Microstructural characterisation of fibre-reinforced composites

Edited by John Summerscales

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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

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

First published 1998, Woodhead Publishing Ltd and CRC Press LLC
Reprinted 2001

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 240 8
CRC Press ISBN 0-8493-3882-4
CRC Press order number: WP3882

Printed by T.J.I. Digital, Cornwall, England

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Preface

That same day Pharaoh commanded the taskmasters of the people as well as their supervisors "You shall no longer give the people straw to make bricks, as before: let them go and gather straw for themselves. But you shall require of them the same quantity of bricks as they have made previously"

Genesis 5: 6–8

The use of natural composite materials by human kind is almost as old as the species itself, notably through animal and plant fibres adapted to perform each task. The last fifty years have seen a significant advance in the development of artificial fibre-reinforced composite materials for structural applications, notably with continuous glass or carbon fibres at high fibre volume fractions. The effective development of this class of materials involves a close understanding of the micromechanical features and the macroscopic performance. The use of microstructural characterisation techniques has been applied widely in extending this understanding. However, the literature has proliferated primarily in conference proceedings and journals across a wide range of subjects, throughout science, technology and engineering, with marginal coverage in a few books. The purpose of this book is to provide a contemporary review of the application of advanced microstructural characterisation techniques to fibre-reinforced composites. It is aimed at senior undergraduates, research degree candidates and engineers entering, or already practising, in the field of composite materials. It should also prove useful to microscopists who are given the task of examining these novel materials. The chapters are intended to provide a stand-alone introduction to each topic with comprehensive referencing of appropriate basic texts and of the original works for those readers wishing to pursue the subject further.

The introductory chapter presents an overview of the need for appropriate microscopical techniques, together with a brief consideration of textures and of those advanced techniques which are not covered elsewhere in the book and are current (image processing and three-dimensional visualisation) or are likely to become important in the near future (optical coherence tomography, microradiography, magnetic resonance). Chapter 2 considers the microscopy of flexible textile composites: this important area has much to offer those of us who have traditionally been associated with 'structural' rigid composites. Chapter 3 describes the novel 3D confocal laser scanning microscopes at the University of Leeds. To date, these have been primarily employed in the characterisation of short fibre composites: the systems have high potential for detailed characterisation of continuous unidirectional and fabric-reinforced composites. Chapter 4 describes the work of the University of Delaware in the geometric modelling of yarn and fibre assemblies, whilst Chapter 5 extends this by considering the yarn shape in woven fabric composites. Chapter 6 deals with the quantitative microstructural definition of fabric-reinforced composite materials and the potential to develop process–property–microstructure relationships. The final three chapters each consider the use of a specific microstructural characterisation technique: Chapter 7 deals with electron microscopy in the study of interfacial bonding, Chapter 8 deals with the measurement of strain or stress by Raman microscopy and last (but by no means least) Chapter 9 looks at the acoustic microscope in the context of ceramic-matrix and metal-matrix composites.

The authors selected for each chapter were most often the first choice of the editor, but a couple were either too busy or otherwise could not give their commitment to the project. The editor thus accepts any blame for imbalance in the coverage of the book. Extreme care has been taken in creating this volume, but inevitably there will be faults which escape proofreading. I should be most grateful to receive notice of any such problems which readers might detect, and especially to receive reprints of appropriate key papers which might point the way to any future volume.

John Summerscales

Acknowledgements

The editor would like to acknowledge the help of colleagues in the School of Manufacturing, Materials and Mechanical Engineering for their assistance in the preparation of this book, either through direct advice on points of detail or simply for being there to share the work of the School equitably. Further, I should like to express personal gratitude to Mr David Short (now Head of SMMME) for his encouragement and guidance throughout the twenty years which have elapsed since I adopted composites as a career.

Thanks are also due to Patricia Morrison at Woodhead Publishing Limited for supporting the idea of this book and for undertaking much of the routine administrative work in a cheerful and professional manner.

Last, but by no means least, I should like to thank each of the chapter authors for producing manuscripts in a timely manner which required minimal editing and who thereby made a pleasure of the task of putting the book together.

Where figures have not been originated by the chapter authors, we are grateful to the originators and publishers for permission to reproduce the figures included in the book. They are acknowledged in the individual chapters. The authors and publishers have attempted to trace the copyright holders of all figures reproduced in this publication and apologise to any copyright holders if permission to publish has not been obtained through error or omission.

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1
Introduction

JOHN SUMMERSCALES

1.1 Introduction

Fibre-reinforced composites have risen from very small beginnings around the time of the Second World War to an industry which is now delivering in excess of one million tons of material in each of Europe and the United States every year. The vast majority of these materials are polymer-matrix composites. The percentage of metal-matrix and ceramic-matrix composites is still in single percentage figures by volume and weight, but represents a larger proportion in value.

The intention of this book is to highlight some of the current state-of-the-art in the microstructural characterisation of fibre-reinforced materials. The increasing interest in mesostructures (intermediate between micro- and macrostructures) is reflected here.

Those readers who are not familiar with the basic principles are referred to the recent literature for microscopy [1–35] and for microprobe analysis [36–43]. Flewitt and Wild [28] offer a good balance of comprehensive coverage and realistic price, albeit that polymers and composites are given only superficial treatment. A biennial annotated bibliography of chemical microscopy is produced by Cooke [44].

1.2 Microscopy of polymers and composites

Three existing books provide a very strong background for the microscopy of polymers. They are recommended sources for that broader topic. Hemsley [45] has described the more advanced techniques of polymer microscopy using light (or optical) microscopy. He outlines the principles and practice of individual methods which may be applied to various forms of specimen. His book gives only fleeting coverage of composites; sectioning of composites (pages 33/34) and ultraviolet and fluorescence microscopy for curing and permeability of thermosetting resins (pages 266–269).

Sawyer and Grubb [46] provide a slightly more extended coverage; specimen preparation (first edition pages 82/84), etching of reinforcement fibres (pages 111/112), fibre, plastics and composites fracture (pages 131–136), application of microscopy to fibres and fabrics (pages 155–176) and to composites (pages 214–230), the surface coatings on fibres (page 238) and liquid crystalline polymer composites (pages 252–254).

Roulin-Moloney [47] describes the techniques that are currently employed in fractographic investigation, including case histories of specific polymeric and composite materials to which the techniques have been applied. The book opens with six chapters on the microscopical techniques including image analysis, and closes with six chapters on fractography and failure mechanisms. Of the latter six, three are specific to carbon fibre composites, to short-fibre reinforced semi-crystalline thermoplastics and to stress corrosion cracking in glass reinforced plastics.

Engel *et al.* [48] have published a book which shows different kinds of damage in polymers that can often only be interpreted by microscopic examination combined with experience. Some 40 examples are relevant to this book, notably:

- fracture of unsaturated polyester resin matrix composites (plates 196, 197, 346–348 and 420–421),
- fracture of fibre-reinforced nylon 6 (plates 296–298, 404 and 405),
- fracture of fibre-reinforced PTFE (plate 300),
- fracture of fibre-reinforced polypropylene (plates 301, 302, 338–340, 384 and 385),
- fracture of fibre-reinforced polycarbonate (plates 305–307, 356–358 and 429–430),
- fracture of cotton-reinforced phenolic resin (plates 349 and 350),
- weathering of glass-reinforced nylon (plates 205–207),
- frictional wear of carbon fibre-reinforced epoxy resin (plates 111–114) and
- a glass fibre-reinforced PTFE bearing cage (plates 117 and 118).

A companion book exists [49], but does not include micrographs of either fibre- or whisker-reinforced composites. The Royal Microscopical Society organises a biennial International Conference on Microscopy of Composite Materials [50], although the polymer composite content is often low.

1.2.1 Defects

Adams and Cawley [51,52] reviewed defect types and non-destructive testing techniques for composites and bonded joints.

Summerscales [53] recently reviewed the manufacturing defects which are likely to occur in fibre-reinforced plastics composites. Special emphasis was laid