Hot Ductility Loss and Recovery in the CGHAZ of T23 Steel during Post-weld Heat Treatment at 750°C

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The coarse grained heat affected zone (CGHAZ) of T23 steel was assessed by strain-to-fracture (STF) tests during post-weld heat treatment (PWHT) at 750°C to investigate the microstructure changes and corresponding hot ductility. Scanning electron microscope (SEM), transmission electron microscope (TEM), and small angle x-ray scattering (SAXS) combining with JmatPro was used to study the characteristics of carbides, dislocation density, and lath size. Voids and micro-cracks were also analyzed by SEM. The results showed that sample with 1 min PWHT lost its hot ductility and intergranular cracking occurred. The hot ductility recovered with increasing PWHT time. However, the mechanisms were quite distinct during different time periods. Within PWHT time range from 0.5 h to 1.5 h, the recovery of hot ductility resulted from decreasing dislocation density. The hot ductility exhibited an opposite tendency with hardness. By contrast, the hot ductility and the hardness increased together after 2 h PWHT. It was resulted from the formation of lots of V-rich MC carbides in grain and coherent/semi-coherent smaller M23C6 carbides at the prior austenite grain boundaries (PAGBs). The strength of the grain interior and PAGB increased together. Plastic deformation was accommodated only by the cracking or shearing of low strength blocks. The fracture surface exhibited quasi-cleavage cracking together with dimples, which represented of good hot ductility. It could be concluded that the drastic changes of microstructure altered the manner of plastic deformation accommodation during stress relief, which led to the hot ductility loss and recovery during PWHT at 750°C.

KEY WORDS: hot ductility; T23 steel; CGHAZ; carbides.

1. Introduction

T23 is used at 500 to 600°C in modern power station. The chemical composition is designed with respect for manufacture of membrane walls without preheating and, if possible, without heat treatment.1) The weldability is improved by decreasing carbon content to under 0.1% and economical alloying with Cr, Mo, V, W and B. The reduction of these elements to a bare minimum guaranteeing creep resistance also decreases the carbon equivalent, and thereby the preheating is not necessary during welding.2) The joints are typically multipass butt welds and are laid down under conditions of severe structural restraint. The residual stresses thus developed may be of the same order of magnitude as the yield stress, and are relieved by applying a post-weld heat treatment (PWHT) which involves heating for several hours3) in the temperature range from 720 to 800°C.4) 750°C or 760°C is usually selected as the PWHT temperature at present.2)

Due to the metastable nature of the strengthening precipitates, the carbides undergo undesirable transformation during elevated temperature service or during PWHT. The type and size of carbides change with PWHT time and temperature.5,6) After long time tempering, the volume fraction of carbides in lath will decrease, and carbides at grain boundary (GB) will coarsen.5) Meanwhile, the dislocation density will decrease and consequently the microstructure recovery.7) Therefore, the mechanical properties must have changed greatly, which plays an important role in the course of stress relief during PWHT and future service at high temperature.

For the heat-resistant steel which is susceptible to reheat cracking, the hot ductility of coarse grained heat affected zone (CGHAZ) is poor. Therefore, the operating stress and residual stress might lead to reheat cracking during PWHT or high temperature service.8–12) In our previous work, the reheat cracking mechanism depended on the PWHT temperature.13) Similarly, the microstructure and hot ductility would also be affected by the PWHT time. Thus the nucleation and propagation of cracks will be various with different PWHT time.

In addition, for welded assembly with wall thickness less than 10 mm, 2 h is enough for PWHT.14) However, microstructure evolution and mechanical properties changes of base metal during long time aging and creep are paid more attention than those of CGHAZ during PWHT.1,15,16) Similarly, few researches are reported to discuss the evo-
olution of microstructure and corresponding hot ductility in the CGHAZ of T23 steel during PWHT. In this study, microstructural characterization was carried out before and after STFs by scanning electron microscope (SEM), transmission electron microscope (TEM), and small angle x-ray scattering (SAXS). JmatPro was used to calculate the precipitation evolution during PWHT, which was helpful for the secondary phase particles identification. Combing the mechanical properties obtained by STF and hardness tests, the mechanisms of the PWHT time on hot ductility of T23 steel were then clarified.

2. Experimental Procedures

2.1. Materials Preparation and Mechanical Testing

CGHAZ preparation, PWHT and STF tests were conducted on Gleeble 3500. The dimensions of the specimens, parameters of the thermal cycle and loading are shown in Fig. 1, which have been described in detail in our previous studies. The chemical compositions are listed in Table 1. Considering the emphasis of this work, specimens were prepared with different PWHT time: 1 min, 0.5 h, 1 h, 1.5 h, 2 h at 750°C, and followed by STF tests at the same temperature. Three sets of samples with same PWHT time were prepared. Two sets were used to study the microstructure before STF tests. The third set was used to access the hot ductility and microstructure after fracture.

The hardness of samples was obtained by a Vickers diamond indentation test with a load of 4.9 N. The hardness reported was the average of five indentations results per specimen.

2.2. Microstructural Observation

Observation of the microstructural characteristics of the specimens was carried out using a NOVA NanoSEM 230 SEM and JEOL-2100F TEM. Metallographic specimens were prepared by grinding and polishing then etched with 4% nital. The secondary phase particles identification, dislocation density and lath size measurements were performed on TEM with energy dispersive spectrometer (EDS) operated at 200 kV. Thin foils were prepared in a twin-jet electropolisher, operated at 50 V, in a solution consisting of 95% acetic acid and 5% perchloric acid. The electrolyte temperature was maintained below −30°C. Dislocation density measurements were performed for TEM foil thicknesses between 180 and 220 nm. For the TEM micrographs evaluated in the present study a g of [220] was used. One micro grain was identified which contained different dislocation densities at five different locations within this scatter range. Additionally, the dislocation density of 1 min samples was too high to measure. It was calculated by empirical formula. The statistics of lath size was conducted in ten zones per foil with a magnification of 8000 times. After STF tests, the fracture surface and locations of cracking initiation and propagation on the axial-sections are studied by SEM.

2.3. Physicochemical Phase Analysis

The phase identification of the precipitates was also done on the extracted residues obtained by electrolytically dissolving the matrix in a solution consisting of 1% tetramethylammonium chloride, 10% acetylacetone and methanol in volume fraction. The electric current was less than or equal to 0.6 A and the temperature was in the range of −5°C to 0°C. The residues were collected and washed out. Then the residues extracted were identified by SAXS performed on a PANalytical X’Pert MPD diffractometer at 40 kV and 40 mA. Cu Kα radiation was used and a 2θ range from 20–120° was step-scanned with a scanning step of 0.0167° and scanning time of 20 seconds.

3. Results and Discussion

Before STF tests, the microstructure at different PWHT time was examined. Figure 2 shows the distribution, evolution, types, and relative quantity of precipitates after different PWHT time. There was carbides uncovered GB with 1 min PWHT. With increasing PWHT time, the GB was nearly covered by coarsened carbides. As shown in Figs. 2(a)–2(e) and Table 2, the average size of precipitates at prior austenite grain boundary (PAGB) increases after 1 min PWHT and keeps stable within the PWHT time range from 0.5 h to 1.5 h. After 2 h PWHT, the average size of precipitates at PAGBs decreased and some of these precipitates were darker than others. It stated that the precipitates at PAGBs were unstable and their type could have changed after 2 h PWHT.

The contrast of secondary electron image depended on the surface topography of the specimen. Carbides with high

Table 1. Chemical composition (wt%) of the T23 steel.

| C   | Mn  | P   | S   | Si  | Cr  | Mo | W  | V  | Nb | N   | Al | B  | Ni | Ti | Tr/N |
|-----|-----|-----|-----|-----|-----|----|----|----|----|-----|----|----|----|----|-----|
| 0.06| 0.37| 0.012| 0.002| 0.28| 2.32| 0.08| 1.56| 0.22| 0.044| 0.0035| <0.015| 0.0026| 0.05| 0.009| 3.9 |

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Cr content were resistant to the natal corrosion and their surface was high and bright. Otherwise, carbides with low Cr content have similar height and brightness with matrix. It was declared that M$_3$C and M$_2$3C$_6$ were Fe-rich carbides, M$_7$C$_3$ were Cr-rich carbides. Therefore, it was speculated that the bright carbides and dark ones at PAGBs in Fig. 2(e) were Cr-rich M$_7$C$_3$ and Fe-rich M$_2$3C$_6$, respectively.

Though it was difficult to identify incoherent M$_7$C$_3$ carbides from magnetic thin foil by TEM, the types of carbides could be still determined indirectly. Figure 2(f) shows the precipitates evolution computed by JmatPro. It was found that M$_3$C carbides dissolved quickly during PWHT and more stable carbides (M$_2$3C$_6$, M$_7$C$_3$, MC) formed. M$_7$C$_3$ carbides completely transformed to M$_2$3C$_6$ and MC carbides when the PWHT time reached 1 000 s. Though the cooling rate of model used in JmatPro was slightly faster than that in our study, the precipitation sequence was not affected. The results above agreed well with previous study. It can be deduced that the time 1 000 s in Fig. 2(f) corresponds to a certain time after 2 h PWHT in our research.

Figure 3 also presents three types of carbides which are in agreement with the results obtained by JmatPro. Due to the high volume of MC carbides in extracted residues, only MC was determined by SAXS as VC carbides. It was worth noting that the diffraction peak of V-rich MC increased obviously with 2 h PWHT. Due to the same formation tendency of MC and M$_2$3C$_6$ carbides shown in Fig. 2(f), M$_2$3C$_6$ (darker carbides) must precipitate greatly at PAGBs while M$_7$C$_3$ (brighter carbides) decreased after 2 h PWHT (as shown in Fig. 2(e)).

The mechanical properties affected by the microstructure could be also helpful for the carbides identification. Figure 4 shows the mechanical properties at different PWHT time. The stress-displacement curves in Fig. 4(a) reflect the strength and hot ductility of material. The sample with 1 min PWHT presented obvious brittleness. With increasing time, the hot ductility of material was improved. It was noted that the slope of the latter part of the curves had changed. With increasing PWHT time, the slope tended to be stable. It reflected the drastic changes of microstructure which gradu-
ally became stable with increasing PWHT time.

Figure 4(b) shows the hardness and reduction in area of samples at different PWHT time. The hardness fell quickly and then kept low falling rate. However, the hardness increased after 2 h PWHT. The maximum of stress in Fig. 4(a) also shows the higher strength with 2 h PWHT than that with 1.5 h PWHT. The reduction in area, which represented for hot ductility, exhibited an opposite tendency with hardness within the PWHT time range from 1 min to 1.5 h. However, the hot ductility and the hardness increased together after 2 h PWHT.

The fracture modes in Figs. 5(a)–5(f) can verify the hot ductility. The sample with 1 min PWHT exhibited obvious intergranular cracking. With increasing PWHT time, most of the surfaces presented dimples and only a few intergranular cracking occurred. However, beyond a certain time, the dimples became shallow and quasi-cleavage cracking appeared. According to the reference,\textsuperscript{18} the quasi-cleavage cracking do not reduce the hot ductility as cleavage cracking. Additionally, there were still dimples in the fracture surface, which was on behalf of good hot ductility. The fracture surface analyses were in agreement with the hot ductility results in Figs. 4(a) and 4(b).

In order to explain the unusual behavior between the hot ductility and the hardness in Fig. 4(b), the crystal structure of M$_2$3C$_6$ at PAGBs, dislocation density, and lath size were studied. Figures 6(a)–6(e) show one of the TEM analyses of
Table 3. Dislocation densities and lath size determined in the present study by TEM.

| Heat treatment | $\rho_{TEM} \times 10^{14}$ m$^{-2}$ | Size of lath [nm] |
|---------------|-----------------------------------|------------------|
| 1 min         | 11.14                             | 435.0            |
| 0.5 h         | 1.89                              | 474.8            |
| 1 h           | 1.45                              | 479.8            |
| 1.5 h         | 1.31                              | 489.3            |
| 2 h           | 0.83                              | 496.0            |

M$\text{23C}_6$ carbides. They were Fe-rich carbides with some Cr, W, V, Mo, Nb. Parts of them possessed twin substructure. M$\text{23C}_6$ has good coherent relation with ferritic matrix and its twin structure is beneficial for the interface strength.\(^{19}\)

That may be why Nawrocki\(^{6}\) proposes to increase the proportion of M$\text{23C}_6$ at PAGBs to reduce the reheat cracking susceptibility.

The dislocation density and lath size after different PWHT time are listed in Table 3. Figures 6(f) and 6(g) display one TEM result of dislocations after 2 h PWHT. The
dislocation density decreased with increasing PWHT time while the lath size just slightly increased. Therefore, the decreasing of hardness had a close relation with the quick recovery of dislocation density.

However, when PWHT time reached 2 h, a large number of V-rich MC carbides precipitated at lath boundaries (stabilize the lath structure) and dispersive distributed within laths. Therefore, the hardness increased when the speed of dislocation recovery was slower than that of precipitation of VC carbides. Meanwhile, coherent/semi-coherent smaller M_23C_6 carbides precipitated at PAGBs, which increased interface strength of PAGBs. Due to the increased strength of grain interior and PAGBs, plastic deformation during STF test was accommodated only by the cracking or shearing of low strength blocks. Intergranular cracking did not occur and good hot ductility retained.

Cracking mechanisms and corresponding axial section SEM images at different PWHT time are summarized in Fig. 7. The left part and right part showed the distribution and size of carbides and cavities when the cracks initiated and after fracture. The SEM images in fracture mode shown in Fig. 7 could be found in Fig. 5. It was found that the location where cavities nucleated determined the fracture modes. The cavities tended to nucleate at the weak interface determined by the comprehensive role of carbides and dislocation density. Sample with 1 min PWHT lost its hot ductility because of weakened PAGBs by M_3C.6,12) Thus cavities nucleated at PAGBs. Within PWHT time range from 0.5 to 1.5 h, the recovery of hot ductility resulted from decreasing dislocation density. Cavities nucleated in soft-

![Fig. 7](image_url)

Fig. 7. Cracking characteristics and corresponding SEM images of (a) 1 min; (b) 0.5 h, 1 h; and (c) 1.5 h, 2 h failure samples. Carbides and voids were represented by red empty circles and blue filled circles, respectively. Line type of arrows standed for different fracture modes.
ened grain interior. After 2 h PWHT, the strength of grain interior and PAGBs both increased, the cavities tended to nucleate at block boundaries. It led to the cracking or shearing of low strength blocks and the fracture surface showed quasi-cleavage cracking. As can be seen, drastic changes of microstructure altered the cracking mechanisms, which led to the hot ductility loss and recovery during PWHT at 750°C.

4. Conclusions

(1) With increasing PWHT time, M₃C and M₇C₃ carbides dissolve while smaller M₂₃C₆ carbides precipitate at PAGBs. V-rich MC, which strengthens the grain interior, has similar precipitation tendency with M₂₃C₆.

(2) Sample with 1 min PWHT loses its hot ductility because of weakened PAGBs by M₃C carbides. Within PWHT time range from 0.5 to 1.5 h, the dislocation density decreases greatly, which effectively softens the grain interior. It leads to the plastic deformation happens mainly in grain rather than at PAGB, which results in the recovery of hot ductility.

(3) After 2 h PWHT, a large number of V-rich MC carbides precipitate at lath boundaries and dispersive distribute within laths. The strength in grain increases. Meanwhile, coherent/semi-coherent smaller M₂₃C₆ carbides precipitate at PAGBs, which increases interface strength of PAGBs. Due to the increased strength of grain interior and PAGBs, plastic deformation during STF test is accommodated only by the cracking or shearing of low strength blocks. Intergranular cracking does not occur and good hot ductility retains.

The results in this study provide an instruction for heat treatment process of T23 steel and other heat resistant steel, which can ensures the safety after the welded joint was put into operation.

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