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Friction and wear properties of black oxide surfaces in rolling/sliding contacts

Carl H. Hager Jr.*, Ryan D. Evans

The Timken Company, North Canton, OH 44720, USA

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ABSTRACT

Black oxide chemical conversion treatment of steel surfaces was originally developed as a low-cost method to improve the corrosion resistance of the base material. Over time, this surface treatment was introduced on tribological components amid claims that it improved galling and smearing resistance. In rolling element bearings, smearing damage can occur during high transient slip events between roller bodies and raceways, and black oxide surface treatments have recently been prescribed as a solution for this mode of adhesive wear damage. Therefore, fundamental friction and wear experiments using uni-directional sliding and rolling/sliding contacts were conducted to explore the performance of black oxide surfaces in tribological contacts.

From this work, it was concluded that mated black oxide surfaces yield similar or lower friction as compared to mated steel surfaces (in lubricated contacts), exhibit high wear in low λ conditions, and can mitigate adhesive wear damage as long as the oxide layer remains intact.

1. Introduction

The service life of a rolling element bearing is often limited or reduced by component damage or material fatigue. Component damage can result from wear, external contaminant ingress, severe application conditions, neglect, or misuse. One particular type of life-limiting bearing damage is called “smearing”. Smearing is a term that is often used to describe adhesive wear damage in rolling/sliding contacts when sliding is substantial and the lubricant film thickness is insufficient to adequately separate the contacting surfaces [1]. Damage associated with smearing may occur when rolling elements slide along the raceway surface as they enter or exit the loaded zone [2]. In some cases, this type of damage can be mitigated through component and/or system design. However, there are some instances where the damage cannot be avoided without modifying the tribological contact surfaces.

If smearing damage cannot be avoided from a component or system design approach, another way to mitigate adhesive wear damage is to change the chemical composition of one or both of the surfaces in contact. In 1978, Gregory wrote a paper providing a brief summary of conversion coatings available to improve adhesive wear resistance [3]. Gregory suggests that black oxide surfaces (when oiled or waxed) can yield low friction and resistance to adhesive wear damage. In 1998, Philström and Ström published a master’s thesis where black oxidizing was applied to spherical roller bearings (Part number 222 22E/C3 – 110 mm bore diameter – 200 mm outer diameter) [4]. In their work, the authors conducted smearing tests in bearings by periodically stopping the rollers while the shaft was still rotating. This was done for a total of 100 cycles at eight different shaft speeds (100, 200, 400, 800, 1600, 2400, 3200, and 4500 rpm). If no smearing damage was found on the bearings after each shaft speed, the shaft speed was raised to the next level. Upon the completion of this testing with and without black oxidized rollers, Philström and Ström found that black oxidized rollers yielded a small improvement in smearing resistance as compared to uncoated rollers [4].

A few years later, Scherb and Zech developed a smearing test using cylindrical bearings from which the authors concluded that black oxide can prevent smearing damage and reduce frictional torque by up to 20% [5]. In 2010, Mihailidis et al. conducted smearing tests with a two-disc machine where the entrainment velocity was held constant and the slide-to-roll ratio was increased until severe adhesive wear occurred between the mated samples [6]. These tests demonstrated that the black oxidized discs could survive higher levels of slide-to-roll ratio compared to the untreated discs. In addition, tests were also conducted with a constant slide-to-roll ratio and demonstrated that the black oxidized discs exhibit higher wear rate and lower friction as compared to the untreated discs during a wear-in cycle.

Although the aforementioned work found that black oxide surfaces yield some benefit in reducing adhesive wear or smearing

* Corresponding author. Tel.: +1 234 262 2077; fax: +1 234 262 2282.
E-mail address: carl.hager@timken.com (C.H. Hager Jr.).

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damage compared to untreated surfaces, in 2013 Evans et al. concluded that black oxide treatment of rollers and raceways did not provide additional smearing protection in cylindrical roller bearing tests conducted under severe conditions [7]. In this work, the authors conducted bearing tests where the load zone was shifted 180° every 15 s. In three separate tests, the bearings with black oxidized raceways and rollers exhibited smearing damage after just one load cycle. Bearings that were untreated with ground raceways exhibited smearing after just one load cycle as well. This conclusion seems contrary to the previous bearing tests by Scherb and Zech [5] and the two-disc experiments by Mihailidis et al. [6], but seems to agree with the spherical bearing tests conducted by Philström and Ström [4].

Although there are conflicting results in the literature (which may be due to the severity of the testing), black oxide surface treatments have recently been prescribed as a solution to mitigate various types of damage in rolling element bearings [8]. In addition, the literature also suggests that there may be other benefits to using black oxide such as low friction and high wear rates during break-in [5,6]. Therefore, the focus of this work was to conduct bench-level tribological experiments to determine the friction and wear properties of black oxide surfaces in rolling/sliding contacts. Several different experimental techniques and results are described in sequence and then drawn together with discussion and general conclusions.

2. Black oxide

Although black oxide or black oxidizing can be used to describe a number of surface coatings on steel, the term "black oxide" in this work refers to the conversion of a steel surface to Fe_3O_4 or magnetite using a hot alkaline bath. This type of black oxide surface is created using a process that was developed in Germany in the early 1900s and has been used to produce an attractive black finish, impart moderate corrosion resistance (with the addition of secondary oil impregnation or wax), enhance lubricity, and resist galling [9]. It should be noted that the extent of these claimed benefits rely on a secondary process of oil or wax impregnation. In fact, many vendors state that the anti-galling properties of black oxide surfaces allow for the outer layer to be sacrificed during initial contact. Therefore, the black oxide layer is galling resistant in that it wears away without adhesive wear damage to the mated surfaces.

The black oxide layer is generated by placing the steel components into a hot aqueous alkaline nitrate bath. The concentration of the bath is controlled by boiling the solution at a desired temperature between 285 °F (141 °C) and 310 °F (154 °C) [10]. This can be done using either a single-bath or double-bath process. The single-bath process typically uses one tank with a temperature range from 285 °F (141 °C) to 290 °F (143 °C) [10]. The double-bath process typically uses a second tank with a temperature range from 305 °F (152 °C) to 310 °F (154 °C) [10]. Depending on the process, the black oxide layer thickness can range from 0.5 to 3 μm. A common standard for defining black oxide surface treatments on ferrous materials is DIN 50938.

Although the black oxide process is well documented, there is comparatively less data published on the properties and microstructure of the black oxide surface layer. In this work, a through hardened AISI 52100 steel disk (HRC 58-62) was metallographically polished and then black oxidized using a double-bath process. The microstructure of the black oxide surface layer was analyzed using a focused ion beam to mill a trench followed by inspection of the cross section with a high-resolution scanning electron microscope (SEM). Fig. 1 shows a secondary electron SEM image of the black oxide surface layer on the AISI 52100 steel disk.

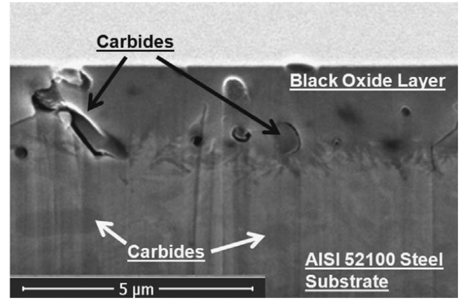

Fig. 1. High-resolution scanning electron microscope image of black oxide surface layer.

Table 1
Nanoindentation mechanical property comparison for untreated AISI 52100 steel and black oxide layer [11].

Sample	Avg. hardness (GPa)	Depth range for avg. hardness (nm)	Avg. elastic modulus (GPa)	Depth range for avg. modulus (nm)
Untreated AISI 52100 steel	10.6 ± 0.5	100–200	230 ± 10	100–200
Double-bath black oxide	2.6 ± 0.3	200–300	61 ± 5	80–100

The oxide layer itself is dense, but there are pre-existing carbide particles contained within the microstructure that exhibit voids at the boundaries between the carbide and oxide materials. Measurements of the coating thickness varied from ~1.4 to 2.2 μm for this sample. In comparison, Evans et al. [11] published a transmission electron micrograph of a single-bath black oxide surface cross section. The view shown has a thinner (200–500 nm) black oxide layer that did not exhibit voids at the carbide/oxide interface like those observed for the double-bath layer in Fig. 1.

The mechanical properties of the black oxide layer were measured with a nanoindenter using the continuous stiffness technique [12] and a Berkovich tip. The indentation parameters included a depth limit of 2500 nm, a strain rate of 0.1 s^{-1}, a harmonic displacement target of 1 nm, a frequency of 45 Hz, and an assumed Poisson's ratios for black oxide (0.25) and untreated steel (0.3). Fifteen replicate tests were performed on each sample. In addition to measurements on the black oxide surface, a second through hardened AISI 52100 steel disk was metallographically polished and analyzed along with the black oxidized sample. The measurement results from both surfaces are summarized in Table 1. Based on these measurements, the black oxide surface layer has approximately 25% of the hardness and 27% of the modulus of the base steel surface. These results were also published by Evans et al. in 2014 [11].

3. Experimental details

All sliding and rolling sliding wear experiments were conducted using a Wedeven Associates Inc. manufactured WAMsc4 test machine. This machine is able to run block-on-ring experiments with a stationary block and a rotating cylinder, and can also perform rotating cylinder-on-cylinder or rotating ball-on-cylinder rolling/sliding wear experiments. The machine has two opposing spindles that can be controlled independently. One of the spindles is mounted on three load cells and an actuator that allows for it to move toward the opposing spindle in the contact plane. Moving one spindle toward the fixed second spindle allows for a normal load to be applied between the test surfaces while the spindles are