
Bimodal microstructure and fatigue properties of nanocrystalline and ultrafine grained nickel

Dissertation

Zur Erlangung des Grades des
Doktors der Ingenieurwissenschaften (Dr.-Ing.)
der Naturwissenschaftlich-Technischen Fakultät III
Chemie, Pharmazie, Bio- und Werkstoffwissenschaften
der Universität des Saarlandes

Von
M.Sc. Tao Qian
Saarbrücken, 2013

Eingereicht am: 07.02.2013
Tag des Kolloquiums: 24.07.2013
Dekan: Prof. Dr. Volkhard Helms
Berichterstatter: Prof. Dr. H. Vehoff
Prof. Dr. R. Busch
Vorsitzender: Prof. Dr. C. Boller
Akad. Mitarbeiter: Dr. C. Gachot

Saarbrücker Reihe

Materialwissenschaft und Werkstofftechnik

Band 39

Bimodal microstructure and fatigue properties of nanocrystalline and ultrafine grained nickel

Tao Qian

Herausgeber:

Prof. Dr. rer. nat. Horst Vehoff

Prof. Dr. Eduard Arzt
Prof. Dr.-Ing. Dirk Bähre
Prof. Dr.-Ing. Christian Boller
Prof. Dr. rer. nat. Ralf Busch
Prof. Dr. rer. nat. Rolf Clasen
Prof. Dr.-Ing. Stefan Diebels
Prof. Dr.-Ing. Frank Mücklich
Prof. Dr. rer. nat. Martin Müser
Prof. Dr. rer. nat. Wulff Possart
Prof. Dr.-Ing. Markus Stommel

Shaker Verlag

Aachen 2014

Bibliographic information published by the Deutsche Nationalbibliothek

The Deutsche Nationalbibliothek lists this publication in the Deutsche Nationalbibliografie; detailed bibliographic data are available in the Internet at <http://dnb.d-nb.de>.

Zugl.: Saarbrücken, Univ., Diss., 2013

Copyright Shaker Verlag 2014

All rights reserved. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording or otherwise, without the prior permission of the publishers.

Printed in Germany.

ISBN 978-3-8440-2656-6

ISSN 1860-8493

Shaker Verlag GmbH • P.O. BOX 101818 • D-52018 Aachen

Phone: 0049/2407/9596-0 • Telefax: 0049/2407/9596-9

Internet: www.shaker.de • e-mail: info@shaker.de

To my wife Ying and my son Haoting

Acknowledgments

Looking back on my time in the Institute of Materials Science and Methods in Saarland University, I would like to express my sincere thanks to all of those who have supported me and have thereby contributed to the accomplishments in this thesis. It is my distinct pleasure to acknowledge here my coworkers, friends and family, without whom I would not have chance to finish this work.

In the first place I would like to express my gratitude to Prof. Dr. rer. nat. Horst Vehoff for providing me the opportunity and the guidance of my Ph.D study. He has an exceptional insight in the material science and his helpful discussions and scientific comments inspired me to continue my research.

Equally important, I am sincerely grateful to Dr. Michael Marx for his supervision and support to my work and research. He was always very patient to take discussions with me and to propose many meaningful suggestions. From a beginner to a person who acquires good skills on the electron microscope I learned much from him. Besides, he spent lots of time to read this thesis and gave his critical comments.

I gratefully acknowledge Prof. Dr. rer. nat. Ralf Busch for being part of the Reading Committee and his comments about this thesis.

I am grateful to the financial support from the German Research foundation (DFG) with the researching project MA 3322/3-1.

I would like to thank Philipp Kerger, my assistant, for preparing my specimens, conducting the heat treatment experiments and processing the experimental data, Chen Chen for preparing my specimens, and Halima Bayad for supporting in the in-situ annealing.

My thanks go particular to Kerstin Schüler for being a such nice colleague and friend and helping me in different areas, to Dr. Nousha Kheradmand for sharing the office and many interesting talks, to Dr. Afroz Barnoush for sharing of his knowledge, to Dr. Wolfgang Schäf for introducing the FIB-crack method and performing the hydraulic testing machine, and to Mohammad Zamanzadeh, Dr. Camille Perrin, Dr. Markus Welsch and Alain Knorr for their help in the institute. I would also like to thank my friend Dirk Hillerbrecht for his patience improving my German speaking and our

Acknowledgments

long-standing badminton playing.

I am very grateful to our technicians Peter Limbach and Stephan Schmitz who were always ready for preparing my specimens and had good ideas and workmanship for my specimen holders, to Andreas Kirsch from whom I could get my support and help anytime and anywhere, and to Rita Maron for her availability and support in the laboratories. I would like to thank Mrs. Elisabeth Ohm and Miss Diana Born for their support in the institute life and making the administrative work.

I would like to than Dr. I. Karaman in Texas A&M University for processing of the ECAP nickel billets, to thank Dr. Flavio Soldera and Dipl.Ing. Christoph Pauly from the Institute of the functional Materials for their support in the FIB operation and the preparation of my first microcracks, to thank Dipl.Ing. Benjamin Bax from the Institute of the functional Materials for conducting the surface laser heat treatment, and to thank Dipl. Ing. Jörg Schmauch from the Institute of Technical Physics for his support in performing the EBSD measurements and the other REM and TEM measurements.

I would like to thank my parents who give me everything but never expect anything in return. Their support, encouragement and love make me go further in my life. I would like to thank my parents in-law for their understanding and supporting to take care of my wife and my son when I was so long absent.

Finally, I am so grateful to my wife Ying for her love, without her understanding and encouragement it is impossible for me to finish this work, and to my lovely son Haoting for bringing me so much happiness and giving me courage and power to keep on going.

Contents

Acknowledgments	iii
List of Figures	ix
List of Tables	xvii
Abstract	xix
Zusammenfassung	xxi
Acronyms	xxiii
1. Introduction	1
2. Literature Review	5
2.1. Nanocrystalline and ultrafine grained materials	5
2.1.1. Electrodepositon and equal channel angular pressing	5
2.1.2. Mechanical properties	9
2.1.3. Bimodal grain size distribution	13
2.2. Fatigue properties and crack behaviour of the NC and UFG materials . .	18
2.2.1. Fatigue properties	18
2.2.2. Crack initiation and growth	24
2.3. Annealing phenomena and nanoscale grain growth kinetics	28
2.3.1. Recovery, recrystallization and grain growth	28
2.3.2. Nanoscale grain growth kinetics	36
3. Experimental	39
3.1. Materials investigated	39
3.2. Specimen preparation	40
3.2.1. Specimens for mechanical tests	40
3.2.2. Surface preparation	40

Contents

3.3. Microstructural analysis	41
3.4. Heat treatment	43
3.5. Mechanical property measurements	46
3.5.1. Microindention	46
3.5.2. Tensile tests	46
3.6. Fatigue experiments	46
4. Results	51
4.1. Microstructural characterization	51
4.2. Microstructures after heat treatment	55
4.2.1. PED nickel annealed at 250 °C	55
4.2.2. ECAP nickel annealed at 250 °C	60
4.2.3. PED nickel annealed at 500 °C	65
4.3. Microhardness	75
4.4. Tensile tests	77
4.5. Fatigue experiments to different microstructures	80
4.5.1. PED NC, NC/UFG and UFG nickel with FIB microcracks	80
4.5.2. In-situ fatigue experiment of the bimodal PED NC/UFG nickel	86
4.5.3. ECAP UFG nickel with FIB microcracks	87
4.5.4. ECAP UFG nickel with macro-notches	95
4.5.5. Fatigue behaviour of the bimodal ECAP UFG/CG nickel	100
5. Discussion	105
5.1. Initiation of bimodal NC/UFG microstructure from PED NC nickel	105
5.2. Initiation of bimodal UFG/CG microstructure from ECAP UFG nickel	111
5.2.1. Influence of the annealing temperatures	111
5.2.2. Influence of the annealing time	112
5.2.3. Correlation between the microtextures and recrystallization	114
5.2.4. In-situ annealing of the ECAP nickel	118
5.2.5. Conclusion to the annealing of ECAP UFG nickel	121
5.3. Normal and abnormal grain growth of PED nickel at elevated temperature	122
5.4. Influence of the microstructures to the microhardness	126
5.5. Influence of the microstructures to the tensile properties	132
5.6. Influence of the microstructures of PED nickel to the fatigue properties	136
5.6.1. Crack growth behaviour of different microstructures	136
5.6.2. In-situ fatigue experiment for the bimodal NC/UFG nickel	147

5.6.3. Conclusions to the fatigue properties of the fine grained PED nickel	152
5.7. Influence of the microstructure of ECAP nickel to the fatigue properties	154
5.7.1. Fatigue of the ECAP UFG nickel with microcracks	154
5.7.2. Fatigue of the notched specimens	162
5.7.3. Fatigue of the ECAP bimodal UFG/CG nickel	164
5.7.4. Conclusions to the fatigue properties of the ECAP nickel	165
6. Conclusion and Outlook	167
6.1. Conclusions	167
6.2. Outlooks	169
A. Parameters for the PED NC nickel	173
B. Analysis of the microstructure of the ECAP nickel	175
C. In-situ fatigue experiment of the bimodal NC/UFG nickel	179
D. ECAP nickel heat treated with laser beam	183