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ACTIVATION EN COUCHE ULTRA MINCE PAR IMPLANTATION PAR RECUL D'IONS LOURDS RADIOACTIFS : APPLICABILITE POUR LES ETUDES D'USURE ET DE CORROSION

ULTRA THIN LAYER ACTIVATION BY RECOIL IMPLANTATION OF RADIOACTIVE HEAVY IONS : APPLICABILITY IN WEAR AND CORROSION STUDIES

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EDF
Electricité de France

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GUINARD L.
LACROIX O.
SAUVAGE T.
BLONDIAUX G.

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SYNTHÈSE :

Une nouvelle procédure d'étalonnage est proposée pour l'application de l'implantation par recul d'ions lourds radioactifs (énergies entre quelques centaines de keV et quelques MeV) dans la proche surface des matériaux, dans le cadre d'un programme de recherche sur les phénomènes d'usure ou de corrosion submicrométrique. Le profil en profondeur des radioéléments implantés est déterminé à l'aide de films ultraminces déposés par pulvérisation cathodique sous plasma d'argon. Les deux courbes de l'ion ^{56}Co dans le nickel relevées pour des profondeurs d'implantation de 110 et 220 nm respectivement démontrent la faisabilité et la reproductibilité de cette méthode pour ces profondeurs activées. Les sensibilités atteintes par la détection des pertes de surface sont d'environ 1 et 2 nm respectivement. La détection en ligne s'effectue directement sur l'échantillon étudié.

Une description générale de la méthode est présentée. Une étude de la cinématique de la réaction, suivie d'un traitement général des paramètres d'irradiation à adopter, sont également développés avec l'intention d'employer la méthode d'activation des couches ultraminces (UTLA) dans d'autres applications de recherche et industrielles.

EXECUTIVE SUMMARY :

A new calibration procedure is proposed for the application of recoil implantation of radioactive heavy ions (energies between a few hundred keV and a few MeV) into the near surface of materials as part of a research programme on submicrometric wear or corrosion phenomena. The depth profile of implanted radioelements is performed by using ultra thin deposited films obtained by cathode sputtering under argon plasma. Two curves for ^{56}Co ion in nickel have been determined for implantation depths of 110 and 200 nm, respectively, and stress the feasibility and reproductibility of this method for such activated depths. The achieved surface loss detection sensitivities are about 1 and 2 nm respectively. The on line detection mode is performed directly on the sample of interest.

A general description of the method is presented. A study of the reaction kinematics followed by a general treatment on the irradiation parameters to be adopted are also developed with the intention of using the ultra thin layer activation method (UTLA) to further applications in research and industry.

Ultra thin layer activation by recoil implantation of radioactive heavy ions: applicability in wear and corrosion studies

O. Lacroix [a], T. Sauvage [a], G. Blondiaux [a], L. Guinard [b]

[a] *CNRS, Centre d'Etudes et de Recherches par Irradiation, 3A, rue de la Férollerie, 45071 Orléans, France*
[b] *EDF, Direction des Etudes et Recherches, Département Etude des Materiaux, 77250 Moret-sur-Loing, France*

1. Introduction

Technological improvements in the fields of passivation and treatment of surfaces lead to development of increasingly sensitive methods of surface wear and corrosion measurements. For about twenty years, activation by neutrons or charged particles have been applied to tribological and corrosion phenomena studies. The best sensitivities obtained are of the order of a nanometer, but they require a detection mode on fluids [1,2]. The application of these methods is therefore limited to phenomena where a fluid is available to transport radioactive wear debris to a detection point. However, these very sensitive methods are rather imprecise because of sedimentation and trapping phenomena along the closed fluid circuit [3].

Thereby, the UTLA method based upon recoil implantation of radioactive heavy ions into the near surface of materials has been developed with the intention of having access to nanometer sensitivities without use of fluids as a transport vector. The method enables an on-line detection on the experimenal sample for nanometer wear or corrosion rates. In 1980, Conlon [4] introduced the ultra thin layer activation and proposed a general treatment. A few experiments concerning implantation rates of radioactive recoiling ions by the nuclear reaction $^{56}Fe(p,n)^{56}Co$ have also been performed by Leterrible [5], and a theoretical depth profile determination of the recoiling radioelements has been considered. Yet, according to the literature, it appears that no experimental calibration curve and no development of UTLA techniques to wear or corrosion rates studies have been realized.

The near-surface activated depth (usually some tens to several hundreds nanometers) does not permit the use of calibrated stacked thin foils for the depth profile determination of the implanted radioactive heavy ions. The originality of the calibration procedure presented in this report resides in the use of very thin films deposited on a substrate. The films are deposited on a light backing by cathode sputtering and have a similar composition to the sample to be investigated. The thicknesses are measured by Rutherford backscattering.

By associating the calibration procedure with the near surface activation, the UTLA method opens new perspectives and turns out to be a prolific tool, henceforth applicable to the study and measurements loss of material in the nanometer range. The fields of application involved are numerous: biomaterials, micromecanics, corrosion, etc.

2. Ultra thin layer activation method

2.1. Principle

The UTLA method is based upon the principle of recoil implantation by recoiling applied to radioactive heavy ions

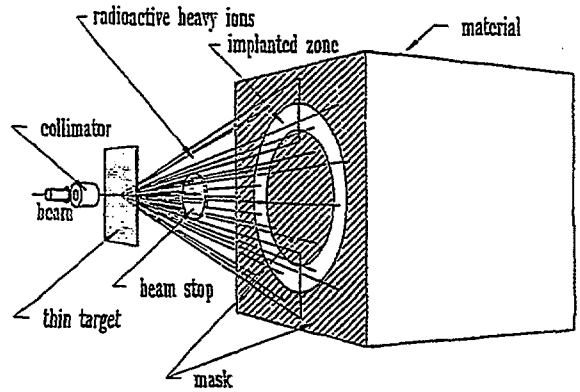

Fig. 1. Frontal implantation geometry.

generated by a beam of light mass particles (p, d, ^{3}He, ^{4}He). These charged particles are easy to produce by an isochronous variable energy cyclotron. The most commonly used energies do not exceed 40 MeV.

A thin target (a few micrometers) of elementary composition A is bombarded by the primary beam and is activated following the nuclear reaction **A(a,b)B**. Some generated radioactive heavy ions B acquire sufficient kinetic energy (maximum energies of a few hundred keV to a few MeV) to recoil out of the target and be implanted in the material. The implantation may be realized with the sample of interest in frontal (Fig. 1) or in tubular geometry (Fig. 2). After having penetrated the thin target, the beam is stopped by a Faraday cup, which can be located upstream or downstream of the implanted zone.

2.2. Calibration procedure

Films of composition similar to the sample of interest are deposited on silicon substrates by cathode sputtering. The pair film–substrate is positionned in a well defined implantation zone. This irradiation geometry must be similar to the investigated sample set-up.

After irradiation, the total radioactivity B_0 implanted in the pair film–substrate is measured with an HPGe detector.

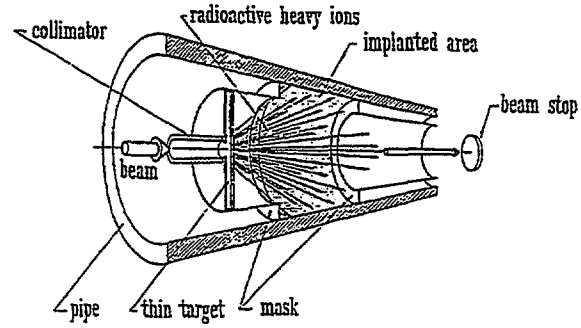

Fig. 2. Tubular implantation geometry.

A chemical attack specific to the pair film–substrate enables to dissolve the film avoiding any transfer of radioactive ions from the substrate to the chemical solution. The radioactivity B of the one substrate is then measured under the same detection conditions. The ratio B/B_0 expresses the relative remanent radioactivity after a surface loss equal to the film thickness. The irradiation of various thickness films enables to establish the correlation between the remanant radioactivity and the surface loss $B/B_0 = f(x)$.

Several reasons have led us to choose silicon as substrate. For instance, the low atomic number ($Z = 14$) of silicon allows us to obtain a good precision of the thickness measurements by the RBS method for most metallic films. In addition, the silicon reacts weakly with common acids and facilitates the films chemical dissolution.

The oxidization rate in the volume and the homogeneity in the depth of the films are important parameters to obtain a depth profile of the implanted radioactive ions similar to the one of the samples to be investigated.

3. Energy and angle spectra of the radioactive ions

3.1. Kinematic study of nuclear reactions: theoretical energy-angle spectrum

In case of a two-body reaction which may be written as A(a,b)B, the rules of energy and momentum conservation enable to calculate the kinetic energy of the final state particles as a function of their emission angle. The kinetic energies of the incident particles (p, d, ^{3}He, α) accelerated in a cyclotron being low, the kinematic study of such a nuclear reaction where B is the radioactive recoiling ion is realized by using classical mechanics.

By calling M_1, M_2, M_3, M_4, E_1, E_2, E_3, E_4 the respective masses and energies of the nuclei a, A, b, B and ϕ, θ the emission angles of the nuclei B and b (Fig. 3), the recoiling energy E_4 of the radioactive heavy ion is given as a function of its emission angle in the laboratory frame by the following equation:

$$\sqrt{E_4} = \frac{\sqrt{M_1 M_4 E_1}}{M_3 + M_4}\cos\phi \pm \left\{ \frac{M_1 M_4 E_1 \cos^2\phi}{(M_3 + M_4)^2} + \frac{M_3 Q + E_1(M_3 - M_1)}{M_3 + M_4} \right\}^{1/2} \quad (1)$$

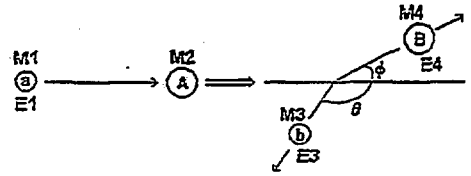

Fig. 3. Laboratory frame.

with

$$Q = M_1 + M_2 - (M_3 + M_4). \quad (2)$$

From Eq. (1), we can deduce:

if $[M_3 Q + E_1 (M_3 - M_1)] > 0$, there exists for an angle ϕ a single energy value of the recoiling ion.

$$\sqrt{E_4} = X \cos\phi + \sqrt{X^2 \cos^2\phi + Y}$$

with

$$X = \frac{\sqrt{M_1 M_4 E_1}}{M_3 + M_4},$$

$$Y = \frac{M_3 Q + E_1 (M_3 - M_1)}{M_3 + M_4},$$

if $[M_3 Q + E_1 (M_3 - M_1)] < 0$, there exists for an angle $0 \le \phi < \phi_{crit}$ two possible energy values of the recoiling ion.

The critical angle ϕ_{crit} i.e. maximum emission angle of the recoiling ion is defined by the following equation:

$$\phi_{crit} = \arccos\sqrt{\frac{M_3 + M_4}{M_1 M_4 E_1}\left(-M_3 Q - E_1 (M_3 - M_1)\right)}.$$

The energy E_{crit} relative to the critical angle is given by the following relation:

$$E_{crit} = \frac{M_1 M_4 E_1}{(M_3 + M_4)^2} \cos^2\phi.$$

A minimum energy E_{4min} and a maximum energy E_{4max} hence appear at the emission angle $\phi = 0$ given by:

$$\sqrt{E_{4_{min}}} = X - \sqrt{X^2 + Y},$$

$$\sqrt{E_{4_{max}}} = X + \sqrt{X^2 + Y}.$$

In the previous formula, the masses M_1, M_2, M_3, M_4 are the nuclei ones and are expressed in MeV/c^2 with $c^2 = 1$, the kinetic energy E_1 of the incident particle is expressed in MeV.

For the above calculations, the particles are supposed to be in their ground state. In the UTLA method, this condition is valid except for the radioactive heavy nuclei which can present an excited state [4,6]. The value of the energy balance Q is thus modified and defined by the following relation:

$$Q = M_1 + M_2 - (M_3 + M_4^*)$$

with $M_4^* = M_4 + E_4^*$ where E_4^* is the excitation energy.

3.2. Emerging angle and energy spectra

As the range of generated ions is less than the target thickness, the emerging energy spectrum of the recoils is continuous, ranging from the maximum energy E_{4max} to zero. Due to multiple scattering the emerging angular spectrum is not limited to the range [0, ϕ_{crit}].

For a nuclear reaction at a given energy, the differential cross sections relative to each excited state of B affects directly the angle and energy distributions of the ions emerging from the target. In the UTLA method, the calibration curves depend on these distributions and on the irradiation geometry (beam diameter, implantation cone, incidence angle). To determine the importance of each parameter, a computer Monte Carlo simulation, associated to TRIM95 [7] for the transport of particles in matter, is under study.

4. Application of the UTLA method

The nuclear reaction ^{56}Fe(p,n)^{56}Co has been chosen to apply the UTLA method because of its high cross section. Moreover, the half-life $T = 77.1$ days and the 846 keV gamma ray (branching ratio $f_\gamma = 99.9\%$) of the ^{56}Co radioelement is well suited for this study. The energy spectrum obtained from Eq. (1) gives for 13 and 18 MeV primary beam, respectively, a critical angle of 49.6° and 56.7° (Fig. 4).

4.1. Experimental set-up

The recoil implantation revealing a cylindrical symmetry about the beam axis, six implantation areas of a 6 mm diameter have been arranged according to the cylindrical geometry.

Their positions from the beam axis define an implantation cone whose half angle is between 30 and 40° (Fig. 5). A collimator system (diameter 2.5 mm) positioned upstream of the thin iron foil (thickness 8 μm) allows a maximum angular deviation of the incident protons of less than one degree. The centering of the implantation set-up

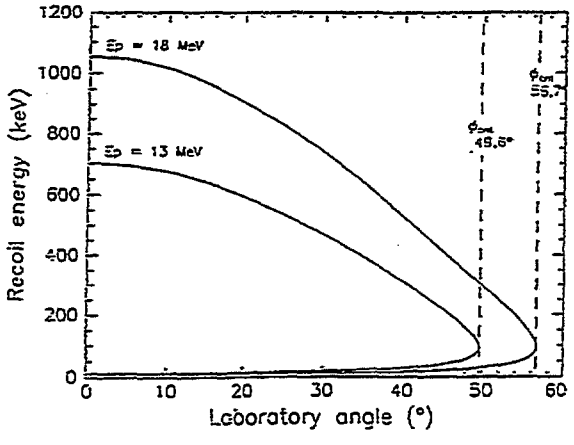

Fig. 4. Theoretical energy spectrum versus laboratory angle for the ^{56}Fe(p, n)^{56}Co nuclear reaction.

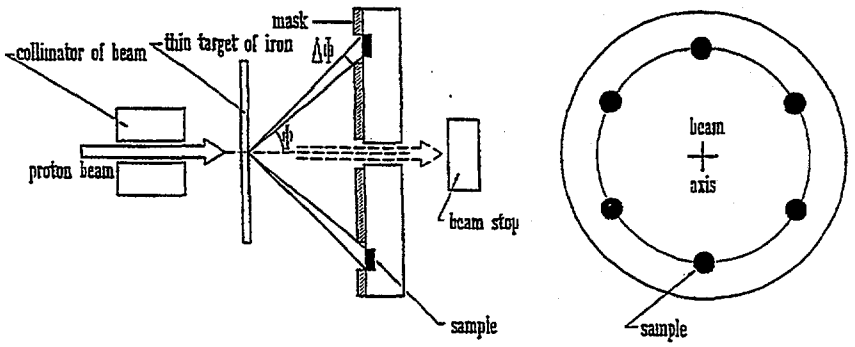

Fig. 5. Recoil implantation set-up.

is accomplished with a mechanical tolerance less than 0.1 mm.

A beam scanning upstream of the collimator system has been performed to secure a homogeneous irradiation of the six samples. Each sample receives qualitatively as well as quantitatively the same flux of recoiling radioactive ions. It is only for this condition that the calibration curve can be well determined.

The implantation set-up is inserted in a vacuum chamber. A secondary vacuum ($< 10^{-4}$ mbar) is sufficient to avoid any energy loss of the recoil products.

4.2. Depth profile of the ^{56}Co implanted in nickel

Thin films of nickel are deposited on a silicon substrate whose roughness does not exceed 2 nm. Each film thickness is measured by the Rutherford backscattering technique with an alpha particle beam of 2 MeV produced by a Van De Graaff accelerator. The beam current is limited to 20 nA to avoid all damage of the deposited material and to minimize the dead time.

Considering the very small thicknesses of the films, the uncertainties of the RBS measurements are primarily due to the error made on the charge measurement and on the solid detection angle of the backscattered alpha particles. The accumulated error in the thickness measurements is estimated to be at most 10% of the deposited thickness.

For each experiment, six films of nickel are irradiated simultanuously in the conditions previously described. The ^{56}Co activities B_0 and B are measured with a HPGe detector (31% relative efficiency) at the 846 keV gamma ray. The statistical errors of the gamma peak areas are given as two standard deviations.

4.2.1. Results

Two implantation experiments have been performed with a 13 MeV incident proton beam. The experimental points are presented on Fig. 6.

Fig. 7 presents the calibration curve with a 18 MeV incident protons beam.

The energy loss of the 13 or 18 MeV protons over the iron target thickness (8 μm) is insignificant.

At 13 MeV, the low dispersion of the experimental points obtained by two different experiments reveals the good reproductibility of the irradiation set-up and also the good quality of the films deposited by cathode sputtering.

At 13 and 18 MeV protons, the maximum implanted depths are respectively of about 110 and 200 nm. The depth resolutions are respectively close to 2 and 4 nm.

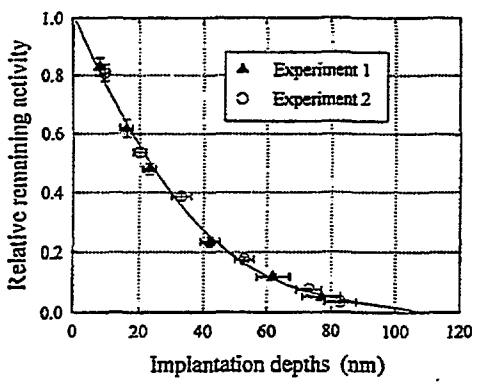

Fig. 6. Calibration curve for 13 MeV protons.

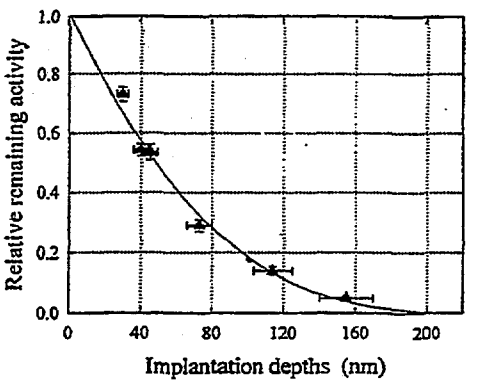

Fig. 7. Calibration curve for 18 MeV protons.

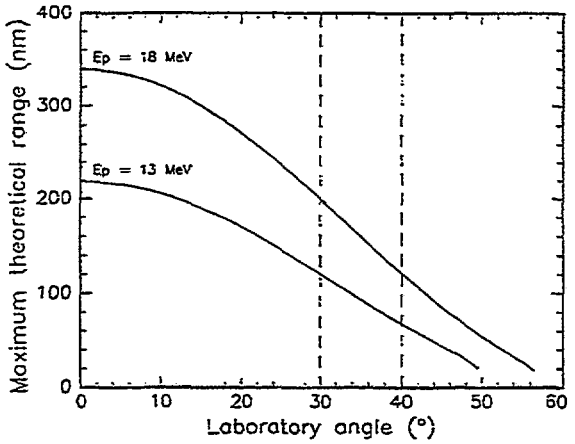

Fig. 8. Theoretical maximum range of ^{56}Co in nickel versus laboratory angle (TRIM95).

4.2.2. Discussion

Fig. 8 represents the maximum theoretical range $R_{max}(\phi)$ of ^{56}Co ions in nickel as a function of the emission angle ϕ and for the energies 13 and 18 MeV. These maximum ranges have been estimated both from the nuclear reaction kinematics and the TRIM95 software. The theoretical energy spectrum reveals a double energy of the recoiling ion for a fixed angle $\phi < \phi_{crit}$ (Fig. 4). The maximum range is calculated by considering the higher energy value and the implantation geometry, i.e.

$$R_{max}(\phi) = R(E_4(\phi))\cos\phi$$

for a frontal implantation geometry (Fig. 1),

$$R_{max}(\phi) = R(E_4(\phi))\sin\phi$$

for a tubular implantation geometry (Fig. 2).

At 13 and 18 MeV protons, the maximum experimental implanted depths obtained for the ^{56}Co ion in nickel are compared to the maximum theoretical range (Table 1). For the 30– 40° implantation cone, this range is defined for the $\phi = 30°$ emission angle because it is greater than the one at 40° (Fig. 8); the implanting theoretical energy spectra are from 0 to 470 keV at 13 MeV protons and from 0 to 750 keV at 18 MeV protons.

The experimental values are in good accordance with the theoretical ones. This can be justified by the fact that the implanting ions of high energy are produced within the iron foil in the last nanometers of its external side. Their emerging angle and energy are close to the theoretical ones.

Table 1
Comparison between experimental implanted depth and maximum theoretical range

Energy of incident protons (MeV)	13	18
Experimental implanted depth (nm)	110	200
Maximum theoretical range (nm)	120	200

The study of the nuclear reaction ^{56}Fe(p,n)^{56}Co kinematics has enabled us to determine the maximum implanted depth of the ^{56}Co ions in nickel. If it is not possible so far to generalize this agreement to all nuclear reactions, it appears that the nuclear reaction kinematics gives significant information on the activated thickness and proves to be an essential tool for the application of the UTLA method. Moreover, it enables to select the nuclear reaction and the incident particle energy appropriate to the study requirements. Generally, the nuclear reaction kinematics using light mass particle beams on a heavy-nuclei target present a critical emission angle ϕ_{crit} and two possible values of energy of the recoiling ion for an angle $0 \le \phi < \phi_{crit}$. The calculations of the critical angle and the recoil energies inform the user of the implantation geometry to be adopted and of the implanted depth. For the final choice of the nuclear reaction, this information should be coupled to the size of the reaction cross section.

For reactions with more than two particles in the final state, the kinematic equations have not been solved. Nevertheless, a few experiments with the ^{59}Co(p, pn)^{58}Co reaction reveal that by assimilating the light particles of the final state to a single equivalent mass particle, the theoretical implanted depth is in accordance with the experiment. However, this single case does not allow to generalize to such nuclear reactions.

The nuclei ^{56}Co backscattering effect induces a systematic error in the calibration procedure. Substantially less low energy nuclei are backscattered from silicon than from a nickel substrate. Each measured activity B is overestimated. The total activity B_0 is also overestimated specially for very thin nickel films. Calculations on sandwich targets using TRIM95 reveal that the low energy nuclei ^{56}Co backscattering effect on B/B_0 ratio is negligible for incidence angles between 0 and 40°. Consequently, in the described irradiation condition, the calibration curves realized with silicium substrate are well representative of ^{56}Co depth profile in bulk nickel.

For grazing angles, this effect must be taken into account; a convenient way is to select a substrate whose atomic number is close to the one of the materials to be implanted.

For each sample (6 mm diameter disc), the implanted activities corresponding to a 40 mC charge on the production target are presented in Table 2 for 13 and 18 MeV incident protons. The maximum range (a few hundred nanometers) of the recoiling radioactive ions in iron target are very much smaller than the target foil thickness (8 μm). Only the ions produced near the downstream side of the target foil recoil out of the latter and lodge in the material. The implanted radioactivity is therefore dependent on the range of the recoiling ions in the target foil. The rates registered for the energies of 13 and 18 MeV point out to this dependence. For the same charge, the

Table 2
Implanted ^{56}Co activity for the 13 and 18 MeV protons beams

Energy of the incident protons (MeV)	Cross section (mb)	Implanted depth (nm)	Implanted activity (Bq)
13	410	110	40
18	100	200	23

radioactivity implanted by the 13 MeV protons is greater by a factor of 1.7 whereas the cross section is greater by a factor of 4.1. This important difference is mostly explained by the fact that at the energy of 18 MeV the ^{56}Co ions are more energetic and thus more easily escape from the iron target. This phenomenon thus compensates for the decrease in the cross section. This tendency is important for the choice of the nuclear reactions because it allows for cases of implanted thicknesses close to a micrometer to select nuclear reactions whose cross sections are low.

5. Applicability of the UTLA method

5.1. Implantation rates

For a given nuclear reaction, the implantation rate depends on the cross section relative to the incident beam energy, on the recoiling energies, on the angular acceptance and on the implantation angle.

For the reactions presented in Table 3, the number of heavy ions recoiling out of the target is about 10^{10} to 2×10^{11} for 2×10^{17} incident particles. In practice, for the experimental set-up described in Figs. 1 and 2, only a fraction of these ions is implanted in the samples. To preserve an on line detection mode, a few microampere beam current is necessary.

In the experiments performed with the ^{56}Fe(p,n) ^{56}Co nuclear reaction, the implanted radioactivity is about 1 kBq for a charge of 40 mC received by the iron foil and for an implantation cone of 30– 40° and then sufficient to obtain good detection conditions.

5.2. Interference

A parasite and/or interfering activity can be generated in the sample by the primary beam particles which are scattered from the target to the acceptance cone.

In case of ^{56}Co implantation experiments using ^{56}Fe(p, n)^{56}Co nuclear reaction, if the implanted sample contains the iron element, scattered protons create a ^{56}Co interferent activity through the same nuclear reaction.

The choice of the nuclear reaction and therefore of the implanted radioelement must take into account the elementary composition of the investigated sample.

To reduce these interferences, the choice of an implantation cone with a large half angle is adviced, if it corresponds to the requirements of the implanted depth and of the implantation rate. More, the scattering of the incident particles may be reduced by minimizing the target thickness, although not to a point where microscopic perforations are present. However, the target thickness must be greater than the maximum recoil range in the production target to avoid any reduction of implantation rate.

The presence of parasite and/or interfering activities does not allow the use of a NaI detector during the on-line measurements because of its low energy-resolution and high background. The use of a HPGe detector is strongly advised.

Table 3
List of suitable nuclear reactions for the application of the UTLA method

Nuclear reaction	Threshold energy (MeV)	Beam energy (MeV)	Cross section (mbam)	Critical angle	Implantation cone	Implanted depth (nm)
^{65}Cu(p, n)^{65}Zn	2.2	10	800	62°	30–40°	80
		15	300	68°	30–40°	140
^{56}Fe(p, n)^{56}Co	5.4	13	410	50°	30–40°	120 *(110)*
		18	100	57°	30–40°	200 *(200)*
^{48}Ti(p, n)^{48}V	4.9	13	500	52°	30–40°	170
		20	100	60°	30–40°	290
		20	350	–	30–40°	260 *(280)*
^{59}Co(p, pn)^{58}Co	10.6	26	600	–	30–40°	410
		34	400	–	30–40°	580
^{55}Mn(α, n)^{58}Co	3.8	24	100	27°	10–20°	940

5.3. Implantation depth

In case of light incident particles on a heavy mass target, the maximum energies transferred to a recoiling ion are generally between a few hundred keV and a few MeV, which confers them maximum implantation depths in the material from a few tens to several hundred nanometers. Table 3 shows some theoretical values calculated for the frontal geometry (Fig. 1) for a nickel sample. A few experimental values measured for the same geometry and implanted material are given in brackets.

As the angle and energy spectra of the recoils depend on the implantation angle, the conditions of the sample irradiation must be rigorously similar to the ones defined during the calibration process. As the depth implanted by the UTLA method is often less than a micrometer, the sample roughness may influence the implantation profile and therefore the calibration curve. In a following report, the variation of the depth profile as a function of the standard film roughness will be studied.

6. Conclusion

This study reveals that surface depth profiles of recoiling heavy ions can be determinated for hundred nanometers activated dephts and easily used for very sensitive wear and corrosion studies. Generally, the methods of deposition by cathode sputtering or evaporation coupled to the RBS technique for the measurement of film thickness enables to obtain depth calibration curves ranging from a few tens to several hundred nanometers. Subsequently, material loss may be followed in most cases in on line detection mode on the piece exposed to wear or corrosion phenomena with nanometric sensibilities.

The kinematic of the nuclear reaction coupled with the TRIM95 software gives essential indications to the choice of the nuclear reaction, the incident particle energy, the implantation geometry and therefore facilitates the application of the UTLA method.

The UTLA method presents numerous advantages compared to direct activation methods:

(1) The activation being independent on the activated material composition and the deposition methods permitting to deposit a wide range of materials, the UTLA method may be applied to all kinds of materials. Moreover, the potentially damaging effects are far less than those induced by direct activation techniques using light or heavy ions. The method is thus applicable to polymers [8,9].

(2) The wide range of radioisotopes that can be generated and implanted allows to carry out experimental requirements (depth to activate, chemical nature of the radioelement in the case of selective corrosions, etc.).

(3) The generated activities are very low (a few kBq). Radioprotection precautions are therefore considerably reduced.

(4) The use of light particle beams (p, d, ^{3}He, α) is common to a vast majority of accelerators.

Nevertheless, as the implantation takes place in a vacuum chamber, the application fields of this method are limited to reduced-dimensions samples.

An important parameter in the UTLA method is the sample roughness. To complete these studies realized with very low roughness films, experiments on significantly rougher films will be carried out to determine the influence of roughness on the depth profile of the implanted radioactive ions.

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