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Procurement and Screening Test Data for Advanced Austenitic Alloys for 650°C Steam Service (Part 2, Final Report)

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Metals and Ceramics Division

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CONTENTS

LIST OF FIGURES

LIST OF TABLES

PROCUREMENT AND SCREENING TEST DATA FOR ADVANCED AUSTENITIC ALLOYS FOR 650°C STEAM SERVICE (PART 2, FINAL REPORT)*

R. W. Swindeman, G. M. Goodwin, P. J. Maziasz, and E. Bolling

ABSTRACT

The results of screening tests on alloys from three compositional groups are summarized and compared to the alloy design and performance criteria identified as needed for austenitic alloys suitable as superheater/reheater tubing in advanced heat recovery systems. The three alloy groups included lean (nominally 14% Cr and 16% Ni) austenitic stainless steels that were modifications of type 316 stainless steel, 20Cr-30Ni-Fe alloys that were modifications of alloy 800H, and Ni-Cr aluminides, $(Ni,Cr)_3Al$. The screening tests covered fabricability, mechanical properties, weldability, and oxidation behavior. The lean stainless steels were found to possess excellent strength and ductility if cold-worked to an equivalent strain in the range 5 to 10% prior to testing. However, they possessed marginal weldability, poor oxidation resistance, and sensitivity to aging. The modified alloy 800H alloys also exhibited good strength and ductility in the cold-worked condition. The weldability was marginal, while the oxidation resistance was good. The aluminides were difficult to fabricate by methods typically used to produce superheater tubing alloys. The alloys that could be worked had marginal strength and ductility. An aluminide cast alloy, however, was found to be very strong and ductile. Compositions of the stainless steel and modified alloy 800H were selected for the production of tubing and for further studies of weldability, surface treating, corrosion in simulated fireside environments, and mechanical behavior. Work on the aluminide tubing was deferred pending improvements in the fabrication technology.

INTRODUCTION

This report contains the experimental results of a screening investigation to select a small group of austenitic alloys for evaluation as superheater/reheater tubing in advanced steam cycle applications. The rationale for such an undertaking and the details of the program plan are

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contained in other reports.[1-4] The scope of the evaluation includes four groups of alloys based on type 316 stainless steel, alloy 800H, aluminum-bearing iron-chromium-nickel alloys, and chromium-nickel alloys intended for cladding. In addition to cladding for fireside protection, the scope of the evaluation includes an assessment of surface treatment measures to protect against steamside corrosion for stainless steels containing less than 25% chromium.

An important aspect of the investigation is the requirement that it be directed toward a basic understanding of the materials and their response to the complex thermal, mechanical, and corrosive environments of interest to the Advanced Research and Technology Development (AR&TD) Fossil Energy Materials Program sponsored by the U.S. Department of Energy (DOE). To conform to this requirement, the investigation is focused on determining the underlying issues related to the serviceability of each class of materials, rather than selecting one specific alloy for commercialization and production.

The report describes the results of the screening tests on three of the four alloy groups of interest to the program. Since the level of effort was different for the three groups, the report is separated into three appropriate sections, and a fourth section is added to compare the performance of one group with another.

LEAN STAINLESS STEELS

The lean stainless steels were essentially modifications of type 316 stainless steel and were based on compositions developed by Maziasz and coworkers for use in nuclear applications.[5-6] For superheater tubing, changes were made to allow for silicon and nitrogen additions and a reduction of phosphorus for improved weldability. Along with the developmental alloys, several commercial alloys were included in the evaluation. Commercial alloys of interest included type 316 stainless steel, 17-14CuMo stainless steel, Esshette 1250, Sandvik 12R72 (equivalent to the German alloy Din. 1.4970), the Nippon Kokan alloys Tempaloy A-1 and A-2,[7] and a radiation-resistant modified type 316 stainless steel alloy identified as PCA.[5-6] Compositions of some of these alloys are given in Table 1.

Table 1. Compositions of the modified type 316 stainless steels (wt %)

Alloy	C	Si	Mn	Ni	Cr	Ti	Nb	V	Mo	P	B	S	N
316 ES1	0.059	0.56	1.79	13.2	16.5			0.02	2.24	0.013	0.001	0.020	0.043
316 ES2	0.058	0.54	1.78	12.6	16.8	0.01	0.10	0.10	2.00	0.030	0.001	0.009	0.062
PCA	0.048	0.52	1.83	16.6	14.3	0.31	0.02	0.04	1.95	0.014	0.001	0.002	0.008
17-14CuMo	0.098	0.95	0.83	13.8	16.5	0.21	0.45	0.07	1.96	0.014	0.005	0.005	0.025
CE0	0.072	0.41	1.80	16.0	14.2	0.24	0.10	0.57	2.45	0.071	0.005	0.007	0.015
CE1	0.085	0.21	1.64	16.2	13.1	0.21	0.12	0.52	2.30	0.076	0.005	0.008	0.016
CE2	0.079	0.26	1.89	16.0	16.1	0.31	0.11	0.58	2.26	0.069	0.007	0.008	0.017
CE3[a]	0.086	0.21	1.75	16.2	14.5	0.27	0.11	0.56	2.41	0.071	0.005	0.008	0.012
AX5	0.076	0.12	2.04	16.2	13.9	0.27	0.15	0.52	2.46	0.024	0.005	0.015	0.021
AX6	0.074	0.12	1.96	16.0	14.3	0.28	0.15	0.51	2.48	0.041	0.005	0.015	0.020
AX7[b]	0.073	0.11	2.00	16.0	14.2	0.18	0.15	0.53	2.48	0.073	0.005	0.014	0.024
AX8	0.074	0.12	2.05	15.9	13.9	0.24	0.08	0.15	2.48	0.043	0.005	0.015	0.022

[a]Contains 1.96% Cu.

[b]Contains 1.5% Cu.

Eight experimental alloys were identified, and compositions are listed in Table 1. The reference composition was alloy CE1. Other compositions included CE0 to study the effect of a silicon increase, CE2 to study the influence of higher chromium, CE3 and AX7 to examine the strengthening influence of copper, AX5 and AX6 to examine performance with reduced phosphorus, and AX8 to study the influence of reduced phosphorus and vanadium.

FABRICABILITY

Information on the fabrication methods used to produce the lean stainless steels was provided in an earlier report.[4] The CE series of alloys were produced by Combustion Engineering, where the production of small tube hollows by centrifugal casting was examined. Because of potential problems associated with TiO_2 formation, the alloys were melted by an air-induction process and cast as 25-kg solid ingots that were subsequently electroslag remelted. The ingots were hot-rolled with 10% reductions in thickness per pass and 1200°C reanneals between passes. The finished products were 13-mm-thick plates, approximately 100-mm wide. Plates were delivered in a mill-annealed condition with an equivalent 5 to 10% cold work. The AX series of alloys, produced at the AMAX Research Laboratory, were vacuum-induction melted, poured as 20-kg ingots, and homogenized at 1250°C for 2 h. Hot-rolling was performed at 1100°C with 10% reduction in thickness per pass. Intermediate reanneals at 1200°C were introduced between each pass. The plates were delivered in the mill-annealed condition that involved a 1200°C reanneal for 0.5 h followed by a 10% cold reduction in thickness. No problem was encountered in producing the plates.

THERMAL-MECHANICAL TREATMENTS

Most of the mechanical testing was performed on materials in the mill-annealed condition; however, exploratory testing was undertaken to examine the sensitivity of several alloys to thermal-mechanical treatment. In this exploratory testing, specimens were individually annealed at 1115 and 1200°C in argon and rapidly cooled. Cold-work effects were introduced by tensile-straining machined specimens to levels of 2 and 5%. Some aging effects were also examined by furnace-aging specimens in the creep testing machine for 24 h at 850°C prior to testing. Other aging studies involved tests on specimens from furnace plates aged to 10,000 h at 700°C. Specimens from this task have yet to be tested.

MECHANICAL BEHAVIOR

The screening test program for mechanical behavior consisted of tensile testing at room temperature and 700°C and creep testing at 700°C at 170 MPa. Additional testing of selected alloys was undertaken to establish trends in regard to stress and temperature effects. Supplementary studies included relaxation testing, variable stress and temperature testing, and creep crack growth testing.

Tensile test results are summarized in Fig. 1 and Table 2. These results indicated that all eight experimental alloys had adequate strength and ductility in the mill-annealed condition at room temperature to meet the requirements of ASTM A 213 (205-MPa minimum yield, 515-MPa minimum ultimate, and 35% elongation). The yield strengths reflected varying degrees of work in the mill-annealed condition. Solution-treating at 1115°C reduced the yield and ultimate strengths of those heats that were examined in this condition, but the strengths at room temperature remained acceptable. Solution-treating at 1200°C drastically reduced the room-temperature yield strengths, and none of the heats met the 205-MPa minimum yield expected for stainless steels conforming to ASTM A 213. The tensile strength of all mill-annealed alloys at 700°C was excellent relative to austenitic stainless steel meeting ASTM A 213. Some trends are shown in Fig. 1.

Creep rupture data are provided in Table 3. For materials in the mill-annealed condition, the rupture lives at the screening test condition (170 MPa at 700°C) ranged from 1432 h (heat CE1) to beyond 15,000 h (heats AX5 and AX7). These times were substantially better than the lives for type 316 stainless steel (110 h) and PCA (340 h) and were equivalent to, or better than, the life for 17-14CuMo stainless steel (1463 h). Comparisons are shown in Fig. 2. All heats exhibited excellent creep ductilities, measured in terms of the reduction of area, and had ductile, transgranular

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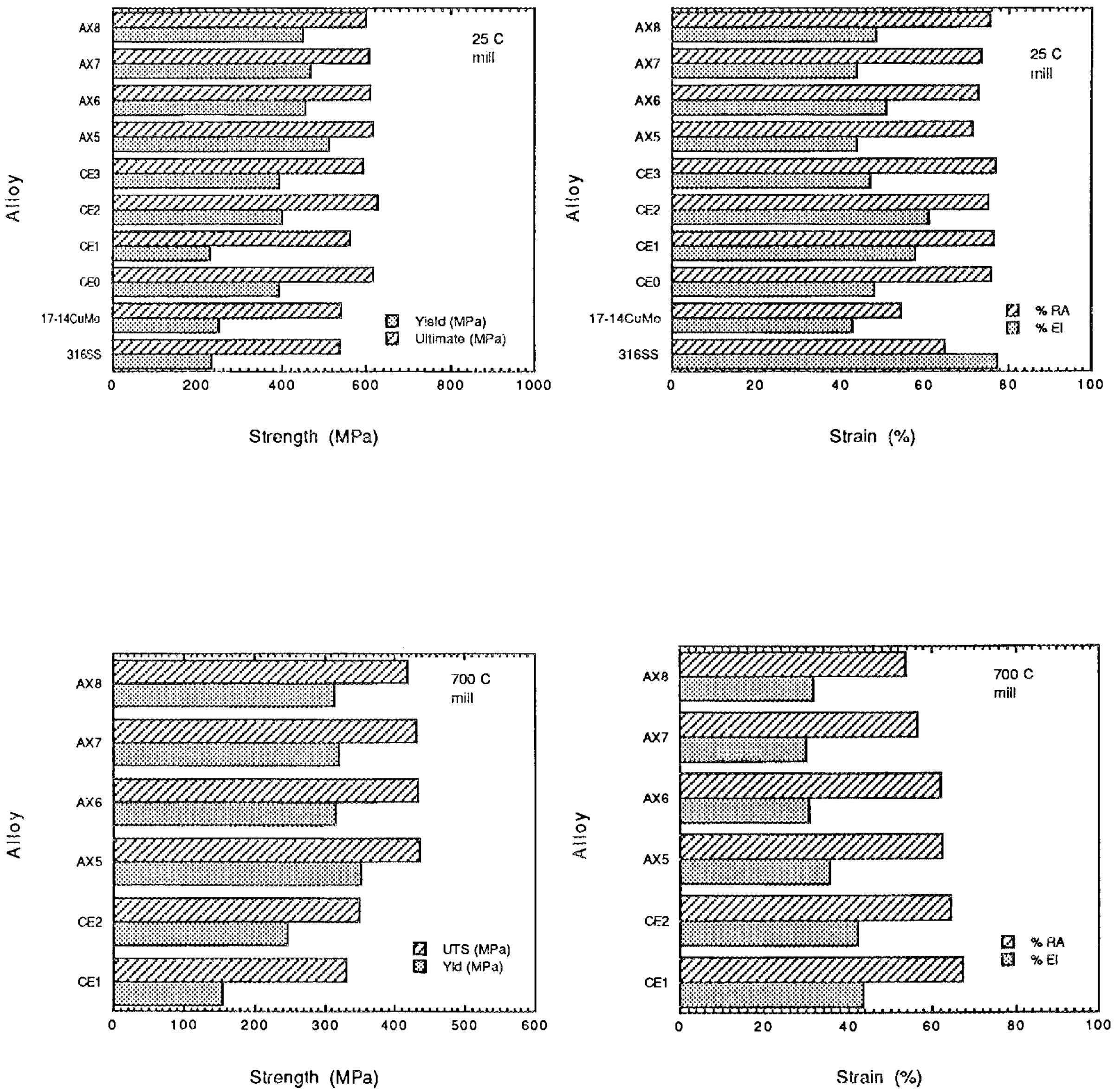

Fig. 1. Comparisons of the tensile properties of mill-annealed modified type 316 stainless steels at 25 and 700°C.

Table 2. Tensile data for the modified type 316 stainless steels

Alloy	Specimen	Condition[a] (°C)	Temperature (°C)	Yield (MPa)	Ultimate (MPa)	Elongation (%)	Reduction of area (%)
316SS	ES1-10	Mill	25	233	536	77.2	64.7
PCA	PCA-12	1050	25	219	577	57.0	77.5
PCA	PCA-01	1115	25	169	537	71.6	81.2
PCA	PCA-07	1200	25	159	520	75.0	76.2
17-14CuMo	CuMo26	Mill	25	250	541	42.8	54.5
17-14CuMo	CuMo30	1200	25	176	508	52.1	46.7
CE0	CE0-05	Mill	25	395	615	48.2	75.9
CE0	CE0-08	1115	25	228	580	56.1	75.0
CE1	CE1-15	Mill	25	231	561	58.0	76.5
CE1	CE1-13	1115	25	213	565	55.6	75.0
CE1	CE1-18	1200	25	175	551	72.3	75.1
CE2	CE2-08	Mill	25	402	628	61.0	75.3
CE2	CE2-09	1115	25	225	585	47.7	75.4
CE3	CE3-13	Mill	25	392	592	47.1	77.1
CE3	CE3-09	1115	25	222	567	55.1	74.0
AX5	AX5-05	Mill	25	511	616	44.0	71.6
AX5	AX5-02	1200	25	179			
AX6	AX6-05	Mill	25	457	609	51.0	72.8
AX7	AX7-05	Mill	25	467	607	43.8	73.4
AX7	AX7-03	1200	25	183			
AX8	AX8-05	Mill	25	451	601	48.6	75.5
PCA	PCA-04	1115	700	77	344	59.0	71.3
PCA	PCA-08	1200	700	67	327	50.0	55.8
17-14CuMo	CuMo25	Mill	700	157	299	28.4	31.1
CE0	CE0-01	1115	700	157	358	44.1	60.0
CE1	CE1-17	Mill	700	155	330	43.8	67.4
CE1	CE1-11	1200	700	123	359	26.3	45.8
CE2	CE2-01	Mill	700	246	350	42.1	64.5
CE2	CE2-04	1115	700	149	323	51.9	67.3
CE2	CE2-07	1200	700	120	345	38.2	43.0
AX5	AX5-06	Mill	700	352	434	25.4	62.4
AX6	AX6-06	Mill	700	316	432	30.7	62.1
AX7	AX7-06	Mill	700	319	431	30.0	56.4
AX8	AX8-06	Mill	700	313	419	31.7	53.6

[a]Mill anneal was 1200°C plus 10% hot roll for CE alloys and 1200°C plus 10% cold roll for AX alloys.

Table 3. Creep rupture data for the modified type 316 stainless steels

Specimen	Condition[a] (°C)	Temperature (°C)	Stress (MPa)	Minimum creep rate (h)	Time to 1% creep (h)	Time to rupture (h)	Elongation (%)	Reduction of area (%)	Status[b]	Comment
CE0-02	1115	700	170	1.8E-4	700.0				D	
CE0-03	Mill	700	170	5.6E-5					D	
CE0-04	1115/age	700	170	2.9E-4	4.0	238	43.0	81.6	R	
CE0-06	1115/5%/age	700	181	1.3E-3	260.0	502	35.5	74.7	R	
CE0-07	1115/2%/age	700	170	9E-3	7.0	189	23.0	71.1	R	
CE0-10	1115/5%	700	179	3E-4	1,000.0	1,519	26.5	77.9	R	
CE0-11	1200/5%	700	170	6.5E-5	4,400.0	6,981	29.5	58.3	R	
CE0-12	1115/2%	700	170	1E-5	1,200.0	1,722	30.2	81.0	R	
CE0-13	Mill	700	170	1E-5	3,200.0	6,174	28.8	61.5	R	
CE0-14	1200	700	170	1.4E-5	3,450.0	5,098	28.0	58.0	R	
CE1-01	Mill	700	100	3.2E-5					D	7,315 h
CE1-02	Mill	700	172						D	3,662 h
CE1-05	1115/800/168[c]	700	100	1.55E-3	310.0				D	1,247 h
CE1-08	1200/5%	700	170	5E-5	3,100.0	5,693	23.6	56.5	R	
CE1-09	Mill	700	172	1E-4	730.0	1,432	40.1	71.3	R	
CE1-12	1200	700	170	6E-5	2,350.0	3,728	35.3	63.8	R	
CE2-02	Mill/age	700	170	1E-4	2,750.0	4,134	25.0	58.1	R	
CE2-06	1200/age	700	170	7.2E-4	530.0	771	34.5	73.1	R	
CE2-10	1200/age	700	170	6E-4	200.0	464	35.0	74.8	R	
CE2-12	1200	700	170	1.4E-4	1,950.0	2,713	36.0	70.8	R	
CE2-14	1115	700	170	1.6E-4	900.0	1,415	31.5	78.3	R	
CE2-15	Mill	700	170	1.1E-4	1,910.0	2,365	14.4	41.5	R	
CE3-01	Mill	700	170	6E-5	2,900.0	4,941	13.2	45.8	R	
CE3-02	Mill/age	700	170	1.1E-4	2,200.0	3,795	23.0	53.0	R	
CE3-03	Mill	730	170	8.9E-4	960.0	1,776	17.5	54.0	R	
CE3-04	Mill	700	200	3E-4	1,240.0	1,828	26.8	70.4	R	
CE3-05	Mill	760	138	4.1E-4	950.0	1,175	13.6	41.6	R	
CE3-06	Mill	760	200	5.2E-3	40.0	54	25.2	74.2	R	
CE3-07	1115	700	100	1.8E-5	4,400.0	9,392	24.9	58.3	R	
CE3-08	1115	760	140	4E-3	45.0	100	54.4	75.4	R	
CE3-10	Mill	760	170	2.4E-2	122.0	229	23.3	70.2	R	
CE3-11	Mill	700	100	8E-6					I	20,000 h
CE3-12	1115	700	170	2.9E-4	900.0	1,319	29.2	34.7	R	
CE3-14	Mill	700	140	3.7E-5	15,500.0				I	20,000 h
CE3-15	Mill	760	170	6E-4	205.0				D	
CE3-16	Mill	800	100	6.4E-4	925.0				D	Oxidation
CE3-17	Mill	700	240	1.1E-3	375.0	630	20.0	44.6	R	
CE3-18	1200	700	170	1.4E-4	2,200.0	3,309	20.0	49.8	R	
CE3-19	Mill	700	117	1.8E-5					I	20,000 h
CE3-20	Mill	760	100	1.7E-4	4,300.0				D	Oxidation
CE3-20E	1200	700	100	2E-5					I	18,000 h
CE3-21E	1200	650	100						I	15,000 h
CE3-22E	Mill	700	100						I	20,000 h
CE3-25E	Mill	650	100						I	2,000 h
AX5-01	1200	700	170	1.1E-4	2,500.0	4,171	33.9	61.6	R	
AX5-02	1200/2%	700	170	3E-5	2,700.0	4,692	39.2	66.9	R	
AX5-03	1200/2%/age	700	170	2E-1	1.8	94	67.8	69.6	R	
AX5-07	Mill	700	170	4.5E-5	13,000.0				I	16,000 h
AX5-09	1200	600	350	1.4E-4	1,500.0	1,956	45.0	59.6	R	
AX5-12	Mill	600	300						I	3,000 h
AX5-14	Mill	600	350						I	5,000 h
AX6-01	1200	700	170	1.2E-4	2,900.0	4,919	38.6	62.3	R	
AX6-07	Mill	700	170	4E-5					D	Oxidation
AX6-13	1200	800	100	1.6E-2	26.0	127	63.3	71.9	R	
AX7-01	1200	700	170	1E-4	2,000.0	2,804	32.7	63.6	R	
AX7-02	1200	650	200						I	10,000 h
AX7-03	1200/2%	700	170	1.5E-4	2,000.0	2,888	33.3	69.6	R	
AX7-04	1200/2%/age	700	170	3E-4	20.0	910	36.2	65.1	R	
AX7-07	Mill	700	170	3,6E-5	6,500.0	6,694	13.6	45.4	R	
AX7-08	Mill	650	240						I	8,000 h
AX7-09	1200	675	200	9E-5	8,200.0				I	9,000 h
AX7-10	Mill	700	190	2.2E-5	4,400.0	4,590	10.1	44.7	R	
AX7-11	Mill	700	140	2E-5					I	8,000 h
AX7-12	Mill	650	200						I	8,000 h
AX7-13	1200	700	200	1.5E-4	1,350.0	1,978	26.8	61.6	R	
AX7-14	1200	700	140	6E-5	2,500.0	5,186	29.6	55.9	R	
AX8-01	1200	700	170	5E-4	2,700.0	4,633	26.2	68.4	R	
AX8-07	Mill	700	170	1.5E-5					I	15,000 h
AX8-08	Mill	700	200						I	2,500 h
AX8-09	Mill	730	170						I	2,000 h
AX8-11	Mill	700	240						I	2,500 h
AX8-13	Mill	760	140						I	1,500 h

[a]Mill = mill annealed, 1115 and 1200 = annealing temperature, age = 850°C for 24 h. Mill anneal for CE alloys was 1200°C plus 10% hot roll; for AX alloys, 1200°C plus 10% cold roll.

[b]D = discontinued, R = ruptured, I = in test.

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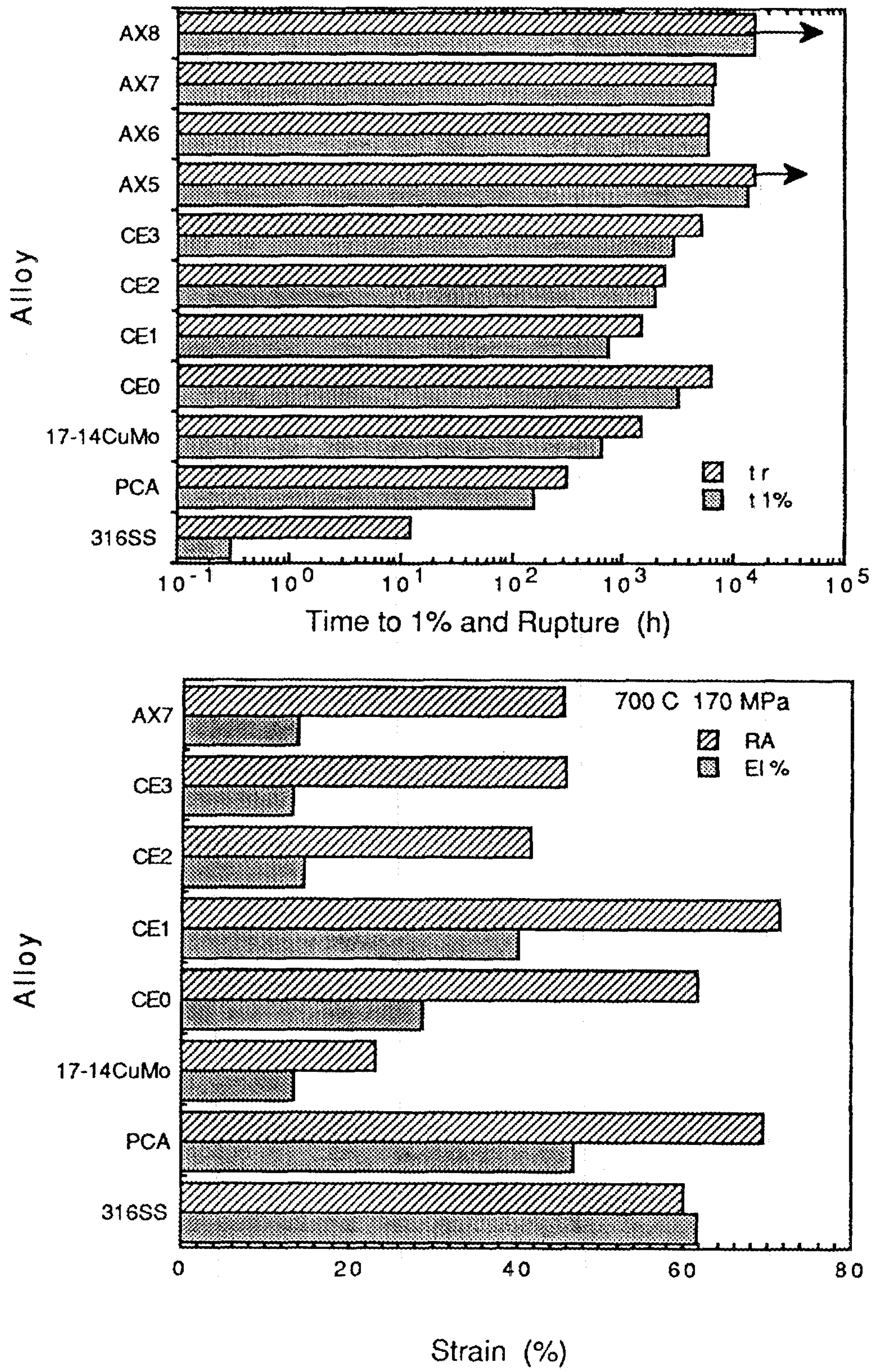

Fig. 2. Comparisons of the creep rupture properties of mill-annealed modified type 316 stainless steels at 170 MPa and 700°C.

failure modes. Creep rates were low and the times to 1% creep strain were about 75% of the rupture lives. Minimum creep rate was not a satisfactory parameter for describing creep resistance since tests on some heats exhibited a period of near zero or even negative creep not long after loading.

Solution-treating the CE alloys at 1115°C produced both a lower ultimate strength and a significant reduction in rupture life for most heats tested at the screening test condition (170 MPa and 700°C). Reasons for this poorer performance were discussed in an earlier report[4] and seem to be due to the production of coarse, nonstrengthening MC carbides during solution treatment. Solution-treating at 1200°C restored the life at the screening test condition (170 MPa at 700°C). Comparisons of the annealing effects are shown in Fig. 3.

A few exploratory tests were performed to examine the influence of cold work on the creep rupture of material reannealed at 1115 and 1200°C. Tensile strains of 2 and 5% were introduced in annealed test bars, and the samples were tested at the screening test conditions (170 MPa at 700°C). Straining to 2% raised the yield strength, reduced the inelastic loading strain, and reduced the primary creep in most cases. However, the rupture life was not greatly extended. The 5% tensile strain had a much greater effect on rupture life and, in some instances, restored the rupture life to values approaching that for the mill-annealed material. The effects of cold work are illustrated in Fig. 3. Much more information on cold-working effects can be found in the publication of Li and coworkers.[8]

Several aging treatments were examined. These were intended to explore the tendency for time-dependent strengthening, weakening, and embrittlement. Also, if tubing were to be subjected to a commercial chromizing treatment, it would necessarily experience long times at high temperature followed by a slow cool. This chromizing condition was simulated by annealing at 1115°C and aging at 850°C for 24 h. The influence of the aging (at 850°C for 24 h) on alloys with various processing histories is shown in Fig. 3(*c*): aging produced only slight changes in the creep behavior of mill-annealed alloys but large reductions in the life of annealed alloys. Again, Li and coworkers have studied aging effects in detail.[8]

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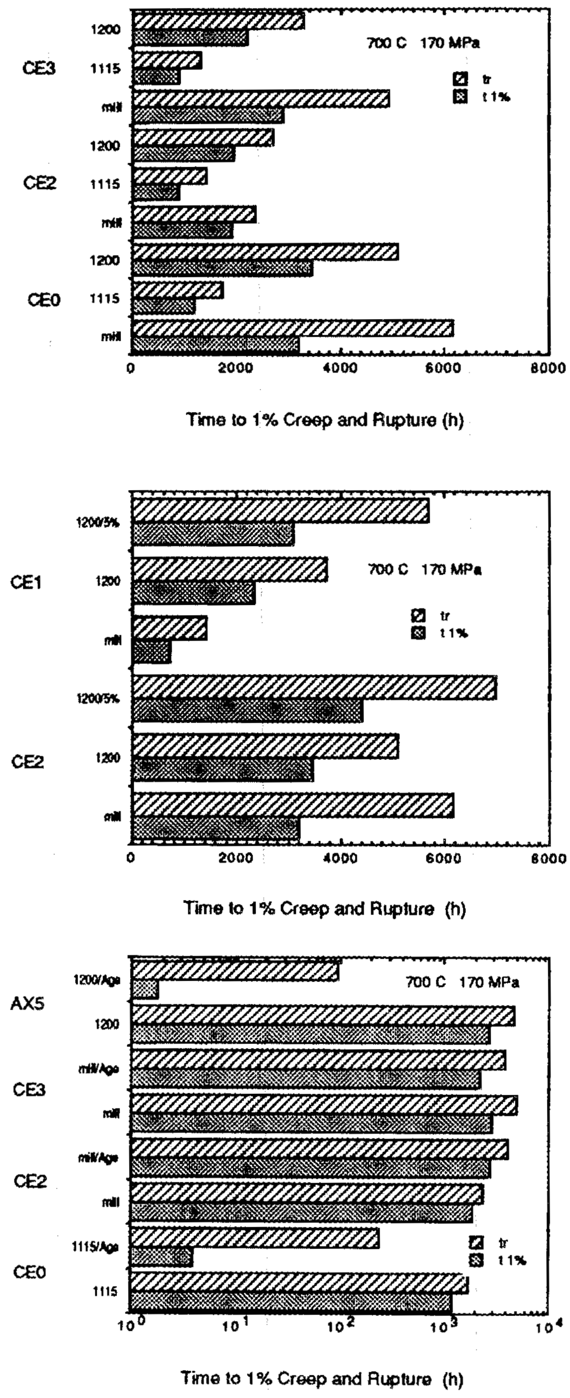

Fig. 3. Effect of annealing temperature, aging (850°C/24 h), and cold work on the creep rupture properties of modified type 316 stainless steels at 700°C and 170 MPa.

Alloys selected for studies of the effect of stress and temperature on creep rupture included CE1, CE3, AX5, AX7, and AX8. Of these, the most extensive testing was performed on CE3, which accumulated test times to beyond 20,000 h. Data for heat CE3 in the mill-annealed condition are summarized in Fig. 4, which shows the variation in the time to 1% creep and rupture life with stress and temperature. Data for the other alloys were insufficient to establish long-time strength, but several tests are still in progress.

The creep and tensile ductilities of all alloys were found to be excellent when measured on the basis of reduction of area. Values typically ranged from 30 to 80%, as indicated in Fig. 5. Elongations were as low as 10% in some alloys.

Relaxation tests on heat AX5 indicated a very low relaxation rate at 600 and 700°C. Generally, the relaxation strain rate was equivalent to that for creep and tensile testing conditions when compared on the basis of the same stress and accumulated strain. The results of these studies will be the subject of a separate report.

Exploratory creep crack growth tests were performed at 700°C on heat AX5 in both the mill-annealed and 1200°C condition. Excellent resistance to crack growth was found relative to other stainless steels and high-temperature alloys. These tests will also be the subject of a separate report.

WELDABILITY

The weldability of the CE series of alloys was described in a previous report.[4] Screening tests consisted of hot-cracking evaluations of thin sheets using the Sigmajig,[9] thermomechanical simulation (Gleeble) tests of 6.3-mm-diam bars, side bend tests of butt-welded 13-mm plate, tensile tests of weldment specimens at 25 and 700°C, and creep rupture tests of weldment specimens at the screening test conditions (700°C at 170 MPa). A few additional creep tests were performed at other conditions.

All of the lean stainless steels were rolled to 2-mm (0.010-in.) sheet and tested in the Sigmajig at applied stresses in the range 25 to 200 MPa. All heats exhibited hot cracking at the weld centerline for stresses in excess of 50 MPa with 100% cracking at stresses above 100 MPa. Thus, the

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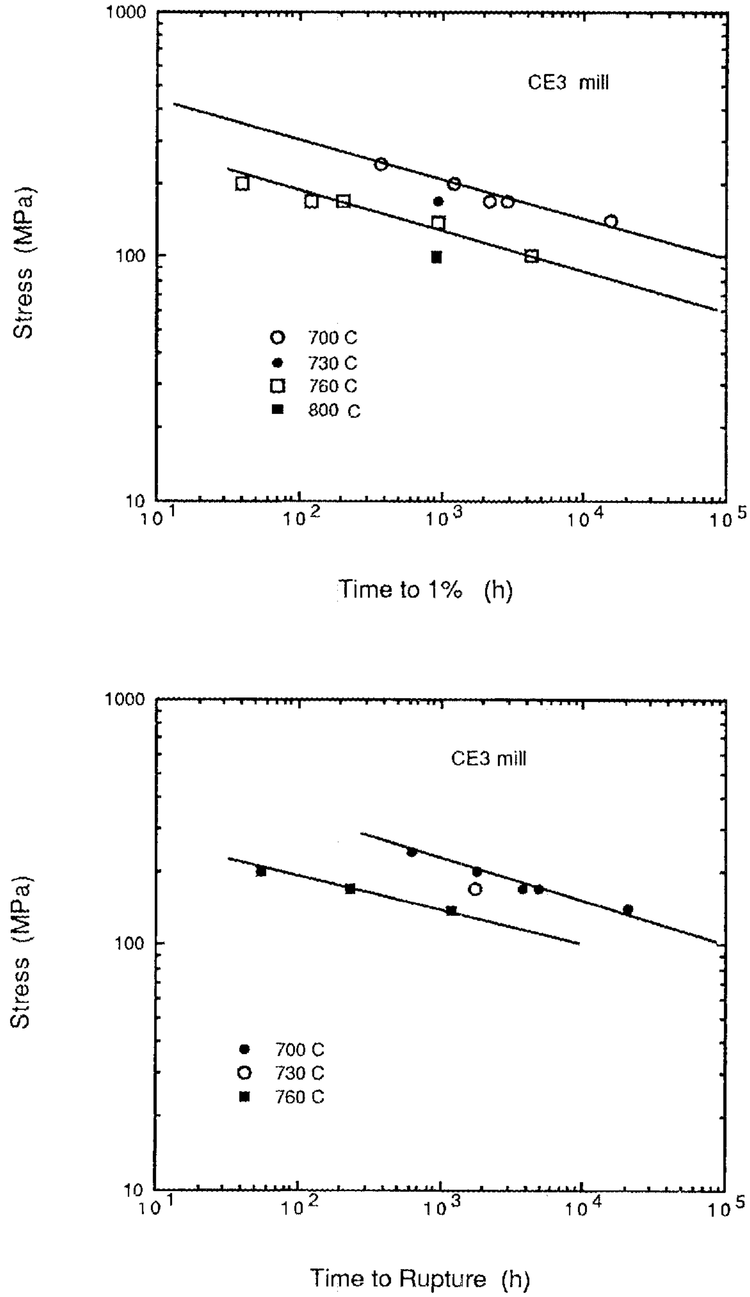

Fig. 4. Effect of stress and temperature on the creep rupture of mill-annealed heat CE3.

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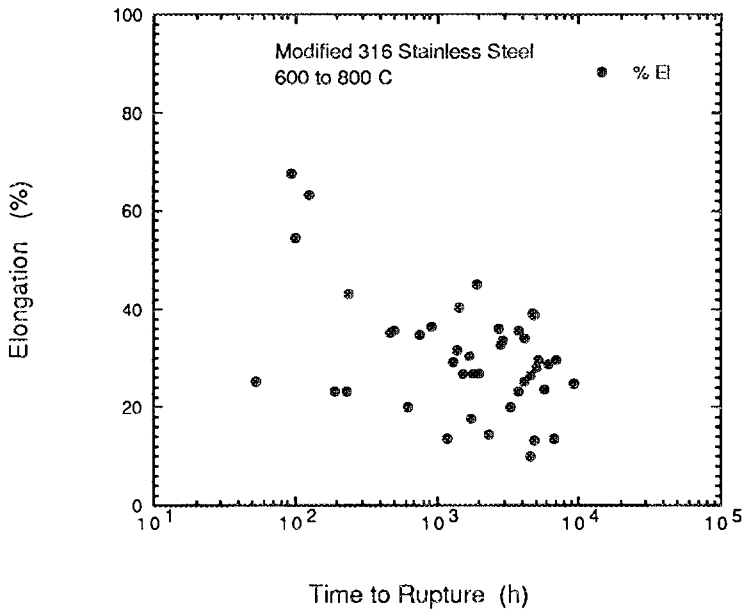

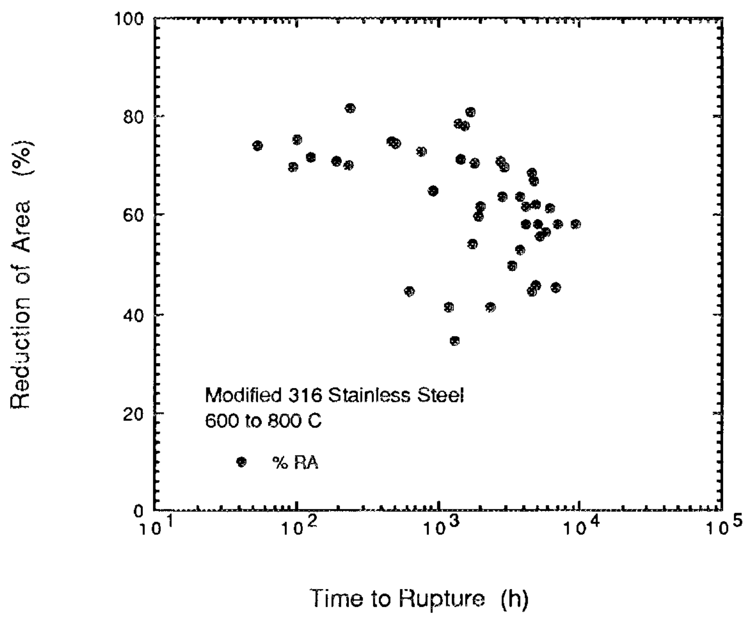

Fig. 5. Creep rupture ductility of modified type 316 stainless steels.

results on the AX series of lean stainless steels confirmed the findings on the CE series reported previously.[4] Results were interpreted to indicate that filler metals of different compositions would be needed to join the lean stainless steels. The CE alloys were joined with Inconel 82 (ERNiCr3), 17-14CuMo stainless steel wire [gas tungsten arc (GTA) process], and 17-14CuMo stainless steel electrodes [shielded metal arc (SMA) process]. Of these, the 17-14CuMo stainless steel SMA welds were the strongest relative to the base metal.

The AX heats (AX5 through AX8), along with 17-14CuMo stainless steel, were Gleeble tested to determine the sensitivity of the heat-affected zone (HAZ) near the fusion line to cracking and low ductility failure. All alloys, including the 17-14CuMo stainless steel, exhibited low ductility failures at a temperature 100°C below the melting temperature. Again, the results indicated the potential for welding problems in this class of alloys, especially for heavy-section welding where high restraint could result in high thermally induced stresses during welding.

The AX heats were welded as 13-mm-thick plates using 17-14CuMo stainless steel electrodes, as described in the previous report for the CE heats.[4] Sections taken from the welds were examined metallographically for indications of hot cracking. Typical results for alloys AX6 (0.04% P) and AX7 (0.07% P) are shown in Fig. 6. The high-phosphorus AX7 alloy exhibited extensive cracking, while the lower-phosphorus-containing heats (AX5, AX6, and AX8) revealed fewer cracks in the metallographic and side bend examinations. The weld fusion line in the 17-14CuMo stainless steel was sound, but cracking was observed near the center of the weld after aging.

The AX6 alloy was selected for producing welds with controlled residual element (CRE) type 316 stainless steel.[10] Excellent welds were produced using this GTA filler metal which contains Ti, B, and P additions to improve the strength and ductility of type 316 stainless steel weldments. There were no indications of cracking in either side bend or metallographic investigations.

Tensile tests were performed at 25 and 700°C on specimens taken from weldments. Data are summarized in Table 4 and Fig. 7. Fusion line failures were observed in heat AX7, with high phosphorus, while centerline weld metal failures occurred in the other alloys. Evidence of hot cracking was found on the fracture surface of heat AX7.

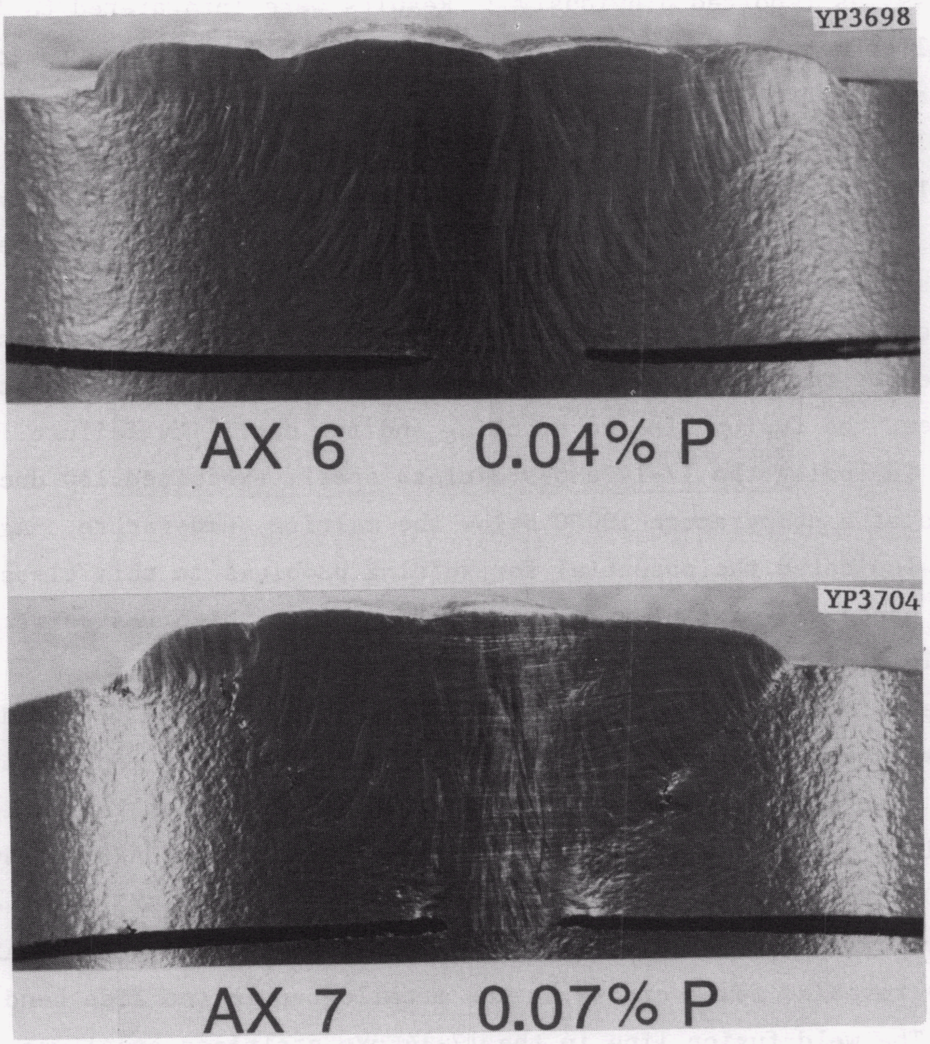

Fig. 6. Side bend test samples of weldments in heats AX6 and AX7.

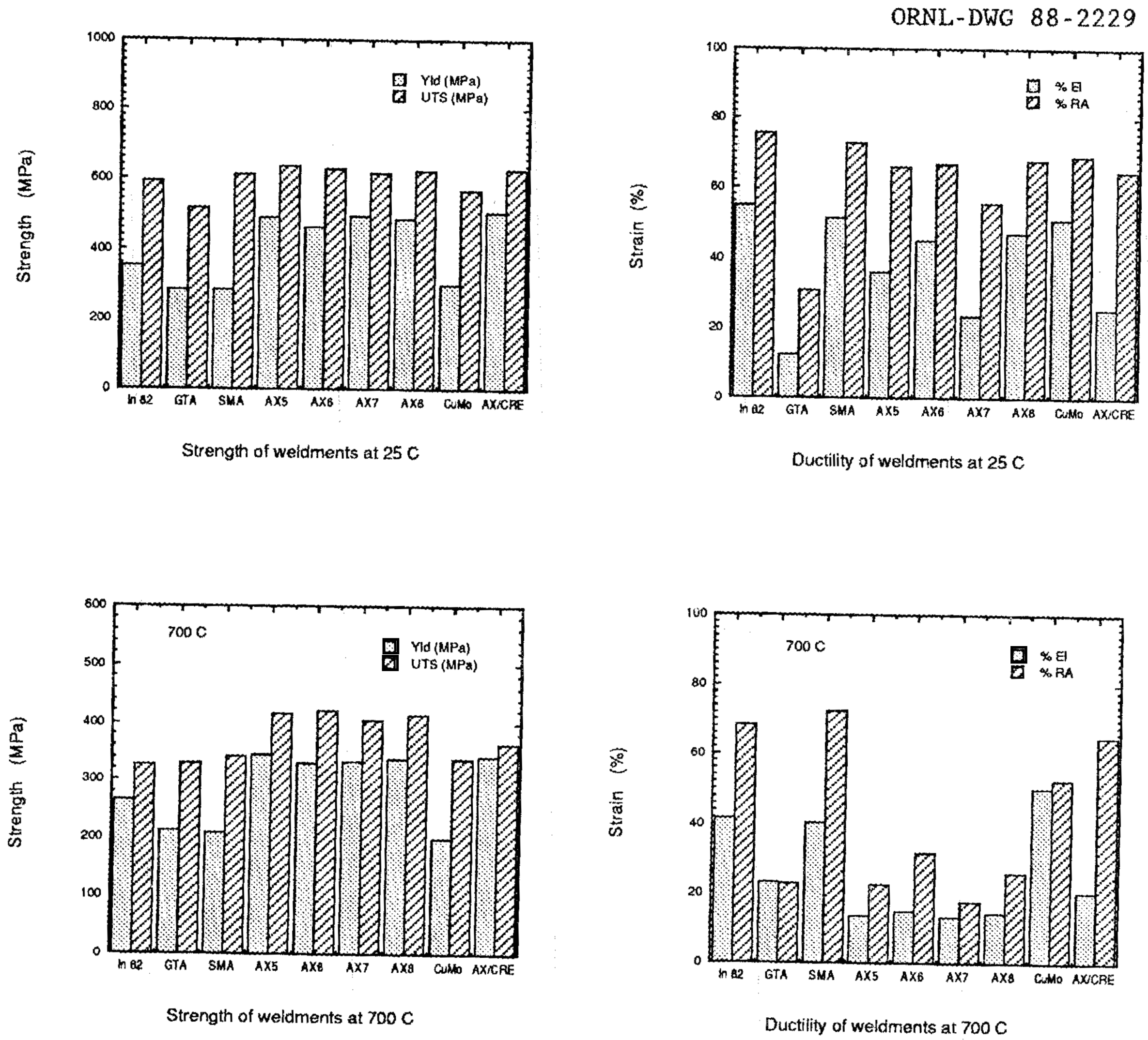

Fig. 7. Comparisons of the tensile properties of weldments in modified type 316 stainless steels at 25 and 700°C.

Table 4. Tensile data for modified type 316 stainless steel weldments (All specimens in as-welded condition)

Specimen	Temperature (°C)	Yield (MPa)	Ultimate (MPa)	Elongation (%)	Reduction of area (%)	Failure location	Type weld
CEW01	25	351	594	55.0	75.5	Weld	In 82 GTA
CEW03	700	266	327	41.5	68.4	Weld	In 82 GTA
CEW08	25	284	518	12.2	30.7	Weld	CuMo GTA
CEW05	700	212	331	23.0	22.6	Weld	CuMo GTA
CEW12	25	283	612	51.4	72.9	Weld	CuMo SMA
CEW09	700	208	341	40.1	72.7	Base	CuMo SMA
AX5-1	25	492	638	35.7	66.1	Base	CuMo SMA
AX5-2	700	344	415	13.5	22.5	Weld	CuMo SMA
AX6-1	25	463	628	45.0	66.8	Base	CuMo SMA
AX6-2	700	330	421	14.7	31.4	Weld	CuMo SMA
AX7-1	25	494	618	23.4	55.6	FL[a]	CuMo SMA
AX7-2	700	333	405	13.2	17.6	FL	CuMo SMA
AX8-1	25	488	627	47.2	68.0	Weld	CuMo SMA
AX8-2	700	337	414	14.4	26.0	Weld	CuMo SMA
CuMo1	25	299	568	51.0	69.5	Base	CuMo SMA
CuMo2	700	199	336	50.0	52.7	Base	CuMo SMA
AX/CRE1	25	505	630	25.4	65.0	Base	316 CRE GTA
AX/CRE2	700	342	364	20.2	64.8	Weld	316 CRE GTA

[a]FL = fusion line.

Creep tests at 700°C and 170 MPa were performed on all alloys. Specimens welded with 316 CRE (GTA) and 17-14CuMo stainless steel (SMA) filler metals failed at the centerline of the weld metal with lives ranging from 2332 to 3567 h. All failures in the 17-14CuMo stainless steel weld metal were low ductility failures with reductions of area less than 5%. The CRE/AX6 weldment failed with the lowest life (2332 h) but with a much improved ductility (39.6% reduction of area). Creep rupture tests are summarized in Table 5 and comparisons are made for the screening test conditions in Fig. 8. Creep rupture tests for other stresses are in progress for both the 17-14CuMo stainless steel and the CRE 316 stainless steel filler metals. The longest weldment tests are approaching 5000 h. Several weldments were placed into aging furnaces to produce 10,000-h exposures.

Table 5. Creep rupture data for modified type 316 stainless steel weldments
(All specimens in as-welded condition)

Specimen	Temperature (°C)	Stress (MPa)	Rupture life (h)	Reduction of area (%)	Status[a]	Failure location	Type weld	Comment
CE2W-01	700	170	771	35.7	R	Weld	In 82 GTA	
CE1W-01	700	170	834	0.6	R	Weld	CuMo GTA	
CE1W-02	700	170	1758	40.0	R	Base	CuMo SMA	
AX5W-03	700	170	2827	0.4	R	Weld	CuMo SMA	
AX5W-04	700	140			I		CuMo SMA	4,000 h
AX6W-03	700	170	2919	4.2	R	Weld	CuMo SMA	
AX6W-04	700	240	259	9.0	R	Weld	CuMo SMA	
AX7W-03	700	170	3645	4.0	R	Weld	CuMo SMA	
AX8W-03	700	170	2819	4.0	R	Weld	CuMo SMA	
CuMo-03	700	170	1681	25.3	R	Base	CuMo SMA	17-14CuMo base
AXCR-03	700	170	2332	39.6	R	Weld	316 CRE GTA	
AXCR-04	700	240	211	23.4	R	Weld	316 CRE GTA	
AXCR-05	730	170	282	49.2	R	Weld	316 CRE GTA	
AXCR-06	730	140	1885	39.0	R	Weld	316 CRE GTA	
AXCR-07	700	140			I		316 CRE GTA	3,000 h
AXCR-08	650	240	620	13.2	R	Weld	316 CRE GTA	
AXCR-09	650	200			I		316 CRE GTA	3,000 h

[a]R = ruptured, I = in test.

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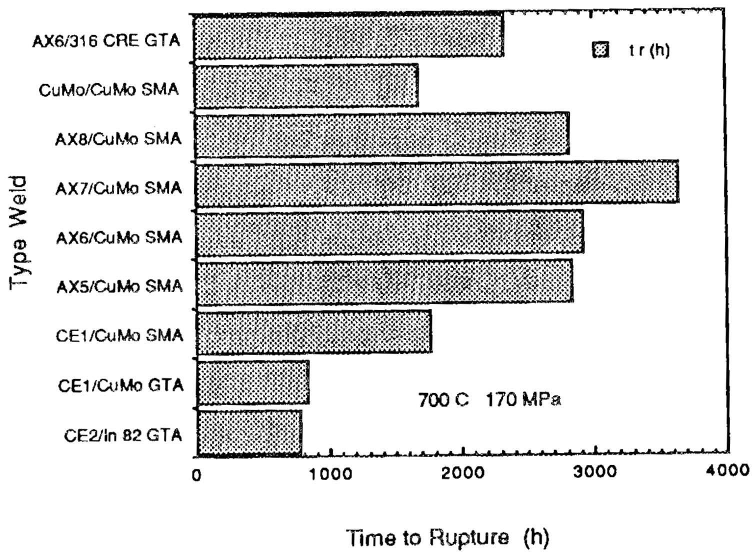

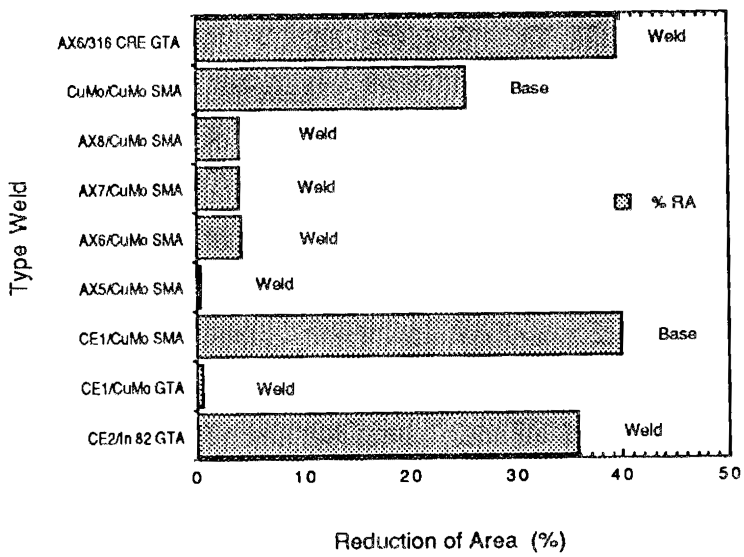

Fig. 8. Comparisons of the creep rupture properties of weldments in modified type 316 stainless steels at 700°C and 170 MPa.

OXIDATION

In the previous report,[4] it was observed that severe oxidation occurred in some specimens of the CE series of alloys during creep rupture testing in the range 700 to 800°C. This phenomenon has also been observed in the AX series of alloys both with and without stress. Generally, the time required for catastrophic oxidation decreased with increasing temperature and occurred in approximately 1000 h at 800°C, 4000 h at 760°C, and 7000 h at 700°C. Even so, most specimens have survived to produce good rupture lives and ductilities. Many samples have exceeded 10,000 h, and a few are still in test after 20,000 h at 700°C with no apparent deterioration in mechanical properties.

Samples of heat AX6 and 17-14CuMo stainless steel were chromized to produce coatings approximately 100 μm deep with an average chromium content of 25%. Peak chromium content reached 35% [see Fig. 9(*a*)]. Creep tests after chromizing were performed at several temperatures and stresses. Results showed that the conditions needed to produce the chromized coating greatly reduced the life and ductility of both alloys. The 17-14CuMo stainless steel was weakened far more than heat AX6, as indicated by the comparative creep curves in Fig. 9(*b*). The superior performance of heat AX6 was attributed to the resistance of this material to creep cavitation and creep crack growth. Cracks that developed in the sigma phase associated with the coating did not propagate in heat AX6. Additional testing of the chromized AX6 alloy is in progress.

MICROSTRUCTURAL OBSERVATIONS

The microstructure that developed as a result of heat treatment and thermal mechanical exposure has been described in a number of papers.[4,8,11,12] The alloys were designed to produce a fine MC precipitate within the matrix and a mixture of coarse $M_{23}C_6$ and fine MC precipitates on the grain boundaries. The MC was expected to stabilize a fine dislocation network produced in the material by hot or cold working to a modest 5 to 10%. Additional stabilization of the microstructure was produced by the formation of phosphide needles throughout the matrix. The high nickel equivalent specified through the alloy composition suppressed the formation

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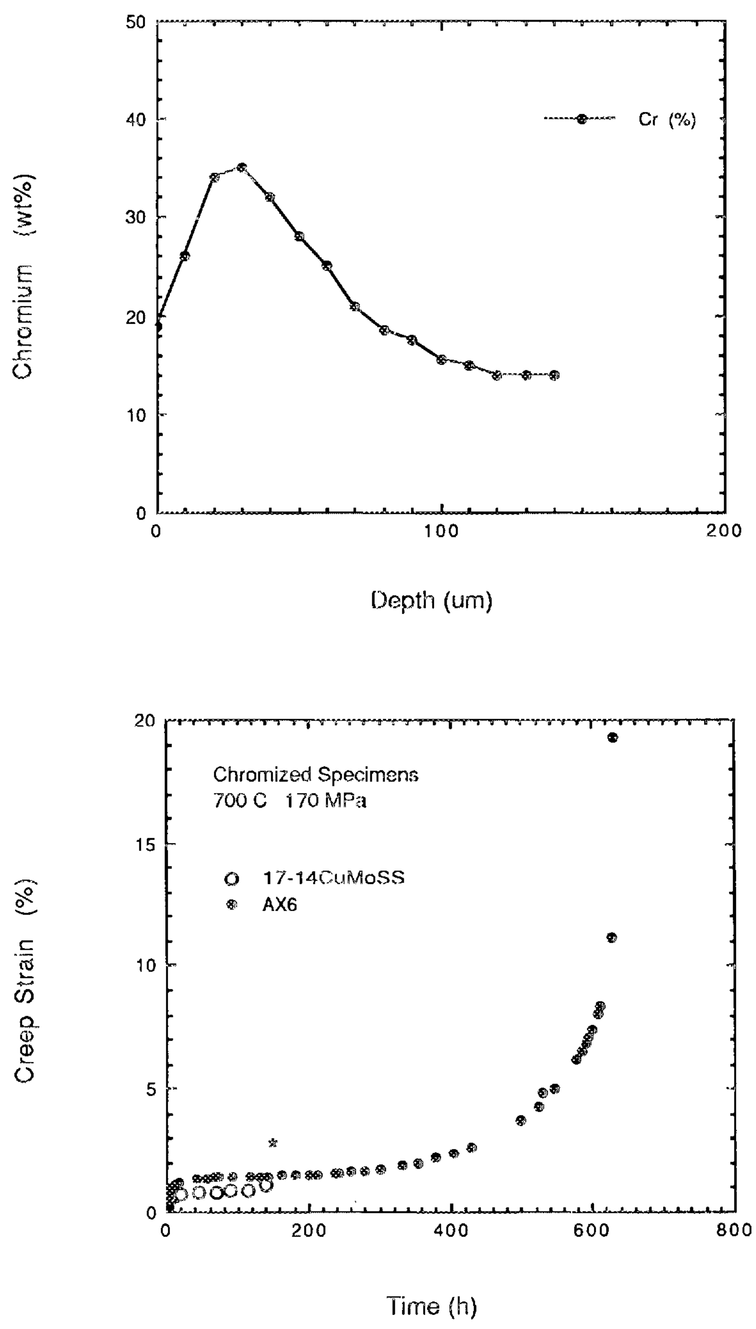

Fig. 9. Results of chromizing studies on modified type 316 stainless steels: (*a*) chromium concentration versus depth in a creep rupture specimen, (*b*) comparison of creep curves for 17-14CuMo stainless steel with heat AX6 at 700°C and 170 MPa.

of sigma phase. Transmission electron microscopy of specimens aged or creep tested confirmed prior expectations. Todd found that aging for 3000 h at 800°C produced little or no change in the size of matrix precipitates.[12] We found that the fine precipitate microstructure remained fairly stable (showing neither coarsening nor dissolution) through the time range 700 to 6000 h at 700°C.

MODIFIED ALLOY 800H

The compositions for the modified alloy 800H series of alloys were selected with the expectation of better resistance to steamside corrosion, by virtue of the 20% Cr and 30% Ni content, and good creep strength, by virtue of the additions of MC forming elements (Ti, Nb, and V). The chromium was kept low to avoid intermetallic phase formation. The compositions of the four alloys, identified as AX1 through AX4, are indicated in Table 6. AX1, AX2, and AX3 were produced to examine the advantages and disadvantages of phosphorus additions, while AX4 was selected to examine the influence of higher chromium. The only commercial alloy produced in the United States known to approach the selected compositions is alloy 800H. Alloys under development by others have some degree of commonality with the modified alloy 800H selections in Table 6,[13-15] but none of them include vanadium additions.

Table 6. Compositions of modified alloy 800H (wt %)

Alloy	C	Si	Mn	Ni	Cr	Ti	Nb	V	Mo	P	B	S	N	Other
800H	0.080	0.24	0.90	31.9	19.5	0.42						0.003		0.43Al
AX1	0.087	0.20	1.99	29.8	19.6	0.27	0.21	0.52	1.98	0.074	0.005	0.012	0.003	
AX2	0.090	0.23	1.96	30.4	20.4	0.36	0.24	0.53	1.96	0.045	0.011	0.009	0.028	
AX3	0.092	0.22	2.00	30.6	20.6	0.36	0.24	0.52	2.00	0.031	0.010	0.010	0.029	
AX4	0.091	0.22	1.97	30.3	25.2	0.36	0.24	0.53	1.97	0.072	0.011	0.009	0.030	

FABRICABILITY

The four alloys were produced by the AMAX Research Laboratory by vacuum-induction melting to produce 20-kg ingots. These were homogenized at 1250°C for 2 h, machined to remove entrapped surface inclusions, and hot rolled at 1100°C with 10% reduction in thickness per pass. Anneals at 1200°C were introduced between passes. The plates were given an anneal at 1200°C for 0.5 h then cold rolled 10% to the final 13-mm thickness. Two plates from each heat were produced.

THERMAL-MECHANICAL TREATMENTS

As with the lean stainless steels, most of the testing on the modified alloy 800H alloys was performed in the mill-annealed condition. A few tests were performed after a 1115°C reanneal, and one heat (AX2) was tested extensively in the 1200°C annealed condition. Tensile strains and aging treatments similar to those introduced into the lean stainless steels were examined in a cursory way.

MECHANICAL BEHAVIOR

The screening test program for mechanical behavior consisted of tensile tests at room temperature and 700°C and creep testing at 700°C and 170 MPa. Additional testing of heats AX2 and AX3 was performed at other stresses and temperatures. A few relaxation tests and creep crack growth tests were performed on heats AX2 and AX3, but the results of these tests will be not be reported here.

Tensile test results are summarized in Table 7 and Fig. 10. All heats were found to possess excellent strength and acceptable ductility in the mill-annealed condition. When solution-treated at 1200°C the yield strength decreased to marginal levels, just as was seen in the case of the lean stainless steels.

A summary of creep rupture data produced on the modified alloy 800H heats is provided in Table 8. Under the screening test conditions (170 MPa at 700°C), the alloys showed a wide range of behavior. The

Table 7. Tensile data for modified alloy 800H

Alloy	Specimen	Condition[a] (°C)	Temperature (°C)	Yield (MPa)	Ultimate (MPa)	Elongation (%)	Reduction of area (%)
800H	800H-08	Mill	25	180	538	54.5	61.1
AX1	AX1-01	Mill	25	456	639	46.3	74.9
AX2	AX2-02	Mill	25	533	690	34.6	62.4
AX2	AX2-06	1115	25	289	643	44.1	64.1
AX2	AX2-01	1200	25	214	598	46.5	61.3
AX3	AX3-08	Mill	25	494	659	35.6	59.1
AX3	AX3-09	1115	25	355	634	38.5	61.7
AX3	AX3-10	1200	25	213	605	53.3	65.8
AX4	AX4-04	Mill	25	507	690	39.5	64.5
AX4	AX4-05	1115	25	368	702	39.3	56.6
AX4	AX4-03	1200	25	230	620	54.2	58.4
AX1	AX1-09	Mill	700	306	451	32.3	42.3
AX2	AX2-11	Mill	700	378	418	37.2	56.0
AX3	AX3-05	Mill	700	347	473	39.5	53.9
AX3	AX3-12	1200	700	128	407	51.6	50.3
AX4	AX4-11	Mill	700	330	478	36.8	56.6

[a]Mill anneal was 1200°C plus 10% cold rolled.

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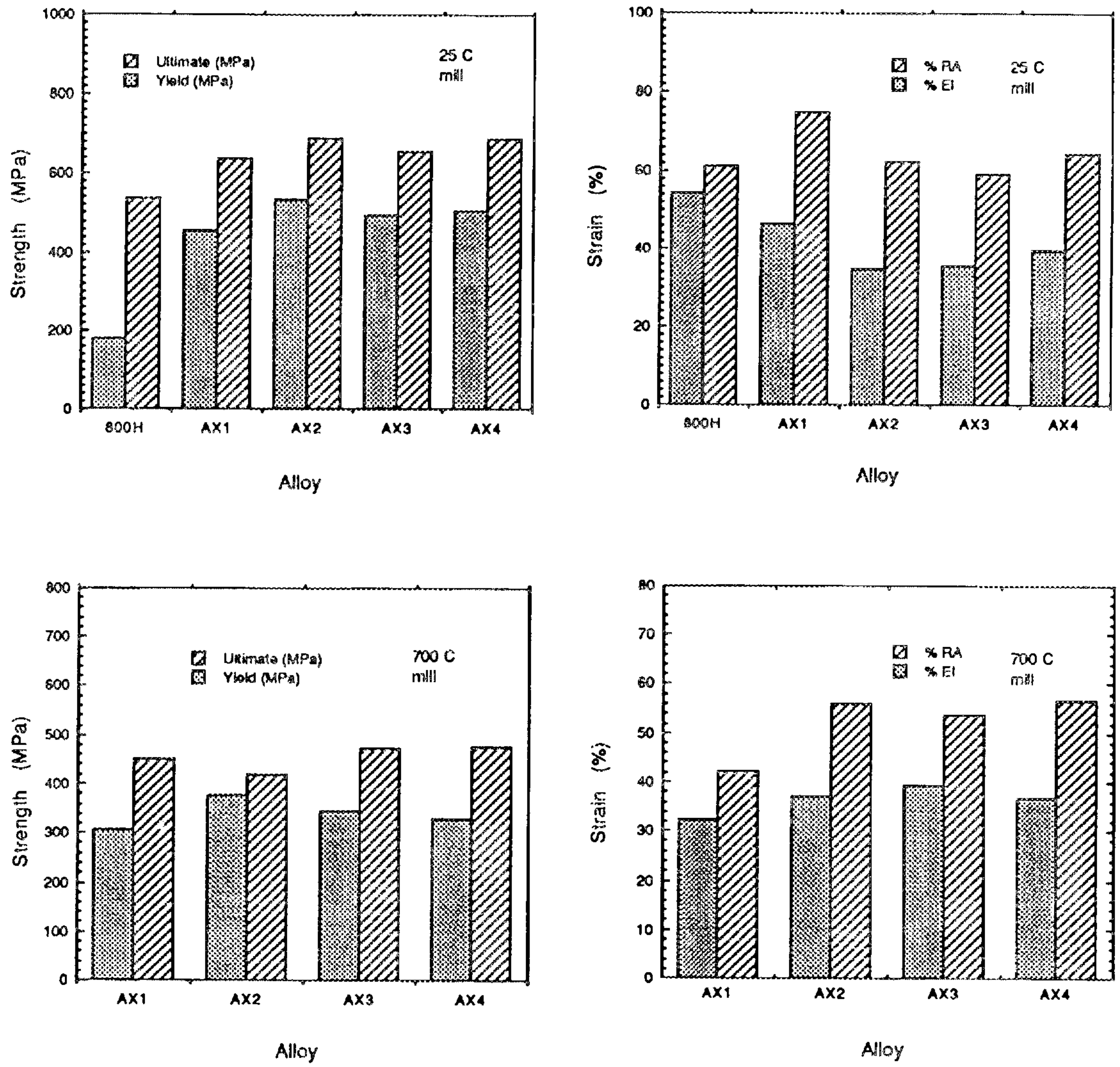

Fig. 10. Comparisons of the tensile properties of mill-annealed modified alloy 800H at 25 and 700°C.

Table 8. Creep rupture data for modified alloy 800H

Specimen	Condition[a]	Temperature (°C)	Stress (MPa)	Minimum creep rate (%/h)	Time to 1% creep (h)	Time to rupture (h)	Elongation (%)	Reduction of area (%)	Status[b]	Comment
AX1-06	Mill	700	170	1.1E-4	6,800.0	8,969	12.8	33.9	R	
AX1-11	1200	700	170	1E-4	2,100.0	2,888	31.1	74.6	R	
AX1-12	1200/2%	700	170	1.2E-4	1,900.0	2,572	23.7	45.6	R	
AX1-13	1200/2%/age	700	170	2E-4	200.0	1,061	39.2	74.8	R	
AX2-03	1200	700	170	1.7E-4	1,800.0	2,987	34.4	70.0	R	
AX2-04	1200	760	200	1.4E-2	0.6	43	32.9	68.6	R	
AX2-05	1200	760	140	6E-3	18.0	207	49.3	74.2	R	
AX2-07	1200	760	170	4E-2	6.2	88	43.0	72.7	R	
AX2-08	1200	700	200	4.6E-4	50.0	1,605	33.0	72.2	R	
AX2-09	1200	700	170	1.8E-4					D	
AX2-10	1200	700	140	8E-5	3,000.0	4,831	46.6	46.7	R	
AX2-12	Mill	700	170	1E-5	6,000.0	12,325	15.8	64.3	R	
AX2-13	1200	760	120	2E-3	147.0	342	70.0	74.2	R	
AX2-15	1200	650	170	1E-5					I	12,000 h
AX2-16	1200	650	240	1E-5	150.0				I	12,000 h
AX2-17	1200	650	200	1E-5					I	12,000 h
AX2-18	1200	760	100	6.4E-4	300.0	1,072	49.5	76.6	R	
AX2-20	1115	700	170	1E-4	60.0	1,040	54.0	74.0	R	
AX2-21	1115	700	240	4E-3	0.4	234	30.1	73.8	R	
AX3-01	Mill	650	300						I	1,000 h
AX3-02	Mill	730	200						I	1,000 h
AX3-03	Mill	700	170	8E-6					I	15,000 h
AX3-07	1200/2%	700	170	1E-4	750.0	2,341	39.0	74.8	R	
AX3-13	1200	700	170	2E-4	200.0	2,743	32.9	67.4	R	
AX3-14	Mill	700	240	1.2E-4	2,750.0	3,041	20.2	63.1	R	
AX3-16	Mill	730	120						I	1,000 h
AX3-17	Mill	760	140						I	1,000 h
AX4-01	1200	700	170	7E-5	475.0	2,232	8.5	9.2	R	
AX4-06	Mill	700	170	1.3E-4	1,090.0	1,090	3.4	2.4	R	

[a]Mill = mill annealed, 1115 and 1200 = annealing temperature, age = 850°C for 24 h. Mill anneal for CE alloys was 1200°C plus 10% hot roll; for AX alloys, 1200°C plus 10% cold roll.

[b]R = rupture, D = discontinued, I = in test.

high-chromium-content alloy (AX4) failed after only 1090 h in a creep brittle manner, while one of the low-phosphorus-content heats (AX3) was still in test after 16,000 h. The other two heats, AX1 and AX2, exhibited exceptionally good lives (8969 and 12,325 h, respectively) and ductilities. Under the same conditions, alloy 800H failed in 110 h. Comparisons are made in Fig. 11 for the time to 1% creep, the rupture life, the elongation, and the reduction of area for the mill-annealed alloys. Most of the life for the AX heats was spent at low strains, and the time to 1% creep was about 75% of the time to rupture. The 1200°C anneal was examined for all four AX heats at the screening test condition and found to decrease the rupture life for heats AX1, AX2, and AX3 to times in the range 2000 to 3000 h. Heat AX4 was improved by the 1200°C anneal to produce a similar rupture life, although the rupture ductility remained low. Specimens of heat AX2 were tested after annealing at 1115°C and found to be weaker than those in the mill-annealed or 1200°C annealed condition. The age at 850°C for 24 h was introduced into one specimen of heat AX2 that had been annealed at 1200°C then cold strained to 2%. The treatment further reduced the rupture life at 700°C and 170 MPa. The 2% cold strain of heats AX1 and AX3 after the 1200°C anneal produced little or no effect on the rupture life. Thus, the screening tests on the modified alloy 800H heats showed that the alloys containing 20% Cr were better than the alloy containing 25% Cr and that the mill-annealed condition (1200°C plus 5 to 10% cold rolling) was superior to other thermal-mechanical treatments.

The creep rupture data for heat AX2 in the 1200°C annealed condition and over a range of stresses and temperatures are summarized in Fig. 12, where they may be further compared with the response for mill-annealed material. Here, it is clear that the benefits of the mill-annealed starting condition extend over a broad range of testing conditions.

WELDABILITY

The modified alloy 800H series of alloys were rolled to 2-mm-thick sheet and evaluated for hot cracking susceptibility in the Sigmajig[9] device. Extensive hot cracking was found for stresses in excess of 50 MPa. Tests on 6-mm-diam bars in the Gleeble machine revealed very little ductility after heating to 1300°C and testing at 1200°C. Thus, data

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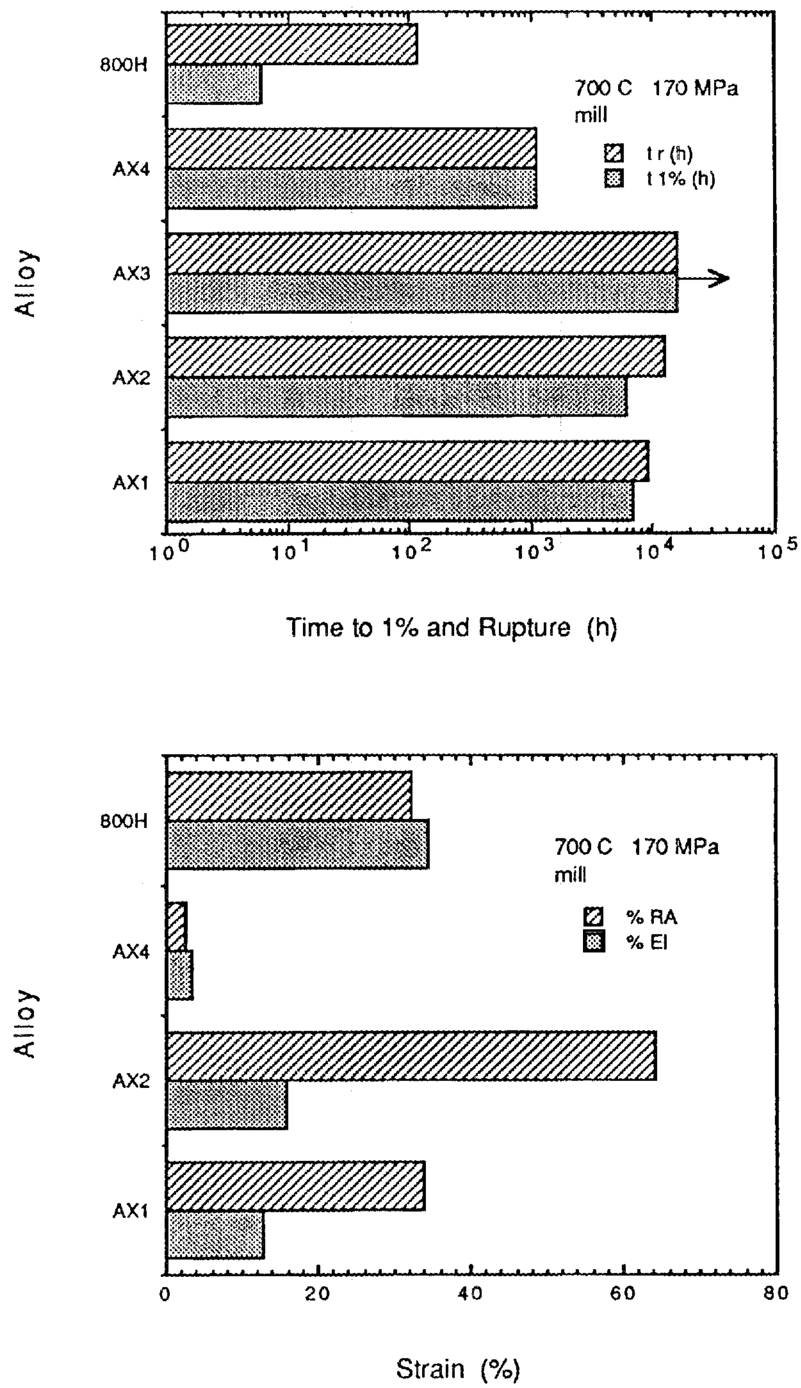

Fig. 11. Comparisons of the creep rupture properties of mill-annealed modified alloy 800H at 700°C and 170 MPa.

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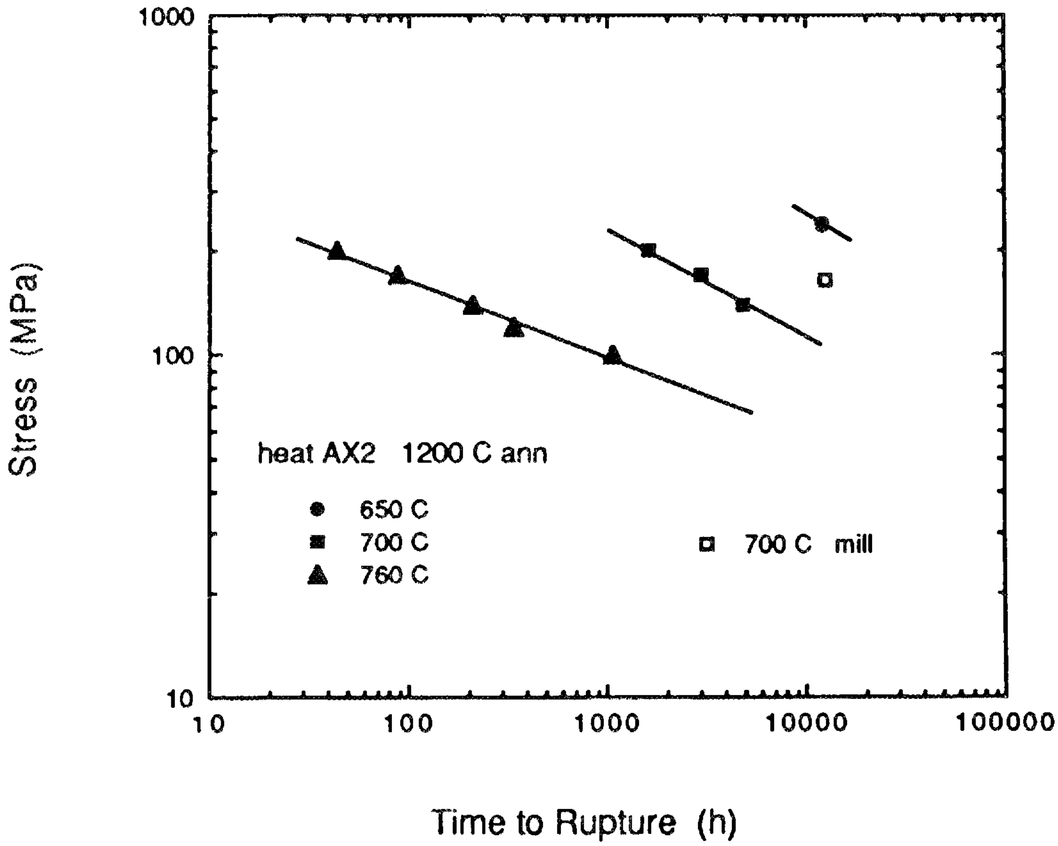

Fig. 12. Effect of stress and temperature on the creep rupture of annealed heat AX2.

indicated that significant problems would be encountered if the alloys were to be autogenously welded in thin sections or welded with filler metal of similar composition in thicker sections. Therefore, the 17-14CuMo stainless steel electrode alloy was selected to make butt welds in 13-mm-thick plates by the SMA process. Side bend tests on the welded samples revealed severe fusion line cracking in the heats containing 0.07% P (heats AX1 and AX4). The two heats with lower phosphorus content (AX2 and AX3) revealed no macroscopic cracks. A comparison is shown in Fig. 13.

Tensile tests on weldment specimens revealed fusion line failures in the heats with high phosphorus content at both room and elevated temperatures. Heat AX4 was the most brittle. Of the two low-phosphorus heats, AX2 was the least prone to fail near the fusion line. Tensile data are summarized in Table 9 and Fig. 14.

Creep rupture tests on weldment specimens at 700°C and 170 MPa produced a fusion line failure in AX4 after 172 h. The other weldments failed at the centerline of the 17-14CuMo stainless steel weld metal after times in the range 2841 to 3222 h (see Fig. 15 and Table 10). The lean stainless steel weldment data, when combined with the results on the modified 800H alloys, suggest that the rupture life increases with phosphorus content when failure takes place in the weld metal. Additional data will be obtained on weldments from the modified alloy 800H aged at 700°C.

OXIDATION

Observations of the surfaces of creep specimens of the modified alloy 800H heats tested at 700°C revealed no unusual features, and it is expected that the alloys would possess corrosion resistance equivalent to alloy 800H.

MICROSTRUCTURAL OBSERVATIONS

Metallographically, creep processes in the modified alloy 800H heats resulted in more grain boundary cavitation than the lean stainless steels. Even so, creep failures were primarily due to plastic instability and gave rise to excellent creep ductilities. Analytical electron microscopy revealed essentially the same type of microstructures as was observed in

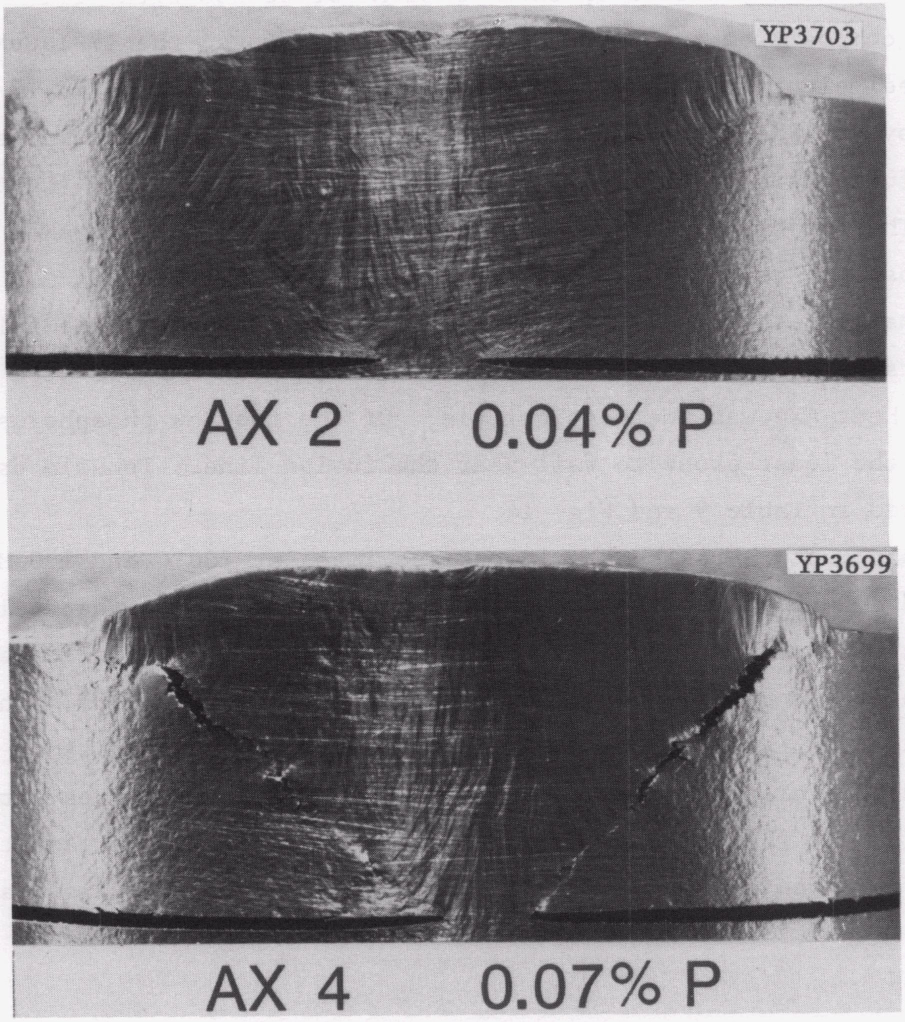

Fig. 13. Side bend test samples of weldments in heats AX2 and AX4.

Table 9. Tensile data for modified alloy 800H weldments

(All specimens in as-welded condition)

Specimen	Temperature (°C)	Yield (MPa)	Ultimate (MPa)	Elongation (%)	Reduction of area (%)	Failure location	Type weld
AX1W-01	25	461	603	9.0	18.3	FL[a]	CuMo SMA
AX1W-02	700	316	414	17.5	17.6	FL	CuMo SMA
AX2W-01	25	515	684	19.6	47.6	Weld	CuMo SMA
AX2W-02	700	345	414	13.4	39.2	Weld	CuMo SMA
AX3W-01	25	494	639	11.9	28.1	Weld	CuMo SMA
AX3W-02	700	329	428	20.1	21.1	FL	CuMo SMA
AX4W-01	25	474	510	1.2	2.5	FL/weld	CuMo SMA
AX4W-02	700	264	264	0.8	0.4	FL	CuMo SMA
CuMoW-1	25	299	568	51.0	69.5	Base	CuMo SMA
CuMoW-2	700	199	336	50.0	52.7	Base	CuMo SMA

[a]FL = fusion line.

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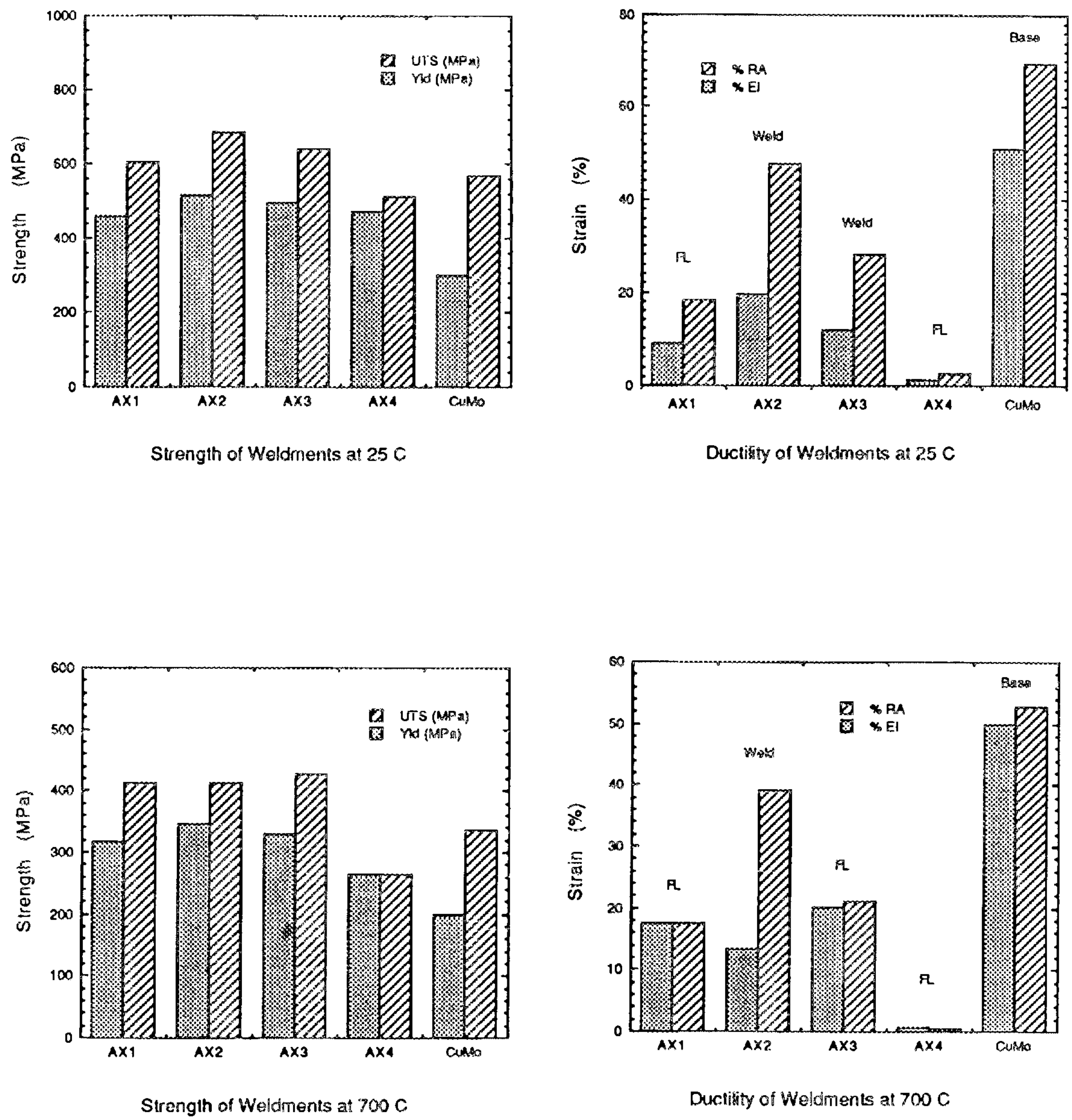

Fig. 14. Comparisons of the tensile properties of weldments in modified alloy 800H at 25 and 700°C.

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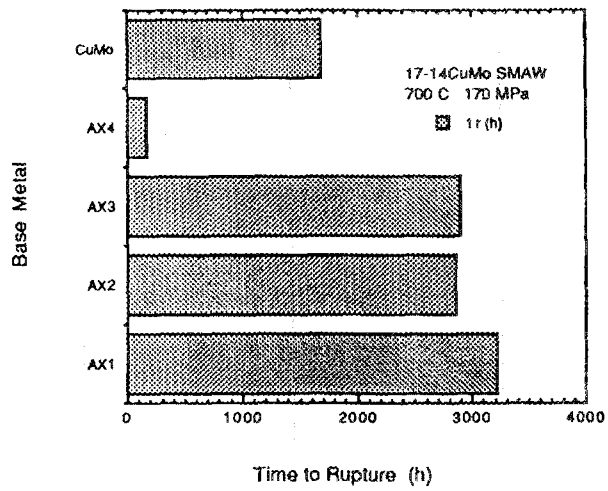

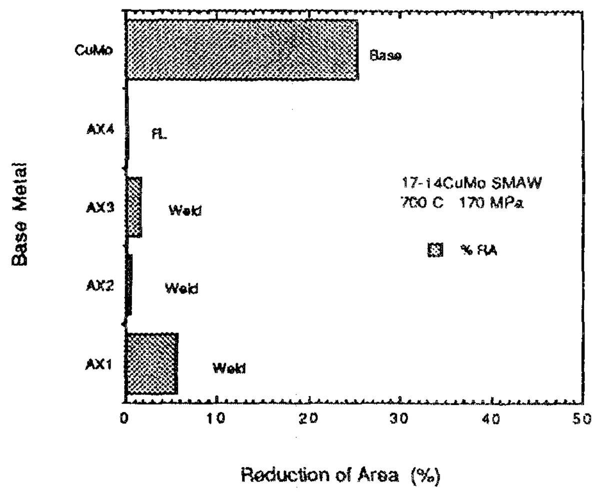

Fig. 15. Comparisons of the creep rupture properties of weldments in modified alloy 800H at 700°C and 170 MPa.

the lean stainless steels. Hardening was due to the development of fine precipitate-stabilized dislocation microstructures within the matrix and large blocky carbide precipitates along the grain boundaries. Some phosphide needles were observed. The microstructures of the modified alloy 800H heats were quite different compared to alloy 800H. The latter had fine titanium-rich MC and coarser $M_{23}C_6$ in the matrix, and $M_{23}C_6$ aligned along grain boundaries. Although the matrix MC was fine, it seemed ineffective at pinning dislocations. By contrast, the Ti-, Nb-, and V-rich MC found in the modified alloy 800H heats was extremely effective at pinning dislocations. This confirmed the important synergistic effects between the combination of Ti, Nb, and V in strengthening the modified alloys.

ALUMINUM-BEARING AUSTENITIC ALLOYS

The aluminum-bearing austenitic alloys that were initially of interest fell into two groups. The first group consisted of essentially 300 series stainless steels in which some of the chromium was replaced by aluminum. These included an alloy developed by Foster-Wheeler identified as FW4C (ref. 16) and one of a series of aluminum-bearing austenitic alloys (identified as heat 898) studied by McCurdy and Moteff.[17] However, before these alloys could be produced and evaluated as part of the screening program, a new assessment report issued by ORNL concluded that exceptionally high strength would be needed in superheater tubing alloys for service above 600°C and that only materials like nickel aluminide and structural ceramics offered such potential.[18] In response to this, emphasis was placed on alloys in a second group of aluminum-bearing austenitic alloys, namely nickel aluminides.[19-21] Over 300 compositions were available for selection, but after comparing the alloy design criteria[3] with the expected properties, the choices were narrowed to three. The compositions for these alloys are provided in Table 10, which include one aluminum-bearing austenitic stainless steel (alloy 898), one wrought Ni-Cr aluminide (IC218), two wrought Ni-Cr-Fe aluminides (IC283 and IC357), and one investment cast Ni-Cr aluminide (IC221).

Table 10. Creep rupture data for modified alloy 800H weldments

Specimen	Temperature (°C)	Stress (MPa)	Rupture life (h)	Reduction of area (%)	Failure location	Type weld
AX1W-03	700	170	3,222	5.6	Weld	CuMo SMA
AX2W-03	700	170	2,858	0.6	Weld	CuMo SMA
AX3W-03	700	170	2,899	1.6	Weld	CuMo SMA
AX4W-04	700	170	172	0.1	FL[a]	CuMo SMA
CuMoW-3	700	170	1,681	25.3	Base	CuMo SMA

[a]FL = fusion line.

FABRICABILITY

The modified 300 series stainless steel (alloy 898) was supplied by the University of Cincinnati as 25-mm-diam rod produced by the Hoskins Manufacturing Company. The alloy was air-induction melted as a 700-kg heat and cast as several 170-kg ingots, normalized at 1250°C, and hot rolled to the final product. The IC218 alloy was a reduced zirconium version of a high strength Ni-Cr aluminide with the same designation that had been produced as both wrought and cast products. At the zirconium level specified in Table 11, it was expected that the material would be readily workable. Combustion Engineering produced a 38-kg ingot by argon induction melting virgin materials. The ingot could not be hot rolled without cracking; hence, samples were sawed from the ingots and ground to the following dimensions: 25 × 70 × 200 mm. These were solution-treated at 1175°C for 0.5 h. Subsequent efforts to cold roll and hot roll produced severe cracking for even small reductions (10%). Similar surface-conditioned samples were delivered to ORNL for fabrication, and identical

Table 11. Compositions of aluminum-bearing austenitic alloys

Alloy	Al	Cr	B	Zr	Fe	Mo	Ni	Other
898	4.8	14.6		0.20	Bal		26.0	0.5Si, 0.025C, 0.01Y
IC218	8.5	8.0	0.026	0.38	0.2		Bal	
IC283	9.0	7.1	0.026	0.33	13.3		Bal	
IC357	9.5	7.0	0.020	0.30	11.2	1.30	Bal	
IC221	8.2	7.8	0.020	1.70			Bal	

problems were encountered. Work on this heat of IC218 was abandoned. The fabrication route for IC283 at Combustion Engineering was similar to that for the IC218 alloy and produced similar results. The conditioned samples were rolled at ORNL, where it was found that the IC283 could be hot rolled with less than 20% through-thickness cracking when stainless steel cladding was used to minimize surface cooling from the rolls. Plate, approximately 13-mm thick, was produced for testing. The IC357 heat was melted at Combustion Engineering and delivered as an ingot to ORNL where it was hot extruded to square bar. This was subsequently hot rolled to 7-mm-thick plate. Surface cracks extending to 1 mm were observed, but there was sufficient material for mechanical and corrosion testing.

THERMAL-MECHANICAL TREATMENTS

The aluminides were annealed at temperatures in the range 1050 to 1200°C. However, the addition of chromium and iron to the nickel aluminides produced a detectable amount of disordered phase and beta phase (NiAl) in the ordered matrix;[21] hence, some of the alloys were aged 24 h at 815°C to increase the content of ordered phase.

MECHANICAL BEHAVIOR

The tensile properties produced in the screening tests on the aluminum-bearing austenitic alloys are provided in Table 12 and are summarized in Fig. 16. Characteristically, the aluminides exhibited yield strengths that increased with temperature between 25 and 700°C. The ultimate strengths were very high, but decreased between the same two temperatures, while the ductilities were low and marginal relative to the requirements for most tubing applications.

Creep rupture data are provided in Table 13 and Fig. 17. These include results from one test on alloy 898, the aluminum-modified stainless steel; several tests on IC283, IC357, and IC221; and one test result reported by Liu on IC357 for material from a small 0.5-kg melt.[20] Only heats IC283 and IC357 were tested at the screening conditions used for the modified 316 stainless steels and the modified alloy 800H heats. However, sufficient data were gathered on all alloys to show that alloy 898 was weak

Table 12. Tensile data for aluminum-bearing austenitic alloys

Alloy	Specimen	Condition[a] (°C)	Temperature (°C)	Yield (MPa)	Ultimate (MPa)	Elongation (%)	Reduction of area (%)
898		1200	25	208	615	47.8	66.3
898		1200	700	377	517	18.0	29.0
IC283	283-01	Mill	25	634	852	15.2	15.2
IC283	283-02	Mill	700	667	780	4.9	9.2
IC357	357-X1	1050/age	25	579	1,171	30.6	
IC357	357-X2	1050/age	600	717	1,040	22.0	
IC357	357-05	1100	25	725	1,430	21.8	26.8
IC357	357-01	1100	700	758	897	13.7	19.6
IC221	221-12	Cast/age	25	426	854	17.8	18.8
IC221	221-16	Cast/age	704	604	823	14.2	24.1

[a]Mill = mill annealed, 1115 and 1200 = annealing temperature, age = 850°C for 24 h. Mill anneal for CE alloys was 1200°C plus 10% hot roll; for AX alloys, 1200°C plus 10% cold roll.

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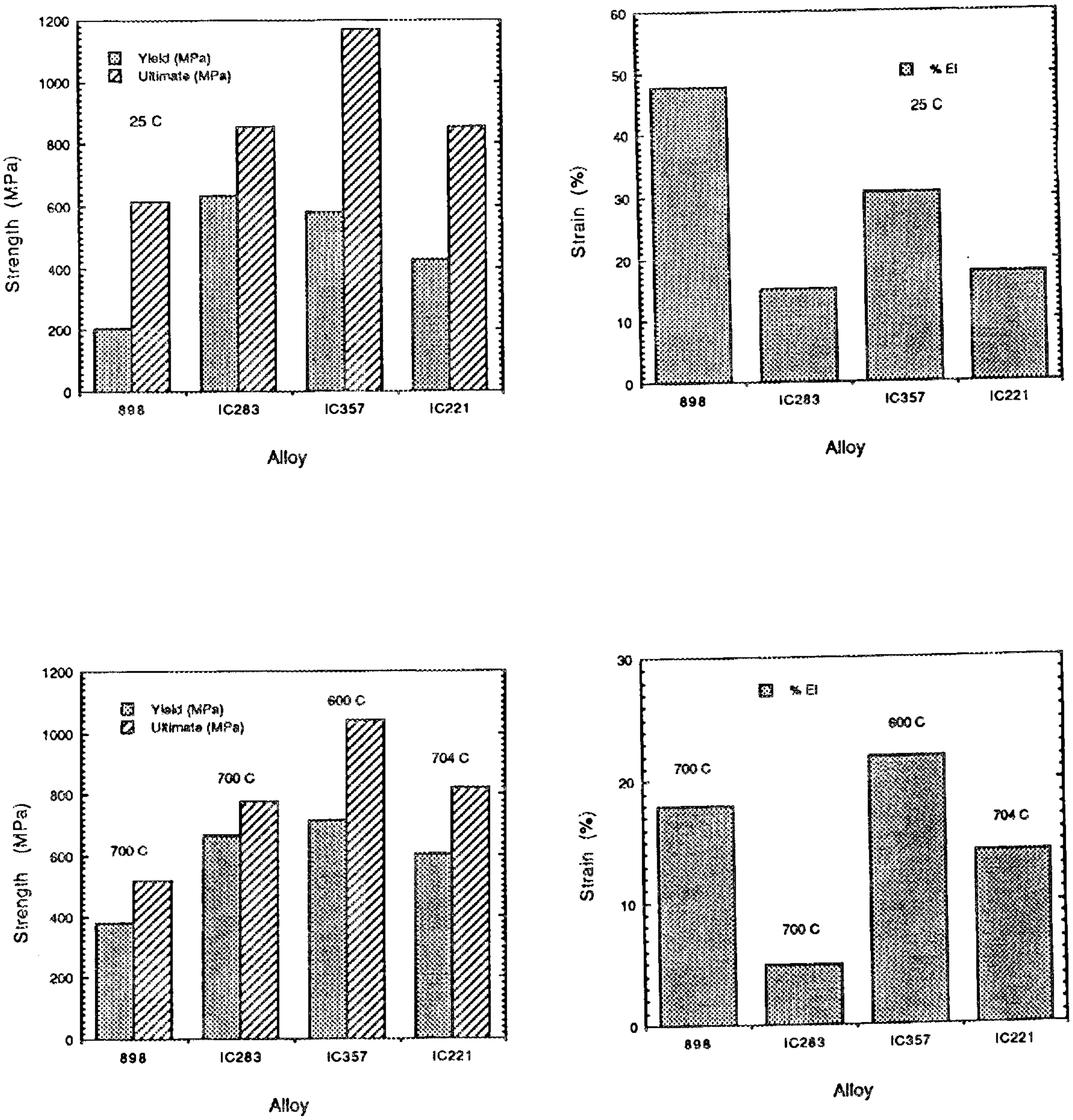

Fig. 16. Comparisons of the tensile properties of aluminum-bearing austenitic alloys at 25°C and elevated temperature.

Table 13. Creep rupture data for aluminum-bearing austenitic alloys

Specimen	Condition[a] (°C)	Temperature (°C)	Stress (MPa)	Minimum creep rate (%/h)	Time to 1% creep (h)	Rupture life (h)	Elongation (%)	Reduction of area (%)	Status[b]	Comment
898-01	1200	649	138	1.3E-3		2,962	4.3	8.6	R	
283-03	Mill	700	170		150.0	150	2.0	0.5	R	
283-04	Mill	700	240	2.5E-3	143.0	143	2.1	0.4	R	
283-05	Mill	700	120	3.8E-4	3,759.0	3,759	2.0	0.1	R	
283-06	Mill	700	240	3E-3	140.0	153	2.8	0.8	R	
283-07	Mill	760	170	1E-2	64.0	67	3.0	0.1	R	
283-08	Mill	700	170	1.6E-3	451.0	451	3.1	1.6	R	
283-09	Mill	730	170	3.4E-3	175.0	189	2.6	0.2	R	
283-10	Mill	760	120	2.8E-3	290.0	395	5.2	1.2	R	
283-11	Mill	800	80	5E-3	200.0	651	9.0	4.7	R	
357-03	1050/age	760	139	NA[c]	NA	220	42.0	NA	R	
357-04	1100	760	170	2E-2	19.0	92	NA	NA	R	
357-05	1100	700	240	1.1E-2	90.0	331	13.4	17.0	R	
357-06	1100	700	170		110.0				I	
221-03	Cast/age	815	310	2.2E-2	41.0	341	21.9	24.9	R	
221-04	Cast/age	760	207	1E-4	4,100.0				I	16,000 h
221-07	Cast/age	760	310	2E-3	440.0	4,217	22.9	20.8	R	
221-08	Cast/age	815	207	3E-3	260.0	1,900	NA	NA	R	
221-14	Cast/age	704	207	2E-5					I	8,000 h

[a]Mill = mill annealed, 1115 and 1200 = annealing temperature, age = 850°C for 24 h. Mill anneal for CE alloys was 1200°C plus 10% hot roll; for AX alloys, 1200°C plus 10% cold roll.

[b]R = ruptured, I = in test.

[c]NA = not available.

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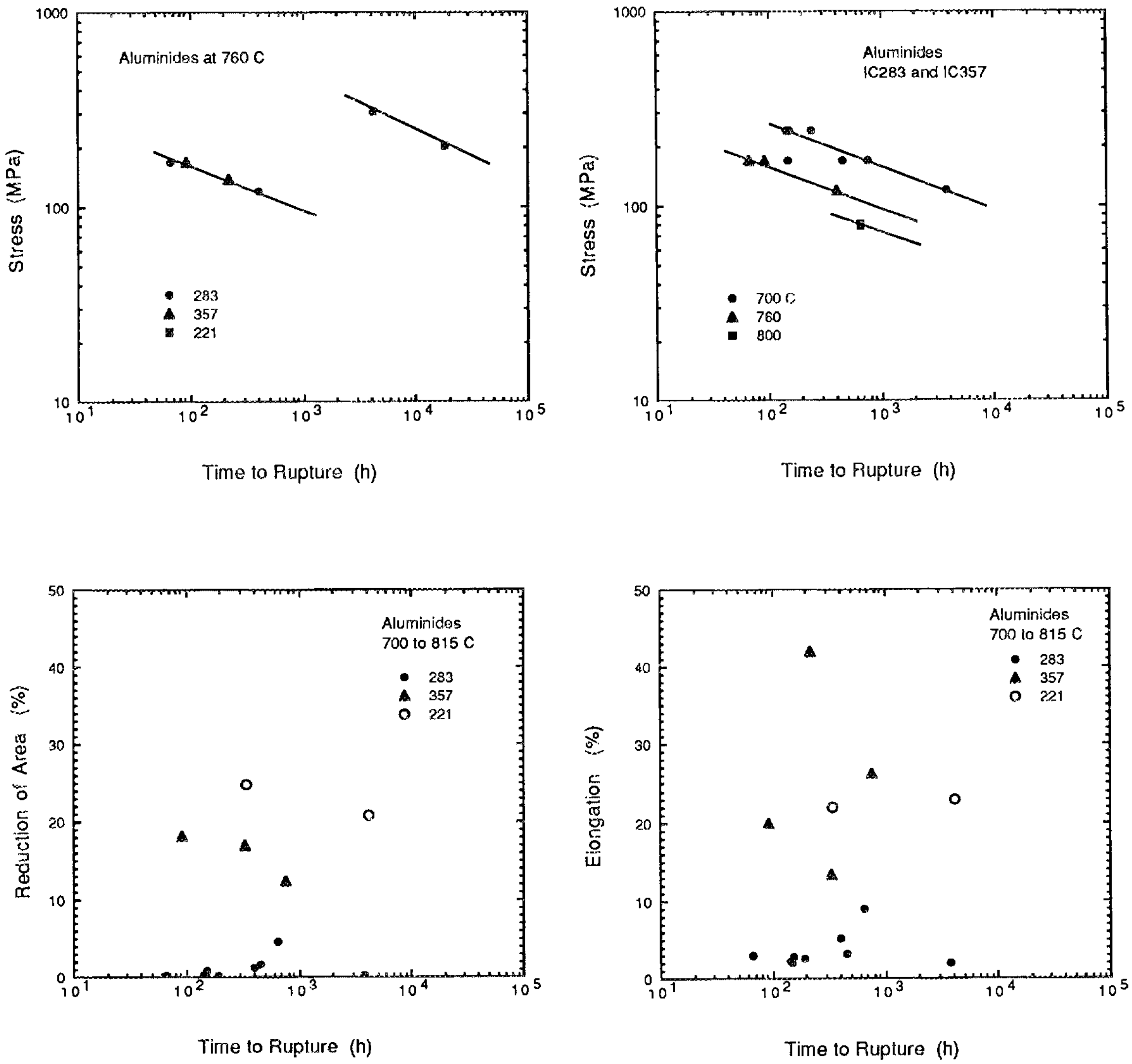

Fig. 17. Comparisons of the creep rupture behavior of aluminum-bearing austenitic alloys at several temperatures and stresses.

with low ductility, IC283 had moderate strength and poor ductility, IC357 had moderate strength and fair ductility, and the cast IC221 alloy had excellent strength and ductility. Additional testing data for IC357 will be reported at a later date.

WELDABILITY

The weldability of the aluminides was assessed by running an autogenous GTA weld across machined blocks of the IC218 and IC283 alloys. Both showed cracks in the melted material running several millimeters into the HAZ. By comparison, the modified lean stainless steels and modified alloy 800H under similar welding conditions exhibited centerline cracking in the melted material with no macroscopically detectable extension into the HAZ. The weldability of the aluminides is being evaluated separately from this activity.[2]

OXIDATION

The aluminides exhibited excellent resistance to oxidation at all temperatures of creep testing (700 to 815°C).

MICROSTRUCTURAL OBSERVATIONS

Microstructural studies were limited to the examination of creep-tested specimens. It was found that all creep failures were intergranular. Second phases in IC221 and IC283 were often present on these grain boundaries, but it is not known whether these precipitates contributed to the failure mechanism.

ALLOY COMPARISONS

The alloy design criteria on which the individual alloys and alloy groups are being evaluated have been specified in a previous document.[3] Criteria fall into five categories: metallurgical stability, fabrication and joining, mechanical properties, fireside corrosion, and steamside corrosion. Results that have been produced on the alloys have largely covered the metallurgical stability, fabrication, joining, and mechanical properties areas. Work on fireside corrosion testing will begin in early 1989, and significant information on alloy stability should be produced by mid-1989. Nonetheless, all criteria will be discussed in this section on alloy comparisons.

METALLURGICAL STABILITY

The first three criteria specified regarding metallurgical stability were concerned with the tendency of Fe-Cr-Ni alloys to form embrittling intermetallic phases such as sigma, Laves, and chi. The low chromium and high nickel contents of the modified 316 compositions virtually assure the absence of massive sigma phase, and no sigma phase has been observed in specimens aged to long times either with or without stress. Laves phase and sigma phase formation could be enhanced by minor additions of titanium and niobium to the alloy. However, the entire combination of minor residual elements, including carbon, boron, phosphorus, and reduced silicon, was specifically designed to counteract the intermetallic phase promotion of titanium and niobium, and the alloy modification was successful in this. More detailed analysis of the stability in this respect falls under the work scope of a subcontract at the Cornell University.[2,8] Good metallurgical stability was also expected in the case of the modified alloy 800H compositions AX1 through AX4. In contrast, the higher chromium steels being developed elsewhere, such as the modified 310 stainless steel composition,[14] could be susceptible to sigma formation. Because of the higher niobium and silicon levels permitted in 17-14CuMo stainless steel, both sigma and Laves phase could be expected, and they are observed in this alloy,[6] with concomitant embrittlement. Similar problems could be developed in niobium-modified 20Cr-25Ni stainless steel and 310 stainless steel. The ORNL-modified steels in both the 14Cr-16Ni-Fe and 20Cr-30Ni-Fe groups contain molybdenum, which is known to promote chi phase formation in type 316 stainless steel. However, microstructural examination, including X-ray energy dispersive spectroscopy, revealed no chi phase. Again, the minor alloying element combinations used to modify the alloys were specifically selected to eliminate chi phase. The aluminides are not expected to form sigma, Laves, or chi, but they contain a significant amount of disordered gamma, some beta phase which is also brittle, and can contain a zirconium precipitate from the disordered phase on the grain boundaries. These phases are probably the cause for the low ductility observed in IC283. Vigorous efforts are under way to find ways to avoid these problems in the aluminides.[19,20]

The fourth and fifth criteria specified regarding metallurgical stability were concerned with the nature and stability of the fine strengthening precipitates in the modified 316 stainless steels and the modified alloy 800H compositions. The original alloy design concepts called for the introduction of several MC-forming elements that could lead to the development of a fine matrix precipitate that would stabilize the dislocation network and resist creep. Further stabilization of the matrix microstructure was expected from the formation of phosphide needles. The CE and AX series of alloys were found to develop this microstructure, provided that a high solution temperature could be used in combination with enough warm or cold work to develop a dislocation density approaching $10^{14}/m^2$ prior to service.[6] This microstructure is characterized by a room-temperature yield strength which exceeds 300 MPa. However, the long time stability of such a matrix substructure at service temperatures was of serious concern, and it fell within the work scope of the subcontract at Cornell University to develop models to predict aging effects. This work is still in progress. Data produced to date from creep testing indicate that microstructures were far more stable than would be expected from the experience gained on other alloys, but much study is needed in this area. Commercial alloys, such as the 300 series stainless steels and alloy 800H, were observed to be unstable if cold worked to high strength levels.[21,22] Recrystallization and grain boundary migration often produce problems in commercial steels and alloys that have not been observed in the CE and AX series of alloys at 700°C. The aluminides were not precipitation hardened; hence, there is no reason to expect loss of strength or embrittlement on aging.

The sixth criterion addressed the need to clad alloys for protection against fireside corrosion. The screening task work activity did not examine this problem. However, cladding of commercial stainless steels, such as Esshete 1250 and 17-14CuMo, has been accomplished elsewhere;[7] hence, there is reason to expect that the CE series and AX5 through AX8 series could also be clad. Similarly, claddings on alloy 800H have been produced,[15] so there is reason to be optimistic about cladding the AX1 through AX4 series of alloys. No efforts have been made to clad the aluminides, although coextrusion with 300 series stainless steels has been used in producing material.[23] Pack-aluminizing of $(Ni,Cr)_3Al$ to produce a NiAl surface layer affords an additional approach for combatting fireside corrosion.

The final criterion for metallurgical stability concerns the ability to tolerate at least 15% cold work without the need for annealing. The CE and AX series of alloys demonstrated this capability. The low-room-temperature ductility of the aluminides was marginal, at best, and cold straining of tubing could reduce ductility to marginal levels. Powder-derived aluminide products apparently have excellent ductilities and should tolerate high levels of cold work.[23]

FABRICATION AND JOINING

The first three alloy design criteria applicable to fabrication and joining technology concerned the need to produce tubing of the size and quality required for superheater/reheater construction. For the modified austenitic stainless steels, the applicable specification was chosen to be ASTM A 213, while for the modified alloy 800H the applicable specification was ASTM B 407. Since the aluminides represent an emerging technology, no specifications are considered applicable. The melting practices utilized for the CE and AX series of alloys were selected to produce residual element contents typical of commercial practices, and the subsequent chemical analyses indicated that the composition ranges originally specified[4] could be met. Two problems were identified that had to be addressed. The first involved the loss of titanium during attempts to centrifically cast tube hollows at Combustion Engineering. The problem was surmounted by casting solid ingots, cropping the top, and subsequently rolling sound material, free of titanium dioxide. The second problem, observed by AMAX, was a poor surface condition of as-cast ingots that produced poor surface finishes after hot rolling. The problem was solved by surface machining before hot rolling. Using these approaches, it should be possible to produce tubing of these materials to the applicable specifications. The aluminides, however, remain a formidable fabrication problem. Although the alloys could be cast without severe cracking, the subsequent hot or cold working produced cracking. Alloy IC218, which was expected to have the best strength, could not be worked, and alloys IC283 and IC357 exhibited extensive surface cracking that suggested problems would be encountered in producing defect-free tubing. Much work remains in the fabrication technology of the aluminide alloys.

The fourth criterion on fabricability addressed the need for compatibility with corrosion-resistant cladding. This issue was not resolved by the screening program but will be included in the work scope of a subcontract on fabrication technology undertaken by Babcock and Wilcox Research Laboratory.[2]

The fifth criterion identified the need for chromizing the lean stainless steels to protect against corrosion on the steamside. It was demonstrated that plate and rod samples could be chromized to levels that met the target concentrations and depths. However, the subsequent tests on the chromized samples clearly revealed that the high chromium coatings had a deleterious affect on base metal strength. The 17-14CuMo stainless steel behaved very poorly from a mechanical point of view, although it has been shown to perform well under stress-free, short-time fireside corrosion testing conditions.[7] Work on surface treating will continue under a subcontract with Babcock and Wilcox Research Laboratory and another with Ohio State University,[2] but for the short term it is assumed that none of the austenitic stainless steels are amenable to chromizing. If this assumption holds true, the usefulness of the lean stainless steels in superheater/reheater applications will depend greatly on their innate steam corrosion resistance, which will be assessed under an industrial subcontract at some future date.[2]

One of the most important alloy design criteria for superheater tubing is that the alloy be weldable by procedures specified in the *ASME Boiler and Pressure Vessel Code*, Sect. I, on power boilers. The CE and AX series of alloys were joined as 13-mm plate in a restrained configuration using three different filler metals - Inconel 82, 17-14CuMo stainless steel, and CRE 316 stainless steel. Both GTA and SMA welding processes were examined. Although the CE alloys with high phosphorus (0.07%) were joined with no severe problems, the high-phosphorus AX series of alloys exhibited hot cracking and were judged to be nonweldable for this reason.

All of the modified alloy 800H alloys were judged to be susceptible to hot cracking. Welding of actual tubing would be required to make meaningful evaluations of the weldability. No successful welds have been produced in thick-section aluminides using any type of filler metal. Thus, the weldability of this class of alloys remains an unsettled issue, although work to develop joining methods is under way under the sponsorship of another program.[2] Of the filler metals that were examined in connection with the modified 316 alloys and modified alloy 800H compositions, the Inconel 82 appeared to be too weak, the 17-14CuMo stainless steel too brittle, and the CRE 316 stainless steel a reasonable compromise for the near term. The fact that the weldments in alloys containing 0.04% P or less could not be ruptured near the fusion line was taken as an encouraging sign, but full-scale testing of weldments would be necessary before any meaningful assessments could be made. A subcontract to develop a joining technology for the modified alloys is being placed at the University of Tennessee.[2]

The seventh alloy design criterion relating to fabrication and joining identified the requirement to make tube-to-header joints. Included was the possible need for dissimilar metal joints. Again, the satisfaction of the criterion for any of the alloys must be demonstrated on tubing, and such a demonstration did not form part of the screening evaluation.

MECHANICAL PROPERTIES

Eight criteria were identified that involved mechanical properties. All criteria addressed performance requirements for long times and high temperatures, since the short-time performance requirements were covered by the applicable ASTM specifications on fabrication. The screening testing conditions were not adequate to assure that the promising heats would meet the criteria, but were useful in eliminating some alloy compositions and thermal-mechanical treatments that would not meet the strength and ductility requirements. It was established earlier[4] that the rupture life, rather than the time to produce 1% creep, was the proper index for ranking the alloys from the fact that the long-time design stress would be controlled by either the minimum or the average strength to produce rupture in the reference time frame (100,000 h). Based on this assumption, Larson-Miller parametric curves were constructed that represented the minimum and average rupture strengths required for an alloy that would meet the target design strength level of 60 MPa at 700°C.[3] Following rules often used in setting code allowables, the target design strength conformed to the lower value of either 80% of the minimum or 67% of the average strength at the time and temperature of interest. Of course, insufficient data existed for the heats to establish the minimum or average strengths in a statistical sense, but for assessing the potential of the various alloys and heat treatments it was assumed the alloys with the best potential would have average or better-than-average strengths. The design curves needed for the assessment are shown in Fig. 18. The target strength is shown as the symbol, while the dashed line represents the trend for minimum strength, and the solid line represents the trend for average strength. The slopes of the curves were based on the trend of the design strength versus temperature for type 316 stainless steel. Any heat or heat treatment that produced rupture strength data falling near or above the trend for average strength (solid line) was judged to be worthy of consideration on a rupture strength basis. Heats and alloys that produced data that fell near the minimum strength or crossed the minimum strength curve were judged to be unacceptable. Other considerations, such as tensile properties, notch toughness, creep embrittlement, or oxidation resistance, of course, would enter into the final assessment of the heats acceptable from a strength viewpoint, but such factors were not considered here. Comparisons are made in Figs. 18 and 19. Those alloys that showed promise were three heats in the mill-annealed CE series (CE0, CE2, CE3); all four modified 316 stainless steel heats in the mill-annealed AX series (AX5, AX6, AX7, and AX8); two heats in the modified alloy 800H mill-annealed series (AX2 and AX3); and the cast IC-221 alloy. Note that the requirements for strength in weldments (Fig. 18) were reduced to 90% of the strength of base metal.[3] It was assumed that, for butt-welded joints in tubing, the weld crown will provide added strength and the maximum principal stress (hoop stress) will be orientated in a direction parallel to the weld. Also, the Larson-Miller parameter with a constant of 20 did a poor job of correlating creep and rupture data, and an optimized constant would be required before using parametric correlations to set design stresses.

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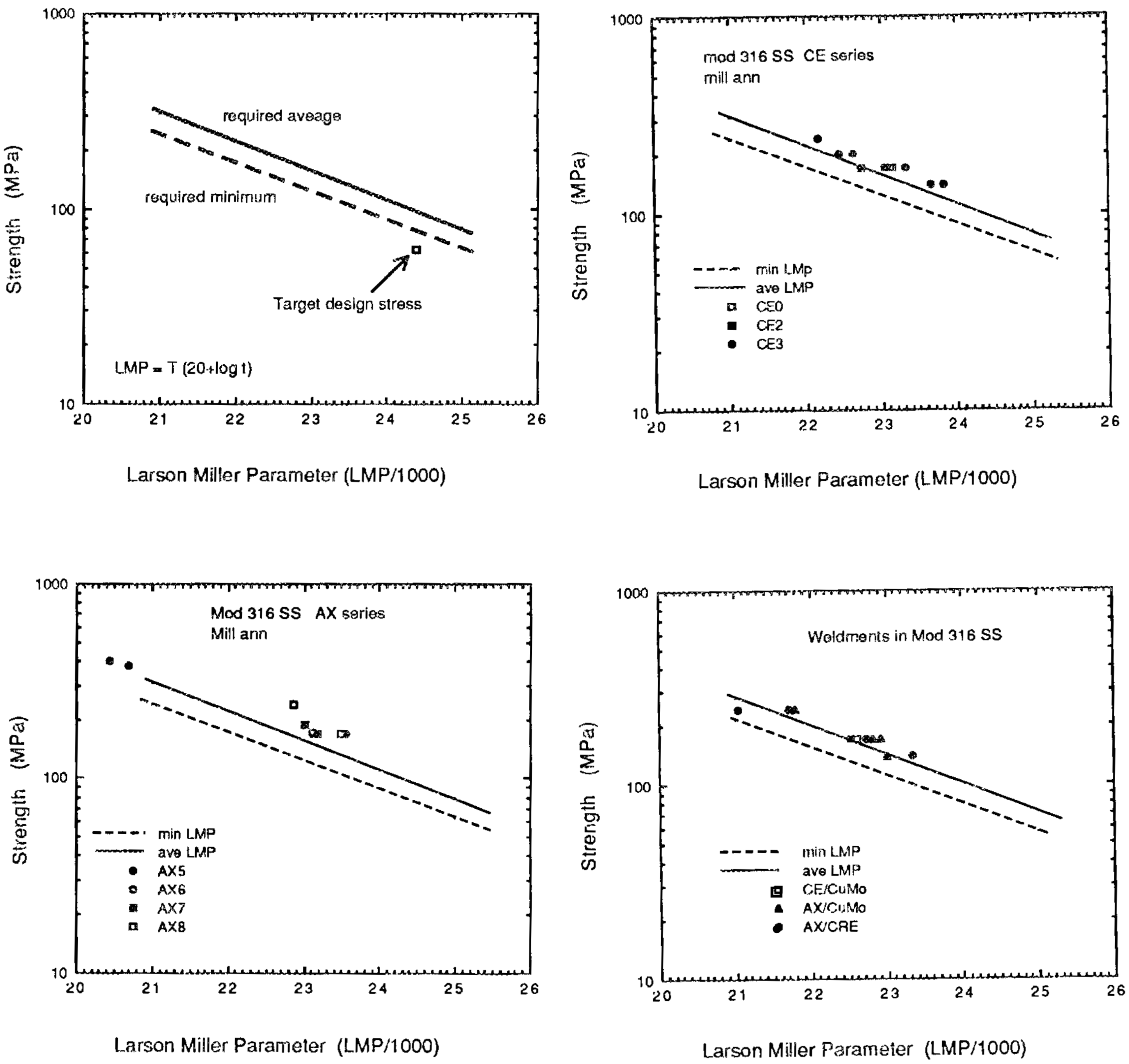

Fig. 18. Stress versus Larson-Miller parameter plots showing the creep rupture strength of modified type 316 stainless steels relative to the alloy design criteria curves.

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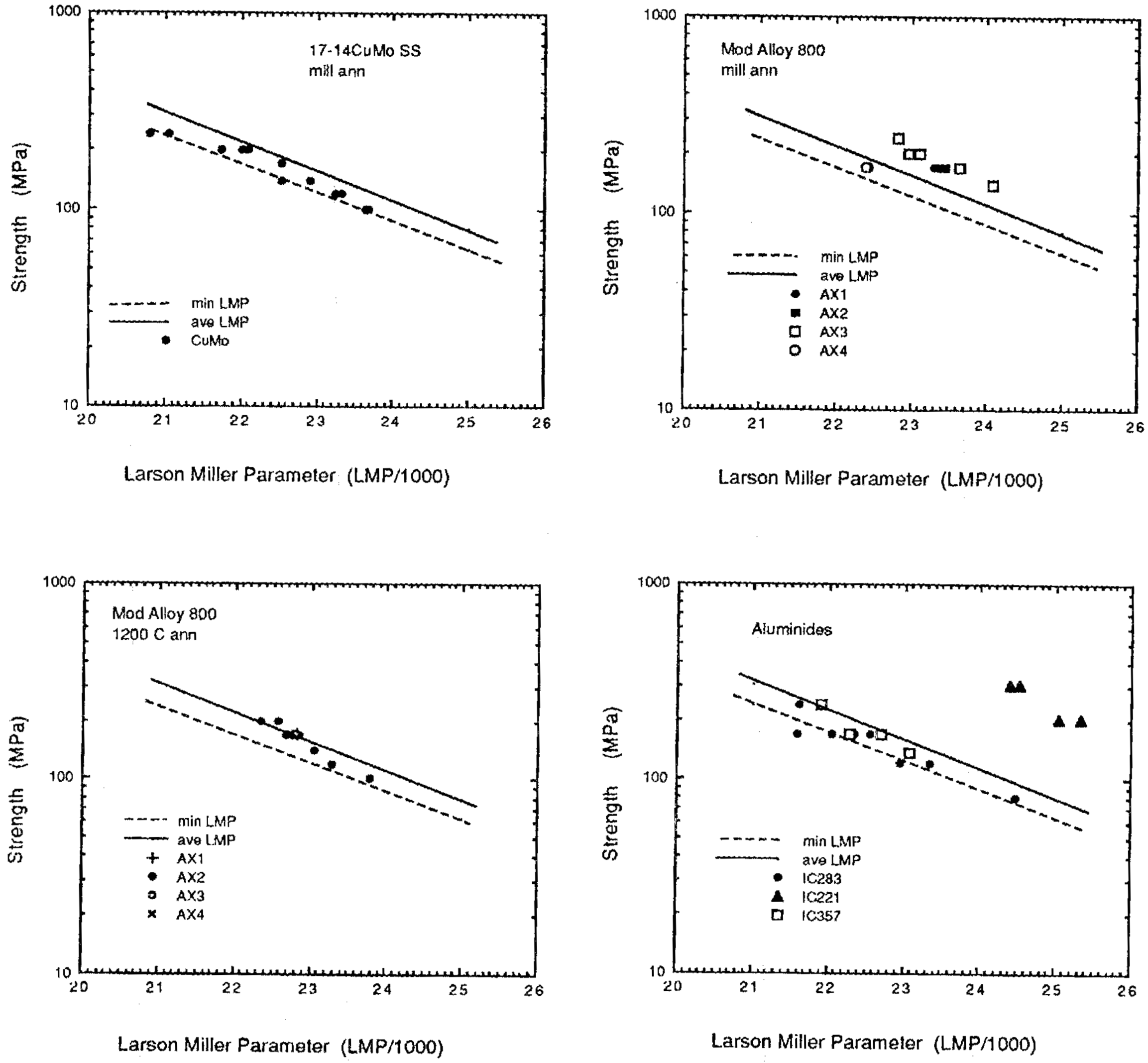

Fig. 19. Stress versus Larson-Miller parameter plots showing the creep rupture strength of several candidate alloys relative to the alloy design criteria curves.

FIRESIDE CORROSION

Four alloy design criteria were identified with respect to fireside corrosion. These addressed the need to clad or surface coat alloys containing less than 25% Cr, the ability to protect tubing with metallic or ceramic shields, the performance of alloys in combustion atmospheres produced by beneficiated coal with low sulfur and alkali content, and the performance of alloys in a limestone-scavenged environment. The screening test data provide very little information regarding the resolution of these issues. The chromizing efforts, described earlier in the report, produced discouraging results and pointed toward the need to clad alloys from all three groups. Definitive test data will be forthcoming from the subcontract at Foster-Wheeler.[2] The problem of cracking in autogenous welds indicated that care would be needed in the design of shield attachments, with the aluminides presenting the greatest problem. The modified 316 alloys would probably be the least corrosion resistant in the beneficiated coal and fluid bed combustion environments. Again, test data will be produced under subcontract.[2]

STEAMSIDE CORROSION

Both the modified alloy 800H and the aluminides are expected to do well in steam. The modified 316 alloys are expected to be marginal. A testing program to evaluate relative performance will be undertaken at some future date.[2]

CONCLUSIONS AND RECOMMENDATIONS

1. Significant improvements in high-temperature strength and ductility were produced in modified type 316 stainless steel by reducing chromium; increasing niobium; increasing nickel; adding combinations of MC-forming elements that include titanium, niobium, and vanadium; adding boron; permitting phosphorus to reach 0.04%; and introducing enough cold work to assure a minimum yield strength of 300 MPa. Tubing with a composition similar to heat AX5 or heat AX6 should be produced for further evaluation.

2. Significant improvements in high-temperature strength and ductility were also produced in modified alloy 800H by reducing aluminum content; adding MC-forming elements that include titanium, niobium, and vanadium; adding molybdenum and sufficient levels of carbon and boron; and introducing sufficient work to assure a minimum yield of 300 MPa. Tubing with a composition similar to heat AX2 or heat AX3 should be produced for further evaluation.

3. The copper-bearing modified 316 stainless steel (heat CE3) was creep tested to beyond 20,000 h and exhibited excellent strength in the mill-annealed condition. A small, lower-phosphorus-containing heat should be produced and assessed for weldability.

4. $(Ni,Cr)_3Al$ fabrication technology has not advanced to a stage where strong, ductile material can be produced as tubing by methods practical to the boiler tubing industry.

5. Although it is possible to join the type 316 stainless steel and alloy 800H modified for high strength and ductility, the standard tools to predict weldability indicate that potential problems exist for both alloys. New filler metals need to be developed, and testing of full-scale weldments in tubing is required.

6. Chromizing of austenitic stainless steels is feasible in terms of reaching desirable chromium levels and penetration depths, but loss in mechanical strength must be expected. Efforts to clad and chromize tubing heats should be continued.

7. Enough fabrication technology exists to produce coupons for laboratory fireside corrosion testing. Materials for testing will include at least one modified type 316 stainless steel (AX6), one modified alloy 800H (AX2), a Ni-Cr-Fe aluminide (IC357), coupons clad with alloy 691, and chromized coupons.

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report was reviewed by J. H. DeVan and R. R. Judkins, and prepared for publication by D. J. Walmsley.

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