

Springer Series in Materials Science

Volume 247

Series editors

Robert Hull, Troy, USA

Chennupati Jagadish, Canberra, Australia

Yoshiyuki Kawazoe, Sendai, Japan

Richard M. Osgood, New York, USA

Jürgen Parisi, Oldenburg, Germany

Tae-Yeon Seong, Seoul, Republic of Korea (South Korea)

Shin-ichi Uchida, Tokyo, Japan

Zhiming M. Wang, Chengdu, China

The Springer Series in Materials Science covers the complete spectrum of materials physics, including fundamental principles, physical properties, materials theory and design. Recognizing the increasing importance of materials science in future device technologies, the book titles in this series reflect the state-of-the-art in understanding and controlling the structure and properties of all important classes of materials.

More information about this series at <http://www.springer.com/series/856>

Wolfgang Grellmann · Beate Langer
Editors

Deformation and Fracture Behaviour of Polymer Materials



Springer

Editors

Wolfgang Grellmann
Centre of Engineering,
Martin-Luther-University Halle-Wittenberg
Halle, Saxony-Anhalt
Germany

Wolfgang Grellmann
Polymer Service GmbH Merseburg,
Associated An-Institute of University of
Applied Sciences Merseburg
Merseburg, Saxony-Anhalt
Germany

Beate Langer
Department of Engineering and Natural
Sciences
University of Applied Sciences Merseburg
Merseburg, Saxony-Anhalt
Germany

Beate Langer
Polymer Service GmbH Merseburg,
Associated An-Institute of University of
Applied Sciences Merseburg
Merseburg, Saxony-Anhalt
Germany

ISSN 0933-033X

Springer Series in Materials Science

ISBN 978-3-319-41877-3

DOI 10.1007/978-3-319-41879-7

ISSN 2196-2812 (electronic)

ISBN 978-3-319-41879-7 (eBook)

Library of Congress Control Number: 2017938288

© Springer International Publishing AG 2017

This work is subject to copyright. All rights are reserved by the Publisher, whether the whole or part of the material is concerned, specifically the rights of translation, reprinting, reuse of illustrations, recitation, broadcasting, reproduction on microfilms or in any other physical way, and transmission or information storage and retrieval, electronic adaptation, computer software, or by similar or dissimilar methodology now known or hereafter developed.

The use of general descriptive names, registered names, trademarks, service marks, etc. in this publication does not imply, even in the absence of a specific statement, that such names are exempt from the relevant protective laws and regulations and therefore free for general use.

The publisher, the authors and the editors are safe to assume that the advice and information in this book are believed to be true and accurate at the date of publication. Neither the publisher nor the authors or the editors give a warranty, express or implied, with respect to the material contained herein or for any errors or omissions that may have been made. The publisher remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Printed on acid-free paper

This Springer imprint is published by Springer Nature

The registered company is Springer International Publishing AG

The registered company address is: Gewerbestrasse 11, 6330 Cham, Switzerland

Preface

The growing demands on the reliability, safety and lifetime of machines, equipment and components made of polymers and composites make it necessary to develop meaningful test methods for the assessment of fracture properties. For this purpose, polymer-specific evaluation methods and concepts of the field of technical fracture mechanics and polymer diagnostics/polymer testing are used. Within polymer sciences, these areas of research have emerged as separate disciplines over recent years, evidenced by curricula of polymer engineering programmes at universities and universities of applied sciences.

The present status report on the current state of knowledge of technical fracture mechanics of polymers and composites with polymer matrix has been supplemented by revised presentations from the 14th discussion conference on “Deformation and Fracture Behaviour of Polymers” and by contributions describing our own research. By including additional contributions dealing with the investigation into the toughness of polymers with time-dependent fracture mechanical characteristics and the use of crack resistance concepts for polymers and elastomers, we aim to provide a comprehensive overview of the current state of knowledge.

The discussion conference on “Deformation and Fracture Behaviour of Polymers” has been taking place every two years in Merseburg for more than 30 years and has become a recognised scientific conference.

The 2014 conference was held jointly with the international scientific congress “PolyMerTec 2014”, which was organised by the Merseburg University of Applied Sciences and focused on engineering topics for the first time.

The conferences aim at showcasing the progress in fundamental research and applied research in this scientific discipline. This is accomplished by means of plenary talks, short contributions and lively discussions among the large number of expert colleagues.

Essential topics are as follows:

- Polymer testing, damage analysis and polymer diagnostics of components
- Toughness characterisation of polymers with fracture mechanics concept

- Hybrid methods for polymer testing and diagnostics
- Non-destructive polymer testing (ultrasound)
- Long-term static behaviour and ageing

The conference programme also includes exhibitions of equipment used for non-destructive and destructive material testing, polymer analytics and elastomer and film testing.

This book follows the status reports that have already been published by Springer:

- Deformation und Bruchverhalten von Kunststoffen
Hrsg: W. Grellmann und S. Seidler
1998, ISBN 3-540-63671-4
- Deformation and Fracture Behaviour of Polymers
Eds.: W. Grellmann and S. Seidler
2001, ISBN 3-540-41247-6

A comprehensive compilation of mechanical and fracture mechanical properties from the literature and own research is documented in the following encyclopaedia:

- Mechanical and Thermomechanical Properties of Polymers
Group VIII Advanced Materials and Technologies Volume VIII/6A3
Eds.: W. Grellmann and S. Seidler
2014, ISBN 978-3-642-55165-9

In addition to the aforementioned books on the deformation and fracture behaviour of polymers, the Merseburg School edited the textbooks *Polymer Testing* for students at universities and universities of applied sciences. These textbooks were published by Hanser in German (2005, 2011, 2015) and English (2007, 2013). A Russian translation appeared in 2010.

These textbooks on polymer testing and diagnostics and on technical fracture mechanics of polymers and composites with polymer matrix also form the basis of an online encyclopaedia on “polymer testing and diagnostics”. This online encyclopaedia follows the wiki system known from Wikipedia and is available for free at <http://wiki.polymerservice-merseburg.de> in Version 4.0 (2014).

With the edition of this status report, we hope to contribute to an enhanced understanding of specific problems of the discipline among colleagues from different research institutes and the polymer industry.

The editors would like to express their special thanks to Dr.-Ing. Ralf Lach, Polymer Service GmbH Merseburg, Associate Institute of the Merseburg University of Applied Sciences, for his comprehensive support and critical advice.

Halle and Merseburg, Germany
Merseburg, Germany
September 2016

Wolfgang Grellmann
Beate Langer

Contents

Part I Modern Aspects of Fracture Mechanics in the Industrial Application of Polymers

1 Time-Dependent Fracture Behaviour of Polymers at Impact and Quasi-Static Loading Conditions	3
R. Lach and W. Grellmann	
1.1 Comparison of Methods for the Determination of R-Curves for Polymers at Impact Loading	4
1.2 Material Samples	5
1.2.1 Semicrystalline Polymers with Particle–Matrix Structure: PP/EPR/PE Copolymers	5
1.2.2 Short-Glass Fibre (GF)-Reinforced Semicrystalline Polymers: PP/GF	6
1.2.3 Nanophase-Separated Amorphous Polymers: Binary Blends of PS–PB Block Copolymers	7
1.2.4 CTOD Rate Under Quasi-Static Test Conditions: PP/EPR Blends	10
1.2.5 Influence of the Temperature: Amorphous Polycarbonate (PC)	11
1.3 Crack Propagation Kinetics of Polymers at Impact Loading Conditions	12
1.4 Discussion of Literature on Crack Propagation Kinetics	15
1.5 Stable Crack Propagation as Kinetic Phenomenon—An Outlook	19
References	20
2 Fatigue Crack Growth Behaviour of Epoxy Nanocomposites— Influence of Particle Geometry	23
M.H. Kothmann, G. Bakis, R. Zeiler, M. Ziadeh, J. Breu and V. Altstädt	

2.1	Introduction	23
2.2	Experimental	25
2.2.1	Materials	25
2.2.2	Preparation of Nanocomposites	26
2.2.3	Characterisation Methods	26
2.3	Results and Discussion	27
2.3.1	Organophilisation of Nanoparticles	27
2.3.2	Morphology	27
2.3.3	Fatigue Crack Propagation Behaviour	28
2.4	Conclusion	30
	References	31
3	Fracture Mechanics Methods to Assess the Lifetime of Thermoplastic Pipes	33
	F. Arbeiter, G. Pinter, R.W. Lang and A. Frank	
3.1	Failure Behaviour of Polymer Pipes	34
3.2	Fracture Mechanics Approach for Pipe Lifetime Calculations	36
3.3	Crack Growth in Polyethylene	39
3.4	Extrapolation to Static Crack Growth Behaviour from Fatigue Tests	41
3.5	Lifetime Calculation of PE Pipe Grades	44
3.6	Lifetime Calculation of a PE Pipe Grade at 80 °C Using Cyclic CRB Tests	45
3.7	Conclusion and Outlook	48
	References	49
4	Thermographic Characterisation of the Deformation and Fracture Behaviour of Polymers with High Time and Spatial Resolution	55
	M. Stein and K. Schneider	
4.1	Introduction	55
4.2	Experimental	56
4.2.1	Materials	56
4.2.2	Methods	57
4.3	Results	59
4.3.1	Thermomechanical Characterisation of PET	59
4.3.2	Thermomechanical Characterisation of PC	65
4.4	Discussion	67
4.4.1	Polycarbonate—Affine Deformation with Uniform Energy Dissipation	68
4.4.2	Poly(Ethylene Terephthalate)—Localised Deformation and Complex Influence of Two Phases	68

4.5	Conclusion	71
	References	71
5	Mechanical and Fracture Mechanical Properties of Polymorphous Polypropylene	73
	A. Monami, B. Langer, J. Sadilek, J. Kučera and W. Grellmann	
5.1	Introduction	73
5.2	Experimental Part	75
5.3	Results and Discussion	77
5.3.1	Degree of Crystallinity	77
5.3.2	Influence of Cooling Rate on the Resistance Against Stable Crack Initiation and Crack Growth	78
5.4	Conclusion	80
	References	80
6	Numerical Modelling of Damage Initiation and Failure of Long Fibre-Reinforced Thermoplastics	83
	L. Schulenberg, D.-Z. Sun and T. Seelig	
6.1	Introduction	83
6.2	Problem Formulation	84
6.2.1	Experimental Observation	84
6.2.2	Microscopic Observation	84
6.2.3	Numerical Microstructural Model	86
6.3	Numerical Results	87
6.3.1	Single-Fibre Unit Cell Under Uniaxial Tension	87
6.3.2	Unit Cells Containing Three Fibres	88
6.3.3	Variations of the Fibre Overlapping Length and Load Direction	90
6.4	Discussion	91
6.5	Summary	91
	References	92
Part II Advanced Structure-Sensitive Methods for Analysing Cracks and Fracture Surfaces		
7	Characterisation of Polymers in the Scanning Electron Microscope—From Low-Voltage Surface Imaging to the 3D Reconstruction of Specimens	95
	A. Zankel, M. Nachtnebel, C. Mayrhofer, K. Wewerka and T. Müllner	
7.1	Introduction	95
7.2	Low-Voltage Mode of the SEM	96
7.3	Low-Vacuum Mode of the ESEM	97
7.4	The ESEM Mode	100

7.5	Artefacts and Beam Damage	101
7.6	3D Information Using In Situ Ultramicrotomy	103
7.7	Conclusions	106
	References	107
8	3D Reconstruction of Cracks in Polymers—New Insight into the Fracture Behaviour?	109
	M. Nachtnebel, A. Zankel, C. Mayrhofer, M. Gahleitner and P. Pölt	
8.1	Introduction	109
8.2	Preparation and Image Processing	111
8.3	Results and Discussion	112
8.4	Conclusion	117
	References	118
9	Determination of the Stable Crack Growth by Means of the Fluorescence Adsorption-Contrast Method (3D-FAC Method)	121
	M. Kroll, B. Langer and W. Grellmann	
9.1	Introduction	122
9.2	Experimental	123
9.2.1	Materials	123
9.2.2	<i>J-R-Curve</i> Determination	124
9.3	Development of a Fluorescence Microscopy Procedure for Δa Measurement	125
9.3.1	Fluorescent Penetration Dye Colouring	125
9.3.2	Optimisation of the Fluorescent Application Process	126
9.3.3	Fluorescence Adsorption-Contrast Method (3D-FAC Method)	129
9.4	Results and Discussion	133
9.5	Conclusions	135
	References	136
10	Acoustic Emission Analysis for Assessment of Damage Kinetics of Short-Glass Fibre-Reinforced Thermoplastics—ESEM Investigations and Instrumented Charpy Impact Test	139
	M. Schoßig, A. Zankel, C. Bierögel, P. Pölt and W. Grellmann	
10.1	Introduction	140
10.2	Theoretical Background	141
10.2.1	Acoustic Emission (AE) Analysis	141
10.2.2	Frequency Analysis—Wavelet Transform (WT)	144
10.3	Experimental Details	145
10.3.1	In Situ Tensile Tests in ESEM Coupled with AE Measurement	145
10.3.2	Coupling ICIT and AE Analysis	147

10.4	Results	149
10.4.1	ESEM Investigations—Coupling the In Situ Tensile Test with AE Analysis	149
10.4.2	AE Measurements During ICIT	157
10.5	Summary	161
	References	162
11	The Fractography as a Tool in Failure Analysis—Possibilities and Limits	165
	I. Kotter and W. Grellmann	
11.1	Introduction	165
11.2	Fractography—Fracture Surface Structures	166
11.2.1	Waves and Grid Lines	166
11.2.2	Fracture Parabola Respectively U- or V-Shaped Ramps	167
11.2.3	Ramps, Bars or Steps	169
11.2.4	Example: Fracture of a Multi-Layer Pipe	169
11.3	Limits of Validity of the Fractography for Filled and Reinforced Plastics	171
11.4	Summary	173
	References	174
Part III Fracture Mechanics and Related Methods for Analysing the Fracture Safety and Lifetime of Plastic Pipe Materials		
12	Slow Crack Growth of Polyethylene—Accelerated and Alternative Test Methods	177
	B. Gerets, M. Wenzel, K. Engelsing and M. Bastian	
12.1	Introduction	177
12.2	Slow Crack Growth	178
12.3	Test Methods to Determine Slow Crack Growth Behaviour of PE	178
12.3.1	Conventional Test Methods	178
12.3.2	Accelerated Test Methods: (Accelerated) Full Notch Creep Test (FNCT and aFNCT)	179
12.3.3	Alternative Test Methods: Strain Hardening Test (SHT)	182
12.3.4	Alternative Test Methods: Cracked Round Bar Test—CRB Test	185
12.4	Conclusions	186
	References	187
13	Polypropylene for Pressure Pipes—From Polymer Design to Long-Term Performance	189
	L. Boragno, H. Braun, A.M. Hartl and R.W. Lang	

13.1	Introduction PP Market Overview	189
13.2	Morphology and Polymorphism of PP	191
13.3	Short-Term Properties—Charpy and Pipe Falling Weight	192
13.4	From Microstructure to Final Properties	194
13.5	Influence of Processing	196
13.6	Long-Term Behaviour—Pressure Resistance and Slow Crack Growth in PP Materials	197
13.7	Conclusions	199
	References	200
14	Lifetime of Polyethylene (PE) Pipe Material—Prediction Using Strain Hardening Test	203
	E. Nezbedová, J. Hodan, J. Kotek, Z. Krulis, P. Hutař and R. Lach	
14.1	Introduction	203
14.2	Conventional Assessment of Long-Term Performance and Lifetime: The Pennsylvania Edge Notch Tensile Test and the Tensile Full Notch Creep Test	205
14.3	Accelerated Assessment of Long-Term Performance and Lifetime: The Strain Hardening Test	206
14.4	Results	207
14.5	Conclusions	209
	References	210
15	Influence of Welding and Composition on the Short-Term Stable Crack Propagation Through Polyolefin Single- and Bilayered Structures	211
	R. Lach, T. Krolopp, P. Hutař, E. Nezbedová and W. Grellmann	
15.1	Introduction	212
15.2	Experimental	212
	15.2.1 Materials and Specimen Preparation	212
	15.2.2 Equipment and Data Analysis	214
15.3	Results and Discussion	215
	15.3.1 Influence of Specimens Shape, Orientation, Welding and Loading Speed on Stable Crack Initiation and Propagation Behaviour in Single-Layer Pipes Made from PE 100, PE 80 and PP Materials	215
	15.3.2 Influence of Interlayers and Crack Propagation Direction on Stable Crack Initiation and Propagation Behaviour in Bilayer Pipes Made from PP Materials	220
15.4	Summary	224
	References	226

16 Influence of Different Welding Conditions of Polyolefin Pipes on Creep Crack Growth	229
J. Mikula, P. Hutař, M. Ševčík, E. Nezbedová, R. Lach, W. Grellmann and L. Náhlík	
16.1 Introduction	230
16.2 Welding of Polyolefin Pipes	230
16.3 Material Properties of the Welded Region	231
16.4 Numerical Model Description	233
16.5 Location of Crack Initiation	234
16.6 Stress Intensity Factors for Different Configurations	235
16.6.1 Influence of Material Inhomogeneity	235
16.6.2 Influence of the Weld Bead Radius	236
16.6.3 Influence of Different Weld Bead Shape	236
16.7 Crack Trajectories for Different Welds	238
16.8 Lifetime Prediction	239
16.9 Conclusion	239
References	240
17 Epoxy Modifications—A Novel Sealing Material for Rehabilitation of Pipe Joints	243
C. Schoberleitner, T. Koch and V.-M. Archodoulaki	
17.1 Introduction	244
17.2 Experimental Section	245
17.3 Results and Discussion	247
17.4 Conclusion	251
References	252
Part IV Deformation Behaviour and Fracture Mechanics Characteristics of Polymer Films and Adhesive Systems	
18 Approaches to Characterise the Mechanical Properties of Films and Elastomers	257
K. Reincke and W. Grellmann	
18.1 Introduction	257
18.2 Experimental Opportunities of Mechanical Films and Elastomers Testing	258
18.2.1 Conventional Tensile and Notched Impact Test After ISO 8256	258
18.2.2 Instrumented Notched Tensile Impact Test	259
18.2.3 Instrumented Puncture Impact Test	260
18.2.4 Tear Test	261
18.2.5 Peel Tests	262
18.3 Examples of Use	264
18.3.1 Assessment of the Toughness Properties of Elastomers	264

18.3.2	Influence of Chemical Loading on the Mechanical Properties of a Thermoplastic Film	265
18.3.3	Influence of Chemical Loading on the Toughness Properties of Elastomers	266
18.3.4	Evaluation of a PE/PB-1 Peel System	267
18.4	Conclusions	269
	References	269
19	Fracture Mechanics Characterisation of Peelfilms	271
	M. Nase, M. Rennert, S. Henning, A. Zankel, K. Naumenko and W. Grellmann	
19.1	Introduction	272
19.2	Experimental	274
19.3	Results and Discussion	276
	References	280
20	Fracture Mechanics Characterisation of Low-Adhesive Stretch Films	283
	M. Rennert, M. Nase, K. Reincke, R. Lach and W. Grellmann	
20.1	Introduction	284
20.2	Experimental	286
	20.2.1 Material and Composition of the Films	286
	20.2.2 Cling Test According to ASTM D 5458	287
20.3	Results and Discussion	291
20.4	Conclusion	295
	References	295
21	Thermal Stability and Lifetime Prediction of an Epoxide Adhesive System	297
	R. Tiefenthaler, R. Fluch, B. Strauß and S. Hild	
21.1	Introduction	297
21.2	Materials and Methods	299
	21.2.1 Material and Samples	299
	21.2.2 Spectroscopic Techniques and Mechanical Analyses	299
	21.2.3 T-Peel Test and Tensile Lap-Shear Test	300
21.3	Results and Discussion	300
	21.3.1 Thermomechanical Analysis	300
	21.3.2 ATR-IR Spectroscopy	301
	21.3.3 Raman Spectroscopy and Thermogravimetric Analysis	304
	21.3.4 Lifetime Prediction: T-Peel Test and Tensile Lap-Shear Test	304
21.4	Conclusions	309
	References	309

Part V Fatigue Crack Propagation, Lifetime and Long-Term Mechanical Behaviour of Thermoplastics and Elastomers

22 Morphology and Fatigue Behaviour of Short-Glass Fibre-Reinforced Polypropylene	315
M. Palmstingl, D. Salaberger and T. Koch	
22.1 Introduction	315
22.2 Analysis of Morphology of SFRP	316
22.3 Determination of Fatigue Behaviour	323
References	331
23 Characterisation of the Deformation and Fracture Behaviour of Elastomers Under Biaxial Deformation	335
K. Schneider, R. Calabrò, R. Lombardi, C. Kipscholl, T. Horst, A. Schulze, S. Dedova and G. Heinrich	
23.1 Introduction	335
23.2 Concept of the Biaxial Test Stand	336
23.3 Upgrading of a Biaxial Testing Method	338
23.3.1 New Clamping System for High Biaxial Deformation	339
23.3.2 Specimen Geometry	340
23.3.3 Crack Propagation with the New Specimen	341
23.4 Material	341
23.5 Results	342
23.5.1 Material Behaviour Under Biaxial Load	342
23.5.2 Strain Amplification at the Crack Tip of a SENT Sample	343
23.5.3 Crack Propagation Under Biaxial Load	344
23.5.4 Crack Propagation and Estimation of the Tearing Energy	345
23.6 Conclusion	348
References	348
24 Influence of Thermal Ageing Process on the Crack Propagation of Rubber Used for Tire Application	351
R. Stoček, O. Kratina, P. Ghosh, J. Maláč and R. Mukhopadhyay	
24.1 Introduction	351
24.2 Theoretical Background	354
24.2.1 Dynamic-Mechanical Analysis (DMA)	354
24.2.2 Fracture Crack Growth (FCG)	355
24.3 Experimental Details	356
24.3.1 Material Preparation	356
24.3.2 Ageing	356
24.3.3 DMA	357
24.3.4 FCG	357

24.4	Results and Discussion	358
24.4.1	DMA	358
24.4.2	FCG	360
24.5	Conclusion	362
	References	363
25	Development of Magnetorheological Elastomers (MREs) for Strength and Fatigue Resistance	365
	J. McIntyre and S. Jerrams	
25.1	Introduction	366
25.2	Preparation of Materials	368
25.3	Experimental Methodology	369
25.4	Results and Discussion	371
25.5	Summary and Conclusions	373
	References	374
26	Fibre-Reinforced Polyamides and the Influence of Water Absorption on the Mechanical and Thermomechanical Behaviour	377
	P. Guttmann and G. Pilz	
26.1	Introduction and Objectives	378
26.2	Experimental	378
26.2.1	Materials	378
26.2.2	Experimental Procedure	379
26.3	Results and Discussion	380
26.3.1	Water Absorption	380
26.3.2	Dynamic-Mechanical Analysis (DMA)	381
26.3.3	Monotonous Tensile Tests	382
26.3.4	Media Creep Tests	385
26.4	Summary and Outlook	387
	References	388
27	Accelerated Measurement of the Long-Term Creep Behaviour of Plastics	389
	F. Achereiner, K. Engelsing and M. Bastian	
27.1	Introduction	389
27.2	Principle of the Stepped Isothermal Method	391
27.3	Creep Testing Using SIM	392
27.4	Construction of a Master Curve	395
27.5	Assessment of the Method	397
27.6	Applications of SIM	398
27.7	Conclusions	400
	References	401

Part VI Influence of Ageing on Mechanical and Fracture Mechanics Performance of Thermoplastics and Elastomers	
28 Hygrothermal Ageing of Injection-Moulded PA6/GF Materials Considering Automotive Requirements	405
T. Illing, M. Schoßig, C. Bierögel, B. Langer and W. Grellmann	
28.1 Introduction	405
28.2 Material and Experiments	407
28.3 Results and Discussion	408
28.4 Summary and Conclusion	416
References	417
29 Ageing of Polymer Materials—Testing, Modelling and Simulation Considering Diffusion	421
H. Baaser	
29.1 Introduction	421
29.2 Test Method	423
29.2.1 Change in Stiffness Over a Long Period of Time	423
29.2.2 Diffusion	423
29.3 Mechanical Model and Numerical Application	425
29.4 Computational Results	426
29.4.1 O-Ring Application	426
29.4.2 Compression Test Specimen—Surface–Volume Ratio	426
29.5 Conclusions and Discussion	428
References	429
30 Investigations of Elastomeric Seals—Low-Temperature Performance and Ageing Behaviour	431
M. Jaunich, A. Kömmling and D. Wolff	
30.1 Introduction	431
30.2 Behaviour at Low Temperatures	432
30.3 Methodology for the Ageing of Elastomeric Seals	435
30.4 Conclusion	442
References	442
Part VII Mechanical Properties and Fracture of Elastomers—Influence of Composition, Reinforcement and Crosslinking	
31 Mechanical Reinforcement in a Polyisoprene Rubber by Hybrid Nanofillers	447
S. Agnelli, V. Cipolletti, S. Musto, M. Coombs, L. Conzatti, S. Pandini, M.S. Galimberti and T. Riccò	
31.1 Introduction	447
31.2 Experimental	449

31.3	Results and Discussion	451
31.3.1	Transmission Electron Microscopy Analyses	451
31.3.2	Mechanical Behaviour	452
31.4	Conclusions	458
	References	458
32	Structure–Property Correlations of SSBR/BR Blends	461
	K. Reincke, W. Grellmann, S. Ilisch, S. Thiele and U. Ferner	
32.1	Introduction	462
32.2	Experimental	462
32.3	Results	464
32.3.1	Influence of the Composition on the Processing-Related Properties	464
32.3.2	Influence of Composition of the Rubber Mixture on the Physical Properties	467
32.4	Structure–Property Correlation	470
32.5	Conclusions	472
	References	473
33	Comparison Between Peroxide and Radiation Crosslinking of Nitrile Rubber	475
	K. Bandzierz, D.M. Bielinski, G. Przybytniak, M. Jaszczałk and A. Marzec	
33.1	Introduction	475
33.2	Experimental	477
33.2.1	Materials and Samples Preparation	477
33.2.2	Radiation Crosslinking	477
33.2.3	Peroxide Thermal Crosslinking	478
33.2.4	Crosslink Density Determination	478
33.2.5	Chain Scission and Crosslinking Ratio Determination	479
33.2.6	Mechanical Properties Test	480
33.3	Results and Discussion	480
33.4	Conclusion	482
	References	482
34	Wood Flour as a Filler of Natural and Epoxidised Natural Rubber	485
	A. Smejda-Krzewicka, W.M. Rzymski and P. Dmowska-Jasek	
34.1	Introduction	485
34.2	Materials and Methods	486
34.2.1	Materials	486
34.2.2	Sample Preparation	486
34.2.3	Testing Methods	487

Contents	xix
34.3 Results and Discussion	487
34.3.1 Effect of Wood Flour Derived from Coniferous Trees (CF) on Properties of NR and ENR	487
34.3.2 Effect of Wood Flour Derived from Deciduous Coniferous Trees (DF) on Properties of NR and ENR	489
34.4 Conclusion	491
References	491
35 Characterisation of the Ultimate Tensile Properties of Elastomers by a Dimensionless Hooke Number—A New Approach to Failure Envelopes	493
N. Rennar and P. Kirchner	
35.1 Introduction	493
35.2 Theoretical Background	494
35.3 Experimental Part	497
35.3.1 Selection of Polymers and Recipes of Test Compounds	497
35.3.2 Mixing Procedure, Crosslinking and Testing	497
35.4 Results and Discussion	498
35.5 Summary and Conclusions	505
References	506
36 Thermomechanical Analysis Strategies for Elastomer Components Under Dynamic Loading	507
R. Behnke and M. Kaliske	
36.1 Introduction and Overview	507
36.2 Simultaneous Solution Scheme	509
36.3 Sequential Solution Scheme	511
36.4 Conclusion and Outlook	515
References	516
37 Influence of Selected Silica Fillers on the Properties of Vulcanised Rubber Blends	517
W.M. Rzymski, A. Smejda-Krzewicka, J. Rogoża and A. Ochenduszko	
37.1 Introduction	517
37.2 Materials and Methods	518
37.3 Results and Discussion	519
37.4 Conclusion	524
References	524

List of Authors

F. Achereiner SKZ—German Plastics Center, Würzburg, Germany

S. Agnelli Department of Mechanical and Industrial Engineering, University of Brescia, Brescia, Italy

V. Altstädt Department of Polymer Engineering, University of Bayreuth, Bayreuth, Germany

F. Arbeiter Department Polymer Engineering and Science, Montan University Leoben, Leoben, Austria

V.-M. Archodoulaki Institute of Materials Science and Technology, Vienna University of Technology, Vienna, Austria

H. Baaser Freudenberg Technology Innovation, FTI, Weinheim, Germany; Professor for Engineering Mechanics & FEM, University of Applied Sciences, Bingen, Germany

G. Bakis Department of Polymer Engineering, University of Bayreuth, Bayreuth, Germany

K. Bandzierz Faculty of Chemistry, Institute of Polymer & Dye Technology, Łódź University of Technology, Łódź, Poland

M. Bastian SKZ—German Plastics Center, Würzburg, Germany

R. Behnke Faculty of Civil Engineering, Institute for Statics and Dynamics of Structures, Dresden University of Technology, Dresden, Germany

D.M. Bielinski Faculty of Chemistry, Institute of Polymer & Dye Technology, Łódź University of Technology, Łódź, Poland; Division of Elastomers & Rubber Technology, Institute for Engineering of Polymer Materials & Dyes, Piastow, Poland

C. Bierögel Centre of Engineering, Martin-Luther-University Halle-Wittenberg, Halle/Saale, Germany; Polymer Service GmbH Merseburg, Associated An-Institute of University of Applied Sciences Merseburg, Merseburg, Germany

L. Boragno Borealis Polyolefine GmbH, Linz, Austria

H. Braun Borealis Polyolefine GmbH, Linz, Austria

J. Breu Department of Inorganic Chemistry I, University of Bayreuth, Bayreuth, Germany

R. Calabro Department of Chemistry, Materials and Chemical Engineering, Polytechnic University of Milan, Milan, Italy

V. Cipolletti Department of Chemistry Materials and Chemical Engineering, Polytechnic University of Milan, Milan, Italy

L. Conzatti Institute for Macromolecular Studies, National Research Council, Genova, Italy

M. Coombs Pirelli Tyre, Milan, Italy

S. Dedova Leibniz Institute for Polymer Research Dresden e.V., Dresden, Germany; Dresden University of Technology, Dresden, Germany

P. Dmowska-Jasek Faculty of Chemistry, Institute of Polymer & Dye Technology, Łódź University of Technology, Łódź, Poland

K. Engelsing SKZ—German Plastics Center, Würzburg, Germany

U. Ferner Trovotech GmbH, Bitterfeld-Wolfen, Germany

R. Fluch voestalpine Stahl GmbH, Linz, Austria

A. Frank Polymer Competence Center Leoben GmbH, Leoben, Austria

M. Gahleitner Borealis GmbH, Linz, Austria

M.S. Galimberti Department of Chemistry Materials and Chemical Engineering, Polytechnic University of Milan, Milan, Italy

B. Gerets SKZ—German Plastics Center, Würzburg, Germany

P. Ghosh Hari Shankar Singhania Elastomer & Tyre Research Institute, Kankroli, Rajasthan, India

W. Grellmann Centre of Engineering, Martin-Luther-University Halle-Wittenberg, Halle/Saale, Germany; Polymer Service GmbH Merseburg, Associated An-Institute of University of Applied Sciences Merseburg, Merseburg, Germany

P. Guttmann Department Polymer Engineering and Science, Montan University Leoben, Leoben, Austria

A.M. Hartl Institute of Polymeric Materials and Testing, Johannes Kepler University Linz, Linz, Austria

G. Heinrich Leibniz Institute for Polymer Research Dresden e.V., Dresden, Germany; Faculty of Mechanical Science and Engineering, Dresden University of Technology, Dresden, Germany

S. Henning Fraunhofer Institute for Microstructure of Materials and Systems (IMWS), Halle/Saale, Germany

S. Hild Institute of Polymer Sciences, Johannes Kepler University Linz, Linz, Austria

J. Hodan Institute of Macromolecular Chemistry, Academy of Sciences of the Czech Republic, Prague, Czech Republic

T. Horst Faculty of Automobile and Mechanical Engineering, University of Applied Sciences Zwickau, Zwickau, Germany

P. Hutař Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno, Czech Republic

T. Illing Valeo Schalter und Sensoren GmbH, Bietigheim-Bissingen, Germany

S. Ilisch Trinseo Deutschland GmbH, Schkopau, Germany

M. Jaszczałk Faculty of Chemistry, Institute of Polymer & Dye Technology, Łódź University of Technology, Łódź, Poland

M. Jaunich BAM—Federal Institute for Material Research and Testing, Berlin, Germany

S. Jerrams Centre for Elastomer Research, Dublin Institute of Technology, Dublin, Ireland

M. Kaliske Faculty of Civil Engineering, Institute for Statics and Dynamics of Structures, Dresden University of Technology, Dresden, Germany

C. Kipscholl Coesfeld GmbH & Co. KG, Dortmund, Germany

P. Kirchner Plastics and Elastomer Technology, University of Applied Sciences Würzburg-Schweinfurt, Würzburg, Germany

T. Koch Institute of Materials Science and Technology, Vienna University of Technology, Vienna, Austria

A. Kömmling BAM—Federal Institute for Material Research and Testing, Berlin, Germany

J. Kotek Institute of Macromolecular Chemistry, Academy of Sciences of the Czech Republic, Prague, Czech Republic

M.H. Kothmann Department of Polymer Engineering, University of Bayreuth, Bayreuth, Germany

I. Kotter Polymer Service GmbH Merseburg, Associated An-Institute of University of Applied Sciences Merseburg, Merseburg, Germany

O. Kratina Centre of Polymer Systems, Tomas Bata University in Zlín, Zlín, Czech Republic; Department of Polymer Engineering, Faculty of Technology, Tomas Bata University in Zlín, Zlín, Czech Republic

M. Kroll BASF Leuna GmbH, Leuna, Germany; Institute of Macromolecular Chemistry, Academy of Sciences of the Czech Republic, Prague, Czech Republic

T. Krolopp Polymer Service GmbH Merseburg, Associated An-Institute of University of Applied Sciences Merseburg, Merseburg, Germany

Z. Kruliš Institute of Macromolecular Chemistry, Academy of Sciences of the Czech Republic, Prague, Czech Republic

J. Kučera Polymer Institute Brno, Brno, Czech Republic

R. Lach Polymer Service GmbH Merseburg, Associated An-Institute of University of Applied Sciences Merseburg, Merseburg, Germany

R.W. Lang Institute of Polymeric Materials and Testing, Johannes Kepler University Linz, Linz, Austria

B. Langer Department of Engineering and Natural Sciences, University of Applied Sciences Merseburg, Merseburg, Germany; Polymer Service GmbH Merseburg, Associated An-Institute of University of Applied Sciences Merseburg, Merseburg, Germany

R. Lombardi Leibniz Institute for Polymer Research Dresden e.V., Dresden, Germany; University of Naples Federico II, Naples, Italy; Bridgestone Technical Center Europe, Rome, Italy

J. Maláč Centre of Polymer Systems, Tomas Bata University in Zlín, Zlín, Czech Republic; Department of Polymer Engineering, Faculty of Technology, Tomas Bata University in Zlín, Zlín, Czech Republic

A. Marzec Faculty of Chemistry, Institute of Polymer & Dye Technology, Łódź University of Technology, Łódź, Poland

C. Mayrhofer Austrian Centre for Electron Microscopy and Nanoanalysis, Graz, Austria; Institute for Electron Microscopy and Nanoanalysis, Graz University of Technology, Graz, Austria

J. McIntyre DKI—The German Institute of Rubber Technology, Hanover, Germany

J. Mikula Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno, Czech Republic; Brno University of Technology, Brno, Czech Republic

- A. Monami** Polymer Service GmbH Merseburg, Associated An-Institute of University of Applied Sciences Merseburg, Merseburg, Germany
- R. Mukhopadhyay** Hari Shankar Singhania Elastomer & Tyre Research Institute, Kankroli, Rajasthan, India
- T. Müllner** Department of Chemistry, Philipps University, Marburg, Germany
- S. Musto** Department of Chemistry Materials and Chemical Engineering, Polytechnic University of Milan, Milan, Italy
- M. Nachtnebel** Institute for Electron Microscopy and Nanoanalysis, Graz University of Technology, Graz, Austria
- L. Náhlík** Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno, Czech Republic
- M. Nase** University of Applied Sciences Hof, Hof, Germany
- K. Naumenko** Institute of Mechanics, Otto Von Guericke University Magdeburg, Magdeburg, Germany
- E. Nezbedová** Polymer Institute Brno, Brno, Czech Republic
- A. Ochenduszko** Synthos S.A., Oświęcim, Poland
- M. Palmstingl** Institute of Materials Science and Technology, Vienna University of Technology, Vienna, Austria
- S. Pandini** Department of Mechanical and Industrial Engineering, University of Brescia, Brescia, Italy
- G. Pilz** Department Polymer Engineering and Science, Montan University Leoben, Leoben, Austria
- G. Pinter** Department Polymer Engineering and Science, Montan University Leoben, Leoben, Austria; Polymer Competence Center Leoben GmbH, Leoben, Austria
- P. Pölt** Institute for Electron Microscopy and Nanoanalysis, Graz University of Technology, Graz, Austria
- G. Przybytniak** Institute of Nuclear Chemistry and Technology, Warsaw, Poland
- K. Reincke** Centre of Engineering, Martin-Luther-University Halle-Wittenberg, Halle/Saale, Germany; Polymer Service GmbH Merseburg, Merseburg, Germany
- N. Rennar** Plastics and Elastomer Technology, University of Applied Sciences Würzburg-Schweinfurt, Würzburg, Germany
- M. Rennert** Polifilm Extrusion GmbH, Südliches Anhalt, Germany; University of Applied Sciences Hof, Hof, Germany

T. Riccò Department of Mechanical and Industrial Engineering, University of Brescia, Brescia, Italy

J. Rogoża Synthos S.A., Oświęcim, Poland

W.M. Rzymski Institute of Polymer & Dye Technology, Łódź University of Technology, Łódź, Poland

J. Sadilek Polymer Institute Brno, Brno, Czech Republic

D. Salaberger University of Applied Sciences of Upper Austria, Wels, Austria

K. Schneider Leibniz Institute for Polymer Research Dresden e.V., Dresden, Germany

C. Schoberleitner Institute of Materials Science and Technology, Vienna University of Technology, Vienna, Austria

M. Schoßig Polymer Service GmbH Merseburg, Associated An-Institute of University of Applied Sciences Merseburg, Merseburg, Germany

L. Schulenberg Fraunhofer Institute for Mechanics of Materials (IWM), Freiburg, Germany

A. Schulze Leibniz Institute for Polymer Research Dresden e.V., Dresden, Germany; Chemnitz University of Technology, Chemnitz, Germany

T. Seelig Institute of Mechanics, Karlsruhe Institute of Technology (KIT), Karlsruhe, Germany

M. Ševčík Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno, Czech Republic

A. Smejda-Krzewicka Institute of Polymer & Dye Technology, Łódź University of Technology, Łódź, Poland

M. Stein Institute of Processing Machines and Mobile Machinery, Dresden University of Technology, Dresden, Germany

R. Stöck PRL Polymer Research Lab s.r.o., Zlín, Czech Republic; Centre of Polymer Systems, Tomas Bata University in Zlín, Zlín, Czech Republic

B. Strauß voestalpine Stahl GmbH, Linz, Austria

D.-Z. Sun Fraunhofer Institute for Mechanics of Materials (IWM), Freiburg, Germany

R. Tiefenthaler Institute of Polymer Science, Johannes Kepler University Linz, Linz, Austria; voestalpine Stahl GmbH, Linz, Austria

S. Thiele Trinseo Deutschland GmbH, Schkopau, Germany

M. Wenzel SKZ—German Plastics Center, Würzburg, Germany

K. Wewerka Institute for Electron Microscopy and Nanoanalysis, Graz University of Technology, Graz, (Austria)

D. Wolff BAM—Federal Institute for Material Research and Testing, Berlin, Germany

A. Zankel Institute for Electron Microscopy and Nanoanalysis, Graz University of Technology, Graz, Austria; Austrian Centre for Electron Microscopy and Nanoanalysis, Graz, Austria

R. Zeiler Department of Polymer Engineering, University of Bayreuth, Bayreuth, Germany

M. Ziadeh Department of Inorganic Chemistry I, University of Bayreuth, Bayreuth, Germany

Abbreviations

2NCT	Double Notch Creep Test
3D-FAC	Fluorescence adsorption contrast method
ABS	Acrylonitrile–butadiene–styrene
ACN	Acrylonitrile
ACT	Accelerated Creep Test
AE	Acoustic emission
AFM	Atomic force microscopy
aFNCT	Accelerated Full Notch Creep Test
ALE	Arbitrary Lagrangian Eulerian
ATBN	Amine-terminated butadiene–acrylonitrile copolymer
BET	Brunauer–Emmett–Teller
BIIR	Brominated poly(isobutylene-co-isoprene)
BOPET	Biaxial oriented poly(ethylene terephthalate)
BR	Butadiene rubber
BSE	Backscattered electrons
C(T)OD	Crack-(tip)-opening displacement
CA	Coupling agent
CB	Carbon black
CCG	Creep crack growth
CD	Particle distance
CEG	Cation exchange capacity
CF	Coniferous trees
CIP	Carbonyl iron powder
CNT	Carbon nanotubes
CRB specimen	Cracked round bar specimen
CRB test	Crack Round Bar Test
CS	Compression set
CSEM	Conventional scanning electron microscopy
CT specimen	Compact tension specimen
CT	Computed tomography

CTOA	Crack-tip-opening angle
DCP	Dicumyl peroxide
DENT specimen	Double-edge-notched tension specimen
DGEBA	Diglycidyl ether of bisphenol-A
DI	Macro dispersion index
DIC	Digital image correlation
DIE	Digital image elaboration
DMA	Dynamic-mechanical-analysis
DMTA	Dynamic-mechanical-thermal analysis
DSC	Differential scanning calorimetry
EB	Electron beam
EDS	Energy-dispersive X-ray Spectrometry
EDZ	State of plane strain
EFTEM	Energy-filtered transmission electron microscopy
ENR	Epoxidised natural rubber
EPDM	Ethylene-propylene-diene rubber
EPFM	Elastic-plastic fracture mechanics
EPR	Ethylene-propylene rubber
ESEM	Environmental scanning electron microscopy
ETD	Everhart-Thornley detector
EVA	Ethylene(vinyl acetate)
FBA	Formerly bonded area
FCG	Fatigue crack growth
FCP	Fatigue crack propagation
FE	Finite element
FEA	Finite element analysis
FEG	Field-emission gun
FEM	Finite element method
FKM	Fluorocarbon rubber
FNCT	Full Notch Creep Test
FSBR	Styrene–butadiene–butylacrylate mix polymer
FTIR	Fourier transform infrared spectroscopy
FT-model	Folgar-Tucker model
FW	Impact falling weight
GF	Short glass fibre-reinforced
GPC	Gel permeation chromatography
HNBR	Hydrogenated nitrile butadiene rubber
ICIT	Instrumented Charpy impact test
IR	cis-1,4-polyisoprene; synthetic rubber
IRHD	International rubber hardness degree
LEFM	Linear elastic fracture mechanics
LFT	Long fibre-reinforced thermoplastics
LVSEM	Low-voltage scanning electron microscopy
MAH	Maleic anhydride
micro-CT	Microcomputer tomography

MR	Magnetorheological
MRE	Magnetorheological elastomer
MTS criterion	Maximum tangential stress criterion
nanoG	Nano-graphite
NBR	Nitrile–butadiene rubber
NCTL Test	Notched Constant Tensile Load Test
NIS	Notched impact strength
NPT	Notch Pipe Test
NR	cis-1,4-polyisoprene; natural rubber
PA	Polyamide
PA6	Polyamide 6
PA66	Polyamide 66
PB-1	Polybutene-1
PC	Polycarbonate
PDMS	Polydimethylsiloxane
PE	Polyethylene
PE-HD	High-density polyethylene
PE-LD	Low-density polyethylene
PE-LLD	Linear low-density polyethylene
PE-MD	Medium-density polyethylene
PENT Test	Pennsylvania Edge Notch Tensile Test
PES	Polyethersulfone
PET	Poly(ethylene terephthalate)
PLT	Point Load Test
PMMA	Poly(methyl methacrylate)
PNR	Polynorbornene
PP	Polypropylene
PPA	Polyphthalamide
PS specimen	Pure-shear specimen
PVC	Poly(vinyl chloride)
PXRD	Powder X-ray Diffraction
R-curve	Crack resistance curve
RH	Relative humidity
RSC model	Modified model of Wang and Jin
SBEM	Serial block face scanning electron microscopy
SBR	Styrene–butadiene rubber
SCG	Slow crack growth
SE	Secondary electrons
SEM	Scanning electron microscopy
SENB specimen	Single-edge-notched bending specimen
SENT specimen	Single-edge-notched tension specimen
SF	Single fibre
SFRP	Short fibre-reinforced polymers
SHT	Strain Hardening Test
SIM	Stepped Isothermal Method

SMART	Small accelerated reliable test
SZH	Stretch zone height
SZW	Stretch zone width
TEM	Transmission electron microscopy
TGA	Thermogravimetric analysis
THF	Tetrahydrofuran
TMT	Thermomechanical treatment
TOR	Polyoctenamer
TPE	Thermoplastic elastomer
TPU	Thermoplastic polyurethane
TTSP	Time–temperature superposition principle
UD	Unidirectional
VPSEM	Variable pressure scanning electron microscopy
WF	Wood flour
WLF	Williams-Landel-Ferry
WOL	Wedged open loading
WT	Wavelet transform
X-CT	X-ray computed tomography