EFFECTS OF THE TEMPERING TEMPERATURE ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF X70 DUAL PHASE STEEL

Mansouri Tahar, Zidelmel Sami^{*}, Allaoui Omar

Laboratory Process Engineering, University of Laghouat, B.P. 37G route de Ghardaia 03000 Laghouat, Algeria

> Received 24.10.2021 Accepted 25.04.2022

Abstract

This study uses direct quenching (DQ) heat treatment at an intercritical annealing temperature (IAT) of 800 °C to form a martensite-ferrite dual phase microstructure of X70 steel. The effects of tempering temperatures ranging from 200 to 500 °C on tensile properties in a dual-phase X70 steel are investigated. Carbon diffusion and redistribution in the microstructure are influenced by tempering. It was discovered that the amount of carbides increases with the tempered temperature, resulting in depleted carbon in martensite. Conversely, increasing the temperad temperature causes a decrease in ultimate tensile strength and yield strength while increasing elongation.

Keywords: dual phase steel; intercritical annealing; tempering; mechanical properties.

Introduction

Martensite-Ferrite Dual phase steel, which is interesting engineering steel due to their excellent mechanical properties is composed of a softer ferrite phase and the harder martensite phase [1–4]. Dual phase microstructures can be developed by heating in the austenite-ferrite region and by quick quenching to room temperature austenite changes to martensite. The formation of martensite leads to residual stresses and a high density of dislocations in ferrite which can be reduced by tempering treatment [5]. The martensitic transformation is the source of particular mechanical properties of dual phase (DP) steels, such as high tensile strength, continuous yielding behavior, low yield strength and high strain hardening rate [6-9]. The mechanical behavior of (DP) steel is affected by many factors, such as grain size [10, 11], dislocation density [12, 13], constituent phase properties [14], martensite volume fraction [15, 16], and martensite morphology [17]. To improve ductility and formability of high strength DP steels,

^{*} Correspending author: Zidelmel Sami, s.zidelmel@lagh-univ.dz

tempering process could be proposed. During tempering, the changes in microstructure that occur induce improvement of both martensitic and ferritic phases. Many authors reported, in the literature, that an increase of the tempering temperature causes a decrease in the yield strength and the tensile strength [18, 19]. *Anazadeh Sayed and Kheirandish* [20] found that the tempering treatment affects the martensite phase and also the dislocation density of the ferrite phase. *Baltazar Hernandez et al.* [21] discussed the microstructural evolutions during tempering treatment. *Li et al.* [22] observed carbide precipitation and coarsening of martensite structures during tempering process.

Precipitation during the tempering treatment of dual phase X70 steel with a (ferrite+martensile) microstructure has been rarely studied. However, there is little work on detailed tempering behavior, such as carbide precipitation and its influence on softening, of Dual Phase pipeline steel. Thereby, the purpose of this article was to confirm our recent study by exploring the evolution of the softening phenomenon as a function of tempering of martensite in X70 Dual Phase steel. Hence, to determine the ability of the tempering temperature to modify the microstructure and the mechanical properties of the two-phase X70 steel, we studied the tensile properties and the microstructures of certain samples quenched at several temperatures.

Experimental procedure

The steel that was the subject of this study is of grade X70 according to the API standard, the chemical composition of which is given by Table 1. The steel is provided by Alpha pipe gas society, Ghardaia, Algeria. The specimens were intercritically annealed at 800 °C followed by water quenching to obtain a martensite ferrite dual phase structure. They were then tempered at various temperatures ranging from 200 to 500 °C as illustrated in Figure 1. The microstructures of the specimens were identified using scanning electron microscopy (SEM). Before microstructure observations, specimens have undergone mechanical polishing using abrasive papers and etched in a 3% Nital solution. Tensile tests on flat specimens were carried out at room temperature using a computer-assisted Mohr Federhaff Lasenhausen System machine.

С	Mn	Si	S	Р	Nb	V	Ti	Al
0.07	1.52	0.34	0.001	0.012	0.045	0.048	0.003	0.035

Table 1. Chemical composition of X70 steel (wt.%).



Fig. 1. Schematic illustration of the heat treatment routes.

Results and discussion

Microstructure

Figure 2. shows the microstructure by Scanning Electron Microscope (SEM) of API X70 steel after having undergone the direct quenching (DQ) heat treatment with an annealing temperature of 800 °C. It was observed that the microstructure is consisting of coaxial grains of ferrite (F) and martensite islands (M). It is noted that the volume fraction of martensite (VFM) of the specimen treated at 800 °C is 45%. During the quenching which follows an intercritical annealing, there is a change in volume which induces a plastic deformation. Consequently, this creates a high density of free dislocation in the ferritic grains in the vicinity of the martensite [13]. The increase in strength and reduction in ductility of steel is provided by the formation of martensite which generates a large amount of distortion and internal stresses. The large number of free dislocations cannot be obstructed by the few solute atoms at the ferrite / martensite interface. Therefore, the phenomenon of diffusion compensates the rest of the atoms coming from the interior of the ferritic grains. This can be achieved by tempering.



Fig. 2. SEM image showing Martensite-Ferrite dual phase microstructures of the specimen intercritically annealed at 800 °C.

Figure 3. shows an SEM microstructure of specimens that underwent intercritical annealing at 800 °C followed by tempering at 300 °C and 400 °C. The microstructure consists of a ferrite matrix (F) and islands of hard martensite (M). During the process, the carbides (C) can be observed in the microstructure. The increase of the tempering temperature induces carbide precipitation which leads to an increase of its size. The ferrite is covered with numerous carbide particles when the tempering temperature reaches 300 °C. This is in accordance with what is reported in the literature as several researches [19, 23], assert that the carbides start to precipitate inside the ferrite grain when the tempering temperature is increased to about 300 °C.



Fig. 3. Microstructures by SEM of the specimens annealed at 800 °C and followed by tempering for 1 h, and temperatures: a) 300 °C, and b) 400 °C.

Mechanical properties

Figure 4. shows the effect of tempering temperature on the evolution of strength (yield strength, ultimate tensile strength) and total elongation. We can notice that Yield stress (YS) and ultimate tensile strength (UTS) decrease with the increase of temperature due to the diffusion of carbon atoms from their stressed interstitial lattice sites to ensure the formation of second-phase carbide precipitates. On the other hand, the diffusion of carbon atoms increases with the increase of temperature. From a temperature of 200 to 500 °C, we observe the reduction of both stresses from 730 to 640 MPa for (UTS), and from 550 to 480 MPa for (YS) as it is shown in the figure 4a, while the elongation rises sharply and continuously from 24.5% to 26.3% as it is shown in the figure 4b.

When the tempering temperature reaches about 300° C, the precipitation of carbides is started and the residual stresses and the dislocation density decrease further, which allowed the lattice of the martensite to be less tetragonal. Therefore, the (YS) and (UTS) decrease and the elongation increases. According to *Zamani et al.* [24], there is evidence that the precipitating carbides nucleate at dislocations during thermal processing of DP steels. However, the tempering allows the reduction of residual constraints and the density of the free dislocations that surround the martensite islands [18, 20].



Fig. 4. Change in mechanical properties in the DP X70 steel as a function of tempering temperature.

(a) Yield Stress and Ultimate Tensile Strength (b) total elongation.

Failure mode during a tensile test

In order to clarify the effect of tempering temperature on ductility, tensile fractures were studied using a scanning electron microscope (SEM) for all the heat-treated samples. The tensile fracture specimens broken at room temperature for X70 steel, obtained at different tempering temperatures, are shown in Figure 5. The presence of the dimples in the fractured surfaces reveals that the rupture is ductile, which is managed by the nucleation, the growth and the coalescence of the voids. This is a typical appearance of ductile failure due to the high ductility reported with this type of heat treatment. A closer observation of the fractographs indicates that the increase in tempering temperature causes the formation of larger sized dimples due to the presence of coarser martensite in the microstructure at high tempering temperature.

Multiple failure modes (voids nucleation, their subsequent growth and the ultimate coalescence of the most voids at fracture) provides a multitude of fractureproperty, such as the total elongation of this DP steel, as shown in the Figure 4b. This means that the increase in the ductility of the steel leads to an increase in the size of the dimples formed during the tensile test. The coarser dimples dominant in the DP steel aged at 400 °C, can be attributed to the particle (carbides) distribution. At this temperature, the size of the carbides was larger than that of 300 °C, which in turn left more space for the growth of voids. The work of Bag et al. [16] on fractographic observations of (DP) steels indicates that, during plastic deformation, ferrite deforms before martensite and makes cracks germination easy either at the level of the precipitates present, or at the ferrite-martensite interfaces. Subsequently, according to the state of the constraints present in the microstructure, the cracks propagate either by cleavage or by dimples.

Many researchers [14, 26] have observed that the formation of micro-cavities results both from the breakdown of martensite particles and from the decohesion of the interface. They consider that martensite can be a site for the initiation of micro-cavities. Thus, during plastic deformation, the stress concentrations in the ferrite near the

martensite particles increase rapidly, which leads to the formation of microcavities at the onset of plastic deformation.



Fig. 5. SEM micrographs of fracture surface for different tempering temperatures: a) 300 °C, and b) 400 °C.

Conclusions

The evolution of microstructure and tensile properties as a function of tempering temperature of X70 Dual phase steel was investigated in this study. The obtained results lead to the following conclusions:

- 1. Intercritical treatment at 800 °C leads to a structure (martensite-ferrite) whose martensitic phase is well distributed in the ferritic matrix.
- 2. An increase in the tempering temperature induces a decrease in yield strength (YS) and ultimate tensile strength (UTS) and a decrease in total elongation.
- 3. From the tempering temperature 300 °C, there is a precipitation in the ferrite because of the diffusion of carbon. Furthermore, the precipitation grows with the tempering temperature.

Acknowledgements

The facilities received by alpha pipe gas society and the financial support of the Directorate General for Scientific Research and Technological Development (DGRSDT) to carry out this work are gratefully acknowledged by authors.

References

- M. Azuma, S. Goutianos, N. Hansen, G. Winther, X. Huang: Mater Sci Technol, 28 (2012) 1092-1100.
- [2] W. Wang, M. Li, C. He, X. Wei, D. Wang, H. Du: Mater Des, 47 (2013) 510-521.

- [3] S. Oliver, T.B. Jones, G. Fourlaris: Mater Sci Technol, 23 (2007) 423-431.
- [4] C. Tasan, M. Diehl, D. Yan, M. Bechtold, F. Roters, L. Schemann, C. Zhang, N. Peranio, D. Ponge, M. Koyama, K. Tsuzaki, D. Raabe: Annu Rev Mater Res, 45 (2015) 391-431.
- [5] M. Erdogan, R. Priestner: Mater Sci Technol, 18 (2002) 369-376.
- [6] M. Sarwar, R. Priestner: J Mater Sci, 31 (1996) 2091-2095.
- [7] H. Seyedrezai, A.K.Pilkey, J.D.Boyd: Mater Sci Eng, A 594 (2014) 178-188.
- [8] M. Asadi, B.C.Decooman, H.Palkowski: Mater Sci Eng, A538 (2012) 42-52.
- [9] R.R. Queiroz, F.G.Cunha, B.M.Gonzalez: Mater Sci Eng, A543 (2012) 84-87.
- [10] M. Calcagnotto, Y.Adachi, D.Ponge, D.Raabe: Acta Mater, 59 (2011) 658-670.
- [11] Y. Kang, Q. Han, X. Zhao, M. Cai: Mater Des, 44 (2013) 331-339.
- [12] Y.Bergström, Y.Granbom, D.Sterkenburg: J Metall, Journal of Metallurgy (2010) 1-16.
- [13] M. Calcagnotto, D. Ponge, E. Demir, D. Raabe: Mater Sci Eng, A527 (2010) 2738-2746.
- [14] J. Kadkhodapour, S. Schmauder, D. Raabe, S. Ziaei-Rad, U. Weber, M. Calcagnotto: Acta Mater, 59 (2011) 4387-4394.
- [15] M.R. Akbarpour, A. Ekrami: Mater Sci Eng, A 477 (2008) 306-310.
- [16] A. Bag, K.K. Ray, E.S. Dwarakadasa: Metall Mater Trans, A 30 (1999) 1193-1202.
- [17] Q.H. Han, Y.L. Kang, P.D. Hodgson, N. Stanford: Scr Mater, 69 (2013) 13-16.
- [18] X. Fang, Z. Fan, B. Ralpha, P. Evans, R. Underhill: J Mater Process Technol, 132 (2003) 215-218.
- [19] S. Gunduz: Mater Lett, 63 (2009) 2381-2383.
- [20] A. A. Sayed, Sh. Kheirandish: Mater Sci Eng, A 532 (2012) 21-25.
- [21] V.H. Baltazar Hernandez, S.S. Nayak, Y. Zhou: Metall Mater Trans, A 42 (2011) 3115-3129.
- [22] H. Li, S. Gao, Y. Tian, D. Terada, A. Shibata, N. Tsuji: Mater Taday Proc, 2 (2015) S667-S671.
- [23] A. Kamp, S. Celotto, D.N. Hanlon: Mater Sci Eng, A 538 (2012) 35-41.
- [24] M. Zamani, H. Mirzadeh, M. Maleki: Mater Sci Eng, A 734 (2018) 178-183.
- [25] J. Kang, Y. Ososkov, JD. Embury, DS. Wilkinson: Scr Mater, 56 (2007) 999-1002.

Creative Commons License

This work is licensed under a Creative Commons Attribution 4.0 International License.