Construction of an ultrahigh vacuum facility for elastic record detection analysis of hydrogen in metal alloys

by Robin Hugh Barnes, Jr

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Physics
Montana State University

Abstract:
Construction and calibration of a laboratory facility to study the presence of hydrogen in and on metal surfaces in an ultrahigh vacuum environment are described. This facility is used for Elastic Recoil Detection Analysis of hydrogen in the surface layers of titanium alloys. The facility was calibrated by means of mylar targets with known hydrogen concentration. Questions posed to the calibration involved the analysis methods to be used on spectra obtained and the accuracies possible with an aperture of extended dimension collecting recoiled protons from an extended surface on the sample. Rigorous numerical analyses were applied to determine the acceptable applications for the simplifying approximations customarily used with smaller diameter apertures and smaller probing beam diameters. The approximation methods were acceptably accurate for near-surface analysis of a sample, but were of a limited value for determination of the characteristics of non-homogeneous hydrogen concentrations at greater depths. Accurate analysis of hydrogen concentration profile in a sample was limited to a depth dependent upon the material of the sample and the range of the ions in that material.

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APPROVAL

of a thesis submitted by

Robin Hugh Barnes, Jr.

This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

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Date July 23, 1996

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ABSTRACT

Construction and calibration of a laboratory facility to study the presence of hydrogen in and on metal surfaces in an ultrahigh vacuum environment are described. This facility is used for Elastic Recoil Detection Analysis of hydrogen in the surface layers of titanium alloys. The facility was calibrated by means of mylar targets with known hydrogen concentration. Questions posed to the calibration involved the analysis methods to be used on spectra obtained and the accuracies possible with an aperture of extended dimension collecting recoiled protons from an extended surface on the sample. Rigorous numerical analyses were applied to determine the acceptable applications for the simplifying approximations customarily used with smaller diameter apertures and smaller probing beam diameters. The approximation methods were acceptably accurate for near-surface analysis of a sample, but were of a limited value for determination of the characteristics of non-homogeneous hydrogen concentrations at greater depths. Accurate analysis of hydrogen concentration profile in a sample was limited to a depth dependent upon the material of the sample and the range of the ions in that material.

INTRODUCTION

The current state of technology is such that higher demands are placed on the materials used to construct devices employed in everyday commerce. One of the most demanding fields of production is that of aerospace vehicles. The high specific energy required to transport a unit of mass from earth surface to orbit makes the vehicle structure one of the most carefully controlled weight elements of the mission. The materials used in the vehicle structure are subjected to great ranges of temperature variation and considerable stress. In addition to the mechanical and thermal stresses imposed during a mission, the material is expected to be exposed to hydrogen-rich environments throughout the range of temperatures and mechanical stresses. Hydrogen as a fuel provides the highest specific impulse to the mission, and so best fulfills the requirement of minimizing the weight of the fuel component of the mission, and reduces the cost of transportation of the payload. The materials that best withstand the range of temperatures and stresses imposed are titanium alloys. The difficulty arises from the affinity of titanium for hydrogen, and the wide range of hydrogen solubility in titanium.[3] Large quantities of hydrogen are readily absorbed by titanium at high temperatures, and, if this hydrogen does not outgas before the material cools, the lower solubility of the hydrogen at lower temperatures forms titanium hydrides within the metal matrix, causing the structure to fail. It is therefore highly desirable to determine parameters of hydrogen absorption and retention into the surfaces of titanium alloys.

Elastic Recoil Detection Analysis is one of the most convenient means of studying the concentration of hydrogen within a metal surface.

CHAPTER 1

THEORY OF ANALYSIS METHODS

Elastic Recoil Detection Analysis

Elastic Recoil Detection Analysis (ERDA) is an ion beam surface analysis technique.[6] The incident ions are presented to the target surface at a shallow angle. Forward-scattered ions are collected in a particle detector under known geometrical conditions that permit analysis of their energies to determine the concentration of the species with depth in the target surface. The geometry of the ERDA technique is presented in Figure 1. The primary advantage of ERDA is that it permits the detection and analysis of light nuclei, in the case of these experiments, hydrogen. This technique is readily performed with relatively low energy ion beams, and permits probing to some depth into the sample to establish a profile of hydrogen concentration with depth.

ERDA permits probing a material to determine the presence and concentration of hydrogen at various depths. The data obtained from an ERDA experiment are a spectrum of particle counts versus energy of detected particles. Two examples of ERDA spectra are provided in Figure 2. The analysis is not entirely straightforward, and the purpose of this work is to describe the considerations that have been applied to this experimental method to determine the limits of accuracy that are available.

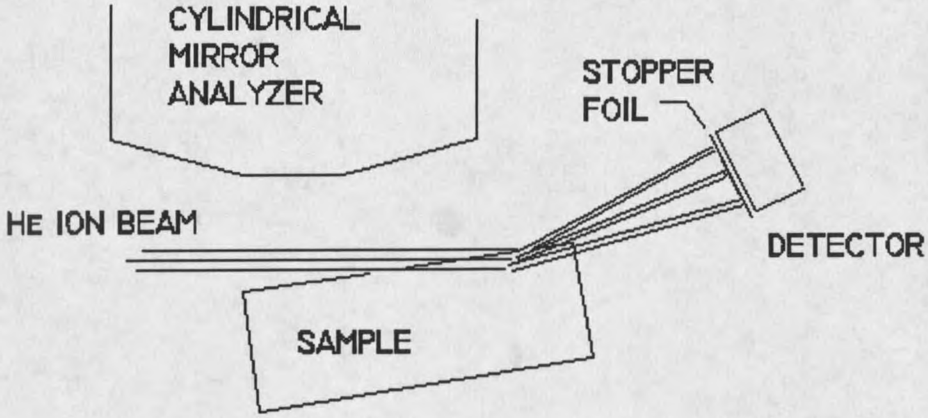

Figure 1. Schematic view of the ERDA experiment. The helium ion beam is admitted through an aperture on the left. The scattering of the protons on the surface of the sample and to a depth within the surface is detected by means of the particle detector. The stopper foil is used to prevent elastically scattered He ions from penetrating to the detector. The Cylindrical Mirror Analyzer is used for Auger Spectroscopy.

<u>Implementation</u>

In our experiments, the incident ion is Helium (He), with energies limited to less than 1.8 MeV. This is a selection made primarily to avoid neutron-producing reactions in the target chamber and beam line component and secondarily to reduce the electrical stress imposed upon the accelerator. The operations reflected in this work were primarily for calibration; to increase the consistency of the results as much as practicable, most experiments were done at approximately 1.6 MeV for the incident He ions.

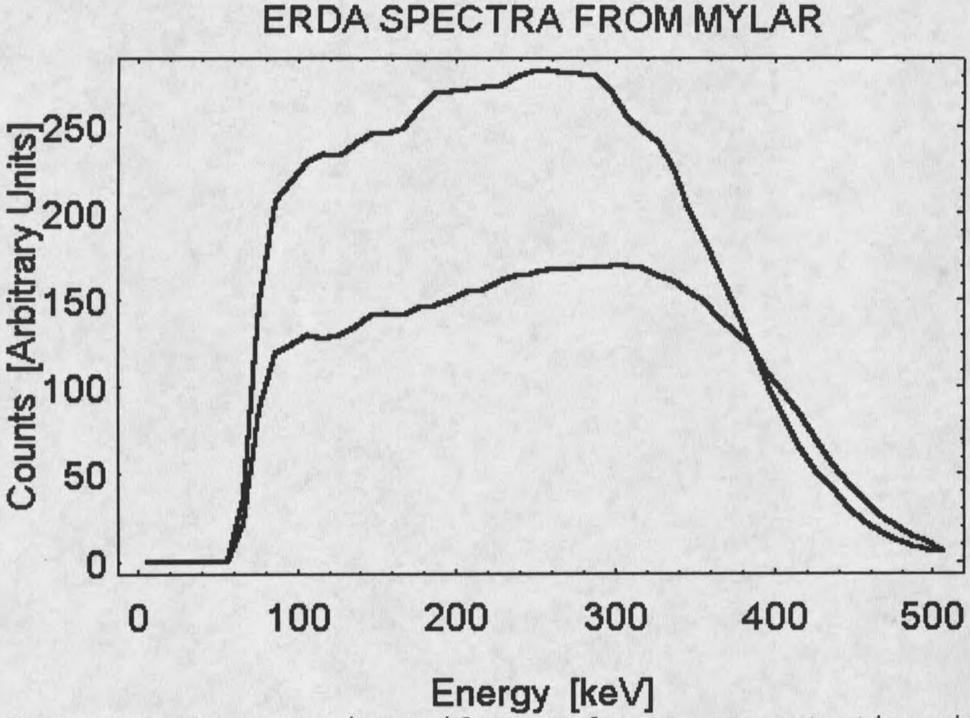

Figure 2. Spectra demonstrating uniform Hydrogen concentration with range of depths sampled. The higher yield spectrum is obtained from a sample placed closer to the detector. The spectrum with the higher energy at the leading edge is obtained by placing the sample closer to the chamber center.

Kinematic Factor. The primary energetic effect of the He ions upon Hydrogen (H) in the target is expressed by the product of the Kinematic Factor (KF) and the incident energy of the He ions (Equation 1). The KF is entirely dependent upon the geometry of the collision and the masses of the particles involved. The angle from the direction of motion of the incident ion through which the recoiling ion scatters is referred to as the scattering angle. In our experiments, we have used scattering angles in the range of twenty to thirty degrees. In this region, Kinematic Factor is represented by a slightly convex curve that can be immediately described as a straight line. The slight disadvantage faced is that the KF is never greater than .568 of the incident particle energy in this range, and to assure adequate range of energies of the scattered H ions for detailed analysis, we are required to use higher energies of incidence for the He ions.

$$KF = \left[\frac{4 M_1 M_2}{(M_1 + M_2)^2} \right] \cos^2(\varphi)$$

Equation 1. Forward Recoil Scattering Kinematic Function for the Kinematic Factor (KF). Subscript 1 indicates the incident ion, 2 the scattered ion. The angle is the scattering angle in the lab frame, measured from the direction of motion of the incident ion.

Scattering Cross Section. Given the characteristics of the forward-scattering collisions, an essential consideration is the quantitative description of the proportion of the total of original particles that may be expected to scatter in a specific direction. Due to the fact that high incident energies are required and the nuclei are of the two lightest species, simple Coulomb field scattering does not adequately describe the interactions. To address this phenomenon theoretically requires the rigors of partial wave analysis, as nuclear attraction potentials affect the scattering geometry in the close approach of the light nuclei. This work will utilize an application of an extensive and quite comprehensive series of experiments conducted and reported by Baglin, et al.[1] The intent of their work was to provide quantitative data on the absolute scattering cross-sections for H from a carefully controlled hydrocarbon polymer. These data are presented for a range of energies and scattering angles, along with coefficients describing arbitrary polynomial fits