MICROSCOPY TECHNIQUES FOR MATERIALS SCIENCE

A R CLARKE AND C N EBERHARDT

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Materials science has witnessed incredible change and technological development over the past few decades and no more so than in the application of optical microscopy. So fast has been the pace of this change that the authors felt there was a real need for a new book that discusses the potential of the latest optical microscopes for materials science research. The main objective for computer-assisted microscopy is to make the most effective measurements of the 3D structure of materials and to make the measurement as quickly and efficiently as possible so as to interpret the resulting images without undue bias. The authors hope this book will do justice to the types of measurement that are now possible and point the way to uses of novel optical microscopy techniques in materials science.

The book is in three main parts. In Part I the authors review the basic principles behind the traditional view of optics (leading on to optical microscope design) and microelectronics (leading on to computer hardware, software and image analysis). In Part II, the practical issues involved with the acquisition and interpretation of images from standard optical reflection microscopes and also confocal laser scanning microscopes (CLSMs) are presented. Also, two major case studies are discussed which aim to show the reader how ideas from Part I are synthesised into real applications. In Part III, the emphasis is on alternative, non-optical microscopy techniques for the characterisation of material structures and two further case studies are presented on the use of X-ray microtomography and low frequency ultrasonics for fibre-reinforced polymer composites research.

Dr Ashley Clarke graduated from Imperial College, London in 1967 and stayed on to gain his doctorate in space physics in 1972. After a brief research assistantship at Imperial College he later joined the University of Leeds as a lecturer in physics in 1972. In the 1980s he designed the microprocessor-based Vela datalogger for school science laboratories. He was appointed Senior Lecturer in 1991. For the past thirty years, he has been actively involved with various research activities: ground-based astronomy, balloon-based and far-infrared astronomy and computer-assisted microscopy for materials science. He has taught physics, electronics and microelectronics and is on the Editorial Board of the Journal of Microscopy.

Dr Colin Eberhardt graduated from the University of Leeds in 1996 with a degree in Physics and Mathematics. He later undertook his PhD under the supervision of Dr Clarke. His PhD thesis involved work with ICI and DuPont, focusing on automated microscopy techniques for materials characterisation. Following the completion of his PhD in 1999, he continued to work within the group as a Research Fellow and his interests broadened to include studies of textiles using X-ray microtomography. Dr Eberhardt is currently a Product Specialist within the microscopy and imaging company, VisiTech International and is a Light Microscopy Section committee member in the Royal Microscopical Society.

Woodhead Publishing Ltd
Abington Hall
Abington
Cambridge CB1 6AH
England
www.woodhead-publishing.com
ISBN 1 85573 587 3

CRC Press LLC
2000 Corporate Blvd, NW
Boca Raton
FL 33431
USA
CRC order number WP1552
ISBN 0-8493-1552-2

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CRC Press
Boca Raton Boston New York Washington, DC

WOODHEAD PUBLISHING LIMITED
Cambridge England

Published by Woodhead Publishing Limited
Abington Hall, Abington
Cambridge CB1 6AH
England
www.woodhead-publishing.com

Published in North America by CRC Press LLC
2000 Corporate Blvd, NW
Boca Raton FL 33431
USA

First published 2002, Woodhead Publishing Limited and CRC Press LLC

British Library Cataloguing in Publication Data
A catalogue record for this book is available from the British Library.

Library of Congress Cataloging-in-Publication Data
A catalog record for this book is available from the Library of Congress

Woodhead Publishing ISBN 1 85573 587 3
CRC Press ISBN 0-8493-1552-2
CRC Press order number: WP1552

Cover design by The ColourStudio
Project managed by Macfarlane Production Services, Markyate, Hertfordshire (macfarl@aol.com)
Typeset by MHL Typesetting Limited, Coventry, Warwickshire
Printed by TJ International Limited, Padstow, Cornwall, England

Microscopy techniques for materials science

To our wives and children

Sue, Hannah, Gemma, Emily and Rosalie Clarke

and

Sue Eberhardt

Preface

At last the book is finished – and I have now been asked to put my mind to the Preface! It occurs to me that writing a Preface is a unique art form. Admittedly, after limited research into Preface-writing, I propose, like innumerable authors before me, to start with the usual whinge – yes, to paraphrase Mrs Beeton from the Preface of her famous cookbook, if we had known '... what courageous efforts were needed to be made', I am quite sure that we would never have started this enterprise. However, it is clear that one of the sensible reasons for co-authorship is that it has, at least, halved the agony for each of us.

In a sense, our book could be considered a kind of 'cook-book' – a cook-book for any reader who is interested in image processing and microscopy as a means to a materials science research end. Over the past two years, the original form of the book, as previously discussed with Patricia Morrison in 1999, has changed dramatically, but we hope, for the better. We have tried to minimise the mathematics that underpins these topics, and have concentrated on the practical issues (and pitfalls) one comes across when acquiring and analysing image data using various microscopic and tomographic measurement techniques. We have also made a real effort to expunge all spelling and grammatical *faux pas* and believe that, scientifically, the equations are correct and the conclusions presented (such as they are) are sound. In view of the speed of evolution in microscopic measurement techniques, this book can only hope to be a snapshot of the current situation and we suggest that readers follow the latest research articles in journals like the Royal Microscopical Society's *Journal of Microscopy* to keep abreast of future developments. Also, you might like to keep checking our Department of Physics and Astronomy website here at the University of Leeds to see our latest 3D reconstructions.

Recently, a colleague of mine in the Molecular Physics and Instrumentation Group said that he did not have a clue what I had been doing (research-wise) for the past ten years – well, this book is for him. In effect, the various case studies, in the latter two thirds of this book, catalogue my research team's attempts to try to understand how to make the best measurements of fibre orientations and their spatial distributions in both composites and textiles materials. They provide

testimony to just how far we have progressed from the initial 2D image analyser (which was attached to a standard optical reflection microscope), via the confocal laser scanning microscopes (Biorad MRC500 and Noran Olympus) and finally onto the X-ray microtomography and ultrasonic 'time of flight' systems (whose full potential we are still exploring).

One of the most difficult decisions we had to make was where to start the book. We eventually decided, without wishing to insult the intelligence of the reader, that a good place to start would be to review some background topics. Therefore, the first part of the book sets the scene for what later follows and is a rapid canter through historical developments in both optics and electronics, which have naturally led to the current computer-assisted, microscopy techniques that exist today.

We hope you like our sprinkling of apt quotations. Most books that deal with science, computers or computing are often incomprehensible and tedious. It is a pity that the surrealism of Lewis Carroll's works cannot be sustained throughout a 'scientific' book like this one. I have been looking for literary allusions to microscopy and, as might be expected, Carroll has something appropriate to say (aimed at children) in 'The Professor's Lecture' from one of his lesser well-known works, *Sylvie and Bruno Concluded*:

> ... he beckoned the Gardener to come up on the platform, and with his help began putting together what looked like an enormous dog-kennel, with short tubes projecting out of it on both sides.
>
> 'But we've seen elephants before,' the Emperor grumbled.
>
> 'Yes, but not through a ***Megaloscope***!' the Professor eagerly replied. 'You know you can't see a Flea, properly, without a ***magnifying-glass*** – what we call a ***Microscope***. Well, just in the same way, you can't see an Elephant properly without a ***minimifying-glass***. There's one in each of these little tubes. And this is a Megaloscope! ..."

Lewis Carroll then goes on to describe a hilarious scene with the megaloscope, and a second passage from the same Professor's Lecture also touches upon the central theme of our book.

> ... 'Our Second Experiment', the Professor announced, as Bruno returned to his place, still thoughtfully rubbing his elbows, 'is the production of that seldom-seen-but-greatly-to-be-admired phenomenon, ***Black Light***! You have seen White Light, Red Light, Green Light, and so on: but never, till this wonderful day, have any eyes but mine seen ***Black Light***! This box', he said, carefully lifting it upon the table, and covering it with a heap of blankets, 'is quite full of it ... would anyone like to get under the blankets and see it?'
>
> Dead silence followed this appeal: but at last Bruno said 'I'll get under ...'
>
> ... 'What did you see in the box?' Sylvie eagerly enquired.
>
> 'I saw nuffin!' Bruno sadly replied. 'It were too dark!'
>
> 'He has described the appearance of the thing exactly!' the Professor exclaimed with enthusiasm.

> ***Black Light***, and ***Nothing***, look so extremely alike, at first sight, that I don't wonder he failed to distinguish between them! We will now proceed to the Third Experiment. ...'

Perhaps, with good reason, he was also taking the rise out of the university lecturing profession here! Having quoted already from Lewis Carroll, it seems appropriate that I conclude this hastily conceived piece with some good advice that Carroll has to offer in his Preface to the *Sylvie and Bruno Concluded* story:

> ... Let me here express my sincere gratitude to the many Reviewers who have noticed, whether favourably or unfavourably, the previous Volume. Their unfavourable remarks were, most probably, well-deserved; the favourable ones less probably so. Both kinds have no doubt served to make the book known, and have helped the reading Public to form their opinions of it. Let me also here assure them that it is not from any want of respect for their criticisms, that I have carefully forborne from reading ***any*** of them. I am strongly of the opinion that an author had better ***not*** read any reviews of his books: the unfavourable ones are almost certain to make him cross, and the favourable ones conceited; and ***neither*** of these results is desirable ...

So, dear reader, if (for some inexplicable reason) you do not like our book or find any glaring errors, do not bother to tell us, just use the book as an expensive door-stop. If you do like the book, please do not bother to let us know, but for sure, tell all your friends and colleagues about it, because both Dr Eberhardt and I have expanding families to support and any extra royalties would be most welcome.

Ashley Clarke, September 2002

Acknowledgements

I have indeed been fortunate to be the supervisor of some very talented people, whose special software skills, physical insight and attitude towards their research work has resulted in our development of the measurement techniques described by the case studies in this book. I owe a great debt to my Ph.D students over the past 12 years: Dr Nic Davidson for the development of the 2D image analyser system; Dr Geoff Archenhold for his work on the Biorad MRC500 confocal system and the initial work into pattern matching; Dr Mike Enderby for automating the first version of the ultrasonic testrig and especially my co-author, Dr Colin Eberhardt for extending the confocal work with the Noran Odyssey and, latterly, the X-ray microtomography research. Also, I would like to thank my current Ph.D students: Andrew Schwarz, who has helped tremendously with the necessary corrections to some of the figures through his knowledge of the Adobe Illustrator software package (and his digital photography of some of the test equipment within the department) and Mat Harper for his input to the final case study on 3D elastic stiffness constants and the ultrasonic testrig.

For our recent foray into the X-ray microtomography research, my special thanks go to Nishanth Gopinathan and Dr Jia, the Institute for Particle Science and Engineering, School of Process, Environmental and Materials Engineering, for their assistance with, and access to, the University of Leeds, Skyscan 1072 X-ray system. Also, Professor Ryszard Pyrz at the Institute of Mechanical Engineering, Aalborg University for many years of fruitful collaboration, exchange of postgraduate students and access to their Skyscan machine.

With a small team like ours, the research contribution from short-term, undergraduate project students can be significant and we have been fortunate with visiting French project students over the years: Georges Bervin, Dan Gutknecht, Noe Poffa and Homig Lamon. There have also been numerous Leeds 3rd year project students who were persuaded to contribute to our confocal and ultrasonic work, amongst them were Sze Wei Ku, Andrew Johnston, James Watt and Kathryn Morris. We have also been assisted by French postgraduate students from Dr Michel Vincent's group at the École des Mines; Thomas Giroud and Sylvain Fluoret.

Special thanks go to our colleagues in the Polymer IRC and Polymer Group at the University of Leeds: Dr Alan Duckett, Dr Peter Hine and Professor Ian Ward who have vastly more experience than I with the measurement and modelling of fibre reinforced polymer composites. Their interaction with us over the years has sparked off many useful developments and I am indebted to Dr Duckett for steering me onto the problem of fibre orientations over 13 years ago during a memorable coffee break in 1989! I am also indebted to our colleagues in the Molecular Physics and Instrumentation Group within the Department of Physics and Astronomy: Professor David Batchelder, Dr Alastair Smith, Dr Simon Webster, Dr Kurt Baldwin and Ph.D student Kevin Critchley for their help on Raman microscopy which has made a valuable contribution in this book. My special thanks also go to Dr Mike Ries for the NMR photograph in Chapter 6.

I would like to now take the opportunity to acknowledge the skills of our Mechanical and Electronic Workshops (led by Mr Jack Coley and Mr Mansukh Patel respectively) within the Department of Physics and Astronomy, especially the skills of Trevor Haines, Stewart Weston, Andrew Price and Paul Ogden who have all contributed directly to our ultrasonic testrig design over the years.

On the practical side of microscopy, my special thanks go to Dr Vyvyan Howard (University of Liverpool) for introducing me to the mysteries and potential of stereology, Dr Torsten Mattfeldt for collaborating with us on a test for isotropy within fibre-reinforced composites and Dr Alan Entwistle (Ludwig Institute and the Royal Microscopical Society) for his many useful discussions and invaluable contribution to team funds through his annual microscope workshop demonstrations!

Many organisations have supplied us with samples over the years, some of which figure in this book, but my particular thanks go to: Cranfield University (Professor Phil Irving), IKP University of Stuttgart (Dr Gunther Fischer), École des Mines, Sophia-Antipolis (Dr Michel Vincent), University of Bristol (Professor Mike Wisnom), ICI Technology (Dr Bill Meredith and Dr Simon Allen) who have all provided us with a broad range of samples (ranging from bread, wood adhesive, foam and thin films), DuPont (Dr Paul Mills) and the Leeds School of Textile Industries (Dr Stephen Russell).

Like most small research teams, the financial assistance needed to keep together a team and its collective expertise has always been a struggle. My especial thanks go to Professor Paul Curtis (DERA Farnborough) for large and small research contracts granted to us over the years; also our unseen sponsors in Brussels, who were persuaded to fund our work under the Brite-Euram initiative; the UK Science Research Council for the numerous MoD/EPSRC joint grants that have been awarded to us over the past 10 years, and the assistance of liaison officer, Dr Matthew Hiley on the latest grants. Without the timely assistance of Dr Andrew Dickson (Biorad) and his free loan of the Biorad MRC500 confocal system for a number of years, we could never have performed our initial CLSM

studies on composites. Then, without Noran UK's half-price offer of an Olympus CLSM system and Dr Garry Burdett's (Health and Safety Executive, Sheffield) significant contribution towards the Olympus, we would never have been able to explore the automation of confocal systems. My co-author (CNE) is also grateful for being awarded ICI Technology's Case Quota Ph.D Studentship on thin films.

Our grateful thanks go to Dr Bill Meredith for providing the phase contrast, DIC and crossed polar images shown in Figures 1.62 and 1.63; Professor Tony Wilson (University of Oxford) for permission to show his tilted microcircuit in Figure 4.5; Professor Gwynne Morgan (Leeds) for his photograph of Sir William Bragg (Figure 5.1); Dr Kurt Baldwin and Dr Simon Webster for the Raman figures (Figures 5.6, 5.7 and 5.14) and LEO Electron Microscopy Ltd for the picture of the first commercial electron microscope in Figure 6.3. Other figures in the book have been recreated, from many referenced sources, by Colin Eberhardt using Adobe Illustrator software. Nearly all of the figures in most of the case studies have been generated from our own image data using the Leeds 2D system, the Noran Odyssey CLSM, the Skyscan 1072 system or the Leeds ultrasonics testrig.

Finally, our thanks to Gwen Jones, Patricia Morrison and Stuart Macfarlane for keeping us on schedule.

Ashley Clarke, September 2002

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