Measurement of surface wear using the thin layer activation (TLA) technique

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Abstract. The thin layer activation (TLA) technique is being used to measure the continuous wear of piston rings and cylinder liners in the nm to μm range. The procedure, effectively using a radioisotope tracer, involves two main steps. Firstly the surface of interest is activated using a charged particle beam and secondly its wear characteristics are tested using a special tribometer set-up. The whole procedure will be discussed along with technical factors to be considered (including the different nuclear reactions used to study different elements) and its limitations. Other known and potential applications will also be described.

1. Introduction

There are many instances when it is important to measure the wear of surfaces. One particular area of interest is the study of engine components such as piston rings and cylinder liners. This enables the motor car industry to investigate the wear resistance and tribological properties of new materials and surfaces with respect to modern lubricants, corresponding fuel dilution and combustion products.

The thin layer activation (TLA) technique [1-8] was developed in the early 1970s. The surface of interest e.g. a piston ring from an engine is labelled with a gamma-ray emitting radioisotope tracer by activating it using a charged particle beam from an accelerator. The piston ring is then used and whilst wear takes place the loss of activity of the piston ring (direct measurement) or the activity of the removed layer (indirect measurement, wear products in the oil) is measured using gamma-ray spectrometry systems. The change in activity indicates how much wear has taken place. This technique has been used to study the wear of engine components [4,9], railway rails [4], cutting edges of turning tools [4] and the corrosion of metallic samples [5,10].

The cyclotron (type MGC-20E) at ATOMKI has been used for several years to carry out TLA studies. It was installed in 1985 with the technical assistance of the IAEA who also supported its reconstruction from 1997 to 1999.

A variation of the above standard TLA procedure to measure the continuous wear of piston rings and cylinder liners outside of an engine in the nm to μm range has previously been developed [11]. It is different in two ways. Firstly the activity of the samples is below the "free handling limit" (FHL) [12] meaning that they are exempt from the normal regulations that apply to radioactive materials and secondly the wear is measured using a modified tribometer (developed at AC^2T research). As part of the WEMESURF (characterization of WEar MEchanisms and SURface Functionalities with regard to lifetime prediction and quality criteria) European research training network, ATOMKI and AC^2T research are using this procedure to study the wear of cylinder liners focusing on the different material phases [13] and on the effect of laser dimples to increase the tribological performance of this tribo-system, and also to study the irradiation of coated piston rings.

2. Experimental

2.1. Thin layer activation (TLA)

The surface of interest is activated using the external beam line of the cyclotron at ATOMKI. The particle beams and energies available are shown in TABLE I. The beam, of diameter 2 - 5 mm, exits into air through a Duratherm window. During irradiations both this window and the sample are cooled using a flow of air. The choice of nuclear reaction (some are shown in TABLE II) to use for the activation depends on several different factors [4]:

- Elements within the sample that can be activated using the available particle beams.
- Half life of the produced radioisotope – it should be long enough to enable the wear measurement to be performed but short enough for cost effective sample handling. In the case of this variation of the standard procedure there has to be a good balance between half life, FHL and necessary minimum activity for a desired resolution of wear rate. In the case of other wear measuring methods using TLA the half life may have a tremendous effect on handling of the radioactive waste.
- Cross section of production of the radioisotope – this should be high to ensure relatively low costs, which is of special importance for routine testing in industry.
- Energy of emitted gamma rays – it is important that the used gamma-ray lines from different radioisotopes are clearly separated from each other and do not overlap.

TABLE I: Particle beams available.

Particle	**Energy (MeV)**
p	2.5 – 18
d	1 – 10
^{3}He	4 – 26
α	2 – 20

TABLE II: Some of the nuclear reactions used.

Nuclear reaction	**Max. x-section (mbarn at MeV)**	**Half life (days)**	**γ-ray lines (keV)**	**FHL (MBq)**
$^{nat}Fe(p,x)^{56}Co$	396 at 12.5	77.3	847, 1238	0.1
$^{nat}Fe(d,x)^{57}Co$	310 at 7.2	271.8	122, 136	1
$^{nat}Cu(p,x)^{65}Zn$	220 at 10.5	244.3	1115	1
$^{nat}Cr(d,x)^{51}Cr$	700 at 6.7	27.7	320	10

Normally the energy of the beam should be chosen so that the cross section is near the maximum and the calibration curve (activity versus depth which is obtained from the cross section and energy loss of the incident particles) is as linear as possible. *FIG. 1* shows the calibration curve for a Cr layer i.e. that of a coated piston ring that has recently been irradiated at an angle of 15°. Here a deuteron beam of energy 7.3 MeV was chosen which has resulted in a homogenous ^{51}Cr activity distribution from the $^{nat}Cr(d,x)^{51}Cr$ nuclear reaction down to

~4 μm i.e. unit loss of activity corresponds to unit loss of material due to wear. For these tests the maximum wear of interest is less than 4 μm.

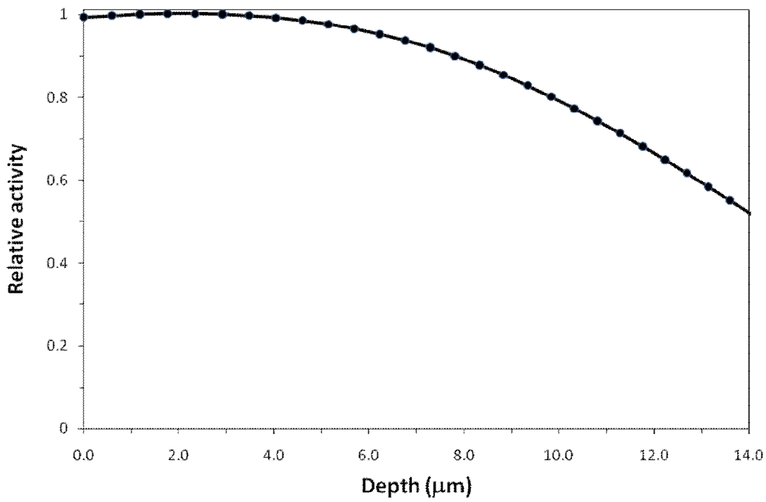

FIG. 1. Calibration curve for Cr from the $^{nat}Cr(d,x)^{51}Cr$ *nuclear reaction for a deuteron beam of 7.3 MeV and an irradiation angle of 15°.*

For this procedure the activity of the sample is also kept below the FHL. This means that the radiological risks to individuals handling the samples are sufficiently low to be of no regulatory concern and that the samples are therefore exempt from the regulations that apply to usual radioactive materials [12]. Due to this they do not require any special handling and can be used in any laboratory. This limit varies for different radioisotopes and depends on their gamma-ray energies and half life. Compliance with this requirement is checked using gamma-ray spectroscopy with a HPGe detector. Straight after irradiation there is normally a “cooling down” period to allow short lived radioisotopes to decay ensuring that the activity is below the FHL.

For wear measurements in the nm to μm range the activity should be concentrated near the surface of the sample to increase the amount of activity removed during the wear process. This can be achieved by decreasing the irradiation angle from normal to glancing incidence e.g. 15° as was done for the Cr coated piston ring, which has the effect of reducing the depth of activation and increasing the sensitivity of the technique thus allowing a lower sample activity to be used.

2.2. Measurement of wear using the tribometer set-up

The components of interest e.g. piston ring and cylinder liner are mounted in the modified tribometer at AC^2T research, with the activated zone located at the contact point, as shown in *FIG. 2*. The tribometer simulates the running of an engine by moving, in this case, the piston ring back and forth on the cylinder liner while a flow of lubricant passes through the contact zone. The lubricant goes to a reservoir which is located inside a NaI(Tl) gamma-ray well detector and is then pumped back to the contact zone. As wear takes place activated particles are detached from the surface of interest and taken away by the lubricant. The number of gamma rays of a particular energy from the chosen radioisotope detected in the lubricant indicates how much wear of that selected element has taken place. Software is used to record the number of gamma rays detected as a function of time (typically integrated over periods of tens of seconds). From this and with knowledge of the depth profile of the radioisotope in the material (calibration curve) and the contact area the wear depth and rate can be calculated. This set-up and the wear depth calculation method are known as nano-scale wear Volume Coherence Technology (nVCT) [11, 13-15].

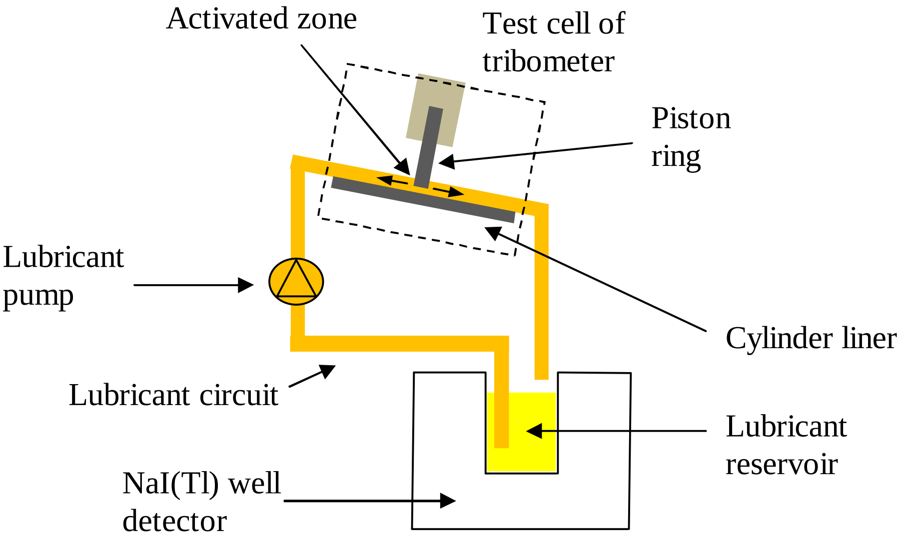

FIG. 2. Schematic of the tribometer set-up.

In order to simulate different engine conditions the parameters of the tribometer such as the load on the contact point, stroke width and frequency, and length of running time can be varied. The lubricant can also be heated before entering the contact zone in which case it is water cooled in the gamma-ray detector to prevent damage to the NaI(Tl) crystal.

3. Applications

With the set-up described, wear of Al-Si-Cu alloy (main composition 76% Al, 18% Si and 3.5 - 4.5% Cu) cylinder liners with engine oil or diesel as the lubricant have been studied [13] by activation of the Cu content using the $^{nat}Cu(p,x)^{65}Zn$ reaction. Cu was activated because of the high cost of ^{3}He for using the $^{nat}Al(^{3}He,x)^{22}Na$ reaction and because the $^{nat}Al(p,x)^{22}Na$ reaction has a threshold of 25 MeV which cannot be reached with the present cyclotron. The $^{nat}Si(p,x)^{22}Na$ reaction has a threshold of around 30 MeV and hence could not be used.

Piston ring (CrMoV steel type) wear for different engine oil – biodiesel lubricant mixtures [14] has also been studied using the $^{nat}Fe(d,x)^{57}Co$ nuclear reaction.

Other components presently under investigation include Cr carbide steel (100Cr6) ball bearings and Cr coated piston rings that have been activated using the $^{nat}Cr(d,x)^{51}Cr$ reaction. In the case of coated samples it is important to avoid activating the bulk material, gamma rays from which may interfere with those from the activated coating or cause the FHL to be exceeded. The depth of activation can be reduced by decreasing the irradiation angle or by decreasing the energy of the beam. If there is a choice of possible reactions using different incident particles then the penetration depth of these in the sample should also be taken into consideration.

4. Discussion

Using TLA with a tribometer set-up, engine components can be tested continuously outside of an engine hence making tests easier, quicker and cheaper to perform. The capability to measure continuously avoids problems associated with starting and stopping of the tests which can occur with other non-continuous methods [11].

Through careful selection of the nuclear reactions used, it is possible to study the wear of different elements simultaneously either in the same or different samples e.g. elements in the piston ring and cylinder liner. The Fe in both of these could be studied simultaneously by using the two nuclear reactions $^{nat}Fe(p,x)^{56}Co$ and $^{nat}Fe(d,x)^{57}Co$ which result in radioisotopes that emit gamma rays of different energies.

The cyclotron at ATOMKI is also used to measure the cross sections of nuclear reactions that are of relevance to TLA e.g. (d,x) reactions on Fe [16] among numerous others [17]. These cross sections are available from the EXFOR library, Nuclear Data Services section of the IAEA [18].

The TLA technique is only suitable for studying the wear of metals and alloys i.e. materials that can be activated by small accelerators. Polymer materials can be studied by implanting suitable radioisotopes using a recoil implantation technique [19-20]. This has been used to study polyethylene components for use in artificial joints [21].

5. Acknowledgements

The authors would like to acknowledge the support of the European Commission through the project MRTN-CT-2006-035589 WEMESURF.

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