

PURE Award Final Report

Carolyn Pethrick

Award Term: June – August, 2018

Introduction

This project investigates the effectiveness of boride coatings on the wear and friction properties of carbon steel. In particular, the behavior of coated steel in corrosive environments was a key focus of this project. Boride-coated steel is used in many applications, such as manufacturing equipment, and oil extraction (Fichtl, 1981; An, 2008). The industry partner which supplies the coated samples has a focus on industrial applications in the oil sands. Low-friction and wear-resistant parts are important for their resilience. Corrosion resistance is also important, as metal parts need to withstand corrosive environments such as dirty and salty water. In industrial applications, replacing a worn part means loss of profit while a plant is down. Innovating and refining steel coatings is highly sought after by oil companies.

Tribology is the study of friction and wear. Methods of this field can be applied to examine the effectiveness of metal coatings supplied by our industry partner. The Egberts group at the Schulich School of Engineering Department of Mechanical Engineering recently developed a home-built tribometer capable of performing linear reciprocations across the surface of a metal sample while applying a constant normal force. This tribometer is controlled by software developed in LabVIEW, which can be customized with relative ease. Corrosion and tribology merge in the field of tribocorrosion, where electrochemical techniques are used to examine corrosive wear while mechanical wear is examined using tribological methods.

The aim of this project was to design a container to perform reciprocations while taking measurements of the material's corrosion rate. Software developed in LabVIEW was modified to control the apparatus and collect data. The proposal also discussed possibilities of collecting data for the tribocorrosion experiments if time allowed. During the PURE award term, the scope of the project expanded due to limitations imposed by equipment availability and delays in manufacturing the corrosion-testing container. The additional tasks included refining and developing data collection techniques for dry testing of the coatings, such as surface imaging and processing.

Design Process

Corrosion-Testing Container

The tribocorrosion experiments had several requirements which guided the design of a container for the corrosive liquid. The container must hold liquid, a 1"x2"x1/4" coated sample, and two electrodes. The assembly would move along a single axis, on top of an existing stage moved with servo motors. First and foremost, the new container had to be compatible with the stage of the dry-testing apparatus, and prevent the costly force-torque sensor from getting wet. Samples need to be mounted, held, and removed without

damaging the wear track. In order to make meaningful measurements, a reference electrode and platinum mesh had to be suspended in the solution. The reference electrode provides a comparison point for potentials to be measured against. A silver-silver chloride electrode was chosen for the reference electrode because of ease of storage. Because current must flow to complete the circuit, an inert platinum mesh should be suspended in the solution as a counter electrode.

The design went through six iterations: Figure 1 shows the final design. The container sits on top of the sample, with a space for the reservoir above the sample. The container is fastened to the plate screws. An O-ring is held in place between the container and sample to ensure the assembly is watertight. Acrylic was chosen for the container because of its strength and chemical inertness to salt solutions.

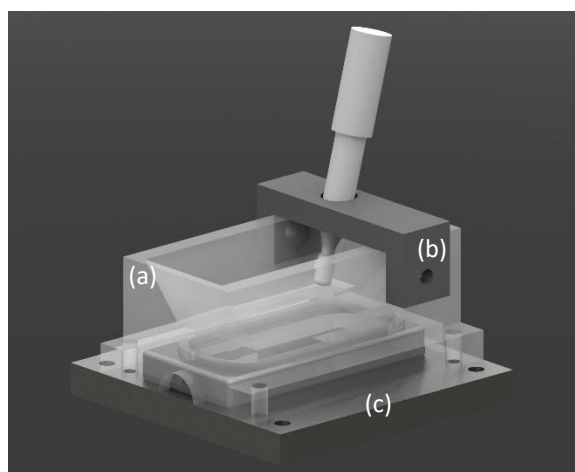


Figure 1. The design for the corrosion-testing apparatus and the manufactured apparatus. The designed components are (a) the container, (b) the reference electrode clip, and (c) the plate for mounting on the existing stage.

LabVIEW Software Modifications

The existing software controlled the servo motors which move the stage and used feedback from the force-torque sensor to maintain a constant normal force. It also recorded data so that the friction coefficient could be calculated.

The software had to be heavily modified to carry out tribocorrosion experiments. Code modifications allowed measurements from the potentiostat, which externally controls the applied potential, as well as the force-torque sensor. The modified software first waits for the metal to reach equilibrium, lowers a ruby sphere to the metal. The sphere moves across the surface of the metal a specified number of times, then stops movement as the metal returns to equilibrium. During reciprocations, the software uses feedback from the force-torque sensor to ensure that the normal force is constant for reliable results. The applied current and the forces on the sample allow for quantification of corrosion and wear behavior.

Results

Surface Profile Measurements and Data Collection Techniques

A Zeta-20 surface profilometer was used to characterize the wear track after reciprocations across the surface of a sample. During a friction test, a ruby sphere moves back and forth across a metal sample, forming a groove. Ultimately, the goal was to calculate the amount of volume lost. This method characterizes how well a material resists wear. The surface image and height profile of a coarsely-polished sample is shown in Figure 2. A MATLAB script was used to calculate the negative wear, where the ruby sphere had worn away material, and positive wear, where material had built up due to deposition of material or stresses due to applied forces in the friction test.

All of the images taken with the Zeta-20 profilometer were of samples tested in dry conditions. The same methods will be applied in the future to samples used in tribocorrosion experiments.

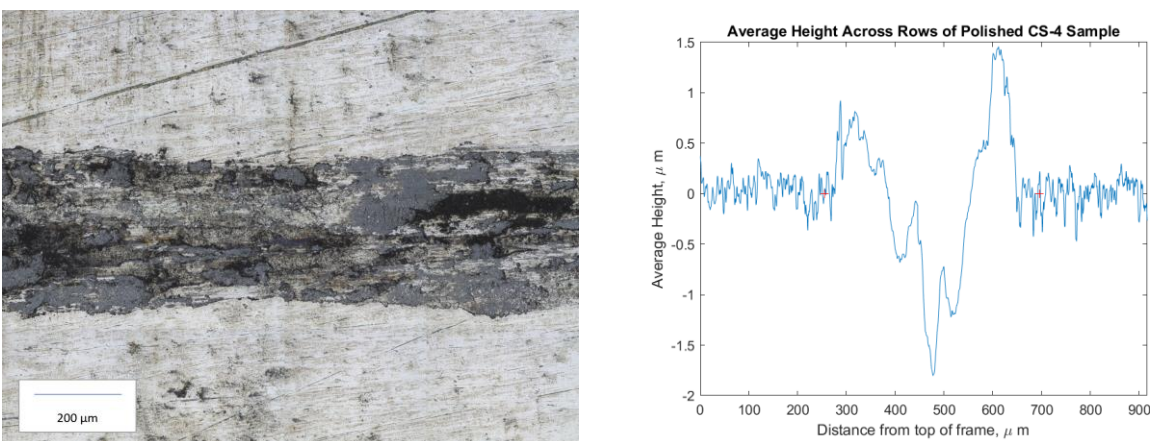


Figure 2. The optical image of the surface of the polished metal sample and the corresponding average height profile across the wear track.

Tribocorrosion Experiments

I aimed to conduct potentiostatic tribocorrosion experiments, during which a fixed potential is applied to accelerate corrosion while a ruby sphere is moving across a metal surface. The expected outcome of tribocorrosion experiments is that current will increase from its equilibrium value when a pin is moving across the surface (Mischler, 2008). This increase is caused by the removal and reformation of a coating which forms on the surface of many metals while the metal is oxidizing. The coating does not corrode like the metal does, thus it is a passive coating, contrasting with the active surface of the metal. While the pin is linearly reciprocating back and forth across the surface of the metal, the passive coating can be scratched off. The metal will re-form the passive coating in order to be most stable (Mischler, 2008). While re-passivating, current must flow through the potentiostat to complete the reaction. Thus, the

current should increase during reciprocations due to the continued removal and formation of the passive coating.

Ultimately, two tribocorrosion experiments were done. The intention of these experiments was to verify that the software and equipment were capable of producing useful results. As well, the data collected during the experiments will be used to identify areas to investigate in more depth. Figure 3 shows data collected during reciprocations in the two tribocorrosion experiments. The friction coefficient is calculated using the ratio of forces measured by the force-torque sensor parallel and perpendicular to the metal. It describes the resistance to movement between two surfaces in contact.

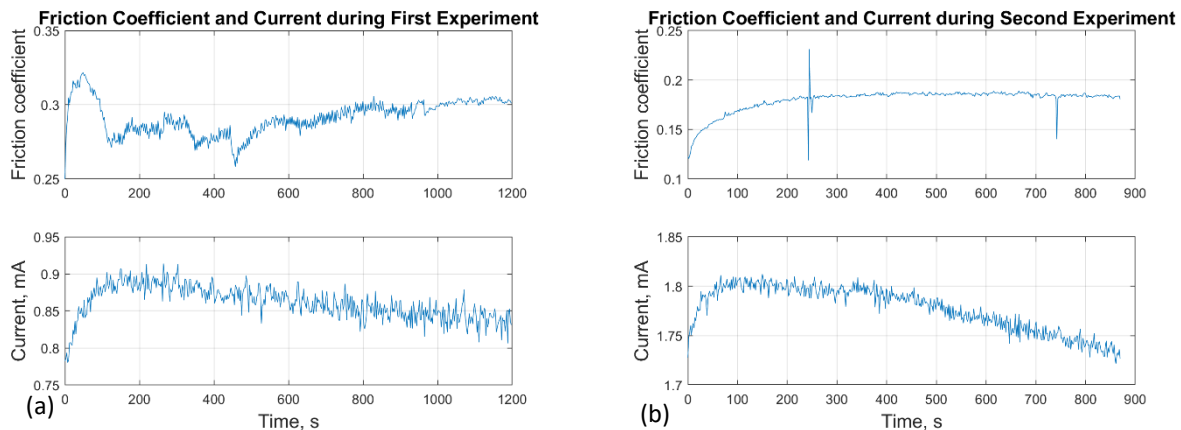


Figure 3. Friction coefficient and current during reciprocations of the first (a) and second (b) tribocorrosion experiments.

The results of the tribocorrosion experiments were successful in demonstrating the capability of the instruments and software. Some modifications were made to the software between the two tests, such as fixing a data collection problem and increasing the reciprocation speed. The test results mostly agree with theory (Mischler, 2008): at the beginning of the reciprocation period, current rapidly increases from its equilibrium value, however the decline in Figure 3 (b) was unexpected. It is possible that the current has not reached a steady-state value in the time frame used, or that a film formed during wear is causing unexpected behavior. The friction coefficient also had interesting behavior during the tribocorrosion experiment. The friction results in Figure 3 (b) were more consistent and reached a steady-state value faster than most of the fry tests. The first experiment friction coefficient data in Figure 3 (a) not very meaningful due to set-up errors: the pin may have been partially rubbing against tape used to reduce surface area. More tests must be conducted to know if the behavior exhibited in Figure 3 is typical for boronized steel.

Significance

The project that I have been working on this summer is part of ongoing research at the Egberts Group to investigate the wear and corrosion behavior of coated metal. My will initiate further investigations into the corrosive properties of the coatings. The apparatus and software which I have contributed to will be used in the upcoming months to generate high-quality data. Within the next year, a paper will be written using data collected with the apparatus developed in this project.

The field of tribocorrosion is new and evolving rapidly. There are still many unknowns about the interactions between mechanical and corrosive wear. The links between corrosion and tribology are very important to our industry partner because of the applications of their coatings in harsh, corrosive environments. The tribometer which I have modified is the only instrument in Calgary which links a traditional reciprocating tribometer with a potentiostat.

Reflection on my Experience

Receiving a PURE award has allowed me to gain valuable design experience at an early stage in my engineering career. I was able to design an apparatus which is capable of taking sophisticated measurements for future publications. It was immensely rewarding, albeit frustrating at times, to be able to go through the design process from 3D modeling to using the apparatus to make real measurements.

The input of my supervisor and the graduate student who I was working under helped persevere to the successful realization of my objective. I feel that I gained professional experience by working with the other members of the Egberts group.

Summer research has allowed me to learn valuable skills which I will hopefully be able to apply in the future to graduate studies. I feel I have an advantage for graduate studies and in my future career because of my experience during this award term.

References

- An, J., Tian, X., Lu, Y., Sun, S. J., Wang, Z. G., Hao, W. Q., Zhu, X. D., & Yang, Y. L., (2008). Effect of boronising on mechanical properties, wear and corrosion of N80 steel. *Materials Science and Technology*, 24(3), pp. 314-319.
- Fichtl, W. (1981). Boronizing and its practical applications. *Materials in Engineering*, 2, pp. 276-286.
- Mischler, S. (2007). Triboelectrochemical techniques and interpretation methods in tribocorrosion: A comparative evaluation. *Tribology International*, 41, pp. 573-583.