Doped Beam Very Low Energy Particle Induced X-Ray Emission

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Under the supervision of Milos Toth and John A. Scott

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Certificate of original authorship

I, Daniel Totonjian declare that this thesis, is submitted in fulfilment of the requirements for the award of Doctor of Philosophy, in the Faculty of Science at the University of Technology Sydney.

This thesis is wholly my own work unless otherwise referenced or acknowledged. In addition, I certify that all information sources and literature used are indicated in the thesis.

This document has not been submitted for qualifications at any other academic institution.

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Glossary

PIXE Particle Induced X-Ray Emission

VLE-PIXE Very Low Energy PIXE

SEM Scanning Electron Microscope

EDS Energy Dispersive Spectroscopy

FIB Focused Ion Beam

IBA Ion Beam Analysis

BSI Backscattered Ions

PFIB Plasma Focused Ion Beam

LMIS Liquid Metal Ion Source

GFIS Gaseous Field Ionisation Source

ICP Inductively Coupled Plasma

PWBA Plane Wave Born Approximation

eECusPSShsR **e**xact, **E**nergy corrected, **C**oulomb corrected, **u**nited-**s**eparated, **P**erturbed **S**tationary **S**tate, **h**artree-**s**later, **R**elativistic correction

XRPCS X-Ray Production Cross Section

MO Molecular Orbital

SDD Silicon Drift Detector

LOD Limit of Detection

SNR Signal To Noise Ratio

RF Radio Frequency

amu Atomic Mass Unit or Dalton

TMP Turbo Molecular Pump

IGP Ion Getter Pump

SRM Standard Reference Material

C-K Coster-Kronig

LEIS Low Energy Ion Scattering

QCM Quartz Crystal Monitor

AFM Atomic Force Microscope

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Abstract

Particle Induced X-Ray Emission (PIXE) is a spectroscopic technique where characteristic X-Rays are generated from a sample by the impact of high energy particles. PIXE is typically performed with protons in a particle accelerator at energies in excess of 1MeV and is used for the detection of trace elements due to the lower background compared to complementary techniques such as Scanning Electron Microscope (SEM) Energy Dispersive Spectroscopy (EDS). PIXE performed at energies of less than 1MeV is sometimes used to enhance sensitivity to light elements, however very low energy PIXE (VLE-PIXE) performed at energies available to a commercial focused ion beam microscope of $\leq$30keV was considered impossible due to the extremely low X-Ray production at these energies.

In this research, VLE-PIXE was made possible by doping a hydrogen focused ion beam with a small proportion of a heavier ion species such as Ar or Xe. The characteristic X-Ray signal was shown to increase dramatically, allowing trace element analysis in the low parts per million range, offering performance comparable to proton only PIXE performed at much higher energies. This thesis outlines the implementation, characterisation, and application of the doped beam VLE-PIXE technique in a commercial focused ion beam microscope utilising available hardware and little to no modification to the instrument.

An investigation into the beam doping technique led to an interpretive model which considers various physical mechanisms which may be responsible for the increased performance which includes: the formation of quasi-molecules between the heavy projectile ion and the target atom, the suppression of non-radiative transitions, and vacancy lifetime modification due to multiple ionisation. These mechanisms may arise from the coincident impact of protons and a heavy ion species upon the same region of the sample.

The ions backscattering from the surface during VLE-PIXE analysis were also analysed to provide additional information regarding the sample thickness and composition. This leads to the possibility of several new techniques such as simultaneous doped beam VLE-PIXE and backscattered ion spectroscopy for real-time tomography, or endpointing during Focused Ion Beam (FIB) milling.