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AUSTENITIZING AND MICROSTRUCTURE
OF A HT-9 STEEL

Roy C. Wilcox and Bryan A. Chin

Department of Mechanical Engineering
Auburn University
Auburn, Alabama 36849

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Abstract

The effects of austenitizing temperatures and times on the microstructure of a 12Cr-1Mo-V-W steel are reported. Austenitizing temperatures covering the range of 900 to 1225°C and times of 30 minutes to 100 hours were used. Equilibrium microstructures were difficult to obtain because delta ferrite initially present in the steel resists dissolution during austenitizing.

Introduction

The ferritic 12Cr-1Mo-V-W steel known as HT-9 has been selected for investigation as an alternative to austenitic stainless steel for duct applications in liquid metal fast breeder reactors [1]. The HT-9 steel shows resistance to swelling [2] and irradiation creep [3-4] in neutron environments. However, irradiation produces an increase in the ductile-brittle transition temperature [5] which could result in failures during offnormal events. Therefore, an investigation was initiated to study the effects of heat treatment on the structure and properties of HT-9. This paper investigates the effect of austenitizing temperatures and times on the microstructure of a 12Cr-1Mo-V-W steel.

Procedure

All specimens were cut from a 5.1 cm diameter bar manufactured by Carpenter Technology Corporation, Heat no. 84425. The chemical composition is given in Table I. Metallographic samples were either cut from standard Charpy specimens or were 1 cm cubes to approximate the heating and cooling gradients in the Charpy specimens.

Heat treating was conducted in fused quartz capsules. All specimens were ultrasonically cleaned and placed with Ta and Zr foils in the capsules. The foils served as oxygen getting materials. The capsules were evacuated and flushed with high purity Ar prior to final sealing at a pressure of 4 psia.

Specimens were austenitized at temperatures over the range of 900 to 1225°C for times of 30 minutes to 100 hours. The austenitizing treatment was followed by air cooling. Tempering when used was conducted at 700°C for 2 hours followed by air cooling.

Standard metallographic procedures were used for specimen preparation and for grain size and volume amount determinations. However, several etchants were used to reveal different features of the microstructure. A staining etchant [6] composed of 80 ml H₂O, 20 ml HCl, 2 gm ammonium bifluoride and 1 gm potassium metabisulfide was used to reveal delta ferrite and carbides. Prior austenitic grain size was determined using an electrolytic etch consisting of 65 ml H₂O, 65 ml HCl, 12 ml acetic, and 20 ml HNO₃ at .65 volts for 30 to 100 seconds [7]. Electrolytic extraction of carbides were performed. The electrolyte was 10% HCl-methanol using a current of 100-120 milliamperes for 12 hours with platinum electrodes [8]. Carbide particle size and structure were determined using optical microscopy with a HP9836 Digitizer and x-ray powder techniques.

Results

The microstructure of the as-received bar is shown in Figure 1. The structure was tempered martensite with a prior austenite grain size of 0.02 mm. Delta ferrite comprised 2-4% by volume of the structure and was present in the form of stringers elongated in the direction of fabrication. The delta ferrite stringers were not completely distributed uniformly throughout the structure on the microscopic scale. Large carbides were found at the delta ferrite-matrix interface and were evident in both the transverse and longitudinal sections.

Figure 2 gives the phases produced on air cooling as a function of the austenitizing temperature and times used in this investigation. X-ray analysis of extracted carbides from tempered structures revealed only the $M_{23}C_6$ type to be present. Other investigators have found that the $M_{23}C_6$ type carbide is produced by tempering at temperatures greater than 600°C for 12Cr-1Mo steels [9,10].

Prior austenite grain growth was studied as a function of both austenitization temperature and time (Figures 3 and 4). For 30 minute austenitizing times, a constant grain size of 0.02 mm was observed in the temperature region of 900 to 1025°C. Above 1025°C, increasing the austenitizing temperature resulted in a linear increase in grain size. These results are similar to those of previous investigators [11,12]. Fabrication of the original material caused the grains to be elongated. This grain elongation decreased as the austenitizing temperature increased. Equiaxial

grains were observed at temperatures of 1100°C or greater. Below 1025°C, no change in grain size was observed for austenitizing times out to 30 hours (Figure 4). Above 1025°C, grain growth is relatively slow within the temperature region studied.

Figure 5 shows the dependence of delta ferrite on austenitization temperature for a constant time of 30 minutes. Volume fractions determined from examination of ten longitudinal and transverse sections are plotted. Differences in the volume percentage as determined from transverse and longitudinal sections result from the directionality of the delta ferrite caused by processing of the initial ingot. A minimum amount of delta ferrite was achieved at a temperature of 1100°C, the volume fraction being reduced from 4% to less than 1%. Above 1175°C, the amount of delta ferrite again increases which is consistent with passing into the austenite plus ferrite region of the phase diagram for 12% Cr steels [13].

The time of austenitization also effects the volume fraction of delta ferrite as shown in Figure 6. Increasing time results in a decrease in delta ferrite. Even at temperatures as low as 1000°C, the delta ferrite can be reduced below 1% by an austenitization time of 3 hours. Delta ferrite is reduced to nearly an undetectable volume fraction by annealing to a time of 10 hours or greater for temperatures between 1000 and 1125°C. This reduction in delta ferrite is shown in Figure 7 for the austenitizing temperature of 1000°C for times of 30 minutes and 10 hours.

Figure 8 shows the microstructure as a function of austenitization temperature for a constant time of 30 minutes. At 900°C, the matrix is tempered martensite. After cooling from temperatures of 1000 to 1200°C a martensitic matrix is produced. Within the temperature range of 900 to 1200°C, 30 minute heat treatments did not eliminate delta ferrite. The morphology of the delta ferrite does change slightly with austenitizing temperature. As the temperature increases, the ferrite stringers become rounded with more ferrite grain boundaries being evident. Thus, the stringers tend to break into a number of distinct grains. The smaller ferrite stringers tend to spheroidize with time.

The large carbides present at the delta ferrite-matrix interface in the as-received material (Figure 1) are influenced by the austenitization treatment. Metallographic examination of samples austenitized for 30 minutes revealed these carbides were present at all temperatures from 900 to 1225°C. However, the amount of total carbides present decreased with increasing austenitizing temperature as seen in Figure 9. These results are similar to those obtained by other investigators [14,15]. Figure 9 also shows that the ferrite boundary carbide size decreased as austenitizing temperature increased. These results indicate dissolution of the carbide.

The time required to dissolve the carbides is temperature dependent and decreases with increasing austenitizing temperature. Complete dissolution of the carbides was obtained within 10 hours

for temperatures between 1050 and 1125°C (Figure 2). At 1000°C, delta ferrite is eliminated within 10 hours but the carbides are still present as seen in Figures 2 and 7b. These carbides were not dissolved in 30 hours.

Discussion

Several phase diagrams [13,16,17] have been developed for use with 12% Cr steels. The diagram shown in Figure 10 [16] for Fe-Cr-0.2C was used as the basis for this investigation. The austenite field extends to about 14% Cr at 1100°C and is restricted by delta ferrite at higher temperatures and by carbides at lower temperatures. Thus at 12% Cr, pure austenite should exist between 950 and 1250°C. The presence of delta ferrite and/or large carbides after austenitizing for 30 minutes indicates that equilibrium structures were not obtained in the steel investigated. The presence of ferrite forming alloying elements such as Mo [10,18] tend to effectively move the steel to a higher chromium equivalent content decreasing the ability to form a fully martensitic structure in this 12% Cr. steel.

The phases observed by metallographic examination for this steel, as reported in Figure 2, indicate the ferrite + austenite + carbide region extends over the range of 1000 to 1225°C for austenitizing times of 30 minutes. The upper limit of this three phase region decreases in temperature with increasing austenitizing times and drops below 1050°C at about 10 hours. However, at 1000°C the austenite + carbide region appears between 3 and 10 hours and persists past 30 hours.

The minimum delta ferrite found for austenitizing 30 minutes at 1100°C, indicates that the outermost limit of the full austenite region is close to 1100°C. However, times of greater than 10 hours were required to obtain a completely austenitic structure at 1100°C. The presence of delta ferrite and/or residual carbides at the lower austenitizing temperatures points out the difficulty in obtaining equilibrium structures when delta ferrite is initially present in the starting material.

Conclusions

The following conclusions resulted from this investigation.

1. When delta ferrite is initially present in HT-9 steels, equilibrium is difficult to obtain on austenitization.
2. A constant austenite grain size was obtained at 1000°C but increased with both time and temperature at higher temperatures.
3. The weight percent of extracted carbides in untempered steels and the size of the ferrite boundary carbides decrease with increasing austenitizing temperature.
4. Austenitizing in the region of 1050 to 1125°C required 10 hrs to completely dissolve residual carbides and 10 to 30 hrs to eliminate ferrite.
5. Residual carbides were present past 30 hrs when austenitizing at 1000°C

TABLE 1
Chemical Composition of HT-9 (wt.%)

Fe	Ni	Cr	Mo	W	Si	MN	C
Bal	0.51	11.8	1.03	0.52	0.21	0.50	0.21
Cu	Ti	Al	B	V	S	P	N
0.04	0.002	0.031	0.0008	0.33	0.003	0.008	0.006

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a

b

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a

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a

b

c

d

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