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Chapter

Tribology: The Tool to Design Materials for Energy-Efficient and Durable Products and Process

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Abstract

This chapter describes a summary of the main tribological achievements carried out in TEKNIKER during the last 37 years. It covers the description of commercial and newly developed tribological test benches and case studies for a wide variety of applications. The examples refer to different tribological characterization tools for material selection (e.g., composition, surface treatments, lubricants). It makes emphasis in the failure mechanisms (pitting, scuffing, abrasion, adhesion, thermal fatigue, tribocorrosion, etc.) and friction simulation of a wide range of materials (seals, textiles, steels, cast iron, light alloys, ceramic, composites), tribological systems (mechanical components, biomaterials, tribolubrication), and environments (vacuum, ultrahigh vacuum, low or high temperature, and corrosive). A huge range of new testing equipment and protocols have been developed to simulate the mentioned failure mechanisms and working environments. This knowledge will make possible, in the future, to simulate at laboratory a still wider list of tribological systems and develop new standards. Tribology will help to implement materials solutions into energy and resource efficient products and process, to reduce carbon footprint.

Keywords: tribology, tribocorrosion, lubricants, materials, pitting, scuffing, abrasion, adhesion, erosion, thermal fatigue, die soldering, vacuum, ultrahigh vacuum, tribodesorption, outgassing, high temperature, corrosive

1. Introduction

TEKNIKER is a nonprofit applied research institution located in the armoring city of Eibar specialized in manufacturing, precision engineering, and tribology, with a history of 37 years. It is a founded member of the IK4 Research Alliance, a private alliance of seven R&D centers with 1158 persons and 308 PHD, with the mission to generate, capture, and transfer scientific and technological knowledge to industries. The tribology unit from TEKNIKER is working on failure diagnosis, surface characterization, and solving tribological problems in direct collaboration with other units from TEKNIKER like surface physics and chemistry, lubricant chemistry, laser, additive and micro-nano manufacturing, maintenance, mechanical design, sensors, and robotics. TEKNIKER is a European referent in the field of tribology with participation in more than 130 projects (EU, Spanish, and regional)

with more than 250 scientific contributions in congresses, journals, and books and 3 patents. It has been active in **several international working groups** and associations, in some of them, acting as Spanish representative:

- The COST 516 about tribology (1995–2000)
- The COST 532 about tribotechnology of engines and transmissions (2002–2007)
- The COST 533 on biotribology (2003–2008)
- The Virtual Tribology Institute (VTI) involving 22 centers of tribology (2005–2016)
- The steering committee member (2005–now) of the European Materials Platform (EuMaT) holding since 2017 the co-secretary
- The NANOMAT (Basque materials and nanotechnologies team (2005–2006))
- The E!-ENIWEP, the Eureka Network for wear prevention (2006–2010)
- The MATERPLAT Spanish Materials Platform (2009–now)
- The E!-SURF, the Eureka Umbrella on Surfaces (2010–2016)
- The Network 2B Funtex on Functional Textiles (2011–2014)
- The steering committee of EMIRI, the materials for Low Carbon Energy Industrial Initiative (2012–now)
- The International Tribology Council (2014–now)
- The Austrian COMET Action X-Tribology (Excellence Center for Tribology (2012–now))
- The BIC, Biobased Industries Consortium (2014–now)
- The Knowledge-based Multifunctional Materials Virtual Institute (KMM-VIN) (2016–now)
- The ECP4 Network about plastics and composites (2015–now)
- The CAPTECH Materials for Defense of the European Defence Agency (EDA) (2016–now)
- The Coordination and Support Action (CSA) MATCH, Materials Open House (2014–2017), promoted by the Alliance of Materials A4M that was created by Suschem, Nanofuture, EMRS, FEMS, EMIRI, and EUMAT
- The European Materials Modeling and European Materials Characterization Councils (EMMC-EMCC) (2017–now)
- The materials for construction team of the ECTP Construction Platform (2018–now)

- The European Pilot Projects Network (EPPN) (2017–now)
- The Basque Country representation in the materials for batteries S3P Smart Specialization Region initiative (2018–now)

TEKNIKER has been active in the **organization of Congress** (LUBMAT, IBERTRIB, COST 516, COST 532, COMADEM, EUMAT, NANOFILMS); international meetings in cooperation with MATERPLAT, EMRS, FEMS-SOCIEMAT, EMCC, and EPPN; and **courses** such Marie Curie (Oct. 2011), LUBMAT (2012, 2016, 2018), or Erasmus+ (2018).

2. Commercial equipment

The philosophy from TEKNIKER has been to buy commercial equipment when available and to develop and construct new testing benches, when they are not easily available. Thus, TEKNIKER is equipped with the **most advanced commercial equipment** covering a huge range of testing standards such as:

- FALEX **high-performance** and FALEX multispecimen test machines (ASTM G99, ASTM D3702, ASTM D5183, ASTM D2266, ASTM D4172)
- FALEX **Four-Ball** Machine to measure antiwear properties (ASTM D 2266 for greases, ASTM D 4172 for oils) and extreme pressure properties (ASTM D 2596 for greases, ASTM D 2783 and ASTM D-7421 for lubricants)
- FALEX **tapping torque test machines** (ASTM D5619)
- OPTIMOL **SRV-III machine** to measure antiwear properties (ASTM D 5707 for greases, ASTM D 6425 and DIN 51834 for lubricants) and extreme pressure properties (ASTM D 5706 for greases, ASTM D 74121 for lubricants)
- CETR UMT-3 **reciprocal tribological test**, Block on Ring Test (ASTM G77), scratch test (ASTM D7027, ASTM G171, ISO 20502), Ball on Flat (ASTM G-133), Friction dissipation (ASTM G-203), Pin Abrasion (ASTM G132), Pin on Disc (ASTM G99), Scratch hardness (ASTM G171), Thrust washer (ASTM D3702), Rolled web friction (ASTM G143), Viscoelastic properties (DIN 53513), Indentation hardness (ISO 14577), Adhesion and mechanical failures (ASTM C 1640)
- CSM **nanotribometer** (Load from 20 μ N-2N)
- **Twin disc** machine with Block on Ring configuration (e.g., ASTM G176, ASTM G77)
- STRAMA FZG **gear test machine**, wear (ASTM D 4998), Micropitting (FVA 54/I-IV), DGMK 575), Pitting (FVA 2/V), Scuffing (DIN 51354-1/2, ISO 14635-1, CEC-L-07-96, IP 334/93, ASTM D5182-91), grease (DIN 74), Extreme Pressure test (FVA 243)
- **Two bearing tests** by Elgeti Engineering to characterize bearings lubricated with greases and oils (DIN51819) and to develop new lubricants, coatings, rolling bearings designs, and predictive maintenance activities

- Ducom **Erosion Tester** (ASTM G-76)
- TABER **abrasion machine** (many standards, e.g., ASTM 4060 (paints), ASTM D3884 (textiles))
- **Pendulum slip** safety testing (UNE-ENV 12663, CEN/TS 16165, BS7976-2, ASTM E303, BS EN 13748-1)
- GMG **tribometer test for flooring** security (DIN 51131, CEN/TS 16165)
- Microtest **tribometer** transformed by TEKNIKER in a tribocorrosion device
- NEURTEK **Washability** Test, humid rubbing test (UNE-EN ISO 11998, DIN 53778, ASTM D 2486, ASTM D 4213)
- CORMET **stress corrosion cracking** in liquid media with electrochemical control until 300°C and autoclave to test stress corrosion at high temperature (800°C) and gaseous media (SO₂, NO₂, CO₂, O₂) (ASTM Practice G 129, ASTM Test Method E8). NACE Standards (TM0198, TM0177)
- Corrosion test salt spray (ISO 9227, ASTM B117)
- Metrohm Autolab **potentiostats**, Electrochemical corrosion (ISO 16773-1-4), liquid corrosion (ISO 2812-1, ISO 2812-2), corrosion classification (ISO 12944-6), others (ASTM G1, ASTM G3, ASTM G5, ASTM G61, ASTM G96, ASTM G102, ASTM G106)
- **Climatic chambers** (UV, temperature, humidity, condensation), (e.g., ISO 6270, ASTM D4329, ASTM D4587, ASTM D4799, ASTM D5208, ASTM G151, ASTM G154, DIN EN 12224, DIN EN 1297, DIN EN 13523-10, DIN EN ISO 4892-1, EN 927-6, ISO 11997-2, ISO 16474-3, ISO 4892-3, ISO 20340 (Off shore, Norsok), D4585)
- **INSTRON mechanical tests** at different temperatures at 1–50 kN with speed range 0.005–500 mm/min (many, e.g., tear test (ISO 13937-2:2000))
- **Compatibility of seals** with lubricants (ASTM D471)
- Complete laboratory for **paint characterization** to measure impact (e.g., ISO 6272), falling sand abrasive test (e.g., ASTM D 968, ASTM D333; ASTM D1395; ASTM D 2205), crosscut test (e.g., ASTM 3359), bend test (e.g., ISO 6860), Persoz and Kónig hardness (e.g., ISO 1522), pencil scratch test (e.g., ISO 1518-4), cupping test (e.g., EN ISO 1520), pull off test (ISO 4624; ASTM D4541), brightness loss (Crock meter)

When possible, round robin tests have been carried out (pin on disc tests, tribocorrosion tests) with different organizations having similar commercial machines.

3. New testing benches

The differential from TEKNIKER is the **capability to develop unique tribological test machines** and new testing configurations to design a wide variety of working conditions (temperature, load, speed) in **different environments** (atmospheric,

inert, vacuum, ultrahigh vacuum, corrosive, etc.) in order to cover different tribological problems to analyze the **tribomechanism taking** place on the **rubbed surfaces** (adhesion, die soldering, galling, abrasion, pitting, micropitting, fretting, tribocorrosion, tribodesorption, tribolayer formation, impact, erosion, tactile friction, corrosion, fatigue corrosion, fretting-corrosion, thermal fatigue, fading, etc.).

The objective of the measurements carried out with the tribological testing machines is to measure differences in the material behavior. For that, a reference is always used that corresponds to a material with a well-known behavior. The standard deviation of the measurement is established with the reference material. The value of the standard deviation depends on (a) the working conditions (pressure, velocity, temperature, etc.) and (b) the properties (roughness, texturing, composition, etc.). The difference between two materials is significative, if it is higher than three times the standard deviation of the measurements. In some cases, also standard protocols have been developed (e.g., tribocorrosion tests) in collaboration with ISO/CEN groups.

The **new test benches developed by TEKNIKER** have been the following:

- New atmospheric, vacuum, and ultrahigh **vacuum tribometer** with capacity to test friction and wear at different temperatures and also analyze the tribodesorption of gases, assisted by mass spectra.
- New DEMETRA machine, to measure the **outgassing** of the lubricants, polymeric materials, and coatings that need to work under vacuum conditions (ASTM E 595, ECSS-Q-ST-70-02C).
- **New drag friction** tester to measure drag friction for different applications (e.g., paints in the ships, friction losses in heat exchangers).
- **Tribocorrosimeters** under oscillatory and rotatory conditions, where it is possible to calculate mechanical wear, corrosive (chemical) wear, and the synergy between mechanical wear and chemical wear (UNE 112086, ASTM G119 and internal protocols).
- **Bearing** tests (radial and axial), to measure torque and wear (now in Mecauto-Wisco Company).
- **Real bearing friction and wear** test configuration adapted to a Twin Disc and Falex machines.
- **Ball on rod** machine to test point contact fatigue at high pressure and speeds (12000rpm, reference STP 771).
- **Cylinder on cylinder** to test line contact fatigue at high pressure and speeds (reference STP 771).
- **Thermal fatigue** test bench, to simulate cycles of high temperature (up to 900°C) and low temperature (the T°C depends on cooling unit selected). One machine is available in TEKNIKER and another in AUDI.
- **Gravelometer** to test the stone-chip (abrasives, screws) resistance to impact of coatings (paints, varnish) (reference ISO 20567-1:2005). Internal protocols have been developed to test impact resistance in polymer injection molds.
- **Clutch dynamometer** test, to measure torque and wear (now in GOIZPER Company).

- **Disc brake** dynamometer test bench, to measure the friction, wear, and thermal fatigue of disc brakes (now in Edertek-Fagor Ederlan company).
- Testing configurations of **engine component** wear and friction test have been adapted to the SRV, FALEX, and CETR machines (e.g., ASTM G181-11, ASTM G206-17). Several new protocols have been developed to simulate valve stem/guide, gudgeon pin/piston, piston ring/cylinder liner, piston ring/piston, piston skirt/cylinder liner, impact of valve seat/valve head, etc.
- **New seals** test bench under reciprocating and oscillatory movement with capacity to test with different lubrication systems and different temperatures.
- **Prosthesis simulators** involving real components for evaluating tribological properties of hip prosthesis coatings and synovial fluids, adapted to FALEX and SRV machines.
- **Car wash type test**, cleaning and abrasive testing of glass (ISO 20566) and solar mirrors (internal protocols).

4. Description of the main new test benches

4.1 CATRI (CA3UHV) machine

CATRI (CA3UHV) machine has been developed by TEKNIKER to determine the friction and wear properties in atmospheric, vacuum, and ultrahigh vacuum conditions. It is also possible to measure the gas tribodesorption detected by mass spectra, during friction and wear tests. Both the force sensor and sample holder of the experimental system have been patented, PCT/ES2009/070635 and PCT/ES2010/07273, respectively [1–4]. In this machine, the patents from CSIC ES200700480 and ES2320513B1 have also been validated. This machine allows complex tribological characterization of materials and coatings in a wide range of gas pressures from atmospheric to ultrahigh vacuum and loads (0.1–20 N). This system can be used to study tribological behavior of the coatings under gradually varying gas pressure and composition, simulating the environments on the various stages of the service life of the real aerospace systems. The machine can work under ultrahigh vacuum (5×10^{-10} mbar) and inert gas in controlled atmosphere (from 10^3 to 5×10^{-9} hPa). T°C can reach up to 350°C. It can be possible to quantify the gas tribodesorption from materials and coatings (**Figure 1**).

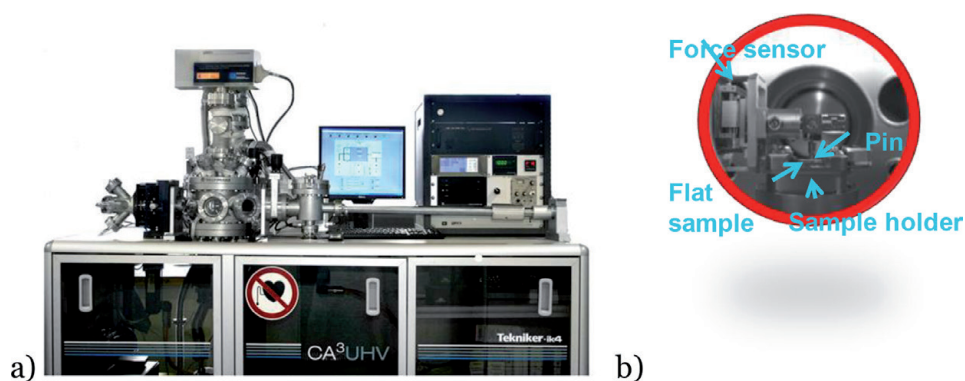


Figure 1.
(a) CATRI machine, (b) detail of the sample holder, TEKNIKER®.

4.2 DEMETRA machine

DEMETRA machine is developed for the analysis of the outgassing from lubricants and materials (e.g., coatings, textiles, and plastics) in vacuum conditions. The bench test has been built using the ASTM E595-07 and ECSS-Q-ST-70-02C Reference Protocols. In the test, the volatile components of the materials and lubricants are determined at 125°C in vacuum conditions. The flexibility of the machine allows to modify working conditions. According to the standard, the Recovered mass loss (RML) should be <1% and the Collected volatile condensable material (CVCM) should be <0.1% (**Figure 2**) [3].

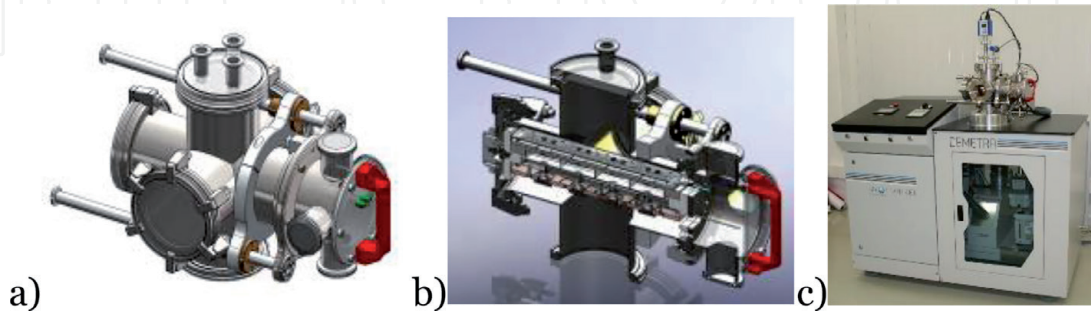


Figure 2.
Details of (a) vacuum chamber, (b) evaporation unit with four testing stations, and (c) DEMETRA machine developed by TEKNIKER.

4.3 Drag friction test

Drag friction test is for the determination of the drag friction torque of antifouling paints or surface treatments (e.g., texturing). The test samples are hollowed cylinders of 200 × 200 mm that can be either located in ships or in the port to be covered with fouling after a specific time period. The torque before and after fouling and with different surface treatments can be compared at different rotating speeds, simulating either laminar or turbulent flow. Two test configurations are available with gaps of 5 and 10 mm. The machine has been developed by TEKNIKER in the frame of EU Project Foul X PEL (<https://cordis.europa.eu/project/rcn/101484/reporting/en>) (**Figure 3**) [5, 6].

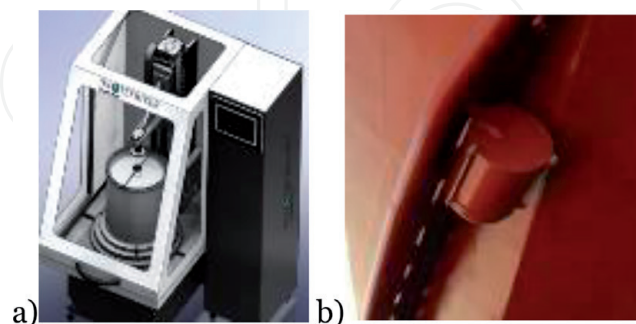


Figure 3.
(a) Drag friction tester, TEKNIKER®. (b) The image shows the testing cylinder location in a ship.

4.4 Tribocorrosion testers

Tribocorrosion testers have been developed combining a tribometer and a potentiostat. The test equipment allows monitoring and control both mechanical and electrochemical parameters. A tribometer creates relative motion, either unidirectional (rotatory) or bidirectional (reciprocating), rubbing two surfaces against each

other. The electrochemical cells are used to record and control the electrochemical parameters. These cells are usually composed of three electrodes: a reference electrode, a counter-electrode, and the working electrode. The reference electrode has a stable, well-defined potential, and it is used to register the potential of the working electrode, i.e., the test sample. Typical reference electrodes are saturated calomel electrodes (SCE) and silver/silver chloride electrodes (Ag/AgCl). The counter-electrode is used to measure or control the current and is usually made of inert materials such as platinum, gold, or graphite. The electrodes are connected to a potentiostat to register the potential between the reference electrode and the working electrode or the current between the counter-electrode and the working electrode. A typical tribocorrosion test setup is schematically shown in **Figure 4** [7–16].

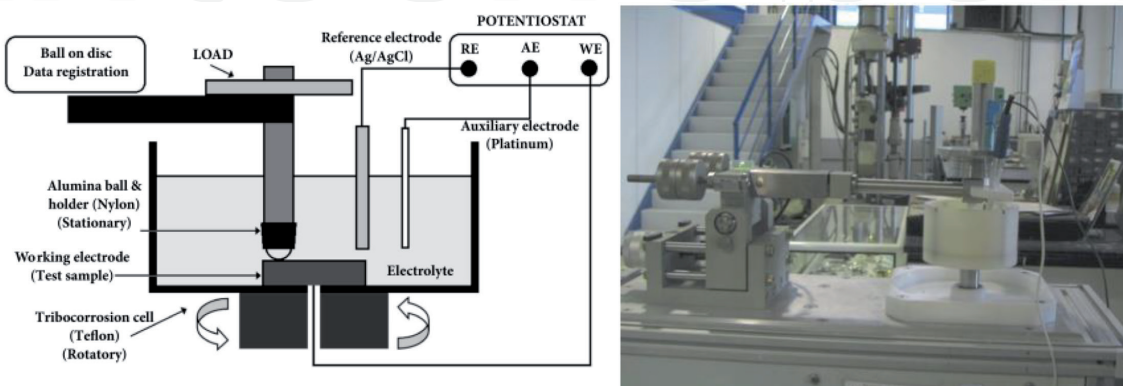


Figure 4. Experimental setup of a tribocorrosion device for a unidirectional tribometer under ball on disc configuration, TEKNIKER®.

4.5 Test bench for plain bearings

A test bench is built with the objective to carry out friction test and wear test in plain bearings. Alternative movement has a stroke of 120–125 mm, a speed of 1–40 m/min, and a maximum load of 3000 kgf (**Figure 5**) [17, 18].



Figure 5. Plain bearing testing machine, designed and constructed by TEKNIKER for Mecauto-Wisco company.

4.6 Ball on rod machine

Ball on rod machine is built with the objective to study the point rolling contact fatigue of the bearing materials and coatings, to apply to ball rolling bearings. It follows

the protocol from Douglas Glover, “A Ball-Rod Rolling Contact Fatigue Tester” (ASTM STP 771, J. J. C. Hoo, D., American Society for Testing and Materials, 1982, pp. 107–124). It has a variable speed, reaching up to 12,000 rpm instead of 3000 rpm of the documented protocol. The typical pressure applied is 5.5 MPa. The lubrication is an air/oil system with controlled flow, typical lubrication regime for high-speed rolling bearings. Alternatively, other lubrication systems can be selected. The balls can be made from steel and ceramic and with or without coatings with a diameter of 12.7 mm. The roller has a diameter of 10 mm and length of 125 mm. The ball on rod machine has a double testing device for parallel testing. The time to reach the micropitting or pitting is recorded and monitored by an increase of the vibrations. The machine was developed by TEKNIKER in collaboration with SNR, in the frame of EU Project Eurobearing (<https://cordis.europa.eu/project/rcn/6657/factsheet/es>) (**Figure 6**) [19].



Figure 6.
Ball on rod machine, TEKNIKER®.

4.7 Cylinder on cylinder machine

Cylinder on cylinder machine, built to study the pitting phenomena in roller bearings, simulates the line contact rolling fatigue behavior of the materials. The machine is based on ASTM STP 771 protocol, with the advantage of reaching a maximum speed of 12,000 rpm with air/oil lubrication. The roller's size is 12x12mm. The duration of the test until reaching the micropitting or pitting phenomena is recorded for different types of materials and coatings of the rollers. The test stops, when the level of vibration increases, due to generation of pitting in the contact. The machine was developed by TEKNIKER in collaboration with SKF, in the frame of EU Project REFINE (**Figure 7**) (<https://cordis.europa.eu/project/rcn/36428/factsheet/en>).

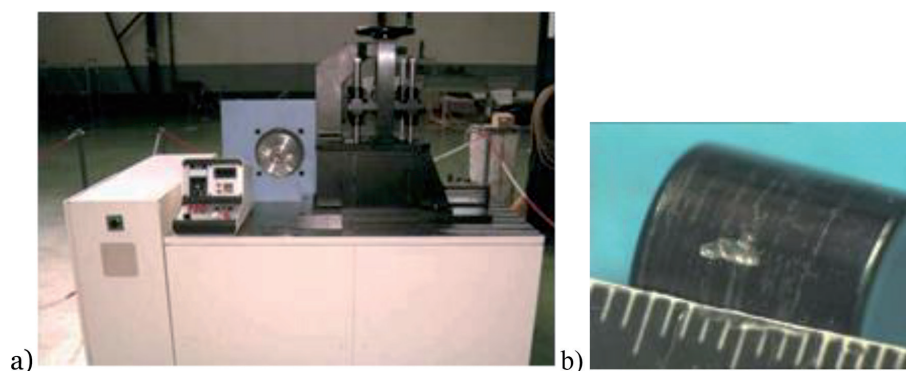


Figure 7.
(a) Cylinder on cylinder machine, TEKNIKER®, (b) pitting in a roller.

4.8 The thermal fatigue

The thermal fatigue test bench has been built to simulate thermal cycles of high temperature generated by induction heating and low temperature cooled with spray. There is one machine in TEKNIKER and one in AUDI. The machine has been developed to simulate the heating and cooling cycles of the high pressure die casting dies. The time to thermal fatigue crack failure mainly depends on (a) the maximum temperature, (b) the temperature gradient, (c) the geometry, and (d) the designed cooling channels inside the mold (testing block). The machine can simulate a time to thermal fatigue failure similar to the real applications (**Figure 8**) [20].

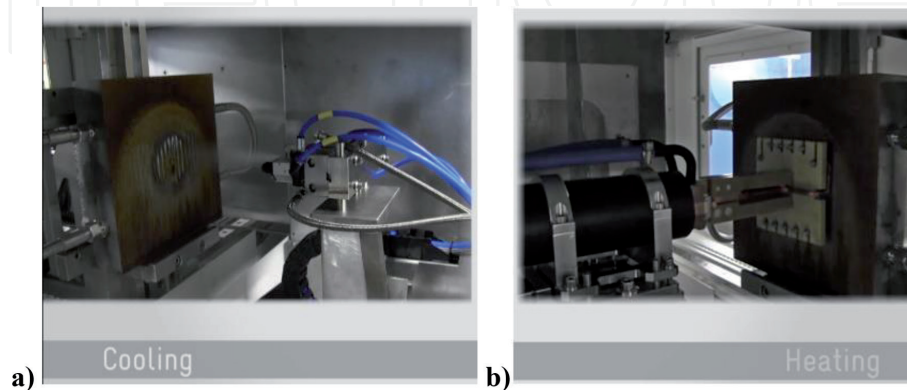


Figure 8.
(a) Cooling process, (b) heating process of the thermal fatigue machine TEKNIKER®.

4.9 Brake disc testing machine

Brake disc testing machine is built with the objective of carrying out friction, wear, and thermal fatigue tests of disc brakes and brake drums. The maximum velocity is 2500 rpm and the maximum load is 105 kg/m². During the test, the torque and the temperature and wear are recorded. Different protocols from automotive suppliers can be applied (e.g., AUDI, Volkswagen) (**Figure 9**) [21].



Figure 9.
Disc brake testing machine, designed and constructed by TEKNIKER for Fagor Ederlan company.

4.10 Clutch dynamometer test

Clutch dynamometer test has been built with the objective to measure the friction and wear of the clutches at fixed or variable conditions of load and speed.

Measurement of force, the friction loss between the cylinder and braking films, and temperatures at different points, forces, torque, and speeds are recorded. The specific power, specific energy, the dynamic and static torques, the sliding time, and initial speed can be monitored during braking or evaluated during the test (**Figure 10**) [22].

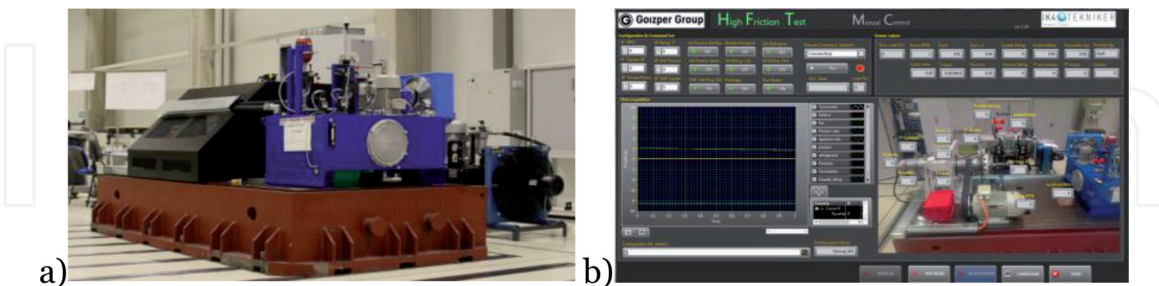


Figure 10.
 (a) Clutch dynamometer test bench. (b) Control unit of the dynamometer, constructed by TEKNIKER for GOIZPER company.

4.11 The TESSA machine

The TESSA machine has been developed by TEKNIKER to measure the friction, wear, and leakage of the dynamic seals [23–28]. It can reach a maximum speed of 6 m/s. The machine has been adapted to work under variable temperature integrating a climatic chamber. The test chamber is modular, and it has the capability to measure the differential pressure between the two sides of the seals for pistons and rollers and evaluate the leakage. Oils sensors, developed by TEKNIKER (www.tekniker.es/en/) and ATTEN2 (<https://atten2.com/en/>), can be used to monitor the generation of wear particles in lubricated seals (**Figure 11**).

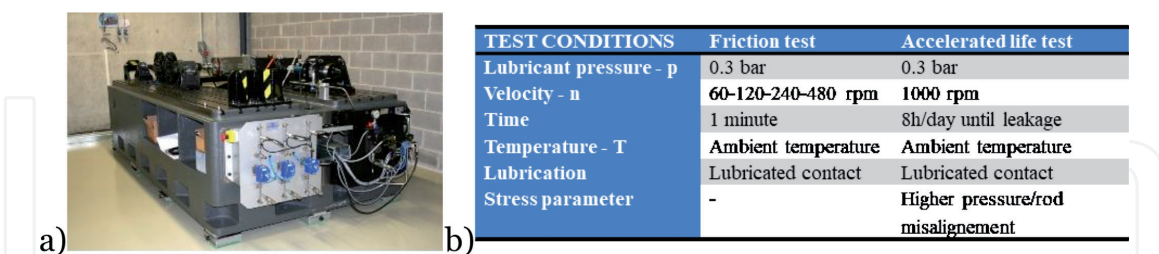


Figure 11.
 (a) New seal test bench constructed by TEKNIKER with two units with oscillatory and reciprocal movements and climatic temperature control (low and high temperatures). (b) Example of test conditions.

4.12 Glass and mirror wash test

Glass and mirror wash test has been built to evaluate the scratch abrasion resistance of coatings used in glass of the cars and solar mirrors. In automotive sector, the glass car wash resistance is assessed. A machine-based washing is simulated in the laboratory environment using a rotating brush and synthetic dirt. The test conditions have been designed to be as close as possible to the real conditions in a car wash and solar mirrors, in order to evaluate the cleanability, the abrasion resistance, and their durability. Protocols for solar mirror evaluation are under development by TEKNIKER in the frame of EU Project In Power (<http://in-power-project.eu/>) (**Figure 12**).

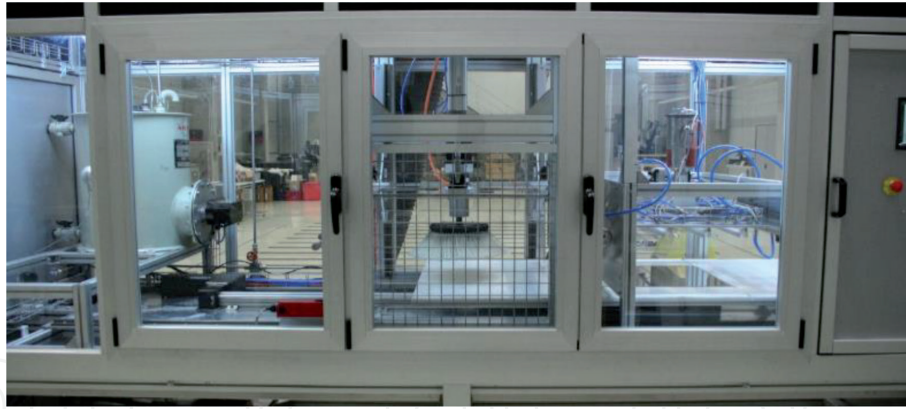


Figure 12.
Glass and solar mirror testing device TEKNIKER®.

5. Case studies

Different case studies have been selected to cover examples of different tribological characterization tools and equipment used for material selection (composition, topography, surface treatments, lubricants), making emphasis in the failure mechanism and friction simulation of a wide variety of materials, tribological systems, and environments.

5.1 Tribology for aggressive environments

5.1.1 Vacuum and ultrahigh vacuum tribology

The **CATRI tribometer** (see **Figure 1**) has been used to screen the tribological properties under vacuum and ultrahigh vacuum of the steel/coating and titanium materials lubricated with different fluids (oils, greases, or ionic liquids). For example, it has been observed that it is possible to find non-halogen ionic liquids to lubricate steel under vacuum and ultrahigh vacuum conditions (e.g., phosphonium phosphate) and to lubricate titanium; up to now, better results have been obtained with halogen-containing ionic liquids or lubricants (e.g., $[\text{Bu}_3\text{MeP}][\text{TF}_2\text{N}]$). The desorption of CF_3 detected in the mass spectra occurred just in the moment when the friction was reduced (see **Figure 13a**). During the test, a TiFx tribolayer was generated. Titanium and inorganic F and S(II) were detected by XPS. The reaction mechanism that explained the TiFx layer generation was elucidated (see **Figure 13c**) thanks to the fragments of the mass spectra (see **Figure 13b**) detected during the tribo-desorption process. The machine and protocols have been used in the National Project CATRI [1], the EU Project MINILUBES [2], the Austrian X-Tribology COMET Initiative [3], the Eurostars Project VACUUM DLC [4], and industrial projects.

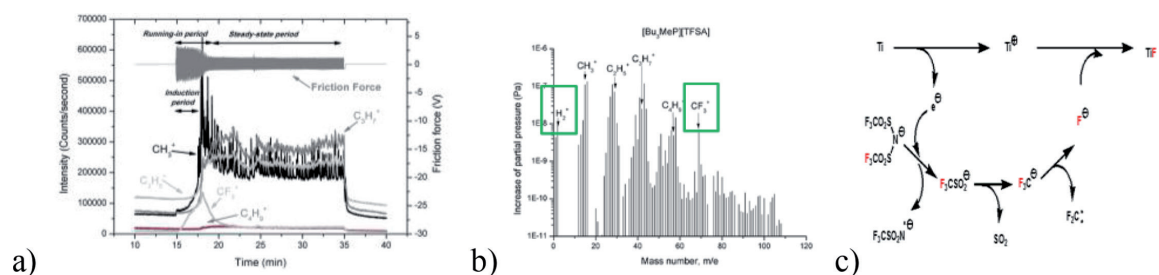


Figure 13.
(a) Friction coefficient during ultrahigh vacuum tribotest (b) mass spectra, highlighting the hydrogen and CF_3 fragments from the F-containing lubricant. (c) Proposed tribomechanism for the TiFx generation.

5.1.2 The DEMETRA machine

The DEMETRA machine (see **Figure 2**) was built to measure the **outgassing under vacuum** to screen and select the materials and lubricants that can work under these conditions. The volatility data of the outgassing measurements of the ionic liquid $[\text{Bu}_3\text{MeP}][\text{Tf}_2\text{N}]$ has been compared to the reference lubricant, a perfluoropolyether (PFPE). It can be stated that the ionic liquid exhibits acceptable outgassing data according to the requirements of the standard ASTM E595 (RML <1.0% and CVCM <0.1%) [3]. The machine has been used in EU Projects VACUUM DLC and Austrian X-Tribology COMET Initiative. It has also been used to study dependence of outgassing of thermal fluids in function of the viscosity for heat exchangers (see Section 11.2) in EU Project SUSPIRE (<http://suspire-h2020.eu/>) (**Figure 14**).

Lubricants	Total mass loss (TML) %	Recovered mass loss (RML) %	Collected volatile condensable material (CVCM) %
PFPE	0.15	0.01	<0,1%
$\text{Bu}_3\text{MeP Tf}_2\text{N}$	<0.1	<0.1	<0,1%

Figure 14.
 Outgassing data of the PFPE oil and the ionic liquid (ASTM E595).

5.1.3 Drag friction tribology in marine environments

The development of the **drag friction test** (see **Figure 3**) has been carried out in the frame of EU Project FOUL X PEL [5, 6]. In this study, the torque of different antifouling paints before and after immersion in a ship or in the Motrico Port has been compared. The tests have been carried out with artificial sea water fluid (ASTM D1141) in a container under rotation at different speed, generating laminar or turbulent regime, using different gaps of 5 and 10 mm. The effect of biofouling, roughness, and fluid properties can be measured. In the example, two fluids FX1 and FX2 were tested and being selected FX2 due to their lower drag friction in all the range of speeds tested. The machine has also been used in the EU Project SUSPIRE to test thermal fluids with different viscosities to select those with lower drag friction loss and higher energy efficiency (see Section 11.2) (**Figure 15**).

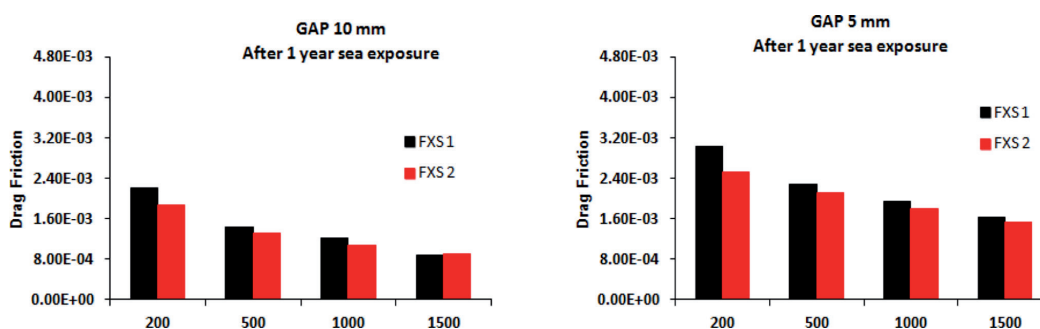


Figure 15.
 Comparative drag friction measurement of two antifouling coatings, before and after 1-year sea exposure in a ship.

5.1.4 Tribocorrosion

The coupling of standard tribometers with a potentiostat has been done to measure **wear-corrosion** behaviour, that means to study the mechanical, the chemical wear and the synergy between both phenomena. In the frame of RAMPE Project [11], the tribocorrosion properties of Inconel + Cr electrodeposited coating is compared with CrN+TiN coating deposited by Physical Vapour Deposition (PVD) developed by TEKNIKER. It is observed that the impedance (corrosion resistance) after the wear test, is considerably reduced for Inconel + Cr Coating. In case of CrN+TiN PVD coating, the impedance curve resulted unaltered after wear test. These results correlate well with the wear scars measured after the test, for both coatings (see **Figure 16**). The technique has been used also in EU Projects FUNCOAT [7, 29–31], FRONTIERS [10], NADIA [32], in Regional project TRICONDEX [8], FRONTIERS [12], and National project NANOTRIBOCOR [9].

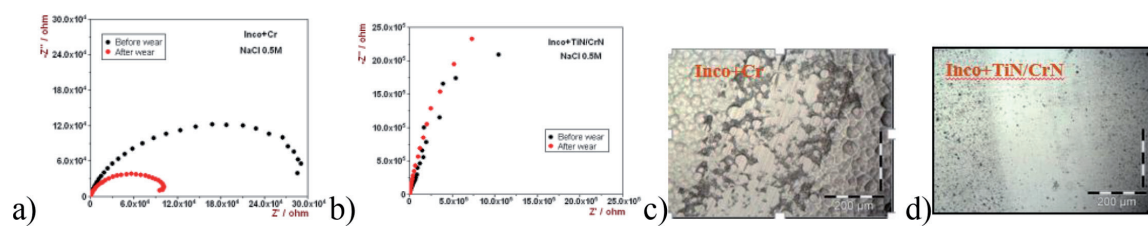


Figure 16. Impedance measurements before and after wear test for (a) Inconel + Cr coating and (b) Inconel + PVD TiN + CrN coating. Wear scars: (c) Inconel + Cr and (d) Inconel + TiN/CrN coating.

5.1.5 Tribocorrosion in marine and offshore environments

High-strength low-alloy (HSLA) steels employed in offshore applications suffer high degradation due to their high corrosivity in seawater and the wear generated during mooring chain movement. The results, obtained in the frame of the FONDEO Adhoc project financed by the Spanish Minister of Science, showed an acceleration of corrosion during wear experiments, as consequence of the synergism generated between wear and corrosion. In **Figure 17(i)**, it can be seen, how the sliding has modified the electrochemical behavior of both steels. The effect of several parameters such as seawater temperature or salinity have also been studied to understand their

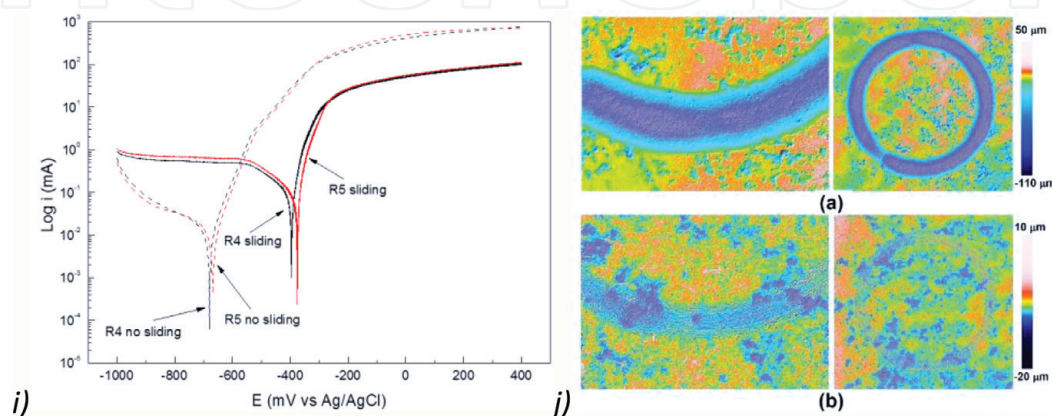


Figure 17. (i) Potentiodynamic polarization curves obtained for two HSLA steel grades (R4 and R5) under corrosion (without sliding) and under tribocorrosion (with sliding) in synthetic seawater. (j) Topography of the wear tracks obtained after tribocorrosion tests on: (a) the TSA and (b) PEO-treated TSA.

influence in the steel degradation [13, 14]. In order to protect steels in mooring lines of floating structures, it has been developed some surface treatments based on thermally sprayed aluminum (TSA) coating with and without post-treatment by plasma electrolytic oxidation (PEO). In **Figure 17(j)**, it can be highlighted the reduction of wear scar after tribocorrosion tests, can be highlighted and produced when the TSA coating on the steel is additionally treated by plasma electrooxidation (**Figure 17(jb)**) [15, 16].

5.2. Tribology of mechanical components

5.2.1 Tribology of journal bearings

Two testing benches have been constructed (see **Figure 4**) for Mecauto company to study the torque and wear in axial and radial bearings. Also, a standard tribotest has been adapted to study the limits of pressure and velocity of the tribological pair. In the frame of BELEADFREE Project, a test has been designed to evaluate the tribological properties of real bearings. In the frame of EREBIO EU Project, the reduction of friction and contact temperature has been proven when using a biodegradable low-viscosity and environmentally friendly engine oil for the lubrication of bearings using a basic thrust washer configuration [17]. In the project ECOBEARINGS [18] and BELEADFREE, the lead content of the bearings have been reduced or eliminated (**Figure 18**).

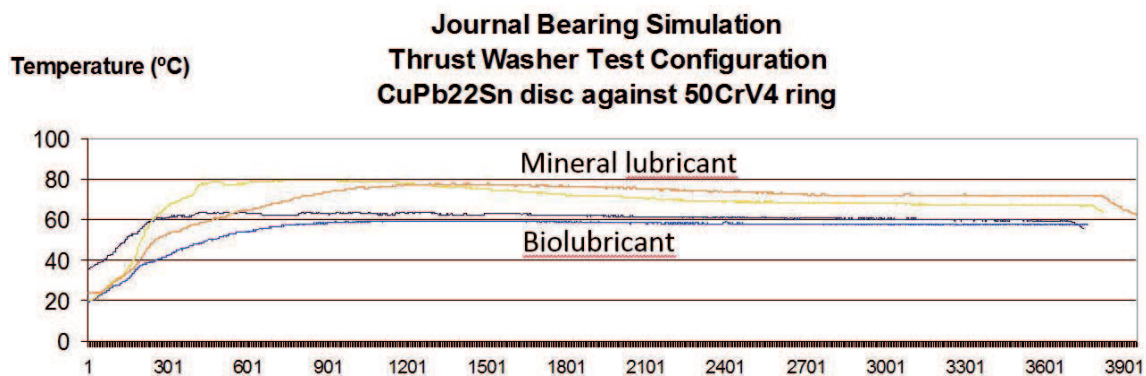


Figure 18. Evolution of temperature when using biodegradable and mineral lubricants to lubricate journal bearings.

5.2.2 Tribology of rolling bearings

As described before, the ball on rod (**Figure 6**) and cylinder on cylinder machines (**Figure 7**) have been constructed by TEKNIKER to study point and line contact rolling fatigue to understand the pitting failure behavior of rolling bearing materials, under high contact loads and high speeds. In the frame of EUROBEARING project, different PVD coatings were studied, measuring the load-carrying capacity, the adherence, the abrasion resistance, and the hardness, as shown in the next table. Their fatigue resistance was also tested using the ball on rod machine. The long fatigue lifetime (1000 million cycles duration) of the TiN + C coating and their good behavior against abrasion and adhesion make this coating a promising alternative to be applied in high-speed rolling bearings [19] (**Table 1**). In this subject, (a) the relationship between vibration generation and oil particle generation (EU Projects TESS) [33], (b) the fatigue behavior of roller bearings (REFINE), and (c) the behavior of different PVD coatings and lubricants for rolling bearings (National Project HIEFBE) have been studied, and (d) a test

configuration to study the tribology of real bearings has been developed, adapted to FALEX tribological test (Project BIOMON).

Coating type	Load-carrying capacity	Adherence	Wear protection	Hardness	Fatigue resistance
CrN	Good	Good	Good	Medium	Bad
Mo	Good	Good	Bad	Low	Bad
TiAlN	Good	Medium-good	Good	High	Good
TiCN	Bad-medium	Bad	Good	High	Good
TiAlCN	Medium	Medium	Good	High	Good
TiN + C (C sputtered)	Medium-good	Good	Medium-good	Medium-high	Very good

Table 1.
Qualitative behavior of coatings to increase lifetime of rolling bearings [19].

5.2.3 Tribology of clutches and brakes

Brakes and clutches require high stable friction coefficient. To understand the tribology phenomena of these systems, testing protocols have been developed for the standard FALEX (high-performance and multispecimen) tribometers to study the friction wear mechanism of brakes and clutches (high temperature abrasion, adhesion, and fading) and to study the relationship of the friction coefficient with the squeal generation. Additionally, a dynamometer test bench has been constructed to test disc brakes (**Figure 9**) and clutches (**Figure 10**). In the frame of the i-SINTER Project, the correlation between the friction measured in pin on disc tests and the torque measured in clutch tests has been studied, observing very similar values, especially at low loads (**Figure 19**). These conditions were selected for screening of the materials with pin on disc tests, before final validation in the clutch test. The tribological knowledge on dry friction has been used in the projects M-eranet JOLIE (brakes) [21], Manunet BRAKE SQUEAL (clutches) [22], and Industrial Project I-SINTER (clutches).

Clutch test vs Pin on Disc

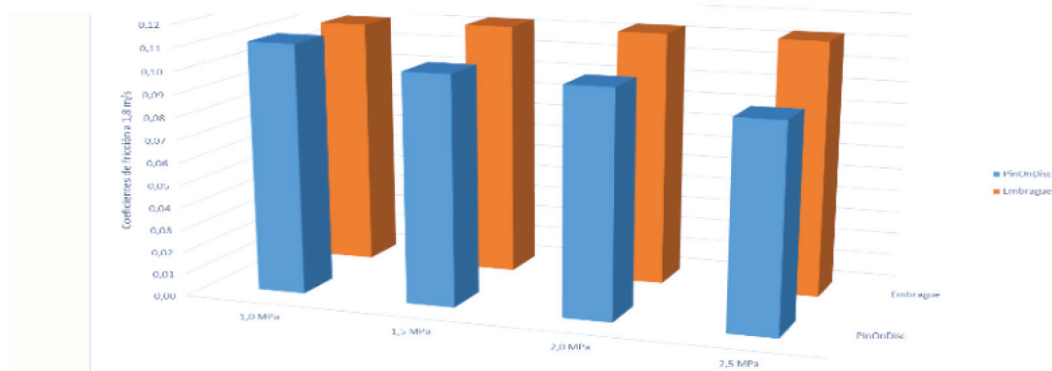


Figure 19.
Coefficient of friction during clutch test and pin on disc test carried out with the same materials.

5.2.4 Tribology of gears and transmissions

In the frame of the EU Project OPTIMIZE, TEKNIKER has upgraded the FZG machine, to measure the vibrations, noise, and transmission losses and to complement the study of micropitting, pitting, or scuffing phenomena (**Figure 20**). In this topic, the team has been working in the EU Projects TESS [33], OPTIMIZE [34], EREBIO [35–37], VOSOLUB [38], SUNOIL [39], LUBRICOAT [40], BIOGREASE [39], and BIOMON [41] and the industrial projects AEROHUMs and SELENA.

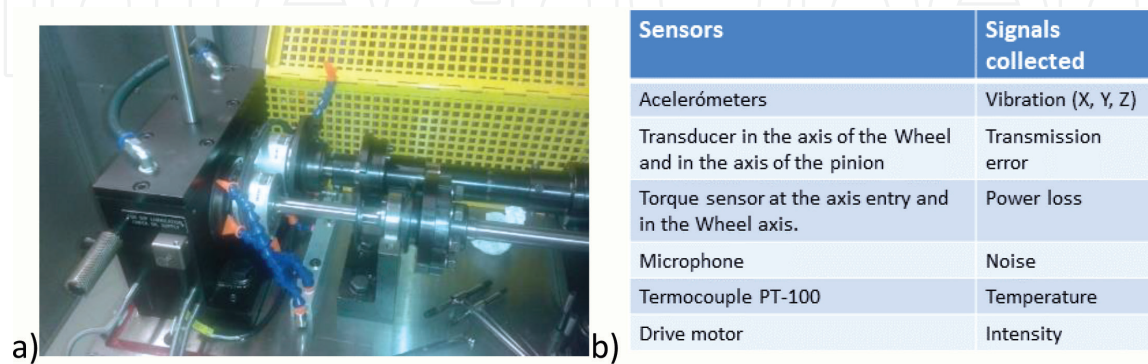


Figure 20.
 a) Upgrading of FZG machine to monitor vibration, transmission error, power losses, noise, temperature, and drive motor intensity and (b) sensors and signals collected [34].

5.3 High-temperature tribology

5.3.1 Tribology of high-pressure die casting (HPDC) and polymer injection molds (PIM)

In the frame of MUSIC Project, a thermal fatigue machine has been constructed to reproduce the main failure of the high-pressure die casting molds. In **Figure 21a**, the thermal cycles can be seen in high-pressure die casting process reproduced in the thermal fatigue machine, and in **Figure 21b**, the thermal fatigue mechanism can be observed, reproduced in a flat specimen with internal cooling circuits, representing the die. The thermal fatigue mechanism can also be reproduced in 3D complex geometries. Setup of testing protocols has been carried out to simulate also other failure mechanisms of HPDC and PIM molds such as mechanical and chemical die soldering, abrasion and erosion resistance, and the corrosion behavior. The knowledge generated in MUSIC project has been applied to the EUROSTAR Project

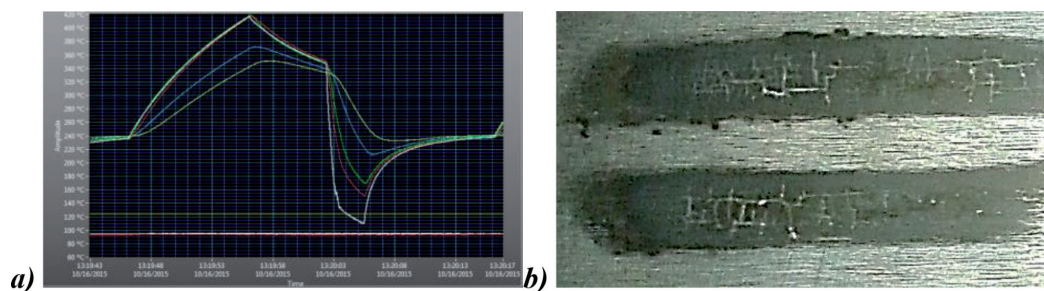


Figure 21.
 (a) Thermal cycles in the thermal fatigue machine TEKNIKER®, (b) thermal cracks reproduced in the tested block of the mold material after 10,000 thermal cycles.

SUPERSLIP (to select surface treatments for polymer injection molds) and to the industrial projects HARCO, ALEPRE, MEFOLUB and MMPUL [20, 42–45].

5.3.2 Tribology of engine and turbine components

The development of testing configurations adapted to standard tribological testing machines can be highlighted in order to test real parts of engine and turbine components (e.g., valve/guide contact, piston ring/cylinder liner contact, the piston skirt/cylinder liner contact, valve head/valve inserts, gudgeon pin/piston, gland/piston rod, piston rod/seal, turbine support, etc.). In **Figure 22**, the results of scuffing tests of the piston ring/cylinder liner used to screen suitable biodegradable oils compatible with bioethanol for two stroke engine oils can be seen. Most of the lubricants suffered scuffing, except the one called SEMO36 that was suitable for the application. The testing protocols developed has been used in the EU Projects POWERFUL [46], EREBIO [47, 48], Nano-HVOF [49], EFCAP [50], COST [51], CLEANENGINE [52, 53], NANOMAG, and NADIA [32]; in the regional project TRIBORE and MOTOLURE [47]; in the industrial projects REMTRAL [51], AUMORE, RECOLURE, EQUIMOTOR and EQUIMOTOR PLUS [54], ABADIE, and 2 MW engine; and in the national projects RAMPE and ALELLA.

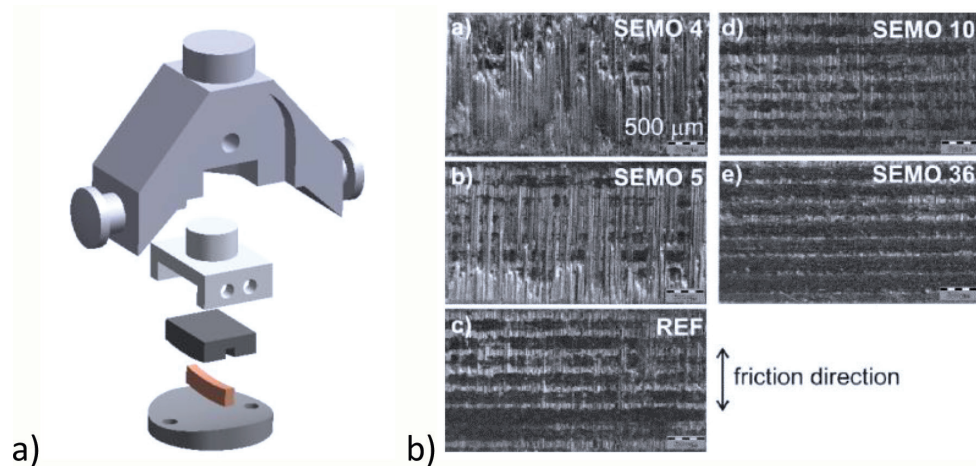


Figure 22. (a) Piston ring/cylinder liner configuration developed by TEKNIKER. (b) Selection of SEMO 36 oil to avoid scuffing of bioethanol/biolubricant mixture, in piston ring/cylinder liner tests.

5.4 Tribology of special materials

5.4.1 Tribology of seals and elastomers

As already described (see **Figure 11**), a test bench has been constructed to test real seals to study their friction, wear, and leakage under oscillatory and reciprocal motions. A simplified tribological test has also been developed to reproduce the failure mechanism and the friction and wear of the seals. In the frame of EU Project KRISTAL, the laser texturing was applied by TEKNIKER directly on seals materials or transferred through the molds. In **Figure 23a**, it can be observed the effect of surface laser texturing on seals materials reducing significantly the friction coefficient. In the EU project STOKES, different surface texturing geometries

were tested in the seals test bench constructed by TEKNIKER, selecting also the most appropriate texturing geometry. This knowledge has been applied to EU Projects FUNDTRIBO [26], ISSELUB, the regional projects MODELOST [23] and EMAITEK [24, 25], and the industrial projects 3D FLEX and ELASWEAR.

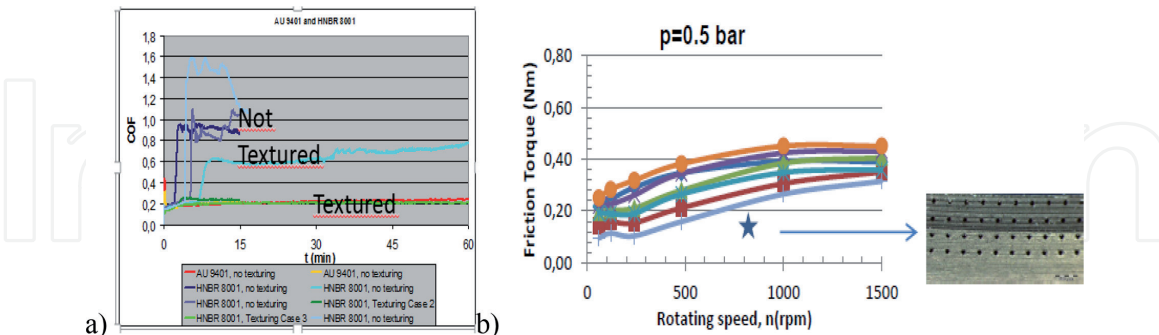


Figure 23. (a) Effect of texturing to improve the tribological properties of different types of seals. (b) Results of the TESSA seal test bench TEKNIKER® with different texturing geometries and selection of the one reaching a minimum torque.

5.4.2 Tribology of anti-slippery floorings

In this context a test protocol has been developed to evaluate the friction (static and dynamic) and wear of the floorings determining the anti-slippery properties simulating the lifetime of the floor (**Figure 24**). This knowledge has been used in the national project TRIBOSTAND, in the EU Project SLIP SAFE, and in a regional project SEGURPAV [55].

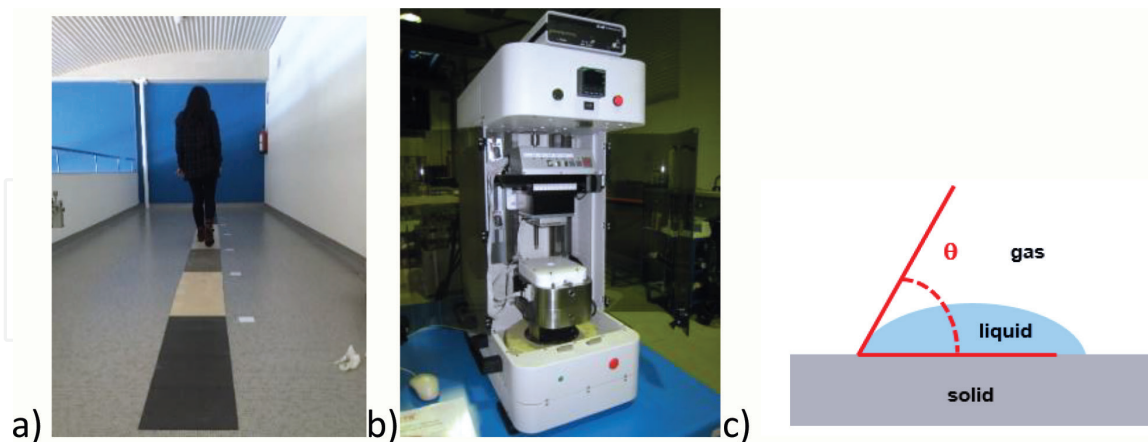


Figure 24. Test protocols have been developed to evaluate the durability, the cleanliness, and the comfort of the floorings (a) involving persons, (b) tribometers, and (c) contact angle measurements.

5.4.3 Tribology of textiles

Testing protocols have been set up to evaluate mechanical properties, abrasion, and tactile properties of the textiles (**Figure 25**). This knowledge has been used in the EU Project 2B FUNTEX [56].

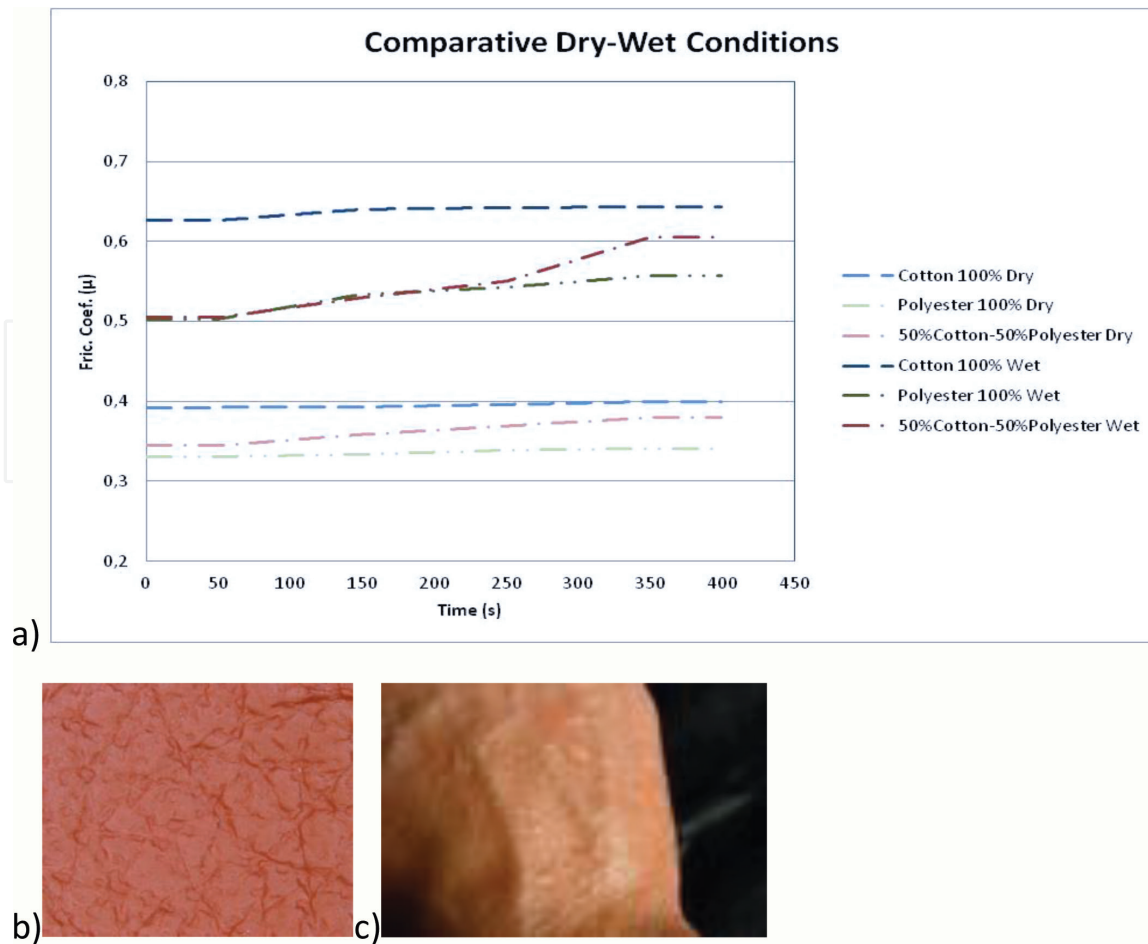


Figure 25. (a) Friction measurements as indicator of textile tactility in dry and wet conditions for cotton, polyester, and the 50% mixture. (b) Material used to simulate the skin at laboratory. (c) Real skin.

5.5 Biotribology

5.5.1 Tribology of autologous bones

The use of impact and compression tests to understand the effect of thermal treatments in artificial bones and inserts has been studied in the frame of the EU Project AUTOBONE. In **Figure 26**, it can be appreciated that the thermal treatment reduces the wear caused by the impact, improving the toughness of the artificial inserts composed of hydroxyapatite and collagen [57].

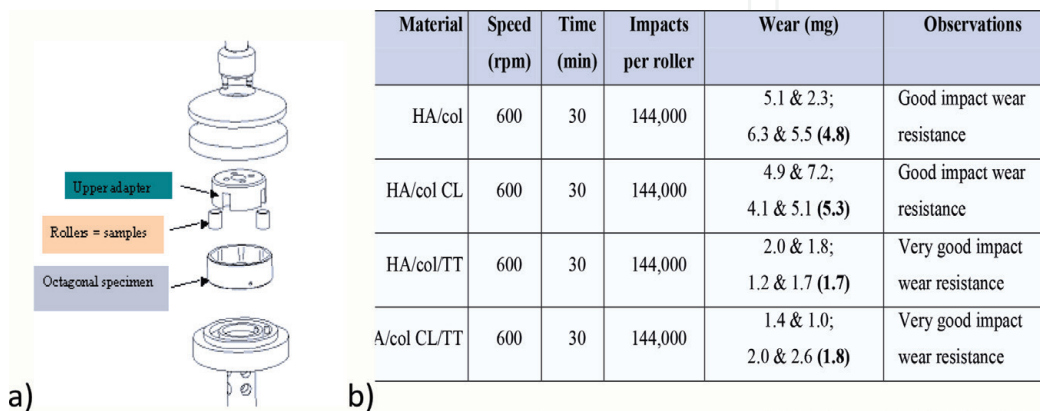


Figure 26. (a) Configuration of impact test. (b) Impact resistance of the hydroxyapatite/collagen bone inserts with and without thermal treatments.

5.5.2 Tribology of hip and knee implants

A testing configuration has been designed adapted to a commercial FALEX tribometer to simulate the wear of hip/acetabular cup. Friction, wear, and tribocorrosion protocols have also been developed to simulate the lifetime of the hip and knee prosthesis. In **Figure 27**, it is possible to see how the TiCN-2 coating deposited by Physical vapor deposition (PVD) by TEKNIKER can enhance the tribocorrosion properties and antibacterial properties. The antibacterial properties are still enhanced when adding a sacrificial silver top layer, also applied by PVD. The Ag layer might be active as anti-infection layer, during implantation, since it will be easily removed due to their poor wear resistance. The protocols have been used in the national projects FUNCOAT, DELECA, SINOVIAL, FUNMAT in the industrial BIOTIDE project, and the Eurostars INNOVATIDE Project [29–31].

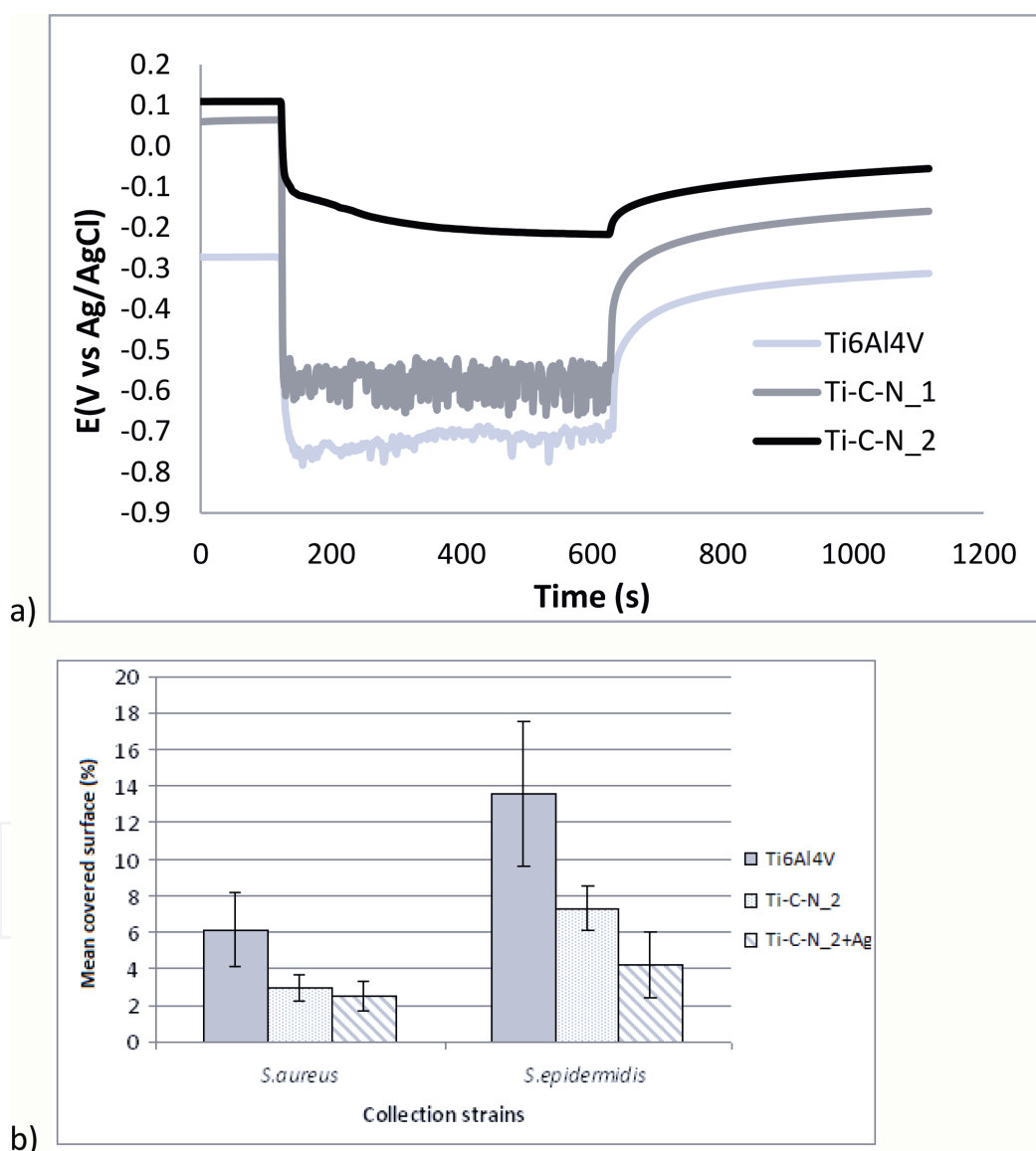


Figure 27. (a) Program for coating evaluation. (b) Tribocorrosion resistance of Ti_6Al_4V alloy before and after coating with TiCN-1 and TiCN-2. (c) Antibacterial properties of Ti_6Al_4V , after coating with TiCN-2 without and with a top silver layer.

5.5.3 Dental tribology

The development of testing protocols can be highlighted to simulate friction, wear, impact, and tribocorrosion in dental implants. In **Figure 28**, the results of

tribocorrosion tests carried out to compare the behavior of titanium before and after treatment with different plasma electrooxidation coatings (PEO) developed by TEKNIKER using different process conditions are represented. The behavior of the coatings improved when increasing the plasma intensity. Also, the wear volume of a reference and new electrolyte was compared observing a significant improvement with the new electrolyte developed in TEKNIKER. This knowledge has been applied to the national project FUNCOAT and industrial projects [58].

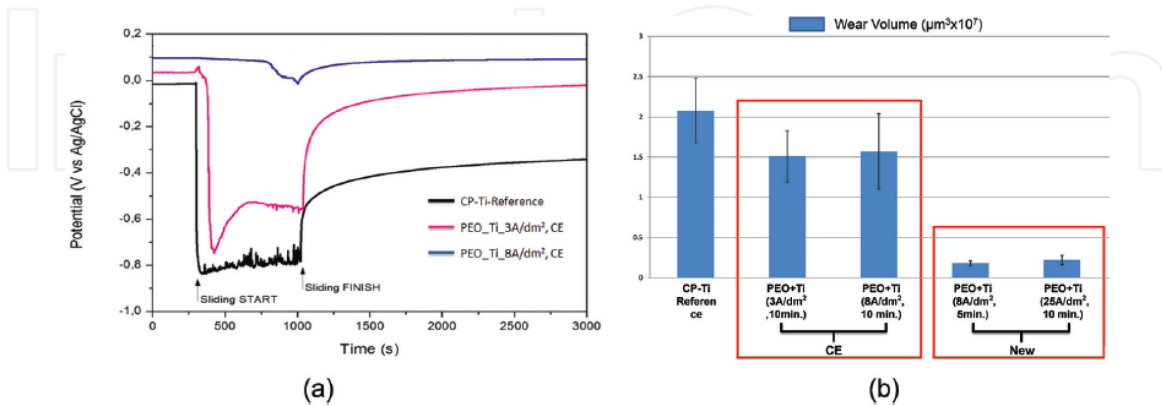


Figure 28.

(a) Tribocorrosion test of titanium (Ti) coated with two types of PEO coatings. (b) Wear volume of tribocorrosion test of Ti and PEO coatings using two electrolytes.

5.5.4 Tactile properties of steel

A similar test protocol mentioned in Section 5.4.3 has also been used to evaluate the steel sheet tactility. The effect of the textured surface geometries of steel sheet surfaces transferred by lamination with laser textured rollers has been studied. The laser texturing was carried out by TEKNIKER. This knowledge has been used in the EU Project STEELTAC [59].

5.6 Tribolubrication

5.6.1 Tribology of environmentally friendly lubricants for cutting and forming applications

Testing protocols has been developed to compare tapping torque results and center of numeric control (CNC) machines to screen torque and wear of cutting and forming fluids and different coatings to increase the efficiency. In the EU

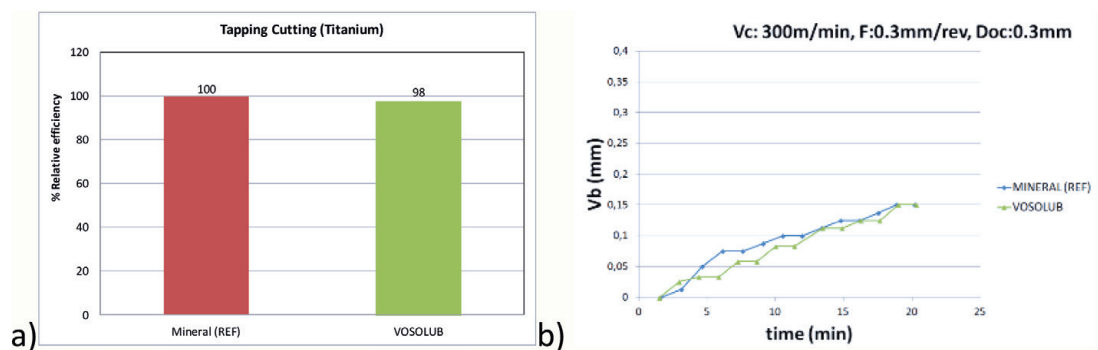


Figure 29.

Comparison of mineral and vegetable lubricants measuring (a) relative efficiency in tapping torque machine and (b) flank wear in CNC grinding machine.

Project VOSOLUB [60], similar tribological properties have been shown between mineral- and vegetable-based cutting lubricants. Both the efficiency of the fluids measuring the torque with the tapping torque machine and the flank wear with a CNC controlled grinding machine have been evaluated (**Figure 29**). The knowledge has been applied to EU Projects IBIOLAB [60], DECOLUB [61], and other related projects such as ECOLUBRO, NANOCOMP, MEFOLUB, and CLAREFOSS.

5.6.2 Tribology of biolubricants for bearings and gears

Testing protocols have been developed to compare the behavior of the biolubricants as alternative to mineral-based oils to lubricate mechanical components (e.g., wind mills, hydraulic pumps, excavators). In the example, a glycol-free lubricant has been developed increasing their load-carrying capacity in comparison with the glycol-containing one. The glycol-free lubricant is biodegradable, and it can be an interesting alternative as a fire-resistant hydraulic oil (**Figure 30**). The protocols have been used in the EU Projects VOSOLUB [38], SUNOIL and BIOGREASE, [39], LUBRICOAT [40], CTVO-NET and LLINCWA [62], BIOMON and IBIOLAB [41], the national projects LUVE and BIOVESIN [63, 64], and the industrial projects ADIVINA, PAVIA, and NANOINTECH.

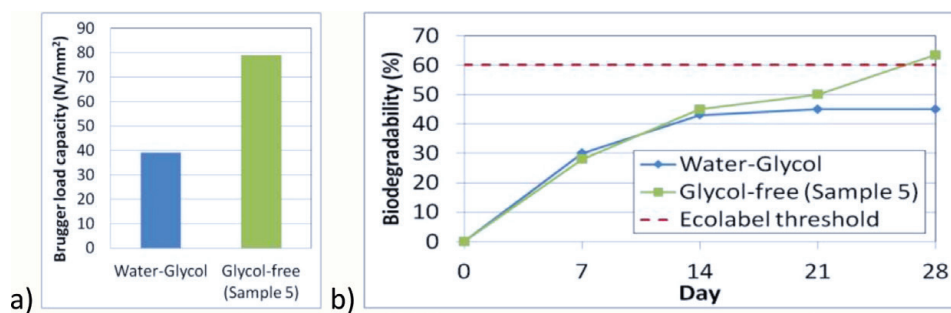


Figure 30.
 (a) Load-carrying capacity of glycol and glycol-free lubricants. (b) Biodegradability of glycol-free lubricants >60%.

5.6.3 Tribology of ionic liquids

Testing protocols has been developed also to evaluate the behavior of ionic liquids as lubricants in different environments studying friction, wear, and tribocorrosion behavior. In **Figure 31**, it is represented the friction and wear scar of the Ionic Liquids DIL3 and DIL7, patented by TEKNIKER, are compared with the hybrid ionic liquid synthesized by TEKNIKER.

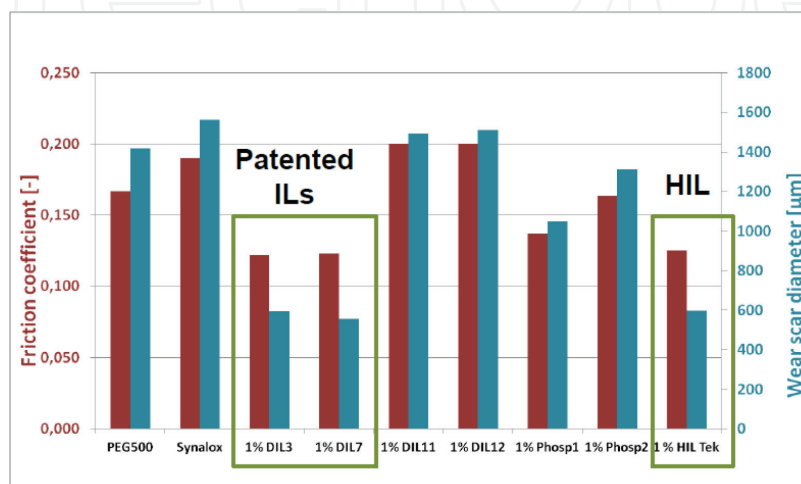


Figure 31.
 In the figure, the friction and wear scar of the ionic liquids DIL3 and DIL7, patented by TEKNIKER, are compared with the hybrid ionic liquid synthesized by TEKNIKER.

DIL7, patented by TEKNIKER (EFS/ID/12720945) and developed in the frame of EU Project MINILUBES Project has been compared with a hybrid ionic liquid synthesized by TEKNIKER, developed in the EMAITEK project, are represented. In this subject, the activity carried out in Eurostar Project VACUUM DLC, the EMAITEK Basque Country Initiative, and the Austrian COMET Project can be also mentioned [65–67].

5.7 Tribology of materials for energy

5.7.1 Tribology for biogas, biodiesel, and bioethanol

Testing protocols has been set up to determine the durability of fuel injectors and other critical engine components of engines working with biogas or suffering lubricant dilution with biodiesel, bioethanol, or their mixtures. In **Figure 32**, it is represented the friction coefficient of the PVD coating Ti-DLC coating deposited by TEKNIKER on injectors, comparing the behavior between diesel and biodiesel (B50, 50% diesel and 50% biodiesel). The protocols have been used in the EU Project Cleanengine and in the industrial project IDEA [68].

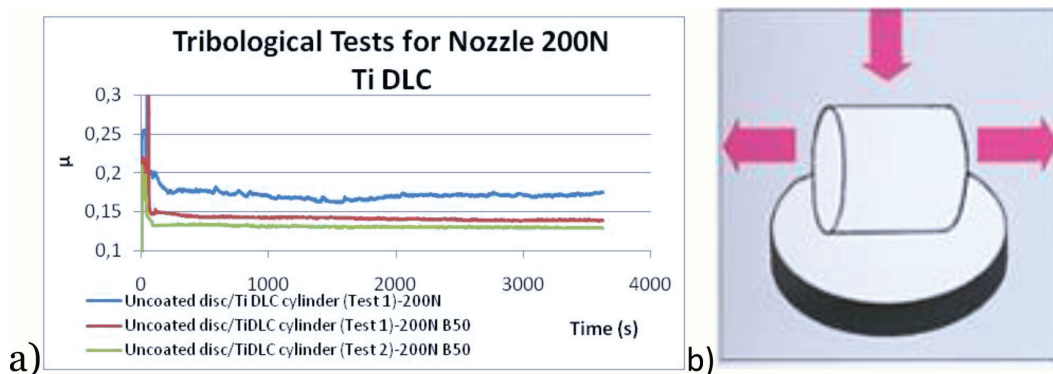


Figure 32.

(a) Tribological properties of the Ti-DLC coating, simulating the contact of injector nozzles with diesel and biodiesel B50; and (b) testing configuration.

5.7.2 Tribology in energy-efficient systems

Testing protocols have been set up to compare the lifetime of critical components of heat exchangers, compressors, gensets, and microturbines. In the example,

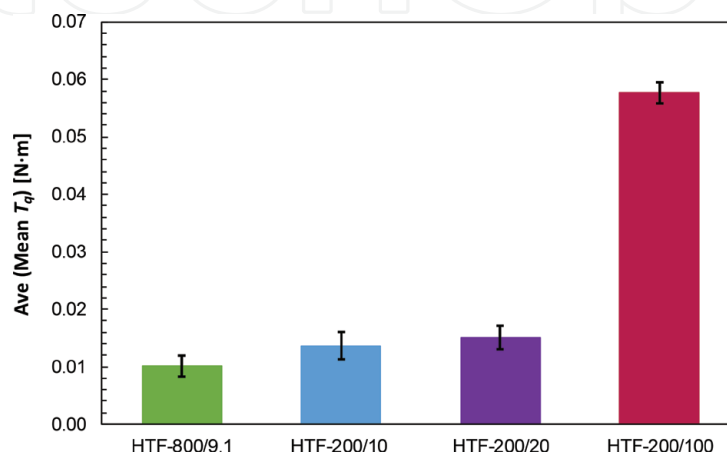


Figure 33.

Average main torque obtained with the drag friction test with different heat exchange fluids of different viscosities (mPa). Error bars represent average standard deviation.

the drag friction test developed by TEKNIKER (**Figure 3**), has been used to evaluate the average torque of different heat exchanger fluids, named as HTF-800 and HTF-200. The last one was tested at different viscosities from 10 to 100 mPa s. The minimum torque was measured for the HT-800 fluid, being finally selected for the application in SUSPIRE Project (**Figure 33**). The protocols have been applied to EU Project HEGEL [68] and to the industrial project ABADIE PLUS [69].

6. Conclusions

Attention to tribological problems would imply worldwide annual savings of 970,000 M€ (1.39% GDP) [69]. A huge range of new testing equipment and protocols have been developed to simulate the friction and wear (mechanical and chemical) of a wide range of mechanical components and systems, in different environments. The knowledge about the working conditions and the failure mechanisms will make possible in the future to simulate at laboratory a still wider list of tribological systems. This will also help us to make a step forward, toward modeling the extrapolation of research results to real systems and the generation of new standards. Artificial intelligence can be also a tool that can facilitate this step forward. Tribology will help to design low carbon footprint materials and implement them in a cost-efficient, predictive, and safe way [70–75]. Tribology will assist also the development of energy- and resource-efficient products and processes, in real systems.

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A summary of 37 years' experience in TEKNIKER.

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