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USE OF RADIOACTIVITY FOR DETERMINING MECHANICAL PROPERTIES IN SURFACE ANALYSIS

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The study of mechanical properties of surfaces has been facilitated in recent years by nondestructive radioactive techniques, which monitor the wearing away of γ-emitting species from the surface. Such species can be produced directly by surface-layer activation or indirectly by implanting the products of a nuclear reaction. Each technique has its strengths and weaknesses, and together they allow the study of wear in a complex device such as an engine without having to disassemble it.

The study Of beam interactions with materials and surfaces is an immense field of study; indeed, all of *Nuclear Instruments & Methods in Physics Research, Section B,* is devoted to this field. Most research, however,: has been on the chemical, defects, impurities, or electronics properties, with relatively little study of simple physical and mechanical properties, such as hardness and wear. One might well ask, why bother with ion beams and/or radioactivity for the study of such straightforward mechanical properties? Is this not a case of overkill, of having an available technique and then trying to find a use for it? In this paper I shall try to justify and promote two viable and economical techniques for the study of wear in surfaces.

Inherent in the design and construction of internal combustion engines, rocket engines, and many other complex devices subject to long-term, isolated mechanical use is how to improve the wearing properties of areas under high stress or friction. One needs to be able to measure the wear under laboratory conditions in a reasonable amount of time, then project these results to field conditions and long-term expectations. This has led to the new science of "tribology," or wear studies, where many ingenious techniques have been developed. Many of these. such as laser techniques, are extremely sensitive, measuring wear well down into the nm range or lower. Unfortunately, most of these techniques require access to and disassembly of the apparatus in order to make direct measurements on the affected components. In addition to requiring expensive downtime and access, this introduces serious uncertainties into the data, for exact reassembly and repositioning is all but impossible. Thus, reproducibility from such techniques tends to be rather poor. One might also ask, why not install some sort of permanent sensor in order to provide continuous monitoring? Again. installing permanent internal sensors not only can alter normal wear patterns, but also most sensors are not capable of withstanding the extremes of temperature and stress present in, say, an engine.

This leads us to investigate techniques that monitor wear through the disappearance of radioactive species from activated parts; these techniques eliminate some of the above

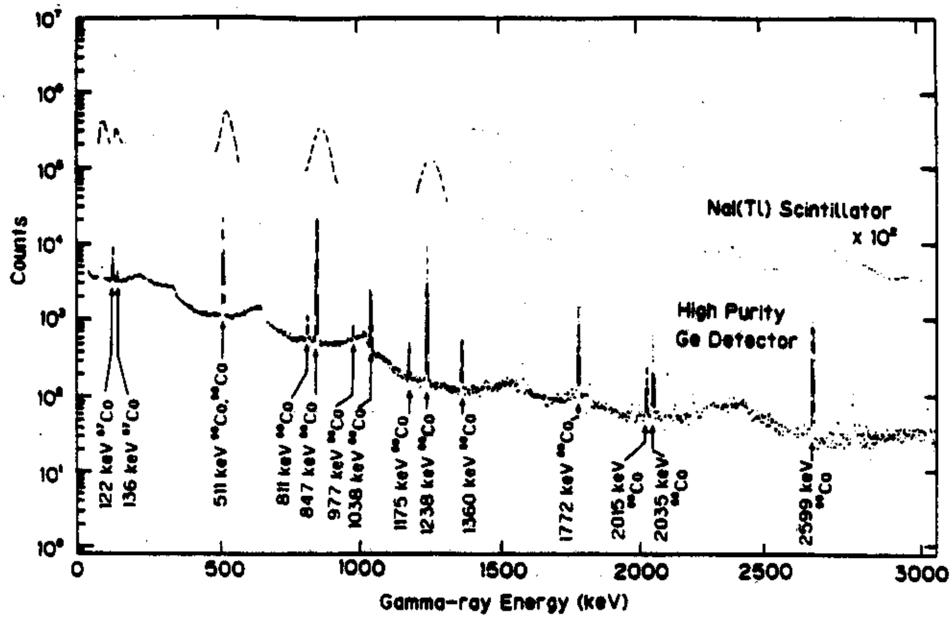

Fig. 1. Gamma-ray spectra of Co activities produced by SLA on Fe samples.

clearly, the NaI(Tl) detector was not able to resolve the peaks satisfactorily. Figure 2 shows the dose-depth profiles for all three radioactive species obtained using the Ge detector. The dose-depth profile for the foils is a true dose-depth profile, while that for the Fe block was obtained by polishing away the surface (using a polishing machine with 600-mesh emory paper) and counting the residual activity point by point.

From Fig. 2 we see that the dose-depth profile for ^{56}Co differs considerably from those for the other two isotopes and disappears at a depth of about 50 μm. This comes about because of the higher Q value for the reaction producing ^{56}Co (5.4 MeV as opposed to ≈1.6 MeV for the other two). Actually, this could be turned into a sort of experimental advantage, for one can determine the depth from a ratio of the isotopes rather than from a more tedious and careful experiment. However, this advantage is more theoretical than real, for it involves counting the very low levels of low-energy γ rays coming from the other two isotopes.

One further observation should be made at this point. Proponents of SLA have made a particular point about the fact that their dose-depth profiles are "rectangular." This has the advantage that it allows one to study the "average wear" of a system. There are two distinct schools of thought on studying the wear in an engine: direct monitoring of the residual activity remaining on a part (a "differential" technique) vs monitoring the activity present in an effluent, such as the engine oil (an "integral" technique). The obvious advantage of the integral technique is that it does not require taking the engine apart and can be performed continuously as the engine is running. Now, if one activates multiple spots on the inside of an engine, then, *provided the dose-depth profiles are all rectangular*, the integral technique allows one to measure the wear averaged over these various spots; otherwise, the deconvolution process is too complicated to be practical. However, from Fig. 2 we can see that the dose-depth profiles are by no means rectangular, especially the one for ^{56}Co, the major activity of interest. Thus, SLA may or may not be a viable technique for easily measuring average wear.

An in-the-field set of measurements[6] is summarized in Figs. 3 and 4. At Fleetguard Corp. (Tennessee, USA), heavy-duty diesel engine components were irradiated as follows:

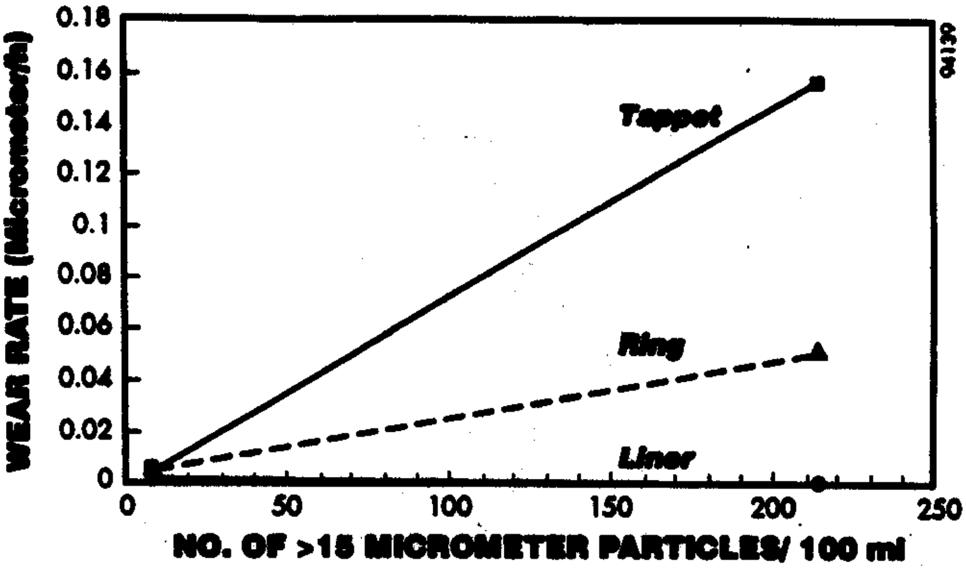

Fig. 4. Relationships between wear rate and concentration of >15 μm particles in oil for components of the activated top ring in a heavy-duty diesel engine. Laboratory simulated operation for 96,000 km of vehicle travel. Obtained by SLA.

a top piston ring, to produce 3 μCi of 312-d ^{54}Mn deposited to a depth of 35 μm; the corresponding cylinder liner, 10 μCi of ^{56}Co to 15 μm; and the sliding tappet cam follower, 3 μCi of ^{57}Co to 25 μm. The engine was then run under laboratory conditions, and the effluent oil was monitored by a NaI(Tl) γ-ray detector. As shown in Fig. 3, this detector was adequate to monitor the three individual activities, although a fair amount of peak resolution and background subtraction was necessary. Many different experiments were performed, using a variety of different loads, different oils, and different levels of particulate impurities in the oils. The results of one such experiment are shown in Fig. 4. Here the engine was run under conditions simulating 96,000 km of travel with oil containing >15 μm particulate impurities (normal engine wear particulates, the size selected by filters). The figure shows a significant difference in wear rate before and after cleaning the oil, and the wear rates for the different components also differ significantly. Thus, here is a situation where the integral SLA technique could and did simultaneously monitor wear rates on different parts, made possible by selectively producing different radioactive species in the different parts.

Radioactive-Ion Implantation

A few years ago a group of us at NSCL[7] developed RII as an alternative method to SLA. When a relatively high-energy (several tens of MeV or greater) beam of heavy-ions strikes a foil, it fragments, and many of the resulting fragments travel forward (in the beam direction). A graphic illustration of this is shown in Fig. 5, which shows a ^{12}C striking an atom in a photographic emulsion.[10] Depending on the nature of the beam itself, of the target, and to some extent on the energy of the collision, a variety of radioactive fragments can be obtained, but the most useful by far turn out to be 53-d ^{7}Be and 2.6-y ^{22}Na, both of which have easily detectable, intense γ rays and long enough half-lives to make their use practicable. Figure 6 shows the type of spectrum one can obtain with a Ge detector.

In Fig. 7 we show a schematic representation of our current experimental apparatus. The components are mounted inside a stainless steel evacuated chamber attached near the

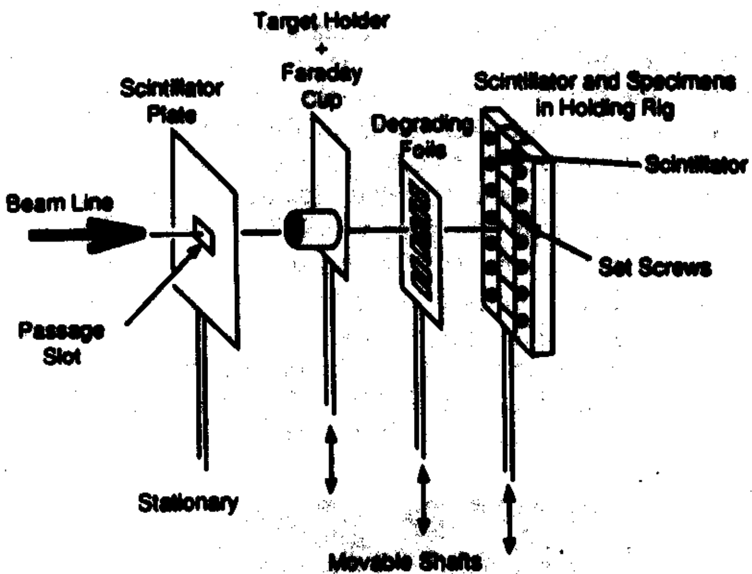

Fig. 7. Schematic representation of the components inside the bombardment chamber for producing radioactive fragments for implantation.

with a Ge detector, and the doses produced in the bombardments were determined by correlating the spectra for each foil and making the standard corrections for half-lives, branching ratios, and efficiencies.

The dose-depth profiles for both ^{7}Be and ^{22}Na are presented in Fig. 8. For ^{7}Be the profile sharpens with decreasing bombarding energy, but so does the production cross-section. Of these three beam energies, 30 MeV/*A* offers the best compromise, but an energy between 18 and 30 MeV/*A* may prove optimum. All three ^{22}Na profiles are considerably simpler than those for ^{7}Be. Here the lowest beam energy also produces the best (sharpest and closest to the surface) profile. (This undoubtedly comes about because ^{22}Na is produced by much simpler nuclear reactions than the complex fragmentation processes necessary to produce ^{7}Be. With lighter beams such as ^{14}N, whose *A* lies closer to Be, the profiles for ^{7}Be also become simpler.[7])

^{7}Be and ^{22}Na complement each other as implanted ions for tribology:

1) The ^{22}Na dose-depth profiles tend to be sharper, making it more suitable for obtaining "average wear" by integral techniques and as discussed in the section on SLA.

2) ^{22}Na can be produced at lower bombarding energies, viz., with cheaper accelerators, and with shorter bombardments, because of its larger production cross-sections -- at least, as indicated thus far by the particular beams we have studied. With currently available beams at NSCL, we can produce a *usable* implanted dose ($\approx$0.1 μCi) with a bombardment time of considerably less than 1 h.

3) On the other hand, it is more difficult to stop -- or separate out -- the primary beam in ^{22}Na production. The d*E*/dx values, on which these separations depend, differ more as the percentage differences in *A* between target and fragments increase, and this is easier to arrange for lighter fragments, such as ^{7}Be, for similar types of reactions. As an example, the difference in d*E*/dx values for ^{20}Ne and ^{22}Na is not great enough to allow separation, so if one were to use a ^{20}Ne beam (^{22}Ne would be even worse), one would have to use additional elements, such as a bending magnet between target and implantation site, to filter out the primary beam.

One other illustrative experiment: To demonstrate the viability of RII for ceramics, we implanted a 1.91-cm (3/4 in.) diameter, 1.27-cm (1/2 in.) thick disk of silicon carbide,

Determining Surface Damage

Since we advertise that one of the advantages of RII over SLA is that RII does not produce appreciable surface damage with ion doses at the level needed for wear studies, we needed to demonstrate this experimentally. We ran a series of experiments in which we bombarded a metastable material, which would be easily susceptible to induced surface damage.[12] For this, we chose a system produced by quenching specimens of Fe-17Cr-4Ni-Cu stainless steel, which produces a metastable martensite structure. This microstructure, consisting of a lath martensite matrix containing δ-ferrite stringers, is subject to substantial changes in mechanical properties when given isothermal annealing treatments at temperatures as low as 755 K. Thus, it should be particularly susceptible to beam damage, and the changes should be readily measurable by techniques such as nanoindentation hardness studies.

Blocks measuring $15.85 \times 12.45 \times 3.18$ mm machined from an as-quenched bar of 17-4PH stainless steel were polished using Buehler 30 μm, 6 μm, 1 μm, and Mastermet diamond abrasives. These were then irradiated, using direct implantation of 2.5-MeV/*A* $^{20}Ne^{2+}$ ions at an average beam current of 5.8 nA, which required ~3 h to accumulate a dose of 2×10^{14} ions/cm^2. ^{20}Ne ions were chosen because they were readily available and approximate the effects of implanting ^{22}Na very well.

Nanoindentation hardness and modulus measurements were carried out at the High Temperature Materials Laboratory at Oak Ridge National Laboratory, using the Mechanical Properties Microprobe nanoindentor. Indentations were made with a constant displacement rate of 10 nm/s, and hardness and elastic modulus calculations were made at indent depths of 50, 100, 150, and 200 nm. Both the irradiated regions and controls taken from unirradiated regions on the same materials were examined. In addition, optical and scanning electron microscopic examinations were made, and the modified region was investigated for new phases by x-ray diffration.

Some results are shown in Fig. 9, which shows data for nanoindentation hardness traverses with 200-nm indents for doses of 2×10^{12}, 2×10^{13}, and 2×10^{14} ions/cm^2. A pronounced dip in the hardness profile is apparent for the 2×10^{14} ions/cm^2 irradiation,

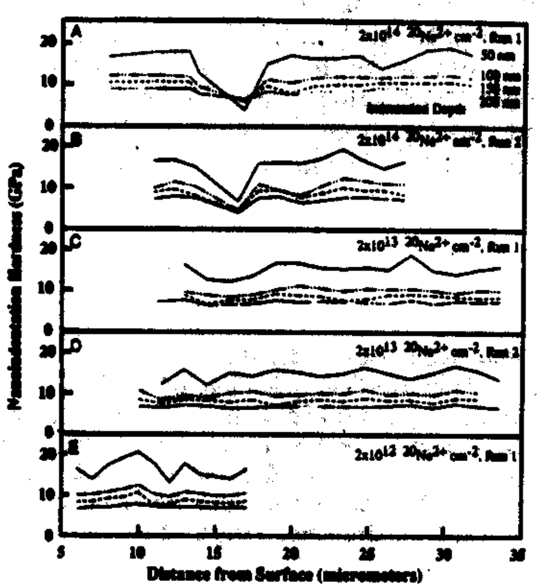

Fig. 9. Nanoindentation hardness profiles for matermsitic 17-4PH stainless steel irradiated with differing doses of 2.5-MeV/*A* $^{20}Ne^{2+}$ ions, demonstrating that RIII does not produce appreciable surface alteration at useful dose levels.

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