

Topics in Current Chemistry

85

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G.K. Wolf
Chemical Effects
of Ion Bombardment

R.W. Kiser
Doubly-Charged Negative Ions
in the Gas Phase

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Chromatography
in Inorganic Trace Analysis



Instrumental Inorganic Chemistry

Springer-Verlag Berlin Heidelberg New York

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Topics in Current Chemistry

Fortschritte der Chemischen Forschung

**Instrumental
Inorganic Chemistry**



Springer-Verlag
Berlin Heidelberg New York 1979

This series presents critical reviews of the present position and future trends in modern chemical research. It is addressed to all research and industrial chemists who wish to keep abreast of advances in their subject.

As a rule, contributions are specially commissioned. The editors and publishers will, however, always be pleased to receive suggestions and supplementary information. Papers are accepted for "Topics in Current Chemistry" in English.

ISBN 3-540-09338-9 Springer-Verlag Berlin Heidelberg New York
ISBN 0-387-09338-9 Springer-Verlag New York Heidelberg Berlin

Library of Congress Cataloging in Publication Data. Main entry under title: Instrumental inorganic chemistry. (Topics in current chemistry ; 85) Bibliography: p. Includes index. CONTENTS: Wolf, G. K. Chemical effects of ion bombardment. – Kiser, R. W. Doubly-charged negative ions in the gas phase. – Schwedt, G. Chromatography in inorganic trace analysis. I. Chemistry, Analytic – Addresses, essays, lectures. 2. Chemistry, Inorganic – Addresses, essays, lectures. I. Wolf, Gerhard K., 1935 – Chemical effects of ion bombardment. 1979. II. Kiser, Robert Wayne, 1932 – Double charged negative ions in the gas phase. 1979. III. Schwedt, Georg, 1943 – Chromatography in inorganic trace analysis. 1979. IV. Series. QD 1.F 58 vol. 85 [QD 75.2] 540'.8s546 79-14 180

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Typesetting and printing: Schwetzingen Verlagsdruckerei GmbH, 6830 Schwetzingen. Bookbinding: Konrad Triltsch, Graphischer Betrieb, 8700 Würzburg
2152/3140-543210

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Springer-Verlag New York Inc.	175, Fifth Avenue · New York, NY 10010 Telephone 4 77-8200

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Chemical Effects of Ion Bombardment

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1 Introduction and Survey

The techniques of ion bombardment and ion implantation are receiving increasing interest in different fields of science. Their application for the solution of problems in basic science and their potential utilization in applied science are numerous. Some years ago the bombardment of semiconductors was the only widely used application. While this is still the field of the most practical importance the implantation of materials in order to improve their resistance against mechanical and chemical attack is receiving more and more attention.

An energetic particle hitting a surface or interacting with a gas may

- merely cause damage,
- itself act as an important quantity for the process,
- end up as a reaction partner during the formation of a new alloy or compound.

Table 1 represents an attempt to classify the studies using ion beams according to their field of science and according to the main effects caused by the ions. The arrangement is somewhat arbitrary. But because of the great overlap between the different kinds of studies, this cannot be avoided.

Therefore, ion implantation is a real interdisciplinary field in which engineers as well as physicists and chemists work. The chemists were in a minority until recently and the chemical aspects of ion implantation received much less attention than the physical and technological ones. Nevertheless, they are just as interesting and of practical importance for its future development. For these reasons it seems justified to present the most important works with chemical significance done during the last decade by chemists, physicists and engineers.

Unfortunately, it is very difficult to demarcate the area of ion bombardment from the surrounding area and to distinguish the more chemical effects from the other effects induced by bombardment. Therefore one has to try to find a definition of "chemical effects of ion bombardment" in the sense of the present article. It includes accordingly all studies concerned with:

- a) Chemical effects of the radiation or, in other words, changes in the chemical structure and composition of the targets induced by the bombardment. The relevant chapters are "Radiation Damage" and "Surface Chemistry of Materials Irradiated with Ions".
- b) The chemical state and the chemical reactions of the bombarding ions themselves. Examples are found mainly in the chapters "Solid State Chemistry of Implanted Atoms" and in "Surface Chemistry of Materials Irradiated with Ions".
- c) The use of implanted ions as a probe for the state of a system. The chapters "Solid State Chemistry of Implanted Atoms" and "Radiation Chemistry" contain a few examples of interest for chemists.
- d) The applications of ion-irradiated materials in chemistry independent of the irradiation effects being of chemical or physical nature. These studies are mentioned in the chapter "Surface Chemistry of Materials Irradiated with Ions".

Semiconductor and thin film technology is an area too large to be included in this survey, apart from a few comments in Sect. 5. This is also true for "Implantation Metallurgy", which has been covered by a number of recent review papers^{1, 2)}.

Table 1. The application of ion beams in different areas of science

Effects caused by the bombardment	Solid state physics	Electrical engineering	Material science	Solid state chemistry	Surface chemistry and physics	Atomic physics physical chemistry
Radiation damage	Superconductivity of bombarded metals	Variations of thin film properties	Simulation of radiation damage e.g. in nuclear power plants Wear, friction and lubrication of materials	Radiation Chemistry	Ion sputtering Ion reflection	Radiation decomposition of gases Ionization phenomena Charge exchange studies
Insertion of impurities	Superconductivity of bombarded metals Doping of optical materials	Variations of thin film properties Doping of semi-conductors	Wear, friction and lubrication of materials Formation of non-equilibrium alloys	Chemical state of implanted atoms	Reactivity of ion-bombarded surfaces Corrosion of materials Electrochemical properties of materials	
Reactions			Formation of non-equilibrium alloys	Syntheses of non-conventional compounds Simulation of "Hot Atom" reactions Simulation of high-energy reactions in space		Simulation of "Hot Atom" reactions Ion-molecule reactions

The radiation effects and ion-molecule reactions in gases are other topics which, strictly speaking, belong to the subjects of this article. But being much too voluminous the subject would by far exceed the extent of this paper. Fortunately, there are several relevant books available^{3, 4)}.

Since there exist quite a number of other methods for surface modification and for introducing foreign atoms into host lattices, one should discuss briefly the advantages of ion implantation:

- a) It is possible to dope every host material with every element. There is no need to consider the question of solubility or other thermodynamic quantities.
- b) Ion implantation is the only method allowing the doping of a host material at any temperature one likes. Studies of the recovery behavior of materials in the low-temperature region and of the low-temperature chemistry of foreign atoms in hosts are thus possible without prior treatment of the system.
- c) One may study the behavior and the reactions of single atoms because the number of ions interacting with the solids or gases may be limited to avoid mutual influence.
- d) Ion bombardment of solids leads to variations of very thin surface layers of a few Å to a few 100 Å, while the bulk of the material remains unchanged.
- e) The density of defects in the implanted region of solids is very high.

The last two arguments are controversial. In certain cases the impossibility of doping the bulk of a material or the creation of defects through the implantation could be disadvantageous. This statement indicates the limitations of the method as well as the fact that it is an experimentally pretentious technique.

The following three chapters provide an outline of the fundamentals of the interactions of heavy ions with matter and a description of the most important experimental methods for the production and acceleration of ions as well as for the analysis of the products of the bombardment.

In Chaps. 5 and 6 the experimental work with chemical relevance to radiation effects as well as the chemical “fate” of the bombarding particles and the applications of the irradiated material are covered.

The last chapter proposes some future trends in this field.

2 Fundamentals of the Interaction of Heavy Ions with Matter

2.1 Energy Loss

An energetic ion passing through matter loses energy through interaction with the surrounding atoms. The mechanism of this energy loss was the subject of many years of investigation, starting in 1913 with Bohr⁵⁾ and most completely developed in 1963 by Lindhard⁶⁾. The treatment by different authors contains many assumptions and correction factors, but the agreement within the experiments is rather good in general, taking into account a number of additional processes not included in the theory.

The following summarizes the theory of Bohr, Bethe and Lindhard et al. The major processes of energy loss are:

- Excitation of the electrons of the surrounding atoms by the energetic ion. This process is called electronic energy loss or electronic stopping.
- Collisions of the ions with the atoms, called nuclear energy loss or nuclear stopping.
- Charge exchange between the ions and the atoms.

The total energy loss is the sum of the three fractions, and may be written

$$\left(\frac{dE}{dx}\right)_{\text{total}} = \left(\frac{dE}{dx}\right)_{\text{electronic}} + \left(\frac{dE}{dx}\right)_{\text{charge exchange}} + \left(\frac{dE}{dx}\right)_{\text{nuclear}}$$

Since charge exchange represents only a very small fraction of the total energy loss, it will be neglected in the following considerations.

Electronic energy loss is the prevailing fraction at high energies or low mass numbers; nuclear energy loss dominates at low energies and high mass numbers. Thus He ions of 100 keV lose their energy nearly exclusively by electronic excitation, 10 keV Xe ions mainly by elastic collisions.

At even higher energy another mechanism starts to play a role, namely Rutherford scattering at the atomic nuclei. But for the energy region we deal with it has not to be taken into account.

2.1.1 Electronic Stopping

In the theory of Bohr⁷⁾ the energy loss of an ion interacting with an atom is

$$\left(\frac{dE}{dx}\right)_{\text{electronic}} = \frac{4 \pi Z_1^2 e^4}{mv^2} B$$

This is only valid for a fully ionized atom moving with a higher velocity than the K-shell electrons. B is an interaction parameter for which Bethe^{8,9)} gave the expression

$$B = Z_2 \ln \left(\frac{2 mv^2}{I} \right)$$

Z_1, Z_2	= nuclear charge of the ion and atom
m	= electronic mass
v	= projectile velocity
I	= average excitation energy of the atom

For lower velocities one has to apply inner shell corrections as well as for the capture of electrons by the primary ion¹⁰⁻¹²⁾.

At low velocities ($v_1 \ll Z_1 \cdot v_0$; $v_0 = Z_1 e^{2/h}$) the electronic stopping power is proportional to v_1 .

For this region Lindhard and Scharff¹³⁾ gave the formula

$$\left(\frac{dE}{dx} \right)_{\text{electronic}} = \xi \frac{8 \pi e^2 N a_0 Z_1 Z_2 v}{e (Z_1^{1/3} + Z_2^{2/3})^{3/2} v_0}$$

$$\xi \sim Z^{1/6}$$

$$a_0 = \text{first Bohr orbit}$$

$$N = \text{number of atoms per unit volume}$$

$$v_0 = \text{Bohr velocity}$$

2.1.2 Nuclear Stopping

Nuclear stopping is only important at low velocities ($v_1 < Z_1 \cdot v_0$) where collisions between the projectile and the target atom as a whole take place. They can be treated using classical mechanics if the energy is above a certain value, a condition being fulfilled nearly in all cases of interest for the present paper. The most difficult task in this treatment is the proper choice of an interatomic potential between two colliding atoms. Bohr¹⁴⁾ as well as other authors discussed this question. There have been several attempts using Born-Mayer-, Coulomb-, Nielson-, Brinkman-, and Fiersov-potentials¹⁵⁾.

Lindhard et al. used a Thomas-Fermi potential and calculated the differential scattering cross section for multiple collisions⁶⁾ as

$$\sigma = \frac{\pi \cdot a^2}{2 t^{3/2}} f(t^{1/2})$$

where f is a numerically evaluated function for which Winterbon et al. give an analytical approximation¹⁶⁾, and t a dimensionless variable and a a measure for the size, connected to the first Bohr orbit a_0 :

$$a = \frac{a_0 \cdot 0.8853}{(Z_1^{2/3} + Z_2^{2/3})^{1/2}}$$

2.1.3 Energy Loss Expressed in Universal Constants

The most interesting feature of the work of Lindhard et al.⁶⁾ is the possibility of expressing the electronic and the nuclear stopping power in terms of universal constants, a form independent of the mass and charge of the individual atoms. A "universal" energy ϵ and a universal range ρ are defined, and the energy loss is expressed in terms

$$\text{of } \left(\frac{d\epsilon}{d\rho} \right) \text{ instead of } \left(\frac{dE}{dx} \right).$$

The electronic energy loss takes the form

$$\left(\frac{d\epsilon}{d\rho} \right)_{\text{electronic}} = k \epsilon^{1/2}$$

$$\text{with } k \approx \frac{0.079 Z_1^{1/6} Z_1^{1/2} Z_2^{1/2} (M_1 + M_2)^{3/2}}{(Z_1^{2/3} + Z_2^{2/3})^{3/4} M_1^{3/2} M_2^{1/2}}$$

The nuclear energy loss is related to the stopping cross section σ by

$$\left(\frac{d\epsilon}{d\rho} \right)_{\text{nuclear}} = \sigma \frac{(M_1 + M_2)}{4 \pi e^2 Z_1 Z_2 M_1}, \text{ where}$$

the universal energy is calculated by

$$\epsilon = E \frac{a M_2}{Z_1 Z_2 e^2 (M_1 + M_2)}$$

and the universal range is calculated by

$$\rho = R N M_2 \frac{4 \pi a^2 M_1}{(M_1 + M_2)^2}$$

M_1, M_2 represent the masses of the projectile or target respectively,

R is the normal range of the projectile in matter and

N is the number of “stopping atoms” per unit volume.

Figure 1 shows the nuclear and electronic specific energy loss.

From the curves it is possible to calculate the “normal” stopping using the above equations. Note that the curve for the nuclear stopping is really universal, whilst the curve for the electronic stopping has a different slope (k) for every combination projectile-target. The figure shows very clearly that nuclear stopping is the dominant mechanism at low energies and electronic stopping at high energies.

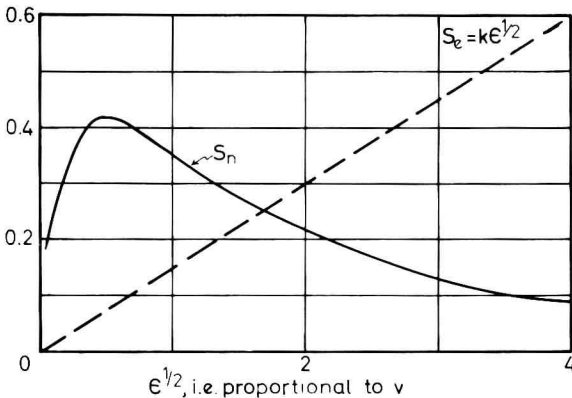


Fig. 1. Nuclear and electronic specific energy loss S as a function of energy in dimensionless units

2.2 Ion Ranges and Profiles

2.2.1 Ranges

The treatment covered in the preceding chapter allows the prediction of ranges of implanted atoms. The range is the total path of the ion or the integral over all $\frac{dE}{dx}$.

If one uses the expression range, one has to define the kind of range meant. Figure 2 tries to clarify the different types. One might be interested in the total range R_L or, more often, in the projected range R_p which is a measure of the distance of the ion from the surface and has the advantage of being easily accessible to measurements.

An approximate relation for the region of nuclear stopping is given by Lindhard¹³⁾

$$\frac{R_L}{R_p} = 1 + \frac{M_2}{3 M_1}$$

The electronic stopping does not lead to major deflection and does not contribute very much to the lateral spread R_L . Another important quantity is the range straggling ΔR or ΔR_p . It contains information about the actual distribution of the implanted ions in a solid. They also have been calculated by various authors, e. g. Schiott¹⁸⁾.

In practice, one normally uses tables or graphs presenting ranges, range straggling, and lateral spread as a function of projectile-target combination and energy. In general, the energy is given in keV and the range or projected range in $\mu\text{g cm}^{-2}$, which allows an easy comparison of different materials. Some range tables are presented in Table 2.

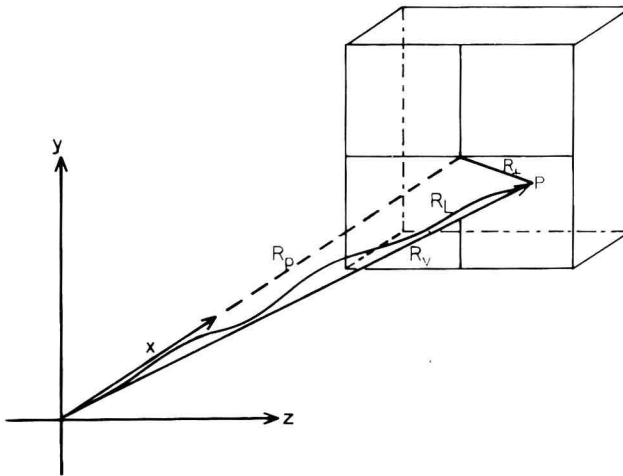


Fig. 2. The different types of ranges for an ion starting from $x=y=z=0$ ¹⁷⁾ in the x direction

$$\begin{aligned} R_L &= \text{total range,} & R_p &= \text{projected range,} \\ R_v &= \text{vector range,} & R_L &= \text{lateral range,} \\ R_v^2 &= R_p^2 + R_L^2 \end{aligned}$$

Table 2. Range-energy tables

Author	Quantities listed	Targets	Ref.
G. Dearnaley et al.	$R_p + \Delta R_p$	$Z_1, Z_2 = 5, 10, 15 \dots$	19)
J. Gibbons et al.	$R_p, \Delta R_p$ $R_L, \Delta R_L$ $\frac{dE}{dR_{\text{nucl.}}}, \frac{dE}{dR_{\text{el.}}}$	Selected elements and compounds	20)
R. G. Wilson, G. R. Brewer	$R_p + \Delta R_p$	Selected elements and compounds	21)

Another possibility is the use of graphs where the reduced range is plotted versus the reduced energy ϵ , as derived from the theory of Lindhard. They have the advantage of containing all possible information on one page, the disadvantage that one has to calculate the “normal” units from the equations given in the preceding section. In most cases the values given in the tables are only for elemental targets or semiconductor compounds. For other compounds one may estimate the ranges assuming a fractional contribution from the different elements.

$$\frac{1}{R_p} = \sum_i \frac{x_{1,2\dots i}}{R_{p1,2\dots i}}$$

$R_{p1,2\dots} =$ Range of the elements 1, 2 . . .

$x_{1,2\dots} =$ Fraction of the component in question at the total mass

The accuracy of this estimate is about $\pm 10\%$ ²²⁾.

2.2.2. Range Profiles

Range or concentration profiles contain information about the distribution of the bombarding ions in the solid target. Since the mapping of the distribution in three

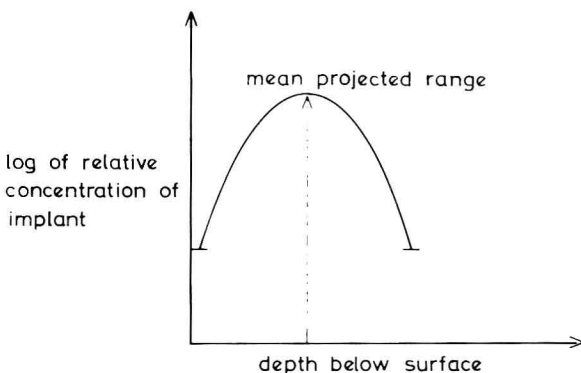


Fig. 3. Theoretical concentration profile of implanted atoms in a solid target

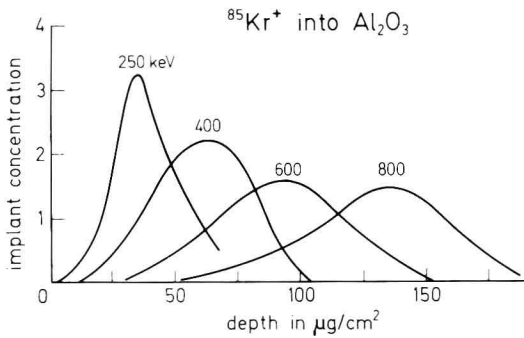


Fig. 4. Range distribution of ^{85}Kr ions implanted at different energies in Al_2O_3 ^{23, 24)}

dimensions is quite intricate, one normally presents the intensity distribution in the direction normal to the surface. This gives a picture of the actual depth distribution of the ions in the target. The theoretical range profiles are very similar to a Gaussian distribution around the mean projected range (Fig. 3).

The energy of the ions is responsible for the penetration depth and the depth distribution.

Figure 4 shows the experimental distribution of ^{85}Kr ions implanted into amorphous Al_2O_3 at different energies.

Such distribution data can also be presented as integral curves. Here the concentration remaining beyond a certain value at any penetration depth is plotted. This kind of description makes it very easy to deduct the variations between two depth values or the concentration at high depth values. Figure 5 shows the same distributions as Fig. 4 in the integral form.

The range profiles measured experimentally very often show considerable deviations from the Gaussian form. The reasons for these deviations are:

- a) In single crystals or polycrystalline material an alignment of the ion beam with a crystallographic axis leads to an enhanced penetration of the ions due to the channeling effect (Sect. 2.4). Figure 6 is a presentation of results using such a system.

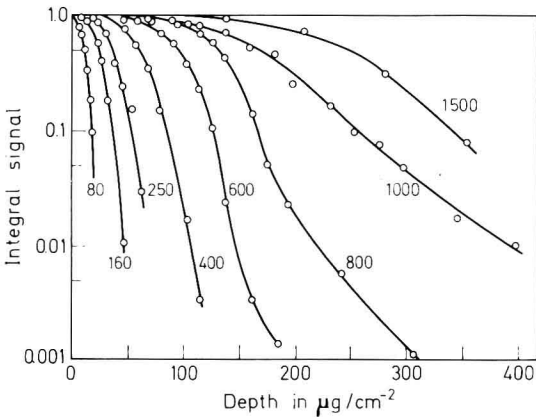


Fig. 5. Range distribution (integral) for ^{85}Kr in Al_2O_3 ^{23, 24)}