



Micromechanical Characterization of Ceramic Solid Electrolytes for Electrochemical Storage Devices

Juliane Franciele Nonemacher

Energie & Umwelt / Energy & Environment

Band / Volume 490

ISBN 978-3-95806-461-4

Forschungszentrum Jülich GmbH
Institut für Energie- und Klimaforschung
Werkstoffstruktur und -eigenschaften (IEK-2)

Micromechanical Characterization of Ceramic Solid Electrolytes for Electrochemical Storage Devices

Juliane Franciele Nonemacher

Schriften des Forschungszentrums Jülich
Reihe Energie & Umwelt / Energy & Environment

Band / Volume 490

ISSN 1866-1793

ISBN 978-3-95806-461-4

Contents

Abstract	i
Kurzfassung	iii
Contents	v
List of Tables	ix
List of Figures	xi
1. Introduction	1
2. Literature Review	3
2.1. Batteries.....	3
2.1.1. Conventional and solid-state batteries	3
2.1.2. Solid-state electrolytes	7
2.1.3. Properties of solid-state electrolytes (physical, chemical and mechanical properties)	8
2.2. Mechanical characteristics	14
2.2.1. Elastic behavior (elastic modulus).....	14
2.2.2. Plastic behavior (hardness)	16
2.2.3. Fracture properties (fracture toughness).....	16
2.2.3.1. Vickers indentation fracture	17
2.2.3.2. Micro-pillar Indentation Splitting test	19
2.2.4. Mechanical considerations of solid-state electrolytes.....	21
2.2.4.1. Dendrite suppression	23
2.2.4.2. Mechanical properties of $\text{Li}_7\text{La}_3\text{Zr}_3\text{O}_{12}$ (LLZO)	26
3. Experimental.....	31
3.1. Materials synthesis	31
3.1.1. Cold pressed.....	31

3.1.2.	Hot pressed.....	33
3.2.	Microstructural characterization	34
3.2.1.	X-Ray diffractometry.....	34
3.2.2.	Inductively coupled plasma optical emission spectrometry	35
3.2.3.	Electrochemical impedance spectroscopy	35
3.2.4.	Metallographic preparation.....	36
3.2.5.	Scanning electron microscope and electron backscatter diffraction.....	36
3.2.6.	Focused ion beam	36
3.3.	Mechanical characterization.....	37
3.3.1.	Depth-sensitive indentation test.....	37
3.3.2.	Indenation fracture toughness	38
3.3.3.	Micro-pillar splitting test	39
4.	Results and discussion.....	44
4.1.	Substitution effect of $\text{Li}_7\text{La}_3\text{Zr}_2\text{O}_{12}$	45
4.1.1.	Conductivity, phase composition and microstructure.....	45
4.1.2.	Mechanical testing results.....	50
4.1.3.	Conclusions and outlook.....	55
4.1.4.	Porosity effect	57
4.1.5.	Moisture associated degradation.....	78
4.2.	Fracture toughness Al-substituted $\text{Li}_7\text{La}_3\text{Zr}_2\text{O}_{12}$ (Al:LLZO) by utilizing micro-pillar indentation splitting test	81
4.2.1.	Conductivity, phase composition and microstructure.....	81
4.2.2.	Mechanical testing results.....	83
4.2.3.	Conclusions and outlook.....	92
4.3.	Hot pressed LLZO.....	94

4.3.1.	Conductivity, phase composition and microstructure.....	94
4.3.2.	Mechanical testing results.....	97
4.3.2.1.	Vickers indentation toughness.....	99
4.3.2.2.	Micro-pillar splitting test.....	102
5.	Conclusions and Outlook.....	110
6.	References	113
	Appendix.....	125
	Publications & Conferences.....	128
	Acknowledgements.....	130

Energie & Umwelt / Energy & Environment
Band / Volume 490
ISBN 978-3-95806-461-4