Effect of Inter Critical Annealing on Microstructure and Wear Behaviour of En-8 Steel

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Abstract - The objective of the present work is to study effect of microstructure on abrasive wear resistance of EN-8 steel which were given inter-critical annealing heat treatment. The samples were heat treated to produce dual phased structure of hard martensite islands embedded in soft ferrite matrix. The results of the indicated that abrasive wear loss increased with decrease in hardness as well as increase in grain size of initial microstructure. The wear loss also increased with applied load. The phase analysis of wear debris revealed the presence of $\text{Fe}_2\text{O}_3$ indicating a tendency towards oxidative mechanism. The variation in wear loss with sliding length and applied load was correlated with microstructure of the material and distribution of phases.

Keywords - Heat treatment, Intercritical annealing, Abrasive wear, Microstructure. Martensite, Ferrite

I. INTRODUCTION

Steels containing hard martensite islands embedded in ferrite matrix are known as dual phase steels. For automobile and other engineering applications, these materials opened up a new dimension due to their formability and high toughness [1]. Low carbon dual phase steels have been touted as a potential material found application in making pipelines for transportation of mineral slurry and other wear resistant applications [2]. In a recent study, potential for use as farm implements where strength and wear resistance is of great concern was explored [3]. Because of their high strength-to-weight ratio and good formability, these steels show a great promise in automobile industry [4]. Despite these manifold applications, tribological properties of dual phase steel have not been studied thoroughly.

Jis and Toji et al have studied evolution of microstructure of dual phase steel with time for intercritical annealing cycle [5,6]. Guo et al have extensively studied effect of intercritical annealing on toughness of martensitic stainless steel [7]. Modi has worked comprehensively on effect of microstructure on high stress abrasive wear properties of plain carbon steel [8]. Abouei et al have studied oxidative wear mechanism for wear of dual phase steel to a great extent [9]. However, effects of prior microstructure on abrasive wear properties of dual phase steel, particularly that of hardened structure remains a grey area. The present investigation was carried out to examine initial microstructure of En 8 steel before intercritical annealing and an attempt was made to correlate the abrasive wear properties of dual phase steels with its microstructure.

II. EXPERIMENTAL PROCEDURE

En 8 steel samples were cut from a rod of 10mm diameter with a length of 25 mm using EDM. The heat treatment cycles involved subjecting the steel samples to annealing (furnace cooling), normalizing (air cooling) and hardening (oil quenching) treatments after austenitizing at 850 °C for 45 minutes. The samples were further subjected to intercritical annealing at 760 °C for 45 minutes. Table 1 shows the heat treatment cycles adopted in this investigation. Samples were polished using standard metallographic procedures prior to optical microscopy, hardness measurement and abrasive wear testing. For optical microscopy, specimens were etched using 5% nital solution. Hardness was measured by Vickers macro-hardness tester by applying a load of 30 kg.

Specimens were subjected to abrasive wear test using pin on disk abrasion tester using SiC abrasive paper (150 μm as abrasive. The abrasive wear tests were carried out with load of 29.42 N and 49.03N. The
sliding velocity was kept constant at 1.67 m/s. The abrasive paper was changed after sliding a distance of 500 m to eliminate the effect of change in morphology of abrading agent. The sample was cleaned before and after wear using ethyl alcohol. The weight loss was measured using digital electronic balance to an accuracy of 0.1 mg to measure the weight loss.

The load was varied to study the effect of load variation on wear loss and to calculate wear coefficient for each sample.

The initial samples and wear debris collected were subjected to XRD analysis in a PANalytical X’Pert Pro MPD diffractometer and phase analysis was carried out using X’Pert Highscore. The wear debris analysis was done to study mechanism by which wear takes place under given conditions.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heat Treatment</th>
<th>Phases Present</th>
<th>Hardness (HVN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Annealed</td>
<td>Austenized at 850°C and furnace cooled</td>
<td>Ferrite and pearlite</td>
<td>180.67</td>
</tr>
<tr>
<td>Normalized</td>
<td>Annealed and austenized at 850°C and air cooled</td>
<td>Ferrite and pearlite</td>
<td>185.67</td>
</tr>
<tr>
<td>Hardened</td>
<td>Annealed and austenized at 850°C and oil quenched</td>
<td>Martensite</td>
<td>347.67</td>
</tr>
<tr>
<td>IA1</td>
<td>Annealed, heated to 760°C and oil quenched</td>
<td>Ferrite and martensite</td>
<td>197.28</td>
</tr>
<tr>
<td>IA2</td>
<td>Annealed and austenized at 850°C and air cooled, heated to 760°C and oil quenched</td>
<td>Ferrite and martensite</td>
<td>211.58</td>
</tr>
<tr>
<td>IA3</td>
<td>Annealed and austenized at 850°C and oil quenched, heated to 760°C and oil quenched</td>
<td>Ferrite, martensite and tempered martensite</td>
<td>238.00</td>
</tr>
</tbody>
</table>

Table 1: Heat treatment schedule, phases and hardness of En 8 steel

III. RESULTS AND DISCUSSION

The annealed structure consists of relatively coarse grains of ferrite with coarse pearlite. The normalised structure consists of finer grains of ferrite with fine pearlite; the hardened structure consists of martensite. The intercritically annealed IA1 microstructure consists relatively coarse ferrite grains and martensite in a lower proportion. The intercritically annealed IA2 microstructure consists of finer ferrite grains and relatively more martensite as compared to IA1. The intercritically annealed IA2 microstructure consists of fine grains of ferrite, tempered martensite and martensite. The proportion of martensite was higher than IA2. It was noticed that martensite content depends to a great extent on microstructure prior to intercritical annealing.

Dual phase steels consisting of soft and ductile ferrite and strong and hard martensite were produced by heating. Initially heat treated En 8 carbon steels at temperatures between Ae1 and Ae3 (i.e. in the austenite + ferrite phase regions) for a predetermined period followed by quenching. This process of heat treatment is also called intercritical annealing. In this process, austenite transforms to martensite while ferrite remains unchanged. Ferrite attains some degree of super saturation of carbon because quenching enabling the ferrite to retain the same amount of carbon at room temperature as present at the treatment temperature [10].

As the temperature is raised above Ae1, ferrite (in case of annealed and normalised samples) or martensite (in case of hardened samples) begins to transform to austenite. New grains of austenite begin to form at nucleation sites which lie along grain boundaries. These nucleated sites grow further to form austenite grains. In case of annealed structure, as the grain boundary area is lower due to coarser grain size, number of nucleation sites is lower. Also austenisation is a diffusion controlled phenomenon, where alloying elements, especially manganese and silicon diffuse preferentially in a particular phase (Mn in austenite and Si in ferrite.) [5, 6] Hence as the nucleation sites are far apart and time required for growth will be higher as elements have to diffuse through a greater distance. Hence during austenisation, as nucleation sites are fewer and rate of growth is lower, for a particular soaking time and temperature, amount of austenite formed will be lower and grains would be coarser [11]. Hence, upon quenching a relatively lower amount of relatively coarser martensite would be obtained.

In case of normalised structure, due to lower grain size and higher grain boundary area, more nucleation sites are available and growth rate is higher. Hence martensite formed will be higher in amount and finer in nature. In hardened structure, correspondingly more and finer martensite will be formed. Presence of residual stresses in martensite assists in diffusion and hence
further increases growth rate. The untransformed martensite is tempered. Hence upon intercritical annealing, hardened sample showed three phases, namely ferrite, martensite and tempered martensite. This parent and newly formed martensite may be differentiated on basis of their response to etching treatment. It has been observed that due intercritical annealing, hardness of annealed and normalised samples increased due to introduction of martensitic phase in the structure, while that of hardened sample decreases due to tempering of parent martensite and introduction of ferrite phase in the structure. The effect of grain size on hardness as studied by Sieboda et al for polycrystalline materials which states that hardness of the material increases in accordance with the Hall-Petch Equation [12].

Figure 2 shows that annealed sample showed highest wear rate, followed by normalised, IA1, IA2, IA3 and hardened. It was also observed wear rates increased with increase in load applied. The wear resistance increased with increase in hardness, martensitic content and decrease in martensite colony size.

The testing methodology adopted ensured that the abrasive particles formed a groove of considerable depth in both harder and softer phases due to uniform motion of the disc and abrasive media being fixed with respect to disc. It can be observed that wear rate depends on abrasive’s ability, under given parameters, to scoop out and form a groove in harder material, i.e. presence of harder phase protects the softer phase in case of steel where the two phases adhere properly and the instances of ‘pull out’ of hard phase out of soft matrix are rare. Thus the depth of groove formed, and implicitly the wear loss from the material depends on both hardness as well as amount of harder phase i.e. martensite [8].

The abrasive wear rate [14] is given by:

\[ Q = \frac{(K \times l)}{h} \]

where, Q is the volume loss per unit sliding distance, l is normal load applied, h is hardness and K is wear coefficient. The wear coefficient K is given in Table 2.

For abrasive wear K was found to depend on fraction of displaced material removed \( f_{ab} \) and on the geometry of abrasive particles. It may be assumed that geometry of abrasive particles and frequency distribution of particles with respect to attack angle (θ) is constant within experimental limits as abrasive used is similar. However, \( f_{ab} \) changes significantly. This change is incorporated in the model proposed by Zum Gahr [14]; which defines wear loss as

\[ W = f_{ab} \times A_v \times d \]

where, W is volumetric wear loss, \( A_v \) is area of groove formed and d is depth of groove and \( f_{ab} \) is defined as

\[ f_{ab} = \frac{A_v(A_1+A_2)}{A_v} \]

where, A1+A2 are the total cross sectional area of material displaced to the sides of the groove. It can be envisioned that softer materials would have lower \( f_{ab} \) as it would rather undergo ploughing than cutting. However, softer material will also have a deeper groove as per aforementioned reasons. Also, as the geometry of abrasive remains more or less constant, shape and size of groove and hence it’s cross sectional area is almost constant. Hence, volumetric wear loss would vary as a combination of both \( f_{ab} \) and depth of groove.

The variation in above result may be explained in following manner. In annealed and normalised structures, due to higher ductility, ploughing would take place rather than cutting and hence \( f_{ab} \) would be low. However, increase in depth of groove due to lack of martensitic phase would dominate over lowering of \( f_{ab} \)and hence wear loss is high. On the other hand, in hardened structure, \( f_{ab} \) would be high and almost all the material displaced is removed. However, as depth of penetration is lower, material displaced itself is lower. However, in this case increase in \( f_{ab} \) dominates and hence wear coefficient is high. However, it is noteworthy that due to lower true area of contact, the wear rate for hardened sample is lowest. By intercritical annealing, both these parameters are optimised, i.e. in annealed and normalised microstructures, due to presence of martensitic phase depth of groove is reduced while in hardened structures, a ferrite matrix imparts ductility to reduce \( f_{ab} \). Hence intercritical annealing decreases wear coefficient.

However, it can be seen that effect of varying wear coefficient is offset by change in true area of contact. It can further be concluded that wear rate is more appropriate measure of severity of wear than wear coefficient.

Also it may be seen that as load applied increases, wear loss increases. This happens because as load is increased, true area of contact increases. Hence, grooves, which are formed by abrasive, have contact as a prerequisite. As contact area increases, number of abrasive particles interacting with the surface increase and so does average depth of groove. Due to this two concomitant effects, wear loss increases with increased load.

XRD analysis indicates presence of Fe2O3 in wear debris. This is an indication of a combined mechanism of cutting as well as oxidative wear. However, the critical oxide film thickness is very low and hence film breaks rapidly. This can be interpreted as inability of
oxide film to prevent wear loss due to high hardness of SiC (about 2400 HV.) This can further aid in explaining higher wear of softer specimen. It has been shown that a harder substrate is able to hold a thicker transfer layer of oxide more firmly as compared to a softer one. The reason is that a soft substrate shows lower mechanical support to hold the oxide layer due to larger change of plastic deformation [14, 15]. Hence, the critical thickness of the oxide may be lesser in a material of relatively lower hardness as compared to a material of relatively higher hardness; hence, there will be a higher probability the flaking off of this layer in the steel having a relatively lower hardness. Thus, a higher wear rate in materials of comparatively lower hardness may be attributed to the increase in the flaking off of the transfer layer during sliding. [9]

IV. CONCLUSION

1. The prior microstructure played an important role in determining the final microstructure after intercritical annealing of En 8 steel which influenced the abrasive wear behaviour of the material.
2. The decrease in initial grain size leads to decrease in abrasive wear rate of the material.
3. The extent of harder phase present and its size was dependent on initial microstructure of the material. An increase in martensite content as well as decrease in martensite size increases wear resistance.
4. The abrasive wear rate increases with applied load.
5. The tendency towards oxidative wear was indicated due to presence of Fe₂O₃ in wear debris. The abrasive wear mechanism was ploughing, cutting and oxidative wear.

REFERENCES