Effect of heat treatment on wear behaviour of rolled carbon steel in DOT4 brake fluid

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Abstract. The wear of heat-treated rolled carbon steels (HTRCSs) were performed under dry and wet sliding in DOT4 brake fluid is successfully investigated and reported in the study. The HTRCSs samples were obtained by heating to and soaking the as-rolled carbon steel (ARCS) at 950 °C prior to quenching or cooling in different media at different selected temperatures between (20 and 250 °C) and atmospheric conditions. The microstructures and wear track patterns of specimens were evaluated using SEM and high-resolution metallurgical microscope. The sliding wear behaviour and wear track patterns of the specimens impressed under 10N normal loads and reciprocating mode was used to assess the wear damages. The obtained wear properties were compared in air and DOT4 fluid environments using Anton Paar TRB tribometer. The results obtained showed that enhanced wear behaviours were obtained from the oil and water quenched samples as compared with the air and furnace cooled samples. The HTRCSs samples are more tolerable than as-rolled sample (ARCS) under dry sliding. However, beside the influences of the heat treatments, the lubricating effect of DOT4 is more pronounced in the results. The CoF ranges from 0.105 to 0.137 for wet sliding while higher CoF values (0.378-0.934) were obtained in the dry sliding.

1. Introduction

Apart many essential factors are considered by the automotive industry in a manufacturing of modern cars which include the high material strength-density ratio, reduced fuel consumption, safety and health enhancement and control of the dangerous exhaust gases [1]. Among various alloy, steel, cast iron and aluminium are widely used in making automobile automotive engine parts such as the engine blocks, cylinders and pistons of which the hydraulic brake cylinder is renowned. The high strength-weight ratio gives better advantage to aluminium alloy but very prone to wear problems and limitation to high temperature application. In contrast, cast iron and steel can withstand more aggressive application conditions. Rolled steel is famous in structural and industrial usage due to its combination of enhanced engineering properties controlled by several factors [2]. The heat treatment produces enhanced mechanical properties in steel alloys due to microstructural transformations [3-4]. Reinforced components can be made of multiphase structured steel alloys [5-6]. The displacement of the phase transformation proceeds with increasing Al, P and Si contents in the steel [7-8].
Until the recent time, reports were scarcely available on investigations involving wear and corrosion metal alloys such as (aluminium alloy) in mineral oils used for most hydraulic fluids (DOT3) and DOT4 fluids. Little explanations are made on wear theories of micromechanical surface contact [9] of aluminium alloy in hydraulic fluid. Thus, the current work is a fraction of the large study focusing on the alloys behaviour in hydraulic fluids (DOT3 and DOT4) of which some are available in literature [10-13]. In the present work, the heat treatment of carbon steel was done with the view to improving the hardness combined with toughness and strength for wear resistance hydraulic master brake application.

2. Experimental procedure

2.1 Material, Testing and Characterization

The 12 mm diameter low carbon steel specimen was received as rolled rod. Chemical analysis was done with Arc-Spectrometer (2000-3 Spetro-CJRO model). The chemical compositions of the bulk material and surface products were determined by Energy Dispersive X-Ray Spectrometer (EDS) facilities. The hardness test was perform using Innovatest - (Falcon 500 series) with dwell time of 10 sec on HV/0.2 Vicker’s scale.

2.2 Heat treatment process

In all cases, the as-rolled specimens (12 mm diameter by 10 mm thick) were heated to 950 °C and homogenize at same temperature for 2Hr 30mins after which the samples were withdrawn from the furnace. H2 was vigorously quenched in oil bath (SAE-40 grade) pre-heated to and maintained at 250 °C constant temperature and left in the oil bath for about 30 mins before finally removing it. H3 sample was quenched in preheated water (105 °C) and removed from the water after 30 mins. The quenched H2 and H3 attained the baths temperatures within short times without the baths losing much heat. H4 was left in the open air to cool to the room temperature for 40 mins after the removal from the furnace whereas H5 was cooled in the furnace for 70 mins. The specimens were washed in benzene. Table 1 and Figure 1 present the conditions for the heat treatment and the cooling regimes for the experiment.

| Sample | Cooling media/Temp. (°C) | Cooling time (min) |
|--------|--------------------------|-------------------|
| H1     | Control                  | -                 |
| H2     | Oil 250                  | 30                |
| H3     | Water 20                 | 30                |
| H4     | Air 20                   | 40                |
| H5     | Furnace 950–20           | 70                |

Table 1. Heat treatment cycles of rolled carbon steel.
2.3 Sample preparation and Metallographic examination
The samples were cut to 10 mm thickness using Brilliant 220, ATM, Germany model automatic precision cutter and the surfaces were rough ground with P320 SiC grit paper and with Dia-max 9 µm poly suspension on Aka-Allegan 9 plate; polished with 3 µm poly suspension on Aka-Moran plate and with 0.2 µm fumed silica on Aka-Chemal plate on automatic polishing machine (Saphir 550, ATM, Germany). The surfaces were water washed for 3 sec and in methylated spirit to remove water on the surfaces before drying in warm fanned air. The samples were swab in Nital etchant according to ASTM-E407-70 [14]. The wear track patterns were examined by Scanning Electron Microscope (SEM-Vega3 Tescan model) and high-resolution metallurgical microscope.

2.4 Tribological tests of heat-treated samples
The wet frictional tests were performed on H1-H5 samples immersed in DOT4 fluid fed into the tribometer cup. The tribometer was set operating in the reciprocating mode using 10N normal load rubbed via 6 mm alumina ball against the test specimen surfaces. The obtained CoF values were plotted against the sliding time. The extent of wear damages and wear products were assessed based on the CoF values and visual examination of the wear track patterns observed on microscopes, SEM with EDS facilities. The obtained CoF ($\mu$) values were compared under the dry and DOT 4 fluid environments.

3. Results and discussion
The chemical compositions and the micro-hardness of H1-H5 specimen are in Tables 2 and 3 respectively.

| Table 2: Chemical compositions of as-rolled carbon steel |
|----------------|----------------|----------------|----------------|----------------|
| %C            | %Mn            | %Si            | %P            | %S            |
| 0.357         | 0.63           | 0.101          | 0.016         | 0.015         |
| %Ni           | %Cu            | %Co            | %W            | %Fe           |
| 0.08          | 0.276          | 0.016          | 0.158         | 97.661        |

| Table 3. Micro-hardness tests of rolled carbon steel |
|----------------|----------------|
| Samples        | HV/0.2 (Average) |
| H1             | 168.74          |
| H2             | 181.29          |
| H3             | 289.88          |
| H4             | 158.49          |
| H5             | 115.68          |
3.1 CoF-sliding time patterns for H1-H5 under 10N load

The wear parameters of H1-H5 under the 10N reciprocating loading in dry and wet DOT4 fluid sliding are presented in Table 4. It relates the CoF with the track radius, wear cycle and sliding time.

Table 4. Wear parameters of steels under 10N load in dry and wet DOT4 sliding

| Samples | Radius (mm) | Cycle Slid time (s) | CoF | CoF max |
|---------|-------------|---------------------|-----|---------|
| H1_DOT4 | 3.99        | 1434                | 0.131 | 0.227  |
| H2_DOT4 | 11          | 5200                | 0.128 | 0.177  |
| H3_DOT4 | 10          | 5712                | 0.105 | 0.216  |
| H4_DOT4 | 7.63        | 7501                | 0.137 | 0.213  |
| H5_DOT4 | 2.51        | 22780               | 0.128 | 0.319  |
| H1_Dry  | 7.39        | 5997                | 0.934 | 1.118  |
| H2_Dry  | 11          | 4685                | 0.471 | 0.990  |
| H3_Dry  | 10.5        | 4906                | 0.510 | 0.762  |
| H4_Dry  | 4.62        | 12389               | 0.378 | 0.533  |
| H5_Dry  | 9.50        | 6028                | 0.741 | 1.118  |

Figure 2. CoF variation in dry and wet DOT4 fluid sliding

Table 3 presents the micro-hardness tests of H1-H5 steel samples. The result demonstrates the effects of heat treatments on the hardness of the steel samples. The effects of oil and water quenching were more obvious on the higher micro hardness values obtained for H2 and H3 as compared with air and furnace cooled heat treated H4 and H5 samples. The H2 and H3 hardness increased due to quenching whereas the H4 and H5 samples hardness reduced due to softening. The wear parameters including the CoF of H1-H5 samples subjected to sliding wear under 10N loading in reciprocating mode of dry and wet sliding in DOT4 fluid are compared Table 4. It was observed that for all the samples (H1-H5), the CoF have better steady state in DOT4 fluid than the dry sliding wear. In Figure 2, the CoF and CoF max obtained from the dry and wet sliding in DOT4 fluid are compared for all the H1- H5 steel samples. In all cases, the dry wear test presents H1 as most affected. The mean CoF increased in the order of H1 > H5 > H3 > H2 > H4 for the dry sliding wear and H4 > H1 > H5 > H2 > H3 in the wet DOT4 fluid sliding wear condition. The effects of the heat treatments make H2-H5 samples to be more tolerable than H1 under dry sliding. However, beside the influences of the heat treatments, the lubricating effect of DOT4 is more pronounced in the results (Table 4 and Figure 2). The CoF ranges from 0.105 to 0.137 for wet sliding while higher CoF values (0.378-0.934) were obtained in the dry sliding.
3.2 Optical microscopy and SEM of microstructures of wear tracks

Fig. 3 shows the OM of microstructures of H1 to H5 test sample surfaces rubbed with alumina ball under the 10N load in dry and wet DOT 4 sliding contact environments. The effects of heat treatments are observed on Fig. 3. It illustrates and compares the severities of the wear in the dry sliding as shown in Fig. 3(a)-(e) and wet sliding in Fig. 3(a1)-(e1) conditions.

In Fig. 4(a)-(f1) the SEM and EDS of the microstructures and wear tracks of H1-H5 are presented. The EDS compares the compositions of wear products observed on the steel surfaces after dry sliding wear. Adhesive wear with particles formed at the tip of the asperities is observed and seem to be more pronounced in dry sliding whereas abrasion is significant in the wet sliding in DOT4 fluid [15-17].

![Figure 3](image-url)

**Figure 3.** Optical micrographs showing the wear tracks of H1 (a, a1), H2 (b, b1), H3 (c, c1), H4 (d,d1) and H5 (e,e1) surfaces before and after wet sliding wear in DOT 4 fluid.
Figure 4. SEM/EDS of wear tracks of H1 (a, a1). H2 (b, b1). H3 (c, c1). H4 (d, d1) and H5 (e,e1) steels surfaces before and after dry sliding wear.
4. Conclusion
The following conclusions can be drawn from the results of the tests obtained: The heat treatments influenced the hardness of the steel samples. The oil and water quenching gave higher micro hardness values for H2 and H3 as compared with air and furnace cooled heat treated H4 and H5 samples. The H2 and H3 hardness increased due to quenching fine grained microstructures whereas the H4 and H5 samples hardness reduced due to softening. The CoF is in the order of H1 > H5 > H3 > H2 > H4 for the dry sliding wear and H4 > H1 > H5 > H2 > H3 for wet DOT4 fluid sliding. The heat treatments make H2-H5 to be more tolerable than H1 under dry sliding whereas the lubricating effect of DOT4 is more pronounced in the wet DOT4 fluid sliding results. The wear behaviour of H1 to H5 in Dot 4 brake fluid are largely depended on two wear mechanisms; adhesive wear and surface plastic deformation. The other important wear mechanisms such as spalling, and pitting were observed on dry samples. To conclude on the tribological behaviour of heat-treated steel in Dot 4 brake fluid environment, the selection of materials for this behaviour should mainly depend on the tribological sliding wear in dry and the lubricative wear. Surface fatigue resistance should be the major determinant for selecting a material for dry and lubricated wear.

5. References
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