared with the average and average - 1.65 standard error of estimate (SEE)* curves for various product forms of several commercial heats in Figs. 9 through 12. These figures show the following.

1. The tensile properties of the longitudinal and transverse specimens and ends A and B of the pipe are the same. This implies that the pipe was uniform from one end to the other and had isotropic properties.

*SEE = Standard error of estimate is an exact definition of standard deviation.

Fig. 6. Effect of tempering temperature on hardness and room-temperature tensile strength properties of hot-extruded 413-mm-OD by 25-mm-wall pipe of modified 9 Cr-1 Mo steel.

Fig. 7. Effect of tempering temperature on total elongation and reduction of area of hot-extruded pipe of modified 9 Cr-1 Mo steel.

Fig. 8. Comparison of tempering response between hot-extruded pipe and cold-rolled and hot-rolled products of modified 9 Cr-1 Mo steel.

2. Yield and ultimate tensile strengths for the extruded pipe were between the average and average - 1.65 SEE curves for various product forms of several commercial heats.

3. Total elongation and reduction of area for the extruded pipe are close to the average or between the average and average - 1.65 SEE for various product forms of commercial heats.

CHARPY IMPACT DATA

Charpy impact tests were conducted on longitudinal and circumferential (tangential) specimens (Table 4). Impact energy is plotted against test temperature for various specimens in Fig. 13, and fracture appearance is plotted against test temperature in Fig. 14. The solid and dashed

Table 3. Tensile properties of 9 Cr-1 Mo hot-extruded pipe
(Normalized at 1040°C and tempered at 760°C for 1 h;
all tests conducted at Cameron Iron Works)

Test temperature (°C)	Strength (MPa)				Total elongation (%)		Reduction of area (%)	
	0.2% Yield		Ultimate		Transverse	Longitudinal	Transverse	Longitudinal
	Transverse	Longitudinal	Transverse	Longitudinal				
A-end specimens								
21	539	525	713	692	25.9	26.7	73.4	71.0
21	536	523	705	686	25.9	25.7	73.4	73.0
21	531	525	705	692	25.3	26.3	74.2	73.0
93	487	498	636	646	23.5	24.0	74.4	74.6
93	488	484	641	642	24.0	24.4	75.2	75.4
204	459	460	596	596	23.1	24.2	75.8	75.4
204	456	456	596	603	22.9	24.7	75.0	74.2
316	442	439	566	565	21.8	21.2	73.4	74.6
316	456	436	566	564	20.4	20.4	72.1	74.2
427	421	421	537	536	20.6	21.0	72.6	73.0
427	417	414	530	529	20.3	22.1	71.3	70.9
538	357	366	418	424	31.8	29.3	85.6	84.3
538	348	362	420	431	32.0	29.3	85.5	84.0
649	238	233	283	296	37.0	37.5	92.3	92.8
649	241	243	283	301	36.8	36.0	93.3	93.3
760	83	87	152	148	48.0	48.0	96.7	97.0
760	78	77	148	146	48.1	48.0	96.9	97.2

Table 3. (Continued)

Test temper- ature (°C)	Strength (MPa)				Total elongation (%)		Reduction of area (%)	
	0.2% Yield		Ultimate					
	Trans- verse	Longitu- dinal	Trans- verse	Longitu- dinal	Trans- verse	Longitu- dinal	Trans- verse	Longitu- dinal
B-end specimens								
21	538	525	710	703	25.2	25.5	72.1	77.3
21	542	526	716	696	25.1	27.3	73.0	72.4
21	525	529	706	710	26.2	25.5	74.3	72.5
93	459	490	646	663	25.3	24.7	74.6	67.8
204	438	446	604	552	25.0	24.5	75.4	76.6
316	443	419	581	567	21.2	25.1	72.6	73.4
427	419	430	536	536	20.1	20.0	73.1	71.9
538	366	370	441	436	27.3	27.5	83.3	83.9
649	213	202	281	302	39.5	38.3	99.3	99.4
760	79	81	150	141	50.8	50.0	99.7	99.7

Fig. 9. Yield strength versus test temperature for the hot-extruded pipe. The average (solid) and average - 1.65 standard error of estimate (dashed) curves for various product forms on several commercial heats are included for comparison.

Fig. 10. Ultimate tensile strength versus test temperature for the hot-extruded pipe. The average (solid) and average - 1.65 standard error of estimate (dashed) curves for various product forms on several commercial heats are included for comparison.

Fig. 11. Total elongation versus test temperature for the hot-extruded pipe. The average (solid) and average - 1.65 standard error of estimate (dashed) curves for various product forms on several commercial heats are included for comparison.

Fig. 12. Reduction of area versus test temperature for the hot-extruded pipe. The average (solid) and average - 1.65 standard error of estimate (dashed) curves for various product forms on several commercial heats are included for comparison.

Table 4. Impact properties of hot-extruded pipe of modified 9 Cr-1 Mo steel

(All tests conducted at Cameron Iron Works)

Test temper- ature (°C)	A-end LC orientation ^a			A-end CR orientation ^b			B-end CR orientation ^b		
	Impact energy (J)	Fracture (% shear)	Lateral expansion (mm)	Impact energy (J)	Fracture (% shear)	Lateral expansion (mm)	Impact energy (J)	Fracture (% shear)	Lateral expansion (mm)
21	275	100	2.46	215	100	2.18	241	100	2.39
21	262	100	2.46	242	100	2.24	245	100	2.41
21	264	100	2.39	245	100	2.29	265	100	2.46
4.4	242	100	2.18	223	100	2.18	218	100	2.21
4.4	258	100	2.29	248	100	2.18	220	100	2.36
4.4	272	100	2.36	239	100	2.18	227	100	2.21
-6.7	248	100	2.26	218	100	2.18	234	100	2.44
-6.7	242	100	2.24	245	100	2.26	223	100	2.34
-6.7	239	100	2.21	231	100	2.16	196	83	2.16
-17.8	262	100	2.29	231	100	2.03	214	100	2.36
-17.8	250	100	2.26	223	100	2.13	231	100	2.34
-17.8	248	100	2.31	228	100	2.13	218	100	2.51
-45.6	163	64	2.01	160	60	1.83	121	49	1.65
-45.6	147	56	1.85	152	56	1.78	133	56	1.73
-45.6	159	67	1.78	151	61	1.80	125	52	1.68
-73.3	109	37	1.47	99	30	1.27	101	27	1.35
-73.3	120	43	1.52	117	41	1.47	101	34	1.37
-73.3	120	35	1.60	113	37	1.40	103	29	0.86

^aLC = longitudinal orientation with circumferential notch.

^bCR = circumferential orientation with radial notch.

Fig. 13. Impact energy versus test temperature for longitudinal and circumferential specimens of hot-extruded pipe.

Fig. 14. Fracture Appearance (% shear) versus test temperature for longitudinal and circumferential specimens of hot-extruded pipe.

curves are drawn to show the range of impact values. Figure 13 shows the 68-J transition temperature to vary from -115 to -138°C . Figure 14 shows the 50% shear transition temperature to vary from -44 to -64°C . The transition temperature values for the extruded pipe are much lower than values of -36 to -37°C for 68 J and 0 to -24°C for 50% shear observed for the saddle forging.¹

CREEP-RUPTURE DATA

Eight creep-rupture tests were conducted on the hot-extruded pipe (Table 5). Rupture data on the extruded pipe are compared with the average curve for several commercial heats (Fig. 15). This figure shows that the creep-rupture strength of the extruded pipe is near the average curve observed for other product forms. The total elongation and reduction of area values for the extruded pipe exceed the minimum values of 20 and 60%, respectively, observed for data on various product forms of commercial heats.

Table 5. Creep-rupture data on hot-extruded pipe of modified 9 Cr-1 Mo steel

(All tests conducted at Cameron Iron Works)

Test temperature ($^{\circ}\text{C}$)	Stress (MPa)	Time to rupture (h)	Elongation (%)	Reduction of area (%)
Transverse orientation				
593	193	65.7	40.3	92.1
593	193	63.6	37.7	92.1
649	131	52.7	43.7	74.4
649	131	58.9	43.7	71.3
Longitudinal orientation				
593	193	82.4	37.0	90.5
593	193	67.6	43.0	90.7
649	131	52.7	45.7	99.2
649	131	64.8	44.3	72.1

Fig. 15. Comparison of creep-rupture data on hot-extruded pipe with the average curves for various product forms of several commercial heats of modified 9 Cr-1 Mo steel.

MICROSTRUCTURE

Optical photomicrographs of longitudinal and transverse specimens at various magnifications from A- and B-ends of the pipe are presented in Figs. 16 through 21. These figures show identical microstructure for longitudinal and transverse specimens from both A- and B-ends of the pipe. The grain size for all specimens was measured to be ASTM 8, which is very fine. The carbide distributions of longitudinal and transverse specimens from the A- and B-ends of the pipe also were very similar (Figs. 20 and 21). The microstructural observations are consistent with the mechanical properties, which were the same for specimens of various orientations and from A- and B-ends of the pipe.

Y-200285

Y-200274

100 μ m

Fig. 16. Low magnification photomicrograph of longitudinal specimens from the (a) A-end and (b) B-end of the hot-extruded pipe of modified 9 Cr-1 Mo steel.

Y-200288

Y-200278

100 μ m

Fig. 17. Low magnification photomicrograph of transverse specimens from the (a) A-end and (b) B-end of the hot-extruded pipe of modified 9 Cr-1 Mo steel.

Y-200286

Y-200276

Fig. 18. Intermediate magnification photomicrograph of longitudinal specimens from the (a) A-end and (b) B-end of the hot-extruded pipe of modified 9 Cr-1 Mo steel.

Y-200289

Y-200275

Fig. 19. Intermediate magnification photomicrograph of transverse specimens from the (a) A-end and (b) B-end of the hot-extruded pipe of modified 9 Cr-1 Mo steel.

Y-200287

Y-200277

Fig. 20. High magnification photomicrograph of longitudinal specimens from the (a) A-end and (b) B-end of the hot-extruded pipe of modified 9 Cr-1 Mo steel.

Y-200290

Y-200279

Fig. 21. High magnification photomicrograph of transverse specimens from the (a) A-end and (b) B-end of the hot-extruded pipe of modified 9 Cr-1 Mo steel.

SUMMARY AND CONCLUSIONS

Hot-extruded pipe of modified 9 Cr-1 Mo steel was fabricated at Cameron Iron Works and evaluated for tempering response in tensile, Charpy impact, creep, and microstructural evaluations. The tensile properties of the hot-extruded pipe were compared with the average and average - 1.65 SEE curves for various product forms of several commercial heats of this alloy. The creep-rupture properties were compared with the average curve for various product forms of the commercial heats. The following conclusions are possible from this work.

1. No unusual problems were encountered nor were unusual procedures required to fabricate the hot-extruded pipe from a modified 9 Cr-1 Mo steel ingot.

2. The tempering response of the hot-extruded pipe was similar to that observed for hot-rolled or cold-rolled plates of other commercial heats of this alloy.

3. Tensile properties of the extruded pipe were similar for both longitudinal and transverse specimens from both ends of the pipe. The strength and ductility properties of the pipe were between the average and average - 1.65 SEE curves for various product forms of commercial heats.

4. Charpy impact properties of the extruded pipe were superior to those observed for a saddle forging from the same heat. The 68-J transition temperature for the extruded pipe was between -115 and -138°C.

5. The creep-rupture strength of the hot-extruded pipe matched the average curve for various product forms of commercial heats.

6. The microstructure was the same for longitudinal and transverse specimens from both ends of the pipe. The mechanical properties of the extruded pipe were consistent with the microstructure observed.

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