Studying the Influence of Tempering Conditions on Tempered Martensite Embrittlement of Low Alloy Steel

NC Wijesinghe¹ and GIP De Silva²

^{1, 2} Department of Materials Science and Engineering, Faculty of Engineering, University of Moratuwa, Sri Lanka

#1 nuwanthi.wijesinghe@gmail.com, 2<indikagip@uom.lk>

Abstract— This work focused on studying the effects of quenching mediums and tempering conditions on the tempered martensite embrittlement of SA335P11 low alloy steel that is mainly used for high pressure steel pipes in thermal power plants. The specimens are quenched in AH metal working fluid that is generally used to quench metal samples and tempered 150 - 500 °C to assure that embrittlement occurred at the temperature range of 200-350 °C. Heat treated samples are subjected to hardness test and fracture toughness test to determine the degree of tempered martensite embrittlement. Effects of tempering temperature and time, and quenching medium on tempered martensite embrittlement are discussed based on the fracture toughness and hardness of the heat treated samples. The results showed that under the tempering temperatures of 200°C to 350 °C, fracture toughness and hardness of samples are rapidly decreased. Furthermore, fracture toughness increases and hardness decreases with increasing the tempering time. Moreover, higher the cooling rate of quenching medium, fracture toughness of the specimen is decreased and hardness is increased.

Keywords— Tempered Martensite Embrittlement, Fracture Toughness, Quenching Medium

I. INTRODUCTION

SA335P11 is a ferritic alloy steel that is widely used for high temperature services such as nuclear and coal power plants, industrial boilers and petro chemical plants. This alloy steel is suitable for the above applications because of their proper bending, flanging (vanstoning), and similar forming operations, and for fusion welding. Since the material consists of Molybdenum (Mo) and Chromium (Cr) elements that enhance the strength as well as the elastic limit, resistance to wear, impact qualities, hardenability, restraining of grain growth, high temperature creep strength and corrosion resistance of steel. These properties make the material ideal for using in power plants where fluids and gases are transported at extremely high temperatures and pressures. Accordingly, P11 steel pipes are used in Lakvijaya Power plant, Sri Lanka to circulate high temperature steam. Therefore, the working temperature of the pipe is above 520 °C where the embrittlement is not severely pronounced. (Hu and Yang, 2004) However, during the reduction of working temperature to 250 - 350 °C range embrittlement can be detectable and it ultimately leads to reduce the shelf life of the material(Darwish et al., 1991).

The mechanism of this tempered martensite embrittlement is not cleared and there have been no reports on studying the effects of quenching mediums and tempering condition on tempered martensite embrittlement.

R. M. Horn et al. have shown that the embrittlement of steel can be undergo through several mechanisms such as interlath carbide precipitation, decomposition of retained austenite, or Prosperous (P), Sulphur (S) and Tin (Sn) in grain boundaries, and is almost due to combination of above factors. Among these factors they have proven that retained austenite has a major contribution to promote tempered martensite embrittlement of AISI 4340 steel(Horn R.M., 1977).

P11 steel consists of 0.11% C, 0.49% Mn, 0.20% Si, 0.87% Cr, 0.45% Mo, S and P. Generally, Carbon content effects the hardness, tensile strength, ductility, toughness and machinability of steel. (Ohtani, 1976) There Mn, Cr and Si contents improve the hardenability and also enhance the production of martensite throughout the volume of specimen and strengthen the material uniformly.

The objective of this study is to determine the degree of embrittlement of P11 steel at different tempering time and temperatures under different cooling rates. (Peters et al., 1989). It could be useful to develop a model to predict the fracture life of pipes used in real life applications under different conditions.

II Methodology

2.1. Assuring the Chemical Composition of obtained samples

P11 test samples were obtained from Lakwijaya Power Plant and full chemical analysis was conducted by Atomic Absorption spectrometer (Avanta Branded – AAS), Carbon – Sulphur Analyzer, and UV- Vis Spectrophotometer (Sonaso Branded – UV-Vis). The obtained results were

SA335P11 low alloy steel.

2.2. Metal Specimen Preparation for Testings

Standard specimens with the dimensions of 10mm \times 10mm × 50mm and 2mm notch were prepared for the charpy impact test. The same specimens were used for Vicker's Hardness test prior to the charpy impact test.

2.3. Analysis of the Effects of Tempering Temperature and Time on Tempered Martensite Embrittlement

Test specimens were austenitized at 870 °C for 1 hour and quenched the sample for 2 hours in AH metal working fluid. The quenched samples were tempered at 8 distinct temperatures of 150, 200, 250, 300, 350, 400, 450 and 500 °C for 4 hours. The experiment was repeated varying the tempering time for 2, 4, 6 and 8 at 300°C tempering temperature. The tempered samples were subjected to Charpy impact test and Vicker's hardness test.

2.4. Analysis of the Effects of quenching medium on **Tempered Martensite Embrittlement**

Test specimens were austenitized at 870 °C for 1 hour and quenched in 4 different quenching mediums i.e. air, water, brine and metal working fluid and 2 hours tempered samples at 300 °C were subjected to toughness and hardness tests.

III Results and Discussion

3.1. Assuring the Chemical Composition of obtained samples

Table 01: Composition of SA335P11 material in weight percentages

Element	Standard ASME SA335P11 composition %	Test Results %
С	0.05 - 0.15	0.11
Mn	0.30 - 0.60	0.49
Si	0.50 – 1	0.62
Cr	1 - 1.50	1.15
Мо	0.44 – 0.65	0.49
S	0.025 max	0.006
Р	0.025 max	0.008

The test results were matched with the standard chemical composition of SA335P11.

compared with the standard chemical composition of **3.2.** Analysis of the Effects of Tempering Temperature and Time on Tempered Martensite Embritlement

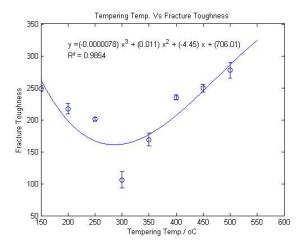


Figure 01: Variation of fracture toughness with tempering

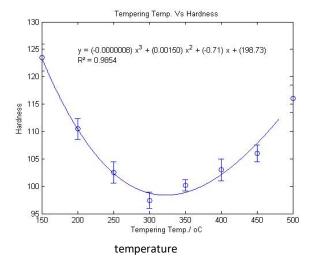


Figure 02: Variation of hardness with tempering temperature

The variation of toughness and hardness of SA335P11 steel with tempering temperature are shown in Fig. 1 & 2. These results indicates toughness and hardness decrease as the tempering temperature increases from 200 to 350 °C. The minimum toughness and hardness was observed when tempered at 300 °C. Therefore, tempered martensite embrittlement in P11 steel is more pronounced at 300 °C. However, tempering at temperatures above 350 °C leads to a significant increase in toughness. This loss in toughness within the range of 200 to 350 °C could be occurred due to formation of carbides of other alloying elements austenite grain boundaries and segregation of impurity elements i.e. P, S, N, Sb, and Sn at grain boundaries. (McMahon, 1977) However, when temperature increases above 350 °C, there is a possibility to dissolve the deposited carbides and segregated impurity elements throughout the austenite grains.

In addition, loss in toughness at 300 °C is correlated with the segregation of carbon to lattice defects and the precipitation of carbides (Fe₃C) along prior austenite grain boundaries during tempering, the decomposition of retained austenite and segregation of impurity elements (i.e. P, S, N, Sb, and Sn) in the grain boundaries during austentization. The exact phenomena and possible mechanisms of embrittlement associated with P11 steel can be identified by microstructure analysis. The extent of impurity segregation and thus the embrittlement is enhanced by the presence of Mn, and Si in the alloy.(Lee and Su, 1999)

The extent of embrittlement also depends on the austenization temperature. As the austenitization temperature raised, the grain size increases, and thus grain boundaries decreases. When the grain boundaries become more enriched with impurities, and the embrittlement phenomenon is more pronounced. (Ennis, 2014)

The above mentioned segregation of impurities and deposition of carbides within the temperature range of 200 to 350 °C may cause weakening of the specimens due to the highly stressed areas associated with some part of the grain boundaries. There is a possibility to affect this phenomena for reducing the hardness within this temperature range.(Joshi, 1975)

The exact phenomena and possible mechanisms of embrittlement associated with P11 steel can be identified and investigated by microstructural analysis of the cross sections of tempered specimens and fractured surfaces obtained after the Charpy impact test. This microstructural analysis would be planned to conduct using high magnification microscope and SEM/EDS. (Zia-Ebrahimi and Krauss, 1984)

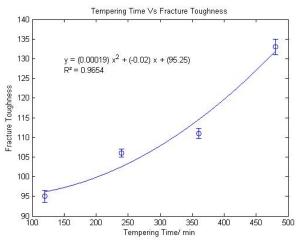


Figure 03: Variation of fracture toughness with tempering time

Fig. 03 indicates that fracture toughness increases as the tempering time is increased. However, the effect of tempering temperature is more significant than that holding time. When specimens are kept at tempering

temperature of 300 °C and increase the tempering time, martensite is transformed to pearlite and ferrite, and internal stresses associated with the structure are released. These microstructural changes result in improving the toughness and reducing the hardness.

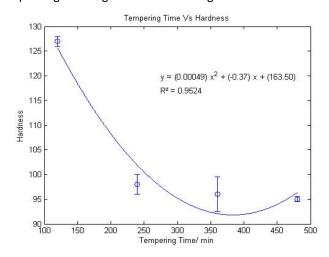


Figure 04: Variation of hardness with tempering time

3.3. Analysis of the Effects of quenching medium on Tempered Martensite Embrittlement

Table 02: Experimental data of fracture toughness and hardness with quenching medium

Exp.	Quenching	Average	Average
No	medium	Toughness	Hardness
1	Air	289.5	102.5
2	Water	248.0	119.0
3	Brine	175.5	124.5
4	AH metal	95.0	127.0
	working fluid		

According to the experimental data in Table 2, fracture toughness was changed under the different quenching mediums. Generally, higher cooling rate produces large content of martensite prior to the tempering process. Therefore, relatively higher amount of martensite is remained even after transform it to the pearlite and ferrite during the tempering process. It is obvious that remained large content of martensite under AH metal working fluid causes reducing the fracture toughness and increasing the hardness of specimens.

Water has relatively high heat capacity that can provide high cooling rate to the heated specimens. However, water absorbs large quantities of atmospheric gases, and when a hot piece of metal is quenched, these gases have a tendency to form bubbles on the surface of the metal which reduces the heat transfer between heated specimen and quenching medium and reduce the cooling rate. Slow cooling facilitate the carbon diffusion and formation of more perlite and bainite. It improves the toughness and reduces the hardness of specimen compare to air quenching.

10% Brine is prepared by dissolving sodium chloride in water. This mixture reduces the absorption of atmospheric gases that reduces the amount of bubbles in quenching medium. As a result, brine wets the metal surface and cools it more rapidly than water. Rapid cooling increase the heat transfer between specimen and quenching medium. It facilitates the martensite formation and creates hard and brittle material while reducing toughness. However, the rapid cooling rate of brine can cause cracking in high-carbon or low-alloy steels.

AH metal working fluid used for this work was highly refined mineral oil. According to experimental results specimens under AH metal working fluid showed the highest cooling rate to produce lowest fracture toughness and highest hardness.

IV Conclusion

This study showed that the tempering conditions and quenching mediums have a significant effect on to the tempered martensite embrittlement of SA335P11 high strength low alloy steel which used in high pressure steel pipes of thermal power plants. As per the experimental results embrittlement of SA335P11 steel has pronounced at $200-350\,^{\circ}\text{C}$ range. Therefore, fracture toughness and hardness have decreased within this tempering temperature range. However, hardness decreases and fracture toughness increases with tempering time. In addition, effect of the quenching medium to the fracture toughness and hardness of this steel have identified.

ACKNOWLEDGEMENT

The authors would like to offer their deepest gratitude to the Department of Material Science and Engineering, University of Moratuwa for giving the sample testing facilities. Furthermore, the gratitude goes to Mr. N.A.S.K.S. Chandrasoma (Chief Engineer-boiler and Oklahoma, Lakvijaya Power Plant) to his kind help in providing samples and required technical information. Thanks are extended to Mr. Sanjeewa Kalansooriya for his contribution to the chemical analysis.

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