# **REVIEW OF 20 YEARS OF INDUSTRIAL APPLICATIONS OF ION BEAM AND RADIATION TECHNIQUES**

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### Abstract

The present work summarizes the applicative researches performed during 1978s and 2006s in Romania using Ion beam based technologies, radiation based technologies and many other measurement techniques to characterize various technological objects as engines, gear boxes, pumps, tools and materials with emphasis on material loss vs. performances. Tribology studies were performed together with material studies, and assembly performances using a large variety of measurement methods and devices. In order to improve speed and accuracy of tribology thin and Ultra-Thin Layer Activation (U-TLA) method have been used producing a radioactive layer on a selected surface of a technological part, measured for quality assurance with auto-radiography and spectroscopy. The part was mounted in the installation and its radioactivity decrease faster than the natural decay was considered to be due to material loss by wear, corrosion or abrasion.

Measuring in real time all operational parameters opens possibility of determining inter-correlations among them, and improves quality of information obtain by testing. During this 20+ y period several hundred experiments have been performed and testing procedure have been gradually developed following quality assurance principles, being transformed into a turn-key procedure, with clear steps, stages, control means and methods, integrating other nuclear and non-nuclear technologies. Methods as XRF, PIXE, RBS, UTLA, NAA, Gama Spectrometry, were integrated with optical, acoustical, mechanical and electrical methods using automated data acquisition, computer processing and results interpretation, benchmarking the computer simulation and using lessons learned for further improvements, following a spiral of evolution. The results were outstanding, assuring industrial customer with the capability to shorten the time from design to market, cheaper and more accurate than with usual methods which were integrated in the measurement process, together with quality assurance tests, increasing the market competitiveness. This activity ended after 2000 due to drastic decay in demand during EU integration.

# 1. INTRODUCTION

The beginning of tribology one may be considered by year 1900BC during Djehutihotep reign, is known best for the famous decoration inside his tomb that represents the transport of a colossal statue of him that was nearly 6.8 metres (22.3 ft) high, 58 t being transported by 172 workers using ropes and a slide, in an effort that is facilitated by pouring water in front of the slide.

## 1.1. Brief history

By 1943Ferris, is patenting a method to measure material loss using radioactive tracers [1], and since late 1960's neutron activation was used, in spite of its disadvantage of making the labelled parts highly radioactive.

Starting from 1960s, with Cyclotrons started to be used in these tribology applications [2], because of the opportunity to create a thin layer of radioactive material, labelling surfaces of interest for tribology, only and drastically reducing overall part's radioactivity, and implicitly gaining several orders of magnitude in sensitivity and easiness of operation. Renowned nuclear laboratories as KFK [3] and Harwell [4] pioneered the field

By 1977 NIPNE-HH Cyclotron was directed to develop economic contracts to accelerate industrial development by knowledge transfer in joint studies. Staring by 1978 under the leadership of Dr. Ivanov and Plostinaru, Phys. Petru Racolta started a new group aiming to TLA development and pursuing applications in economy. For the beginning the Accelerator Applications group started with the development and homologation of the nuclear method and meanwhile performing incipient studies of wear to industrial research institutes [5-7], as seen in FIG. 1,2. By 1981 he was granted a research stage at KFK Cyclotron, where Dr. Herman Schweickert introduced him to their approach in labelling and tribology related measurement that stimulated his imagination and inspired him in the future work, having KFK as a model. The work environment in Romania was different, from Germany, and he had to improvise and bridge many gaps to get practical results. The early studies started with feasibility tests on rotation of piston rings inside a ICE (Internal Combustion Engine) and lubricant characterization [8] and by 1987 Dr. Racolta sustained his PhD. Dissertation work [9].

#### IAEA-CN-301-143

The NIPNE-Cyclotron Application group developed, and by 1984, I was admitted in, developing engine related applications [10-12]. These researches have been developed in cooperation with specialists from Machines Building Industry and R&D units as Polytechnic Institute, Universities, Engine and hydraulic equipment research groups.

## 1.2. TLA/UTLA project evolution

The research development was random, based on the research contracts and interest of various industrial partners, but was coherent into setting in place all needed pieces of knowledge and procedures as finally to obtain a "turn-key", service performed following Quality assurance norms.





FIG. 2 Main collaborators and project phases map

During 1980s, maturation phase was reached, the method was diversified and applied to a wide range of materials and applications, research team was stable and reached up to 6 researchers at Cyclotron accelerator only, working in interdisciplinary teams with industrial partners, involving technicians also[13,14]. During mid 1980s complementary ion based Analysis (IBA) methods as XRF, PIXE, CPAA and RBS were added complementary [15, 16]. The interest for micro and nano particles analysis in filtration systems and other fluid environments was, addressed in almost every experiment [17], and many communications was made, having many industrial research collaborations as seen in FIG. 1,2. UTLA technique was gradually developed up to mid-1990's [18] as recoil was used for sub-micron surface labelling and for isotope production self-separation in heterogeneous structures. IAEA supported our research efforts with research grants for wear of industrial parts, and characterization of material surface hardening by ionic implantation. BY 1993, we boosted CNRS-CERI activities in the field, during a reciprocal know-how exchange stage.

# 1.2.1. Charged particles Thin Layer Activation dimming and final stages

Starting from 1990 the Romanian industry started its decay, on the premises of industrial equipment scrap & salvage in a wild capitalism as preliminary stage for European Integration mainly as a consumption market as it is today. The decay in the industry was reflected in decay in the demand for new research that continued up to 2000 aggravated by the "brain drain", where most of the researchers left to do "programmatic work" in western laboratories. In spite the method reached the "turn-key", stage with a high level of quality assurance, the industrial research demand plummeted.

After Romania's integration in the European Union, the TLA method was applied to some fundamental researches in bio-medical devices, nano-tribology and calibrations of the method [19], mainly under "life-support" funds from IAEA, as seen in FIG.1. In the new context of global economy, based on very few manufacturers of originally, local researched products, the demand for such measurements drastically decreased, the domain becoming dormant where this domain qualified by government as an expensive service for private sector was not funded from research budgets, that has driven to an end of these activities and an opportunity for closing remarks and lessons learned.

## 2. TYPES OF APPLICATIONS

The "QA turn-key" test protocol presented before have been developed and used for each type of industrial or military application, being diversified and developed based on the previous work lessons learned. There are many applications developed over the time that were improved continuously following an evolution curve, and drawing near to complex application of quality standards.[13,14]

# 2.1. Thermal engines

Various ICE (Diesel and Otto) were studied with respect to improving the running-in, where was possible to bring cost avoiding to company, FIG. 3,4 and lubricant oil wear measurement and classification [10].



FIG. 3. The Crankshaft bearing and Cylinder liner wear measurement

FIG.4 The wear of crankshaft bearing and cylinder liner as a function of running time

Engine operating life study and wear rate dependence on various operating parameters as revolution speed, torque, temperature in water and lubricant, lubricant consumption by burning in engine as function of torque and revolution speed [13,14].



#### FIG. 5 Schematic block diagram of wear measurement by TLA

FIG. 6 The engine test bench – radiation detection setups, electronic control equipment and data acquisition

Various materials and surface treatments used for engine betterment as surface hardening by thermos-chemical treatments as annealing, nitration, boron implant etc., were tested over the time together with a large range of lubricants brands. Equipment evolution runs parallel with test programs developments, allowing us to reach high performances. General measurement scheme is seen in FIG.5, and a complex structure using 26 SCAs, 1 MCA and was measuring wear on 4 parts simultaneously measured in a 4' shielding low background detection system, with combined paper and data recording is shown in FIG.6, where the engine is placed on a test bench left outside the picture. Using the developed equipment in various applications basically we determined the charts briefly presented in FIG.7 where one may see the evolution of wear in the first 90 min. from start of a new engine measured with about 30 s response time, and ng/cm2 sensitivity. Then, in FIG.8 are presented the results for the

entire engine life measurements, (1-2 years 24/7), where all parameters are plotted as function of time and engine's dynamic parameters as (RPM, Torque, temperatures, pressures, flows, etc.). Various lubricant characterization and optimization are made, materials and surface's treatments optimization, is performed.



*Fig. 7 real time running-in measurement the first 1.5h* 

Fig. 8 Engine life-time wear measurements, lubricant and material surface characterization, wear particle magnitude

Using complementary methods as stack filtrations we obtained wear particle's dimensional distributions [14] resulted in various engine circumstances, and further by mili-PIXE and RBS we analysed elemental concentrations and combinations giving the right importance to corpuscular aspects, and related phenomena.



FIG. 9 PIXE on an oil filter deposition FIG. 10 – Pitting detection by double 13MeV d labeling

FIG.11 L27 Engine wear rate as function of Torque & RPM

FIG.9 shows that inside the oil filter the wear particle deposition is larger in the fold due to paper structure and lubricant flow modifications. In FIG.11 is presented a wear rate dependence on engine's RPM and torque, with engine regulated fluids pressure, temperature and flows. To detect the wear type in a surface 13MeV deuteron labelling is used, where by measuring the ratio between the 122keV and 847 keV gamma lines is possible to have a clue of the wear particle shapes and position during dislocation shown in FIG.10 [13].



FIG.13 – Transmission lubricant scuffing pressure measurements.

# 2.2. Gears and mechanical transmissions were studied in various devices

Study of gear's tooth wear as a function of lubricant's scuffing properties, FIG.13 shows a FZG testbed modified for TLA and the results obtained in about 2 weeks to characterize the variation of the scuffing pressure at lubricant film breakdown with aging at a constant average temperature (red horizontal curve).

a. Worm gears' materials and operating regimes

- b. Differential transmissions wear as function of torque and revolution speed,
- c. Study of effect of geometric aberrations on differential wear at microbuses
- d. Ferguson wet transmission wear made by XRF and TLA

# 2.3. c) Study of ball bearings and seals used in engines as:

- a. Ball bearing fabrication dispersion induced wear
- b. Rubber wear of flywheel seal

Gear measurements represented a large activity, studying the tribology aspects that included:

# 2.4. Study of lubricants for various applications as:

a. Lubricants used in gears using TLA enhanced FZG test rig for scuffing load tests

b. Lubricants used in engines as mineral and synthetic oils, additives, by measuring the wear of various engine parts, and

c. Lubricants consumption inside the thermal engine using tritium labelling



FIG.14 –Differential Worm gear details and measurement testbed



In FIG. 14 a worm-gear testbed is presented, and in FIG. 15 is given the wear of a hydraulic pump used in tanks hydraulic transmission on tooth front and back side and on the bearing.

# 2.5. Fundamental Material study on tribologic machines simultaneous with machine improvement by implementing computer control and TLA method as:

- a. Timken machine was used to study
  - i. Surface enhancement by thermo-chemical annealing
  - ii. Wear Mechanism To Friction Contact Of Thermoplastic / Steel Couple
  - iii. Metallic Surface's Wear Mechanism
- b. Falex machine, used to study wear as well material transfer between parts in friction contact
- c. Pin on disc
- d. Four Ball Machine

In FIG. 16 there are given the results obtained measuring the material loss by 4 new methods not usually employed, because traditionally the wear is measuring by weighting the sample disc before and after the wear process. The upper left side is by differential autoradiography, underneath is by profile-meter, by TLA and by height of wheel lever. The most complete was the TLA which when desired the shapes of grains could be also known. The other 4 methods where added as supplementary controls in the novel computer controlled machine which had been built. The TLA chart shows the variation of wear with contact pressure too.



FIG.16 – Timken measurements

Fig. 17. Falex material transfer RBS measurements

### IAEA-CN-301-143

An interesting fact that was shown in FIG. 17, is material transfer by wear. Measured by radioactivity as well by RBS, for a Falex machine using a bronze alloy the peaks of phosphorus (P), Iron (Fe), copper, zinc (Cu, Zn), and tin (Sn) that may be interpreted against Abbott roughness profiles. This result was much more than the researches expected, who were very happy with the measurement speed and accuracy only.

e. Technologic material loss in Fluid environments was studied for Diesel injection systems as Rotary pumps using nitride parts as cam, roles pistons, injectors pin and seat as function of flow and pressure as shown in FIG.18, running on a testbed shown in FIG. 19, testing gas and liquid nitriding material hardening.



FIG.18 Diesel injector and rotary cam wear

i.

FIG. 19 TLA diesel injector testbed

- f. Hydraulic equipment was studied as:
  - Water jet abrasion corrosion of steel and other materials for underwater applications



FIG.20 shows the water jet testbed that measures corrosion, abrasion in jet as function of liquid's temperature as shown in 3D charts on the right. In FIG. 21 there are presented the results using differential autoradiography, for izo-level wear spot profiling vs. profile-meter. A 10 m/s,  $\phi$ 5mm water jet was applied 10 h on a steal plate leaving a corrosion-abrasion crater that was measured at the end, while during test remnant radioactivity measurements were performed continuously [13, 14].

- ii. Cavitation enhanced corrosion in propellers and reversible pump-turbine devices
- iii. Various hydro-transport pumps

### L. POPA-SIMIL

A more complex testbed was made to optimize to wear and abrasion the internal profile of hydraulic equipment as centrifugal pumps and turbines running at smaller scale 1:5 or 1;10, and as function of the wear density results to modify profile as to obtain an optimum material loss density in order to preserve hydraulic parameters the entire operation life. FIG. 22 shows on the right a technician adjusting flow in the measurement testbed loop. The centrifugal pump's blades and disks were labelled with different radioisotopes <sup>56</sup>Co, <sup>57</sup>Co.



FIG.22 - The hydro-transport and reversible turbines TLA compatible testbed

FiG. 23,24 shows disk autoradiography in the middle chart the evolution of material loss along hydraulic tube.

depth [um]

Corrosi

Level

Fig.23 – TLA measurement results, along the profile along hydraulic channel by differential  $\gamma$ spectroscopy and by remnant and concentration method correlated with pump's hydraulic parameters



Pump's operation parameters

H-0=

Fig. 24 – the use of differential autoradiographic method to map the material loss surface density on disks in order to see its variation along hydraulic channel



A sample of the results is presented in Figs. 23,24 in order to show how the profile of hydraulic channel and material wall thickness was optimized in the process.

- g. Behaviour of material in hot gases and plasma equipment as:
- i. Gas inert generator and Gas turbines and nozzles material qualification





FIG. 26 Experimental testbed and calibration

The research had the purpose to produce advanced inert gas generators of maritime applications, and to test materials resilience in fire for gas turbines construction, as seen in FIG. 26, 27. The test bed was a complex structure as seen in FIG. 25, 26 that had the capability to test more than 20 samples simultaneously, based on advanced radiation interference and background calculations and cancelation. Research extended on 3 y.



FIG.26 Material selection for fire resilience

FIG. 27 - Comparative test results

- h. Air exposed metals' corrosion, as depending on air pollution
- i. Maritime oil extraction rig corrosion inside sea-to-sea floor





FIG. 29 - LBE corrosion measurement

FIG. 28 showed the setup of various steel samples placed with belts on the oil-rig in order to estimate the corrosion speed in the sea waters, while FIG. 29 shows the possible designed performances a TLA/UTLA setup may reach studying the corrosion speed in LBE (Lead Bismuth Eutectic) used at FBR (Fast Breeder Reactor) cooling. Other applications include liquid metal corrosion for fast breeder nuclear reactors.

Methodological aspects of UTLA technique were considered together with labelling process material's properties modifications, starting from the very beginning at measurement method homologation, and were improved all the time, based on the lessons learned after each experiment.

## L. POPA-SIMIL





FIG. 31 – Resistive spot Welding cups wear

Military equipment gun barrels, tank engines were also analysed with interesting results, helping improve production quality. Wear in electric parts had a special attention as, spark plugs' material loss, breaker plots flash erosion, contactors rail trolley, resistive welding caps, etc.

Rail way wear measurement was performed as function of load and traffic FIG.30 shows the rail top surface wear as function of position and traffic load. And FIG. 31 shows the material loss in Cu-Be spot-welding inserts measured using a GM detector.

Tools material loss was an interesting application for lathe insert made of TiC synthetic materials.
 Synthetic Basalt wear measurements in slides applications and study of influence of various machining

process on material's surface composition





FIG. 33 – Lathe ceramic insert wear test

In FIG. 32 is presented the basalt slide wear test equipment and results as function of time and applied pressure, and in FIG. 33 one may see the measurements of the material loss in a lathe ceramic insert, made of TiC as function of time, position, removed material thickness for various ceramics compositions.

In fine mechanisms wear study by TLA was applied on cassette recorder head wear, dot/needle printer, sewing machines, and vacuum pumps tests. Technologic material loss in MEMS and NEMS was a subject for future developments. These applications were performed on regular basis and some of them as demonstration tests aiming to obtain contracts for continuous research.

## 3. CONCLUSIONS

The work on TLA started by 1977 and ended after 2010, was led by Dr. P. Racolta who has >80 communications, Dr. L.Popa-Simil with >55 communications and obtained the PhD on this domain, coordinated by Dr. E. Ivanov with >25 communications, and was based on more than 200 industrial research cooperation. The work has benefited from IAEA support, for developing and passing difficult moments, but its existence was dependent on the vigor of local industrial research, whose disappearance drove these works to an end. The main lesson learned

#### IAEA-CN-301-143

is that the high-tech, no matter its performances it is developed in the areas where there is both the culture and the need for using it, that makes the balance between demand and supply. For the period of applications the knowledge gain leaped forward the customers, creating a competitive edge, in an non-uniform development, with many drawbacks that finally pounded the activity, and showed that narrow, cutting edge knowledge is not enough to sustain and maintain a prosperous development of an activity, and all is submissive to general rules of competition in open markets. With IAEA's support at critical moments the TLA activity had a 20 years of prosperous evolution ending with the knowledge of a quality assured turn-key test and research procedure.

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## L. POPA-SIMIL

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