RESEARCH PROJECT INITIATION

Date: 28 June 1973

Project Title: "Surface Composition Determined by Analysis of Impact Radiation"

Project No: G-41-620

Principal Investigator: Dr. E. W. Thomas

Sponsor: Public Health Service

Agreement Period: From 1 June 1973 Until 31 May 1974

Type Agreement: Internal Grant

Amount: $1,100

Reports Required: Final Report due by 31 August 1974

Sponsor Contact Person (s):

Dr. J. W. Crenshaw
Biomedical Sciences Support Grant
 Committee
School of Biology
Campus

Assigned to: Physics

COPIES TO:

Principal Investigator
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Project File
File G-32-603
Dr. J. W. Crenshaw
Other_____

RA-3 (5-71)

GEORGIA INSTITUTE OF TECHNOLOGY

OFFICE OF RESEARCH ADMINISTRATION

No Action

RESEARCH PROJECT TERMINATION

Date: November 13, 1974

Project Title: Surface Composition Determined by Analysis of Impact Radiation

Project No: G-41-629

Principal Investigator: Dr. E. W. Thomas

Sponsor: DHEW/PHS/NIH; Biomedical Sciences Support Grant funded through G-32-608/ Crenshaw

Effective Termination Date: 5-31-74

Clearance of Accounting Charges: N/A - all cleared

 # 1/LO PRJ.

Grant Closeout Actions Remaining: None

 # 1/LO - MRS
1 LTO - Pers. Svcs.

Assigned to School of Physics

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School Director
Dean of the College
Director of Research Administration
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Patent and Inventions Coordinator

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Photographic Laboratory
Terminated Project File No. _____

Other __File No. G-32-608__

GEORGIA INSTITUTE OF TECHNOLOGY
ATLANTA. GEORGIA 30332
July 26, 1974

MEMORANDUM

TO: Dr. J. W. Crenshaw
                            Director, School of Biology

FROM: Dr. E. W. Thomas

SUBJECT: Biomedical Sciences Support Grant

I submit herewith material for the annual report of this project.

EWT:rh

xc: J. R. Stevenson

Surface Composition Determined by

Analysis of Impact Radiation

by E. W. Thomas

August 1, 1974

(A) Background

It has been shown[1,2] that when a beam of low energy (30-3000 Volt) ions

or neutral atoms impinge on a surface there is a copious emission of light

when the projectiles are heavy (say Argon, or Krypton) the largest source of

emission is from atoms that are collisionally ejected from the surface and

simultaneously excited; this ejection process is commonly known as sputtering.

By analyzing the emission with conventional spectroscopic techniques one can

determine what elements are present in the surface under bombardment. An

important consideration is that the incident projectiles will penetrate only

a very small distance into the target material; the mean range may be readily

calculated and for Argon ions (or atoms) at 3 keV energy this is only some

two or three monolayers (i.e. two or three atomic layers). Thus the source of

excitation is indeed the surface itself and the data provides information on

the surface composition only and not the bulk composition. The projectile impact

provides a continual erosion of the material and so by following the signals

from various elements as a function of time does in fact acquire a depth distri-

1. C. Kerkdijk and E. W. Thomas. Physics 63, 577 (1973).

2. C. W. White, D. L. Simms and N. H. Tolk. Science 177, 481 (1972).

bution profile of the material under study. It should be noted that the technique causes the surface material to be ejected in the atomic state and therefore provides information on individual elements. Thus there is no information on the molecular arrangement of the target; such molecular information must be sought by other techniques.

The objective of this work was to evaluate whether the analysis of the collisionally induced light emission can give quantitative information on the composition of a surface. We discussed in a previous report a number of situations in which this might be advantageous for the analysis of biological materials. For a test case we have in fact decided to use a human tooth.

(B) Progress to Date

The equipment for this work consists basically of a positive ion accelerator firing a beam onto the target which is contained in an ultra high vacuum environment. Emission from the target surface is analyzed by a conventional spectrometer. During the present reporting period the equipment was finally assembled and tested.

On attempting to introduce the tooth into the vacuum system we encountered some substantial vacuum problems due to outgassing, principally of the formaldehyde solution in which the sample had been stored. This was overcome by using "dried" samples that had not been placed in preservative and further removing fluids by maintaining the sample in a subsidiary vacuum system for some days prior to usage.

The impact at 15 keV Ar^+ beam on the tooth surface gave rise to a visible light spot that could readily be analyzed spectroscopically. The spectral range studied was from 3000 to 7000 Å. The principal spectral lines were those of Ca and Ca^+ (i.e. the Ca I and Ca II spectra respectively). These lines

represent sputtered excited calcium atoms. The predominance of calcium is not surprising since some 33 to 39% (by weight) of human tooth enamel is said to be calcium[3]. We do not however observe any Phosphorus (16-18% by weight[3]).

One can provide a reasonably convincing explanation of why phosphorus is not observed. The P I spectrum is in fact to be found at wavelengths above 9000 Å or below 2700 Å; there are in fact no lines in the spectral region accessible to us. The P II spectrum is fairly rich in the visible region but the transitions are between states of fairly high energy (for example the 3715 Å line involves a transition from a state of 12.80 eV down to 9.48 eV). By contrast, the resonance lines of both the Ca I and Ca II spectra lie in the visible and are in fact our most prominent emissions; the excitiation of these resonances lines requires an energy of around 2-3 eV. Thus the total energy transfer required to produce excited Ca is 2 eV, to produce excited Ca^+ is about 9 eV (to both ionize and excite) and to produce excited P^+ is about 24 eV. It is a well known fact in atomic collision studies that the probability of an inelastic collision event decreases rather rapidly as the required energy transfer increases. Thus one in fact expects calcium to be excited far more readily than phosphorus. Allowing for the excitation probability to decrease as the cube of energy transfer, and with the knowledge[3] of the expected ratio of Ca to P one would indeed expect the visible phosphorus lines to be a factor of forty less intense than calcium.

We should now consider whether any of the trace elements are to be found in our spectra. Sodium for example is present in tooth enamel to about 1% of total weight[3], and by the arguments given above should be favorably excited

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3.   R. A. Young, J. Dental Res. **53**, 193 (1974).

3

since the resonance lines of Na I require an energy transfer of only 2 eV.
Indeed we can identify these sodium lines although they are weak.

There remain a number of other observed lines that cannot be ascribed
to Ca, P or Na; we are continuing to analyze these to determine their origin.

(C) Conclusion

The proposed technique clearly is useable and one can detect sodium
that is present in a relative concentration of 1%. It is however our goal
to detect concentrations far less than this and further developments to
improve signal strength are under way.

The method apparently suffers from a severe selectivity in detection of
different elements. Where an excited state can be produced by a small energy
transfer then one does in fact get a reasonable detection sensitivity; if
however a large energy transfer is needed (such as for excitation of phosphorus
in tooth enamel) the sensitivity may be poor. Of course such selectivity in
detection of various elements is common to most analysis schemes; the case of
a mass spectrometer, for example, is directly analogous to the present method
and suffers from the same problem.

We would note in passing that the sample teeth remained apparently
undamaged by the ion bombardment.

(D) Future Work

We intended to pursue this problem further using other sample materials;
for example we shall compare the response of the tooth with that of synthetic
apatites (hydroxyapatite, chlorapatite, and fluorapatite). Hopefully
detection sensitivity can be improved to permit detection of small quantities
of trace elements.

(E) <u>Signature</u>

E. W. Thomas
Professor of Physics
26 July 1974