Effect of Heat Treatment on Corrosion Behavior of Spring Steels

Arun V K Mechanical Engineering Ilahia School of Science and Technology Cochin, India Roshith Raghavan Metallurgy and Materials National Institute of Technology Mangalore, India

Abstract: The experimental work deals with the effect of heat treatment on the corrosion behaviour of spring steels. In this study the heat treatments like hardening, normalizing and tempering were done for spring steels to obtain martensitic matrix, pearlitic structure and tempered martensitic matrix respectively. After heat treatment the microstructural studies were carried out for the samples using SEM. Hardness measurements were done. The corrosion behaviour of all heat treated samples in HCl at different concentration (1.5N, 2N and 2.5N) was determined using Tafel extrapolation technique. The variation in the corrosion rates due to the effect of heat treatment was noted. The results indicate that for fully martensitic matrix the corrosion rate is minimum and for pearlitic structure its maximum. As tempering time is increased the corrosion rate increases correspondingly. The corroded microstructural images were also taken using SEM and analysed.

Keywords: corrosion, spring steel, leaf spring, HCl, Tafel

1. INTRODUCTION

The word Corrosion stands for material or metal deterioration or surface damage in an aggressive environment. Corrosion is a chemical or electrochemical oxidation process, in which metal transfers electrons to environment and undergoes a valence change. Corrosion returns the metal to its combined state in chemical compounds that are similar to the ores from which metals were extracted. Corrosion of structural elements is a major issue for any industry because of the chemical environment of the chemical processing.

Today steel is the most important resource in this industrialized world. It forms the basic building material of today's structure. Moreover steels with large chromium and vanadium percentage can be used as spring steels which form the suspension system. Prevention of wear and increase in steel life depends principally on the design and operation on the component, but providing some pre-use treatment on steel can also improve the quality to a great extent. It has been seen that most of the study focuses on the experimental testing of the steel component and very few focuses on the material testing and improving its properties beforehand. One of the processing routes to alter the properties is heat treatment. Nearly 90% of the springs are used in heat treated conditions. The major requirement for the conventional spring steel is toughness, strength & hardness [11].

Increasing demands of automotive industry on performance improvement, weight reduction and cost savings place a lot of pressure on vehicle components, which require new design concepts and further material development. In this respect, weight reduction is very important as it reduces costs, but more importantly it reduces fuel consumption and CO_2 pollution. The biggest fuel consumers and polluters are trucks, where redesign and use of lighter high strength leaf springs can bring considerable benefits. Parabolic leaf springs used in suspension systems of truck front axles are usually made of two leaves, and serve two main purposes: support the weight of the trailer and provide the spring function in the suspension system. Improved strength of spring steel can be achieved through control of alloy composition, effective heat treatment, micro-alloying, thermo mechanical treatment and shotpeening The main objectives of current research work are to investigate the corrosion behavior of spring steel in acid medium, investigate the effect of concentration of medium on corrosion behavior of spring steel and investigate the effect of heat treatment on the corrosion behavior of spring steel.

2. EXPERIMENTAL PROCEDURE

2.1 Material Preparation

There are many grades of steels used for manufacturing of leaf springs like 9260, 4068, 4161, 6150, 8660, 5160, and 51B60. The leaf spring used in this experiment is 51B60.

The samples were cut from leaf spring and metallographically mounted using cold setting resin. This exposed flat surface of the mounted part was polished using 1/0, 2/0, 3/0, 4/0 grit level and finally disc polished using levigated Alumina, and etched using 2% Nital which was prepared by adding 2 Milliliter of Nitric acid to 98 Milliliter of ehtyl alcohol.

2.2 Medium

The corrosion studies were conducted in Hydro chloric acid solution of different concentrations viz. 1.5N, 2N and 2.5N.

The solutions were prepared from concentrated HCl(almost 32N). 1.5N HCl was prepared by adding 150ML of concentrated HCl to 850ML of distilled water. 2N HCl was prepared by adding 200ML of concentrated HCl to 800ML of distilled water. Finally the 2.5N HCl was prepared by adding 250ML of concentrated HCl to 750ML of distilled water.

2.3 Temperature

The corrosion studies were conducted at room temperatures.

2.4 Heat treatment

The samples were subjected to heat treatments like hardening, normalizing and tempering. The leaf spring of the automobile was first cut into rectangular pieces of area 1.1×1.1 cm² and height of 1 cm. One sample was taken and heated to 920^oC, holding at that temperature for 45 minutes for homogenizing in a resistance furnace. After 45 minutes the sample was taken out and air cooled to obtain Normalized structure for leaf

spring. Another sample was taken and heated to 920° C and after holding 45 minutes at that temperature it is immediately oil quenched to obtain Hardened microstructure for spring steel. Five samples were heated to 920° C and after holding there for 45 minutes the samples were oil quenched immediately. These samples were reheated to a temperature of 200° C. After one hour one sample was removed from furnace and oil Quenched immediately. That sample is tempered for 1 hour sample. Like this after each hour one sample is removed and oil quenched. So that we will get Tempered 1 hour, tempered 2 hour, tempered 3 hour, tempered 4 hour, tempered 5 hour samples.

2.5 Microstructural Examination

These heat treated samples were finely polished using 1/0, 2/0, 3/0, 4/0 grit level and finally disc polished using levigated Alumina, and etched using 2% Nital. SEM images were taken before and after corrosion for the samples.

2.6 Hardness Test

All the heat treated samples were subjected to hardness test. The hardness of the samples was determined using Rockwell c scale.

2.7 Method

Tafel polarization studies were carried out by using CH instrument's electrochemical analyzer and a three electrode cell. An area of 1.1×1.1 cm² of the polished leaf spring specimens were exposed to acid solution (1.5N, 2N and 2.5N) at room temperature. The polarization studies were made from -0.250V to 0.250V against open circuit potential (OCP) with a scan rate of 0.01V/sec and the corresponding corrosion currents, i, recorded. From the potential, E Vs log i plots, corrosion potential, E_{corr}, and corrosion current density, i_{corr}, were determined. The corrosion rate (C.R), in mpy, is calculated using the relation:

Corrosion Rate (mpy) =
$$0.129 \times EW \times i_{corr}/D$$
 (1)

Where, $i_{corr} = corrosion current density in \mu A/cm²$, D=density of the corroding material, 7.16 g/cm³, E.W=27.398g/mol,equivalent weight of corroding material (atomic weight/oxidation number). The experiments were repeated for the hardened, normalized and tempered (1-5 hours) samples. E_{corr}, i_{corr} and C.R for each experiment were determined.

3. RESULT AND DISCUSSIONS

3.1 Microstructural Examination

The spring steel samples were heat treated and the microstructures were viewed under SEM. The results of SEM are shown below.

By analyzing the scanning electron microscopy images of the hardened sample, the lath shape martensite formed due to the hardening process can be clearly seen. The samples were oil quenched after austenizing.



Figure 1. SEM images for hardened sample



Figure 2. SEM images of a normalized sample

Looking at the scanning electron microscopy images of the normalized sample, the layers of ferrite and pearlite can be clearly seen.



Figure 3. SEM images of a sample tempered for 1 hour

Looking at the scanning electron microscopy images of the tempered lhour sample, carbides are distributed along previous martensitic laths.



Figure 4. SEM images of a sample tempered for 2 hour

Looking at the scanning electron microscopy images of the 2 hour tempered sample, it is clearly seen that the along with martensite the carbides are also there but the intensity of carbides has increased as compared with samples tempered for 1 hour.



Figure 5. SEM images of a sample tempered for 3 hour

Looking at the scanning electron microscopy images of the 3 hour tempered sample, it can be clearly seen that the intensity of carbides is increasing as the tempering time is increased.

In figure 6, Looking at the scanning electron microscopy images of the 4 hour tempered sample, it can be clearly seen that the intensity of carbides is increasing as compared to samples tempered for 3 hour.



Figure 6. SEM images of a sample tempered for 4 hour



Figure 7. SEM images of a sample tempered for 5 hour

Looking at figure 7, it can be clearly seen that the distribution of carbides along the martensitic laths is very high. The coarsening of the carbides may occur along the ferritic grain boundaries.



Figure 8. Microstructure of the normalized spring steel after corrosion in 2.5N HCl



Figure 9. Microstructure of the hardened spring steel after corrosion in 2.5N HCl



Figure 10. Microstructure of the tempered 1 hour spring steel after corrosion in 2.5N HCl



Figure 11. Microstructure of the tempered 2 hour spring steel after corrosion in 2.5N HCl



Figure 12. Microstructure of the tempered 3 hour spring steel after corrosion in 2.5N HCl



Figure 13. Microstructure of the tempered 4 hour spring steel after corrosion in 2.5N HCl



Figure 14. Microstructure of the tempered 5 hour spring steel after corrosion in 2.5N HCl

The microstructures of the heat treated specimens of spring steel were observed. Microstructures are of martensitic

structures with some carbides at the grain boundaries, cabides may be occurred due to incomplete austanitization. After tempering for 1 hour the microstructure consist of tempered martensite and the carbides precipitates (epsilon carbides) are seen along the grain boundaries. As the carbon atoms are diffusing from the martensite to form the ε -carbides the overall hardness decreases as the tempering time increases. The main aim of the heat treatment is to modify the strength and toughness of the spring steels. Tempering of hardened steel reduces the brittleness or increase the toughness of the spring steel. So the tempering is selected in such a way to obtain the desired product required for automobiles.

3.2 Hardness Test

After the heat treatment the hardness is measured by using Rockwell c scale. The Figure 15 shows the decrease in hardness as we increase the tempering time. As expected the hardness of the hardened sample is high. The martensite formed due to heat treatment increases the hardness of the leaf spring sample. As we are tempering the sample the hardness of the sample goes on decreasing. This is due to the loss of carbon in the martensite due to formation of carbides.



Figure 15. Variation of hardness with tempering time

3.3 Results Of Corrosion Tests

The Tafel Plots were drawn for each heat treated samples for different normalities of HCl solution as shown below.



Figure 16. Tafel plot for the normalized sample. Plot is drawn for three normalities(1.5N,2N and 2.5N).



Figure 17. Tafel plot for the hardened sample. Plot is drawn for three normalities(1.5N,2N and 2.5N)



Figure 18. Tafel plot for 1 hr tempered sample. Plot is drawn for three normalities(1.5N,2N and 2.5N).



Figure 19. Tafel plot for the 2 hr tempered sample. Plot is drawn for three normalities(1.5N,2N and 2.5N).



Figure 20. Tafel plot for the 3 hr tempered sample. Plot is drawn for three normalities(1.5N,2N and 2.5N).



Figure 21. Tafel plot 4 hr tempered sample. Plot is drawn for three normalities(1.5N,2N and 2.5N).



Figure 22. Tafel plot for the 5 hr tempered sample. Plot is drawn for three normalities(1.5N,2N and 2.5N).

From the Tafel plots it can be seen that the corrosion rates are increasing with the increase in normalities of hydrochloric acid and also the corrosion rates increases with the increase in the tempering time. As we can see the i_{corr} increases with the increase in normalities.

The corrosion rates for the hardened sample is minimum and the corrosion rate of 5 hour tempered sample is maximum. This is because during hardening the microstructure formed is martensitic and as tempering is done carbides start precipitating from the martensite and martensite changes to low carbon martensite. Due to presence of carbides it is more prone to corrosion.

The table 4.2 shows the corrosion rates obtained for various samples. From the table it can be seen that the corrosion rate is maximum for the 5 hour tempered sample. It is evident from the table that with increase in tempering time and the concentration of HCl the corrosion rate in spring steel is increasing.

Sl no	Type of sample	Corrosion rates(mpy)		
		1.5N	2N	2.5N
1	Hardened	9.97	10.63	17.22
2	Tempered for 1 hour	10.35	12.76	21.09
3	Tempered for 2 hour	13.10	13.97	22.35
4	Tempered for 3 hour	13.92	16.10	24.65
5	Tempered for 4 hour	13.69	17.86	24.20
6	Tempered for 5 hour	11.65	20.72	37.05
7	Normalized	22.25	23.33	37.52

Table 4.2 Corrosion rate of heat treated samples

Fig.4.23 & Fig.4.24 shows the variation of corrosion rate with tempering time and variation of corrosion rates with increase in concentration of HCl.



Figure 23. Variation in corrosion rates with tempering time.

It can be also seen that corrosion rates are increasing with increasing concentration.



Figure 24. Corrosion rate with increase in concentration.

In a metal, anodic dissolution starts at the grain boundaries since they are amorphous regions and have high defect energy. Thus, the grain boundaries anodically dissolve as a function of time in an aggressive media having Cl– ions, and corrosion proceeds along the grain boundaries leaving the steel surface covered with corrosion products such as metallic oxides. In this case its inevitable that the original matrix loses weight. The grain boundaries rapidly react with aggressive Cl– ions and form pits. Therefore these regions can be seen as dark regions in the images of corroded sample [3].

As the time progresses, the amount of grain boundaries for anodic dissolution decreases and lath interfaces become new regions to be corroded. The ferrite phase which is having lower hardness behave as anode and cementite phase which is a kind of ceramic component behaves like cathode in corrosive media. In the case of hardened spring steel there martensite and in some cases retained austenite will also be there, the retained austenite phase act as anode and the martensite which is having a body centered tetragonal structure act as cathode. The corrosion occurs only when both anodic and cathodic reactions are simultaneously taking place. If any of the reaction is delayed the corrosion rate decreases. In hardened case the carbon is not available for the cathodic reaction to take place. So the corrosion rate is very low.

In the case of normalized spring steel there will be only ferrite and pearlite present in the microstructure. So the ferrite will act as anode and cementite in pearlitic structure eases the cathodic reaction so corrosion rate is high in the case of normalized steel compared to hardened and tempered case.

In the case of tempered martensitic steels consist of ferrite laths and micron sized iron carbides and nano sized alloy carbides at the boundaries of laths or within the laths. In this case the corrosion rates gradually increases corresponding to tempering time, this is because on tempering it will form low carbon martensite(metastable state) and Fe_{2.4}C and cathodic reaction takes place. As the tempering time increases the corrosion rate also increases.

4. CONCLUSION

The following conclusions can be drawn from the results obtained by experimental work:

- 1. The corrosion rate of spring steel increases with increase in concentration of corrosive media.
- 2. The hardened spring steel is having maximum corrosion resistance whereas the normalized sample shows least corrosion resistance.
- 3. The hardness of the spring steel decreases with increase in tempering time. This is due to loss of carbon in martensite.
- 4. The microstructure observation clearly shows that the corrosion is initiated in the grain boundaries and is propagated.
- 5. The corrosion of spring steel is increasing with increasing tempering time.

5. ACKNOWLEDGMENTS

We are grateful to Dr Jagatnath Naik, HOD, Department of Metallurgy and Materials Engineering NITK Surathkal Inda Inda, for his keen interest and support during the course of the investigation. Also the authors gratefully acknowledge the assistance provided by the Metallurgy department staffs of NITK surathkal India for carrying out heat treatments and corrosion experiments.

6. REFERENCES

- [1] ASM Handbook. 2005. Volume 1, spring steels, ASM International, Ohio.
- [2] ASM Handbook, 2005. Volume 13B, corrosion: Materials, ASM International, Ohio.
- [3] Atapek,S.A, Seyda Polat and Sibel Zor.2013. "Effect of Tempering Temperature and Microstructure on the Corrosion behavior of a Tempered Steel" Protection of

Metals and Physical Chemistry of Surfaces, Vol. 49, No. 2, 240–246.

- [4] Digges, T.G, Samuel, J. R, and Glenn ,W. G.1966. "Heat Treatment and Properties of Iron and Steel." U.S. Government Printing Office, Washington, D.C. ,12-15.
- [5] Fontana, M.G. and Greene, N. D.1978. Corrosion Engineering, McGraw Hill, USA.
- [6] Fontana, M.G.1987. "Corrosion Engineering" McGRAW-HILL Series in Material Science and Engineering, 39-198.
- [7] Gariboldi.E,Nicodemi,W,Silva,G. and Vedani,M.1994.
 "Mechanical Properties Of spring steels at room and low temperatures.",11-14.
- [8] Lee,C.S et al. 1998. "Microstructural influence on fatigue properties of a high-strength spring steel." J.Materials Science and Engineering A241,30-37.
- [9] Podgornik, B., Leskovšek, V., Godec, M. and Senčič, B. 2014.
 "Microstructure refinement and its effect on properties of spring steel." J.Materials Science and Engineering A599,81–86.
- [10] Rajan, T.V, Sharma, C.P and Ashok Sharma. 2011. "Heat Treatment: principles and techniques" PHI learning private limited, New Delhi. Heat treatment processes for steel, 94-104.
- [11] Sharma,S.S et al. 2013. "Effect of Heat Treatment on Mechanical Properties of AISI 4147 Spring Steel" 3rd International Conference on Mechanical, Automotive and Materials Engineering (ICMAME'2013) April 29-30,102-104.