

UNIVERSITI TEKNOLOGI MARA

**FABRICATION AND
CHARACTERIZATION OF POROUS
SILICON BY TWO-STEP
ANODIZATION USING
ALTERNATING CURRENT PHOTO-
ASSISTED ELECTROCHEMICAL
(ACPEC) ETCHING**

FATIMAH BINTI ZULKIFLI

Thesis submitted in fulfillment
of the requirements for the degree of
Master of Science
(Electrical Engineering)

College of Engineering

July 2022

ABSTRACT

There are several reliable etching techniques to produce a uniform porous silicon (Si) structure on the semiconductor. Wet etching technique that is commonly used such as metal-assisted electroless etching and direct current photoelectrochemical (DCPEC) etching techniques often produced a non-uniform porous structure. While alternating current photoelectrochemical (ACPEC) etching technique produced a high porosity distribution of porous Si but with shallow pores. Consequently, to further enhance the porous Si structures and properties, a new etching technique, which is a two-step alternating current photo-assisted electrochemical (two-step ACPEC) etching technique was introduced in this research work. In the first step, the Si sample was left immersed in the electrolyte for a few minutes. Then, in the second step, the ACPEC technique was applied. Therefore, this research aims to synthesize the porous structure of porous Si using the two-step ACPEC technique and characterize its structural properties under different etching parameters. The work of this research was divided into three objectives: the formation of porous Si using different etching techniques, crystal orientations, etching durations, and current densities. In the first objective, porous Si samples were etched using DCPEC, ACPEC, and two-step ACPEC etching techniques. The porous Si sample etched using two-step ACPEC etching had the highest pore density and porosity, higher surface roughness in root mean square (RMS), and improvement in the uniformity of pores compared to the other two techniques. This showed that the two-step ACPEC technique improved the structural properties of the porous Si compared to the other two techniques. Therefore, the two-step ACPEC etching technique was used to further study the properties of porous Si in the next objectives. For the second objective, two different crystal orientations of Si, namely Si n(100) and Si n(111), were used to synthesize porous Si using the two-step ACPEC technique. The pores formed on porous Si n(100) consist of a mix of crisscross-shaped and a few square-shaped pores while porous Si n(111) exhibited irregular-shaped pores. On top of that, porous Si n(100) exhibited higher pore density and surface roughness than porous Si n(111). This showed that different crystal orientations of Si affected the properties of porous structures. Then, the Si n(100) was used in the next objectives. In the third objective, porous Si was etched at different etching durations. Porous Si etched for 30 minutes exhibited the highest average pore diameter (429.69 nm), porosity (26.99%), surface roughness in RMS (44.50 nm), pore depth (42.94 nm), and pore density out of all the samples. The Raman spectra result showed an improvement in surface area to volume ratio for all of the porous Si samples compared to the as-grown Si. Hence, it could be inferred that different etching duration affected the formation of pores on porous Si structures and the optimized etching duration obtained from this work was 30 minutes. For the next etching parameter, porous Si samples were etched using the two-step ACPEC etching technique at different current densities. The results showed that as the current density increased, the estimated porosity, pore density, average pore depth and surface roughness value in RMS increased. However, when the current density exceeded above 20 mA/cm², the estimated porosity decreased significantly along with the average pore depth and surface roughness value in RMS. Therefore, it could be concluded that different current densities strongly affected the porous Si structures. Generally, the research outcomes from all these works show different morphologies can be obtained and altered by the potential of porous Si by varying different etching parameters which are eventually suitable for sensing devices.

ACKNOWLEDGEMENT

Foremost, I would like to express my gratefulness to Allah S.W.T for giving me this chance and strength to complete my Master. I would also like to express my deepest gratitude to my supervisor, Dr Rosfariza Radzali, and my co-supervisors, Dr Alhan Farhanah Abd Rahim, and Dr Puteri Sarah Mohamad Saad, for their full support, expert guidance, understanding, and encouragement throughout my research. Without their incredible patience and counsel, this thesis would not be completed. All the assistance from them is deeply appreciated.

I would also like to take this opportunity to thank all the team members, postgraduate students, laboratory staff from Universiti Teknologi MARA (UiTM) Cawangan Pulau Pinang, and NOR Universiti Sains Malaysia (USM).

My greatest appreciation to all my close friends who have been with me, encouraging me and providing me with ideas and suggestions throughout my Master's journey.

My thanks to all personnel that was involved in helping me complete this thesis of study be it directly or indirectly.

Finally, this thesis is dedicated to my parents and family for their unconditional love and support in completing my study. Alhamdulillah.

TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENT	v
TABLE OF CONTENTS	vi
LIST OF TABLES	ix
LIST OF FIGURES	xi
LIST OF ABBREVIATIONS	xiv
CHAPTER ONE: INTRODUCTION	1
1.1 Background of the Study	1
1.2 Problem Statement and Motivation	2
1.3 Research Objectives	3
1.4 Scope and Limitations of the Study	3
1.5 Significance of the Study	4
1.6 Thesis Organization	4
CHAPTER TWO: LITERATURE REVIEW	6
2.1 Introduction	6
2.2 Overview of The Process of Fabricating Porous Silicon	9
2.2.1 Fabrication of Porous Si by Dry Etching	10
2.2.2 Fabrication of Porous Si by Wet Etching	11
2.3 Principle of Electrochemical Etching in Si	15
2.4 Atomic Force Microscopy	17
2.5 Crystalline Properties	17
2.6 Raman Spectroscopy	19
2.7 Ideal Porous Si Properties	20
2.8 Chapter Summary	21

CHAPTER THREE: METHODOLOGY	22
3.1 Introduction	22
3.2 Flow Chart of the Study	22
3.3 Sample Preparation	24
3.4 Two-Step Alternating Current Photo-Electrochemical (ACPEC) Etching Technique	26
3.5 Characterization Tools	29
3.5.1 Structural and Optical Characterization	29
3.6 Safety Precautions	35
3.7 Chapter Summary	35
CHAPTER FOUR: RESULTS AND DISCUSSION	37
4.1 Introduction	37
4.2 The Effect of Different Etching Techniques on the Properties of Porous Si	37
4.2.1 Fields Emission Scanning Electron Microscopy Analysis	37
4.2.2 Atomic Force Microscopy Analysis	41
4.2.3 High Resolution X-Ray Diffraction Analysis	44
4.3 The Effect of Different Crystal Orientations on the Properties of Porous Si	46
4.3.1 Fields Emission Scanning Electron Microscopy Analysis	46
4.3.2 Atomic Force Microscopy Analysis	48
4.3.3 High Resolution X-Ray Diffraction Analysis	50
4.4 The Effect of Different Etching Durations on the Properties of Porous Si	53
4.4.1 Fields Emission Scanning Electron Microscopy Analysis	53
4.4.2 Atomic Force Microscopy Analysis	56
4.4.3 High Resolution X-Ray Diffraction Analysis	59
4.4.4 Raman Spectroscopy Analysis	61
4.5 The Effect of Different Current Densities on the Properties of Porous Si	63
4.5.1 Fields Emission Scanning Electron Microscopy Analysis	63
4.5.2 Atomic Force Microscopy Analysis	66
4.5.3 High Resolution X-Ray Diffraction Analysis	69
4.6 Chapter Summary	71
CHAPTER FIVE: CONCLUSIONS	72
5.1 Conclusions	72