# Contents

<table>
<thead>
<tr>
<th>List of Figures</th>
<th>i</th>
</tr>
</thead>
<tbody>
<tr>
<td>List of Tables</td>
<td>x</td>
</tr>
<tr>
<td>List of Abbreviation</td>
<td>xi</td>
</tr>
</tbody>
</table>

## Chapter 1: Introduction

1.1 Motivation 1

1.2 Objective of the work 4

1.3 Organization of thesis 5

## Chapter 2: Literature Review

2.1 Overview 6

2.2 Microstructure & Microchemistry 6

2.2.1 Pure Zr 6

2.2.1.2 The Zr-O System 7

2.2.2 Alloying elements of Zr based alloys 10

2.2.3 Impurity elements 16
2.3 Oxidation of Zr and its alloys

2.3.1 Air Oxidation

2.3.2 Aqueous Corrosion

2.3.2.1 Incorporation of SPPs in oxide

2.3.2.2 Correlation of Autoclave Corrosion Testing to In-Reactor Uniform Corrosion Performance

2.3.2.3 Effect of Li on oxidation

2.3.3 Localized Oxidation

2.3.3.1 Nodular Corrosion

2.4 Hydriding of Zr and its alloy

2.5 Microstructure Modeling during phase transformation

2.5.1 Evolution of Phase Field Modeling in solid state

2.6 Effect of Irradiation

2.7 References

Chapter 3: Materials and Methodology

3.1 Material

3.2 Sample preparation and different characterization techniques

3.2.1 Fabrication of coupons and thermo mechanical treatment

3.2.2 Autoclaving
3.2.3 H pick up Measurement 53
3.2.4 Optical Microscope 53
3.2.5 Scanning Electron Microscopy 53
3.2.6 Transmission Electron Microscopy 54
3.2.7 Cross sectional TEM sample preparation technique 54
3.2.8 Micro-hardness measurement 56
3.2.9 Small Angle Neutron Scattering (SANS) 56
3.2.10 X-ray photo Electron Spectroscopy 57
3.2.11 Grazing Incidence X-ray Diffraction (GIXRD) 58
3.2.12 Ion and neutron Irradiation 60
3.2.13 Computational Technique 60
3.2.14 References 61

Chapter 4: Development of PFM to predict microstructure evolution in Zr-Nb alloy during diffusional phase transformation

4.1 Introduction 63
4.2 Phase Field model formulation 66
4.3 Free Energy Functional

4.4 Evolution Equations

4.5 Anisotropy in Interfacial Energy

4.6 Non-dimensionalization & Finite element method

4.7 Initial Microstructure

4.8 Phase-field simulations of growth of single protrusion of grain-boundary $\alpha$

4.9 Phase-field simulations of multiple protrusions in grain boundary $\alpha$

4.10 Incorporation of nucleation event implicitly and explicitly

4.11 Implicit nucleation and subsequent growth

4.11.1 Effect of temperature or undercooling

4.12 Explicit nucleation event and subsequent growth

4.12.1 Interface velocity

4.13 Effect of supersaturation on growth velocity

4.14 Summary

4.15 References
Chapter 5: Precipitation and growth study of intermetallics and their effect on oxidation behavior in Zr-Sn-Fe-Cr alloys

5.1 Introduction 99
5.2 Results 101
5.2.1 Microstructure Characterization 101
5.2.1.1 Cold rolled and as quenched Microstructure 102
5.2.1.2 Annealing at 700\(^\circ\)C for 15 min 102
5.2.1.3 Annealing at 700\(^\circ\)C for 1 hr 103
5.2.1.4 Annealing at 700\(^\circ\)C for 10hrs 106
5.2.1.5 Annealing at 800\(^\circ\)C for 1hr 109
5.2.1.6 Annealing at 800\(^\circ\)C for 10hr 111
5.2.1.7 Annealing in \(\alpha+\beta\) phase field (850\(^\circ\)C/1hr) 112
5. 2. 2 Micro-hardness Study 112
5.2.3 Small Angle Neutron Scattering 113
5.2.4 Oxidation Study 117
5.2.4.1 GIXRD study 120
5.2.4.2 XPS Analysis 125
5.2.4.3 Cross Sectional TEM 127
5.3 Discussions 129
5.4 Summary 133
Chapter 6: Influence of Fe content and β-phase on Corrosion and Hydrogen pick up behaviour of Zr-2.5Nb pressure tube material

6.1 Introduction

6.2 Results

6.2.1 Microstructures & characteristics of the precipitate or second phase

6.2.2 Micro hardness measurement

6.2.3 Oxidation behaviour

6.2.4 Hydrogen pick up behaviour

6.2.5 SEM Observation of oxide

6.2.6 XPS Analysis

6.2.7 GIXRD results

6.3 Discussions

6.4 Summary

6.5 References
Chapter 7: Effect of irradiation on Oxide

7.1 Introduction 175
7.2 Damage profile using TRIM programme 176
7.3 Characteristic of Oxide 178
7.4 GIXRD Results 178
7.4.1 Heavy Ion Irradiation 178
7.4.1.1 Zr-2.5 Nb alloy 179
7.4.1.2 Zr-Sn-Fe-Cr alloy 182
7.4.2 Proton Irradiation 187
7.4.2.1 Zr-Sn-Fe-Cr alloy 187
7.5 In pile oxide formed in fuel tube 190
7.6 XPS analysis of Heavy ion irradiated oxide of Zr-2.5Nb alloy 196
7.7 Summary 197
7.8 References 198

Chapter 8: Conclusions & Scope for further research

8.1 Conclusions 200
8.2 Scope for further research 203

Appendix 205