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Characterisation and wear behaviour of rolled carbon steel in Dot 4 brake fluid

Olawale O Ajibola^{1,2*}, Ojo J Akinribide¹, Samuel O Akinwamide,¹ Peter A Olubambi¹

¹Center for Nanoengineering and Tribocorrosion, School of Mining, Metallurgy and Chemical Engineering, University of Johannesburg South Africa ²Materials and Metallurgical Engineering Department, Federal University of Oye-Ekiti, Nigeria

Corresponding author: olawale.ajibola@fuoye.edu.ng

Abstract. The wear behaviour of as-rolled carbon steel (ARCS) in DOT4 brake fluid is investigated and reported in this study. Sample was characterised using chemical analysis, hardness, impact and tensile strength tests. Arc-spectrometry and the Energy Dispersive X-Ray (EDS) facilities were used to ascertain the chemical compositions. The microstructures and wear track patterns of samples were evaluated using Scanning Electron Microscopy (SEM) and High-resolution metallurgical microscope. The wear of as-rolled carbon steel (ARCS) samples subjected to reciprocating motion of loads at room temperature and constant wear time were compared in air, DOT4 fluid and water. The frictional behaviour and wear track patterns of the specimens subjected to varied normal loads (3, 5 and 10N) under reciprocating sliding wear were employed to assess the wear damage of samples in the three different environments. The coefficient of friction (CoF) obtained tends to be in the order of $\mu_{DOT4 \text{ brake fluid}} < \mu_{water} < \mu_{air}$ comparatively for the three test environments.

1. Introduction

Apart from the aluminium alloys, cast iron and steels are wide recommended for the manufacture of automobile and automotive engine parts such as the engine master cylinders and pistons; master brake and sliding calliper and piston components [1]. To get adequate information on behaviour of materials selected for use as components of automobile and automotive engines; diverse and intensive material characterisation and testing may be applicable. These may include the microstructural analysis, thermal and mechano-chemical behaviour of the materials when subjected to diverse manner of stresses and chemical environments [2-4].

Some automobile and automotive engine parts are subjected to various challenges such as the moderately high range of temperature and friction as in the combustion chamber of the engine block, cylinder and pistons [5]. In braking systems of automobiles, constant rubbing of contact surfaces often results in mild or intensive wear. A study shows that strong adhesive amalgamation appears at the tip of the asperities when two surfaces come in contact [6]. Therefore, adhesive wear particle emanates from shearing the boundary interface caused by sliding [7]. The hydraulic braking system is most famous among the various automobile braking systems. Friction contacts are produced by application of braking force with and without transmission fluids [8]. More research reports are available in the recent times on the characterisation, properties, tribology and corrosion behaviour of aluminium alloys

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applicable to hydraulic brake cylinder pistons [9-10]. However, there is a lack of papers in the literature on the behaviour of steels used in similar service conditions and environments. The present work is a fraction of the extensive research work on the metal alloys - hydraulic fluids (DOT3 and DOT4) interactions, of which reports are available in public domains [11-18].

Wrought iron is a tough malleable, semi fused alloy of iron with fibrous slag inclusion, containing less than 0.08% C; suitable for forging and rolling in contrast to cast iron. Wrought carbon steel, also called the plain carbon steel is malleable with small Mn and some other elements. Rolled steel profiles of different shapes are produced by passing the stock through a single or multiple pair of rolls that reduces the thickness uniformly. The properties of the rolled products (mechanical, wear, weldability, corrosion) are much dependent on the interdependent set parameters which can be any or combination of rolling speed, rolling time and cooling rate. In a study by Adebayo and Ajibola [19], the influence of independent variables on the mechanical properties of rolled carbon steel was reported, while other studies investigated the influence of carbon content [20], cooling rate [21] and grain refinement [22] on the mechanical properties and microstructure of dual phase steels [23]. Steel rolling can be achieved hot or cold by similar processes, though the end products have different qualities. Thus the wear behaviour of as-rolled carbon steel (ARCS) specimen subjected to reciprocating motion of varied loads were compared in air, DOT4 fluid and water. The frictional coefficient and wear track patterns of the specimens under varied normal loads were used to assess the wear damages of the steel samples in the three selected environments.

2. Experimental procedures

2.1 Materials

The carbon steel materials used for the experiment were obtained from the light section rolling mill (LSM) of the Ajaokuta Steel Complex Ltd (ASCL) in Ajaokuta, Nigeria. The material was received as rolled 12mm diameter rods made from the as-cast 100 mm x 100 mm square billets. Arc-Spectrometer (2000-3 Spetro-CJRO model) and the Energy Dispersive X-Ray Spectrometer (EDS) facilities were used to determine the chemical compositions and it is presented in Table 1.

2.2 Sample Preparation and metallographic examination

The steel specimens were cut to 12 mm diameter by 10 mm thickness using the automatic precision cutting machine (Brillant 220, ATM, Germany). The surface was prepared to obtain even and flat mirror-like surface finish. The sample surfaces were water washed for 3 seconds and thereafter washed in methylated spirit to eliminate water stain on the surfaces before drying in warm shield air. The specimens were swabbed in Nital etchant containing 2 % vol. of Nitric acid and 98 % vol. ethanol solution (ASTM, E407-70) [24]. The microstructures were examined following standard procedures at different magnifications.

2.3 Mechanical testing and characterisation

2.3.1 Tensile test. Instron Universal tensile tester of type 3369 model was used to carry out the tensile tests. The test specimens were machined to the machine's specification in accordance with the ASTM standard. Each specimen was gripped at the two ends and then pulled until fracture and the ultimate tensile strength was recorded on the panel.

2.3.2 Impact test. The impact energy was determined by using Tinius Olsen 84-3 Model, Impact tester [18]. The test piece of a square cross-section of 10 mm dimension and 50 mm long, notched at the midpoint was used. The specimen was placed in a vice as a beam fixed at the two ends. The pendulum hammer of 75 kg weight was raised, on releasing; the specimen was hit behind the V-notch. The energy absorbed was read from the dial scale mounted on the machine and computed as a difference between the initial height h_i and final height h_f as the measure of the energy absorbed.

2.3.3 *Micro-hardness test.* The InnovaTest-(Falcon 500 model) was used for the hardness test. Three-point micro-indentations were made on the surface of the sample at regular interval of 3 mm distance apart. The dwell time was 10 s on HV/0.2 Vicker's scale.

2.3.4 Tribological tests. These sliding wear tests were carried out in two different environments. In the case of wet sliding tests, 20 ml of the fluid (DOT4 or water) is fed into the tribo cup, while the asrolled carbon steel samples were immersed into the fluid (DOT4 brake oil or water) at atmospheric temperature of 25 °C and 50 % humidity. The tribometer was run in the reciprocating mode; sequence count of 10, frequency of 10 [Hz] with an acquisition rate of 100.0 [Hz] and fixed at a stop condition of 3 m. Three normal loads (3N, 5N and 10N) were applied via 6 mm alumina ball used to rub the surface of the rolled steel samples. The wear experiments were prepared, performed, analysed and reported considering the ASTM G 115-04, G 40-02; G 99 and G 133, DIN 50324, and ASTM G 99 - 95a. The CoF profiles were plotted against the sliding time. For clarity and easy view of reciprocating analysis, the CoF-sliding time curve on the two-way extraction profile was size filtered to 50 percent. The wear behaviour is evaluated based on the CoF (μ) obtained and compared in the three test environments.

2.4 Microstructural evaluation

The microstructures and wear track patterns of samples were evaluated using Scanning Electron Microscopy (SEM) (Vega3 Tescan model) and high-resolution metallurgical microscope (Zeiss MTB 2011 model). The frictional behaviour and wear track patterns of the specimens subjected to varied normal load under reciprocating sliding wear was employed to assess the wear damages of samples in the three different environments.

3. Results and discussion

The chemical compositions, mechanical properties and the three-point micro-hardness tests results of the as-rolled steel samples are shown in Tables 1-3.

| Elements | Cr | Со | Р | Cu | Si | С |
|----------|------|-------|-------|-------|--------|-------|
| Amount % | 0.69 | 0.016 | 0.016 | 0.276 | 0.101 | 0.357 |
| Elements | Mn | S | Ni | W | Fe | |
| Amount % | 0.63 | 0.015 | 0.08 | 0.158 | 97.661 | |

Table 1. Chemical compositions of as-rolled carbon steels samples.

| Table 2. Mechanical | properties of as-rolled | carbon steel samples. |
|----------------------------|-------------------------|------------------------|
| Lubic 1 Hiteenamean | properties of as follow | earboin breer samples. |

| Properties | Value |
|--|--------|
| Tensile | 673.18 |
| Strength, TS $_{@maxL}$ (N/mm ²) | |
| Tensile Strength, $TS_{@Break}(N/mm^2)$ | 494.83 |
| Yield Strength (N/mm ²) | 486.31 |
| Hardness (HV) | 168.74 |
| Impact Strength (N/m) | 100 |

| Sample: H1 | P 1 | P 2 | P 3 |
|----------------------------|------------|-----------|--------|
| HV/0.2 | 179.4 | 163.5 | 163.5 |
| Analysis: Max 17 | 9.4, Min 1 | 63.34, Av | verage |
| 168.74 | 4, Std.dev | 7.53 | |
| HBS500 | 155.4 | 141.8 | 141.7 |
| MPa | 607.2 | 552.3 | 551.8 |
| Indentation d ₁ | 0.0483 | 0.0484 | 0.0477 |
| (mm) | | | |
| Indentation d ₂ | 0.0427 | 0.0468 | 0.0476 |
| (mm) | | | |

 Table 3. Three-point micro-hardness tests of rolled carbon steel sample.

3.1 CoF-sliding time patterns for ARCS under 3, 5 and 10N reciprocating loads

Table 4 and Figure. 1 illustrate the wear parameters of ARCS samples under the action of 3, 5, 10N reciprocating load in air, DOT4 fluid and water. There are variations in the COFs obtained from the test with respect to the sliding test parameters such as the normal applied load, track radius, slid time or cycle and wear environment/lubrication medium.

The CoF (μ) obtained tends to be the order of μ_{DOT4} (0.112~0.146) < μ_{water} (0.304~0.834) < μ_{air} (0.423~0.934) comparatively in the three test environments. Maximum COF values of 1.214, 1.118 and 0.242 were obtained from the test with water, air and DOT4 fluid respectively.

The graphical representations to compare CoF variation with applied normal loads in air, water and DOT4 fluid are presented in Figure 1. The CoF and CoF maximum values obtained in the sliding wear contacts are compared with respect to the three applied normal loads (3, 5 and 10N) and sliding environments (air, DOT4 fluid and water).

Generally, it is observed from the results (Figure 1) that the as-rolled carbon steel sample has tolerable friction behaviour in the three tested environments. Nonetheless, water shows less lubricating characteristic than the oil.

For the dry air sliding, the CoF fluctuates with increased applied load; it increased with load increase from 3N to 5N; thereafter diminished to a lower value. Increase in frictional coefficient for dry air sliding can be attributed to absence of surface wetting films. Whereas dissimilar CoF behaviour was observed in the wear analysis carried out in DOT4 environment. Decrease in frictional coefficient can be ascribed to resistance to wear resulting from the load bearing capacity of the film formed on the sample surface during wear. Sliding in water environment showed a clear trend of CoF increase as the load increased from 3 to 10N. Increament in CoF value can be as a result of deeper penetration due to increase in applied load. Further observations show a rapid decrease in frictional coefficient in ARCS analysed at various loads in DOT 4 environment in comparison to analysis carried out in dry air and water environments. ARCS have highest CoF in water as compared with air and DOT4 at 3N. Dry sliding in air gave highest CoF among the water and DOT4. Similar result was observed in a study by Duan et al. [25].

| Table 4. Wear param | eters of ARC | S under 3, 5, | 10N reciprocation | ng load in aiı | r, DOT4 and w | ater. |
|---------------------|--------------|---------------|-------------------|----------------|---------------|-------|
| | | | | | | |

| Load | Media | Cycle | Slid | CoF | CoF |
|------|-------|-------|------------|-------|-------|
| (N) | | | time | | max |
| | | | <i>(s)</i> | | |
| 3 | Air | 7059 | 716 | 0.770 | 0.923 |
| 3 | DOT4 | 8168 | 816 | 0.112 | 0.228 |
| 3 | Water | 7147 | 714 | 0.834 | 1.214 |
| 5 | Air | 4088 | 408 | 0.423 | 0.852 |
| 5 | DOT4 | 8412 | 841 | 0.146 | 0.242 |

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| 5 | Water | 9529 | 953 | 0.332 | 0.789 |
|----|-------|-------|------|-------|-------|
| 10 | Air | 5997 | 540 | 0.934 | 1.118 |
| 10 | DOT4 | 14345 | 1434 | 0.131 | 0.227 |
| 10 | Water | 14294 | 1429 | 0.304 | 0.627 |



Figure 1. CoF variation with applied normal loads in air, water and DOT4 fluid.

3.2 Microstructures and wear patterns

Figure 2. shows the SEM and EDS of microstructures of ARCS test sample surface. Figure. 3. show the optical micrographs before and after dry sliding wear, while Figure. 4 are the SEM images of wear profile of ARCS impressed with alumina ball under the 10N load in air, water and DOT 4 sliding contact environments. The severities of the wear in the three environments are revealed in the images in Figures 3 and 4 as compared with Figure 2. Figure. 3c showed mild wear effect of wear in oil as compared with dry sliding (Figures. 3b and 4a) and wet sliding in water (Figure 4a). In Figure 3 the (a) microstructures and (b) dry sliding wear track of ARCS are presented. Strong adhesive union was formed at the tip of the asperities when two surfaces of ball and test sample are contacted and subsequently, adhesive wear particles were formed by shearing the interface caused by sliding in accordance with Akinwamide et al. [26]. In contrast, the strong abrasion is formed at the surface and region of contact between two load-bearing flat surfaces when ball meets the ARCS surface in water (Figure. 4b) and in oil (Figure. 4c) [7, 15-16]. Thus, abrasive wear particle is produced at the interface initiated by sliding.



Figure 2. showing (a) SEM and (b) EDS of microstructures of ARCS.



Figure 3. showing optical micrograph of (a) microstructures and (b) dry sliding wear track of ARCS.



Figure 4. SEM showing wear tracks of ARCS under the (a) dry air (b) water and (c) DOT 4 sliding contacts.

4. Conclusion

The following conclusions are drawn from the results of the tests obtained; the CoF results show that the as-rolled carbon steel sample has the most tolerable sliding friction behaviour in the DOT4 fluid of all the three tested environments. Wear is most severe under dry sliding in air with strong adhesive union formed at the tip of the asperities when two surfaces are contacted. It is demonstrated that strong abrasion was experienced at region of contact between load-bearing ball with the ARCS surface sliding in water and DOT4 fluid. Notwithstanding, mild abrasive wear was experienced on the ARCS surface in oil.

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