Grain boundary-dislocation interaction: A local investigation via micron-sized bicrystals

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

Nousha Kheradmand

Saarbrücken, Mai 2012

Tag des Kolloquiums:	10.05.2012
Dekan:	Prof. Dr. W.F. Maier
Berichterstatter:	Prof. Dr. H. Vehoff
	Prof. Dr. F. Mücklich
Vorsitzender:	Prof. Dr. R. Hempelmann
Akad. Mitarbeiter:	Dr. Isabella Gallino

Saarbrücker Reihe

Materialwissenschaft und Werkstofftechnik

Band 33

Grain boundary-dislocation interaction: A local investigation via micron-sized bicrystals

Nousha Kheradmand

Shaker Verlag

Aachen 2012

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., 2012

Printed in Germany.

ISBN 978-3-8440-1158-6
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

Contents

Acknowledgments

In the first place I would like to express my utmost gratitude to Prof. Dr. rer. nat. Horst Vehoff for his contribution to the completion this research work. I will never forget his insight into the world of science and his unique way of teaching and supervision, which makes one proud to be a scientist. Without his support and understanding during the beginning of the "baby time", I may not have been able to finish my PhD now.

I gratefully acknowledge Prof. Dr. Ing. Frank Mücklich for accepting to being a part of the Reading Committee of this thesis, and also for his support all along my studies at Saarland University, specifically within the AMASE Program.

I would like to thank Christine Welsch, my diploma student, for performing multi-sided experiments, from lithography, to cutting pillars in FIB, to nanoindentation, and Vitoria Boiko, my HiWi, for performing the long-term heat treatments of Ni samples.

Many thanks go in particular to Dr. Ing. Michael Marx, a multi-talented colleague and friend in social life, for constructive scientific comments, to Dr. Ing. Afrooz Barnoush for his fabulous theoretical and experimental ideas anytime and performing the MD simulations of this thesis, to Kerstin Schüler and Dirk Hildebrecht for being such nice friends and having unforgettable time together, to Tao Qian for sharing the office and many interesting discussions about saving the world, to Mohammad Zamanzadeh for his support when I needed his helps and hearing me when I needed somebody to talk, to Alain Knorr for helpful discussions on the heat treatment of Ni and invention of a new imaging equipment for my Ni samples: his scanner!, to Dr. Ing. Wolfgang Schäf for assisting in the start the *theoretische Grundlage der Werkstoffwissenschften*, to Dr. Ing. Markus Welsch, our EBSD measurements master, to Dr. Ing. Mark Henning for leaving so many useful self-developed software behind for us, to Dr. Camille Perrin for bringing the French culture nearer to us.

Collective and individual acknowledgments are also owed to other diploma and master students in our group: Bastian Philippi, Vera Marx, Martin Weinmann, Michael Bick, Velayarce Jorge Rafael, Philip Kerger, Markus Mischo.

Many thanks to Stephan Schmitz and Peter Limbach whose handiwork and good ideas served to design my sample holders, and who were fast if I needed something urgently. I also thank Andreas Kirsch and Rita Maron for their availability and support for laboratory materials. Many thanks to Frau Ohm, who accompanied us in the institute almost until the end of my work, for making the administrative work easier, and Frau Born for being so practical and fast in response. To Frau Galli and Melani Groh many thanks for organizing our library very well.

I would like to thank Prof. Mao Wen from the school of Materials Science and Engineering at Shanghai Jiao Tong University in China for providing us with his MD simulation code and his support during the simulation of the bicrystalline micropillar compression tests.

From the Institute of Functional Materials I would like to acknowledge Dr. Ing. Flavio Soldera, Christoph Pauly, Dominik Britz and Agustina Guitar for their support in the preparation of thin films, and special thanks to Paula Souza for the professional and quick English proof-reading.

From the Institute of Engineering Mechanics I would like to thank Dipl. Ing. Zhaoyu Chen for performing the FEM simulations of micropillar compression tests for this research and from the Institute of Technical Physic many thanks to Dipl. Ing. Jörg Schmauch for his assistance in performing the high resolution EBSD measurements.

Finishing this thesis would not have been possible for me without the incredibly nice people at the “Kindertagesstätte für Bedienstete der Universität des Saarlandes”, specially Frau Hombücher, Nicole Wagner, Regiene Bleyer, Sandra and Manuela. I was extraordinarily fortunate in having you to take care of my child.

I would like to thank my parents, whose education and encouragement, love and sincerity brought me to where I am standing now; my brother and my other family members in Saarbrücken for their supports in all its forms; my parents in-law for their understanding and patience, and an angel in our neighborhood, Frau Hoffmann.

Finally, to my precious husband Afrooz, for your help especially in the crucial moments we spent together, for your permanently *good ideas* and your support in my scientific and daily life. And last but not least, my special thanks will be dedicated to my beautiful son *Suren*, for showing me that there is something more challenging than a PhD thesis.

Thank you all!

List of Tables

List of Figures

Abstract

In this research work, an experimental method is developed at the mesoscopic scale to investigate the interaction of dislocations with a selected grain boundary and its strengthening effect as a function of the grain boundary type.

The local mechanical testing method is based on microcompression tests of Focused Ion Beam (FIB)-cut bicrystalline micropillars with the component crystals oriented for single slip and multiple slip. Orientations identical to the experiments are used to generate models of the bicrystalline micropillars with up to four million atoms (140 nm in diameter) in Molecular Dynamics (MD) simulations. The compression test of these bicrystals is followed by Electron Backscatter Diffraction (EBSD) measurements on the bicrystal cross sections to investigate crystal lattice rotation in correlation with the excess dislocation density.

The microscopic test specimens are fabricated using high-voltage ion beam currents, which leads to the interaction of the ions with the host material. This problem, referred to as "FIB damage", was examined by high-resolution EBSD and nanoindentation techniques. The results show that FIB damage is a function of the ion beam current and the crystallographic orientation of the lattice, and that its main effect is the introduction of surface defects and the facilitation of dislocation nucleation.

Different sized bicrystals, from 1 to 5 μm in diameter, show different deformation behaviors. In bicrystals over 2 μm in diameter, identical flow stresses to single crystals with multiple slip orientation are obtained. These bicrystals resemble two single-crystalline micropillars connected in parallel and Taylor hardening is the responsible mechanism of deformation. Diameters below 2 μm, where the grain boundary-dislocation interaction plays a more crucial role than the dislocation-dislocation interaction, show a pronounced hardening effect of the grain boundary. Our EBSD measurements and the orientation analyses on the bicrystals with 1 μm diameters prove the increase of the misorientation in the vicinity of the grain boundary. In contrast, in a large bicrystalline micropillar with a 5 μm diameter, the orientation gradient is observed only in the bottom-up direction (parallel to the loading axis), which is a clear evidence of the independent deformation of the adjacent crystals. In agreement with the literature, lattice rotation is required for slip transmission and, thus, for compatible deformation of the bicrystals.

Zusammenfassung

Im Rahmen dieser Arbeit wurde eine experimentelle Methode entwickelt, um die Wechselwirkung zwischen Versetzungen und ausgewählten Korngrenzen, sowie ihre Verfestigungseffekte auf einer mesoskopischen Skala als Funktion des Korngrenzentyps zu untersuchen. Die lokale mechanische Testmethode basiert auf Mikro-Drucktests von Focused Ion Beam (FIB) geschnittenen bikristallinen Mikropillars, deren Einzelkristalle für Einfachgleitung sowie für Mehrfachgleitung orientiert sind.

Die gleichen Orientierungen werden benutzt, um Drucktests an bikristallinen Mikropillars mit bis zu vier Millionen Atomen mittels Molekular-Dynamik-(MD) Rechnungen zu simulieren. Im Anschluss an die Druckversuche wurden Electron BackScatter Diffraction (EBSD) Messungen auf der Querschnittsseite der Mikropillars durchgeführt, um die Gitterrotation des Kristalles in Korrelation mit Überschussversetzungen (excess dislocations) zu bestimmen.

Die Mikropillars wurden mit Ionenstrahlen hoher Beschleunigungs-Spannung hergestellt, was üblicherweise zu einer Wechselwirkung zwischen den Ionen und dem Probenmaterials führt. Dieses als “FIB Schädigungseffekt” bekannte Problem wurde durch hoch aufgelöste EBSD-Messungen und durch die Nanoindenter Messungen überprüft. Die Ergebnisse zeigen, dass die FIB Schädigung eine Funktion der Ionenstrahlstärke und der kristallographische Gitterorientierung ist und, dass ihr Haupteffekt die Erzeugung von Oberflächendefekten und dadurch eine erleichterte Versetzungsnukleation ist.

Unterschiedlich große Bikristalle von 1 bis 5 μm Durchmesser zeigen unterschiedliches Verformungsverhalten. In Bikristalle über 2 μm Durchmesser ist die Fließspannung gleich der Fließspannung einkristalliner Mikropillars, die für Mehrfachgleitung orientiert sind. Diese Bikristalle gleichen zwei einkristallinen “parallel geschalteten” Mikropillars wobei die Taylor Verfestigung die Verformung kontrolliert. Bikristalle unter 2 μm Durchmesser zeigen ausgeprägte Verfestigungseffekte der Korngrenze, wobei die Wechselwirkung zwischen Korngrenze und Versetzung eine wesentlich wichtigere Rolle als die Wechselwirkung der Versetzungen untereinander spielt.

Die EBSD Messungen an Bikristallen mit 1 μm Durchmesser und die darauf basierende Orientierungsanalyse weisen eine steigende Fehlorientierung in unmittelbarer Nähe der Korngrenze nach. Im Gegensatz dazu ist in einem großen Bikristall mit 5 μm Durchmesser der Orientierungsgradient nur in der “Bottom-Up”-Richtung (parallel zu der Belastungsrichtung) zu beobachten, was ein klarer Beweis für die unabhängige Verformung beider Einzelkristalle ist. In Übereinstimmung mit der Literatur konnte die Gleittransmission als Ursache für die Gitterrotation bestätigt werden und somit für die kompatible Verformung der Bikristalle als erforderlich identifiziert werden.

I would probably not say everything
I think, but definitely think all I say.

Gabriel Garcia Marquez