A Round Robin to evaluate the degradation of materials in sliding contacts operated in aqueous solutions

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Content of the lecture

• What is a Round Robin Action?
• Test protocol
• Case study: tribocorrosion of a passivating CoCrMo biomaterial
  • Electrochemical aspects
  • Role of surface film on friction and wear
  • Synergism in tribocorrosion
What is a Round Robin Action?

In experimental methodology, a **round robin test** is a test (analysis, measurement, or experiment) performed independently several times.

The purpose is the **verification of a new method of analysis**!

When a new method of analysis is developed, a round robin test involving proven methods would verify whether the new method produces results that agree with established ones.

A modern term dates from the 17th Century French “ruban rond” (round ribbon) = the practice of signatories to petitions appending their names on a document in a non-hierarchical circle or ribbon pattern.

What is a Round Robin Action?

This involves **multiple independent scientists** performing the same test method in different equipment, or a variety of methods and equipment.

A **true round robin** only occurs in the specific case that all participants evaluate or test the **exact same test object**.

- a test methodology described as accurate as possible,
- reporting by each participant according a given template,
- an in-depth analysis by the co-ordinator,
- an improvement of the methodology eventually elaborated for the new round.
Test protocol on tribocorrosion

Starting point: need for appropriate testing tools

• Preliminary approach:
  • identifying existing testing methodologies
    (literature search, standards like ASTM, AFNOR, DIN, BS, …)
  • identifying main process mechanisms
    (physical, mechanical, chemical)
  • identifying main process parameters
    (temperature, pressure, environment, materials, …)

• Development of a test protocol (guidelines)

Tribocorrosion process

- Passive film
- Substrate
- Corrosion
- Repassivation
- Corrosion accelerated by wear
- Corrosion products
  Wear accelerated by corrosion
Experimental fact: 
a synergism ($\Delta W$) between corrosion and wear processes!

$$W_{\text{tribocorrosion}} = W_{\text{corrosion}} + W_{\text{mechanical}} + \Delta W$$

Ways to determine $\Delta W$:

ASTM G119-04 Determining Synergism between wear & Corrosion
drawback: not applicable on passivating materials

New protocol required valid for passivating materials:

A methodology for the assessment of the tribocorrosion of passivating metallic materials

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Objective of the new test protocol:
To allow a fast evaluation of the potential and risks of new materials in sliding contacts operated under wet conditions (tribocorrosion) simulating biological systems

Approach used in the test protocol:
Combination of electrochemical measurements and sliding tests to decouple material losses originating from corrosion ($W_{\text{corrosion}}$) and wear ($W_{\text{wear}}$)
Test protocol on tribocorrosion

Protocol for passivating metallic materials is based on:
- behavior of passivating materials at open circuit potential **without sliding** (phase I)
  ➔ passivation kinetics
- corrosion and wear of materials at open circuit potential **under continuous sliding** (phase II)
  ➔ mechanical and corrosive components of total wear
- repassivation rate of mechanically activated material **under intermittent sliding** (phase III)
  ➔ influence of repassivation on wear

Behavior of CoCrMo at open circuit potential without sliding (phase I)

- good repeatability of Eoc in presence of albumin
- albumin causes a faster stabilization of open circuit potential
Test protocol on tribocorrosion

Behavior of CoCrMo at open circuit potential without sliding (phase I)

Reproducibility?
Test protocol on tribocorrosion
Corrosion-wear of at OCP under continuous sliding (phase II)

- Repeatability is even better in presence of albumin
Test protocol on tribocorrosion

Physical approach:
galvanic coupling between worn and unworn areas !!

![Diagram showing passivation, oxide cracking, repassivation, and mechanical activation processes in tribocorrosion.](image)
Physical approach:
galvanic coupling between worn and unworn areas !!
AISI316 in 0.5 M H$_2$SO$_4$ under continuous unidirectional sliding (120 rpm = 0.5 s)

Counterbody: ZrO$_2$
Normal load: 5 N

Test protocol on tribocorrosion

Wear volume in PBS + albumin = $1.70 \times 10^{-7}$ cm$^3$ +/- $0.05 \times 10^{-7}$
Wear volume in PBS = $1.02 \times 10^{-7}$ cm$^3$ +/- $0.17 \times 10^{-7}$
Test protocol on tribocorrosion

Repassivation rate under intermittent sliding (phase III)

Wear scars on 316L stainless steel after fretting tests done at different holding times in ambient air of 50% RH

- holding time = $t_{off}$
- Scan by profilometer ($\mu$m)

Wear depth ($\mu$m)

- holding time 2 s
- holding time 8 s
Test protocol on tribocorrosion

Repassivation rate under intermittent sliding (phase III)

OCP during continuous ($T_r = 0.5\ s$, $t_{on} = 0\ s$) and intermittent ($T_r = 0.5\ s$, $t_{off} = 4.5\ \text{or}\ 49.5\ s$) unidirectional sliding of AISI316 against zirconia at a normal force of 5 N

Test protocol on tribocorrosion

Repassivation rate of mechanically activated Al-based alloys under intermittent sliding (phase III)

Al$_{71}$Cu$_{10}$Fe$_{9}$Cr$_{10}$

increase of $T_{off}$ improves passivation

Al$_3$Mg$_2$

increase of $T_{off}$ increases corrosion
Tribocorrosion of passivating metallic materials results in a change in friction and wear due to a modification of surfaces:
- friction: affected by removal/re-growth of oxide in sliding contact
- wear: affected by galvanic coupling between worn/unworn areas

Synergism in tribocorrosion can be analyzed by:
- in-situ electrochemical measurements, and
- appropriate test protocol for passivating materials!

Functionalizing the surface of passivating materials is one attractive way to by-pass degradation by tribocorrosion!
Think tank
How would you explain the lowering of material degradation in these different wet media?

Main conclusions and challenges

- Round Robin tests are useful to evaluate repeatability and reproducibility.

- Major attention to be given to possible variations in test conditions at different labs. Therefore: literature data must be analyzed and used critically!

- Tribocorrosion = emerging field of scientific interest

- Industrial recognition is growing (implants in health sector, food industry, electrical connectors in transportation, MEMs technology, environmental issues….)
Main literature for further reading

- J.-P. CELIS, P. PONTHIAUX, F. WENGER
  Tribo-corrosion of materials: Interplay between chemical, electrochemical, and mechanical reactivity of surfaces,
  Wear, 261 (9), 939-946 (2006)

- S. ACHANTA, D. DREES, J.-P. CELIS
  Friction from nano to macroforce scales analyzed by single and multiple-asperity contact approaches
  Surface and Coatings Technology, 202, 6127-6135 (2008)

- N. DIOMIDIS, J.-P. CELIS, P. PONTHIAUX, F. WENGER
  A methodology for the assessment of the tribocorrosion of passivating metallic materials