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Abstract

The effect of a reduced-temperature austenization treatment on the microstructure and strength of two ferritic-martensitic steels was studied. Prototypic 9% and 12% Cr steels, modified 9Cr-1Mo (ASME T/P91) and Type 422 stainless (12Cr-1Mo-W-V), respectively, were austenized at the standard 1050°C and an off-normal 925°C, both followed by tempering at 760°C. The reduced austenization temperature was intended to simulate potential inadequate austenization during field construction of large structures. The microstructure, tensile behavior, and creep strength were characterized for both steels treated at each condition. While little change in microstructure was observed for the modified 9Cr-1Mo steel, the creep strength was reduced at higher temperatures and in long duration tests. The microstructure of the Type 422 stainless in the off-normal condition consisted of polygonized ferrite instead of tempered martensite. In this case the creep strength was reduced for short duration tests (less than ~1000 hr), but not for long duration tests. Slight reductions in tensile strength were observed at room temperature and elevated temperatures of 450, 550, and 650°C.

Introduction

Ferritic-martensitic (F-M) steels with Cr contents ranging from 9 to 12 wt.% (all subsequent compositions are given in wt.%) are widely used in fossil-fueled steam generation plants as tubing and pipework. These heat-resistant steels typically contain 0.1 to 0.2% C and additions of refractory metals such as Mo, V, and W. The steels are used in a normalized-and-tempered condition with an initial microstructure of tempered martensite, $M_{23}C_6$ carbides along prior austenite and subgrain (lath) boundaries, and in some cases fine MX precipitates within the laths. Ferritic-martensitic steels have been extensively developed and studied over the past thirty years due to the need for increasingly advanced steam conditions, i.e. higher temperature and pressure, and find common use in super- and ultrasupercritical plants with steam conditions approaching 630°C and 30 MPa (1,2).

Ferritic-martensitic steels are also being considered for application in various Generation IV advanced nuclear power plant designs as pressure vessels, tubing and piping, and fuel cladding (3,4). They are a particularly attractive substitute for austenitic stainless steels due to their reduced cost, lower thermal expansion coefficient, and better radiation stability.

Although studies on the microstructure, strength, and thermal stability of F-M steels have been widely published in the literature, most report the results of tests performed on laboratory specimens with well-controlled heat treatments. Less documented are the effects of off-normal heat treatments on the properties of these steels, even though many service failures have been attributed to improper heat treatment. Specific examples include the premature rupture of 12Cr superheater tubing due to inadequate austenization treatment, reported by Barraclough and Gooch (5), and the replacement of several pipe bend segments due to improper cooling after welding and prior to post-weld heat treatment (PWHT) in a German plant, as reported by Hald (6). Of particular concern is the fact that these improperly treated steels typically meet the standard certification requirements, e.g., chemistry, hardness, and room-temperature tensile strength.

This paper reports the results of initial work examining the effect of improper heat treatment on two F-M steels, a modified 9Cr-1Mo steel (ASME T/P91) and a 12Cr-1Mo-W-V steel (Type 422 stainless steel). Modified 9Cr-1Mo, a "second generation" F-M steel developed in the 1970's (7,8), is widely used for tubing, headers, and piping in current supercritical fossil plants and is a leading candidate alloy for application in several Generation IV nuclear plants. Type 422 stainless steel finds application as steam turbine blading and high-temperature bolting (9,10). The effect of inadequate austenization (such as might occur in the center of a thick section) on the microstructure, hardness, and elevated-temperature tensile and creep strength of the two steels was determined. Specimens were austenized at the standard temperature of 1050°C and an off-normal temperature of 925°C, both followed by the standard 760°C tempering treatment.

Experimental Procedures

Materials and Heat Treatment

Modified 9Cr-1Mo steel (ASTM A182, F91) was obtained commercially in the form of a hot-rolled round bar 50 mm in diameter in the normalized and tempered condition. Type 422 stainless steel was obtained commercially in the form of a forged and "annealed" plate 50 mm in thickness (the microstructure in the as-received condition was tempered martensite). The measured chemical compositions of both steels are listed in Table I. Both steels were sectioned into billets suitable for heat treatment, and

Table 1: Chemical Composition (wt.%)

Alloy	Fe	C	Cr	Mo	V	W	Ni	Nb	N	P	S
Mod. 9Cr-1Mo	Bal.	0.09	8.11	0.98	0.21	--	0.17	0.08	0.049	0.014	0.003
Type 422	Bal.	0.22	11.76	0.96	0.24	1.02	0.85	0.012	0.036	0.017	0.001

were heat treated with either a standard procedure consisting of austenization at 1050°C for a half-hour, air cooling to room temperature, and tempering at 760°C for two hours, or an "off-normal" procedure in which the austenization temperature was 925°C instead of 1050°C, with the remainder the same.

The effect of heat treatment on microstructure was assessed using light microscopy (LM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) techniques. Mounted and polished metallographic cross-sections were prepared using standard techniques and immersion etched with either Vilella's reagent or a solution of 30 ml ethanol, 3 g picric acid, 0.6 ml nitric acid, and 1 ml hydrochloric acid. A light etch with the latter was found to be effective in revealing carbide precipitates in SEM examination. Precipitate size distributions were determined by analysis of SEM images of etched sections taken at high magnification (20,000 to 40,000 X). TEM foils were prepared from 3 mm discs which were mechanically thinned to 100 µm and electropolished to perforation. TEM examination was performed on a Phillips EM420 STEM operating at 120 kV.

Mechanical Testing

Tensile tests were performed at room and elevated temperatures of 25, 450, 550, and 650°C on cylindrical specimens with 4.1 mm gauge diameter and 17.5 mm reduced section. Specimen extension in room-temperature tests was measured using a standard clip gauge. An averaging, rod-in-tube type extensometer was used for tests at elevated temperatures; the translated displacement was measured outside the furnace using two clip gauges. The specimens were heated with a radiant furnace and allowed to equilibrate for 15-30 minutes prior to testing; specimen temperature was controlled with a type K thermocouple to within 1°C. The tensile tests were performed at a constant cross-head displacement corresponding to an initial strain rate of 1×10^{-3} sec^{-1}.

Constant-load creep-rupture tests were performed at 800, 900, and 1000°C on lever arm creep machines with resistively heated furnaces. Specimens were again cylindrical with 6.3 mm gage diameter and 40 mm reduced section. Specimen temperatures were controlled to within ±5°C by monitoring with two Type K thermocouples wired to the specimen gauge length. Averaging, rod-in-tube type extensometers were used; the displacement was measured outside the furnace by two linear variable differential transducers (LVDTs). The temperature and LVDT readings were periodically recorded using a computer-based data acquisition system. Most creep tests were run to failure.

Selected specimens were examined after testing. Longitudinal cross-sections of the reduced sections were prepared, etched, and examined using SEM. General microstructural changes were noted.

Results

Microstructure

Typical optical microstructures for the Modified 9Cr-1Mo and Type 422 steels in both heat treatment conditions are shown in Figure 1. All show typical tempered martensite structures at this magnification. While the lath structure in the Type 422 stainless steel is visibly more degraded in the off-normal condition with a slight apparent coarsening in the carbides, little difference can be distinguished between heat treatment conditions in the modified 9Cr-1Mo steel except a slight reduction in prior austenite grain size. The large difference in prior austenite grain size between the two steels is also apparent

No clear difference in the standard and off-normal microstructures was discernable for the modified 9Cr-1Mo steel in the TEM. Relatively coarse carbides were observed on the prior austenite and lath boundaries, along with a high dislocation density. A similar tempered martensite structure was observed for the Type 422 stainless steel in the standard condition, although the carbide density was noticeably higher than in the modified 9Cr-1Mo, commensurate with the higher C content of Type 422. Indications of recovery and ferrite polygonization were observed in the Type 422 off-normal condition.

Differences in the microstructures of the two heat treatment conditions were quantified in terms of precipitate size measured using image analysis of high-magnification SEM micrographs. Figure 2 shows typical images for the two steels; Table 2 lists precipitate dimensions, ASTM grain size, and hardness. No difference in mean precipitate diameter was observed for the modified 9Cr-1Mo steel, but precipitates in the Type 422 stainless steel were significantly coarser in the off-normal condition. The precipitate size distribution for the Type 422 stainless in the off-normal condition was slightly bimodal, with a second peak developing at 480 nm due to precipitate impingement and agglomeration. The Rockwell C hardness levels for the two steels were consistent with the observed microstructural differences (or lack thereof): no significant change was observed for the modified 9Cr-1Mo steel, and the hardness of the Type 422 stainless steel was considerably lower in the off-normal condition.

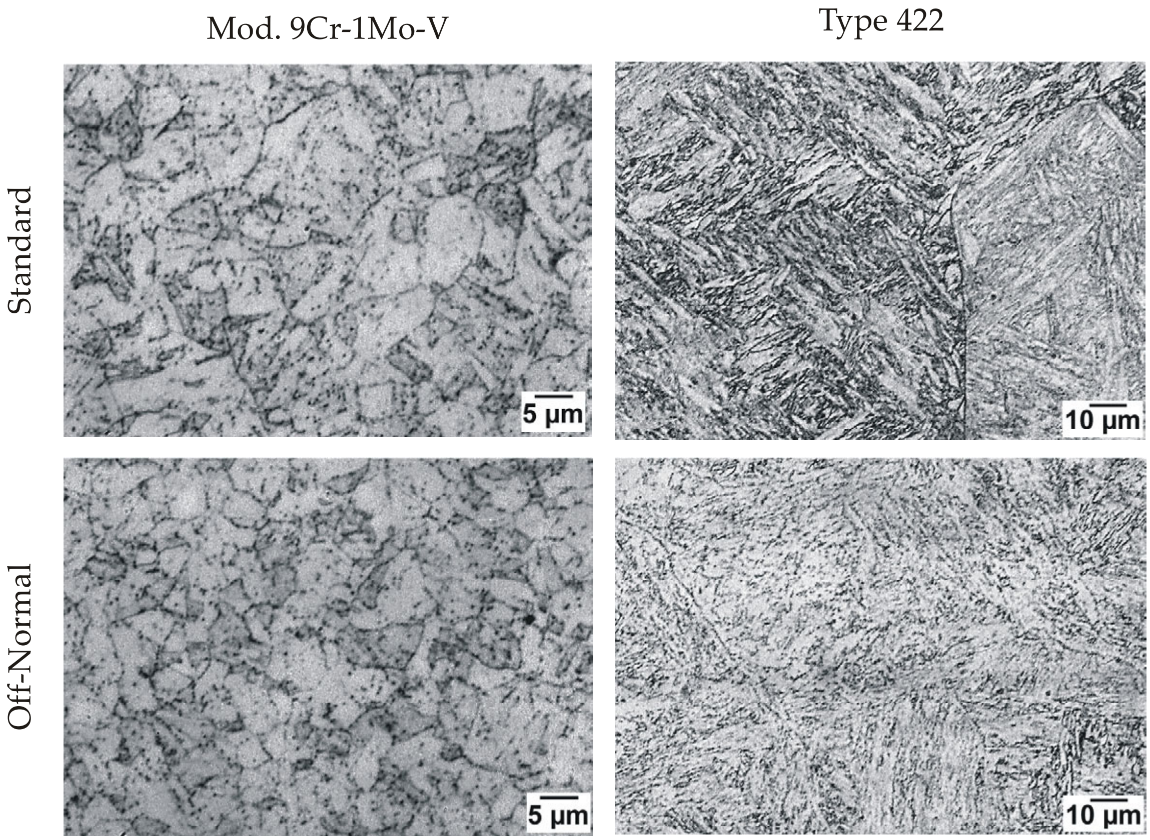


Figure 1: Optical Microstructure

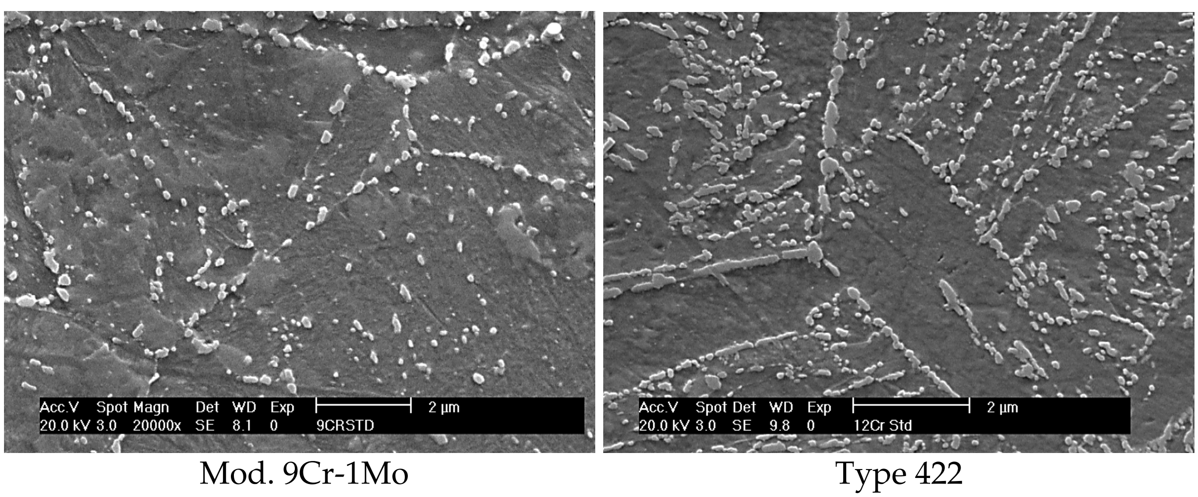


Figure 2: Typical SEM Micrographs Used for Precipitate Size Analysis

Table 2: Heat Treatment Conditions and Microstructural Characteristics

	Mod. 9Cr-1Mo Standard	**Mod. 9Cr-1Mo Off-Normal**	**Type 422 Standard**	**Type 422 Off-Normal**
Normalization	1050°C/0.5 h/AC	925°C/0.5 h/AC	1050°C/0.5 h/AC	925°C/0.5 h/AC
Tempering	760°C/1 h/AC	760°C/1 h/AC	760°C/1 h/AC	760°C/1 h/AC
Prior Austenite Grain Size	11.5	12.5	5	5
Hardness (R_c)	19.1	17.9	27.6	19.9
Mean Carbide Diameter ± 95% CI (nm)	124 ± 8	136 ± 8	134 ± 6	185 ± 12
Mean Carbide Diameter Std. Deviation (nm)	75	69	63	117

Elevated Temperature Strength

Figure 3 shows the elevated temperature tensile strengths of the modified 9Cr-1Mo and Type 422 steels. Small decreases in both yield and ultimate tensile strength (UTS) were observed for each steel; the reduction in strength was greater for Type 422 steel, consistent with microstructure and hardness. The magnitude of reduction (roughly 50 MPa for modified 9Cr-1Mo and 100 MPa for Type 422) was relatively constant with increasing temperature to 650°C, at which temperature the strengths of the two conditions were nearly equal. Commensurate increases in ductility were observed, with the difference again smaller at the highest temperatures.

Figure 4 shows minimum creep rate and stress-rupture data for modified 9Cr-1Mo steel in standard and off-normal conditions. Shown for comparison is early modified 9Cr-1Mo data obtained at Oak Ridge National Laboratory (ORNL) (8). Data for the standard heat treatment shows good agreement with the ORNL data, as expected. The stress exponents, n, in the Norton creep equation ($\dot{\varepsilon}_{\min} \propto \sigma^n$) are relatively high and decrease with temperature, ranging from 16 at 550°C to 8.5 at 650°C. The off-normal data increasingly deviate from the standard data with increasing temperature: the two datasets lie together at 550°C, and the off-normal condition is roughly 30% weaker at

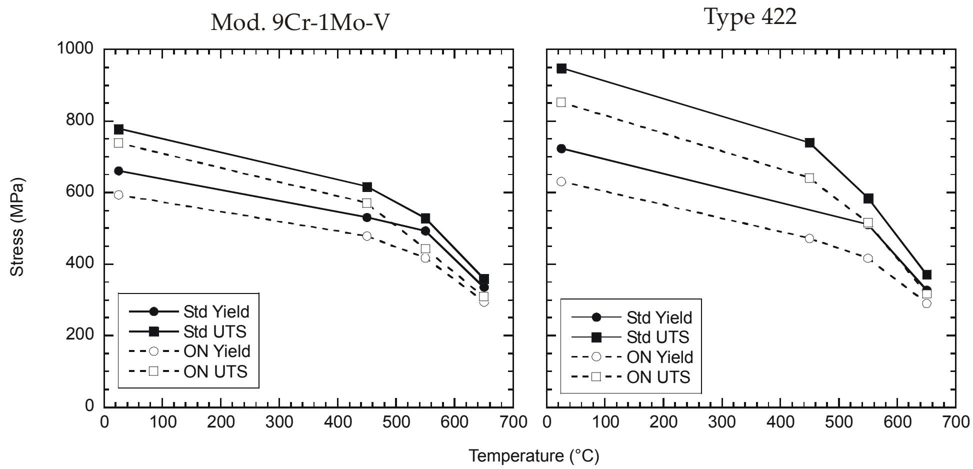


Figure 3: Elevated Temperature Tensile Strength

650°C. Data for the off-normal condition at 600°C fall slightly below the standard data for lower stresses/longer lives. The trends in minimum creep rate and rupture life are consistent.

Creep and stress-rupture data for Type 422 stainless steel in standard and off-normal heat treatment conditions are shown in Figure 5. Baseline data published by the Japanese National Research Institute for Metals (NRIM) (9) are shown for comparison. The NRIM data trends are extrapolated (gray lines) to better delineate the trends for the present standard heat treatment data, which generally agree with the NRIM data. At each temperature, creep rates and rupture lives for the off-normal condition agree with those of the standard condition at the lowest stresses but are markedly poorer at higher stresses. The magnitude of high-stress strength reduction increases with test temperature. Although the data are limited, the creep rate or rupture life at which the off-normal data begins to deviate from the standard data appears to change with temperature. The minimum creep rate at which deviation occurs increases from approximately 10^{-9} sec^{-1} at 550°C to 10^{-8} sec^{-1} at 650°C. Similarly, the rupture life at which deviation occurs varies from 10,000 hr at 550°C to 1,000 hr at 650°C.

Although individual data are not presented, the creep elongation to failure and reduction in area observed in both heat treatment conditions were typical for each alloy. Ductility in the off-normal heat treatment condition was slightly higher than in the standard condition; all failures occurred by ductile rupture. Little change in optical microstructure was observed for either steel after creep-rupture testing at 550 and 600°C, but a degradation of the tempered martensite structure was observed for the modified 9Cr-1Mo steel after creep testing at 650°C for ~ 500 hr, as shown in Figure 6. Spheriodization and recovery appeared to be slightly more advanced in the off-normal condition.

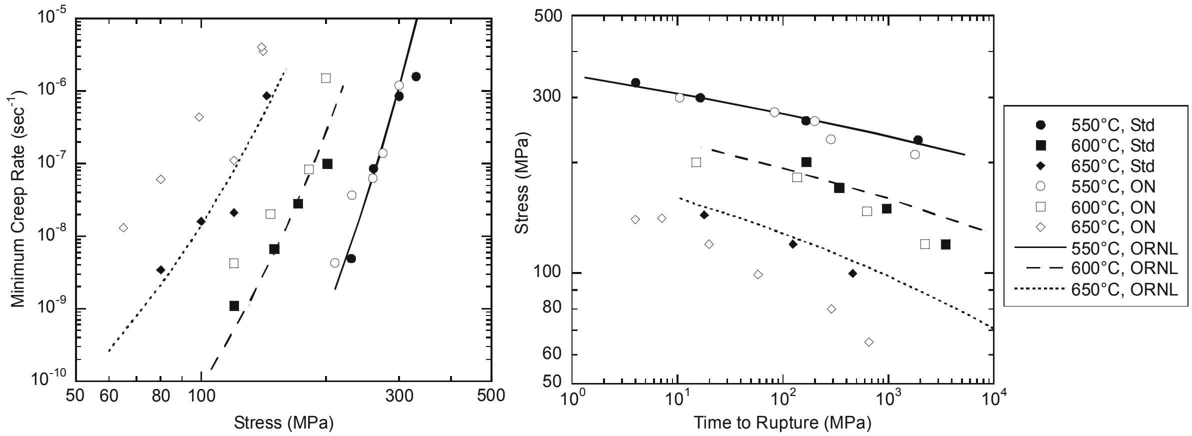


Figure 4: Minimum Creep Rate and Stress Rupture Life for Modified 9Cr-1Mo Steel

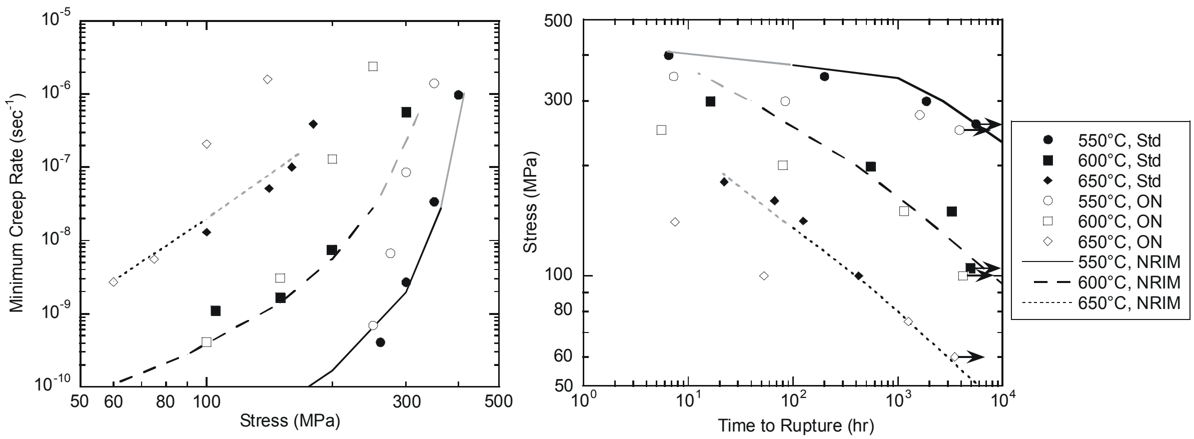
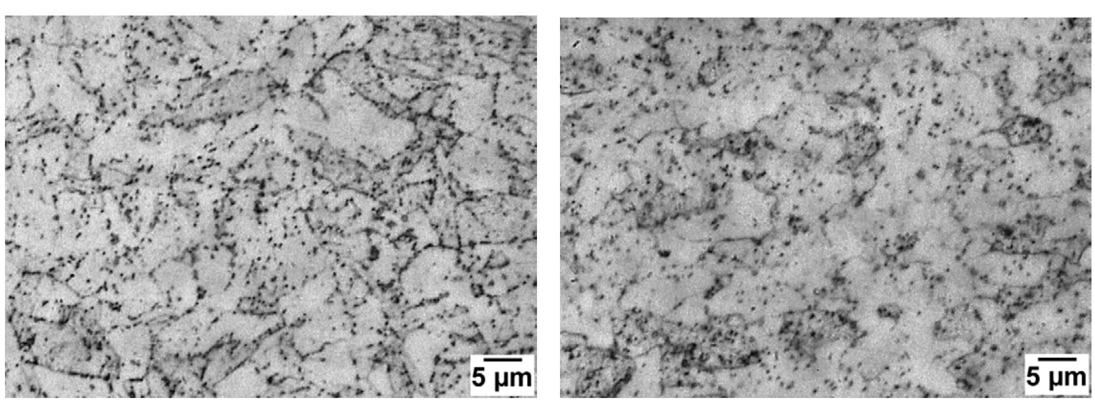


Figure 5: Minimum Creep Rate and Stress Rupture Life for Type 422 Stainless Steel



Standard: 462 hr at 100 MPa　　Off-Normal: 654 hr at 65 MPa

Figure 6: Microstructures of Modified 9Cr-1Mo Steel After Creep at 650°C

Discussion

Modified 9Cr-1Mo Steel

Austenization of modified 9Cr-1Mo steel at 925°C instead of the standard 1050°C resulted in little microstructural change. Off-normal and standard structures observed using LM, SEM, and TEM were nearly identical, and no significant difference was observed in the carbide size distribution. The only quantifiable difference was a small but noticeable reduction in the prior austenite grain size (from ASTM ~11.5 to ~12.5) for the off-normal condition.

The lack of microstructural difference is consistent with the fact that both temperatures lie in the austenite phase field for this alloy (the A_{c3} temperature is reported as 905°C (11)). The only difference expected is less carbide dissolution during the dwell at the lower, off-normal temperature. However, because carbides in the quenched-and-tempered initial condition are fine, the extent of dissolution at the off-normal temperature appears to be not markedly different than that at the standard temperature, and the two final microstructures are nearly indistinguishable. As expected, austenite grain growth was restricted at the lower temperature, reflected in the smaller prior austenite grain size. The small reductions in hardness and tensile strength in the off-normal condition probably result from a slightly reduced extent of carbide dissolution for the lower-temperature austenization, which will give softer martensite and reduced dislocation density after tempering (11). The changes are sufficient to overwhelm the increase in hardness and tensile strength resulting from the decreased prior austenite grain size.

A significant reduction in creep strength at 650°C was observed for the off-normal condition despite the microstructural similarity. At 650°C the strength reduction was clear at all stress levels tested and was apparent at lower stresses (< 150 MPa) at 600°C. These strength reductions are also tentatively attributed to slightly reduced carbide dissolution during austenization at the lower, off-normal temperature. Creep strength in F-M steels at high temperatures is controlled by the subgrain size and dislocation density, which are in turn controlled by carbide precipitates—Nb- and V-containing MX precipitates in particular for modified 9Cr-1Mo (12,13). Although TEM examination of post-crept microstructures is needed, it is plausible that slightly less carbide dissolution at 925°C may ultimately result in fewer fine MX precipitates present to restrict recovery and subgrain growth during creep, thereby resulting in reduced strength relative to the standard condition. Such an effect would be expected to be particularly pronounced for higher temperature or longer duration tests where microstructural changes during creep are significant. The post-test optical microstructures support this hypothesis—after ~500 hr of creep at 650°C the tempered martensite structure appears more degraded in the off-normal condition than in the standard condition (Figure 6).

The observation of reduced creep strength in modified 9Cr-1Mo steel with lowered austenization temperature is consistent with previous findings. Sikka, et al. (8) noted marked reduction in rupture life at 650°C with decreasing austenization temperature, with nearly an order of magnitude difference in life at 90 and 100 MPa stress levels; a similar life reduction was observed in the present study. Other researchers have also observed deleterious effects of exposure to temperatures in the 900-950°C range (11,14,15); such temperatures correspond to those experienced in the fine-grained HAZ of weldments, which are preferential failure locations (Type IV cracking) in creep conditions (16).

Type 422 Stainless Steel

The differences in the 12% Cr Type 422 stainless steel microstructures after austenization at standard and off-normal temperatures were more marked than for the modified 9Cr-1Mo steel. The structure following standard austenization at 1050°C and tempering was tempered martensite with high dislocation density, while polygonized ferrite with low dislocation density was observed after austenization at 925°C and tempering; significant carbide coarsening was also observed.

The microstructural information indicates that austenite did not form at 925°C, even though (as with the modified 9Cr-1Mo steel) this temperature was within the austenite phase field (the A_{c3} temperature is reported as 885°C(10)). The prior austenite grain size in the off-normal condition was essentially the same as the as-received material and after standard heat treatment (the ASTM grain size in all conditions was 4 to 5), contrary to an expected reduction in prior austenite size for the lower austenization temperature. The presence of polygonized and recovered ferrite rather than tempered martensite indicates that martensite was not formed during cooling from the 925°C austenization temperature (and hence austenite was not present prior to cooling). The structure resembled that seen in the intercritically-heated HAZ of weldments (17-19). It is unclear why austenite was not formed at 925°C; either the material did not actually achieve this temperature or the dwell time was not sufficient. The higher alloy and carbon content will result in slower transformation kinetics relative to the 9% Cr steel, perhaps sufficiently slower that austenite was not formed during the half-hour austenization period for Type 422 steel, but was for modified 9Cr-1Mo.

The reduced tensile strength and hardness clearly reflect the microstructural changes. In contrast to the modified 9Cr-1Mo steel, however, the creep strength of Type 422 stainless in the off-normal condition was reduced at all temperatures, but only for high stress, short duration tests. The creep strength at low stresses in the off-normal condition was equivalent to the standard condition. This behavior can be understood in terms of creep degradation of the standard microstructure. For Type 422 stainless the initial microstructure in the off-normal condition (recovered ferrite) is similar to that found after long-term creep testing of the standard microstructure (20,21). In a long-duration creep test the standard microstructure evolves into a microstructure similar to the off-normal microstructure at a relatively small fraction of the test duration. The microstructure controlling the behavior of the majority of a long-term test is therefore similar for standard and off-normal conditions, whereas in shorter tests the initial microstructure dominates, and the standard microstructure is stronger. The rupture time at which the lives of the off-normal and standard conditions become equivalent is shorter at higher temperatures, consistent with thermally-activated recovery of the standard microstructure as the cause of equivalent behavior. Note that in the case of Type 422 stainless MX precipitates do not significantly enhance creep strength by impeding recovery (the alloy is "first generation", not containing Nb), so it occurs more quickly and completely than in modified 9Cr-1Mo.

A similar study of the effect of off-normal austenization treatment was performed on X20CrMoV121 steel by Barraclough and Gooch (5), in response to service failure of this steel at very short life. X20CrMoV121 has a very similar composition to Type 422, but for this alloy the effect of lower-temperature austenization (at 900°C instead of 925°C) was very marked—no sign of a lath microstructure was observed, only equiaxed ferrite. The reduction in strength at 640°C was approximately 20 MPa; nearly 50% of the strength at that temperature. The strength reduction at 650°C in the present study was approximately 30%, but only for test durations shorter than 100 hr. It is unclear whether the considerably greater degradation observed by Barraclough and Gooch resulted from the small alloy composition difference or the small difference in austenization temperature (25°C).

These findings indicate an apparent increased sensitivity of the more highly alloyed 12% Cr steel to variations in heat treatment in comparison with the 9% Cr alloy, at least on a microstructural level. The greater sensitivity to heat treatment variations of 12% Cr steels has been noted by other researchers, particularly for welding processes (16,22,23). It is important to note, however, that for typical service conditions (e.g. lower stress, long duration) the modified 9Cr-1Mo steel is in fact more sensitive to the reduced temperatures than Type 422 stainless. The creep strength reduction for modified 9Cr-1Mo only exists at higher temperature and longer times, while the creep strength of Type 422 is not reduced for these conditions. The creep strength advantage of later-generation F-M steels (such as modified 9Cr-1Mo) over earlier alloys primarily results from increased microstructural stability (resistance to recovery and subgrain coarsening). The stability results from a fine, intra-lath dispersion of MX precipitates. Off-normal conditions which impair the formation of these precipitates have the potential to seriously impair the creep strength of the advanced alloys.

Conclusions

The effect of a lowered austenization temperature, 925°C instead of the standard 1050°C, on the microstructure and elevated temperature strength of modified 9Cr-1Mo steel and Type 422 stainless steel (12Cr-1Mo-W-V) was investigated. Although there was little discernable change in the microstructure of the modified 9Cr-1Mo alloy in the off-normal condition, the creep-rupture strength was significantly reduced at 650°C; slight reductions were observed for low stresses at 600 and 550°C. The creep strength reduction is attributed to decreased carbide dissolution at the lower austenization temperature and a commensurate change in the size and distribution of MX precipitates, which control the recovery and coarsening of subgrain structure in this alloy. For Type 422 stainless, off-normal austenization resulted in a structure of polygonized ferrite and coarsened carbides, indicating that transformation to austenite did not occur even though the A_{c3} temperature for this alloy was exceeded. The recovered structure had significantly reduced hardness and short-term elevated temperature tensile and creep strength, but the long-term creep strength was not affected.

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