DEVELOPMENT OF COMPOSITE ANODE FOR IMPROVING THE PERFORMANCE OF LI-ION BATTERIES

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DEVELOPMENT OF COMPOSITE ANODE FOR IMPROVING THE PERFORMANCE OF LI-ION BATTERIES

by

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Dedicated to my family...

Certificate

This is to certify that the thesis entitled **"Development of composite anode for improving the performance of Li-ion batteries"**, submitted by **Ms. Bhavya Nidhi Vats** to the Indian Institute of Technology Delhi, for the award of the degree of **Doctor of Philosophy**, is a record of the original, bonafide research work carried out by her under our supervision and guidance. The thesis has reached the standards fulfilling the requirements of the regulations related to the award of the degree.

The results in this thesis have not been submitted in part or in full to any other University or Institute for awarding any degree or diploma to the best of our knowledge.

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Abstract

Lithium-ion (Li-ion) batteries have revolutionized the energy storage landscape with their high energy density and long cycle life compared to other commercial battery technologies, making them a popular choice for a wide range of applications. Graphite is the primary choice for anode in commercial Li-ion batteries due to its excellent electrochemical stability, good electronic conductivity, and low cost. However, the low theoretical specific capacity of graphite anode ($\approx$ 372 mAh/g) has limited its use in high-energy applications. To address this limitation, composite anodes have emerged as a promising solution by integrating graphite with high-capacity anode materials. Silicon has the highest theoretical specific capacity ($\approx$ 4200 mAh/g), about ten times that of graphite. However, silicon suffers from significant volume changes during the charging and discharging process, leading to cracking and pulverization of the anode, ultimately degrading the battery performance.

This study presents the synthesis of core-shell nanoparticles, silicon (Si) as core and titania (TiO_2) as shell: Si@TiO_2, which are then reinforced into natural graphite to develop high-performance composite anode materials for Li-ion batteries. The peptization technique is employed to develop TiO_2 shell over silicon nanoparticles, which can effectively control the structural degradation during cycling. The present study reveals that the developed core-shell nanoparticles exhibit $\approx$ 37% improvement in capacity retention as compared to the bare silicon anode.

These core-shell nanoparticles are reinforced into the natural graphite in varying concentrations (5%, 10%, 15% w/w) for developing Graphite/Si@TiO_2 composite anode materials. Additionally, Graphite reinforced with 10% w/w bare silicon is also developed for benchmarking the performance of the developed composite anodes. During electrochemical cycling, graphite composites containing 5% w/w core-shell (GrCS5), 10% w/w core-shell (GrCS10), and 15% w/w core-shell (GrCS15) nanoparticles exhibit initial discharge capacities of 568 mAh/g, 675 mAh/g, and 716 mAh/g, respectively, retaining $\approx$ 76%, $\approx$75%, and $\approx$ 72% of their initial capacity after 100 cycles. In contrast, the graphite composite containing 10% w/w bare silicon (GrSi10) shows an initial discharge capacity of 728 mAh/g but retains only $\approx$ 57% of the initial capacity after 100 cycles. The study identifies GrCS10 as the

optimal core-shell percentage in the natural graphite using quadrant analysis for the development of an efficient composite anode.

Even though composite anodes demonstrate improved performance, they are prone to degradation when undergoing repeated charging and discharging cycles which impacts the overall battery performance. Post-cycling examination of batteries enhances the understanding of complex processes of the composite anodes for advancement in battery technology. Morphological and structural characterizations of GrCS10 and GrSi10 anodes are conducted before and after cycling using field emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), Energy dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD). The chemical changes after cycling are recorded using X-ray photoelectron spectroscopy (XPS).

The FE-SEM micrographs show minimal morphological variations in GrCS10 anode material, but significant changes are recorded in GrSi10 anode material. In case of GrSi10 anode, a layer of distinct physical appearance is noted on the surface. The cross-sectional FE-SEM micrographs reveal the swelling during cycling is more in the GrSi10 anodes compared to GrCS10. Further, the corresponding EDS mapping indicates the change in elemental composition within composite anodes after cycling. The changed electrochemical behavior of the composite anodes is attributed to the change in the composition of the solid electrolyte interphase (SEI) layer, as confirmed by the XPS, and minor loss in crystallinity of GrCS10 anode material, compared to GrSi10, as confirmed by the XRD. The study provides insights into the mechanisms governing material degradation during the electrochemical processes in the composite anodes.

सारांश

लिथियम आयन बैटरियों ने अपनी उच्च ऊर्जा घनता और लंबी चक्र आयु के कारण अन्य पारंपरिक बैटरी तकनीकों की तुलना में ऊर्जा भंडारण के क्षेत्र में क्रांतिकारी बदलाव लाया है। यही कारण है कि ये बैटरियाँ अनेक क्षेत्रों में व्यापक रूप से अपनाई जा रही हैं। ग्रेफाइट को वाणिज्यिक लिथियम आयन बैटरियों में एनोड सामग्री के रूप में प्रमुखता से चुना जाता है, क्योंकि इसमें उत्तम विद्युत-रासायनिक स्थिरता, अच्छी विद्युत चालकता तथा कम लागत होती है। तथापि, ग्रेफाइट की सैद्धांतिक विशिष्ट क्षमता लगभग ३७२ मिली ऐम्पियर घंटा प्रति ग्राम होने के कारण यह उच्च ऊर्जा अनुप्रयोगों के लिए सीमित है।

इस सीमा को दूर करने हेतु, उच्च क्षमता वाली एनोड सामग्री को ग्रेफाइट के साथ सम्मिलित कर सम्मिश्रित एनोड विकसित किए जा रहे हैं। सिलिकॉन की सैद्धांतिक विशिष्ट क्षमता लगभग ४२०० मिली ऐम्पियर घंटा प्रति ग्राम है, जो ग्रेफाइट की तुलना में लगभग दस गुना अधिक है। किंतु चार्ज और डिस्चार्ज की प्रक्रिया के दौरान सिलिकॉन में अत्यधिक आयतन परिवर्तन होता है, जिससे एनोड में दरारें और टूट-फूट हो जाती है, और बैटरी का समग्र प्रदर्शन घट जाता है।

इस अध्ययन में कोर शैल संरचना वाले नैनोकणों का निर्माण किया गया है, जिसमें सिलिकॉन को कोर और टाइटेनिया को शैल के रूप में लिया गया है। इन नैनोकणों को प्राकृतिक ग्रेफाइट में प्रविष्ट कर उच्च क्षमता वाले सम्मिश्रित एनोड विकसित किए गए हैं। सिलिकॉन नैनोकणों के ऊपर टाइटेनिया शैल विकसित करने के लिए पेप्टाइजेशन तकनीक का उपयोग किया गया, जिससे चक्रों के दौरान होने वाले संरचनात्मक क्षरण को नियंत्रित किया जा सके। अध्ययन में पाया गया कि ये कोर- शैल नैनोकण सामान्य सिलिकॉन एनोड की तुलना में लगभग सैंतीस प्रतिशत अधिक क्षमता बनाए रखते हैं।

इन कोर- शैल नैनोकणों को विभिन्न मात्रा में — पाँच प्रतिशत, दस प्रतिशत एवं पंद्रह प्रतिशत भार के अनुसार — ग्रेफाइट में मिलाकर ग्रेफाइट-कोर- शैल सम्मिश्रित एनोड विकसित किए गए हैं। साथ ही, दस प्रतिशत भार के साधारण सिलिकॉन को ग्रेफाइट में मिलाकर एक अन्य तुलनात्मक नमूना भी तैयार किया गया। विद्युत-रासायनिक परीक्षणों के दौरान पाया गया कि पाँच प्रतिशत, दस प्रतिशत और पंद्रह प्रतिशत कोर- शैल युक्त ग्रेफाइट में प्रारंभिक डिस्चार्ज क्षमता क्रमशः ५६८, ६७५, और ७१६ मिली ऐम्पियर घंटा प्रति ग्राम थी, तथा सौ चक्रों के पश्चात ये क्रमशः लगभग ७६, ७५ और ७२ प्रतिशत प्रारंभिक क्षमता बनाए रख पाए। इसके विपरीत, दस प्रतिशत साधारण सिलिकॉन युक्त ग्रेफाइट की प्रारंभिक क्षमता ७२८ मिली ऐम्पियर घंटा प्रति ग्राम थी, किंतु सौ चक्रों के पश्चात यह केवल ५७ प्रतिशत क्षमता बनाए

रख सका। इस आधार पर विश्लेषण से यह निष्कर्ष निकाला गया कि दस प्रतिशत कोर- शैल मिश्रण ग्रेफाइट के साथ सर्वोत्तम प्रदर्शन करता है।

हालाँकि सम्मिश्रित एनोड बेहतर प्रदर्शन दिखाते हैं, परंतु बारंबार चार्ज व डिस्चार्ज से इनकी कार्यक्षमता में गिरावट आती है, जो बैटरी की दीर्घकालिक दक्षता को प्रभावित करती है। चक्रों के पश्चात किए गए अध्ययन से इन सम्मिश्रित एनोडों में हो रहे जटिल परिवर्तनों को समझने में सहायता मिलती है, जो अगली पीढ़ी की बैटरियों के विकास के लिए आवश्यक है। जीआरसीएस१० और जीआरएसआई१० नामक नमूनों की संरचनात्मक एवं रासायनिक जांच चक्रों से पहले और बाद में की गई। इसके लिए क्षेत्रीय उत्सर्जन स्कैनिंग इलेक्ट्रॉन माइक्रोस्कोपी, ट्रांसमिशन इलेक्ट्रॉन माइक्रोस्कोपी, ऊर्जा विसरण एक्स-रे स्पेक्ट्रोस्कोपी तथा एक्स-रे विवर्तन तकनीकों का प्रयोग किया गया। साथ ही, एक्स-रे फोटोइलेक्ट्रॉन स्पेक्ट्रोस्कोपी द्वारा चक्रों के पश्चात रासायनिक परिवर्तनों को दर्ज किया गया।

क्षेत्रीय स्कैनिंग माइक्रोस्कोपी द्वारा यह पाया गया कि जीआरसीएस१० में संरचनात्मक रूप में बहुत कम परिवर्तन हुआ, जबकि जीआरएसआई१० में स्पष्ट भौतिक परिवर्तन दिखाई दिए। जीआरएसआई१० की सतह पर एक अलग प्रकार की परत देखी गई, और क्रॉस सेक्शनल चित्रों में यह स्पष्ट हुआ कि चक्रों के दौरान जीआरएसआई१० में अधिक सूजन हुई। साथ ही, तत्वीय विश्लेषण से यह स्पष्ट हुआ कि चक्रों के पश्चात सम्मिश्रित एनोडों की संघटन में परिवर्तन हुआ है। विद्युत-रासायनिक व्यवहार में आया परिवर्तन सॉलिड इलेक्ट्रोलाइट इंटरफेस परत की संरचना में बदलाव तथा जीआरसीएस१० की क्रिस्टलीयता में मामूली कमी के कारण है, जो कि एक्स आर डी द्वारा पुष्टि की गई। यह अध्ययन बैटरियों के संचालन के दौरान सामग्री क्षरण की प्रक्रियाओं को समझने में महत्वपूर्ण योगदान देता है।

Contents

Certificate

Acknowledgements

Abstract

Contents

List of Figures

List of Tables

Abbreviations

Symbols

1 Introduction **1**
1.1 Overview of battery technologies 1
1.2 Historical development of Li-ion battery 4
1.3 Components of Li-ion battery 7
1.4 Working mechanism 9
1.5 Anode and its materials 10
1.5.1 Classification of anode materials 11
1.6 Emerging challenges in anode materials 12
1.7 Research significance 13

2 Literature Review **15**
2.1 Graphite as a promising anode material 15
2.1.1 Operational mechanism 17
2.1.2 Potential characteristics of graphite as anode 18

2.1.3 Literature review on graphite anodes 19
2.2 Silicon: Opportunities and challenges as anode material 22
2.2.1 Operation mechanism 22
2.2.2 Fundamental challenges 23
2.2.3 Modification strategies for silicon anodes 25
2.3 Graphite/Si composite anode material 29
2.4 Degradation mechanism of composite anodes 33
2.5 Summary of literature review 36
2.6 Research gaps 37
2.7 Problem formulation 38
2.7.1 Problem definition 38
2.7.2 Research objectives 40
2.8 Organization of the thesis 40

3 Materials and Methods 43
3.1 Core-shell nanoparticle and composite anode 43
3.2 Material and structural characterization 46
3.2.1 Electron microscopy 46
3.2.2 Energy dispersive X-ray spectroscopy (EDS) 48
3.2.3 X-ray diffraction pattern(XRD) 48
3.2.4 X-ray photoelectron spectroscopy (XPS) 49
3.3 Coin cell development 50
3.3.1 Electrode preparation and cell assembly 51
3.3.2 Sample nomenclature 53
3.4 Electrochemical testing 55
3.4.1 Cyclic voltammetry (CV) 55
3.4.2 Galvanostatic charge discharge test (GCD) 55
3.4.3 Electrochemical impedance spectroscopy (EIS) 56
3.4.4 Lithium diffusion coefficient test 57
3.5 Quadrant analysis 58
3.6 Post-cycling analysis 59
3.6.1 Disassembly of cells 60
3.6.2 Morphological and structural characterization 60

4 Electrochemical Performance of Si@TiO_2 Anode 63
4.1 Introduction 63
4.2 Results and discussion 65
4.2.1 Structural characteristics 65
4.2.2 Surface chemistry analysis 70
4.2.3 Electrochemical response 71
4.3 Key findings 74

5 Electrochemical Performance of Graphite/Si@TiO_2 Composite Anode 77
- 5.1 Introduction . . . 78
- 5.2 Results and discussion . . . 79
  - 5.2.1 Structural characteristics . . . 79
  - 5.2.2 Electrochemical performance: A comparative evaluation with Graphite/Si anode . . . 81
  - 5.2.3 Optimality in composition of Graphite/Si@TiO_2 composite anode . . . 89
- 5.3 Key findings . . . 91

6 Synergy between Electrochemical Behaviour and Material Characteristics: Post-Cycling Analysis 93
- 6.1 Introduction . . . 94
- 6.2 Results and discussion . . . 95
  - 6.2.1 Effect of cycling on GrCS10 and GrSi10 . . . 95
  - 6.2.2 SEI Dynamics . . . 102
  - 6.2.3 Correlation between electrochemical behavior and material characteristics . . . 105
- 6.3 Key findings . . . 106

7 Conclusions and Future Scope 109
- 7.1 Conclusions . . . 109
- 7.2 Future scope . . . 112

Bibliography 113

List of Publications 139

Brief Biography 141

List of Figures

1.1 Lithium-ion market size and growth forecast, in US$ billions [1, 2] . . 3
1.2 Schematic diagram showing the components of Li-ion battery and its working mechanism . . . 7

2.1 Potential profile of first charge-discharge cycle of graphite [3] 16

3.1 Schematic representation of peptization technique for core-shell (Si@TiO_2) development . . . 44
3.2 Sequential process for the synthesis of Graphite/Si@TiO_2 composite anode . . . 46
3.3 FE-SEM instrument at IIT Delhi . . . 47
3.4 TEM instrument at IIT Delhi . . . 47
3.5 XRD instrument at IIT Delhi . . . 49
3.6 XPS instrument at IIT Delhi . . . 50
3.7 Coin cell development procedure . . . 53
3.8 Sample Nomenclature . . . 54
3.9 Electrochemical performance testing setup (Biologic VMP3) 56
3.10 Schematic diagram of quadrant analysis . . . 59

4.1 FE-SEM micrographs of (a) bare silicon nanoparticles, (b) Si@TiO_2 core-shell nanoparticles . . . 66
4.2 Morphology of Si@TiO_2 core-shell nanoparticles (a) FE-SEM and (b-f) corresponding EDS mapping . . . 67
4.3 TEM micrographs of Si@TiO_2 core-shell nanoparticles at scale of (a) 100nm (b) 20nm . . . 68
4.4 Morphology of Si@TiO_2 (a) STEM micrographs and (b) Corresponding EDS mapping . . . 68
4.5 XRD spectra of bare silicon and Si@TiO_2 core-shell nanoparticles . . 69
4.6 XPS spectra (a) Si@TiO_2 core-shell and silicon nanoparticles, (b-d) HR spectra of silicon, titanium and oxygen, respectively 70
4.7 Cyclic Voltammetry of Si@TiO_2 core-shell nanoparticles 72
4.8 Charge-discharge curve of Si@TiO_2 core-shell nanoparticles 73
4.9 Capacity retention curve of bare silicon and Si@TiO_2 core-shell nanoparticles . . . 74

5.1 FESEM micrograph of (a) bare graphite, (b) Graphite/Si@TiO_2 composite, (c-f) EDS mapping of Gr/Si@TiO_2 composite material 80
5.2 XRD spectra for graphite, silicon, Si@TiO_2 and Graphite/Si@TiO_2 . 80
5.3 Cyclic Voltammetry profiles for different composite anodes (a) GrCS5, (b) GrCS10, (c) GrCS15, (d) GrSi10 82
5.4 Charge/Discharge profiles for different composite anodes, (a) GrCS5, (b) GrCS10, (c) GrCS15, (d) GrSi10 83
5.5 Electrochemical response for different composite anodes (a) specific discharge capacity of 1^{st} and 100^{th} cycle (b) Capacity retention vs cycle number curve 85
5.6 EIS spectra of (a) GrCS5, (b) GrCS10, (C) GrCS15 (d) GrSi10 after 1^{st} and 100^{th} cycle 87
5.7 Lithium diffusion coefficient for all composite anodes after 100 cycles 89
5.8 Quadrant analysis for the identification of optimal composite anode composition 90

6.1 FE-SEM micrographs of GrCS10 anode before and after 100 cycles, (a, b) 100 μm (c, d) 1 μm and GrSi10 anode before and after 100 cycles (e, f) 100 μm (g, h) 1 μm respectively 96
6.2 FE-SEM micrographs of the top anode surface after 100 cycles with EDS mapping (a, b) GrCS10, (c, d) GrSi10 97
6.3 Cross-sectional FE-SEM micrographs of (a, b) GrCS10 (c, d) GrSi10 before and after 100 cycles, respectively 98
6.4 TEM micrographs of (a, b) GrCS10, (c, d) GrSi10 before and after 100 cycles, respectively 99
6.5 XRD spectra of GrCS10 (a, c) and GrSi10 (b, d) before and after 100 cycles 101
6.6 High-resolution XPS spectra for GrCS10 and GrSi10 composite anodes after 100 cycles 104
6.7 Schematic representation of degradation mechanism for GrSi10 and GrCS10 composite anodes during cycling 106

List of Tables

1.1 Properties of anode materials in Li-ion batteries 12

2.1 Literature review for different coating materials 27

3.1 Sample nomenclature for the developed coin cells 54

5.1 Resistance values simulated through equivalent circuits 87

6.1 Variation of electrochemical and physical properties of GrCS10 and GrSi10 composite anodes during cycling 105

Abbreviations

Al_2O_3	**A**luminium **O**xide
CAGR	**C**ompound **A**nnual **G**rowth **R**ate
CMC	**C**arboxy **M**ethyl **C**ellulose
CNT	**C**arbon **N**anotube
Co_3O_4	**C**obalt **O**xide
CV	**C**yclic **V**oltammetry
DEC	**D**iethyl **C**arbonate
DMC	**D**imethyl **C**arbonate
EC	**E**thylene **C**arbonate
EDS	**E**nergy **D**ispersive **X**-ray **S**pectroscopy
EIS	**E**lectrochemical **I**mpedance **S**pectroscopy
EVs	**E**lectric **V**ehicles
Fe_2O_3	**F**erric **O**xide
FE-SEM	**F**ield **E**mission **S**canning **E**lectron **M**icroscopy
GCD	**G**alvanostatic **C**harge **D**ischarge
GICs	**G**raphite **I**ntercalation **C**ompounds
GITT	**G**alvanostatic **I**ntermittent **T**itration **T**echnique
HEBM	**H**igh **E**nergy **B**all **M**illing
HUMO	**H**ighest **O**ccupied **M**olecular **O**rbit
ICE	**I**nitial **C**oulombic **E**fficiency
LCO	**L**ithium **C**obalt **O**xide
$LiAsF_6$	**L**ithium **H**exaflouroarsenate
LiC_6	**L**ithiated **G**raphite
$LiClO_4$	**L**ithium **P**erchlorate
LiF	**L**ithium **F**louride
Li-ion	**L**ithium-ion
LiPAA	**L**ithium **P**olyacrylic **A**cid
$LiPF_6$	**L**ithium **H**exaflourophosphate
LMO	**L**ithium **M**anganese **O**xide

LTO	**L**ithium **T**itanate **O**xide
LUMO	**L**owest **U**noccupied **M**olecular **O**rbit
MO	**M**etal **O**xide
NaCMC	**S**odium **C**arboxymethyl **C**ellulose
NaPAA	**S**odium **P**olyacrylate
NCA	**L**ithium **N**ickel **C**obalt **A**luminium **O**xide
NiMH	**Nickel** **M**etal **H**ydride
NMC	**L**ithium **N**ickel **M**anganese **C**obalt **O**xide
NMP	**N** **M**ethylpyrrolidone
PAN	**P**olyacrylo **N**itrile
PC	**P**ropylene **C**arbonate
PEI	**P**olyethylenimine
PVDF	**P**olyvinylidene **D**iflouride
rGO	**R**educed **G**raphene **O**xide
SCN	**S**ilicon **C**arbon **N**anocomposite
SEI	**S**olid **E]lectrolyte Interphase**
STEM	**S**canning **T**ransmission **E**lectron **M**icroscopy
TEM	**T**ransmission **E**lectron **M**icroscopy
TiO_2	**T**itanium **O**xide
TiS_2	**T**itanium **D**isulfide
TTIP	**T**itanium **T**etra-isopropoxide
UAV	**U**nmanned **A**erial **V**ehicle
XPS	**X** **R**ay **P**hotoelectron **S**pectroscopy
XRD	**X** **R**ay **S**pectroscopy

Symbols

T	Absolute temperature
°C	Celcius
cm	Centi-meter
C	Current rate
F	Faraday's constant
R	Gas constant
g	Gram
h	Hour
K	Kelvin
m	Micro-meter
mA	Mili-Ampere
M	Molar concentration
nm	Nano-meter
n	No of electrons
Ω	Ohm
%	Percentage
V	Volt
σ_w	Warburg's constant