EFFECT OF TEMPERING PROCESS ON HYDROGEN DELAYED FRACTURE SUSCEPTIBILITY OF 4140 STEEL FASTENER

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Abstract - *Delayed fracture is a phenomenon that happens due to the nascent hydrogen attacks at the grain boundaries of the metal slowly and makes the metal weaker thereby prone for fracture initiation. This can also be referred as hydrogen embrittlement. The susceptibility of Hydrogen delayed fracture of 4140 steel fastener has been studied with the influence of tempering temperature by means of hydrogen delayed tests. In order to solve the delayed fracture phenomenon of the steel fastener, the chamber electric furnace was used to carry out the tempering test. The specimen is machined as per ASTM standard for conducting the tensile test and it is immersed in a Hydrochloric acid for steel for the diffusion of hydrogen. The influence of various tempering temperatures on the hardness and tensile strength of steel fastener is analyzed. Fracture modes were investigated with the use of a scanning electron microscope (SEM). The results show that the performance of steel is most excellent when the tempering temperature is around 450°C to 480°C.*

Key Words: **delayed fracture; tempering; hydrogen embrittlement, constant load test microstructure**.

1.INTRODUCTION

Delayed fracture generally comes under brittle fracture. High strength steels are usually mixed with hydrogen during smelting, processing and using. Generally, hydrogen which enters steel is extremely harmful. For many materials, even a trace of hydrogen can induce delay fracture through diffusion and enrichment. Hence the diffusion and enrichment of hydrogen in metals is the premise and bridge for delay fracture. Delayed fracture of high strength steel is mainly because the intrusion of the hydrogen in the steel, which is characterized by brittle, intergranular fracture, so the starting point of improving the delayed fracture performance is to improve the effectiveness of hydrogen trapping in the steel, reduce the concentration of diffusible hydrogen in the matrix and increase the grain boundary binding strength.[1,2]. Hydrogen embrittlement is a permanent loss of ductility in a metal caused by hydrogen in combination with stress, either externally applied or internal residual stress. Hydrogen embrittlement susceptibility of martensitic steel decreases with the increase of the tempering temperature. hydrogen delayed fracture can be identified by intergranular fracture. An Intergranular fracture is a fracture that follows the grains of the material, where cracks that take place along the grain boundary.[3,4]. Susceptibility of steel to hydrogen embrittlement varies with composition, microstructure, and strength level. A354 BD rods are generally made from AISI 4140, a medium-carbon, low alloy steel that is heat treated by austenitizing, quenching and tempering to produce a tempered martensite microstructure. The tempering temperature controls the strength of this material. Strength level can be described in terms of yield strength, ultimate tensile strength, or hardness. Generally, as hardness increases, the resistance of AISI 4140 to hydrogen embrittlement decreases.[5]. The present work was done to investigate the effect of tempering process on the hydrogen –delayed fracture susceptibility of AISI 4140 steel fastener by using HCL solution.

Table -1 Chemical composition of AISI 4140 steel fastener

AISI 4140	C	Mn	Cr	P	S
Wt %	0.35-0.45	0.5-0.8	0.9-1.5	0.04	0.03

Table- 2 Heat treatment of experimental material

<table>
<tr><th>No .of samples</th><th colspan="2">Heat treatment system</th></tr>
<tr><td>1</td><td rowspan="4">850°C * 30 min, water cooling</td><td>350°C* 30min air cooling</td></tr>
<tr><td>2</td><td>400°C* 30min air cooling</td></tr>
<tr><td>3</td><td>450°C* 30min air cooling</td></tr>
<tr><td>4</td><td>520°C* 30min air cooling</td></tr>
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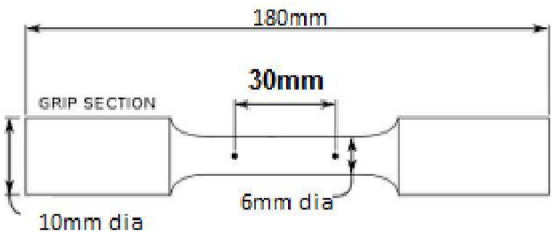


Fig- 1 Tensile test specimen with dimension

2. EXPERIMENTAL PROCEDURE

The chemical compositions of the material used in this work are given in table1. Heating of steel to various temperature can reduce the permeation and absorption rate of hydrogen can be reduced for coated steel.[6].The experimental specimens were quenched at 850°C for 30min and tempered for 30min at 350C, 400°C, 450°C, and 520° in order to achieve various strength and microstructures, in the tempering process. Table 2 shows the heat treatment system involved in this work. All the experimental specimens are allowed for quenching for martensite structure which has high brittleness and tempering process has done for reducing the hardness of the specimens.[7-8]

Fig. 1 and Fig. 2 shows tensile test specimen for the analysis of mechanical properties and the notch tensile specimens for the analysis of the hydrogen-delayed fracture respectively. To conduct the tensile performance of the experimental material it is machined to ASTM E8 standard by using lathe machine. The main reason to use notch tensile specimen for conducting delayed-fracture test is the solubility of absorbed hydrogen is highest in areas which are under stress, especially in notched areas. [9-10]. Experimental specimens were allowed for hardness test using Rockwell hardness tester for all four tempering temperatures.

Notched tensile specimens were immersed in aqueous solution 0.1N Hydrochloric Acid (HCL) for diffusing hydrogen into the steel. Fig. 3 shows the experimental notched specimens immersed in HCL solution for 15min. Microstructural characterization of the fractured surface from the delayed fracture test specimens was analyzed by means of Scanning Electron Microscopy(SEM). If the fracture is due to delayed fracture susceptibility of the steel then it will show the intergranular fracture. The morphology of a fracture surface will vary based on the susceptibility of the material and the degree of embrittlement[11-10].

The delayed fracture test, i.e. a sustained tensile load is applied to the specimens after the injection of hydrogen. The applied stress ranges were varied to determine delayed fracture time characteristics. The use of the applied stress-ratio instead of the absolute value of the stress serves to evaluate the microstructure that has been designed to produce a given strength level.

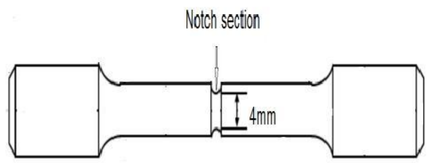


Fig- 2 Notch Tensile test specimen

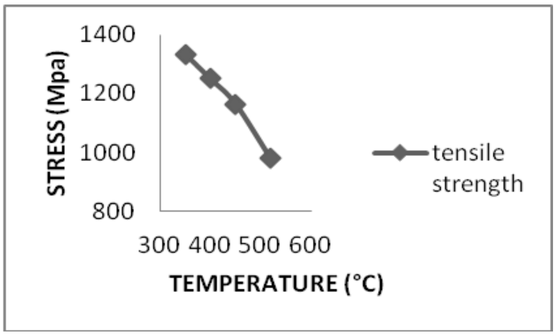
3. RESULTS AND DISCUSSION

3.1 TENSILE TEST

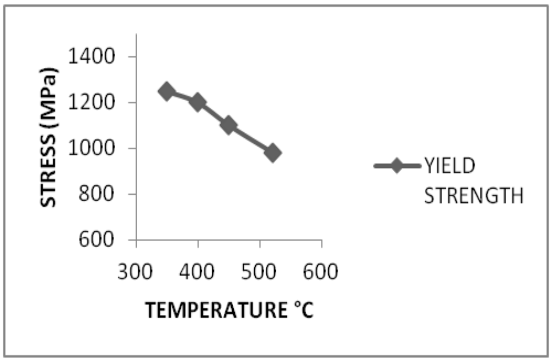
The effect of tempering process on the steel fastener over a range of temperature resulted in substantial changes in mechanical properties such as tensile strength and yield strength. The tensile strength and yield strength decreased with increase in the tempering temperature as shown in Table3.The tensile and yield strength of the steel is gradually decreased with increasing in tempering temperature. Graph has been plotted with results of tensile test as shown in Fig. 3 which shows a gradual decrease in tensile and yield strength.

Table-3 Varying mechanical properties of the AISI 4140 steel fastener after tempering process

Tempering Temp. [°C]	T.S [MPa]	Y.S [MPa]
350C	1333	1250
400C	1250	1200
450C	1165	1050
500C	980	900

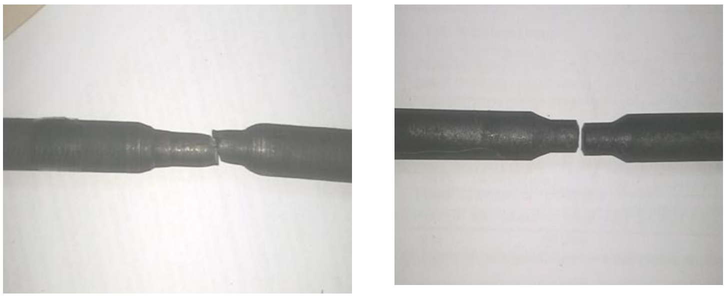


(a)



(b)

Fig-3 (a), (b) Mechanical properties with varying tempering temperature



(a) (b)

Fig- 5 (a), (b) Ductile and brittle fracture respectively

Fig. 4 clearly depicts the ductile and brittle fracture behavior of the tested material without and with hydrogen diffusion. In ductile fracture Fig. 4(a) shows the material is allowed for a plastic deformation before fracture. In brittle fracture Fig. 4(b) shows there is no any plastic deformation till its fracture.

3.2 HARDNESS TEST

Hardness test reveals that how much the tested material resistance to indentation or penetration. Hardness test was conducted for the experimental material to find its tempering temperature effect on the hardness property of the material. [13]. The Hardness value of the experimental material AISI 4140 steel fastener decreases with increasing tempering temperature as shown in Table 4. Reducing hardness of the experimental material helps significantly to reduce the susceptibility of hydrogen delayed fracture because high hardness materials are more susceptible to hydrogen delayed fracture. The Graph has been plotted for hardness value from the hardness test results Fig. 5 shows the relation between hardness and tempering temperature.

With the increase in the Tempering temperature, carbon decreases in the martensite because of carbide precipitation was significant, which led to a general phenomenon of reduced hardness of the base structure.

Table-4 Varying hardness value of steel with different tempering temperature

Samples at °C	Hardness number (HRC)
350°C	41
400°C	38
450°C	35
520°C	33

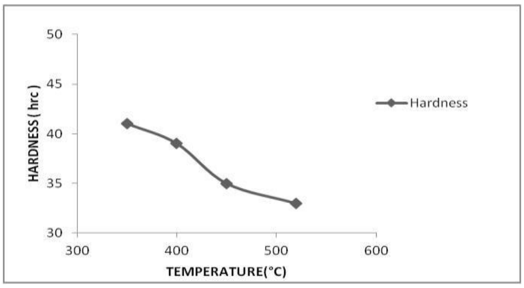


Fig. 5 Variation of Hardness at different tempering temperatures

3.3 DELAYED FRACTURE TEST

The constant loading test was carried out at the notch tensile test specimen in strength-to-stress ratio of 0.8-0.4 after the injection of hydrogen, and the time that it took for the specimen to fracture was measured.

The delayed fracture results are shown in Fig. 6 in terms of time to fracture as a function of applied stress ratio. The time to fracture decreased with increasing applied stress-ratio in HCL solution. With the increase in the tempering temperature, the time to fracture increased at all the stress ratio. Compared to tempering temperatures of 350°C, 400°C, and 450°C, at 520°C, the specimen took relatively long until fracture at a low-stress ratio, which was similar to the pattern shown in the results from the previous mechanical property analysis.

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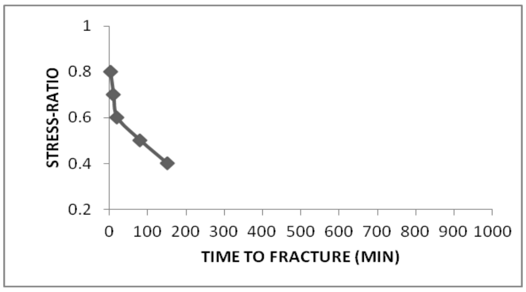
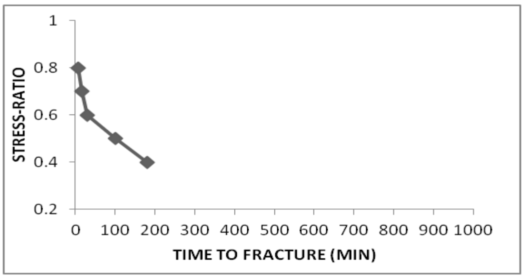
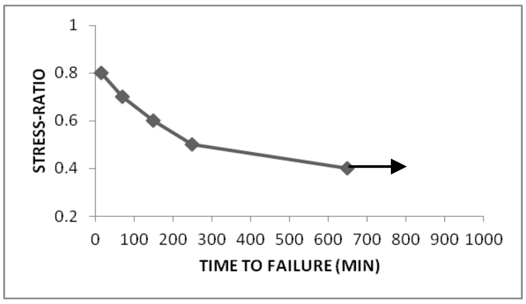


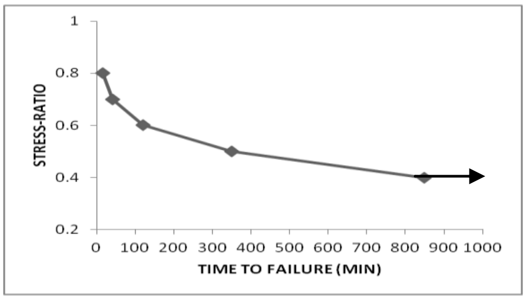
(a) At 350°C



(b) At 400°C



(c) At 450°C



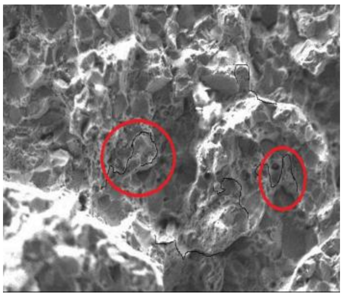
(d) At 520°C

Fig -6 (a), (b), (c), (d) Delayed fracture diagrams of the steels at different tempering

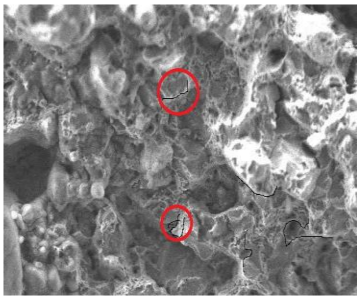
The arrows in the Fig. 6(c), (d) denote a non-fractured specimen at the elapsed time indicated. Compared to the tempering temperature of 350C, 400C, at 450C and 520C, the specimen took relatively long until fracture at a low-stress ratio, which was similar to the pattern shown in the results from the previous mechanical property analysis.

3.4 SEM RESULTS

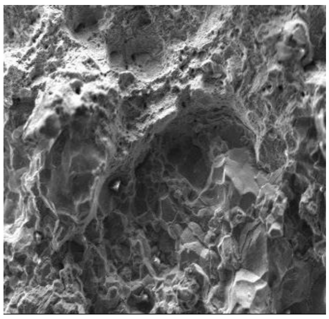
Toughness and tensile properties are the results of various steps leading to fracture, which appear in fractographic features. Usually, hydrogen delayed fracture surface shows intergranular fracture it can also be called as striations or quasi-cleavage fracture. [14]. From the results, we can find the influence of hydrogen diffusion and constant load test in the material at different tempering temperature.



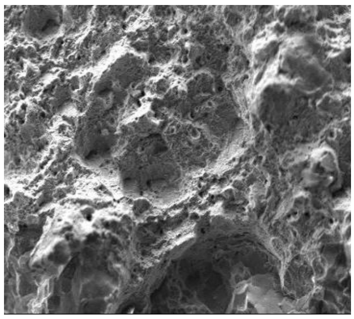
(a) At 350°C



(b) At 400°C



(c) At 450°C



(d) At 520°C

From the results, we can find the influence of hydrogen diffusion and constant load test in the material at different tempering temperature. Fig. 7 shows the SEM view of the fractured surface of the V-notch tensile specimen for the delayed fracture test after injection of hydrogen. The fractured surface of the hydrogen injected constant-loading test specimen displayed almost no plastic deformation but only a typical intergranular hydrogen delayed fracture. Specimens with 350°C and 400°C tempering temperature show more brittle fracture than the specimen with 450°C and 520°C which shows improved ductile fracture. Fig 7(a) and (b) shows a marked area of a brittle intergranular fracture where open grain boundaries are also present.

4. CONCLUSION

Experimental materials were allowed to tempering process. The tempering temperatures were 350°C, 400°C, 450°C, and 520°C and tempering time was 30min. The hydrogen was inserted into the steel using hydrochloric acid. The hydrogen delayed fracture susceptibility of quenched and tempered steel fastener 4140 was investigated under a constant tensile loading using circumferentially notched round bar samples. Mechanical properties of the tested samples were changed with the effect of tempering temperature. From the results, the following were concluded treatment has a clear effect on the delayed fracture susceptibility throughout

(1).One can see that tempering treatment has a clear effect on the delayed fracture susceptibility throughout the entire stress range.

(2).With the increase of the tempering temperature, the tensile strength and hardness of the steel were decreased.

(3).From the fractured surface analysis, it was observed that the fractured surface of the constant loading test after the injection of hydrogen showed an intergranular fractured surface typical of a hydrogen-delayed fracture.

(4).Overall, with respect to the mechanical properties, hardness and characteristics of microstructure, the optimum tempering temperature for AISI 4140 steel fastener used in this experiment were around 450°C to 480°C.

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