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**STAINLESS STEELS WITH IMPROVED STRENGTH
FOR SERVICE AT 760°C AND ABOVE***

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STAINLESS STEELS WITH IMPROVED STRENGTH FOR SERVICE AT 760°C AND ABOVE

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ABSTRACT

An evaluation was undertaken of modified 25Cr-20Ni stainless steels and a modified 20Cr-25Ni-Nb stainless steel for advanced energy applications at 760°C (1400°F) and higher. It was found that good fabricability, strength, and ductility could be produced in the modified steels. Stress rupture data to beyond 10,000 h showed that the strengths of the modified steels were more than double that for type 310H stainless steel.

INTRODUCTION

In the last fifteen years several competing advanced energy technologies have been under development to improve thermal efficiency and reduce emissions resulting from the combustion and conversion of coal (Armor, 1989, Bajura and Webb, 1991, Poe and coworkers, 1991, and Chapman and Johanson, 1991). As these technologies move toward the construction of demonstration plants, the selection of the structural materials becomes of paramount importance. The temperature, pressures, and environments under which the structural materials will operate vary considerably from one concept to another, so one can expect a large range of materials to be utilized (Stringer, 1995). Materials will encompass carbon steels, stainless steels, nickel-based alloys, cobalt-based alloys, intermetallics, ceramics, refractories, and composites. The U.S. Department of Energy (DOE), Office of Fossil Energy Advanced Research and Development (AR&TD) Materials Program is addressing the materials needs for several advanced technology areas and attempting to identify or develop materials that could serve in as many applications as possible (Judkins and coworkers, 1990). Earlier, research was

undertaken by the DOE to assist the Electric Power Research Institute (EPRI) in the investigation of alloys for the advanced steam cycle. Alloy design and evaluation criteria were identified and work was begun to examine four groups of alloys (Swindeman, 1986). These groups included lean stainless steels (containing less than 20% chromium), higher chromium iron-base steels (containing 20% or more chromium), nickel-base alloys, and aluminum-bearing, high-temperature alloys. Over the years, the interest of the DOE in advanced steam cycle has been replaced by increasing interest in combined cycle concepts which include fluidized bed combustors, carbonizers, and gasifiers. The materials performance requirements have changed, and, of the materials included in the DOE-FE AR&TD program on advanced steam cycle materials, the higher chromium-bearing steels and the aluminum-bearing high-temperature alloys take on greater importance. Although most of the effort has been directed toward corrosion resistant materials (Natesan, 1985, Natesan and Podolski, 1991), there are instances where stronger alloys are needed to replace those currently in use. Commercially available higher chromium alloys with improved strength include a modified 310 stainless steel identified as HR3C (Sumitomo Metal Industries, 1987), a modified 20Cr-25Ni-Nb stainless steel identified as NF709 (Nippon Steel, 1996), and a modified 21Cr-11Ni-N stainless steel identified as 253MA. One of these, 253MA, is approved for pressure-boundary service to 900°C (1650°F). The other two stainless steels are judged to have potential for service above 760°C (1400°F). This report summarizes research to evaluate the performance of these higher chromium stainless steels relative to 310H stainless steel and a developmental 25Cr-20Ni-Ta-N stainless steel.

Table 1. Chemical Composition of Stainless Steels

Element	Composition (wt%)					
	310H	253MA	HR3C heat A	HR3C Heat V	NF709	310TaN
C	0.067	0.089	0.06	0.051	0.067	0.051
Mn	1.73	0.55	1.22	1.61	1.03	1.53
Si	0.44	1.73	0.40	0.64	0.41	0.33
P	0.025	0.021	0.014	0.018	0.013	0.01
S	0.001	0.001	0.001	0.001	0.001	0.003
Cr	24.59	20.80	25.65	24.23	20.52	24.9
Ni	19.92	10.99	20.15	18.68	24.88	20.2
Mo	0.36	0.18			1.48	
Ti		0.03			0.06	
Nb			0.47	0.32	0.26	
Ta						1.6
N	0.06	0.15	0.246	0.20	0.16	0.20
B					0.005	
Ce		0.02				

MATERIALS

Commercial alloys that were considered included 310H, 310HCbN (HR3C), 21Cr-11Ni-N (253MA), and 20Cr-25Ni-Nb-N (NF709) stainless steels. For several of the steels, data were available in the literature for temperatures to 815°C and higher. Sources included Prager (1992) for 304H, Sumitomo Metal Industries (1989) for HR3C, Nippon Steel (1996) for NF709, and Kelly (undated) for 253MA. For other steels, supplemental testing was required to extend the database to higher temperatures. Type 310H stainless steel was purchased as a 25-mm (1-in.) diam. bar product meeting ASTM A 479. Type HR3C stainless steel and NF709 stainless steel were obtained as tubing conforming to ASTM A 213. For weldability studies, the tubing was split and rolled to sheet of 1-mm thickness. The 253MA stainless steel was obtained as 1.6-mm (0.063-in.) sheet conforming to ASTM A 240. Compositions for the steels are provided in Table 1. A developmental stainless steel, identified as 310TaN stainless steel, was also examined, and the composition is included in the Table 1. This steel was similar to HR3C stainless steel except that the niobium was replaced by a much higher level of tantalum (1.5%) for additional solid solution hardening. The 310TaN stainless steel was produced as 13-mm plate, 1-mm thick sheet, and 51-mm diam. tubing. All steels with the exception of 310H stainless steel contained more than 0.1% nitrogen. In addition, three of the steels were annealed at 1200°C (2200°F). In the as-received condition, all steels exhibited medium to coarse grain sizes. The commercial alloys were expected to be weldable, but, for comparison purposes, autogenous welds were made in sheets of HR3C, NF709, and 310TaN stainless steels. Butt welds were produced in the 13-mm plate of the 310TaN stainless steel by the gas-tungsten-arc (GTA) process with alloy 556 filler metal and by the shielded-metal-arc (SMA) process using alloy 117 electrodes.

MECHANICAL BEHAVIOR

Mechanical tests were performed on bar and sheet specimens. Depending on the product thickness, tensile and creep specimen diameters were 3.18, 5.71, 6.35, or 12.7 mm (0.125, 0.225, 0.25, or 0.50 in.). The 3.18-mm specimens were button-head ends. All other bar specimens had threaded ends. The sheet specimens were typically 1.02-mm (0.04-in.) thick with reduced section lengths of 32 mm (1.25 in.).

Tensile properties were obtained from literature for the commercial steels (VanEcho, Roach, and Hall, 1967, Sumitomo Metal Industries, 1989, Nippon Steel, 1996, and Kelley, undated). At all temperatures, HR3C, NF709, and 253MA stainless steels exhibited the higher yield strengths than 310H stainless steel. All of the nitrogen-bearing steels exhibited higher ultimate strengths than 310H stainless steel over the entire temperature range of evaluation, as shown in Fig. 1. Tensile ductility of all the stainless steels exceeded 40% over the entire temperature range of evaluation.

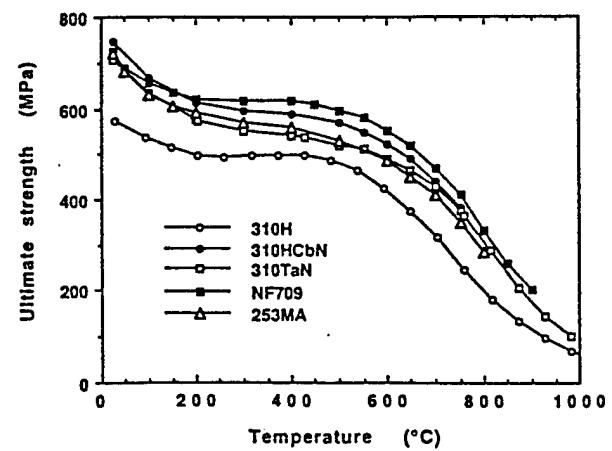


Fig. 1. Ultimate strength versus temperature for five stainless steels.

Creep testing was performed at temperatures in the range of 600 to 927°C (1112 to 1700°F) with emphasis at temperatures above 760°C (1400°F). This testing included a detailed examination of the developmental 310TaN stainless steel and a few exploratory tests on the commercial stainless steels to examine the general features of their creep curves. The first series of tests on the 310TaN stainless steel was aimed at an investigation of fabrication variables on creep strength. Here, the creep data at 871°C (1600°F) and 35 MPa (5.08 ksi) were compared for material after cold rolling, hot rolling, and annealing at 1200°C (2200°F). The creep curves had similar shapes but the annealed (coarse-grained) material possessed the best creep strength. The 1200°C (2200°F) anneal was selected as the reference condition.

The primary creep curves for the 310TaN stainless steel at temperatures ranging from 600 to 815°C (1112 to 1500°F) exhibited hardening in the primary stage, although the primary creep stage ended by 0.5% strain as shown in Fig. 2. Curves at 600 and 650°C (1112 and 1202°F) exhibited sustained periods of secondary creep. In the curves at 700 and 815°C (1292 and 1500°F) the secondary creep stage was very brief and most of the life was spent in the tertiary creep stage.

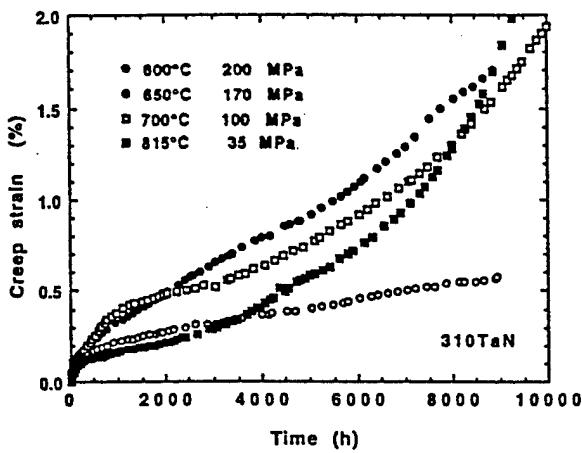


Fig. 2. Typical creep curves for 310TaN stainless steel.

Longer time tests at 815°C and above produced a decrease in the creep rate at high strains. The phenomenon was very apparent in the creep curve in a test at 871°C (1600°F) and 17.5 MPa (2.54 ksi), as may be seen in Fig. 3. Here, the decrease in creep rate occurred at a strain above ten percent. Similar behavior has been observed in other stainless steels tested under similar conditions and has been attributed to the formation of chromium nitride precipitates (VanEcho, Roach, and Hall, 1967, and Swindeman and Marriott, 1994).

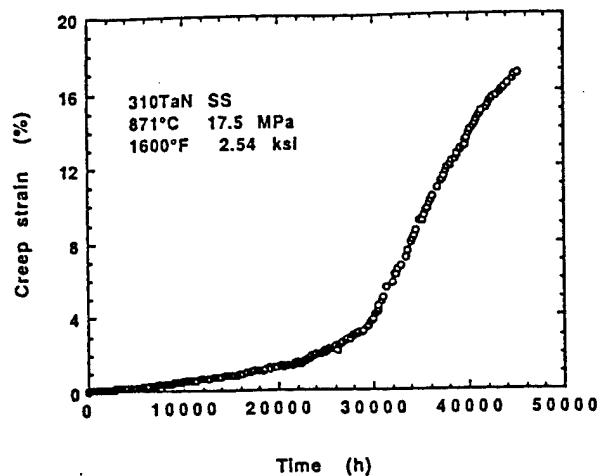


Fig. 3. Long-time creep curve for 310TaN stainless steel at 871°C (1600°F) and 17.5 MPa

Exploratory tests on the HR3C and NF 709 stainless steels showed the same annealing effects as the 310TaN stainless steel, with the high temperature (1200°C) anneal producing the best creep strength. Also, the creep characteristics observed in the 310TaN stainless steel were observed in the other nitrogen-bearing stainless steels. Below 815°C all materials showed hardening during the primary creep stage. With increasing temperature, the primary creep diminished but the extent of secondary and tertiary creep varied with material and testing conditions. In Fig. 4 some comparisons are provided for different materials tested at 927°C (1700°F) and 25 MPa. Here, the two strongest materials, 310TaN and NF 709, spent at least half of their lives in second stage creep.

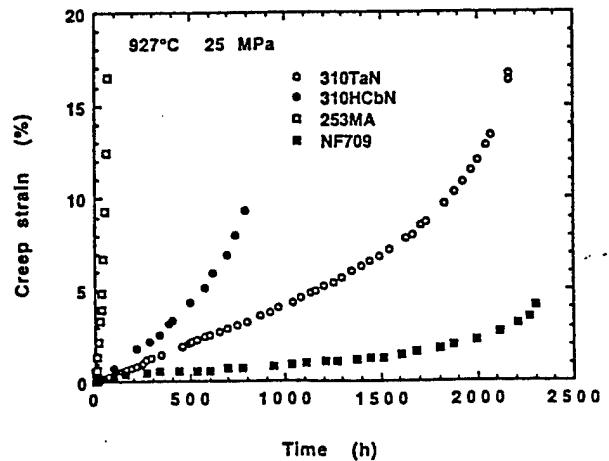


Fig. 4. Comparison of creep curves for five stainless steels at 927°C (1700°F) and 25 MPa

Minimum creep rate (mcr) data were available for several of the stainless steels, and these data were correlated with temperature and stress by means of the Larson Miller parameter (LMP). In some cases it was difficult to define the minimum creep rate since the initial creep rate was negative, similar to the observation of Prager (1992) for 310 stainless steel. Fig. 5 plots the log stress versus LMP for the 310TaN stainless steel using a near-optimum parametric constant of 25. The trend appears linear for data over the temperature range of 600 to 1038°C (1112 to 1900°F). Figure 6 provides the trend curve for HR3C stainless steel published by Sumitomo (1989). The optimum parametric constant found by Sumitomo was 19.61 and, with the exception on one test at 900°C (1650°F), testing was performed at 750°C and below. Data produced in this work over the temperature range of 600 to 982°C (1112 to 1800°F) on two heats of HR3C stainless steel tubing are compared to the trend curve. Most test data fell below of the trend curve, indicating that the test materials possessed about 80% of the expected average strength.

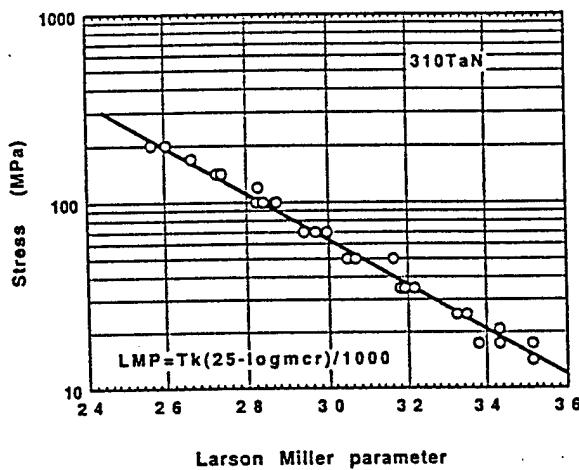


Fig. 5. Stress versus LMP for the minimum creep rate of 310TaN stainless steel

Figure 7 provides the trend for NF709 stainless steel published by Nippon Steel (1996). The optimum parametric constant was found to be 23.34 and the curve had an upward turn with increasing LMP. Data produced in this work generally fell above the curve. Consistent with the observations from comparative creep curves, it was found that the NF709 stainless steel was the strongest of stainless steels in regard to mcr. The creep strengths of the steels are compared in Fig. 8 which provides a plot of log stress versus temperature for a creep rate of 10^{-4} %/h. Here NF709 stainless steel is the strongest followed by 310TaN and HR3C stainless steels.

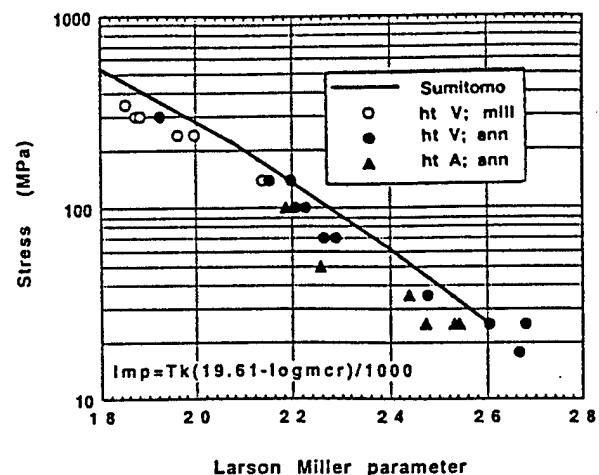


Fig. 6. Stress versus LMP for the minimum creep rate of HR3C stainless steel

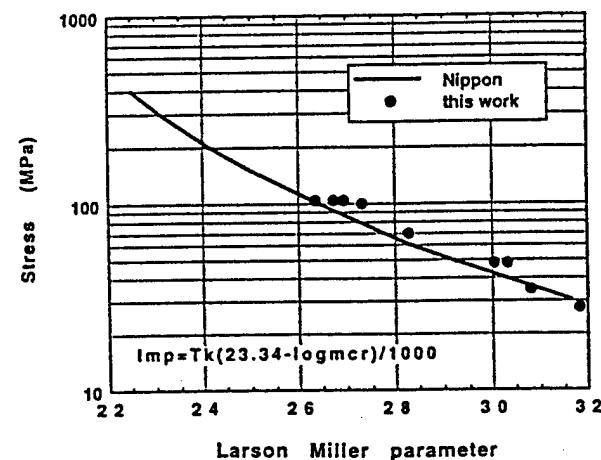


Fig. 7. Stress versus LMP for the minimum creep rate of NF709 stainless steel

Stress-rupture data for the steels were correlated on the basis of the Larson Miller parameter. Figs. 9 through 11 provide log stress versus LMP for three stainless steels. The trend lines are based on the data available in the literature, with the exception of the 310TaN stainless steel, while the symbols indicate data produced in this work. The LMP constant vary from 14 for 310TaN stainless steel to 17.38 for NF709. Exploratory test data produced in this work generally fell within the normal range of variability in stress rupture data which corresponds to a 20% strength variation about the "average" strength.

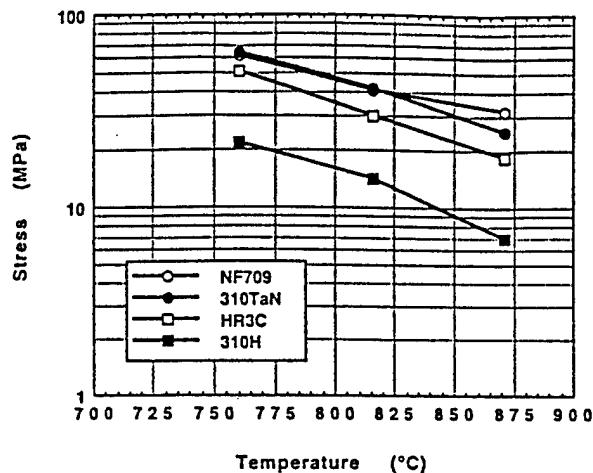


Fig. 8. Comparison of the stress to produce a minimum creep rate of $10^{-4}/\text{h}$ for several stainless steels

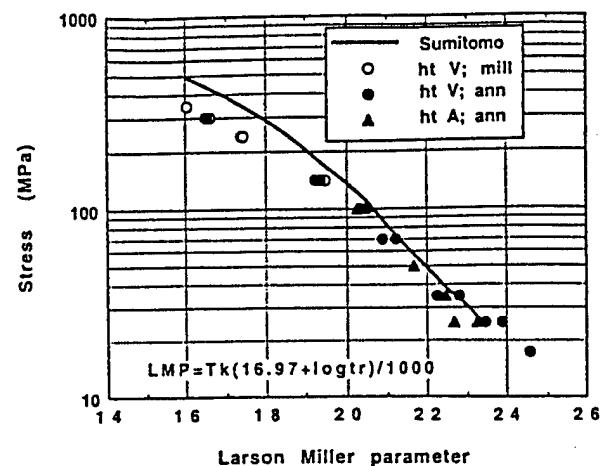


Fig. 10. Stress versus LMP for the rupture life of HR3C stainless steel

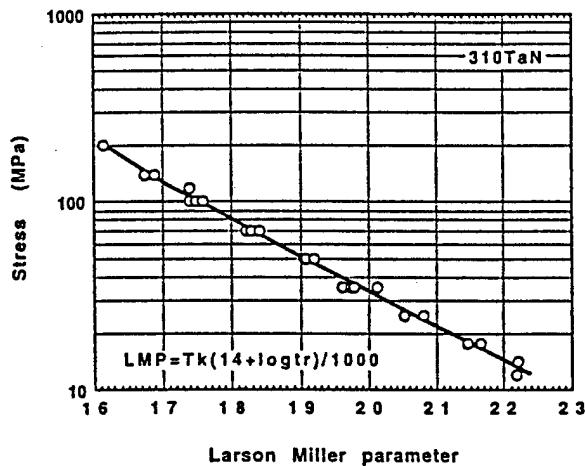


Fig. 9. Stress versus LMP for the rupture life of 310TaN stainless steel

These curves were used to estimate the 10,000 h rupture strength for the different steels, and their comparative strengths are plotted in Fig. 12 for temperatures in the range of 760 to 927°C (1400 to 1700°F). The two strongest steels, 310TaN stainless steel and NF709 possess twice the strength of 310H stainless steel.

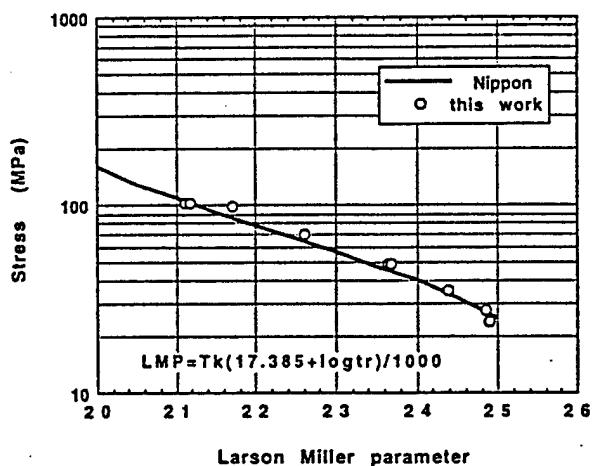


Fig. 11. Rupture stress versus LMP for the rupture life of NF709 stainless steel

The creep ductilities for all of the nitrogen-bearing commercial stainless steels are good. Elongations for HR3C and NF709 stainless steels exceed 10 percent for testing times to 30,000 h and more. The developmental 310TaN stainless steel, however, experienced a minimum creep ductility at most testing temperatures, as shown in Fig. 13. At 700°C (1292°F) creep elongations decreased to 7% at times in excess of 10,000 h. Testing in progress at 650 and 600°C (1202 and 1112°F) give no indications of lower ductilities for longer times.

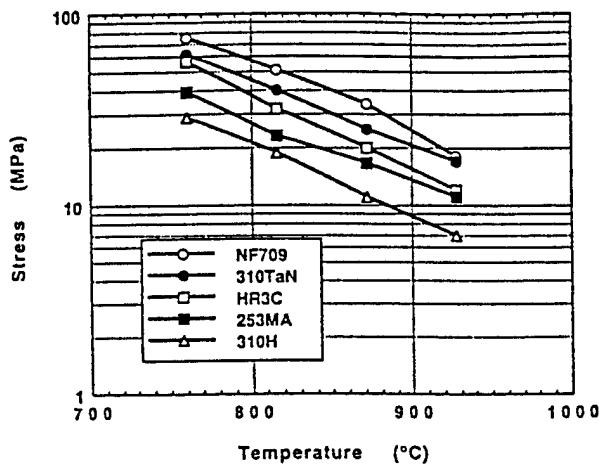


Fig. 12. Comparison of the stress to produce rupture in 10,000 h for several stainless steels

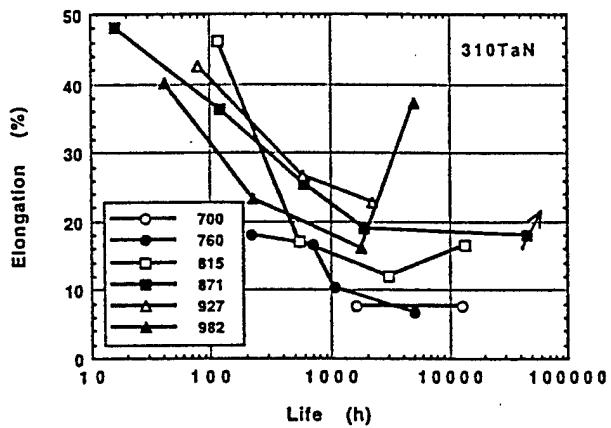


Fig. 13. Stress-rupture elongation versus time for 310TaN stainless steel

All of the stainless steels included in this evaluation lose room-temperature tensile ductility and toughness as a result of prolonged exposure at high temperature. The chemical compositions of HR3C and NF709 stainless steels, however, have been carefully balanced to minimize the quantity and distribution of embrittling phases, and these stainless steels exhibit tensile elongations in excess of 20% after aging. Charpy impact energies for these two stainless steels exceed 50 J after long-time aging. Exploratory aging studies of 310TaN stainless steel indicate that this steel retains 20% or more room temperature ductility after 1000 h aging at temperatures in the range of 760 to 871 °C (1400 to 1600°F).

The nitrogen-bearing steels are readily weldable. The HR3C stainless steel has been joined with alloy 82 filler metal, alloy 625 filler metal, and HR3C containing 0.5% Mo (Sumitomo Metal Industries, 1987). Strength of weldments made with alloy 625 or the "matching filler metal" is comparable to base metal. The NF709 has been joined with alloy 625 filler metal and NF709 stainless steel filler metal (Nippon Steel, 1996). Here too, weldments have strengths comparable to base metal. In this work, stress-rupture specimens from butt welds in 310TaN stainless steel were tested in the cross-weld orientation. Data are compared to the base metal on the basis of the Larson Miller parameter and results are shown in Fig. 14. It may be seen that the weldments have equivalent or better strength than the base metal.

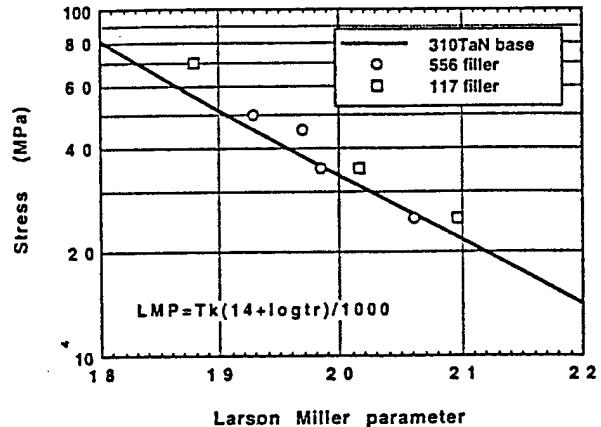


Fig. 14. Stress versus LMP for cross welds in 310TaN stainless steel

DISCUSSION

It is fairly clear that the addition of nitrogen and MC-forming elements along with high-temperature annealing can produce stainless steels with much improved creep strength at 760°C (1400°F) and higher. Over twice the strength of 310H stainless steel has been demonstrated in these steels. The metallurgy and strengthening mechanisms of the commercial nitrogen-bearing, high-chromium stainless steels is well-understood. Bungart and coworkers (1982) examined the solubility of nitrogen in steels whose compositions included the 25Cr-20Ni and 20Cr-25Ni stainless steels, and their work helps to define the nitrogen solubility limits as a function of temperature and chromium content. The subsequent precipitation of carbonitrides during service has been investigated in the HR3C stainless steel by Yoshikawa and coworkers (1988) and in the NF709 stainless steel by Nippon Steel (1996). Yoshikawa and coworkers (1988) examined the stability of HR3C stainless steel at temperatures in the range of

600 to 800°C for times to 10,000 h. They observed both $M_{23}C_6$ and CrNbN precipitation. Precipitates increased with increasing time and temperature, but most of the nitrogen remained in solid solution. They observed that sigma formed when the steel contained less than 20% nickel and less than 0.2% nitrogen. They observed the precipitation of Cr_2N phase when the steel contained more than 22% nickel and 0.25% nitrogen. The appearance of either phase produced lower toughness. Hence, the composition range of HR3C was selected to minimize the quantity of sigma and Cr_2N phases. In the NF709 stainless steel, a Cr_3NiSiC precipitate was observed in addition to the CrNbN and $M_{23}C_6$ precipitates, however, only the CrNbN precipitate persisted for long times above 700°C (1292°F) and was judged to contribute most to the creep strengthening. Details of the precipitation mechanisms in the 310TaN stainless steel are unknown. The presence of sigma phase is expected in the steel, and the general conditions in which sigma forms and its effect on ductility should be similar to 310H stainless steel examined by Menard (1952), Smith and Dulis (1952), Hoke, Eberle, and Wylie (1957), and Barcik (1983). Fine MCN precipitates have been observed in the 310TaN stainless steel to at least 816°C (1500°F), but whether or not these precipitates will lead to long-time strengthening in this high stacking-fault-energy steel (Rhodes and Thompson, 1977) is unknown. Also, a Ni_3Ta intermetallic phase (Weiner and Irani, 1966) was observed in the 310TaN stainless steel at temperatures below 760°C, so strengthening and embrittlement by this precipitate may be expected at lower temperatures. The parabolic hardening observed in primary creep curves at temperatures to 816°C is consistent with the formation of precipitation-stabilized dislocation networks which form early in life. The relatively short secondary creep stage, however, is indicative of an instability and could be due to precipitate coarsening or the loss of solid solution hardening due to the formation of a new but ineffective precipitate such as sigma. Likewise, the tertiary character of creep curve so often observed at temperatures above 760°C could be due to the loss of solid solution hardening elements to coarse precipitates. On the other hand, the turn down in some creep curves at long times and high strains may be related to the internal nitridation of the steels, as observed by VanEcho, Roach, and Hall (1967).

Because of their relatively poor corrosion resistance in air and carburizing environments, these high-strength stainless steels may not be suitable replacements for stainless steels containing silicon, aluminum, or rare earth additions in some applications above 760°C (1400°F), but could replace 304H, 316H, 310H, and 309H stainless steels for limited service life.

SUMMARY

Several austenitic stainless steels developed for advanced steam cycle applications have potential for use above 760°C (1400°F). The steels have approximately twice the strength of 310H stainless steel and have good fabricability, weldability, and resistance to long-time embrittlement. In terms of creep behavior, the strongest is NF709 followed by 310TaN and HR3C stainless steels.

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