## HIGH TEMPERATURE DEFORMATION OF OCTACHLOROPROPANE: A MICROSTRUCTURAL STUDY

bу

Jin-Han Ree

# A Dissertation Submitted to the State University of New York at Albany in Partial Fulfillment of the Requirements for the Degree of Doctor of Philosophy

College of Sciences and Mathematics

Department of Geological Sciences

## HIGH TEMPERATURE DEFORMATION OF OCTACHLOROPROPANE: A MICROSTRUCTURAL STUDY

by

Jin-Han Ree

Abstract of a Dissertation

Submitted to the State University of New York at Albany
in Partial Fulfillment of
the Requirements for the Degree of

Doctor of Philosophy

College of Sciences and Mathematics

Department of Geological Sciences

#### **ABSTRACT**

As an aid to understanding the high-temperature microstructures of rocks, the development of microstructures in the hexagonal organic material, octachloropropane, was studied with *in-situ* optical microscopy. It was found that the deformation behavior of grains in hard and soft orientations for slip is different during simple shearing, although they both grow. Strain heterogeneity is induced by partitioning of deformation into relatively increased components of rigid-body rotation and translation in hard grains and strains in soft grains.

A steady-state foliation, having a constant intensity and orientation was observed in simple shearing. The steady state is maintained by a balance between foliation-strengthening and -weakening processes. The major foliation-strengthening process is intragranular strain, and the major foliation-weakening process is dynamic recrystallization including migration of straight or slightly wavy grain boundaries, grain dissection and rotational recrystallization. Other minor weakening processes are grain amalgamation, relative rigidity of hard grains and grain boundary sliding. Foliation intensity is lower than the axial ratio of the bulk strain ellipse by a factor 0.2 - 0.4 at a total shear strain of 1.3 - 1.8, indicating that grain-shape foliations of this type cannot be used for strain calculation.

Subgrain boundaries which appear similar under optical microscopy originate in seven different ways. They are classical polygonization, kinking, misorientation reduction, grain coalescence, impingement of migrating subgrain boundaries, edgewise propagation, and static development of subgrain boundaries from optically strain-free grains. The preferred orientation of subgrain boundaries with respect to the grain-shape foliation is symmetric in pure-sheared samples and asymmetric in simple-sheared samples.

Grain boundary sliding can occur by discontinuities in the strain, rotation and/or

translation components of deformation across the boundary in deforming samples. Grain boundary diffusion and intragranular plastic deformation are found to be effective in accommodating grain boundary sliding. Grain boundary openings can develop in association with grain boundary sliding, preferentially along grain boundaries at a low angle to the shortening direction. Once grain boundary openings occur, they continuously change their shape and are eventually closed by thrusting of sliding grains and grain overgrowth into the openings. An approximately equal volume of new openings grow in other places, however, maintaining a steady ratio of 0.5 - 3% of the sample volume without development of any large scale fracture. The opening and closing of grain boundaries usually involve neighbor switching of surrounding grains.

#### ACKNOWLEDGEMENTS

This dissertation has benefited from the direct or indirect help of many people. Win Means proposed the topic of this dissertation, and many of the ideas of this study were materialized through discussions with him during our informal 'coin' meetings and 'man-to-man' meetings. I thank him for his continuous support and patience throughout my research at Albany. His financial support made my travels to GSA annual meetings and the Leeds Conference meeting possible, where I was able to present my papers, discuss many topics related to my research with other structural geologists, and open my eyes to current issues of structural geology. Also my two-month visit to the University of Utrecht was possible because of his beneficence. There I learned Utrecht's further development of synkinematic microscopy, computer programs and image analysis, and I conducted a series of creep tests on paradichlorobenzene. I would like to thank Brian Bayly, Jan Tullis, Bill Kidd, Mark Jessell, Janos Urai, Peter Hudleston, Rob Knipe, Jane Gilotti, Paul Bons and Chris Mawer for reading part or all of this work and for making critical comments which resulted in substantial improvements. I also thank Bruce Hobbs and Mike Etheridge for suggesting some helpful ideas for my research project when they visited Albany.

I thank Janos Urai and Cees Passchier for making my visit to Utrecht possible.

During my stay there, discussions with Paul Bons and Coen ten Brink helped to improve my computer programs used in this study. I also thank Janos Urai and Chris Spier for suggesting some helpful ideas for my research project during the stay. Dean D. Wulff of the College of Sciences and Mathematics, SUNYA, also provided some financial support for my visit to Utrecht.

I benefited from discussions with faculty members of the department, John Delano, Steve DeLong, Greg Harper and George Putman, and fellow graduate students, Youngdo Park, Young-Joon Lee, Yun Pan and Rob Alexander through my student

seminars or at my request. Steve Tice, Rob Alexander and Terry Spell helped to improve my English and did not mind my pop-in visits to their office. I particularly thank Youngdo Park and Jaiyoung Rhi, who were always on hand whenever I had a problem with computer programing. Diana Paton kept administrative troubles away from me. Brian Taylor helped lessen technical problems with my experiments.

I thank the late Professor Bong-Soon Park, who was my Master's thesis advisor at Korea University. He ignited my interest in structural geology, and encouraged me to continue studying in the U.S.A. Chris Mawer and Jeff Grambling of the University of New Mexico (Chris is now in Australia), and James Roberston and Paul Bauer of the New Mexico Institute of Mining and Technology helped me to go to the 'right' place for my Ph.D. study when I first came to the U.S.A. I thank all of them.

My study at Albany would not have been possible without the support of my parents and parents-in-law. I particularly thank my mother for coming to Albany to take care of my baby, Hwisoo, while I was in Utrecht and my wife, Boknam, was at school. I also thank Yunmi Kim for taking care of Hwisoo without any problem while we both were at school. Finally I thank Boknam for her patience and love throughout my study. This work was funded by U.S. National Science Foundation grants EAR8506810 and EAR8803096 to Win Means.

### TABLE OF CONTENTS

ABSTRACT	i
ACKNOWLEDGMENTS	iii
TABLE OF CONTENTS	v
LIST OF FIGURES	viii
LIST OF TABLES	хi
CHAPTER 1. INTRODUCTION	1-1
CHAPTER 2. DETAILS OF EXPERIMENTS AND ANALYTICAL METHODS	
2.1 INTRODUCTION	2-1
2.2 EXPERIMENTAL TECHNIQUES	2-3
2.2.1 Sample preparation	2-3
2.2.2 Deformation apparatus	2-4
2.2.3 Photographic recordings	2-6
2.2.4 c-axis measurement	2-6
2.3 ANALYTICAL METHODS	2-13
2.3.1 Calculation of deformation parameters	2-13
2.3.2 Grid and marker particle trajectory maps	2-14
2.3.3 Bulk and intragranular strains	2-15
2.3.4 Measurement of grain-shape foliation and grain boundar	y
orientation	2-16
2.3.5 Deformation of passive grain boundaries	2-18
2.3.6 Measurement of grain area	2-19
CHAPTER 3. STRAIN HETEROGENEITY, DYNAMIC GRAIN GROWTH AND	D
LATTICE REORIENTATION	
3.1 INTRODUCTION	3-1
3.2 OBSERVATIONS AND ANALYSES	3-2
3.2.1 General	3-2
3.2.2 Deformation pattern	3-6
3.2.3 Grain-size history	3-9
3.3 DYNAMIC GRAIN GROWTH AND STRAIN HETEROGENEITY	3-12
3.4 LATTICE REORIENTATION	3-19

3.4.1 c-axis trajectories	3-19
3.4.2 Lattice rotation vs. material-line rotation	3-22
3.5 TEST OF SACHS AND TAYLOR PREDICTIONS	3-28
3.6 DISCUSSION	3-32
3.7 CONCLUSIONS	3-33
CHAPTER 4. DEVELOPMENT OF STEADY-STATE FOLIATION	
4.1 INTRODUCTION	4-1
4.2 EXPERIMENTAL TECHNIQUES	4-2
4.3 OBSERVATIONS AND ANALYSES	4-3
4.3.1 General	4-3
4.3.2 Deformation pattern	4-8
4.3.3 c-axis reorientation	4-8
4.3.4 Grain-size history	4-13
4.3.5 Foliation history	4-13
4.4 PROCESSES FOR STEADY-STATE FOLIATION	4-19
4.5 DISCUSSION	4-27
4.6 CONCLUSIONS	4-32
CHAPTER 5. MULTIPLE ORIGINS OF SUBGRAIN BOUNDARIES	
5.1 INTRODUCTION	5-1
5.2 EXPERIMENTAL DESCRIPTIONS	5-1
5.2.1 Experiment TO-91	5-1
5.2.2 Experiment TO-207	5-6
5.2.3 Experiment TO-11	5-6
5.2.4 Other experiments	5-6
5.3 SEVEN TYPES OF SUBGRAIN BOUNDARIES	5-7
5.3.1 Type I subgrain boundaries	5-9
5.3.2 Type II subgrain boundaries	5-12
5.3.3 Type III subgrain boundaries	5-12
5.3.4 Type IV subgrain boundaries	5-16
5.3.5 Type V subgrain boundaries	5-22
5.3.6 Type VI subgrain boundaries	5-22
5.3.7 Type VII subgrain boundaries	5-27
5.3.8 Population of subgrain boundaries	5-30
5.4 SUBGRAIN BOUNDARY ORIENTATION AND DENSITY	5-30
5.4.1 Subgrain boundary orientation of each type	5-30

5.4.2 Evolution of subgrain boundary orientation	5-30
5.4.3 Evolution of subgrain boundary density	5-33
5.4.4 Comparison of subgrain boundaries between pure shear	
and simple shear	5-33
5.5 DISCUSSION	5-36
5.6 CONCLUSIONS	5-40
CHAPTER 6. GRAIN BOUNDARY DEFORMATION AND DEVELOPMENT OF	
GRAIN BOUNDARY OPENINGS	
6.1 INTRODUCTION	6-1
6.2 GRAIN BOUNDARY SLIDING AND ITS ACCOMMODATION	6-2
6.3 EXPERIMENTAL EXAMPLES	6-6
6.3.1 General	6-6
6.3.2 Experiment TO-110	6-6
6.3.3 Experiment TO-105	6-12
6.3.4 Experiment TO-202	6-23
6.4 EVOLUTION OF GRAIN BOUNDARY OPENINGS	6-29
6.5 DISCUSSION	6-37
6.5.1 Grain boundary sliding and its accommodation	6-37
6.5.2 Implication of grain boundary openings	6-38
6.5.3 Recognition of grain boundary sliding and opening	6-39
6.5.4 Three types of grain boundary migration	6-42
6.6 CONCLUSIONS	6-44
REFERENCES	7-1
APPENDIX 1. EQUATIONS OF DEFORMATION PARAMETERS AND	
COMPUTER PROGRAMS	
A1.1 EQUATIONS OF DEFORMATION PARAMETERS	A1-1
A1.1.1 Equations of Dij and Ti	A1-1
A1.1.2 Equations of D parameters	A1-2
A1.2 COMPUTER PROGRAMS	A1-4
A1.2.1 General	A1-4
A1.2.2 Programs GRID, MPT and TRI	A1-4
A1.2.3 Program GBO	A1-9
A124 Program GRD	A1-13

APPENDIX 2. FURTHER DETAILS OF STEADY-STATE FOLIATION	
A2.1 FOLIATION DEVELOPMENT	A2-1
A2.1.1 Sample TO-109	A2-1
A2.1.2 Sample TO-105	A2-5
A2.2 COMPARISON OF OTHER MICROSTRUCTURES AND	
DEFORMATION PATTERN	A2-9
A2.2.1 c-axis fabrics	A2-9
A2.2.2 Deformation pattern	A2-11
A2.2.3 Grain-size history	A2-14
A2.2.4 Grain boundary migration	A2-16
A2.3 DISCUSSION AND CONCLUSION	A2-18
LIST OF FIGURES	
2.1 Deformation geometry of the OCP sample assembly	2-5
2.2 Urai press	2-7
2.3 Photograph of Leitz Berek compensator	2-9
2.4 Relation between compensator reading and retardation	2-11
2.5 Berek compensator reading vs. c-axis plunge angle in OCP	2-12
2.6 Projection method	2-17
3.1 Maps of OCP sample TO-109	3-5
3.2 c-axis fabric diagrams of OCP sample TO-109	3-7
3.3 Marker particle trajectories of sample TO-109	3-8
3.4 Grid maps of sample TO-109	3-10
3.5 Average grain area vs. bulk shear strain of sample TO-109	3-11
3.6 Maps of TO-109 at a bulk shear strain of 1.2	3-13
3.7 Offset of material lines across grain boundary	3-14
3.8 Plots of $R_f$ vs. angle between $\sigma_1$ and c-axis	3-17
3.9 Plots of grain area vs. bulk shear strain sample TO-109	3-18
3.10 c-axis reorientation trajectories	3-20
3.11 Rotation of c-axis and S <sub>1</sub> vs. bulk shear strain	3-21
3.12 Lattice rotation (R <sub>L</sub> ) and S <sub>1</sub> rotation (R <sub>M</sub> )	3-23
3.13 Plots of S. vs. c-axis rotation of hard grains	2 24

3.14 Model of hard grain deformation	3-25
3.15 Test of model of hard grain deformation	3-27
3.16 Taylor/Sachs effect on dynamic grain growth	3-29
3.17 Plots of R <sub>f</sub> difference vs. orientation function difference	3-30
4.1 Maps of OCP sample TO-110	4-5
4.2 Photomicrographs of sample TO-110	4-7
4.3 Marker particle trajectories of sample TO-110	4-9
4.4 Grid maps of sample TO-110	4-10
4.5 c-axis fabric diagram of sample TO-110	4-11
4.6 c-axis reorientation trajectories of sample TO-110	4-12
4.7 Plots of average grain area vs. bulk shear strain	4-14
4.8 Projection diagrams and rose diagrams of grain boundaries	4-15
4.9 Foliation intensity and orientation plots	4-18
4.10 Trajectories of foliation and bulk finite strain	4-20
4.11 Maps of the central area of the sample	4-21
4.12 Foliation-weakening process by grain boundary migration	4-23
4.13 Foliation-weakening process by dissection	4-24
4.14 Foliation-strengthening process by rotational recrystallization	4-26
4.15 Means' (1981) loop-like plot	4-29
4.16 Plot of aspect ratio of grains against grain area	4-30
4.17 Life history of six grains	4-31
5.1 Maps of sample TO-91	5-5
5.2 Seven types of subgrain boundary development	5-8
5.3 Photomicrographs of Type I subgrain boundary	5-10
5.4 Maps of Type I subgrain boundaries	5-11
5.5 Photomicrographs of Type II subgrain boundary	5-13
5.6 Maps of Type II subgrain boundary	5-14
5.7 Photomicrographs of Type III subgrain boundary	5-15
5.8 Maps of Type III subgrain boundary in TO-91	5-17
5.9 Maps of Type III subgrain boundaries in TO-207	5-19
5.10 Maps of Type IV subgrain boundary	5-23
5.11 Photomicrographs of Type IV subgrain boundary	5-24
5.12 Photomicrographs of Type V subgrain boundary	5-25
5.13 Mans of Type V subgrain boundary	5-26

5.14 Maps of Type VI subgrain boundary in sample TO-11	5-28
5.15 Photomicrographs of Type VII subgrain boundary	5-29
5.16 Orientations of subgrain boundaries of each type	5-31
5.17 Evolution of subgrain boundary orientations	5-32
5.18 Plot of subgrain boundary density vs. bulk shear strain	5-34
5.19 Comparison of subgrain boundary orientations of samples	5-35
5.20 Comparison of subgrain boundary density of samples	5-37
6.1 Schematic diagrams of grain boundary sliding	6-4
6.2 Accommodation mechanisms of grain boundary sliding	6-5
6.3 Offset of material lines in sample TO-110	6-9
6.4 Evolution of grain boundaries in sample TO-110	6-10
6.5 Grain boundary sliding by rotation jump in TO-110	6-13
6.6 Offset of a marker line in sample TO-110	6-15
6.7 Photomicrographs of sample TO-105	6-18
6.8 c-axis fabric diagrams of sample TO-105	6-19
6.9 Grain boundary sliding by translation jump in TO-105	6-22
6.10 Grain boundary sliding by rotation jump in TO-105	6-25
6.11 Grain boundary sliding by translation jump in TO-207	6-28
6.12 Evolution of grain boundary openings	6-30
6.13 Photomicrographs of TO-88 and TO-89	6-31
6.14 Plot of bulk strain vs. grain boundary openings ratio	6-32
6.15 Residence time of grain boundary openings in TO-105	6-33
6.16 Opening and closing of grain boundaries in TO-202	6-35
6.17 Orientations of grain boundary openings	6-36
6.18 Grain boundary orientations	6-41
6.19 Three types of grain boundary migration	6-43
A1.1 Mohr circle for a deformation tensor <b>D</b>	A1-3
A1.2 Flow chart of program GRID	A1-6
A1.3 Flow chart of program MPT	A1-7
A1.4 Flow chart of program TRI	A1-8
A1.5 Flow chart of program GBO	A1-10
A1.6 An example of projection method	A1-11
A1.7 Examples of projection method	A1-12
A1 8 Flow chart of program GRD	A1-14

A2.1 Projection diagrams and rose diagrams of TO-109	A2-2
A2.2 Plots of foliation intensity vs. bulk shear strain of samples	A2-4
A2.3 Plots of foliation orientation vs. bulk shear strain of samples	A2-6
A2.4 Projection diagrams and rose diagrams of TO-105	A2-7
A2.5 Comparison of c-axis fabric diagram of samples	A2-10
A2.6 Comparison of marker particle trajectories of samples	A2-13
A2.7 Comparison of grain size of samples	A2-15
A2.8 Comparison of grain boundary migration of samples	A2-17
LIST OF TABLES	
2.1 Synkinematic microscopy research since 1980	2-2
2.2 Motors for Urai press	2-8
3.1 Conditions of experimental deformation	3-3
5.1 Conditions of experimental deformation	5-2
6.1 Conditions of experimental deformation	6-7
A1.1 Computer programs	A1-5