

Internationale wissenschaftliche
Tagung Polymerwerkstoffe

polymertec²¹ digital

gemeinsam mit der Tagung 17. Problemseminar
“Deformation und Bruchverhalten von Kunststoffe”



Abstractband

Vorträge und Poster

9. – 11. Juni 2021

Impressum

Internationale wissenschaftliche Tagung Polymertec 2021^{digital}
gemeinsam mit der Tagung 17. Problemseminar
"Deformation und Bruchverhalten von Kunststoffen"

9.–11. Juni 2021

Veranstalter:
Kunststoff-Kompetenzzentrum Halle-Merseburg
Institut für Polymerwerkstoffe e. V.
Stiftung Akademie Mitteldeutsche Kunststoffinnovationen
Hochschule Merseburg

Abstractband: Vorträge und Posterbeiträge

Herausgeber:
Prof. Dr.-Ing. Beate Langer
Prof. Dr.-Ing. habil. Peter Michel
Dr.-Ing. Ralf Lach

Redaktion, Satz und Layout: Dr.-Ing. Ralf Lach

Das Werk, einschließlich seiner Teile, ist urheberrechtlich geschützt. Jede Verwertung ist ohne Zustimmung des Verlages und der Herausgeber unzulässig. Dies gilt insbesondere für die elektronische oder sonstige Vervielfältigung, Übersetzung, Verbreitung und öffentliche Zugänglichmachung.

Die innerhalb dieses Buchs genannten oder gezeigten Schutzmarken oder Handelsnamen sind Eigentum der jeweiligen Inhaber. Die Nennung von Markennamen oder Warenzeichen erfolgt unter ausdrücklicher Anerkennung aller Rechte der jeweiligen Rechteinhaber, auch wenn diese nicht unmittelbar gekennzeichnet sein sollten.

Für die sachliche Richtigkeit und die Form der Beiträge in Verwendung von Text und Bild sind die Autoren verantwortlich.

Nach Redaktionsschluss eingegangene Beiträge konnten nicht berücksichtigt werden.

Bibliografische Information der Deutschen Nationalbibliothek:

Die Deutsche Nationalbibliothek verzeichnet diese Publikation in der Deutschen Nationalbibliografie; detaillierte bibliografische Daten sind im Internet über <http://dnb.d-nb.de> abrufbar.

Selbstverlag der Hochschule Merseburg
© Merseburg, Juni 2021

ISBN 978-3-00-069372-4



Prof. Dr. Julia Beate Langer
Professur
Kunststofftechnik/Polymerwerkstoffe
Hochschule Merseburg



Prof. Dr. Peter Michel
Professur Kunststoffverarbeitung
Hochschule Merseburg
Fraunhofer IMWS
Leiter Geschäftsfeld Polymeranwendungen

Vorwort

*Herzlich willkommen zur internationalen wissenschaftlichen Konferenz **Polymertec 2021^{digital}**, die gemeinsam mit der Tagung 17. Problemseminar „Deformation und Bruchverhalten von Kunststoffen“ erstmals als online-Veranstaltung stattfindet.*

Ungeachtet der derzeit schwierigen Situation stellt die Erzeugung von Kunststoffen den wichtigsten Zweig der chemischen Industrie in Sachsen-Anhalt dar. Dementsprechend ist, auch unter dem Gesichtspunkt zukünftiger Erfordernisse von Nachhaltigkeit und Umweltschutz, das wissenschaftliche Interesse an Polymerwerkstoffen ungebrochen und es werden wieder zahlreiche Teilnehmer und sechs Firmenaussteller mit 72 Vorträgen, 23 Postern und einem Firmenkolloquium zum Gelingen der Veranstaltung beitragen. Dafür bedanken wir uns bei allen Beteiligten ganz herzlich.

Unser Dank gilt auch allen Organisatoren und Mitstreitern für das Engagement und den Ideenreichtum bei der Vorbereitung der Tagung. Das Team des Fraunhofer-Instituts für Mikrostruktur von Werkstoffen und Systemen IMWS in Halle hat bei der inhaltlichen Konzeption und operativen Umsetzung entscheidend mitgewirkt und somit, wie unsere anderen Partner, dazu beigetragen, dass wir ein hochkarätiges und attraktives Programm gestalten konnten.

Ohne die aktive Unterstützung durch unsere Förderer wäre eine solche Tagung nicht möglich. Herzlichen Dank an alle, die uns sowohl finanziell als auch moralisch unterstützt haben.

Wir freuen uns auf drei Tage mit wissenschaftlichen Vorträgen, Postern und Firmenpräsentationen aus allen Bereichen der Wertschöpfungskette der Polymerwerkstoffe.

Dieser Beitrag enthält aller Kurzfassungen der Vorträge und Poster entsprechend der wissenschaftlichen Sektionen

- Bruchmechanische Kennwertermittlung
- Struktur und Morphologie
- Kunststoffrecycling
- Charakterisierung von Polymerwerkstoffen
- Polymere Faserverbunde und Nanokomposite
- Zerstörungsfreie Werkstoffprüfung
- Health and Medical Care
- Kautschuk & Elastomere
- Rapid Prototyping

Für alle Autoren besteht zusätzlich die Möglichkeit, im Nachgang zur Tagung die Vortrags- und Posterinhalte in der Zeitschrift Macromolecular Symposia zu veröffentlichen.

Die **Polymertec 2021^{digital}** verfolgt, wie bereits zu den vorangegangenen Veranstaltungen, den Zweck, das breite Spektrum der erkenntnisorientierten Forschung, der angewandten Grundlagenforschung sowie der technologieorientierten Forschung enger zu verknüpfen und als Vermittler zwischen Hochschule und Industrie zu wirken. Parallel zum eigentlichen Tagungsprogramm finden eine Firmenpräsentation und eine Präsentation der Poster, jeweils in digitaler Form, statt.

Im Namen der Veranstalter, dem Kunststoff-Kompetenzzentrum Halle-Merseburg, dem Institut für Polymerwerkstoffe e.V., der Stiftung „Akademie Mitteldeutsche Kunststoffinnovationen“ und der Hochschule Merseburg wünschen wir allen viel Erfolg und Freude an der Tagung

Herzliche Grüße

Ihre Tagungsleitung

Beate Langer und Peter Michel

Inhaltsverzeichnis

Hauptvorträge

F. Dinkel	13
Ist Recycling eine notwendige oder hinreichende Bedingung für eine nachhaltige Verpackung?	
P. Skoczinski, M. Carus, D. de Guzman, H. Käß, R. Chinthapalli, J. Ravenstijn, W. Baltus, A. Raschka	14
Überblick über den Biokunststoffmarkt	
A. Zankel	15
In situ and correlative electron microscopy for the characterization of polymeric materials	
U. Laukant, T. Riedel, C. Woelk	16
Highlights der Schadensanalyse an Kunststoffen	
S. Podzimek	17
Molar mass as the fundamental property of synthetic and natural polymers: Current status of its determination and some historical reminiscence	
L. Kari	18
Modelling of tough, doubly crosslinked, single network hydrogel components in a mechanical context	
J. Stampfl	19
Improving thermomechanical properties of photopolymers for additive manufacturing	

Bruchmechanische Kennwertermittlung

P. Schrader, A. Gosch, M. Berer, S. Marzi	21
Determining the fracture energy of polymeric bulk specimens loaded in modes I and III	
A. Gosch, M. Berer, F. Arbeiter, T. Vojtek, P. Hutař, G. Pinter	22
Mixed mode I/III fatigue testing of unreinforced thermoplastic polymers	
E. Pérez	23
Particle debonding contribution to the fracture toughness of polymer based composites	
W. Li, B. Wang, G. Kalinka	24
Temperature-dependent energy dissipations during epoxy resin fracture	
C. Rosales, A. Costantino, T. Echeverria, C. Bernal, V. Pettarin	25
On the use of fracture mechanics techniques to assess the performance of recycled polymer blends	
E. Nezbedova, M. Kovar, R. Lach, W. Grellmann	26
Informational value of short-term tests to predict the lifetime of less and high oriented PE-HD compared to long-term tests such as FNCT and PENT	
B. Gerets, K. Engelsing	27
Fast testing of the stress crack resistance of polyamide 12	

E. Nezbedova, M. Kovar, R. Lach, W. Grellmann	28
Determination of fracture toughness under plane stress impact conditions of polyethylene (PE)	
M. Chytil, J. Poduška, J. Kučera, P. Hutař	30
Strain hardening test of PE-HD materials in relation to some accelerated lifetime prediction methods	

Struktur und Morphologie (biobasierte Kunststoffe)

M. Du, K. Jariyavidyanont, R. Androsch	32
Effect of molar mass on critical specific work of flow for shear-induced crystal nucleation in poly (L-lactic acid)	
S. McGowan, A. Siebert-Raths	33
Effect of hydrolysis in respect of processability and performance characteristics of polylactic acid (PLA)	
S. Richter, N. Teuscher, M. Heinze, R. Hoffmann, T. Pscherer	34
Atmospheric pressure plasmas for adhesion improvement of natural substances on bio-based polymer films	
R. Pandit, S. Rijal, K. Chapain, R. Adhikari	35
Fabrication and physicochemical characterization of starch based bioplastic films from banana peels	
K. Aslan	36
Influence of chemical and physical treatment on the structure and processability of Kraft lignin for extrusion applications	
D. Zehm, A. Lieske	37
FDCA-based polymer materials beyond PEF	
N. Eversmann, M. Wießner, A. Krombholz	38
Naturbasierte Schäume als funktionalisierte adaptive Elemente	

Kunststoffrecycling

S. Pokhrel, L. S. Rai, A. Sigdel, M. Slouf, J. Sirc, R. Lach, R. Adhikari	40
Fabrication and characterization of starch based bio-degradable polymer: Thermal and biodegradation properties	
J. Giri, R. Lach, S. Henning, J.-M. Saiter, V. Katiyar, R. Adhikari	41
Reinforcement of degradable copolyester with wheat stalk microcrystalline cellulose	
S. Zepnik	42
ALFATERXL® ECO – New ECO TPV from recycled and biobased raw materials	
M. Seitz, V. Cepus, M. Klätte, D. Thamm	43
Chemisches Recycling von Kunststoffabfällen – Chance für eine bessere Kreislaufwirtschaft	
M. Seier, S. Stanic, T. Koch, V. M. Archodoulaki	44
Herausforderungen im mechanischen Recycling von Einweg-Kunststoffen und Möglichkeiten neuer Konzepte	

Charakterisierung von Polymerwerkstoffen

T. Schrank, M. Berer, E. Helfer, M. Feuchter, G. Pinter	46
Analysis of injection molded components of polyoxymethylene by X-ray scattering and diffraction	
J. E. K. Schawe, E. Hempel	47
The influence of fillers and nucleating agents on polypropylene crystallization at high supercooling measured by Fast DSC	
A. Monami	48
Prüfung und Überwachung von Bauprodukten für Gründächer	
A. Flores, M. Gómez-Fatou, H. J. Salavagione	49
Nanoindentation in soft polymer-based composites using a cylindrical punch	
J. Kucera, J. Sadilek, J. Poduska, P. Hutar	50
Real plastic deformation in load separation method used for PE-HD	
J. Oberer, K. Schneider	51
Thermo-mechanical and structural analysis of neck initiation and failure in tensile tests of polyethylene terephthalate	

Polymere Faserverbunde und Nanokomposite

K. Roetsch, T. Horst	53
Mikrostrukturbasierte Festigkeitsbestimmung von endlosfaserverstärkten Duromeren	
M. John, S. Celevicz, I. Jahn, R. Schlimper	54
Challenges in evaluating carbon fiber reinforced thermoplastic tapes	
S. Celevics, S. Richter, I. Jahn, F. Nagel, J. Celevics, S. Pohle	56
Improvement of natural fiber impregnation for biobased sustainable unidirectional tapes	
T. Munkelt, J. Palomino, T. Hedtke, J. Martins de Souza e Silva, C. Schmelzer	57
Vom Samen zur Nanofaser – Gewinnung und Verarbeitung pflanzlicher Proteine zu Nanofaservliesen	
N. L. Bhandari, B. Dhungana, S. Henning, R. Lach, R. Adhikari	58
Structural, thermal and mechanical properties of urea–formaldehyde (UF)-natural fiber composites	
M. E. Frerking, A. Rouyer, M. Butto, J. Bovi, M. Melaj, C. Bernal	59
On the performance of biodegradable self-reinforced composites (SRCs) based on poly(lactic acid) (PLA)	
U. Staudinger, L. Jakisch, B. K. Satapathy	61
Development of block copolymer/carbon nanotube composites: Effects of nanofiller modification on structure, electrical and mechanical properties	
K. N. Dhakal, R. Lach, W. Grellmann, A. Das, H. H. Le, S. Wießner, B. Krause, J. Pionteck, R. Adhikari	62
Strain sensing behavior of piezoresistive flexible copolyester/carbon based filler composites	
J. Giri, S. Pokhrel, S. Henning, R. Lach, W. Grellmann, V. Katiyar, R. Adhikari	63
Compostable polymer composites based on copolyesters and natural fibers	

Zerstörungsfreie Werkstoffprüfung

T. Wagner, Y. Charfi, S. Celevics, R. Schlimper
Inline-Prüfung von UD-Tapes mittels aktiver Thermographie

65

Anton Paar Symposium

A. Braun, J. R. Agudo, G. Arnold
Approaches to overcome rheometric challenges when performing TTS measurements over a large temperature and frequency range

67

J. R. Agudo, M. Trujillo, G. Arnold, L. Ionov, M. Schäffler
Special applications in dynamic mechanical analysis: Combining rotational and axial drives for the characterization of novel materials

68

J. Fischer, M. Pfeiler-Deutschmann
Full Atomic Force Microscopy investigation of a polymer thin film

69

Struktur und Morphologie (allgemein)

T. Abboud, A. Wutzler, T. Groth, H.-J. Radusch
Evaluation of polymer blends for hot melt pressure sensitive adhesives containing tackifying resins and thermoplastic elastomers

71

J. Jůza, I. Fortelný
Compatibilization of polymer melts using block copolymers

72

J. Zidek, F. Kucera, J. Petrus, T. Ondracka, E. Kulovana, M. Novacek, J. Kois, J. John
Co-processing of polypropylene and poly(vinyl chloride) as a model of the compatibilization of separated plastic waste

73

J. Wiener, F. Arbeiter, A. Tiwari, O. Kolednik, G. Pinter
Crack arrester effect in mineral-reinforced polypropylene using soft interlayers

74

R. Landgraf, E. Oelsch, J. Ihlemann
Mechanical behavior of additively manufactured thermoplastic polyurethane: Experiments, constitutive modeling and Finite-Element-Analyses

75

M. Jabłońska, M. Rühl, M. Menzel, U. Hirsch, A. Heilmann
Biofouling studies of zwitterionic coatings of feed spacer for biofouling mitigation

76

K. Krause, T. Neumeyer, V. Altstädt
Hart-Weich-Verbunde mittels Co-Injektionsverfahren: Einfluss der Viskosität auf die Bauteilmorphologie

77

J. Roitner, V. M. Archodoulaki
Betrachtung des Degradationsverhaltens von Polyethylenen im mechanischen Recycling

78

Health and Medical Care

T. Hedtke, J. Martins de Souza e Silva, T. Groth, C. E. H. Schmelzer Elastin as basis for cutting-edge wound dressings	80
K. P. Malla, R. J. Yadav, S. Sakurai, H. Yamane, S. Henning, R. Adhikari Electrospun blends of polycaprolactone/poly-L-lactic acid/gelatin and natural hydroxyapatite for bone tissue engineering	82
G. Hillrichs, R. Lach, G. H. Michler, M. Götze, S. Henning, C. Costa Laser processing of polymeric nanofiber nonwovens for biomedical applications with ultrashort laser pulses	83
A. Narayanan Ramakrishnan, O. Röhrle, C. Ludtka, J. Köhler, A. Kiesow, S. Schwan The role of denture adhesives on the oral health of partial denture wearers: A numerical study	84
F. Du, H. E. Yener, R. Androsch Development of a biopolymer/drug system for fighting mosquito-borne tropical diseases	85
J. Köhler, A. Kiesow, A. N. Ramakrishnan, J. Hey, S. Schwan Der Einfluss von physiologischen Bedingungen auf das mechanische Verhalten von Polymer-Haftcreme-Kompositen	86

Kautschuk und Elastomere

N. Rull, F. Rueda, P. Frontini Crack propagation in isoprene rubber: Experiments and simulation	88
B. Demmel, S. Eibl, T. Förster, M. Johlitz, A. Lion Diffusion characteristics of fuel like substances in aged nitrile rubber investigated by Gas Chromatography/Mass Spectrometry	89
S. Sökmen Methodological characterisation of rubber–filler interactions in silica-filled SBR/BR systems	90
K. Baar RPA-Messungen an treibenden Kautschuk-Mischungen	91
Ü. Tastan, K. Nonnenmacher Ozone testing for rubbers and cable insulations	92
M. Jaunich, A. Kömmling, M. Zaghdoudi, M. Weber, D. Wolff Update on investigations of elastomer O-ring seals at BAM 3.4	93
A. Shahdehi, M. Alimardani, M. Razzaghi-Kashani, H. Roshanaei New insights on dry and wet friction of tire tread rubbers having safe aromatic oils	94

Rapid Prototyping

R. Gopi, R. Schlimper, R. Schäuble, D. Glatz, C. Stüpp Characterization of fiber reinforced 3D printed parts	96
S. Schwan, A. Kumar Majari, A. Krombholz Calculation of the J-integral for 3D printing samples with respect to creep and the production related anisotropy	97
K. Panchasara, K. Mehle, A. Ramakrishnan, C. Ludtka, S. Schwan Modellierung fertigungsbedingter Eigenspannung in 3D-Druck-Bauteilen aus Polyamid	98

Poster

P. Hirsch, T. Theumer Comparative study on polyamide 11 and polyamide 10.10 as matrix polymers for biogenic wood–plastic composites	101
P. Bulacios, C. Bernal, E. Pérez Mechanical performance of bioresorbable maxillofacial implants	102
K. D. T. Kien, N. V. U. Nhi, H. N. Minh, H. X. Tung, D. Q. Minh Effects of the reaction time at 150 °C to the phenolic form from cashew nut shell waste	103
N. Eversmann, A. Krombholz Insekten-Chitin und Chitosan in biofunktionalen Oberflächen	104
S. Wüstenhagen, N. Eversmann, A. Krombholz Optimierter Flammenschutz an einem Naturfaserverbund	105
J. Klehm, T. Theumer, A. Krombholz, K. Thiele, S. Bauer Multifunktionales Beschichtungssystem auf der Basis pflanzenölbasierter kationischer Tenside	106
M. Rühl, J. Klehm, A. Krombholz, S. Gomoll, C. Splith, F. Meier Entwicklung und Bewertung innovativer, robuster und industriell herstellbarer Antifingerprint-beschichtungen aus hybriden Sol-Gel-Nanolacken	107
M. Bräuer, K. Schneider, H. Scheibner Mechanical testing of metal–TPU hybrids	108
P. Hirsch, T. Theumer Effects of UV aging, water storage and recycling on mechanical properties of biogenic wood–plastic composites from polyamide 11	109
C. Mayrhofer, H. Plank, H. Gnägi, I. Letofsky-Papst Ultramicrotomy of polymers at its best: Ultra sonic	110
P. Gautam, K. Shrestha, J. Giri, R. Adhikari Morphology and degradation behavior of ternary blends comprising PLA, PBAT and natural fibers	111
M. Busch, T. Theumer, I. Jahn, A. Krombholz Investigation of the mechanical behavior of partially electron beam cross-linked polyamide	112

S. M. Nagaraja, M. Beiner	113
Different contributions to dissipation in NR and NR–BR blend composites filled with carbon black particles	
B. Tillner, S. Lehmann, I. Jahn, J. Celevics, M. Zschehyge	114
Continuous reinforced polyamide composite materials with unidirectional fiber orientation	
T. Wagner, M. Neul, N. Teuscher, M. Wießner, A. Krombholz, R. Schlimper	115
Entwicklung eines automatisierten Druck-Schmelz-Analysenverfahrens (DSV) für Kunststoffe und Kunststoffgemische zur Ableitung qualitativer und quantitativer Aussagen hinsichtlich der Stoffbeschaffenheit und Verarbeitungsfähigkeit	
T. Theumer	116
Multifunktionales Beschichtungssystem auf der Basis pflanzenölbasierter kationischer Tenside und Lignine	
P. Beckert, A. Ortwein, S. Wüstenhagen	117
Vergleichende Lebenszyklusanalyse von zwei Faserverbund-Konstruktionsweisen für ein Light Electric Vehicle	
R. Lach, L. Castro Key, B. Langer, W. Grellmann, B. Tillner, N. Teuscher, S. Celevics, I. Jahn, P. Michel	118
Fracture mechanics investigations of unidirectionally fibre-reinforced polymer tapes	
S. Sökmen, K. Oßwald, S. Ilisch, K. Reincke	119
Influence of TDAE oil on silica–rubber interactions in SBR/BR homopolymer and binary blends	
J. Posch, K. Oßwald, B. Langer, K. Reincke	120
Evaluation of the fracture behavior of PMMA materials with respect to environmental stress cracking	
R. Schlegel, H. Budde, U. Wendler, N. Mahmood, S. Henning, M. Beiner	121
Adjusting PI–PS-based multigraft copolymers properties via blending	
O. Aghazada, K. Oßwald, K. Reincke	122
Influence of the antioxidant type on the mechanical and viscoelastic properties of silica-filled NR and SBR vulcanizates after thermo-oxidative aging	
M. Seitz, S. Schröter	123
Katalytische Depolymerisation von polyolefinischen Kunststoffabfällen	

Firmenaussteller

Anton Paar GmbH

LIMES Messtechnik und Software GmbH, Krefeld

Mettler-Toledo GmbH

Tosoh bioscience GmbH, Griesheim

ZwickRoell GmbH & Co. KG, Ulm

Polymer Service GmbH Merseburg

Hochschule Merseburg

Institut für Polymerwerkstoffe e.V., Merseburg

F. Dinkel

Ist Recycling eine notwendige oder hinreichende Bedingung für eine nachhaltige Verpackung?

P. Skoczinski, M. Carus, D. de Guzman, H. Käß, R. Chinthapalli, J. Ravenstijn, W. Baltus, A. Raschka

Überblick über den Biokunststoffmarkt

A. Zankel

In situ and correlative electron microscopy for the characterization of polymeric materials

U. Laukant, T. Riedel, C. Woelk

Highlights der Schadensanalyse an Kunststoffen

S. Podzimek

Molar mass as the fundamental property of synthetic and natural polymers: Current status of its determination and some historical reminiscence

L. Kari

Modelling of tough, doubly crosslinked, single network hydrogel components in a mechanical context

J. Stampfl

Improving thermomechanical properties of photopolymers for additive manufacturing

Ist Recycling eine notwendige oder hinreichende Bedingung für eine nachhaltige Verpackung?

**F. Dinkel;
Carbotech AG, Basel (Schweiz)**

Überblick über den Biokunststoffmarkt

**P. Skoczinski, M. Carus, D. de Guzman, H. Käß, R. Chinthapalli, J. Ravenstijn,
W. Baltus, A. Raschka;
nova-Institut GmbH, Hürth (Deutschland)**

Das Jahr 2020 war ein vielversprechendes Jahr für bio-basierte Polymere: Ausverkauftes PLA im Jahr 2019 hat zur Aufstockung der Kapazitäten geführt, PE und PP aus bio-basiertem Naphtha sind auf dem Vormarsch und der zukünftige Ausbau der bio-basierten Polyamide sowie für PBAT, PHAs und Casein-Polymere zeichnet sich ab. Eine geringere Produktion ist nur bei bio-basiertem PET zu beobachten.

Mehrere globale Marken weiten ihr Rohstoff-Portfolio bereits aus, um neben fossilen auch erneuerbare Kohlenstoffquellen zu nutzen, CO₂, Recycling und insbesondere Biomasse, wodurch sich die Nachfrage nach bio-basierten sowie biologisch abbaubaren Polymeren weiter erhöht. Dennoch mangelt es gleichzeitig an Unterstützung durch die Politik, die nach wie vor nur Biokraftstoffe und Bioenergie fördert.

Der neue Markt- und Trendbericht „Bio-based Building Blocks and Polymers – Global Capacities, Production and Trends 2020–2025“ der internationalen nova-Biopolymer-Expertengruppe zeigt Kapazitäten und Produktionsdaten für alle bio-basierten Polymere im Jahr 2020 und eine Prognose für 2025.

Im Jahr 2020 betrug die gesamte Produktionsmenge bio-basierter Polymere 4,2 Mio. Tonnen, was 1 % der gesamten Produktionsmenge der Polymere aus fossilen Quellen entspricht. Zum ersten Mal seit vielen Jahren ist das jährliche Wachstum (CAGR) mit 8 % deutlich höher als das Gesamtwachstum der Polymere (3 – 4 %) – dies wird voraussichtlich bis 2025 anhalten.

Insgesamt beträgt der weltweite Flächenbedarf für bio-basierte Polymere nur 0,006 % der globalen landwirtschaftlichen Nutzfläche. Der wichtigste Biomasse-Rohstoff für die bio-basierte Polymerproduktion ist Glycerin als biogenes Nebenprodukt (37 %).

Im jährlich aktualisierten Marktbericht werden für das Jahr 2020 insgesamt 17 bio-basierte Building-Blocks und 17 Polymere beschrieben, dazu werden umfassende Informationen über die Kapazitätsentwicklung von 2020 bis 2025 sowie Produktionsdaten für das Jahr 2020 pro bio-basiertem Polymer dargestellt. Darüber hinaus enthält die Ausgabe Analysen zu Marktentwicklungen und Produzenten pro Building-Block und Polymer, so dass Leser sich schnell einen umfassenden Überblick über die aktuellen Entwicklungen verschaffen können, der weit über Kapazitäts- und Produktionszahlen hinausgeht.

***In situ* and correlative electron microscopy for the characterization of polymeric materials**

**A. Zankel^{1,2}, R. Schmidt^{1,2}, H. Fitzek², M. Nachtnebel², C. Mayrhofer²
J. Rattenberger², H. Schroettner^{1,2};**

¹ Institute of Electron Microscopy and Nanoanalysis, Graz University of Technology, NAWI Graz (Austria), ² Graz Centre for Electron Microscopy (Austria)

In this work new scanning electron microscopic methods for the investigation of polymeric materials are presented and discussed.

In the last two decades several new methods and techniques in the field of scanning electron microscopy (SEM) were developed and applied to different kinds of materials. This enables especially for polymers new insights into physical and chemical properties accompanied with imaging. In the environmental scanning electron microscope (ESEM) investigations are possible without applying an electrically conductive layer on specimens, which opens the field of new *in situ* methods like *in situ* tensile testing [1], *in situ* ultramicrotomy and even *in situ* experiments with wet specimens. The combination of an SEM with Raman microscopy enables correlative microscopy for the chemical and morphological characterization of complex materials of different length scales down dimensions of nanoplastic particles [2].

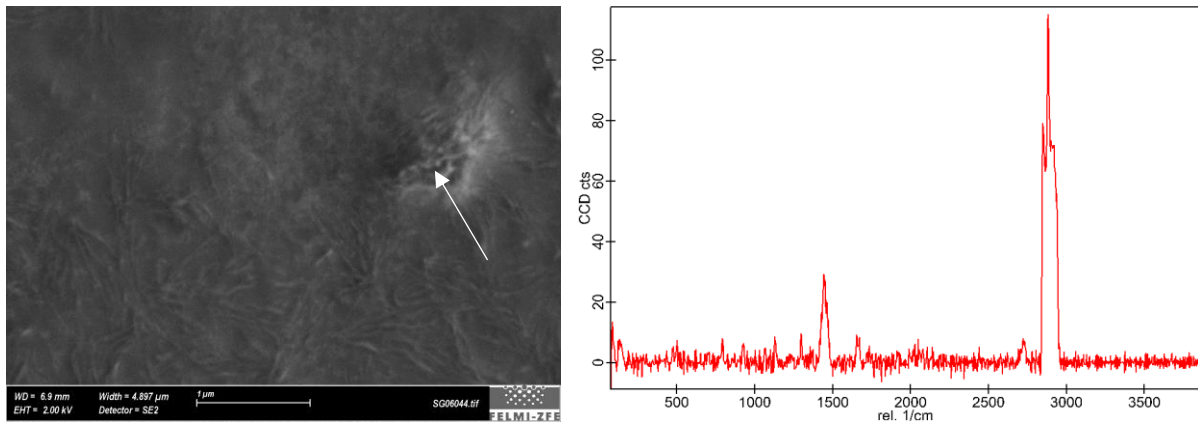


Figure 1: Left: SEM image of a nanoplastic particle (particle position indicated by a white arrow). Right: corresponding Raman spectrum of the particle (polyamide).

Literature

- [1] Heuser, M., Zankel, A., Mayrhofer, C., Reincke, K., Langer, B., Grellmann, W.: Characterisation of the opening behaviour of multilayer films with cohesive failure mechanism by in situ peel tests in the ESEM. *Journal of Plastic Film and Sheeting* (2021) submitted.
- [2] Schmidt, R., Nachtnebel, M., Dienstleder, M., Mertschnigg, S., Schroettner, H., Zankel, A., Poteser, M., Hutter, H.-P., Eppel, W., Fitzek, H.: Correlative SEM-Raman microscopy to reveal nanoplastics in complex environments. *Micron* 144 (2021) 1–7.

Highlights der Schadensanalyse an Kunststoffen

**U. Laukant, T. Riedel, C. Woelk;
Robert Bosch GmbH, Renningen (Germany)**

Die Fertigung von Kunststoffbauteilen aus Thermoplast hat ein bedeutendes Rationalisierungspotenzial bei Großserien, wobei sich der Hersteller im ständig verschärfenden Zielkonflikt zwischen Qualität und Wirtschaftlichkeit befindet. Gleichzeitig werden die Bauteile und die Bauteilanforderungen immer komplexer. Aus diesen Gründen sind leistungsfähige Methoden zur Qualitätssicherung und eine der Erprobung nachfolgende anspruchsvolle Schadensanalyse erforderlich. Nur durch die Kombination verschiedener Analysemethoden und dem entsprechenden Kunststoff-Know-how sind belastbare Aussagen möglich.

Die Schadensanalyse nach Bauteilerprobungen ist eine präventive Qualitätssicherung. Bereits in der Entwicklungs- und Musterphase von Bauteilen werden Optimierungspotenziale erkannt. Außerdem wird ein Erreichen des wichtigen Qualitätsziels „Null Fehler“ in Produktion und Feldeinsatz überhaupt erst möglich. Dazu ist der kontinuierliche Aufbau des Know-hows durch Fehlerdetektion und -analyse, eine ständige Weiterentwicklung der eingesetzten Analysemethoden sowie das Verständnis der Fehlerentstehung und der Wirkzusammenhänge genauso wichtig, wie die Vermeidung der Fehler.

Die Schadensanalyse soll das Schadensbild eindeutig erfassen, die Schadensursache identifizieren, Vorschläge für die Vermeidung der Fehler erarbeiten sowie im Idealfall Empfehlungen für die Optimierung von Fertigungs- und ggf. Einsatzbedingungen oder auch Einsatzgrenzen liefern. Entsprechend den Erfordernissen unterstützt die Schadensanalyse bei der Designoptimierung, der Werkstoffauswahl, der Verarbeitung und den Lebensdaueruntersuchungen.

In diesem Beitrag werden Schadensbilder an Thermoplast-Bauteilen, mögliche Fehlerquellen bei der Spritzgussverarbeitung und verschiedene Analysemethoden in der Schadensanalyse angesprochen. Bei der Auswahl wurde der Fokus auf Analysen gelegt, die durch überlegten Einsatz oder mit Hilfe von kleinen Weiterentwicklungen die Aussagekraft der Ergebnisse deutlich verbessert. Grundlegende Voraussetzung bei allen eingesetzten Analysen ist ein großer Erfahrungsschatz für die Interpretation und Bewertung der Ergebnisse.

Molar mass as the fundamental property of synthetic and natural polymers: Current status of its determination and some historical reminiscence

S. Podzimek^{1,2,3};

¹ Wyatt Technology, Dernbach (Germany), ² SYNPO, Pardubice (Czech Republic), ³ Faculty of Chemical Technology, Pardubice (Czech Republic)

The physical and application properties of all polymers are to a large extent determined by their molar mass distribution, which is also needed for the deduction of kinetics information about polymerization reactions and understanding degradation processes.

Most of the methods used at the beginning of polymer science to determine the molar mass of polymers have been completely or almost forgotten. Such methods are cryoscopy, ebullioscopy, membrane and vapor phase osmometry, and precipitation fractionation. Knowledge of these techniques may be useful just from the view point of understanding fundamental polymer physics and appreciation of the modern analytical instrumentation. Size exclusion chromatography (SEC), despite IUPAC recommendation still being frequently called by its original name gel permeation chromatography (GPC), has taken predominating role in the characterization of complete molar mass distribution of synthetic and natural polymers. The method dates back to the sixties of the last century when its introduction became a revolution in the polymer characterization [1]. However, the method in its original form with calibration of columns with polymer standards suffers from many limitations and the acquired results may be in the case of some polymers even misleading. The combination of SEC with an online multi-angle light scattering (MALS) detector in the eighties of the last century represented the second breakthrough in polymer characterization [2]. The combined SEC-MALS technique provides absolute molar mass distribution and also valuable information about the molecular structure and efficiency of SEC separation. The method can be further coupled with an online viscometer for even more detailed insight into the polymer molecular structure. Alternative analytical separation technique, asymmetric flow field flow fractionation (AF4), can replace SEC when it fails to separate polymers containing branched and/or ultra-high molar mass fractions. The lecture offers a review of basic principles of SEC-MALS and AF4-MALS with examples of the application of the techniques for the characterization of elastomers, polyolefins and acrylic emulsion polymers.

Literature

- [1] Moore, J. C.: Gel Permeation Chromatography. I. A new method for molecular weight distribution of high polymers. *J. Polym. Sci: Part A2* (1964) 835–843.
- [2] Wyatt, P. J.: Light scattering and the absolute characterization of macromolecules. *Anal. Chim. Acta* 272 (1993) 1–40.

Modelling of tough, doubly crosslinked, single network hydrogel components in a mechanical context

L. Kari;

KTH Royal Institute of Technology, Stockholm (Sweden)

Doubly cross-linked, single network hydrogels embody both chemical and physical cross-links. Chemical cross-links are covalent bonds while the physical cross-links may include hydrogen bonds, hydrophobic interactions, π - π interactions, van der Waals interactions and ionic interactions; that is, ion-ion, ion-dipole and dipole-dipole interactions. Only the chemical cross-links are active at very low frequencies while both chemical and physical cross-links are active at higher frequencies, see Figure 1. The bonding and de-bonding activities of the physical cross-links are at their highest at frequency f_{ald} resulting in a maximum loss modulus. A four parameter shear modulus model [1] is presented, displaying an almost clean and constant elastic contribution from the chemical cross-links with an added minor overlaid Rouse kind, half fractional-order frequency contribution to the chemical storage and to the chemical loss modulus. The physical cross-links show a Rouse kind, half fractional-order frequency influence to the physical storage modulus and to the physical loss modulus in the low frequency range, display a fast frequency increase of the physical storage modulus in the medium frequency range where the physical loss modulus shows a peak and, lastly, display a high, plateau like physical storage modulus in the high frequency range while the physical loss modulus decreases with frequency. The shear model results and those of the measurements match very well. Various mechanical components made of this tough hydrogel are modeled in the presentation while using the shear modulus model including vibration isolators and dynamic vibration absorbers.

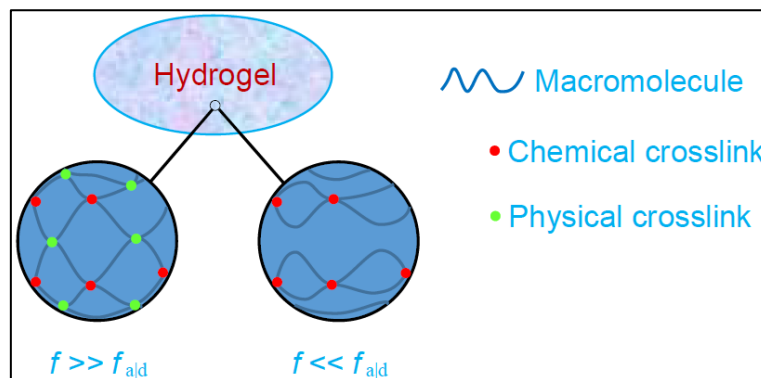


Figure 1: Doubly cross-linked, single network hydrogel excited at high and low frequencies.

Literature

- [1] Kari, L.: Effective visco-elastic models of tough, doubly cross-linked, single-network polyvinyl alcohol (PVA) hydrogels – Additively separable fractional derivative based models for chemical and physical cross-links. Continuum Mech. Therm. (2020) published online.

Improving thermomechanical properties of photopolymers for additive manufacturing

J. Stampfl;

Institute of Materials Science and Technology, TU Wien (Austria)

Lithography-based methods for additive manufacturing of polymers, ceramics and metals enable the fabrication of precise, complex-shaped parts with excellent surface quality in applications like biomedical engineering, electronics and consumer products. Photopolymers contributed 33 % to the worldwide materials sales in 2020, thus making lithography-based AM (L-AM) the most widely used AM technology. Although polymers are the dominating material class for L-AM, it is also possible to manufacture composite, ceramic and metallic materials using L-AM.

The goal in the development of innovative photopolymers for AM is to achieve similar thermomechanical properties like engineering thermoplastics, e.g. ABS or polyamide. In this work, a fracture mechanical approach is used to understand the influence of processing conditions and chemical structure of the utilized monomers on the finally observed mechanical properties.

Traditional photopolymers for L-AM provide high strength and stiffness, but typically lack sufficiently high fracture toughness [1] due to the high cross-link density which prevents plastic deformation. Several approaches are available for increasing toughness [2], e.g. the introduction of cross-linkers with spacers which increase the physical interaction within the final polymer chains (e.g. hydrogen bonds). Dedicated chain transfer agents [3] are also beneficial for improving ductility of the polymer without sacrificing the reactivity of the resin.

In addition to presenting new concepts for improving thermomechanical properties of photopolymers, an overview of recently developed 3D-printable materials will be given, allowing to tackle challenges associated with innovative applications in biomedicine, electronics and engineering.

Literature

- [1] Ligon, S. C., Liska, R., Stampfl, J., Gurr, M., Mühlhaupt, R.: Polymers for 3D printing and customized additive manufacturing. *Chemical Reviews* 117 (2017) 10212–10290.
- [2] Ligon, S., Schwentenwein, M., Gorsche, C., Stampfl, J., Liska, R.: Toughening of photocurable polymer networks: a review. *Polymer Chemistry* 7 (2016) 257–286.
- [3] Seidler, K., Griesser, M., Kury, M., Harikrishna, R., Dorfinger, P., Koch, T., Svirikova, A., Marchetti-Deschmann, M., Stampfl, J., Moszner, N., Gorsche, C., Liska, R.: Vinyl sulfonate esters: Efficient chain transfer agents for the 3D printing of tough photopolymers without retardation. *Angewandte Chemie* 57 (2018) 9165–9169.

P. Schrader, A. Gosch, M. Berer, S. Marzi

Determining the fracture energy of polymeric bulk specimens loaded in modes I and III

A. Gosch, M. Berer, F. Arbeiter, T. Vojtek, P. Hutař, G. Pinter

Mixed mode I/III fatigue testing of unreinforced thermoplastic polymers

E. Pérez

Particle debonding contribution to the fracture toughness of polymer based composites

W. Li, B. Wang, G. Kalinka

Temperature-dependent energy dissipations during epoxy resin fracture

C. Rosales, A. Costantino, T. Echeverria, C. Bernal, V. Pettarin

On the use of fracture mechanics techniques to assess the performance of recycled polymer blends

E. Nezbedova, M. Kovar, R. Lach, W. Grellmann

Informational value of short-term tests to predict the lifetime of less and high oriented PE-HD compared to long-term tests such as FNCT and PENT

B. Gerets, K. Engelsing

Fast testing of the stress crack resistance of polyamide 12

E. Nezbedova, M. Kovar, R. Lach, W. Grellmann

Determination of fracture toughness under plane stress impact conditions of polyethylene (PE)

M. Chytil, J. Poduška, J. Kučera, P. Hutař

Strain hardening test of PE-HD materials in relation to some accelerated lifetime prediction methods

Determining the fracture energy of polymeric bulk specimens loaded in modes I and III

P. Schrader¹, A. Gosch², M. Berer³, S. Marzi¹;

¹ Technische Hochschule Mittelhessen, Institute of Mechanics and Materials, Giessen (Germany), ² Montanuniversität Leoben, Materials Science and Testing of Polymers (Austria), ³ Polymer Competence Center Leoben GmbH (Austria)

Thin-walled polymeric components are used in a wide range of applications. Hence, knowledge about their fracture behavior in bulk is beneficial in practice. Within this study, we present experimental setups to identify the fracture energy of bulk specimens in both mode I and mode III. Double Cantilever Beam (DCB) and Out-of-Plane Loaded Double Cantilever Beam (ODCB) tests are modified to enable testing of such specimens, allowing an evaluation of the fracture energy in both loading modes based on the J-integral. Tests were carried out on a semicrystalline polymer (polyoxymethylene) under quasi-static load conditions. Furthermore, a qualitative fracture surface analysis is performed to examine the dominant crack growth mechanisms.

Mixed mode I/III fatigue testing of unreinforced thermoplastic polymers

A. Gosch¹, M. Berer², F. Arbeiter¹, T. Vojtek^{3,4}, P. Hutař³, G. Pinter¹;

¹ Materials Science and Testing of Polymers, Montanuniversität Leoben (Austria), ² Polymer Competence Center Leoben GmbH (Austria), ³ Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno (Czech Republic), ⁴ Central European Institute of Technology (CEITEC), Brno (Czech Republic)

Mixed mode I/III fatigue fracture investigations of unreinforced thermoplastic polymers are scarcely documented in literature, even though a significant amount of mixed mode loading close to the crack tip is likely to occur in several applications. To name some examples a randomly orientated crack in a component which is used in rolling contact [1–3], twisting of a pipe during installation, etc. Therefore, pure mode I and mixed mode I/III fatigue loading of two unreinforced polymers was investigated in detail in previous studies [4, 5]. The applied mixed mode I/III load leads to both increases and decreases of lifetime, depending on the levels of applied mode I and mode III amount. In the conducted work [4, 5] an equivalent stress intensity factor (K_{eq}) was presented, which takes both loading cases of mode I and mode III into account. Furthermore, the critical influence of friction and the accompanying increase of local temperature between the shear loaded crack flanks in mode III as well as the dominant fracture mechanisms were discussed based on the fracture surface morphology.

References

- [1] Dlhy, P., Poduska, J., Nahlik, L., Berer, M., Gosch, A., Pinter, G., Hutar, P.: Compression-loaded cracked cylinder – Stress intensity factor evaluation. *Key Eng. Mater.* 774 (2018) 331–336.
- [2] Berer, M., Major, Z.: Characterization of the global deformation behaviour of engineering plastics rolls. *Int. J. Mech. Mater. Des.* 6 (2010) 1–9.
- [3] Berer, M., Major, Z.: Characterisation of the local deformation behaviour of engineering plastics rolls. *Strain* 48 (2012) 225–234.
- [4] Gosch, A., Arbeiter, F. J., Berer, M., Vojtek, T., Hutař, P., Pinter, G.: Fatigue characterization of polyethylene under mixed mode I/III conditions. *Int. J. Fatigue* 145 (2021) 106084.
- [5] Gosch, A., Berer, M., Hutař, P., Slávik, O., Vojtek, T., Arbeiter, F. J., Pinter, G.: Mixed mode I/III fatigue fracture characterization of polyoxymethylene, *Int. J. Fatigue* 130 (2020) 105269.

Particle debonding contribution to the fracture toughness of polymer based composites

E. Pérez^{1,2};

¹ National Scientific and Technical Research Council (CONICET), Buenos Aires (Argentina), ² National Institute of Industrial Technology (INTI), Buenos Aires (Argentina)

Incorporation of rigid particle into polymer matrices represents a widely used technic to obtain composite material with enhanced mechanical performance. Rigid fillers can induce energy dissipation mechanisms (plastic void growth, cavitation, shear bands, crazing, localized matrix yielding, among others) conducting to fracture toughness enhancement of polymer based composite materials [1–3]. The properly activation of these mechanisms have been related to the debonding process. In the present work, debonding strength of rigid particles and its contribution to the fracture toughness is investigated.

Debonding process was analyzed by a general solution previously published [4, 5]. For spheres, elliptical particles and fibers increased critical values with reduced filler sizes were observed. Particularly, the highest stresses correspond to stiffer interphases independently of the particle dimension or geometry. The contribution of filler debonding and plastic void growth on the fracture toughness was investigated by an originally proposed analytical solution. In addition, the influence of dissipation zone placed around the fracture plane was also investigated. The obtained results suggest the relevance of involved properties (matrix yielding volume, specific fracture surface energy) which should not be considered as constant values for variable filler content or particle sizes. On this way, more realistic predictions can be obtained with minimal and/or maximal performances varying the filler content or size.

Literature

- [1] Chen, J. K., Huang, Z. P., Zhu, J.: Size effect of particle on the damage dissipation in nanocomposites. *Comp. Sci. Tech.* 67 (2007) 2990–2996.
- [2] Williams, J. G.: Particle toughening of polymers by plastic void growth. *Comp. Sci. Tech.* 70 (2010) 885–891.
- [3] Zapalorto, M., Salviato, M., Quaresimin M.: Influence of the interphase zone on the nanoparticle debonding stress. *Comp. Sci. Tech.* 72 (2011) 249–255.
- [4] Pérez, E., Lauke, B.: Calculation of debonding strength at the interface of particles with different shapes and matrix. *Comp. Struct.* 167 (2017) 262–270.
- [5] Pérez, E., Lauke, B.: Evaluation of the particle geometry and interphase influence on the filler matrix debonding process. *Mater. Res. Express* 6 (2019) 085341.

Temperature-dependent energy dissipations during epoxy resin fracture

W. Li, B. Wang, G. Kalinka;

Federal Institute for Materials Research and Testing, Berlin (Germany)

The polymer, as the matrix material of fiber-reinforced polymer (FRP) composite, influences the micro-scale failure of FRPs strongly. Therefore, knowing the behavior in the polymer matrix is essential for predicting the failure of FRPs.

Epoxy resin is a common matrix material used in FRP. Fracture testing is the common method for studying the material failure. Through this test, the G value (G_{Ic}) is obtained during polymer fracture. In order to obtain a desired stable crack propagation at the crack tip in the fracture test on the epoxy resin, a compact tension (CT) specimen with a designed tapered double cantilever beam (TDCB) geometry (Figure 1A) was employed in this research.

Firstly, G_{Ic} with different pre-crack lengths were measured to find the range of stable crack propagation. The obtained stable range is 18–35 mm away from the origin of crack tip. G_{Ic} including the pre-crack length within this range is $180 \pm 20 \text{ J/m}^2$. In addition, the experimental result is validated by finite element analysis (FEA) simulation using the virtual crack-closure technique (VCCT) (Figure 1B), the corresponding numerical value is $150 \pm 20 \text{ J/m}^2$, which is 20 % lower than the experimental value. The reason is that, the local irreversible deformation occurs at the fracture surface during the fracture at room temperature. Secondly, to investigate the temperature-dependence of the energy dissipation during the epoxy resin fracture, the fracture tests at different temperatures were performed. G_{Ic} values during crack propagation within above-mentioned stable range were measured. The G values decrease with the increasing crack propagation length linearly, and G_{Ic} fitting lines at different temperatures parallel to each other (Figure 1C).

The TDCB specimen provides a stable crack propagation range in polymer. Within this range, the determined G_{Ic} at room temperature is constant. Additionally, the epoxy resin irreversibly deforms during the fracture at higher temperatures (40 °C, 50 °C and 60 °C). The corresponding G_{Ic} decreases during crack propagation. And the tendencies at different temperatures is nearly the same. In the following, fracture tests with TDCB specimens will be extended to more severe temperature ranges. The temperature-dependence of the energy dissipation during crack propagation in the epoxy resin will be thoroughly investigated.

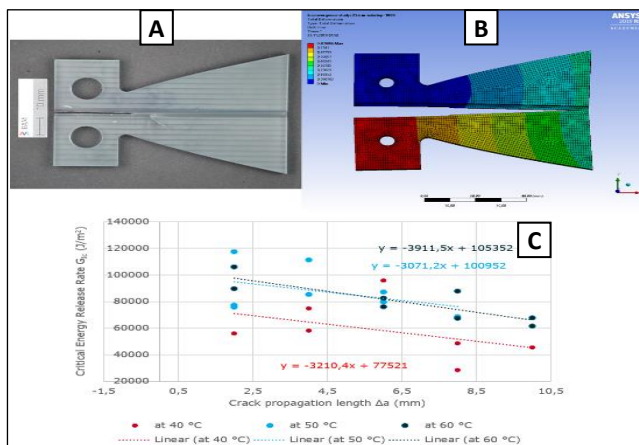


Figure 1: TDCB specimen (A), TDCB model (B), G-crack propagation length diagram (C).

On the use of fracture mechanics techniques to assess the performance of recycled polymer blends

C. Rosales², A. Costantino², T. Echeverria², C. Bernal¹, V. Pettarin²;

¹ Institute of Materials Science and Technology, University of Mar del Plata (Argentina), ² Institute of Polymers and Nanotechnology, University of Buenos Aires (Argentina)

Plastic waste accumulation is one of the main environmental problems of the XXI century. Plastics could be seriously harmful for environment due to their slow and complex degradation process. Some plastics are difficult or expensive to separate in individual materials, and then an effort is done in order to recycle them as blends. The difficult task of dealing with blends is to obtain a material with good mechanical properties, since the presence of a polymer dispersed in a second one may strongly change these properties and limit their use in conventional applications. It is then evident that one of the big engineering challenges in recycling thermoplastic blends is to manufacture competitive products in terms of mechanical properties such as toughness or strength, i.e. to upcycling, understanding upcycling as a process by which recycled waste is transformed into materials with improved performance, adding value and widening their application field. Nowadays, most works which deal with recycled blends evaluate mechanical performance by means of conventional mechanical tests such as tensile, flexural or non-instrumented impact tests which in many cases are not representative of actual loading situations [1]. In a desire to characterize toughness of ductile polymers, we have turned to fracture mechanics, believing that fracture tests, that imply a more severe loading condition, i.e., load triaxiality, are more adequate to evaluate mechanical integrity of final pieces [2]. Through this work, a complete morphological and mechanical evaluation of different recycled thermoplastic blends is presented. Microstructure and morphology of blends were analyzed by XRD, DSC and SEM. Tensile and fracture tests were carried out on mode I double edge-notched tensile specimens (DDENT) at quasi-static loading conditions. Fracture surfaces were studied by SEM. Results arisen from fracture tests, both qualitative and quantitative, were proven to be a very efficient tool to evaluate the performance of this type of blends.

Literature

- [1] Fang, C., Nie, L., Liu, S., Yu, R., An, N., Li, S.: Characterization of polypropylene–polyethylene blends made of waste materials with compatibilizer and nano-filler. *Compos. B. Eng.* 55 (2013) 498–505.
- [2] Anderson, T.: *Fracture Mechanics: Fundamentals and Applications*. 4th ed., CRC Press, Boca Raton (2017).

Informational value of short-term tests to predict the lifetime of less and high oriented PE-HD compared to long-term tests such as FNCT and PENT

E. Nezbedova¹, M. Kovar¹, R. Lach², W. Grellmann²;

**¹UNO Praha, spol. s.r.o., Úhonice (Czech Republic) ²Polymer Service GmbH
Merseburg (Germany)**

Geo-membrane is manufactured only from technically selected, high quality grades of PE-HD, PP, and PE-LD or from some combinations of these grades. The process of manufacturing runs under carefully controlled proven extrusion technology. The durability either of pellet or of geo-membrane is determined by using accelerated test FNCT (ISO 16770) and NCTL (EN 12814-3, ASTM D 5397) test, respectively [1, 2]. For unimodal grade the time to failure according these accelerated tests is about ten hours, for bimodal grade PE 100 it is roughly thousands hours and for the new PE 100-RC grade (RC – Resistant to Cracking) is often greater than 1 year. There has been the need even for these materials to obtain the results in relatively short time. In 2015 a new ISO standard was accepted (ISO 18488: Polyethylene (PE) materials for piping systems – Determination of Strain Hardening Modulus in relation to slow crack growth – Test method) [3]. However this test method still need better understanding with respect to the molecular structure and comparative study with existing test e.g. FNCT and NCTL test, respectively.

This contribution is focused on correlation between SHT and NCTL (FNCT) of PE-HD material to be used for production of geo-membrane. The molecular parameters were used for explanation the discrepancies.

Literature

- [1] Van der Stock, E., Scholten, F.: Strain Hardening Test on PE pipe materials. In: Proceedings of Plastic Pipes XVI (Barcelona, 24.–26.09.2012). Barcelona (2012), 10 pages.
- [2] Nezbedová, E., Kučera, J.: Experimentelle Methoden zur Charakterisierung des Bruchverhaltens von HDPE-Rohren. In: Grellmann, W., Seidler, S. (Eds.): Deformation und Bruchverhalten von Kunststoffen. Springer-Verlag, Berlin Heidelberg (1998), 91–98.
- [3] ISO 18488 (2015): Polyethylene (PE) materials for piping systems – Determination of Strain Hardening Modulus in relation to slow crack growth – Test method.

Fast testing of the stress crack resistance of polyamide 12

**B. Gerets, K. Engelsing;
SKZ – German Plastics Center, Würzburg (Germany)**

Plastic components made of polyamide 12 (PA 12) are frequently used in long-term applications requiring service lifetimes of 50 years or even more. The challenge in material selection, development and quality assurance is to test these properties quantitatively through accelerated / fast tests. An established method to characterize the stress crack resistance respectively slow crack growth behavior of PA 12 does not exist today. Based on the experience with PE characterization [1] and the recently established strain hardening test [2], a method for the characterization of PA 12 materials was developed, which evaluates the stress-strain behavior in the strain hardening region measured by uniaxial tensile tests. For this purpose test and evaluation methods have been thoroughly examined and optimized. [3, 4] The influence of the test temperature is illustrated in Figure 1 for a PA 12 grade.

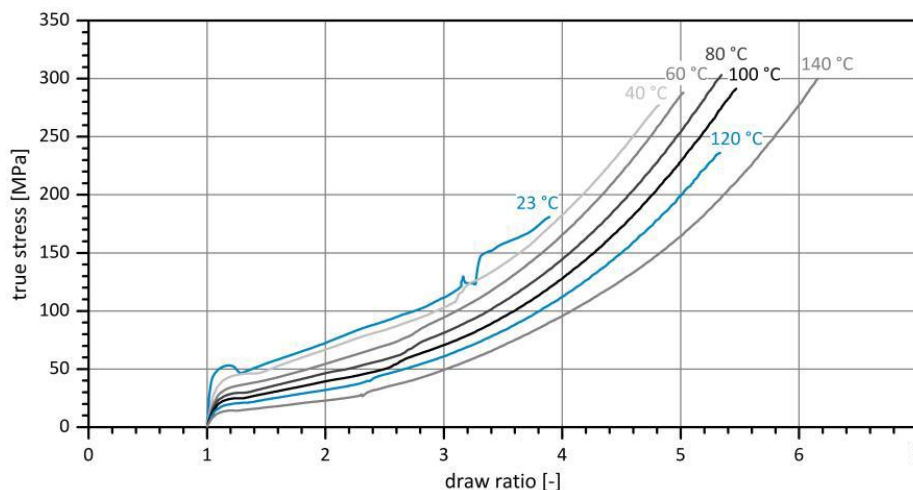


Figure 1: Influence of temperature on the strain hardening behavior of a PA 12 grade.

Literature

- [1] Gerets, B.: Schnelltest-Methode zur Charakterisierung der Spannungsrisssbeständigkeit von Polyethylen (final report IGF-Nr. 18324 N). SKZ – German Plastics Center, Würzburg (2016).
- [2] ISO 14888 (2015): Polyethylene (PE) materials for piping systems – Determination of Strain Hardening Modulus in relation to slow crack growth – Test method.
- [3] Gerets, B., Engelsing, K., Hochrein, T., Bastian, M.: Charakterisierung der Spannungsrisssbeständigkeit von Thermoplasten – Entwicklung eines schnellen Prüfverfahrens (final report IGF-Nr. 18649 N). SKZ – Das Kunststoff-Zentrum, Shaker Verlag, Düren (2019).
- [4] Gerets, B., Messiha, M.: Moderne Prüfmethoden zur Charakterisierung der Spannungsrisssbeständigkeit von PE-HD und PA-U 12. 17. Würzburger Kunststoffrohr-Tagung, Würzburg (25./26.06.2019).

Determination of fracture toughness under plane stress impact conditions of polyethylene (PE)

E. Nezbedova¹, M. Kovar¹, R. Lach², W. Grellmann²;

¹ UNO Praha, spol. s.r.o., Úhonic (Czech Republic), ² Polymer Service GmbH Merseburg (Germany)

In 2015 Czech Republic during the ISO meeting of TC 61 WG 7 proposed a NWI: Plastics – Determination of fracture toughness under plane stress impact conditions of polyethylene (PE). This document describes a test method to measure fracture toughness (G_d) under plane stress impact conditions to be utilized for the analysis of Rapid Crack Propagation (RCP) of polyethylene pipe resins as well as to predict S4 performance of the material. A plane stress impact (PSI) test is offered as an inexpensive way to control RCP phenomena. RCP is typically characterized with notched or un-notched test specimens on pendulum and drop weight type testers [1–3]. Results, usually in form of absorbed energy are used for quality control, screening, material development, or studying the effect of other material characteristics on RCP. The pipe industry uses two qualification tests to characterize RCP, the S4 Test (ISO 13477) and the Full Scale Test (ISO 13478). These tests measure critical pressure and critical temperature for RCP in pipe. The first test is performed in a lab scale, requires pipes length 7 times the pipe diameter and decompression baffles. The second one uses pipe minimally 14 m long. These tests do not directly measure the fracture toughness, G_d , however, their results are affected by it. The method in this standard describes an alternative, inexpensive laboratory method, to characterize Rapid Crack Propagation phenomena in PE pipes. In 2016 a RRT was initiated to verify the use of method not only for PE-HD (PE80, PE100, PE100 RC) material but also for PP-RC, PA 12 and PBT (see Table 1). Four countries took part in this RRT (Czech Republic, Germany, India and China). Basically, the PSI can be used not only for PE but also for PP, PA and PBT. The PBT has the worse dampening for the same thickness as is the thickness for the other materials in study. There is the problem in case if only e.g., K value is estimated, but not for energy. According to the G_{in} as well as G_c the resistance against RCP can be judge.

Table 1: Results of RRT.

material	G_{in} (J/m ²)		G_c (J/m ²)	
	average value	standard deviation	average value	standard deviation
PA	10855	59	23944	4391
PBT	1801	647	2319	143
PE80	6136	382	19305	454
PE100	10067	509	37922	1547
PE100 RC	10384	375	44203	2928
PP-RC	6401	478	6775	481

Literature

- [1] Nezbedová, E., Kučera, J., Vlach, B.: Fracture behaviour of HDPE under dynamic load. *Eng. Mech.* 5 (1998) 169–174.
- [2] Šindelář, P., Nezbedová, E., Šimková, P., Buráň, Z., Bohatý, P.: Effect of structural parameters on rapid crack propagation and slow crack growth in high density polyethylene pipeline materials. *Plast. Rubber Compos.* 34 (2005) 329–333.
- [3] Grellmann, W., Seidler, S. (Eds.): *Polymer Testing*. Carl Hanser Verlag, München (2007).

Strain hardening test of PE-HD materials in relation to some accelerated lifetime prediction methods

M. Chytil¹, J. Poduška², J. Kučera¹, P. Hutař²;

¹ Unipetrol RPA, s.r.o. – Polymer Institute Brno (Czech Republic), ² Institute of Physics of Materials, Czech Academy of Science, Brno (Czech Republic)

For over a decade, Strain Hardening (SH) has been extensively studied as an alternative to the conventional lifetime predicting methods evaluating resistance to slow crack growth (SCG) such as Full Notch Creep Test (FNCT), Pennsylvania Edge Notch Test (PENT), etc. [1, 2] mostly due to its simplicity and less time-demanding preparation. The SH is based on measuring stress–strain curves of a PE-HD at 80 °C and at 20 mm/min elongation rate up to high draw ratio $\lambda = 1 + \varepsilon$, where ε represents relative strain. Strain Hardening modulus $\langle G_p \rangle$ is calculated from the slope of the strain hardening part of the true stress–true strain curve for $\lambda > 8$ and it correlates with SCG [1, 3].

The $\langle G_p \rangle$ determined for the range of PE-HD materials from a sheet extrusion PE-Exthrough PE80 to bimodal new class pipe grades (PE100, PE100 RC) increases within the pipe grades in the PE80–PE100 RC order, which correlates well with FNCT. However, PE-Ex exhibits slightly greater $\langle G_p \rangle$ than PE80 grades, despite its less profound SCG observed by FNCT and PENT. PE-Ex shows also greater yield stress, less short chain branches, although longer than PE80; thus, $\langle G_p \rangle$ seems not being a separated parameter playing a key role in SCG of PE-HD.

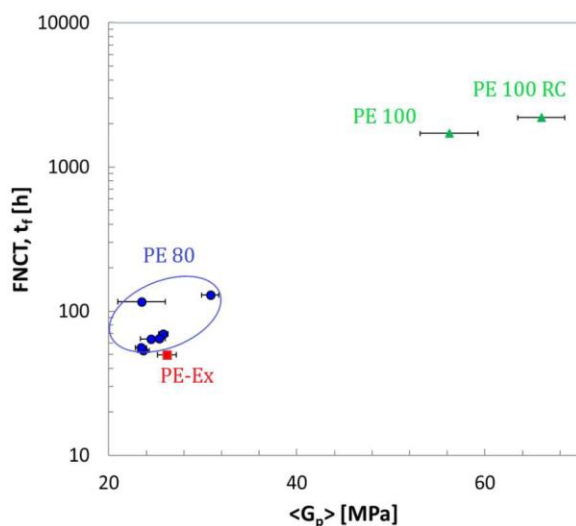


Figure 1: Time to failure t_f of a sheet extrusion PE-Ex, PE80 and bimodal pipe grades PE-HD materials by FNCT vs. correspondent $\langle G_p \rangle$ values.

Literature

- [1] Kurelec, L., Teeuwen, M., Schoffeleers, H., Deblieck, R.: Strain hardening modulus as a measure of environmental stress crack resistance of high density polyethylene. *Polymer* 46 (2005) 6369–6379.
- [2] Havermans-van Beek, D. J., Deblieck, R., Mc Carthy, M., Kloth, R., Kurelec, L.: Strain hardening: An elegant and fast method to predict the slow crack growth behavior of HDPE pipe materials. *Conference Proceedings EUROTEC 2011, Society of Plastics Engineers* (2011), 5 p.
- [3] Deblieck, R. A. C., Beek van, D. J. M., Remerie, K., Ward, I. M.: Failure mechanisms in polyolefines: The role of crazing, shear yielding and the entanglement network. *Polymer* 52 (2011) 2979–2990.

M. Du, K. Jariyavidyanont, R. Andrich

Effect of molar mass on critical specific work of flow for shear-induced crystal nucleation in poly (L-lactic acid)

S. McGowan, A. Siebert-Rats

Effect of hydrolysis in respect of process ability and performance characteristics of polylactic acid (PLA)

S. Richter, N. Teuscher, M. Heinze, R. Hoffmann, T. Pscherer

Atmospheric pressure plasmas for adhesion improvement of natural substances on bio-based polymer films

R. Pandit, S. Rijal, K. Chapain, R. Adhikari

Fabrication and physicochemical characterization of starch based bioplastic films from banana peels

K. Aslan

Influence of chemical and physical treatment on the structure and processability of Kraft lignin for extrusion applications

D. Zehm, A. Lieske

FDCA-based polymer materials beyond PEF

N. Eversmann, M. Wießner, A. Krombholz

Naturbasierte Schäume als funktionalisierte adaptive Elemente

Effect of molar mass on critical specific work of flow for shear-induced crystal nucleation in poly (L-lactic acid)

M. Du, K. Jariyavidyanont, R. Androsch;

Interdisciplinary Center for Transfer-oriented Research in Natural Sciences,
Martin Luther University Halle-Wittenberg, Halle (Germany)

As well known, shear, evident in polymer melt-processing, has a significant influence on polymer crystallization, structure and eventually properties. Therefore, the evaluation of the effect of shear on the crystallization process is an indispensable task for understanding the relationship between processing, structure, and properties. Although many studies reported about the shear-induced crystallization of polymers, quantitative analysis of the effect of molar mass on shear-induced crystallization of poly (L-lactic acid) (PLLA) still is needed.

In this work, the concept of specific work of flow has been applied for analysis of critical shearing conditions for formation of crystal nuclei in PLLA [1, 2]. For the analysed PLLAs with mass-average molar masses of 70, 90, 120, and 576 kDa, critical specific works of flow, above which shear-induced nuclei formation occurs, of around 550, 60, 25, and 2 kPa were obtained, as shown in Figure 1 by the decrease of crystallization times, respectively. The higher the molar mass, the lower is the critical specific work for generation of crystal nuclei. The obtained results are in agreement with additional morphological analyses of the semi-crystalline morphology, showing an increasing number of point-like nuclei after shearing the melt.

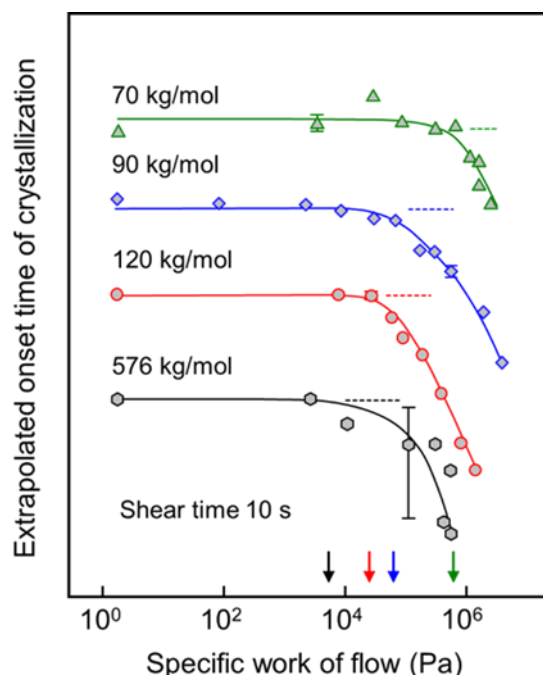


Figure 1: Extrapolated crystallization onset time of PLLA of different molar mass as a function of specific work of flow after shearing the melt at 135 °C for 10 s. The colored arrows at the specific-work-of-flow axis indicate critical specific works of flow for shear-induced crystal nucleation.

Literature

- [1] Janeschitz-Kriegl, H., Ratajski, E., Stadlbauer, M.: Flow as an effective promoter of nucleation in polymer melts: a quantitative evaluation. *Rheol. Acta* 42 (2003) 355–364.
- [2] Iqbal, N., Jariyavidyanont, K., Rhoades, A. M., Androsch, R.: Critical specific work of flow for shear-induced formation of crystal nuclei in poly (L-lactic acid). *Polym. Crystallization* 2 (2019) e10073.

Effect of hydrolysis in respect of processability and performance characteristics of polylactic acid (PLA)

S. McGowan, A. Siebert-Raths;

IfBB – Institute for Bioplastics and Biocomposites, Hannover (Germany)

In order to characterize the degree of hydrolysis of PLA (polylactic acid) during thermal forming processes. Predefined granulates with different degrees of moisture were used in thermal forming processes as extrusion and injection molding. The resulting mechanical characteristic values from these experiments are tested via standard test bars.

To obtain additional information about the hydrolysis during melt injection process further studies were performed on the capillary rheometer. During the tests were also different conditioned PLA granulates on different test temperatures investigated.

The results show that the level of moisture from PLA is of significant importance for the process ability as well as performance characteristics. Especially during the injection molding process, the moisture content of the PLA indicates effects on the hydrolysis. In addition to the moisture content also the processing temperature imply a strong influence on the hydrolysis.

Atmospheric pressure plasmas for adhesion improvement of natural substances on bio-based polymer films

S. Richter¹, N. Teuscher¹, M. Heinze², R. Hoffmann², T. Pscherer³;

¹ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ² POLIFILM Extrusion GmbH, Südliches-Anhalt, OT Weißandt-Göolzau (Germany), ³ TWINscrew GmbH, Hof (Germany)

The EU Plastics Strategy aims to develop 100 % recyclable plastics by 2030 [1]. This poses new challenges, especially for manufacturers and distributors of flexible packaging. Especially for food packaging, new innovative materials have to be developed that are easily recyclable, at best biodegradable and/or made from renewable raw materials. The requirements such as mechanical stability, migration barrier against aroma and taste, but also the printability and sealing must be guaranteed.

A current research project of the Investment Bank Sachsen-Anhalt (FuE No.: 1904/00034) aims at the development and production of new, innovative multilayer polymers made of biopolymers, which have an efficient barrier effect against oxygen and despite which have a high mechanical stability, sufficient transparency and other properties relevant for late use, including antimicrobial properties.

The presentation shows results for the effective plasma modification of the bio-based polymer films for adhesion improvement of natural substances with barrier effect. Relationships between plasma parameters and the surface energy, chemical and morphological properties and their correlation to the adhesion and barrier properties of the applied coatings are presented.

Literature

- [1] Abele, K., Bethke, L.: Pressemitteilung EU Kommission; Kommission legt europäische Plastikstrategie vor. https://ec.europa.eu/germany/news/20180116-plastikstrategie_de (2018).

Fabrication and physicochemical characterization of starch based bioplastic films from banana peels

R. Pandit^{1,2}, S. Rijal¹, K. Chapain¹, R. Adhikari^{2,3};

¹ Department of Chemistry, Tri-Chandra Multiple Campus, Tribhuvan University, Kathmandu (Nepal), ² Nepal Polymer Institute (NPI), Kathmandu (Nepal),

³ Research Centre for Applied Science and Technology (RECAST), Tribhuvan University, Kathmandu (Nepal)

Bioplastic is one of the alternatives of synthetic commodity plastics for short-term packaging applications and can help to reduce the use of non-degradable synthetic materials. In this research, starch was extracted from banana peels and bioplastic films were prepared using various plasticizers such as glycerol, urea, distilled water and glucose by solution casting method. The prepared films were characterized by FTIR spectroscopy, acid-base uptake resistance, soil burial tests and electron microscopy, carbon content and water absorption ability. The FTIR spectra of the films showed the weak peak around 1024 cm^{-1} C–O indicating hydroglucose ring while the peak observed at 2920 cm^{-1} corresponded to CH bond stretching of CH_2 group indicating the presence of amylose and amylopectin. The peak centered at 1598 cm^{-1} indicated the C=O vibration in the amide I region. The film resisted longer days in base uptake as compared to acid. The percentage carbon content in bioplastic film was more than polyethylene and lesser than normal papers. The soil decomposition test showed that the film was decomposed completely within two and half months. The film prepared at low concentration of plasticizer showed the less water absorption as compared to other higher concentration in water absorption ability test.

Keywords: bioplastic films, water absorption, plasticizer, acid-base resistance, polyethylene.

Influence of chemical and physical treatment on the structure and processability of Kraft lignin for extrusion applications

K. Aslan;

Institute for Biopolymers and Sustainability, Hof University (Germany)

Lignin is a by-product of paper and pulp manufacturing. Due to its complex macromolecular structure the Kraft lignin, which accounts for approximately 85 % of the world's total lignin production, could not be used for extrusion. Until now, the Kraft lignin could only be added to a plastic in low concentrations as a filler. Within the research project "LigNutz" the aim of utilizing pure Kraft lignin types was pursued by developing a new and thermally stable lignin compound for extrusion. A detailed analysis of the used lignin types was carried out in relation to the characterization of the structure and the rheological and thermal properties. The modification of the Kraft lignin was carried out using physical and chemical methods. First of all it was investigated to modify the Kraft lignin by means of electron radiation with different radiation doses. Since there was no thermoplastic behavior after the subsequent assessment of the Kraft lignins, chemical modification methods were used. In parallel analysis of the Kraft lignin were carried out in order to be able to define the structure of the Kraft lignin, which served as the basis for the chemical modification. In the chemical modification, the Kraft lignin was treated with chemicals on a laboratory scale. A thermoplastic effect with different adjustable melting temperatures could be successfully created. The new, thermally stable Kraft lignin can thus be processed into compounds and films.

FDCA-based polymer materials beyond PEF

D. Zehm, A. Lieske;

**Fraunhofer Institute for Applied Polymer Research IAP, Potsdam-Golm
(Germany)**

The plastic news reporting about the bio-based building block 2,5-Furandicarboxylic acid (FDCA) are currently dominated by the new thermoplastic poly(ethylene 2,5-furandicarboxylate) (PEF). This is mainly because PEF exhibits superior gas barrier properties than the competing PET when processed as thin film [1], which provides a unique selling point and eases invest decisions for research devoted to enter the huge film and bottle market. However, the variety of polyester-based plastics is large and hence the potential of FDCA might be huge, but has not yet been fully discovered.

With our contribution, we want to shift the focus and share the results obtained during our studies on bio-based unsaturated polyester (UP resin), where we replaced the typically used phthalic- and isophthalic acid by FDCA. Three different commercially relevant petro-based polyester compositions were targeted (suitable for e.g. body parts, repair putties, SMC, relining, chemically resistant resins) and compared with the FDCA-based resins, to unveil the potential of FDCA for such materials. The lecture will show that the mechanical [2] and thermal properties [3] of the new FDCA-based resins are equal or even better than the petro-based resins, which enables FDCA producing companies to tap also into the composite market.

Literature

- [1] Burgess, S. K., Horst, T., Leisen, J. E., Kraftschik, B. E., Mubarak, C. R., Kriegel, R. M., Koros, W. J.: Chain mobility, thermal, and mechanical properties of poly(ethylene furanoate) compared to poly(ethylene terephthalate). *Macromolecules* 47 (2014) 1383–1391.
- [2] DIN EN ISO 527-1 (2012): Bestimmung der Zugeigenschaften – Teil 1: Allgemeine Grundsätze.
- [3] DIN EN ISO 75 (2013): Kunststoffe – Bestimmung der Wärmeformbeständigkeitstemperatur.

Naturbasierte Schäume als funktionalisierte adaptive Elemente

N. Eversmann, M. Wießner, A. Krombholz;
Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS,
Halle (Germany)

Herkömmliche Polymerschäume weisen während der Herstellung einen hohen Bedarf an Energie und Erdöl auf und sind in der Entsorgung problematisch. Ziel des Projekts ist es, einen funktionalisierten adaptiven Schaum auf Basis von lokal anbaubaren Pflanzenölen zu entwickeln. Hier können durch geeignete Pflanzenwahl und Anbaumethoden die langfristigen Folgen der derzeitigen Agrarkulturen abgeschwächt und beispielsweise Zufluchtsorte für Insekten geschaffen werden. Das Öl des Iberischen Drachenkopfs (*Lallemantia iberica*) eignet sich aufgrund des hohen Linolensäureanteils (50–68 %) gut als alternativer Rohstoff zur Herstellung von Polymerschäumen [1]. Im Gegensatz zu Lein ist der Drachenkopf als Bienenweide geeignet. Im Projekt wird Drachenkopföl epoxidiert und auf die Eignung für einen Einsatz in Schäumen geprüft (Bild 1). Bei der Auswahl der Härter und Additive wird strikt auf ihre Umweltverträglichkeit, auch hinsichtlich Herstellung und Transport, geachtet. Hierzu wird der CO₂-Fußabdruck ermittelt und eine LCA-Analyse durchgeführt. Die Fertigung der im Projekt geplanten Schäume kann zu den Drop-in-Verfahren gezählt werden, wobei der Anteil nachwachsender Rohstoffe bei 80–100 % liegen soll. Die Schäume können beispielsweise in Automobilindustrie, Containerbau, Maschinenbau oder Bauwesen als Abdichtung gegen Schlagregen, Lärm, Zugluft und Staub im Innen- und Außenbereich zum Einsatz kommen. Je nach verwendeten Prozessparametern bei der Herstellung der Schäume lassen sich die Elastizität mit verzögerter Dekomprimierung und die Dichte variieren sowie ein Dichtegradient erzeugen. Eine instrumentierte Schaumform soll die Einhaltung der Parameter sicherstellen.

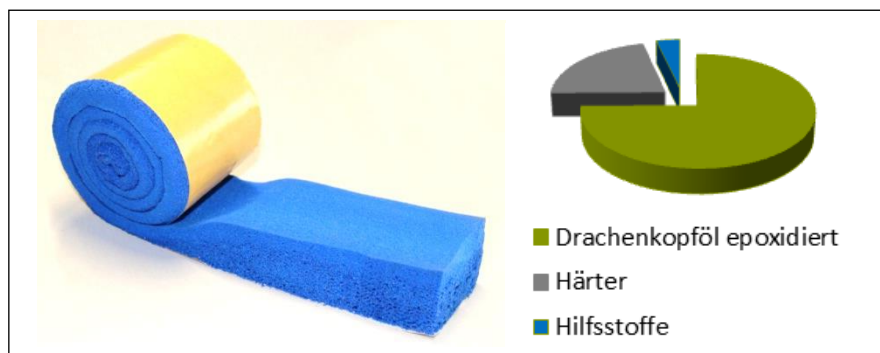


Bild 1: Viscoelastischer Gradientenschaum aus epoxidiertem Drachenkopföl und biogenen Härtern.

Literatur

- [1] Krist, S.: Lexikon der pflanzlichen Fette und Öle. 2. Auflage, Springer-Verlag, Berlin (2013).

S. Pokhrel, L. S. Rai, A. Sigdel, M. Slouf, J. Sirc, R. Lach, R. Adhikari

Fabrication and characterization of starch based bio-degradable polymer: Thermal and biodegradation properties

J. Giri, R. Lach, S. Henning, J.-M. Saiter, V. Katiyar, R. Adhikari

Reinforcement of degradable copolyester with wheat stalk microcrystalline cellulose

S. Zepnik

ALFATERXL® ECO – New ECO TPV from recycled and biobased raw materials

M. Seitz, V. Cepus, M. Klätte, D. Thamm

Chemisches Recycling von Kunststoffabfällen – Chance für eine bessere Kreislaufwirtschaft

M. Seier, S. Stanic, T. Koch, V. M. Archodoulaki

Herausforderungen im mechanischen Recycling von Einweg-Kunststoffen und Möglichkeiten neuer Konzepte

Fabrication and characterization of starch based biodegradable polymer: Thermal and biodegradation properties

S. Pokhrel^{1,2}, L. S. Rai², A. Sigdel¹, M. Slouf³, J. Sirc⁴, R. Lach⁵, R. Adhikari⁶;

¹ Department of Chemistry, Tri-Chandra M. Campus, Tribhuvan University, Kathmandu (Nepal), ² Nepal Polymer Institute (NPI), Kathmandu (Nepal),

³ Institute of Macromolecular Chemistry, Czech Academy of Sciences, Prague (Czech Republic), ⁴ Institute of Polymer Networks and Gels (Czech Republic),

⁵ Polymer Service GmbH Merseburg (PSM) (Germany), ⁶ Research Centre for Applied Science and Technology, Tribhuvan University, Kathmandu (Nepal)

Starch, a renewable and biodegradable polymer, is widely used material for manufacturing bioplastics. It can be prepared from different resources such as cereals (e. g. rice, wheat and maize) and the root vegetables (e.g. potatoes and cassava). In this work, starch was extracted from potato and mango seeds and characterized by different methods (such as by electron microscopy, see Fig 1). A series of starch based composites (PVA/starch and PBAT/starch) were fabricated by solution casting method with the use of glycerol as a plasticizer and citric acid as a crosslinking agent. The properties of extracted starch were evaluated by measuring ash content, moisture content and amylose content. The composites were characterized by electron microscopic, spectroscopic, microindentation techniques and thermal analysis. The FTIR and XRD results of starch confirmed the formation of starch. FTIR results proved the hydrogen bonding interactions in between filler and matrix. DSC analysis yielded glass transition temperature (T_g) and crystallinity. TGA results revealed that thermal stability decreased with the increase of filler content. The biodegradation test of composites (PVA/starch and PBAT/starch) was done under compost burial condition. Hence, the investigated starch based composites with synthetic polymers (PBAT and PVA) can be easily prepared *via* solution casting process with desired thermal and biodegradable properties.

Keywords: Biodegradation, starch, composite, electron microscopy, X-ray diffraction, spectroscopy.

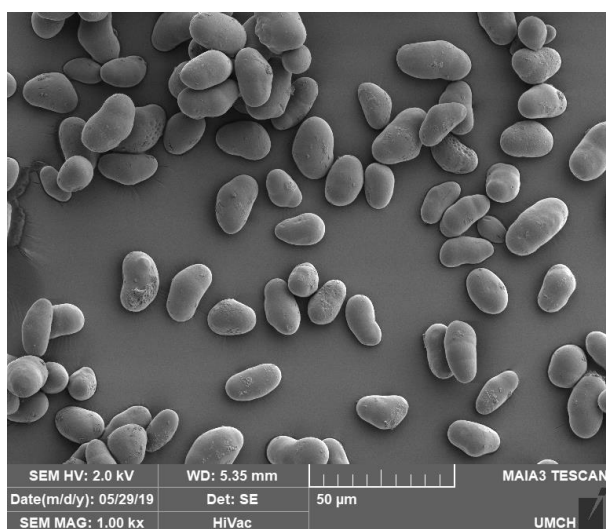


Figure 1: Scanning electron micrograph (SEM) of the potato starch powder.

Reinforcement of degradable copolyester with wheat stalk microcrystalline cellulose

J. Giri^{1,2}, R. Lach³, S. Henning⁴, J.-M. Saiter⁵, V. Katiyar⁶, R. Adhikari^{2,7};

¹ Tri-Chandra Multiple Campus, Tribhuvan University, Kathamandu (Nepal),

² Nepal Polymer Institute, Kathmandu (Nepal), ³ Polymer Service GmbH

Merseburg (PSM) (Germany), ⁴ Fraunhofer Institute for Microstructure of

Materials and Systems IMWS, Halle (Germany), ⁵ Université de Normandie

Rouen Laboratoire SMS Faculté des Sciences and Onyx Developpment,

Groupe-Nutriset, Rouen (France), ⁶ Chemical Engineering Department, Indian

Institute of Technology, Assam (India), ⁷ Research Central for Advance Science and Technology, Tribhuvan University, Kirtipur, Kathamandu (Nepal)

Refusal for degradation of synthetic polymeric materials has become one of the universal issue, which has cause severe land and water pollution throughout the world and led life in critical state. The issue can be solved by using degradable polymer. Therefore, in this work degradable polymer used instead of synthetic polymer and induced early degradation in polymer composites by incorporation of natural fibers Microcrystalline Cellulose (MCC) extracted from agricultural waste wheat stalk (WS) [1].

MCC were extracted from WS by thermo–chemical–mechanical process and compounded with commercially available biodegradable aliphatic–aromatic copolyester, the PBAT, in different proportions [2]. The resulting composites were characterized by various modern analytical techniques. The MCC obtained and polymer composites were analysed by different techniques such as Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Wettability, water absorption, soil composting and Gel Permission Chromatography (GPC). The degradation behavior was measured in term of amount of MCC in the composites and the period of composting. This ultimately concluded that higher amount of MCC and longer composting period in normal agricultural soil descends the molecular weight of the polymer in significant amount still the microscopic amount of polymeric fractions are still in soil.

Literature

- [1] Banerjee, A., Chatterjee, K., Madras, G.: Enzymatic degradation of polymer: A brief review. *Mater. Sci. Technol.* 30 (2014) 567–573.
- [2] Dhar, P., Gaur, S. S., Soundararajan, N., Gupta, A., Bhasney, S. M., Milli, M., Kumar, A., Katiyar, V.: Reactive extrusion of polylactic acid/cellulose nanocrystal films for food packaging applications: Influence of filler type on thermomechanical, rheological, and barrier properties. *Ind. Eng. Chem. Res.* 56 (2017) 4718–4735.

ALFATERXL® ECO – New ECO TPV from recycled and biobased raw materials

S. Zepnik;
ALBIS PLASTIC GmbH, Hamburg (Germany)

Today, circular economy and sustainable material solutions are global key strategies which are in focus not only by companies but also by governments. ALBIS PLASTIC GmbH also addresses these trends in its own global ECO product-strategy. This strategy includes the use of renewable resources as well as post-industrial raw materials for the production of high quality plastics. Brand names such as ALTECH® ECO have been successfully introduced to the market and are nowadays used in various technical applications, e.g. for automotive parts.

Within its ECO strategy, ALBIS PLASTIC has recently developed and commercially launched the ALFATERXL® ECO series, a thermoplastic vulcanizate (TPV). ALFATERXL® ECO is based on 100 % polypropylene (PP) recyclate and/or bio-based ethylene–propylene–diene monomer (EPDM) and produced via a reactive extrusion process (dynamic vulcanization). This contribution will give an overview about the raw material resources of the ALFATERXL® ECO series. General properties such as heat ageing behavior are presented (Figure 1). These properties are also compared to standard TPV materials. Furthermore, the rheology as well as particularities of the thermoplastic processing behavior of ALFATERXL® ECO are discussed. First applications will be presented as well, e.g. 2K molding and injection blow molding parts

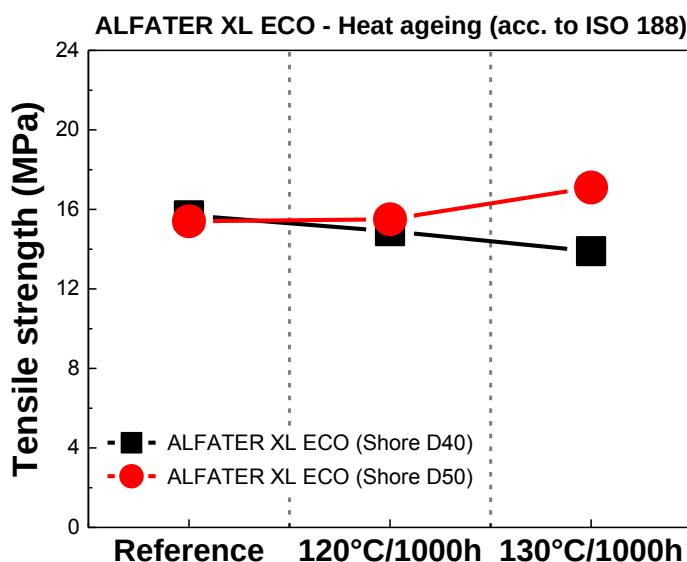


Figure 1: Tensile strength (ISO 37, type 2, 500 mm/min) of two different ALFATERXL® ECO grades before and after heat ageing at 120 °C/1000 h as well as 130 °C/1000 h (acc. to ISO 188).

Chemisches Recycling von Kunststoffabfällen – Chance für eine bessere Kreislaufwirtschaft

M. Seitz¹, V. Cepus¹, M. Klätte², D. Thamm¹;

¹ University of Applied Science Merseburg (Germany), ² Steinbeis R.T.M., Halle (Germany)

Eine möglichst effiziente und vollständige Kreislaufwirtschaft ist bei allen Rohstoffen, nicht zuletzt auch bei Kunststoffen, erklärtes Ziel. Bisher wird in Deutschland nur ein kleiner Teil des Kunststoffabfalls werkstofflich wiederverwertet.

Ziel einer Studie für die DBU war es, die Positionierung der Depolymerisation von Polyolefinen im Gesamtsystem des Kunststoffrecyclings zu betrachten [1]. Dazu wurde untersucht, welche Mengen und welche Qualitäten von Polyolefinabfällen für die Depolymerisation zur Verfügung stehen, sodass entsprechend der Abfallhierarchie keine Konkurrenzsituation zum werkstofflichen Recycling besteht, wie Pyrolyseprodukte hinsichtlich ihrer Marktfähigkeit als chemischer Rohstoff zu bewerten sind, welchen Entwicklungsstand Depolymerisationstechnologien aufweisen, wie sie bewertet werden können und welche Chancen und Risiken für die Depolymerisation von Polyolefinen bestehen und welche Rahmenbedingungen und Maßnahmen für Veränderungen bei Verwertungskaskaden und Kreislaufpfaden beim Kunststoffrecycling hinsichtlich der Depolymerisation notwendig wären.

Depolymerisationstechnologien können einen Beitrag zum Recycling liefern, weil sie eine stoffliche Wiederverwendung von verunreinigten Kunststoffabfällen als Kunststoffneuware ermöglichen. Sie sind damit keine Konkurrenz zum werkstofflichen Recycling, sondern eine Ergänzung. Nur durch das chemische Recycling kann aus verunreinigten Rezyklaten Kunststoff in Neuwarenqualität hergestellt werden. Eine Steigerung der stofflichen Wiederverwertung ist dadurch möglich. Der Erfolg und die Effizienz der Depolymerisationstechnologien hängt ebenfalls wie das werkstoffliche Recycling von der Qualität der Einsatzstoffe ab. Bestimmte Störstoffe wie PVC, PA und PET sollten im Einsatzstoff minimiert oder ganz vermieden werden, sodass nachfolgende Aufbereitungsschritte weniger aufwändig gestaltet werden müssen. Der gesamte Kunststoffkreislauf muss deshalb hinsichtlich Kunststoffdesign, Sammlung und Sortierung so gestaltet sein, dass möglichst reine Kunststofffraktionen für das werkstoffliche aber auch für das chemische Recycling wie der Depolymerisation zugänglich sind. Kann so mehr Masse im Stoffkreislauf gehalten werden, so können auch die Kosten für das Gesamtsystem verringert und klimafreundlichere Produkte hergestellt werden.

Literatur

- [1] Seitz, M., Cepus, V., Klätte, M., Thamm, D., Pohl, M.: Evaluierung unter Realbedingungen von thermisch-chemischen Depolymerisationstechnologien (Zersetzungsverfahren) zur Verwertung von Kunststoffabfällen. Steinbeis-Edition, Stuttgart (2020).

Herausforderungen im mechanischen Recycling von Einweg-Kunststoffen und Möglichkeiten neuer Konzepte

**M. Seier, S. Stanic, T. Koch, V. M. Archodoulaki;
Institut für Werkstoffwissenschaft und Werkstofftechnologie, TU Wien
(Österreich)**

In den letzten Jahren hat die Verwendung von Kunststoffen aufgrund ihrer verschiedenen Eigenschaften und Anwendungen erheblich zugenommen. Zu den weltweit wichtigsten Kunststoffen gehören Polypropylen (PP), Polyethylen (PE), Polyethylenterephthalat (PET) und Polystyrol (PS). Diese Polymere werden in verschiedenen Bereichen eingesetzt, beispielsweise in der Automobilindustrie, im medizinischen Bereich oder im Bauwesen und spielen heute eine wichtige Rolle in unserem Leben. Eine der größten Anwendungen für diese verschiedenen Polymere ist jedoch der Verpackungssektor, in dem die meisten Produkte eine sehr kurze "Lebensdauer" haben oder sogar nur als Einwegprodukte Anwendung finden. [1] Dies ist einer der Gründe, warum das Recycling von derartigen Verpackungen zu einem Problem geworden ist und sogar zu Verboten von Strohhalmen, Besteck, Tüten und anderen Kunststoffprodukten geführt hat. Insbesondere Verpackungen aus Mehrschichtfolien oder solche, die mehrere verschiedene Polymere enthalten, sind nur bedingt recyclingfähig und es wurden bisher keine industriellen Lösungen entwickelt, um dieser Tatsache entgegenzuwirken. [2]

In dieser Arbeit werden mögliche Re- und auch Upcycle-Ansätze mittels reaktiver Extrusion unter Verwendung von Peroxiden gezeigt. Auch die möglichen Potentiale verschiedener Polymermischungen in unterschiedlichen Konzentrationen (binäre bis quartäre Systeme), bestehend aus Polyolefinen, Polystyrol und Polyester, die bei unterschiedlichen Extrusionstemperaturen verarbeitet wurden, werden diskutiert. Weiters wird auch die Sinnhaftigkeit der Substitution einzelner Materialien oder Materialverbünde beleuchtet. Zur Charakterisierung wurden die mechanischen Eigenschaften wie E-Modul, Bruchdehnung und Schlagzugzähigkeit sowie die rheologischen Eigenschaften wie Schmelzverhalten und Schmelzsteifigkeit untersucht.

Literatur

- [1] Yin, S., Tuladhar, R., Shi, F., Shanks, R. A., Combe, M., Collister T.: Mechanical reprocessing of polyolefin waste: A review. Polym. Eng. Sci. 55 (2015) 2899–2909.
- [2] Kaiser, K., Schmid, M., Schlummer, M.: Recycling of polymer-based multilayer packaging: A review. Recycling 3 (2017) 3010001 (26 p.).

T. Schrank, M. Berer, E. Helfer, M. Feuchter, G. Pinter

Analysis of injection molded components of polyoxymethylene by X-ray scattering and diffraction

J. E. K. Schawe, E. Hempel

The influence of fillers and nucleating agents on polypropylene crystallization at high supercooling measured by Fast DSC

A. Monami

Prüfung und Überwachung von Bauprodukten für Gründächer

A. Flores, M. Gómez-Fatou, H. J. Salavagione

Nanoindentation in soft polymer-based composites using a cylindrical punch

J. Kucera, J. Sadilek, J. Poduska, P. Hutar

Real plastic deformation in load separation method used for PE-HD

J. Oberer, K. Schneider

Thermo-mechanical and structural analysis of neck initiation and failure in tensile tests of polyethylene terephthalate

Analysis of injection molded components of polyoxymethylene by X-ray scattering and diffraction

T. Schrank¹, M. Berer¹, E. Helfer¹, M. Feuchter², G. Pinter²;

¹ Polymer Competence Center Leoben GmbH (Austria), ² Chair of Materials Science and Testing of Polymers, Montanuniversität Leoben (Austria)

Polyoxymethylene (POM) is an engineering thermoplastic, which is widely used in structural applications that yield high requirements regarding the performance of the part. The mechanical properties of the final component are strongly dependent on the inner structure of the part, which results from the processing. X-ray measurement methods, such as small angle X-ray scattering (SAXS) and wide angle X-ray diffraction (WAXD), are powerful tools to examine the morphological features of polymers [1, 2].

This study is investigating POM homopolymer components, which were injection molded at different processing conditions, regarding their morphological features. A study on this material processed under moderate conditions was already done by [3]. Contrary, this study focuses on standard injection molding conditions and industrial-near injection molding conditions of selected specimen geometries. Morphological features will be discussed on the basis of SAXS and WAXD measurements. For SAXS, the volumetric degree of crystallinity, the lamellar thickness and the long period were determined by the so-called 1D-correlation. The mass degree of crystallinity was determined from the WAXD measurements by peak fitting. The orientation of the lamellae was characterized by analyzing the azimuthal plots of the SAXS patterns. For the quantification, the so-called ρ -parameter was used. Furthermore, the orientation of the crystal lattice planes was assessed from the azimuthal plots of the WAXD patterns by the determination of the Hermann's orientation function. Additionally, mechanical testing was carried out to rank the different processing conditions. The possibilities of X-ray measurements to examine the structure of POM and corresponding results will be focused in the discussion.

Literature

- [1] Alexander, L. E.: X-ray Diffraction Methods in Polymer Science. Wiley Series on the Science and Technology of Materials, Wiley-Interscience, New York (1969).
- [2] Stribeck, N.: X-Ray Scattering of Soft Matter. Springer Laboratory Series, Springer-Verlag, Berlin Heidelberg (2007).
- [3] Berer, M., Halb, M., Feuchter, M., Pacher, G., Pinter, G.: Fatigue fracture properties and morphology of polyoxymethylene (POM) plates produced under moderate processing conditions. Int. J. Polym. Sci. (2018) Article ID 7410925 (18 pages).

The influence of fillers and nucleating agents on polypropylene crystallization at high supercooling measured by Fast DSC

J. E. K. Schawe, E. Hempel;
Mettler-Toledo GmbH (Switzerland)

The crystallization process can be influenced by nucleating agents added to materials. These additives are classified as: 1) inactive nucleating agents, which have no significant influence on the crystallization kinetics, and 2) active nucleating agents that accelerate the crystallization process. Active nucleating agents can be further classified as particulate additives and additives that self-assemble the structure.

In this example, the nucleating activity of polypropylene (PP) was measured in a wide temperature range (between 0 °C and 130 °C) using Fast Differential Scanning Calorimetry (FDSC) [1]. The isothermal and non-isothermal crystallization kinetics were compared using different types of nucleating agents, including sorbitol-based nucleating agents, different carbon-nanotubes [2] (Figure 1), calcium carbonate [3] and nano-diamonds. All additives influenced the crystallization process of PP in a given temperature range (including calcium carbonate) but were not active outside this range. In addition, the measurements show that the isothermal crystallization process is very sensitive for the characterization of additives' nucleating activity.

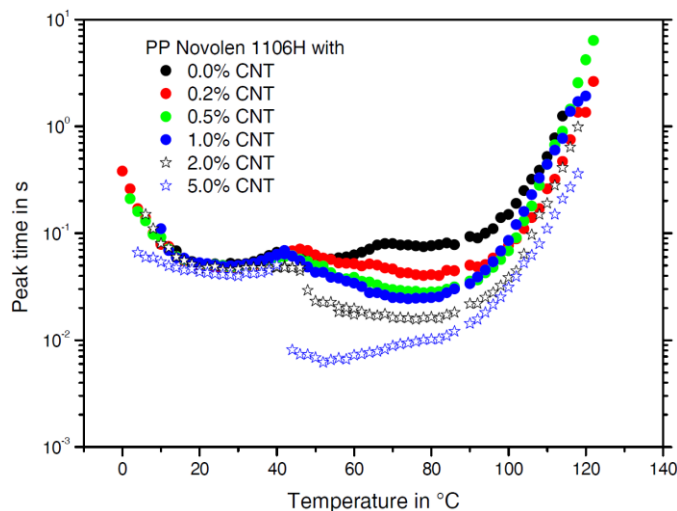


Figure 1: Peak time, t_p , versus the crystallization temperature.

Literature

- [1] Schawe, J. E. K., Budde, F., Alig, I.: Nucleation activity at high supercooling: Sorbitol-type nucleating agents in polypropylene. *Polymer* 153 (2018) 587–596.
- [2] Schawe, J. E. K., Pötschke, P., Alig, I.: Nucleation efficiency of fillers in polymer crystallization studied by fast scanning calorimetry: Carbon nanotubes in polypropylene. *Polymer* 116 (2017) 160–172.
- [3] Schawe, J. E. K., Budde, F., Alig, I.: A new crystallization process in polypropylene highly filled with calcium carbonate. *Colloid Polym. Sci.* 293 (2015) 1607–1614.

Prüfung und Überwachung von Bauprodukten für Gründächer

**A. Monami;
SKZ-Testing GmbH, Würzburg (Deutschland)**

Nanoindentation in soft polymer-based composites using a cylindrical punch

**A. Flores, M. Gómez-Fatou, H. J. Salavagione;
Institute for Science and Technology of Polymers (ICTP-CSIC), Spanish
Research Council, Madrid (Spain)**

Instrumented indentation, or depth-sensing indentation, has become nowadays a fundamental tool for mechanical characterization of materials. For polymer-based composites, it has been shown that the technique can offer a comprehensive understanding of the mechanical changes upon filler incorporation including modulus, hardness and creep enhancement [1]. The case of soft materials is especially challenging mainly due to the difficulty in the detection of the initial contact [1].

The present work addresses the determination of the mechanical properties of a series of flexible conductive elastomers based on poly(styrene-*b*-ethylene-co-butylene-*b*-styrene) (SEBS) and small quantities of graphene (< 2 vol.%). Instrumented indentation with a cylindrical punch is used to evaluate the mechanical enhancement upon filler loading and results are related to structural and morphological changes. SEBS/graphene nanocomposites serve as a model to compare a number of approaches employed to analyse indentation data: assuming pure elastic response during quasi-static loading (i) and unloading (ii); and (iii) by dynamic indentation.

It is found that the three approaches deliver modulus values, E , consistent with tensile testing results [2]. The loading method yields lower E values than the other two approaches and this can be explained as due to a small misalignment of the indenter. In all cases, an initial modulus drop is detected with the incorporation of small amounts of graphene and this can be associated with a distortion of the packing of polystyrene cylinders in the polyethylene matrix. Further addition of graphene up to ≈ 2 vol.% counterbalances the initial E -drop and the SEBS values are eventually restored or even exceeded.

In summary, indentation with a cylindrical punch appears as a convenient strategy to study the mechanical properties of compliant materials and modulus values are found to be consistent with tensile testing analysis.

Financial support from MINECO (Ministerio de Economía y Competitividad), Spain, under grant MAT2017-88382-P is gratefully acknowledged.

Literature

- [1] Díez-Pascual, A. M., Gomez-Fatou, M. A., Ania, F., Flores, A.: Nanoindentation in polymer nanocomposites. *Prog. Mat. Sci.* 67 (2015) 1–94.
- [2] Ania, F., Gomez-Fatou, M. A., Salavagione, H. J., Enrique-Jimenez, P., Quiles-Diaz, S., Flores, A.: Creep behaviour of elastomeric nanocomposites by flat punch indentation: Influence of graphene modification and content. *Comp. Sci. Tech.* 198 (2020) 108311.

Real plastic deformation in load separation method used for PE-HD

J. Kucera¹, J. Sadilek¹, J. Poduska², P. Hutar²;

¹ UNIPETROL RPA – Polymer Institute Brno (Czech Republic), ² Institute of Physics of Materials, AS CR, Brno (Czech Republic)

A new method for evaluating of toughness of ductile polymers, called “load separation method”, has been developed for more over a decade, especially by the group of Prof. F. Baldi, Università degli Studi di Brescia. This method is applied for materials that exhibit stable crack growth during loading of a test specimen. It is based on comparison of compliances of a test specimen with a sharp notch (SN), which exhibits usually a combination of plastic deformation under the notch root and growth of a crack, and an equivalent test specimen with a blunt notch (BN), which usually exhibits only some plastic deformation under the blunt notch root. This conception is used for description of the toughness evaluating the mechanics of elastic–plastic material behavior.

The presented lecture describes results of 3-point bending tests, where Single Edge Notch Bend specimens with SN and BN were applied. The aim was to evaluate the real plastic deformation portion of the whole deformation of each test specimen. For this evaluation, the technique of creep-recovery test following immediately after the bending test was used.

Two high-density polyethylene (HD-PE) pipe grades were selected for testing: monomodal PE80 and bimodal PE100. These polymers are extremely tough, but they differ in stiffness and rheological properties.

Three types of tests were applied: 1) multistep bending with one specimen, 2) tests of single specimen bent to different depths and 3) a combination of these test modes to compare the crack growth under these different conditions.

The standard load–displacement curves were measured, and the plastic separation method was applied to compare the toughness of the two HD-PE grades. Moreover, the crack extensions of the crack growth on the fracture surfaces of the multiple bend specimen and several single bend specimens were measured by the use of a light microscope in the same manner as for the standard J_R evaluation for stable cracks. The main result of this comparison is that the growth of the crack in the multiple bend test specimen is comparable with that in one of the cracks in the single bend test specimens.

It was found that the “real plastic deflection” (Fig. 1) differs significantly from the total deflection having been reduced by the purely elastic part. Thus, the dependences and/or their mutual ratio for SN and BN specimens are completely different from the standard ones.

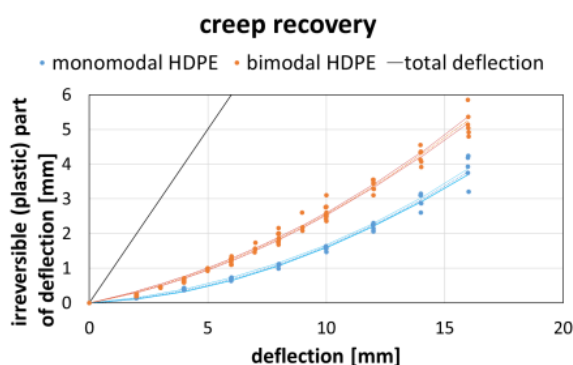


Figure 1: Real plastic deflection vs. total deflection for two PE-HD grades.

Thermo-mechanical and structural analysis of neck initiation and failure in tensile tests of polyethylene terephthalate

J. Oberer¹, K. Schneider²;

¹ Technische Universität Dresden (Germany), ² Leibniz Institute of Polymer Research Dresden (Germany)

The neck formation during tensile tests of amorphous polyethylene terephthalate is observed by IR thermography. The neck formation is found to be highly dependent on the amount of stored elastic energy before yielding and necking can be classified as stable neck formation, followed by neck propagation, unstable neck formation, with cracks at the sample flanks and followed by failure of the sample and spontaneous fracture. The thermographic observation of neck formation shows a major temperature rise to glass transition temperature and above due to dissipation of the unloaded elastic energy of the whole specimen in the necking zone. There are limits of stored elastic energy for stable and unstable neck formation, as well as for spontaneous fracture. Via wide angle X-ray scattering, morphologic changes in the necking zone were studied. For the stable and unstable neck formation, strain-induced crystallization with high orientation was found. At the edges of samples with spontaneous fracture, random crystallization was found.

K. Roetsch, T. Horst

Mikrostrukturbasierte Festigkeitsbestimmung von endlosfaserverstärkten Duromeren

M. John, S. Celevicz, I. Jahn, R. Schlimper

Challenges in evaluating carbon fiber reinforced thermoplastic tapes

S. Celevics, S. Richter, I. Jahn, F. Nagel, J. Celevics, S. Pohle

Improvement of natural fiber impregnation for biobased sustainable unidirectional tapes

T. Munkelt, J. Palomino, T. Hedtke, J. Martins de Souza e Silva, C. Schmelzer

Vom Samen zur Nanofaser – Gewinnung und Verarbeitung pflanzlicher Proteine zu Nanofaservliesen

N. L. Bhandari, B. Dhungana, S. Henning, R. Lach, R. Adhikari

Structural, thermal and mechanical properties of urea–formaldehyde (UF)-natural fiber composites

M. E. Frerking, A. Rouyer, M. Butto, J. Bovi, M. Melaj, C. Bernal

On the performance of biodegradable self-reinforced composites (SRCs) based on poly(lactic acid) (PLA)

U. Staudinger, L. Jakisch, B. K. Satapathy

Development of block copolymer/carbon nanotube composites: Effects of nanofiller modification on structure, electrical and mechanical properties

K. N. Dhakal, R. Lach, W. Grellmann, A. Das, H. H. Le, S. Wießner, B. Krause, J. Pionteck, R. Adhikari

Strain sensing behavior of piezoresistive flexible copolyester/carbon based filler composites

J. Giri, S. Pokhrel, S. Henning, R. Lach, W. Grellmann, V. Katiyar, R. Adhikari

Compostable polymer composites based on copolyesters and natural fibers

Mikrostrukturbasierte Festigkeitsbestimmung von endlosfaserverstärkten Duromeren

K. Roetsch, T. Horst;

Westfälische Hochschule Zwickau, Fakultät Automobil- und Maschinenbau,
Institut für Produktionstechnik (Deutschland)

Bei Faserkunststoffverbunden (FKV) handelt es sich um Verbundwerkstoffe aus Verstärkungsfasern, welche in eine Polymermatrix eingebettet sind. Durch maßgeschneiderte Kombination von Faser und Matrix sind FKV aufgrund sehr hoher spezifischer Festigkeiten als Leichtbauwerkstoff anderen Werkstoffgruppen überlegen. Besonders in Bereichen des extremen Leichtbaus werden Konzepte benötigt mit deren Hilfe die weitere Optimierung eines Verbundes hinsichtlich hoher Festigkeit bei gleichzeitig geringem Gewicht möglich ist.

Mit dem Ziel, die Festigkeit eines FKV unter allgemeinen mehrachsigen Belastungen zu berechnen, wurde ein mikromechanischer Ansatz basierend auf Homogenisierungsmethoden und ihrer Umsetzung mittels der Finite-Elemente Methode (FEM) erarbeitet. Die Belastung eines repräsentativen Volumenelements (RVE) (siehe Bild 1) erlaubt die Berechnung der dadurch im RVE verursachten lokalen Beanspruchungen und somit die Beurteilung von kritischen Beanspruchungen der einzelnen Verbundkomponenten, die zu einem Versagen der Faser (Faserbruch) bzw. der Matrix (Faserzwischenbruch) führen.

Am Beispiel unidirektional verstärkter FKV mit idealer und stochastischer Verteilung der Fasern im Querschnitt werden, unterschiedliche Versagenskriterien hinsichtlich ihrer Eignung zur Vorhersage der Festigkeit des Verbundes analysiert und diskutiert.

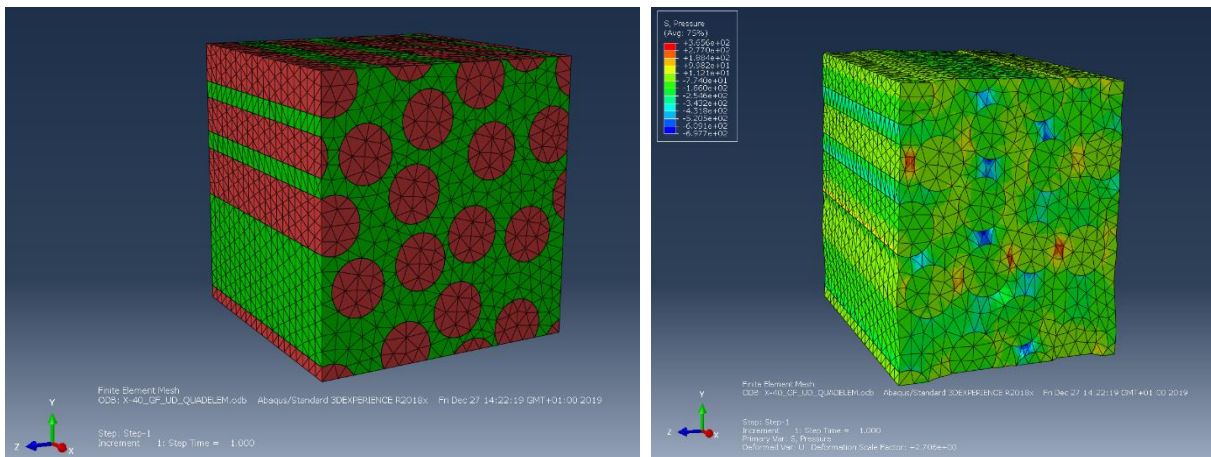


Bild 1: Unverzerrtes RVE (links); verzerrtes RVE mit farblicher Darstellung der Spannungsverteilung in Form des hydrostatischen Anteils des Spannungstensors (rechts).

Challenges in evaluating carbon fiber reinforced thermoplastic tapes

M. John¹, S. Celevicz², I. Jahn², R. Schlimper¹;

¹ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ² Fraunhofer Pilot Plant Center for Polymer Synthesis and Processing PAZ, Schkopau (Germany)

Continuous fiber reinforced thermoplastics are currently facing a broad market launch in the automotive industry, among others [1, 2], see Fig. 1. Research in recent years has so far dealt with individual stages in this process chain. Approaches for a reproducibly high component quality therefore pursue the optimization of individual process stages within this process chain [3, 4]. However, there is no industrial standard for the quality assessment of thermoplastic tapes established. Although a large number of standards are available for evaluating continuous fiber-reinforced composites, most of them are not fully suitable for evaluating thermoplastic-based tapes (TPC) [5]. The standard DIN 65 469 [6] for example was developed in the year 1992 but has been withdrawn. The reason is that they are originally developed and designed on the basis of laminates and continuous fiber-reinforced semi-finished products with a thermosetting matrix.

In particular, when evaluating carbon fiber reinforced thermoplastic tapes, there are some special features to consider. In this article, the methods for determining the fiber volume content (FVC) are evaluated on the one hand, and the determination of tensile stiffness and strength on tape strips, on the other hand. This was done on carbon fiber reinforced tapes with a PA6 matrix. To determine the FVC, the ashing of the matrix (using thermogravimetric analysis) and the solution of the matrix in a wet chemical process were compared. The results from the determination of the fiber volume content were correlated with the determined E-moduli from the tensile tests. When carrying out the tensile tests, some parameters had to be changed compared to the existing standard. In further steps it is examined which influence of tape discontinuities have any influence on the following processing steps. And what relevance, what parameters have in the subsequent processing to the organosheet or as an insert in an injection molding component.

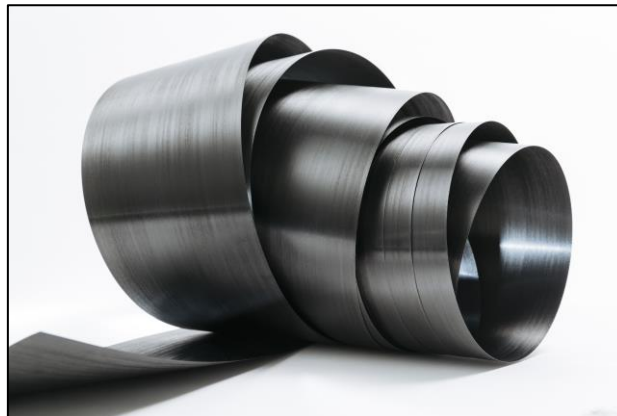


Figure 1: Continuous carbon fiber reinforced thermoplastic tape.

Literature

- [1] Zscheyge, M., Michel, P., Jahn, I.: Bauteile in thermoplastischer UD Endlosfaserverstärkung für die Automobilindustrie. 4. Leichtbau Tagung der Fraunhofer Allianz Leichtbau, Oberhausen(2015).
- [2] Stock, A., Egger, P.: Organobleche verlassen Hochpreissegment. MM Composite World (2011) 12–15.
- [3] Amir Khan, M., Mitschang, P., Schledjewski, R.: Tracing the void content development and identification of its effecting parameters during in situ consolidation of thermoplastic tape material. Polym. Polym. Compos. 18 (2018) 1–15.
- [4] Hofbauer, D., Schramm, N., Kroll, L., Kaufmann, J.: Herstellung endlosfaserverstärkter, thermoplastischer Halbzeuge für Karosseriestrukturbauteile in Großserie. Technologies for Lightweight Structures (2017) 14–23.
- [5] Grellmann, W., Seidler, S. (Eds.): Polymer Testing. 2nd ed., Carl Hanser Verlag, München (2013).
- [6] DIN 65469 (1992-08): Luft- und Raumfahrt – Faserverstärkte Kunststoffe – Zugversuch an einlagigen Zugflachprobekörpern.

Improvement of natural fiber impregnation for biobased sustainable unidirectional tapes

**S. Celevics, S. Richter, I. Jahn, F. Nagel, J. Celevics, S. Pohle;
Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle
(Germany)**

Sustainable and ecologically friendly materials as well as a goal-oriented reduction of CO₂ emissions are focused more than ever in the industry and in society. To challenge the high standards in load bearing or structural parts in light weight applications, where glass fiber (GFRP) or carbon fiber reinforced plastics (CFRP) are dominant, an appropriate performance level of the biobased materials has to be provided. An approach is to utilize continuous fiber reinforced composite materials with unidirectional fiber orientation (UD tapes) [1].

Eligible bio-based textiles for such high-quality applications can be bast fibers like flax or hemp, which can compete with glass fibers in terms of mechanical strength and modulus under optimized processing conditions [2]. Nevertheless, the impregnation of bast fibers with thermoplastic polymers in continuous processes is challenging because of many factors, mainly based on the inherent limitations of fiber and thermoplastic nature. Such factors can be the melt viscosity of thermoplastics, necessary retention times, drying regimes of the fibers and compatibility of matrix to fiber.

In the present work the fiber matrix interface compatibilization is investigated in a continuous film stacking process utilizing wet and dry surface modification mechanisms. Therefore, drafted flax slivers are exposed to atmospheric pressure plasma with air as process gas and additionally containing different coupling agents respectively and are further compressed to composites in a double belt press using biopolymer films. The influence of the surface treatments of the fibers on the water uptake, the surface wettability of the fiber tows (e.g. Fig. 1) and polymer–fiber interface are shown.



Figure 1: Wetting behavior of untreated flax slivers (left), plasma treatment (middle, right).

Literature

- [1] Zschehyge, M., Michel, P., Jahn, I.: Bauteile in thermoplastischer UD Endlosfaserverstärkung für die Automobilindustrie. Oberhausen (2015).
- [2] Joffe, R., Andersons, J., Wallström, L.: Strength and adhesion characteristics of elementary flax fibres with different surface treatments. Compos. Part A Appl. Sci. Manuf. 34 (2003) 603–612.

Vom Samen zur Nanofaser – Gewinnung und Verarbeitung pflanzlicher Proteine zu Nanofaservliesen

T. Munkelt¹, J. Palomino¹, T. Hedtke², J. Martins de Souza e Silva², C. Schmelzer²;

¹ Pilot Pflanzenöltechnologie Magdeburg e.V., Magdeburg (Germany),

² Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS, Halle (Germany)

Pflanzliche Proteine sind biologische Polymere und verfügen über ein enormes Potenzial für unterschiedlichste Anwendungen in technischen, life-science, in food- und feed-Bereichen. Neben der Funktionalisierbarkeit für bestimmte Anwendungen, besitzen pflanzliche Proteine u.a. auf natürlichem Wege potenziell nutzbare antimikrobielle Eigenschaften. Peptide der Proteine von Raps (*Brassica napus*), Kidneybohne (*Phaseolus vulgaris*) und Berglinsen (*Lens culinaris*) weisen interessante antimikrobielle Eigenschaften für Medizin- und Filtertechnik auf [1]. Daher wurden die drei Rohstoffe zur Untersuchung einer möglichen Herstellung von Nanofasern mittels des Elektrospinnverfahrens ausgewählt.

Zunächst wurden die Bedingungen für die nasschemische Extraktion zur Herstellung von reinen Proteinen der drei Rohstoffquellen durch Variation, z.B. von pH-Wert und Temperatur, optimiert.

Beim Elektrospinnverfahren werden die Proteinlösungen oder Suspensionen verwendet, an die bestimmte Anforderungen gestellt werden. Um diese zu erfüllen und einen möglichst hohen Proteinanteil im Vlies zu erreichen, wurden verschiedene organische Lösungsmittel sowie biologisch abbaubare Komposite untersucht. Mit dem Lösungsmittelsystem aus Ameisen- und Essigsäure sowie Polylactidsäure als stabilisierendes Kompositmaterial sind erste Nanovliese gewonnen worden. Die Charakterisierung erfolgte mittels Rasterelektronenmikroskopie und Röntgen-Computertomographie. Der Pflanzenproteinanteil in den Vliesen konnte durch eine Modifizierung auf über 80 % gesteigert werden. Dabei weisen die Vliese unterschiedliche mechanische Eigenschaften auf, die aktuell charakterisiert werden.

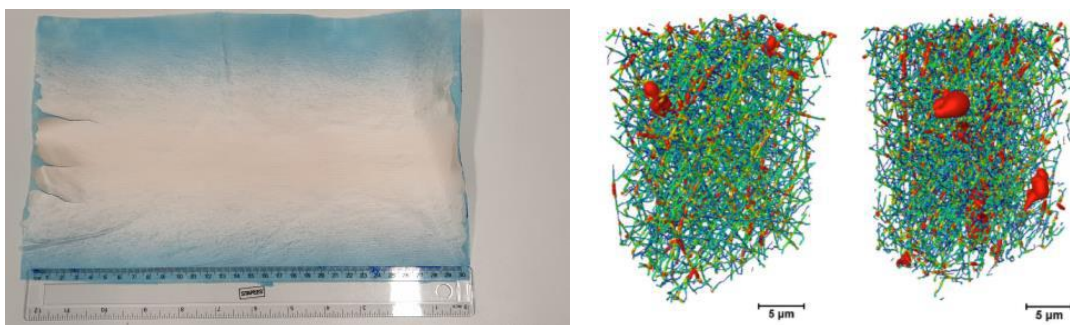


Bild 1: Ein Vlies aus Berglinsenproteinhydrolysat (links, Lineal: 30 cm); eine 3D-Mikrostrukturanalyse eines Proteinvlieses (rechts, Maßstab: 5 µm).

Literatur

- [1] Salas, C. E., Badillo-Corona, J. A., Ramírez-Sotelo, G., Oliver-Salvado, C.: Biologically active and antimicrobial peptides from plants. Biomed. Res. Int. Volume 2015 (2015) Article ID 102129 (11 p.).

Structural, thermal and mechanical properties of urea-formaldehyde (UF)-natural fiber composites

N. L. Bhandari¹, B. Dhungana¹, S. Henning², R. Lach³, R. Adhikari⁴;

¹ Department of Chemistry, Tri-Chandra Multiple Campus, Tribhuvan University, Kathmandu (Nepal), ² Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ³ Polymer Service GmbH Merseburg (Germany), ⁴ Research Center for Applied Science and Technology (RECAST), Tribhuvan University, Kathmandu (Nepal)

The effect of natural fiber surface modification and process variation was studied on different composites (0 %, 15 %, 30 %, 45 % and 60 %) of urea-formaldehyde resin (UF-resin) with allo (*Girardinia diversifolia*) fibers, prepared without using compatibilizers. Composites were set by cross polymerization followed by curing at 60 °C for 7 days [1, 2]. The composites were compared with cotton (*Gossypium arboreum*) fibers composites prepared with same matrix under similar conditions. This study intend to observe the morphology and structure changes of allo fibers after the chemical modification and study the effect of different fibers loading on morphology, thermal, mechanical, and water absorption behaviors of urea formaldehyde composites. The composite materials were characterized by Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), thermogravimetric analysis (TGA), compressive tests, micro indentation hardness tests and water absorption tests [3, 4]. Results showed that treated fibers are found to be more compatible with matrix in composites than neat fibers and effects are distinct on their morphology, mechanical, thermal and water absorption behaviours. The composites are found to be compatible even when prepared without compatibilizers [2, 4].

Keywords: Urea-formaldehyde, polymer composites, compatibilizers, melt mixing.

Literature

- [1] Christjanson, P., Pehk, T., Siimer, K.: Structure formation in urea formaldehyde resin synthesis. Proc. Estonian Acad. Sci. Chem. 55 (2006) 212–225.
- [2] Chiang, T. C., Hamdan, S., Osman, M. S.: Urea formaldehyde composites reinforced with sago fibers: Analysis by FTIR, TGA and DSC. Adv. Mater. Sci. Eng. 2016 (2015) 1–10.
- [3] Zorba, T., Papadopoulou, E., Hatjiissaak, A., Paraskevopoulos, K. M., Chrissafis, K.: Urea-formaldehyde resins characterized by thermal analysis and FTIR method. J. Therm. Anal. Calorim. 92 (2008) 29–33.
- [4] Bhandari, N. L., Dhungana, B. R., Lach, R., Henning, S., Adhikari, R.: Synthesis and characterization of urea-formaldehyde eco-friendly composites based on natural fibers. Journal of Institute of Science and Technology 24 (2019) 19–25.

On the performance of biodegradable self-reinforced composites (SRCs) based on poly(lactic acid) (PLA)

**M. E. Frerking, A. Rouyer, M. Butto, J. Bovi, M. Melaj, C. Bernal;
Instituto de Tecnología en Polímeros y Nanotecnología (ITPN), Facultad de
Ingeniería, Universidad de Buenos Aires (Argentina)**

The growing concern of society on the limited availability of fossil fuel resources has aroused much interest in bio-based materials. At the same time, the extensive use and non-biodegradable character of traditional plastics derived from fossil fuels leading to significant waste accumulation have triggered extensive research on biodegradable polymers which under appropriate conditions will break down far more quickly. Among them, poly(lactic acid) (PLA) is one of the best candidates as a result of its good combination of mechanical properties, renewable origin, biodegradability in compost and relatively low cost. Thus, PLA is an excellent substitute to commodity polymers in different applications such as packaging, textiles, bottles, bags and automotive parts. However, its relatively brittle nature and low thermal stability still limit its use in a great number of applications. Hence, PLA is commonly combined with other polymers, different types of particles or fibres. On the other side, natural fibre-reinforced polymer composites have attracted significant interest in the last decades due to their outstanding environmental-friendly characteristics such as non-toxicity, low density, biodegradability and cheap production cost, being considered as good alternatives to fossil fuel-based polymers. Among natural fibres, the refined form of wood pulp: microcrystalline cellulose (MCC), appears as an appealing reinforcing filler for polymers due to its remarkable properties such as fire resistance, anti-abrasiveness, and biocompatibility [1]. In addition, self-reinforced composites (SRCs) or all-polymer composites have been developed to overcome the difficulty of traditional composites to be recycled. In SRCs, the reinforcement is composed of highly oriented strong fibres or tapes, while the matrix is a polymer of the same chemical nature but with lower melting or softening point. In this way, fully recyclable light composites having excellent adhesion between phases can be obtained. Extensive literature exists regarding the preparation and characterization of self-reinforced composites based on synthetic polymers as well as on bio-based ones [2–5]. Self-reinforced composites compete with traditional composites in many applications depending on their performance/cost ratio. In the present work, biodegradable self-reinforced composites based in a PLA matrix reinforced with high strength PLA fibres were obtained by film-stacking followed by compression molding. PLA was also compounded in an intensive mixer with different contents of microcrystalline cellulose fibres to be used in the form of compression moulded films as the matrix of the SRCs. The morphology-processing-properties relationship of the different developed composites was investigated. The effect of the self-reinforced composites consolidation temperature was also studied. The morphology of the matrix films was analyzed by scanning electron microscopy (SEM) on cryo-fractured surfaces obtained at liquid nitrogen temperature. While the PLA film exhibited a smooth fracture surface, matrix films containing MCC presented increased roughness with significant evidence of cellulose fibres aggregation. In addition, some gaps between the aggregates and the matrix were clearly observed suggesting a weak interaction between the hydrophilic MCC and

the hydrophobic PLA. Differential scanning calorimetry (DSC) analysis indicated that MCC acted as nucleating agent for PLA crystallisation and that, irrespective of the SRCs consolidation temperature, the PLA fibres maintained their structure after moulding. Uniaxial tensile tests were also carried out for both the matrix films and the self-reinforced composites. The results for the matrix films indicated that the incorporation of MCC was detrimental to tensile strength, ductility and toughness, whereas stiffness values remained practically invariable. These results can be attributed to the above mentioned incompatibility between MCC and PLA and the subsequent fibres aggregation. In the SRCs, the increase in the consolidation temperature led to improvements in most of the tensile properties, especially in toughness, while the addition of MCC in the matrix films had a negative effect on these properties. Finally, disintegration tests in composting conditions at 58 °C were carried out for the different SRCs. These tests showed that the material with the highest content of MCC (20 wt.%) was visibly disintegrated after 30 days, while the other composites did not present significant morphological changes after the same burial time. Further work is still needed to obtain SRCs based on PLA which exhibit simultaneous improvements in tensile properties and disintegration ability with respect to neat PLA.

Literature

- [1] Bhasney S. M., Mondal K., Kumar A., Katiyar V.: Effect of microcrystalline cellulose (MCC) fibres on the morphological and crystalline behaviour of high density polyethylene (HDPE)/polylactic acid (PLA) blends. *Compos. Sci. Technol.* 187 (2020) 107941.
- [2] Karger-Kocsis, J., Bárány, T.: Feature article single-polymer composites (SPCs): Status and future trends. *Compos. Sci. Technol.* 92 (2014) 77–94.
- [3] Gao, C., Yua, L., Liu, H., Chen, L.: Development of self-reinforced polymer composites. *Prog. Polym. Sci.* 37 (2012) 767–780.
- [4] Kmetty, A., Barany, T., Karger-Kocsis, J.: Self-reinforced polymeric materials: A review. *Prog. Polym. Sci.* 35 (2010) 1288–1310.
- [5] Mukhopadhyay, S., Adak, B.: *Single-Polymer Composites*. CRC Press, Taylor & Francis Group, Boca Raton (2019).

Development of block copolymer/carbon nanotube composites: Effects of nanofiller modification on structure, electrical and mechanical properties

U. Staudinger¹, L. Jakisch¹, B. K. Satapathy²;

¹Leibniz-Institut für Polymerforschung Dresden e.V. (Germany), ² Indian Institute of Technology Delhi, Department of Materials Science and Engineering, New Delhi (India)

Due to their structural characteristics and interesting designable property profile block copolymers (BCPs) offer high potential as template matrices for controlled and phase-selective incorporation of nano-scaled particles, as the nanofiller dimensions are in the range of the domain sizes of the block copolymers. Via selective incorporation of carbon nanotubes (CNTs) it is aimed to improve the mechanical properties of the BCPs and/or to introduce electrical conductivity, while significantly reducing not only the extent of filler incorporation but also the material costs compared to homogeneous filler dispersion within a polymer matrix. The most significant challenge of using CNTs as nanofiller in BCPs is ensuring their sufficient dispersion and uniform distribution in the polymer matrix in spite of their high aspect ratio (length in micrometer scale) and intrinsic tendency to agglomerate. In this work the dispersion and localization behavior of low contents of multi-walled carbon nanotubes (MWCNTs) in styrene–butadiene based block copolymers were studied. MWCNTs were chemically modified with functional groups to enhance the compatibility with the BCP phases [1]. In addition significantly shortened MWCNTs were used to ensure more favorable size ratios between nanotubes and block copolymer phases in order to increase the possibility of selective localization of the CNTs in one of the BCP phases. The BCP/MWCNT nanocomposites were processed by melt mixing and solution casting. The influence of CNT dispersion and processing conditions on the nanocomposite structure, the electrical and mechanical properties were discussed [2, 3].

Literature

- [1] Staudinger, U., Jakisch, L., Hilbig, L.: Dispersion and localization behavior of modified MWCNTs in immiscible polymer blends of polystyrene and polybutadiene and in corresponding nanostructured block copolymers. *J. Comp. Sci.* 4 (2020) 40.
- [2] Staudinger, U., Satapathy, B. K., Jehnichen, D.: Nanofiller dispersion, morphology, mechanical behavior and electrical properties of nanostructured styrene–butadiene based triblock copolymer/CNT composites. *Polymers* 11 (2019) 1831.
- [3] Staudinger, U., Satapathy, B. K.: Influence of carbon nanotubes on the morphology and properties of styrene–butadiene based block copolymers. *Macromol. Symp.* 373 (2017) 1700030.

Strain sensing behavior of piezoresistive flexible copolyester/carbon based filler composites

K. N. Dhakal^{1,2,3}, R. Lach⁴, W. Grellmann⁴, A. Das¹, H. H. Le¹, S. Wießner¹,
B. Krause¹, J. Pionteck¹, R. Adhikari^{2,3};

¹ Leibniz-Institut für Polymerforschung Dresden e.V. (IPF) (Germany), ² Central Department of Chemistry, Tribhuvan University, Kirtipur, Kathmandu (Nepal),

³ Nepal Polymer Institute (NPI), Kathmandu (Nepal), ⁴ Polymer Service GmbH Merseburg (PSM) (Germany)

Electrical conductive composites of poly(butylene adipate-*co*-terephthalate) (PBAT), an elastomeric aromatic-aliphatic copolyester, with multiwalled carbon nanotubes (MWCNTs) and their hybrids with carbon black (CB) in different proportions were prepared by melt mixing followed by compression molding. Electrical percolation threshold below 1 wt.-% of fillers was achieved depending up on the filler types and proportion in hybrids.

Tensile tests with simultaneous measurement of electrical resistance of the conductive composites were carried out. Exponential increase of relative resistance change ($\Delta R/R_0$) with applied mechanical strain confirmed the piezoresistive properties of the composites. Furthermore, cyclic experiments of these composites with increasing tensile strain (up to 10 %) were performed. Relative resistance change ($\Delta R/R_0$) was found irreproducible with mechanical strain up to 0.5 %, while it was found to be dependent and the most reproducible from 2 to 5 % strain. However, strain dependent change and reproducibility of $\Delta R/R_0$ was found to level off above 8 % strain which is attributed to the irreversible breaking of the conductive filler network due to plastic deformation of polymer matrix. Hence, PBAT/nanocarbons conducting composites along with their high mechanical flexibility demonstrated the potential of applications for low strain sensing purposes.

Keywords: flexibility, polymer composites, electrical conductivity, piezoresistivity, strain sensing.

Compostable polymer composites based on copolyesters and natural fibers

J. Giri^{1,2}, S. Pokhrel^{1,2}, S. Henning³, R. Lach⁴, W. Grellmann⁴, V. Katiyar⁵,
R. Adhikari^{2,6};

¹ Department of Chemistry, Tri-Chandra Multiple Campus, Tribhuvan University, Kathmandu (Nepal), ² Nepal Polymer Institute (NPI), Kathmandu (Nepal), ³ Fraunhofer Institute for Microstructure of Materials and Systems, Halle (Germany), ⁴ Polymer Service GmbH Merseburg (PSM) (Germany),

⁵ Department of Chemical Engineering, Indian Institute of Technology Guwahati, Assam (India), ⁶ Research Centre for Applied Science and Technology, Tribhuvan University, Kathmandu (Nepal)

The structure–properties correlations of composites based on commercial biodegradable copolyester and comprising microcrystalline cellulose (MCC) and chitosan (CS) as fillers were investigated with an emphasis on effect of reinforcing filler on their mechanical behaviour. The materials were characterized by Fourier transform infrared (FTIR) spectroscopy, electron microscopy, tensile as well as microindentation tests.

The morphology of polymer composites studied by scanning electron microscopy (SEM) revealed the incompatibility of the natural fibers. Both MCC and CS were found to macroscopically segregate from the matrix. The CS exhibited elongated, much incompatible morphology compared to more homogeneous and compatibilized structures of the composites with MCC. In both cases, it was demonstrated that MCC and CS could be easily blended up to a pretty high weight fraction into the copolyester matrix even without use of compatibilizers. The polymer composites underwent rapid fragmentation process during biodegradation under soil burial leading to highly brittle behaviour. The molecular weight degradation of the constituent copolymer has been, as shown by gel permeation chromatographic (GPC) studies, found rather insignificant upon a few months of soil burial.

The ductile behaviour of pure PBAT and composites at low filler content has been correlated with large plastic deformation of the matrix polymer as demonstrated by fibrillar morphology on the fracture surface of the morphology of the samples broken during tensile testing. At large filler loading, the large plastic deformation of matrix was hindered by rapid propagation of the cracks that might have formed at weak polymer–filler interface leading eventually to premature failure and hence to low ductility of the materials. The composites demonstrated mechanical properties suitable for low-load bearing applications.

Keywords: natural fiber, cellulose, chitosan, morphology, deformation behaviour.

T. Wagner, Y. Charfi, S. Celevics, R. Schlimper

Inline-Prüfung von UD-Tapes mittels aktiver Thermographie

Inline-Prüfung von UD-Tapes mittels aktiver Thermographie

T. Wagner¹, Y. Charfi¹, S. Celevics¹, R. Schlimper¹;

**Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS,
Halle (Deutschland)**

Unidirektional verstärkte thermoplastische Tapes (UD-Tapes) lassen sich aufgrund der kontinuierlichen Schmelzimpregnierung schnell und wirtschaftlich herstellen und bieten ein großes Leichtbaupotenzial. Für einen sicheren Einsatz im späteren Bauteil ist eine zuverlässige Qualitätskontrolle im Herstellungsprozess unabdingbar. Für strukturelle Bauteile werden mittlerweile digitale Bauteilakten entwickelt, die es ermöglichen alle inneren und äußeren Einflüsse auf ein individuelles Bauteil über den gesamten Produktlebenszyklus zu dokumentieren. Durch den Einsatz von zerstörungsfreier Inline-Prüftechnik lassen sich Qualitätsmerkmale von Materialien im Produktentstehungsprozess erfassen. Verschiedene Fehlertypen von UD-Tapes, zum Beispiel Fasergassen, Welligkeiten und nicht-impregnierte Faserbündel lassen sich mittels der zerstörungsfreien Thermographie, der Lichtmikroskopie sowie der Röntgen-CT sicher detektieren. Dabei bietet die Thermographie diverse Vorteile im Gegensatz zu anderen zerstörungsfreien Prüfverfahren. Mittels der Inline-Thermographie können Fehlertypen im Tape detektiert werden, welche mit optischen Methoden nicht sichtbar sind. Insbesondere bei Tapes mit einer nicht transparenten Matrix bietet diese Methode große Vorteile. In denen am Fraunhofer IMWS durchgeführten Versuchen wurde ein signifikanter Bereich eines Kohlefaser-Tapes nach der Imprägnierung im Werkzeug durch einen IR-Linienstrahler im Produktionsprozess erhitzt. Mit einer Thermographie-Kamera wurde anschließend der Abkühlungsprozess aufgezeichnet. Dabei wurde eine Vielzahl an Thermogrammen aufgenommen, welche anschließend durch einen „Abroll“-Algorithmus zu einem Abkühlungsbild des gesamten UD-Tapes zusammengesetzt wurden. Damit wurde eine materialbedingte Inhomogenität im Tape sichtbar. Markante Bereiche im Thermogramm wurden anschließend charakterisiert und den beschriebenen Fehlertypen zugeordnet. Eine Prüfung im Zugversuch zeigte den Einfluss des Fehlertyps auf die mechanische Performance des UD-Tapes. Durch Bildung von Korrelationen konnten abschließend erste Hinweise darauf gewonnen werden, welche Merkmale im Thermogramm aus der inline Prüfung welchen Fehlertyp darstellt und wie dessen Einfluss auf die Mechanik des Tapes ist.

A. Braun, J. R. Agudo, G. Arnold

Approaches to overcome rheometric challenges when performing TTS measurements over a large temperature and frequency range

J. R. Agudo, M. Trujillo, G. Arnold, L. Ionov, M. Schäffler

Special applications in dynamic mechanical analysis: Combining rotational and axial drives for the characterization of novel materials

J. Fischer, M. Pfeiler-Deutschmann

Full Atomic Force Microscopy investigation of a polymer thin film

Approaches to overcome rheometric challenges when performing TTS measurements over a large temperature and frequency range

**A. Braun, J. R. Agudo, G. Arnold;
Anton Paar Germany GmbH, Ostfildern (Germany)**

Time–temperature superposition (TTS) measurements are a powerful tool to characterize the dynamic mechanical behaviour of samples over a wide range of temperatures and frequencies and, hence, to measure their mechanical properties in different states. In case the results have to describe the materials properties over the largest possible frequency and temperature range rheometer limitations are reached quickly. Because of this, single measurements without having to change the configuration are a challenge.

An example is given by the characterization of pressure sensitive adhesives (PSA). At very low temperatures these materials are characterized by a high shear modulus while the same material is quite soft at elevated temperatures. In case of measurements at low temperatures the torsional compliance of rheometer components is a severe issue: the deflection angle acting within the sample is only a small fraction of the deflection angle generated in the drive unit of the rheometer. To reduce the influence of the torsional compliance, a measuring plate with a rather small diameter is preferred. Additionally, at high temperatures and high frequencies, inertia issues will arise and the sample torque is smaller than the torque necessary to accelerate the measuring geometries. In this case, a large diameter of the measuring plate is preferred. Hence, an optimized rheometer configuration as well as a sophisticated controller being able to control the set parameter acting within the sample are necessary to carry out TTS measurements over wide ranges.

The presentation will show a strategy to perform TTS measurements over a temperature range of more than 200 °C and to measure sample moduli over more than 5 decades without having to change the configuration and the sample. In order to measure precisely at low temperatures, the torsional compliance of each rheometer is determined individually, since small changes of torsional compliance values may have a large effect on the measured moduli. Furthermore, inertia issues have been reduced by using a separate motor transducer mode. This enables to perform precise shear rheological measurements at high frequencies even with moderate plate diameters. The measured data allow to characterize the adhesive sample precisely far below and above its glass transition temperature. Furthermore, the method enables to characterize the impact of the frequency on the glass transition temperature over a broad frequency range.

Special applications in dynamic mechanical analysis: Combining rotational and axial drives for the characterization of novel materials

J. R. Agudo¹, M. Trujillo², G. Arnold¹, L. Ionov², M. Schäffler¹;

**¹ Anton Paar Germany GmbH, Ostfildern (Germany), ² Faculty of Engineering
Sciences, University of Bayreuth (Germany)**

Rheometers or dynamic mechanical analysers capable of combining two drives (rotational and linear drive) have opened new horizons in the characterization of novel materials [1–3]. First of all, combining two motors offers extreme flexibility in the choice of the material deformation mode (shear, torsion, extension, bending, compression). Therefore, these instruments are able to characterize a broad range of different materials from soft matters to stiff composites with Young's modulus above 100 GPa. Furthermore, combining a linear and a rotational measuring drive in one instrument enables the determination of complex Young's modulus $|E^*|$ as well as the complex shear modulus $|G^*|$ with a single sample in a continuous measurement run. These measurements can be performed in compression and shear mode by means of plate–plate measuring geometries and also in torsion and extension mode by means of clamping fixtures for cylindrical or rectangular specimens. The measurements can be further combined with sweeps in frequency, temperature or humidity.

This type of multi-task measurement is interesting for isotropic materials since it is an indirect method to obtain the viscoelastic Poisson's ratio and its dependence on time and temperature [4]. In the case of anisotropic materials, they are particularly useful since it is possible to evaluate extensive information on the viscoelastic properties of the material in longitudinal and transversal direction within one single run. This contribution shows several application examples in which axial and rotational measurements are combined in one single experiment to obtain extra information about the material. Special emphasis is given to anisotropic, bio-inspired and smart materials.

Literature

- [1] Meurer, J., Rodriguez Agudo, J. A., Zechel, S., Hager, M. D., Schubert, U. S.: Quantification of triple-shape memory behavior of polymers utilizing tension and torsion. *Macromol. Chem. Phys.* 222 (2021) 2000462.
- [2] Jiao, D., Lossada, F., Guo, J., Skarsetz, O., Hoenders, D., Liu, J., Walther, A.: Electrical switching of high-performance bioinspired nanocellulose nanocomposites. *Nat. Commun.* 12 (2021) 1312.
- [3] Dingler, C., Müller, H., Wieland, M., Fauser, D., Steeb, H., Ludwigs, S.: From understanding mechanical behavior to curvature prediction of humidity-triggered bilayer actuators. *Adv. Mater.* 33 (2021) 2007982.
- [4] Tschoegl, N. W., Knauss, W. G., Emri, I.: Poisson's ratio in linear viscoelasticity – A critical review. *Mech. Time Depend. Mater.* 6 (2002) 3–51.

Full Atomic Force Microscopy investigation of a polymer thin film

**J. Fischer, M. Pfeiler-Deutschmann;
Anton Paar GmbH (Austria)**

Atomic Force Microscopy (AFM) has been used to characterize mechanical, electrical, magnetic and other surface properties of polymers. In other words, atomic force microscopy can be used to determine various properties of polymer surfaces. When using this microscopy measurement method for characterization, it is often critical that all measurements are performed at the same location on the sample surface. In this case, different surface properties can be correlated accordingly and brought into a quantitative relationship.

Until now, however, AFM measurements with different investigation techniques, so-called AFM modes, at exactly the same location were difficult to perform, since this usually requires a hardware change on the AFM measuring instrument. As the consequence, scanning on the exact same position in nanometer range is not possible anymore after the hardware modification and lifting the AFM head. With a patented AFM-Z sensor technology, we now present a new possibility that allows switching between measurement modes without any hardware exchange. On the example of a thin film polymer blend, we stepwise learn more and more about the sample by using different AFM measurement modes, each step revealing different qualitative and quantitative sample properties, until we have gained all the information we need to unambiguously characterize the sample.

T. Abboud, A. Wutzler, T. Groth, H.-J. Radusch

Evaluation of polymer blends for hot melt pressure sensitive adhesives containing tackifying resins and thermoplastic elastomers

J. Jůza, I. Fortelný

Compatibilization of polymer melts using block copolymers

J. Zidek, F. Kucera, J. Petrus, T. Ondracka, E. Kulovana, M. Novacek, J. Kois, J. John

Co-processing of polypropylene and poly(vinyl chloride) as a model of the compatibilization of separated plastic waste

J. Wiener, F. Arbeiter, A. Tiwari, O. Kolednik, G. Pinter

Crack arrester effect in mineral-reinforced polypropylene using soft interlayers

R. Landgraf, E. Oelsch, J. Ihlemann

Mechanical behavior of additively manufactured thermoplastic polyurethane: Experiments, constitutive modeling and Finite-Element-Analyses

M. Jabłońska, M. Rühl, M. Menzel, U. Hirsch, A. Heilmann

Biofouling studies of zwitterionic coatings of feed spacer for biofouling mitigation

K. Krause, T. Neumeyer, V. Altstädt

Hart-Weich-Verbunde mittels Co-Injektionsverfahren: Einfluss der Viskosität auf die Bauteilmorphologie

J. Roitner, V. M. Archodoulaki

Betrachtung des Degradationsverhaltens von Polyethylenen im mechanischen Recycling

Evaluation of polymer blends for hot melt pressure sensitive adhesives containing tackifying resins and thermoplastic elastomers

T. Abboud¹, A. Wutzler², T. Groth³, H.-J. Radusch^{2,3};

¹ Arakawa Europe GmbH Site Schkopau (Germany), ² Polymer Service GmbH Merseburg (Germany), ³ Martin Luther University Halle-Wittenberg, Halle (Germany)

Resins are materials largely employed in the adhesive industry and when employed in pressure sensitive adhesives they are commonly labeled as tackifiers [1]. There are several types of adhesives and a special group is the hot melt pressure sensitive adhesive. These materials are polymer blends of thermoplastic elastomer, tackifier and, commonly, also a plasticizer. Blends are widely employed in the industry due to their properties improvement achieved for a material but with low efforts and costs involved in their development in comparison to a new designed material. Polymer blends are generally thermodynamically immiscible but they are held to be compatible in concern to the application behavior enabling them to be extensively employed in end-use applications. Hence, understanding the compatibility between the elastomers and the tackifiers is crucial for achieving adequate adhesive performance in pressure sensitive adhesives [2]. The influence of chemically different tackifiers is investigated in this study in order to understand its correlation with polymer blend viscoelastic properties, morphology, surface properties and adhesive performance. Styrenic block copolymers are classically employed in manufacturing hot melt pressure sensitive adhesives. However, developments in elastomeric field suggest the use of poly(ethylene-co-1-octene) block copolymer as an alternative for such styrenic copolymers. A comparison between both thermoplastic elastomers was conducted regarding blend viscoelastic properties, morphology, surface properties and adhesive performance. The results revealed that compatibility between the thermoplastic elastomer and the tackifier highly influences the investigated properties. In summary, the impact of tackifier chemistry in the polymer blends used as hot melt pressure sensitive adhesive could be demonstrated through different levels of interaction with the polyisoprene and the polystyrene contained in the block copolymer by the methods selected. It was also demonstrated that tackifier chemistry is relevant for the performance of hot melt pressure sensitive adhesives when olefinic block copolymer was inserted into the blends.

Literature

- [1] Abbott, S.: Adhesion Science – Principles and Practice, DEStec Publ. Inc., Lancaster (PA/USA) (2015).
- [2] Abboud, T. et al.: Tackifying resins and their polymer blends properties – Application in hot melt pressure sensitive adhesives. 44. Münchener Klebstoff- und Veredelungs-Symposium 2019, Hotmelts – Schmelzhaftklebstoffe – Schmelzmassen, München (28.–30.10.2019).

Compatibilization of polymer melts using block copolymers

J. Jůza, I. Fortelný;

Institute of Macromolecular Chemistry, Czech Academy of Sciences, Prague
(Czechia)

Efficient compatibilisation of immiscible polymer blends is one of the most important tasks in polymer engineering. Block and graft copolymers with blocks miscible or compatible with or the related components of a polymer blend can serve as compatibilisers. Based on models of Leibler [1] and Noolandi [2], we derived our own equations and calculated compatibilising effect for several systems [3] for the dry-brush (copolymer chains shorter than homopolymer chains) and wet brush (copolymer chains longer than homopolymers chains) model as well as for low content of copolymer in the system.

While the wet brush results could be fluently continued as low copolymer content results for lower copolymer concentration, the dry brush model gave similarly compatible results for long homopolymers chains only. For short chains, the dry brush model provides higher copolymer content and higher compatibilisation efficiency than model of low copolymer concentration in whole copolymer concentration range for some systems. The thickness of the interfacial layer in each phase is determined from a condition of minimal Gibbs energy with respect to layer thickness for the wet brush, from length of the stretched copolymer chain (proportional to segments count in the copolymer) for the dry brush, and from Gaussian coil diameter for the low copolymer content.

Therefore, we have tried to leave out some of approximations of the dry brush systems in order to make the results for lower copolymer concentrations more compatible with results of the formula for low copolymer content. The interfacial layer thickness has been expressed from the minimal Gibbs energy condition assuming the difference from the thickness for original model is not strong, similarly as for wet brush model. However, various formulae modifications either brought results with those for low copolymer contents either very slightly closer together, or made them even more different.

Although none of the modifications appeared to bring satisfactory results, we are presenting how some of them affect the computed interface composition and decrease in interfacial tension.

This research was funded by Technology Agency of the Czech Republic (Technologická Agentura České Republiky), project number TN01000008.

Literature

- [1] Leibler, L.: Emulsifying effects of block copolymers in incompatible polymer blends. *Makromol. Chem., Macromol. Symp.* 16 (1988) 1–17.
- [2] Noolandi, J.: Interfacial tension in incompatible homopolymer blends with added block copolymer. *Makromol. Chem., Rapid Commun.* 12 (1991) 517–521.
- [3] Fortelný, I., Jůza, J.: Analysis of the effect of block copolymers on interfacial tension in immiscible polymer blends. *Polymer* 150 (2018) 380–390.

Co-processing of polypropylene and poly(vinyl chloride) as a model of the compatibilization of separated plastic waste

**J. Zidek¹, F. Kucera¹, J. Petrus¹, T. Ondracka², E. Kulovana², M. Novacek²,
J. Kois², J. John²;**

**¹ CEITEC, Brno University of Technology (Czech Republic), ² VIA ALTA a.s.,
Okrisky (Czech Republic)**

Processing the separated plastic waste is a current task because the majority of mixed plastic waste is still combusted or landfilled. It requires co-processing of various polymers such as low-density polyethylene (PE-LD), high-density polyethylene (PE-HD), polypropylene (PP), poly(vinyl chloride) (PVC) or polyethylene terephthalate (PET) with a wide scale of properties. The co-processing of such heterogeneous blend causes problems due to the different nature of the materials. The variability in temperature stability, crystallinity, melt viscosity, and chemical compatibility plays a key role. In the proposed contribution, the mixed polymer was simplified to a binary PP/PVC blend to highlight the theoretical effects of their compatibilization. The PP and PVC were selected due to the fact that these two polymers are probably the most different polymers present in real plastic waste. Polypropylene represents a nonpolar, semicrystalline component that is relatively thermally stable. On the other hand, PVC represents a polar and amorphous polymer, and component with risk of thermal degradation. The contribution summarizes the problems that can be expected during the co-processing of such a diverse mixture. Furthermore, the contribution presents how the co-processing influences the behavior of both polymers, what can be improved by compatibilization, and how processing machines can be set. The data can help one to focus on the problems with polymer recycling.

Funding of this work under the Project FW01010609 from the Technology Agency of the Czech Republic was greatly appreciated.

Crack arrester effect in mineral-reinforced polypropylene using soft interlayers

J. Wiener¹, F. Arbeiter¹, A. Tiwari^{2,3}, O. Kolednik², G. Pinter¹

¹ Materials Science and Testing of Polymers, Montanuniversität Leoben (Austria), ² Erich-Schmid-Institute of Materials Science, Austrian Academy of Science, Leoben (Austria), ³ Materials Center Leoben GmbH (Austria)

Biological materials often show a desirable combination of toughness and strength. Studies on nacre or deep sea sponges [1] show, that a brittle matrix is toughened by thin interlayers (IL) of protein. These soft IL act as crack arresters [2] and prevent catastrophic failure. The same mechanisms shall now be mimicked using industrial materials. Previous contributions [3] suggest, that a dexterous choice of matrix and IL material is necessary. In this contribution, a mineral particle reinforced polypropylene (PP) matrix and an IL made of very soft PP is used. The experimental J-integral, J_{exp} , is determined through single edge notch bending (SENB) experiments to characterise fracture toughness. A normalised parameter for crack advancement, L , has to be used in order to exclude the influence of specimen geometry. A sharp increase in J_{exp} at $L = 0$ can be observed [4], where the crack encounters the IL. This represents the amount of energy required to overcome the IL, demonstrating the effectiveness of the soft IL as crack arrester. The relation between L and the testing time t represents the crack growth rate and can also be used to study crack arrest at an IL. Such investigations [4] show that crack growth comes to a complete halt at the IL. The growth rate remains at 0 mm/s until a new crack initiates in the remaining matrix ligament due to further loading. Since the IL stays intact at all times, this can be considered a re-initiation of a crack instead of continuous crack growth. Even after re-initiation, crack growth is slowed compared to the pure matrix material, because the intact IL functions as stabilizing element.

References

- [1] Miserez, A., Weaver, J. C., Thurner, P. J., Aizenberg, J., Dauphin, Y., Fratzl, P., Morse, D. E., Zok, F. W.: Effects of laminate architecture on fracture resistance of sponge biosilica: Lessons from nature. *Adv. Funct. Mater.* 18 (2008) 1241–1248.
- [2] Sistaninia, M., Kolednik, O.: Effect of a single soft interlayer on the crack driving force. *Eng. Fract. Mech.* 130 (2014) 21–41.
- [3] Tiwari, A., Wiener, J., Arbeiter, F., Pinter, G., Kolednik, O.: Application of the material inhomogeneity effect for the improvement of fracture toughness of a brittle polymer. *Eng. Fract. Mech.* 224 (2020) 106776.
- [4] Wiener, J., Arbeiter, F., Tiwari, A., Kolednik, O., Pinter, G.: Bioinspired toughness improvement through soft interlayers in mineral reinforced polypropylene. *Mech. Mater.* 140 (2020) 103243.

Mechanical behavior of additively manufactured thermoplastic polyurethane: Experiments, constitutive modeling and Finite-Element-Analyses

**R. Landgraf, E. Oelsch, J. Ihlemann;
Chair of Solid Mechanics, Faculty of Mechanical Engineering, Chemnitz
University of Technology (Germany)**

This contribution deals with the combined experimental and numerical investigation on the mechanical behavior of additively manufactured thermoplastic polyurethane (TPU). 3D-printed structures were produced by the Screw Extrusion Additive Manufacturing [3] and test specimens for mechanical testing were extracted in different directions with respect to the layer-wise building direction. The mechanical behavior of these specimens was characterized in uniaxial tension tests with monotonic and cyclic loading, and the results were compared to the behavior of injection molded specimens. The experimental investigation revealed that the strain at failure strongly depends on the production process. Instead, the inelastic properties are nearly unaffected and thus independent of the production process.

These results were further investigated by the help of finite element simulations. To this end, finite element models for tensile test specimens with grooves in different directions were developed. In order to capture the strongly nonlinear and inelastic material behavior of the TPU, the so called Model of Rubber Phenomenology (MOPRH) [1, 2] was applied. By the help of these models, the behavior of the differently prepared specimens was analyzed and compared to experimental findings.

Literatur

- [1] Besdo, D., Ihlemann, J.: A phenomenological constitutive model for rubberlike materials and its numerical applications. *Int. J. Plast.* 19/7 (2003) 1019–1036.
- [2] Landgraf, R., Ihlemann, J.: Application and extension of the MORPH model to represent curing phenomena in a PU based adhesive. In: Lion, A., Jöhlitz, M. (Eds.): *Constitutive Models for Rubber X*. Taylor & Francis Group, London (2017), p. 137–143.
- [3] Oelsch, E., Landgraf, R., Jankowsky, L., Kausch, M., Hoyer, S., Drossel, W.-G., Ihlemann, J.: Comparative investigation on the mechanical behavior of injection molded and 3D-printed thermoplastic polyurethane. *J. Rubber Res.* 24 (2021) 249–256.

Biofouling studies of zwitterionic coatings of feed spacer for biofouling mitigation

**M. Jabłońska, M. Rühl, M. Menzel, U. Hirsch, A. Heilmann (✉);
Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle
(Germany)**

Reverse osmosis (RO) is one of the most energy efficient methods for water desalination [1]. Despite the numerous advantages of RO membrane technology, the problem which affects plenty of desalination plants is fouling. Among all fouling types biofouling, which is an accumulation of bacteria on the membrane and feed spacer surfaces, is particularly challenging. Biofouling leads to the major operational difficulties and increases desalination costs [2]. One of the strategies to mitigate biofouling is the modification of the membrane or feed spacer surfaces.

In this work anti-biofouling modification of the feed spacer surface has been proposed. Hydrophilic and zwitterionic coatings have been generated via the combination of the plasma treatment with dielectric barrier discharge in air and dip coating. The obtained coatings were analyzed by X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM). The anti-fouling properties of the modified samples were studied in batch test with *Pseudomonas fluorescens* GFP. Continuous anti-fouling testing was performed over several days in the constructed for this purpose fouling simulator.

Literature

- [1] Elimelech, M., Phillip, W. A.: The future of seawater desalination: Energy, technology, and the environment. *Science* 333 (2011) 712–717.
- [2] Flemming, H.-C.: Reverse osmosis membrane biofouling. *Exp. Therm. Fluid Sci.* 14 (1997) 382–391.

Hart-Weich-Verbunde mittels Co-Injektionsverfahren: Einfluss der Viskosität auf die Bauteilmorphologie

K. Krause¹, T. Neumeyer¹, V. Altstädt^{1,2};

¹ Neue Materialien Bayreuth GmbH, Bayreuth (Deutschland), ² Lehrstuhl für Polymere Werkstoffe, Universität Bayreuth (Deutschland)

Soft-Touch-Haptik gewinnt derzeit in zahlreichen Anwendungen an Bedeutung, darunter die Fahrzeuginnenausstattung und verschiedenste Artikel des täglichen Gebrauchs. Eine Vielzahl der Teile mit Soft-Touch-Haptik besteht aus zwei Kunststoffkomponenten: einer starren Strukturkomponente, die von einer weichen Komponente bedeckt ist. Die Co-Injektion (auch: Sandwich-Spritzgießen) stellt einen Ansatz zur Herstellung solcher Bauteile dar. Dabei werden zwei thermoplastische Komponenten nacheinander in ein Spritzgießwerkzeug injiziert. Die Hautkomponente wird vorgelegt und die nachfolgend eingespritzte Kernkomponente breitet sich in der noch plastischen Seele der Hautkomponente aus.

Dabei ist eine möglichst homogene Verteilung der Kernkomponente im gesamten Bauteil und eine völlige Umschließung durch die Hautkomponente anzustreben [1]. Die schersensitiven Viskositäten beeinflussen die Ausbildung der Haut-Kern-Struktur maßgeblich: Während ein zu großes Viskositätsverhältnis $\eta_{\text{Kern}}/\eta_{\text{Haut}}$ zu geringer Ausbreitung des Kernmaterials führt, tritt bei zu geringem Verhältnis der sogenannte Fingerfluss auf [2, 3].

Ziel dieser Arbeit ist die Aufklärung des Einflusses der Viskosität der Hautkomponente und damit des Viskositätsverhältnisses auf die Morphologie co-injizierter Formteile. In der vorliegenden Arbeit werden thermoplastische Elastomere auf SEBS (Styrol-Ethylen-Butylen-Styrol)-Basis (TPS) als weiche Hautkomponente und Polypropylen (PP) als Kernkomponente verwendet. Die Viskosität der Hautkomponente wird über den PP-Anteil des TPS variiert, das Masseverhältnis von Öl und SEBS bleibt unverändert. Steigender PP-Anteil führt zu verringerter Viskosität des TPS aufgrund des entschlaufenden Effekts von PP im Weichsegment des SEBS.

Es wird gezeigt, dass eine homogene Verteilung insbesondere dann erzielt werden kann, wenn das Viskositätsverhältnis über einen breiten Scherratenbereich möglichst konstant ist, d. h., die Materialien sich hinsichtlich ihrer Strukturviskosität ähnlich verhalten.

Literature

- [1] Selden, R.: Co-injection molding: Effect of processing on material distribution and mechanical properties of a sandwich molded plate. *Engineering* 40 (2000) 1165–1176.
- [2] Watanabe, D., Ishiaku, U. S., Nagaoka, T., Tomari, K., Hamada, H.: The flow behavior of core material and breakthrough phenomenon in sandwich injection molding. Part II: Influence of mold cavity thickness and core cylinder temperature. *Int. Polym. Process.* 18 (2003) 405–411.
- [3] Zaverl, M., Valerio, O., Misra, M., Mohanty, A.: Study of the effect of processing conditions on the co-injection of PBS/PBAT and PTT/PBT blends for parts with increased bio-content. *J. Appl. Polym. Sci.* 132 (2015) 41278 (11 p.).

Betrachtung des Degradationsverhaltens von Polyethylenen im mechanischen Recycling

**J. Roitner, V. M. Archodoulaki;
Institut für Werkstoffwissenschaft und Werkstofftechnologie, TU Wien,
(Österreich)**

Globale Umweltpläne für die Kreislaufwirtschaft führen zu dem Drang nach Verbesserung des Kunststoffrecyclings, das gerade bei Polyethylenen einige Hürden aufweist. Ein Grund für die Komplexität dieses Themas ist die große Bandbreite an Materialien, die wir als "Polyethylen" bezeichnen. Es gibt nahezu unendlich viele Möglichkeiten für die Hersteller, die Eigenschaften zu modifizieren, um das potenzielle Anwendungsspektrum zu erweitern. Zu diesen Techniken gehören die Einarbeitung von Co-Monomeren, Verzweigungen, Blends und Additiven.

Polyethylen kann zwei unterschiedliche Abbauprozesse aufweisen, Vernetzung und Kettenspaltung, deren Auftreten den Recyclingprozess erheblich beeinflussen kann [1]. Vernetzungsstellen verhindern das Folienblasformen, können aber die mechanischen Eigenschaften von beispielsweise Rohren verbessern. Umgekehrt beeinträchtigt die Kettenspaltung die Langzeitstabilität von Rezyklaten, kann aber die Verarbeitungseffizienz verbessern, indem sie die im Verarbeitungsprozess auftretenden Drücke durch eine geringere Viskosität reduziert. [2]

In dieser Studie wurde die Auswirkung von Mehrfachverarbeitung der Materialien auf die Prävalenz der jeweiligen Degradationsmechanismen für verschiedene Polyethylen-Typen (HD, LD, LLD) untersucht, um die Verarbeitbarkeit und die Eigenschaften des Rezyklats zu analysieren und in einem weiteren Schritt zu optimieren. Unterschiedliche Typen von PE-HD und PE-LD zeigten mittels rheologischer Messmethoden jeweils einen oder eine Kombination aus beiden Abbaumechanismen, während alle verwendeten PE-LLD-Typen eine starke Tendenz zur Vernetzung aufwiesen. Durch Zug- und Schlagzugversuche wurde ermittelt, dass die mechanischen Eigenschaften unabhängig vom Degradationsmechanismus konstant blieben.

Diese Ergebnisse deuten darauf hin, dass die Degradationsmechanismen durch die Verarbeitungsparameter während des Recyclings gesteuert werden können, ohne die endgültigen mechanischen Eigenschaften des Materials offenkundig zu beeinträchtigen. Solche grundlegenden Erkenntnisse in der Polyethylenverarbeitung ebnen den Weg für eine stark verbesserte Verarbeitungseffizienz, Eigenschafts- und Produktoptimierung bei vernachlässigbaren Kosten und könnten sofort und auf breiter Basis in der gesamten Recyclingindustrie umgesetzt werden.

Literatur

- [1] Jin, H., Gonzalez-Gutierrez, J., Oblak, P., Zupančič, B., Emri, I.: The effect of extensive mechanical recycling on the properties of low density polyethylene. Polym. Degrad. Stab. 97 (2012) 2262–2272.
- [2] Vilaplana, F., Karlsson, S.: Quality concepts for the improved use of recycled polymeric materials: A review. Macromol. Mater. Eng. 293 (2008) 274–297.

T. Hedtke, J. Martins de Souza e Silva, T. Groth, C. E. H. Schmelzer

Elastin as basis for cutting-edge wound dressings

K. P. Malla, R. J. Yadav, S. Sakurai, H. Yamane, S. Henning, R. Adhikari

Electrospun blends of polycaprolactone/poly-L-lactic acid/gelatin and natural hydroxyapatite for bone tissue engineering

G. Hillrichs, R. Lach, G. H. Michler, M. Götze, S. Henning, C. Costa

Laser processing of polymeric nanofiber nonwovens for biomedical applications with ultrashort laser pulses

A. Narayanan Ramakrishnan, O. Röhrle, C. Ludtka, J. Köhler, A. Kiesow, S. Schwan

The role of denture adhesives on the oral health of partial denture wearers: A numerical study

F. Du, H. E. Yener, R. Androsch

Development of a biopolymer/drug system for fighting mosquito-borne tropical diseases

J. Köhler, A. Kiesow, A. N. Ramakrishnan, J. Hey, S. Schwan

Der Einfluss von physiologischen Bedingungen auf das mechanische Verhalten von Polymer-Haftcreme-Kompositen

Elastin as basis for cutting-edge wound dressings

T. Hedtke^{1,2}, J. Martins de Souza e Silva^{1,3}, T. Groth², C. E. H. Schmelzer¹;
¹ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ² Biomedical Materials Group, Institute of Pharmacy, Martin Luther University Halle-Wittenberg, Halle (Germany), ³ Institute of Physics, Martin Luther University Halle-Wittenberg, Halle (Germany)

1 Background

The extracellular matrix (ECM) consist of a fibrous network of structural proteins that contributes to the mechanical properties of tissues and provides a scaffold for cell adhesion. Elastin, an essential structural protein and main component of elastic fibers in the ECM, is responsible for the elasticity and resilience of force-bearing tissues and organs. Since elastin production dramatically drops in the early neonatal age, elastin is subject to degeneration associated with functional losses in corresponding tissues that can be addressed by the treatment with complementary biomaterials.

2 Methods

Mature elastin was isolated from porcine aortas that were obtained as by-products from a local slaughterhouse. The isolated and insoluble elastin was partially hydrolyzed to obtain soluble elastin derivatives that differ in the molecular weight distribution of the fragments and thus in solubility. Protein-based fibrous materials were fabricated by electrospinning from elastin blends and stabilized by chemical cross-linking (Fig. 1A,C). Elastin derivatives were mixed with gelatin in a ratio of 3:1 (w/w) and electrospun from a solvent system of non-halogenated carboxylic acids. In further experiments, the elastin content was raised to 90 % (w/w). The fiber fleeces were chemically cross-linked after electrospinning. The microstructure of the fibrous biomaterials was investigated by scanning electron microscopy (SEM) and nano-scale X-ray computed tomography (nano-CT). Cell compatibility was assessed by culturing fibroblasts on the materials.

Furthermore, sponge-like biomaterials were produced by lyophilization of hydrogels and protein solutions comprising equal amounts of elastin and gelatin (Fig. 1B,C). Formaldehyde was optionally added as cross-linker. After casting, the samples were frozen and subsequently freeze-dried. The microstructure of the sponge-like materials was investigated by SEM and micro-scale X-ray computed tomography (micro-CT). Material integrity in aqueous media was evaluated in vitro by time-dependent UV/Vis spectroscopy.

3 Results

This work aimed to design cutting-edge biomaterials based on elastin isolated from biological tissues in order to address functional losses caused by elastin degeneration in biomedical applications. First, we successfully established a cost-efficient procedure to obtain elastin from by-products of the food industry in large quantity as well as methods for elastin solubil-

ization. By means of electrospinning and lyophilization, isolated and processed elastin was successfully converted to into nanofibrous and sponge-like biomaterials that are reasonable stable in aqueous media, cell-compatible and bioabsorbable. Both materials are characterized by a high porosity of $\leq 84\%$ and $\leq 89\%$ for sponge-like and fibrous biomaterials, respectively. Cross-linking is essential for the integrity of electrospun protein-based materials but reduces swellability of sponge-like materials. Preliminary mechanical testing of both revealed a Young's modulus ≤ 50 kPa when wetted. Both materials will be tested for wound healing applications.

4 Results

The materials produced by freeze-drying and electrospinning mimic the ECM in their composition and microstructure. Elastin-like materials have a great potential in modern biomedical applications, in particular as cutting-edge wound dressings in order to address functional losses of tissues and to support the healing process.

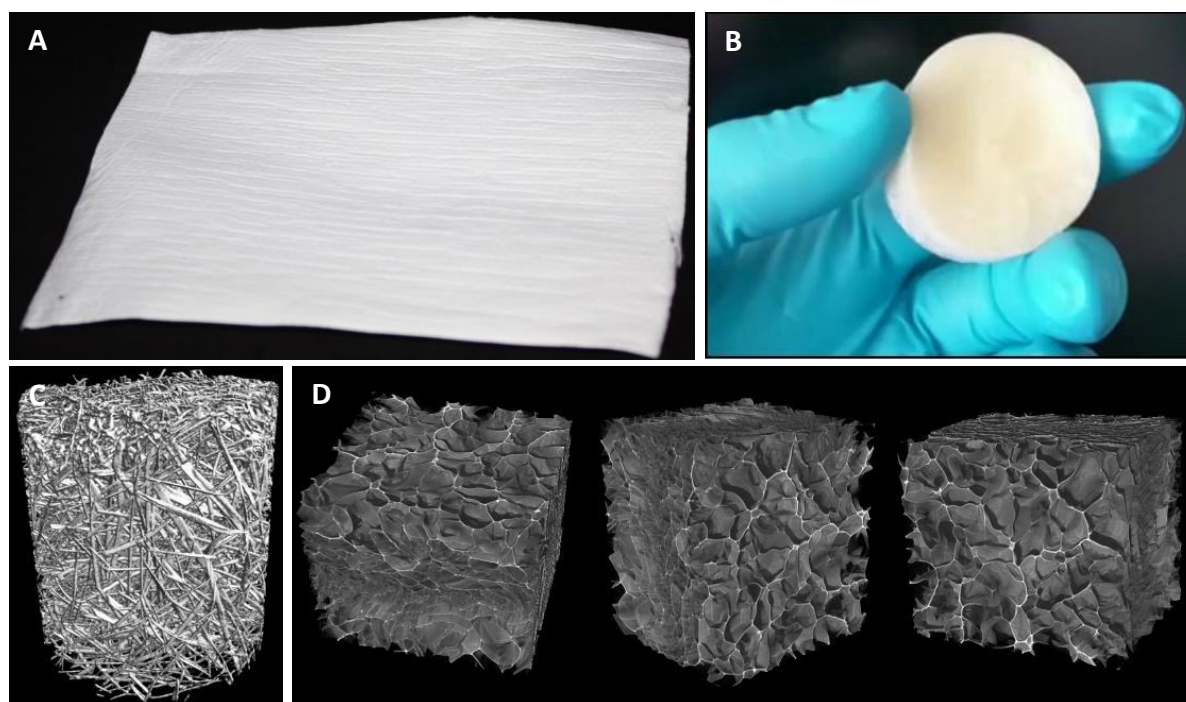


Figure 1: Elastin-based biomaterials. Macroscopic images show a $10 \times 10 \text{ cm}^2$ electrospun nonwoven (A) and a hydrogel sponge (B, 4 cm diameter) with elastin contents of 75 % and 50 % (w/w), respectively. The 3D microstructure of the nonwoven mesh and the hydrogel sponge was visualized by nano-CT (C) and micro-CT (D), respectively. The cylinder's diameter in (C) is $60 \text{ }\mu\text{m}$, the edge length in (D) is 5 mm.

Electrospun blends of polycaprolactone/poly-L-lactic acid/gelatin and natural hydroxyapatite for bone tissue engineering

K. P. Malla^{1,2}, R. J. Yadav³, S. Sakurai⁴, H. Yamane⁴, S. Henning⁵, R. Adhikari²;

¹ Department of Chemistry, Tribhuvan University, Kathmandu (Nepal),

² School of Health and Allied Sciences, Pokhara University, Pokhara (Nepal),

³ Department of Chemistry PNC, Tribhuvan University, Pokhara (Nepal),

⁴ Department of Biobased Materials Science, Kyoto Institute of Technology, Matsugasaki, Sakyo, Kyoto (Japan), ⁵ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany)

The bone tissue repairing demands a material on the principle of biology, clinical science, and engineering [1]. The challenge of the tissue engineering is to fabricate a scaffold that could be effectively mimic to the biophysical environment of natural extracellular matrix (ECM) [2]. In this study, polycaprolactone/poly-L-lactic acid/gelatin/vitamin D3 (PCL/PLLA/GEL/VD3) and various weight percentage (2, 3 and 5 wt. %) of hydroxyapatite (HAp) nanoparticle-incorporated PCL/PLLA/GEL/VD3 scaffolds were fabricated according to modified protocol of Ba Linh et al. [3] by electrospinning technique. Structural and mechanical properties of the scaffolds were determined by scanning electron microscopy (SEM) and tensile testing. The chemical interactions between polymers, VD3 and HAp nanoparticles were analyzed by Fourier transforms infrared spectroscopy (FTIR) and the hydrophilic property of scaffold was analyzed by water contact angle method (WCA). The SEM results confirmed the smooth and bead free morphology with uniform diameter and embedded HAp on the scaffold. Similarly, results of tensile testing indicated that 5 wt.% HAp nanoparticle-incorporated scaffold has higher tensile strength (3.80 MPa) than polymers and VD3-blended scaffold (1.15 MPa). The results of FTIR analysis have shown the chemical interactions between PCL, PLLA, and GEL with VD3 and HAp molecules. The results of contact angle measurements confirmed the decreasing trends of water contact angles from 79.0 to 44.8 degree on increasing weight percentage of HAp nanoparticles from 0 to 5 wt. %, respectively, indicating the better hydrophilicity and biodegradability nature of the scaffolds. These characterization results have shown that natural HAp incorporated with PCL/PLLA/GEL/VD3 composite-blended scaffold will be a promising candidate for bone tissue regeneration study.

Literature

- [1] Sattary, M., Rafienia, M., Kazemi, M., Salehi, H., Mahmoudzadeh, M.: Promoting effect of nano hydroxyapatite and vitamin D3 on the osteogenic differentiation of human adipose-derived stem cells in polycaprolactone/gelatin scaffold for bone tissue engineering. *Mater. Sci. Eng.* 97 (2019) 141–155.
- [2] Remya, K. R., Joseph, J., Mani, S., John, A., Varma, H. K., Ramesh, P.: Nanohydroxyapatite incorporated electrospun polycaprolactone/polycaprolactone–polyethyleneglycol–polycaprolactone blend scaffold for bone tissue engineering applications. *J. Biomed. Nanotechnol.* 9 (2013) 1483–1494; Shitole, A. A., Raut, P. W., Sharma, N., Giram, P., Khandwekar, A. P., Garnaik, B.: Electrospun polycaprolactone/hydroxyapatite/ ZnO nanofibers as potential biomaterials for bone tissue regeneration. *J. Mater. Sci. Mater. Med.* 30 (2019) 17 p.
- [3] Ba Linh, N. T., Min, Y. K., Lee, B. T.: Hybrid hydroxyapatite nanoparticles-loaded PCL/GE blend fibers for bone tissue engineering. *J. Biomater. Sci. Polym. Ed.* 24 (2013) 520–538.

Laser processing of polymeric nanofiber nonwovens for biomedical applications with ultrashort laser pulses

G. Hillrichs¹, R. Lach², G. H. Michler², M. Götze^{1,3}, S. Henning³, C. Costa¹;

¹ University of Applied Sciences Merseburg (Germany), ² Institut für Polymerwerkstoffe e.V., Merseburg (Germany), ³ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany)

Electrospun polymeric nanofiber nonwovens are good candidates to be used as cell scaffolds in tissue engineering. They mimic the extracellular matrix of biological tissues. Also, growth factors or drugs can be implemented during the electrospinning process. However, the spaces between the nanofibers are too small for biological cells to grow into the volume of the nonwovens, so that the cell growth is mainly restricted to the surface of the nonwovens. To fill larger voids, i.e., in bone defects, 3D samples of nanofiber scaffolds with extended capability of cell proliferation into the volume are needed. For this purpose, tubes of nanofiber nonwovens with a diameter of 3.3 mm have been developed [1]. In the present work we investigated a drilling process based on laser ablation to generate microholes in polylactide nanofibers tubes. These nonwovens are very sensitive to heat [2].

By using short picosecond or nanosecond laser pulses the thermal damage and the heat affected zones around the laser generated structures in the nonwovens could be avoided (Figure 1). Also, a laser cutting process for nanofiber tubes into ring scaffolds has been developed (the DFG is thanked for financial funding of project MI 358/37-3).

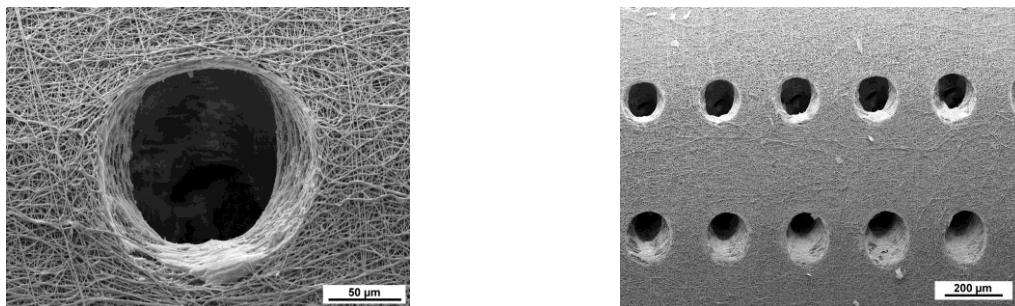


Figure 1: Left: Hole ($\varnothing$ 150 μm) drilled with picosecond laser radiation. Right: Array of holes with $\varnothing$ 150 μm . Hole distance 300 μm .

Literature

- [1] Henning, S., Michler, G. H., Sowislok, A., Jennissen, H. P., Lach, R., Heselich, A., Dohle, E., Al-Maawi, S., Ghanaati, S., Kirkpatrick, J.: Multimodal hybrid materials for regenerative medicine: Manufacturing and morphology of nanofiber scaffolds. POLY-CHAR 2019 [Kathmandu], 19.–23.05.2019, Kathmandu (Nepal).
- [2] Götze, M., Kürbitz, T., Costa, C., Schmelzer, C., Heilmann, A., Hillrichs, G.: Surface modifications of polylactide nanofiber nonwovens and bulk material by short and ultrashort pulsed laser radiation. Proc. SPIE 10906 (2019), DOI: 10.1117/12.2509592 (12 p.).

The role of denture adhesives on the oral health of partial denture wearers: A numerical study

A. Narayanan Ramakrishnan¹, O. Röhrle², C. Ludtka³, J. Köhler⁴, A. Kiesow¹, S. Schwan¹;

¹ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ² Universität Stuttgart (Germany), ³ University of Florida, Gainesville (USA), ⁴ Martin Luther University Halle-Wittenberg, Halle (Germany)

Removable partial dentures significantly influence the mechanical stress characteristics of both the jaw-bone and oral mucosa. The aim of this study was to examine the stress state caused by biting forces upon insertion of partial dentures into the assembly, and to understand the influence of the resulting contact pressure on its retention behavior. For this purpose, computer tomography data of the jawbone and denture was employed to develop corresponding 3D models, which were further used for finite element simulations. The denture system rests on the oral mucosa surface and three abutment teeth. The application of bite forces on the denture generated varying contact mechanical response to the stimulus, across different regions of the denture-mucosa contact space. The contact pressures observed in the regions of the oral mucosa in contact with the denture were below the clinical pressure pain threshold value for soft tissue, which potentially lowers the risk of pain being experienced by denture users. The variation of the retention behavior of the denture to the mucosa was observed across the contact space. Thus, there is a need for restraining mechanisms for the denture system in order to avoid excessive bending forces transmitted to the soft tissue due to the applied bite force.

Development of a biopolymer/drug system for fighting mosquito-borne tropical diseases

F. Du, H. E. Yener, R. Androsch;

Interdisciplinary Center of Transfer-oriented Research in Natural Sciences (IWE TFN), Martin Luther University Halle-Wittenberg, Halle (Germany)

Mosquito-borne tropical diseases, such as malaria, dengue fever, yellow fever, Zika, and lymphatic filariasis, occur at the global scale and involve a wide range of viral and other pathogenic agents, causing hundreds of thousands of deaths each year. Wearable mosquito-repellent personal protection devices appear possible to increase the duration of repellence activity by incorporating repellents into biopolymers [1, 2]. In this paper, the repellent ethyl butylacetylaminopropionate (IR3535[®]) was used as solvent to form a functional poly(lactic acid) (PLA) scaffold by thermally-induced phase separation (TIPS), with PLA regarded as a promising environment-friendly polymeric carrier for the repellent. The miscibility of the polymer/solvent system PLA/IR3535[®] was firstly explored by employing non-crystallizable poly(D,L-lactic acid) (PDLLA), by analysis of the glass transition behavior in the full composition range. The data revealed that the two components are miscible regardless the system composition, with the glass transition temperature of solutions showing a negative deviation from the linear mixing rule, suggesting rather weak enthalpic interactions of the system components [3].

Crystallization-induced solid–liquid (S-L) phase separation, as analyzed by differential scanning calorimetry and cloud-point measurements, occurs on slow cooling of solutions containing crystallizable poly(L-lactic acid) (PLLA) at temperatures higher than ambient temperature. The S-L phase-separation temperature decreases with increasing cooling rate as well as solvent concentration, as expected from the kinetic nature of crystallization and the polymer equilibrium melting point depression according to the Flory theory, respectively. Crystallization of PLLA in solution with IR3535[®] is faster than melt-crystallization of neat PLLA, with the maximum crystallization rate decreasing with decreasing PLLA content in the investigated range from 10 to 50 m% PLLA. In addition, a large sort of spherulitic superstructures were obtained by variation of the crystallization conditions, possibly leading to different 3D scaffold structures and IR3535[®]-delivery characteristics.

In summary, the polymer/repellent system PLA/IR3535[®] has been analyzed regarding the thermodynamics of mixing, with PLLA being identified as a potential candidate as reservoir for the mosquito repellent IR3535[®].

Literature

- [1] Sungkapreecha, C., Iqbal, N., Focke, W. W., Androsch, R.: Crystallization of poly(L-lactic acid) in solution with the mosquito-repellent N,N-diethyl-3-methylbenzamide. *Polym. Crystallization* 2 (2019) e10029.
- [2] Yener, H. E., Hillrichs, G., Androsch, R.: Phase behavior of solvent-rich compositions of the polymer/drug system poly(butylene succinate) and N,N-diethyl-3-methylbenzamide (DEET). *Colloid Polym. Sci.* 299 (2021) 873–881.
- [3] Du, F., Schick, C., Androsch, R.: Full-composition-range glass transition behavior of the polymer/solvent system poly(lactic acid)/ethyl butylacetylaminopropionate (PLA/IR3535(R)). *Polymer* 209 (2020) 123058.

Der Einfluss von physiologischen Bedingungen auf das mechanische Verhalten von Polymer-Haftcreme-Kompositen

J. Köhler¹, A. Kiesow², A. N. Ramakrishnan^{2,3}, J. Hey⁴, S. Schwan²;

¹ Martin-Luther-Universität Halle-Wittenberg, Halle (Deutschland),

² Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS, Halle (Deutschland), ³ Universität Stuttgart (Deutschland), ⁴ Charité – Universitätsmedizin Berlin (Deutschland)

Teil- und Totalprothesen werden als klassische Versorgungen bei Verlust von mehreren oder allen Zähnen zur Behandlung verwendet. Patienten mit solchen Versorgungen benutzen häufig Haftcreme. Sie fühlen sich damit in ihrem sozialen Umfeld sicherer, haben weniger Angst, dass sich die Prothese beim Sprechen oder Essen lockert oder sogar herausfällt. Neben dem positiven Einfluss auf die Zufriedenheit der Patienten zeigten Studien auch, dass die Retention, Stabilität und Kauleistungen mit Haftcreme signifikant verbessert werden.

Verantwortlich für die mechanischen Eigenschaften sind dabei sogenannte aktive Polymerkomposite bestehend aus z.B. Karayagummi, Natriumcarboxymethylcellulose und Polyethylenoxid, Acrylamide, Essigpolyvinyl sowie Füllern wie Petrolatum, Mineralöl und Polyethylenoxid, die als Bindemittel in den Kompositen wirken. In Anwesenheit des Speichels quellen die Polymerkomposite und bilden viskoelastische Filme zwischen Gaumen und Prothese.

Die viskoelastischen Eigenschaften wurde für Haftcremes verschiedener Zusammensetzung mittels Rotationsrheometrie in Abhängigkeit der Quellung, der Temperatur und des pH-Wertes für einen repräsentativen Frequenzbereich bestimmt. Über die Approximation dieser Eigenschaften in einem Materialmodell konnte ein optimaler Anwendungsbereich identifiziert werden, in dem die Polymerkomposite neben einer Reduktion der Druckbelastung des Alveolarknochens auch noch die Lastspitzen, die während des Kauens an den Abutment-Zähnen (Pfeilerzähne an denen die Prothese aufgehängt ist) auftreten, reduzieren können.

N. Rull, F. Rueda, P. Frontini

Crack propagation in isoprene rubber: Experiments and simulation

B. Demmel, S. Eibl, T. Förster, M. Johlitz, A. Lion

Diffusion characteristics of fuel like substances in aged nitrile rubber investigated by Gas Chromatography/Mass Spectrometry

S. Sökmen

Methodological characterisation of rubber–filler interactions in silica-filled SBR/BR systems

K. Baar

RPA-Messungen an treibenden Kautschuk-Mischungen

Ü. Tastan, K. Nonnenmacher

Ozone testing for rubbers and cable insulations

M. Jaunich, A. Kömmling, M. Zaghdoudi, M. Weber, D. Wolff

Update on investigations of elastomer O-ring seals at BAM 3.4

A. Shahdehi, M. Alimardani, M. Razzaghi-Kashani, H. Roshanaei

New insights on dry and wet friction of tire tread rubbers having safe aromatic oils

Crack propagation in isoprene rubber: Experiments and simulation

N. Rull, F. Rueda, P. Frontini;

Instituto de Investigaciones en Ciencia y Tecnología de Materiales (INTEMA),
CONICET, Mar del Plata (Argentina)

The failure of a polyisoprene rubber synthetic rubber elastomer with silica aggregate is assessed under mode I of loading. The mechanical behavior was determined using three different load configurations: uniaxial tension, pure shear (with and without crack) and equibiaxial tension. The resistance to fracture is evaluated under pure shear fracture specimens, by means of the integral-J criterion, and the critical value of tear energy.

This work aims to investigate the predictive capacity of different approaches based on finite element calculations to describe rubber failure. Simulation results are contrast with those obtained experimentally from the pure shear test.

The first approach uses the element elimination technique. A quite good agreement between the simulated and physical experiments is found. The chain elongation failure criterion appears as a valid parameter for the simulation. The drawback of this approach relays on that the implementation of the “elimination of material FE model” leads to a great elimination of elements in the tip of the notch at the time of the initiation of the propagation of the damage.

In a second approach, the problem of crack propagation is addressed using FE models of cohesive elements. The difficulty here is related to their implementation which is more complex and requires the correct calibration of the following properties: stiffness, length of cohesive zone, length of the elements and failure criteria of those elements.

Preliminary results shows that it appears that to extract failure parameters from the pure shear tensile experiment such as fracture energy per unit area, and define the tensile-separation criterion is possible.

Apparently, the cohesive elements of zero thickness embedded in the FE mesh seem to be the most appropriate strategy to approach the simulation of the failure of rubbers. This is because such a setup combines the positive aspects of the two previously proposed models: the crack has the ability to propagate along the preferential way and there is no loss of material due to removal of elements. The disadvantage of this method is the long convergence time.

Finally, it can be concluded that although the non-linear mechanical response of the elastomeric materials makes it difficult to predict their failure, a combination between a constitutive model and a suitable failure criterion into a FE model can lead to a successful result.

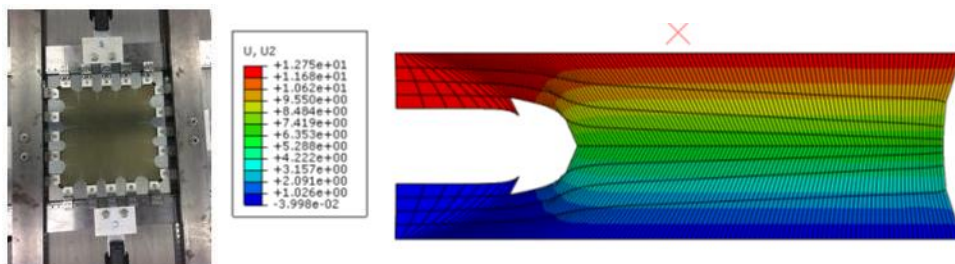


Figure 1:
Biaxial tensile
(left), crack pro-
pagation simu-
lation configura-
tion (right).

Diffusion characteristics of fuel like substances in aged nitrile rubber investigated by Gas Chromatography/Mass Spectrometry

B. Demmel^{1,2}, S. Eibl¹, T. Förster¹, M. Johlitz², A. Lion²;

¹ WIWeB Erding (Germany), ² Universität der Bundeswehr München (Germany)

1 Introduction

Acrylonitrile–butadiene–rubber (NBR) is often used as a material for applications with fuel contact due to its relatively high polarity. In aircrafts with a long service time elastomeric sealings, hoses and linings made of NBR may exhibit ageing with a deterioration of its chemical and mechanical properties. Owing to the introduction of new synthetic fuels in the aircraft sector, it is essential to build a thorough understanding how fuels interact with aged elastomers.

2 Methods

Carbon black filled acrylonitrile–butadiene–rubber (NBR) samples with 18 wt-% acrylonitrile content with ready-to-use formulation were aged at elevated temperatures of 100 and 120 °C in a thermo-oxidative environment. After extraction of the soluble components, classical sorption experiments [1] with model substances like dodecane, decalin and mesitylene as well as Jet A-1 and the synthetic fuel RendiJet were conducted in order to describe the macroscopic behavior. To achieve a deeper understanding of the dynamic diffusion processes of single components furthermore gaschromatography/mass-spectrometry (GC/MS) assisted sorption experiments [2] were carried out.

3 Results

Results show, that there are different sorption periods, in which aromatic, cycloaliphatic and aliphatic compounds redistribute dynamically. It was found that with increasing ageing time there is a discrimination towards sterically demanding and non-aromatic substances. Sorption curves were fitted to Fick's second law of diffusion to obtain diffusion coefficients for quantification.

Literature

- [1] Mathai, A. E., Thomas, S.: Transport of aromatic hydrocarbons through crosslinked nitrile rubber membranes. *J. Macromol. Sci.* 35 (1996) 229–235.
- [2] Blivernitz, A., Förster, T., Eibl, S.: Simultaneous and time resolved investigation of diffusion processes of individual model fuel components in acrylonitrile–butadiene–rubber in the light of swelling phenomena. *Polym. Test.* 70 (2018) 47–56.

Methodological characterisation of rubber–filler interactions in silica-filled SBR/BR systems

**S. Sökmen;
Polymer Service GmbH Merseburg (PSM) (Germany)**

The “green tire” technology gain importance day by day due to its environmental friendly features. Although the substitution of carbon black with precipitated silica in tire treads leads to a simultaneous improvement of rolling resistance and energy saving, silica is not highly compatible with nonpolar rubbers due to its polar nature. Lack of compatibility between silica and nonpolar rubbers results in low rubber–filler interactions which affects various properties of products. In this study, rubber–filler interactions in silica-filled styrene–butadiene rubber (SBR) and butadiene rubber (BR) homopolymer blends were characterized methodologically by rubber layer L concept and dynamic mechanical analysis with the help of rubber process analyzer (RPA). In addition, influence of silanization and polymer chain functionalization on compatibility were examined briefly for silica-filled SBR mixtures. Effects of treated distillate aromatic extract (TDAE) oil content on rubber–filler interactions and viscoelastic properties were investigated in silica-filled SBR mixtures, BR mixtures and SBR/BR binary blends. The experimental results showed a slight improvement in rubber–filler interactions after silanization and polymer functionalization. Viscoelastic properties and rubber–filler interactions were highly dependent on TDAE oil content in silica-filled SBR homopolymer mixtures, BR homopolymer mixtures and SBR/BR binary blends.

RPA-Messungen an treibenden Kautschuk-Mischungen

K. Baar;

Polymer-Technik Elbe GmbH, Lutherstadt Wittenberg (Germany)

1 Einleitung

Bereits 1992 wurde der erste Rubber Process Analyzer (RPA) kommerziell vermarktet (RPA 2000, Alpha Technologies) und fand zunächst vorwiegend in der Reifenindustrie Einsatz. Heute ist das RPA-Gerät eine bekannte, vielseitige Prüfmethode für Kautschuke und deren Mischungen in der gesamten Kautschukbranche.

2 Kurzzusammenfassung

Dieser Beitrag zeigt den Einsatz des RPA in der kautschukverarbeitenden Industrie. Wie auch bei Rotationsrheometern bekannt, wird beim RPA eine oszillierende Messung angewandt, jedoch mit geschlossener Prüfkammer, ähnlich wie bei den bekannten Moving Die Rheometer (MDR). Die Frequenz bzw. die Amplitude sind variabel. Die Temperatur kann während der Messung ebenfalls verändert werden. Durch die große Anzahl der Freiheitsgrade der Messgrößen sind vielfältige Untersuchungen an Polymeren und Mischungen möglich, welche sowohl zur Qualitätssicherung als auch zur Entwicklung von Mischungen eingesetzt werden können. Auch ein möglicherweise notwendiger Polymeraustausch kann mit dem RPA schnell bewertet werden.

Durch die Möglichkeit der Messung des Kammerinnendrucks mit Hilfe eines Drucksensors können treibende Kautschukmischungen und deren Vulkanisationsverhalten sehr gut mit Hilfe des RPA untersucht werden. Die Probenvorbereitung ist für diese Messung ausschlaggebend und muss sorgfältig erfolgen, um reproduzierbare Ergebnisse zu erhalten. Durch die Messung bei verschiedenen Temperaturen und die Untersuchung verschiedener Treibmittel kann auf die Porenentwicklung während des Vulkanisationsprozesses rückgeschlossen werden.

Ozone testing for rubbers and cable insulations

**Ü. Tastan, K. Nonnenmacher;
Anseros Klaus Nonnenmacher GmbH, Tübingen (Germany)**

Industrial plastics have to fulfil a certain number of criteria worldwide. For rubbers, tires and cable insulation, resistance and durability are important parameters for product evaluation. Environmental influences, such as ozone, can strongly influence the durability of these materials. Therefore, ozone test is one of the standard methods for material development and quality assurance of elastomers.

The ozone tests can be carried out in special ozone test-chambers. With this, it is possible to investigate the ozone resistance of all rubber materials under various conditions such as temperature and humidity. The results have great significance for industrial application and allows a prediction of the durability and stability of various elastomers. An example of ozone test chamber SIM7500-TH can be seen in Figure 1. The samples are placed in a closed chamber. The size of the chamber can vary depending on sample size. The use of automation program allows the specified test conditions with defined test parameters, such as ozone concentration, temperature, humidity and the flow rate of ozone–air gas mix in the chamber. The ozone test chambers allow the user to compare a wide range of standards (ISO 1431-1, ASTM D 1149, IEC 60860811-2-1 und DIN 53509) with just one equipment.

The test results of a rubber test for ozone resistance can be seen in Figure 2. The rubber sample is fixed with a bending radius of 90° (same as a real application) and then treated with ozone for total 175 hours in the ozone test chamber. The first cracks can be seen after 39 hours through the window, without opening the test chamber. The results of the tests not only provide information about ozone resistance, but also estimated the durability and stability of various elastomers. This can be done before production, to check whether these materials meet the necessary requirements.



Figure 1: Ozone test chamber SIM7500-TH.

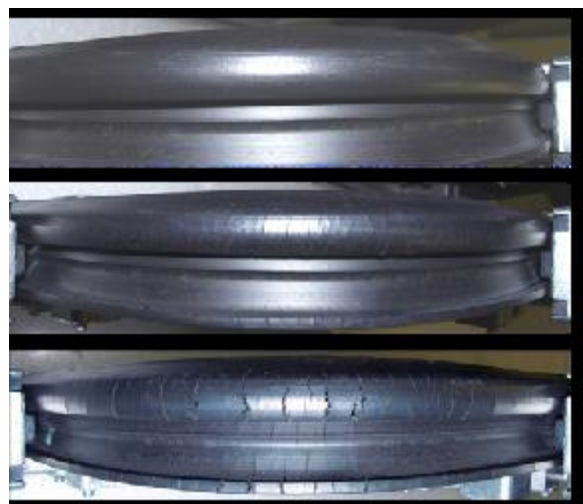


Figure 2: Rubber test for ozone resistance.

Update on investigations of elastomer O-ring seals at BAM 3.4

**M. Jaunich, A. Kömmling, M. Zaghdoudi, M. Weber, D. Wolff;
Bundesanstalt für Materialforschung und -prüfung, Berlin (Germany)**

For many sealing applications rubber O-rings are applied due to their special properties and easy use. In many applications as e.g. in aviation, traffic or process technology the material can be exposed to a variety of conditions during operation as e.g. low temperatures, elevated temperatures and different media. Additionally ageing of seals is an important factor which has an influence on the long term reliability. Therefore, it is important to as well know the material behaviour as to be able to predict the seal performance under those conditions.

In this contribution we would like to discuss different aspects affecting seal performance and the possibility and accuracy of lifetime estimations.

Results of running investigations at the Bundesanstalt für Materialforschung und -prüfung (BAM), Division 3.4 “Safety of Storage Containers” will be presented and discussed.

The work is based on investigations started in 2008 with the investigation of the low temperature behaviour of rubber seals [1, 2], continued with a long term investigation of the ageing behaviour of various seal materials [3–5] and is now including the numerical description of the observed effects [6, 7].

Literature

- [1] Jaunich, M., Tieftemperaturverhalten von Elastomeren im Dichtungseinsatz. BAM Bundesanstalt für Materialforschung und -prüfung. Berlin (2012), p. 1–143.
- [2] Grelle, T., Wolff, D., Jaunich, M.: Temperature-dependent leak tightness of elastomer seals after partial and rapid release of compression. *Polym. Test.* 48 (2015) 44–49.
- [3] Kömmling, A., Jaunich, M., Wolff, D.: Effects of heterogeneous aging in compressed HNBR and EPDM O-ring seals. *Polym. Degrad. Stab.* 126 (2016) 39–46.
- [4] Kömmling, A.: Alterung und Lebensdauervorhersage von O-Ring-Dichtungen. PhD-Thesis, TU Berlin, Berlin (2017), p. 1–121.
- [5] Kömmling, A., Jaunich, M., Pourmand, P., Wolff, D., Hedenqvist, M.: Analysis of O-Ring seal failure under static conditions and determination of end-of-lifetime criterion. *Polymers* 11 (2019) 1251 (19 p.).
- [6] Weber, M., Kömmling, A., Jaunich, M., Wolff, D., Zencker, U., Völzke, H., Schulze, D.: Testing and numerical simulation of elastomeric seals under consideration of time dependent effects, in ASME 2016 Pressure Vessels & Piping Conference (PVP2016). ASME, Vancouver/Canada (2016), Paper 63192 (10 p.).
- [7] Zaghdoudi, M., Kömmling, A., Jaunich, M., Wolff, D.: Scission, cross-linking, and physical relaxation during thermal degradation of elastomers. *Polymers* 11 (2019) 1280 (12 p.).

New insights on dry and wet friction of tire tread rubbers having safe aromatic oils

A. Shahdehi¹, M. Alimardani¹, M. Razzaghi-Kashani¹, H. Roshanaei²;

¹ Polymer Engineering Department, Faculty of Chemical Engineering, Tarbiat Modares University, Tehran (Iran), ² Department of Research and Development, Iran Yasa Tire and Rubber Company, Tehran (Iran)

By substitution of conventional rubber process oils (i.e. DAE: distillate aromatic extract) having high degree of polycyclic aromatic (PCA) content with the safe oils, there have always been a concern that the indispensable tire tread properties are adversely jeopardized. With regard to frictional properties of tire tread, most of previous literature have employed the dynamic properties (loss factor $\tan \delta$ at low temperatures) as indicator of wet grip and predicted an inferior behavior for compounds having safe treated distillate aromatic extract (TDAE) oil. Here, attempts are made to perform direct frictional testing on rubbers having oils with different PCA contents. Mechanisms behind the modification of wet and dry friction were also thoroughly explored by analyzing the adhesion and hysteresis terms of friction. Results indicated that the impact of oil content on coefficient of friction (COF) is more significant than the oil type. The difference in frictional properties of TDAE and DAE samples is in a level that can be readily affected by compound modifications. Interestingly, TDAE samples had a comparable or even better COF than DAE. Exception is for dry friction at high oil loading. The experimental evidence for rubber-oil incompatibility, interfacial work of rubber-counterface adhesion and viscoelastic dissipations support the findings on COF.

R. Gopi, R. Schlimper, R. Schäuble, D. Glatz, C. Stüpp
Characterization of fiber reinforced 3D printed parts

S. Schwan, A. Kumar Majari, A. Krombholz
Calculation of the J-integral for 3D printing samples with respect to creep and the production related anisotropy

K. Panchasara, K. Mehle, A. Ramakrishnan, C. Ludtka, S. Schwan
Modellierung fertigungsbedingter Eigenspannung in 3D-Druck-Bauteilen aus Polyamid

Characterization of fiber reinforced 3D printed parts

R. Gopi¹, R. Schlimper¹, R. Schäuble¹, D. Glatz², C. Stüpp³;

¹ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ² Forschungs-und Beratungszentrum für Maschinen und Energiesysteme e.V., Merseburg (Germany), ³ Brightlands Materials Center (The Netherlands)

Additive Manufacturing (AM), commonly known as 3D Printing is an advanced manufacturing technique which is used to produce parts with complex geometry. Manufacturing a part is simple with 3D printing, it all begins with a simple design of a part with CAD, this design is then sliced into G-codes and these G-codes are conveyed to the 3D printer. The product is manufactured by layer-by-layer deposition of a continuous thermoplastic filament. New development in the Additive Manufacturing sector has been witnessed by the manufacturing of complex fiber-reinforced 3D structures. With increasing need for usage of 3D printing in industries, there is a high need for increasing the reliability of these 3D printed parts. This paper reviews the investigation of Fused Deposition Modeling (FDM) based 3D printed short fiber reinforced polymer structures by first X-ray computer tomography and then tensile testing. Dumbbell shaped specimens have been used for these investigations were produced by 3D printing with varying print process parameters and also the fiber weight fraction. Tested specimens have been compared for the porosity (void content), fiber alignment, fiber volume fraction and also a brief comparison of tensile properties has been done for specimens with different print orientation (Y orientation – 0° raster angle, Z orientation – 90° raster angle). Two sets of specimens were used, one set has been directly printed into a dumbbell shape, another set was milled out of box shaped printed part into a dumbbell shaped specimen. The correlation of the micro-structural properties such as porosity and fiber orientation to the tensile properties has been done. Finally, the effect of the different process parameters on the overall features of the composite has been discussed.

Calculation of the J-integral for 3D printing samples with respect to creep and the production related anisotropy

S. Schwan¹, A. Kumar Majari^{1,2}, A. Krombholz¹;

¹ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ² University of Duisburg-Essen, Institute of Mechanics (Germany)

Extrusion-based additive manufacturing technology, typically known as fused deposition modeling (FDM) is becoming more and more prominent of modern design and manufacturing technology. For the time being, the design of components has been processing using 3D printers with additive manufacturing technology. Nowadays, the materials using for this manufacturing technology are polymers. Polymers are materials fabricated from lengthy, repeating chains of molecules. These substances have particular residences, depending on the type of molecules being bonded. Due to use of polymer materials in fused deposition modeling, notches, delamination of the layers because of the temperature deference are formed inside the components.

Mainly, the aim of this work is to develop a numerical model to calculate the crack propagation parameters in a component with polymer material PA 66 which is manufactured by FDM. To calculate the crack propagation under a monotonic mode I loading J-integral method should be implemented. Moreover, 3D printed components which are manufactured by fused deposition modelling uses polymers, due to the fact that these polymers can go under creep deformation even under room temperature.

Modellierung fertigungsbedingter Eigenspannung in 3D-Druck-Bauteilen aus Polyamid

K. Panchasara^{1,2}, K. Mehle², A. Ramakrishnan¹, C. Ludtka³, S. Schwan¹;

¹ Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS, Halle (Germany), ² Hochschule Merseburg (Germany), ³ University of Florida, Gainesville (USA)

Die Zielstellung der Arbeit ist, den Eigenspannungszustand, der bei der Fertigung von Bauteilen mittels Fused Deposition Modeling (FDM) entstehen kann, zu modellieren. Bedingt durch ungünstige Wärmeleiteigenschaften der im FDM verarbeitbaren Polymere kommt es während des Fertigungsprozesses zu thermisch induzierten Eigenspannungen, die für das spätere Bauteilverhalten von entscheidender Bedeutung sein können.

Dazu wurde der G-Code eines 3D-Druckers zunächst in einen Spalten-Interpreter importiert, so dass er je nach Anforderung neu angeordnet werden kann. Die Neuordnung der Tabelle erfolgt mittels Visual-Basic-Makro. Dieser stellt die Geometriedaten so bereit, dass diese dann mittels eines CAD-Makros (CATIA-Makro) weiterverarbeitet werden können, um ein Bauteil in CATIA zu erzeugen. Vereinfachend wird ein Kreisprofil verwendet, welches über Polymerlinien extrudiert und aufeinandergestapelt wird. Das CAD-Teil wird in ANSYS Workbench importiert. Dort erfolgt die Vernetzung und die zeitlich variierende Zuweisung der Randbedingungen, über die der Druckprozess simuliert wird. Die Berechnungen werden in ANSYS Workbench mit den ANSYS APDL-Befehlen ausgeführt. Dabei wird die Methode der Element-Aktivierung-Deaktivierung in der Simulation verwendet. Die thermische Simulation wird zunächst an einem einfachen Modell (einem einfachen Block) demonstriert und validiert und dann auf das CAD-Teil übertragen. Die Ergebnisse werden aus der Transienten-Analyse importiert und als Lastschritt für die Statisch-Mechanische Analyse verwendet. In der Statisch-Mechanischen Analyse werden die resultierenden Spannungen, Dehnungen und Verformungen für dieses Beispielobjekt mitberechnet.

Das Problem der Verformung ist auf die schnellen Aufheiz- und Abkühlzyklen des Filament-Materials zurückzuführen, die zu einer Akkumulation von Eigenspannungen während des Aufbaus des Teils führen können.

P. Hirsch, T. Theumer

Comparative study on polyamide 11 and polyamide 10.10 as matrix polymers for biogenic wood–plastic composites

P. Bulacios, C. Bernal, E. Pérez

Mechanical performance of bioresorbable maxillofacial implants

K. D. T. Kien, N. V. U. Nhi, H. N. Minh, H. X. Tung, D. Q. Minh

Effects of the reaction time at 150 °C to the phenolic form from cashew nut shell waste

N. Eversmann, A. Krombholz

Insekten-Chitin und Chitosan in biofunktionalen Oberflächen

J. Klehm, T. Theumer, A. Krombholz, K. Thiele, S. Bauer

Multifunktionales Beschichtungssystem auf der Basis pflanzenölbasierter kationischer Tenside

M. Rühl, J. Klehm, A. Krombholz, S. Gomoll, C. Splith, F. Meier

Entwicklung und Bewertung innovativer, robuster und industriell herstellbarer Antifingerprint-beschichtungen aus hybriden Sol-Gel-Nanolacken

M. Bräuer, K. Schneider, H. Scheibner

Mechanical testing of metal–TPU hybrids

P. Hirsch, T. Theumer

Effects of UV aging, water storage and recycling on mechanical properties of biogenic wood–plastic composites from polyamide 11

C. Mayrhofer, H. Plank, H. Gnägi, I. Letofsky-Papst

Ultramicrotomy of polymers at its best: Ultra sonic

P. Gautam, K. Shrestha, J. Giri, R. Adhikari

Morphology and degradation behavior of ternary blends comprising PLA, PBAT and natural fibers

M. Busch, T. Theumer, I. Jahn, A. Krombholz

Investigation of the mechanical behavior of partially electron beam cross-linked polyamide

S. M. Nagaraja, M. Beiner

Different contributions to dissipation in NR and NR–BR blend composites filled with carbon black particles

B. Tillner, S. Lehmann, I. Jahn, J. Celevics, M. Zschoyge

Continuous reinforced polyamide composite materials with unidirectional fiber orientation

T. Wagner, M. Neul, N. Teuscher, M. Wießner, A. Krombholz, R. Schlimper

Entwicklung eines automatisierten Druck-Schmelz-Analysenverfahrens (DSV) für Kunststoffe und Kunststoffgemische zur Ableitung qualitativer und quantitativer Aussagen hinsichtlich der Stoffbeschaffenheit und Verarbeitungsfähigkeit

T. Theumer

Multifunktionales Beschichtungssystem auf der Basis pflanzenölbasierter kationischer Tenside und Lignine

R. Lach, L. Castro Key, B. Langer, W. Grellmann, B. Tillner, N. Teuscher, S. Celevics, I. Jahn, P. Michel

Fracture mechanics investigations of unidirectionally fibre-reinforced polymer tapes

S. Sökmen, K. Oßwald, S. Ilisch, K. Reincke

Influence of TDAE oil on silica–rubber interactions in SBR/BR homopolymer and binary blends

J. Posch, K. Oßwald, B. Langer, K. Reincke

Evaluation of the fracture behavior of PMMA materials with respect to environmental stress cracking

R. Schlegel, H. Budde, U. Wendler, N. Mahmood, S. Henning, M. Beiner
Adjusting PI–PS-based multigraft copolymers properties via blending

O. Aghazada, K. Oßwald, K. Reincke

Influence of the antioxidant type on the mechanical and viscoelastic properties of silica-filled NR and SBR vulcanizates after thermo-oxidative aging

M. Seitz, S. Schröter

Katalytische Depolymerisation von polyolefinischen Kunststoffabfällen

Comparative study on polyamide 11 and polyamide 10.10 as matrix polymers for biogenic wood–plastic composites

P. Hirsch^{1,2}, T. Theumer¹;

¹ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ² Merseburg University of Applied Sciences (Germany)

Biogenic wood–plastic composites from bio-based polymers and wood fibers (bio-WPC) provide an improved sustainability and carbon footprint compared to conventional composites. Recently, such bio-WPCs from bio-based polyamides have gained major interest due to their high mechanical and thermo-mechanical properties. In this study, the resulting mechanical and thermo-mechanical properties of two bio-WPCs from polyamide 11 (PA11) and polyamide 10.10 (PA10.10) with a content of 30 wt.% of beech wood particles (BP) processed by twin-screw compounding and subsequent injection molding were analyzed by tensile testing and dynamic mechanical analysis. For this, two comparable grades of PA10.10 and PA11 were used. It was found that the elastic modulus, tensile strength and the temperature dependent storage modulus of the PA10.10/BP bio-WPC are significantly higher as compared to the PA11/BP material. However, these findings are not addressed to the minor different values of the initial properties of the compared PA10.10 and PA11 but to the different amount of amino groups per repeating unit in these polymers leading to a better adhesion of the matrix and the wood fiber and consequently better mechanical and thermo-mechanical properties of the PA10.10/BP. In consequence, bio-WPCs from PA10.10 should be considered as an interesting alternative with a tendency to higher mechanical and thermo-mechanical properties in comparison to bio-WPCs from PA11.

Literature

- [1] Zierdt, P., Theumer, T., Kulkarni, G., Däumlich, V., Klehm, J., Hirsch, U., Weber, A.: Sustainable wood-plastic composites from bio-based polyamide 11 and chemically modified beech fibers. *Sustain. Mater. Technol.* 6 (2015) 6–14.
- [2] Zierdt, P., Mitzner, E., Gomoll, A., Theumer, T., Lieske, A.: Synthesis of polyamide 6/11 copolymers and their use as matrix polymer in wood-plastic composites. *J. Appl. Polym. Sci.* 133 (2016) 44155
- [3] Zierdt, P., Kulkarni, G., Theumer, T.: Mechanical and thermo-mechanical properties of discontinuously and continuously processed biogenic wood–plastic composites from polyamide 11 and chemically modified beech particles. *Macromol. Symp.* 373 (2017) 118–124.

Mechanical performance of bioresorbable maxillofacial implants

P. Bulacios^{1,2}, C. Bernal^{1,3}, E. Pérez^{2,3};

¹ Institute of Technology in Polymers and Nanotechnology, Buenos Aires (Argentina), ² National Institute of Industrial Technology (INTI), Buenos Aires (Argentina), ³ National Scientific and Technical Research Council, Buenos Aires (Argentina)

Bioresorbable implants for maxillofacial applications has been extensively used in the last decades due to the several advantages observed over titanium fixation systems [1]. Implants based on pure polylactic acid (PLA) or copolymers with polyglycolic acid (PGA) have been successfully applied [1–3]. Independently of the prominent reported data, these polymers exhibit some limited mechanical performance (resistance, fracture toughness, among others) [1]. This disadvantage can be avoided by processing of composite materials based on PLA, PLA/PGA reinforced with calcium phosphates [1, 4]. In the present work, the mechanical performance of maxillofacial implant system was investigated. The components (plate and screw) were fabricated with PLA or PLA-PGA reinforced with different content of β -tricalcium phosphate (TCP).

In general, uniaxial tensile and 3-point bending tests of plates displayed semi-brittle behavior with a precipitous drop of load behind the maximum value. In addition, the maximal strengths were observed for both matrices reinforced with 1 wt.% of TCP. In a similar way, cantilever test of the screws displayed maximal strengths for both matrices reinforced with 1 wt.% of TCP. On the other hand, torque test exhibited improved performance for the PLA based materials compared to the PLA/PGA ones. The obtained results suggest the accuracy to produce composite materials based on PLA, PLA/PGA reinforced with TCP to obtain bioresorbable implants with enhanced performance. Particularly, the torque behavior of PLA/PGA should be considered to warrant the long term success of maxillofacial implant systems.

Literature

- [1] Kanno, T., Sukegawa, S., Furuki, Y., Nariai, Y., Sekine, J.: Overview of innovative advances in bioresorbable plate systems for oral and maxillofacial surgery. *Jpn. Dent. Sci. Rev.* 54 (2018) 127–138.
- [2] Armentano, I., Dottori, M., Fortunati, E., Mattioli, S., Kenny, J. M.: Biodegradable polymer matrix nanocomposites for tissue engineering: A review. *Polym. Degrad. Stab.* 95 (2010) 2126–2146.
- [3] Narayanan, G., Vernekar, V. N., Kuyinua, E. L., Laurencin, C. T.: Poly (lactic acid)-based biomaterials for orthopaedic regenerative engineering. *Adv. Drug Deliv. Rev.* 107 (2016) 247–276.
- [4] Shikinami, Y., Okuno, M.: Bioresorbable devices made of forged composites of hydroxyapatite (HA) particles and poly L-lactide (PLLA): Practical properties of miniscrews and miniplates. *Biomaterials* 22 (2001) 3197–3211.

Effect of the reaction time at 150 °C to the phenolic form from cashew nut shell waste

**K. D. T. Kien, N. V. U. Nhi, H. N. Minh, H. X. Tung, D. Q. Minh;
Ho Chi Minh City University of Technology – VNU-HCM, Hochiminh City
(Vietnam)**

A phenol resin is needed as a binder to make wood ceramics from cashew nut shell waste (CNSW). In this study, this phenol resin was synthesized from the mixture of the CNSW powder, phenol, and sulfuric acid. The performance of the phenolic resin in the reaction was evaluated according to the residue of the unreacted CNSW. The reaction process at 150 °C was investigated at different reaction time intervals. The average number of molecules (M_n) and the average molecular mass (M_w) of the product were determined by gel permeation chromatography (GPC). The molecular structure and functional groups of phenolic resins were determined by infrared spectrometry (FTIR).

Keywords: phenolic resin, liquefied wood, cashew nut shell waste (CNSW).

Insekten-Chitin und Chitosan in biofunktionalen Oberflächen

**N. Eversmann, A. Krombholz;
Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS,
Halle (Germany)**

Im Projekt sollen neue Anwendungsmöglichkeiten für Chitosan untersucht und entwickelt werden. Dieses kann durch Deacetylierung von Chitin aus den Larven der schwarzen Soldatenfliege gewonnen werden. Das allergische Potential ist dabei niedriger als bei Chitosanen aus Krabbenschalen oder Pilzmycel. Ein biogenes Epoxidharz dient als Basis für die Entwicklung einer funktionalisierten Oberflächenbeschichtung. Chitosane und ihre Derivate werden als antimikrobielle/antifungizide Additive eingearbeitet. Obwohl Chitosan eine hohe Fettbindekraft aufweist, ist es in pflanzlichen Ölen nicht löslich. Chitosan-Derivate wie Chitosan-HCl oder Chitosan-Glutamat bilden in Wasser hochviskose gelartige Lösungen. Für tropffreie Beschichtungen an senkrechten Flächen ist die hohe Viskosität vorteilhaft. Weiterhin kann Chitin als Ausgangsstoff für die Herstellung von Härtersubstanzen, z.B. Furandicarbonsäure verwendet werden [1, 2]. Die Chitosan-Derivate beeinflussen die Eigenschaften des Grundharzes dabei sehr stark. Es werden Farbe, Transparenz und Reaktivität verändert. Die neu entwickelten Epoxidmischungen werden hinsichtlich ihres Verarbeitungs- und Aushärteverhaltens, sowie hinsichtlich der Delaminierungsneigung und Rissbildung beurteilt. Die Beschichtung wird auf Haltbarkeit und Langlebigkeit hin optimiert. Die Chitosane sollen oberflächenaktiv fest eingebunden sein und durch Reinigung und Desinfektion nicht ausgewaschen oder inaktiviert werden.



Bild 1: Pflanzenölbasierte Oberflächenbeschichtung mit Chitosan.

Literatur

- [1] Yu, S., Zang, H., Chen, S., Jiang, Y., Yan, B., Cheng, B.: Efficient conversion of chitin biomass into 5-hydroxymethylfurfural over metal salts catalysts in dimethyl sulfoxide - water mixture under hydrothermal conditions. Polym. Degrad. Stab. 134 (2016) 105–114.
- [2] Sajid, M., Zhao, X., Liu, D.: Production of 2,5-furandicarboxylic acid (FDCA) from 5-hydroxymethylfurfural (HMF): Recent progress focusing on the chemical-catalytic routes. Green Chem. 20 (2018) 5427–5453.

Optimierter Flammenschutz an einem Naturfaserverbund

**S. Wüstenhagen, N. Eversmann, A. Krombholz;
Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS,
Halle (Germany)**

Naturfaser-Verbundkonstruktionen (NFK) zeigen gute mechanische Eigenschaften, bei einer vergleichsweise geringen spezifischen Dichte. In der Bilanzierung der Umwelteinwirkungen aus Produktion und Bauteileinsatz (Life Cycle Assessment – LCA) ergeben sich vergleichsweise niedrige Emissionen von Treibhausgasen (Green House Gas Emission – GHG) für den Einsatz von NFK. Das Flammschutz- und Brandverhalten von NFK stellt aktuell eine Herausforderung für bestimmte Einsatzfelder dar.

In einem aktuell laufenden Projekt „CelPreg“ werden Strategien zum verbesserten Flammschutz und Brandverhalten (Fire Smoke Toxicity – FST) für NFK konzipiert, getestet und optimiert. Es kommt ein Epoxidharz-System als Matrixmaterial zum Einsatz, welches zu 90 % aus erneuerbaren Quellen erzeugt wurde. Für die Konsolidierung der imprägnierten Fasermaterialien werden Niedrigdruckverfahren betrachtet (Out of Autoclave – OoA). Das Poster zeigt die ersten Ergebnisse zu erfolgreich flammenschutzmodifizierten Naturfaserverbunden, in Bezug zu den spezifischen mechanischen Eigenschaften.

Multifunktionales Beschichtungssystem auf der Basis pflanzenölbasierter kationischer Tenside

J. Klehm¹, T. Theumer¹, A. Krombholz¹, K. Thiele², S. Bauer³;

¹ Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS, Halle (Deutschland), ² Fraunhofer-Zentrum für Chemisch-Biotechnologische Prozesse CBP, Leuna (Deutschland), ³ bausion Straßenbau-Produkte GmbH, Landsberg (Deutschland)

Ziel des Forschungs- und Entwicklungsvorhabens ist die Entwicklung eines multifunktionalen Beschichtungssystems auf der Basis pflanzenölbasierter kationischer Tenside. Das Beschichtungssystem basiert dabei auf einem kolloidalen Grundsystem und weiteren Additiven. Um eine stabile Emulsion aus Wasser und dem polymeren Verbund bilden zu können, müssen aktuell petrochemische Emulgatoren und Stabilisatoren zugesetzt werden. Im Rahmen des Forschungsvorhabens werden die bisherigen petrobasierten kationischen Tenside durch pflanzenölbasierte Varianten ersetzt und zusätzlich chemisch funktionalisiert. Des Weiteren wird durch den Zusatz von unterschiedlichen Ligninvarianten (beispielsweise Kraftlignin, OS-Lignin) das kolloidale Grundsystem angepasst, um den Anteil der biobasierten Rohstoffe im Beschichtungssystem signifikant zu erhöhen. Damit sinkt der Gehalt der hochmolekularen Aromaten aus petrochemischen Quellen. Ein zusätzlicher Effekt, welcher durch den Zusatz der Lignine erreicht werden kann, ist eine höhere Brandfestigkeit der gesamten Beschichtung. Aktuell erfolgt die morphologische Charakterisierung der Ligninpulver und die Untersuchung des Einflusses der Partikelgeometrie auf das Mischungsverhalten im Verbund. Die Compoundierung erfolgt dabei mittels eines innovativen Ultraschallverfahrens. Die hergestellten Verbundsysteme werden dann sowohl mittels standardisierter Prüfverfahren wie Nadelpenetration, Bestimmung des Erweichungspunktes als auch chemisch-analytisch charakterisiert, um die Struktur-Eigenschafts-Beziehungen untersuchen zu können.



Bild 1: Lichtmikroskopische Aufnahme einer stabilen Wasser-Polymer-Emulsion mit petrobasierten kationischem Tensid.

Entwicklung und Bewertung innovativer, robuster und industriell herstellbarer Antifingerprintbeschichtungen aus hybriden Sol-Gel-Nanolacken

M. Rühl¹, J. Klehm¹, A. Krombholz¹, S. Gomoll², C. Splith², F. Meier²;

¹ Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS, Halle (Germany), ² FEW Chemicals GmbH, Bitterfeld-Wolfen (Germany)

Für die Entwicklung eines industrietauglichen und hochwirksamen hybriden Sol-Gel-Nanolacks für funktionelle Beschichtungen mit Antifingerprint-Effekt (AFP) wird für sprühbare Lacke mit Nano- und Mikropartikeln, welche eine Vernetzung bei Raumtemperatur bis maximal 180 °C mit Härtingszeiten kleiner 15 Minuten aufweisen, eine Prüfvorrichtung entwickelt.

Ausgehend von einer künstlichen Fingerabdruck-Lösung (FADL) und einem strukturierten Fingerabdruck-Stempel soll eine objektivierbare Prüfvorrichtung entwickelt, aufgebaut und systematisch angewendet werden. Als wesentlich wird dabei das Arbeiten mit konstantem Anpressdruck, definierter Druckzeit und Abzugsgeschwindigkeit angesehen.

Die Prüfvorrichtung soll die sich wiederholenden und reproduzierbaren künstlichen Fingerabdrücke auf die beschichteten Oberflächen setzen, diese dann über ein Kamera- und Beleuchtungssystem bildlich dokumentieren. Für eine objektive Bewertung der Abdrücke werden die Bildaufnahmen bei verschiedenen Wellenlängen durchgeführt. Eine anschließende Auswertung der Bilder erfolgt mit Hilfe eines Algorithmus, welcher die Vergleichbarkeit der künstlichen Fingerabdrücke auf den verschiedenen Oberflächen gewähren soll.

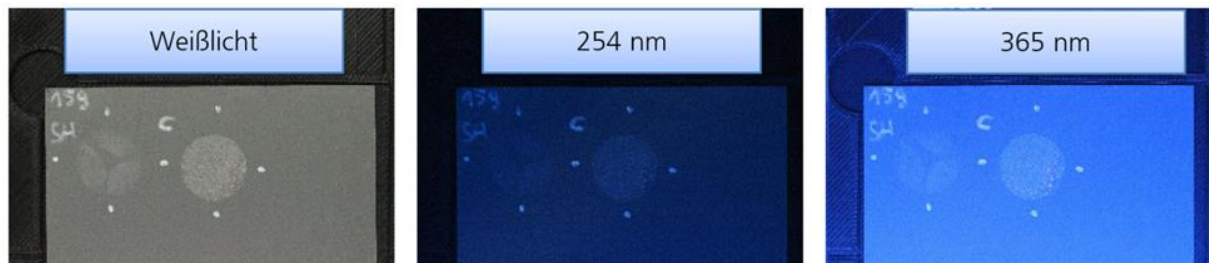


Bild 1: Aufgenommene Bilder eines künstlichen Fingerabdrucks auf einer beschichteten Oberfläche. Der linke Abdruck erfolgte mit synthetischen Handschweiß (SH), der rechte Abdruck mit einer Sonnencreme (C).

Mechanical testing of metal–TPU hybrids

**M. Bräuer, K. Schneider, H. Scheibner;
Leibniz-Institut für Polymerforschung Dresden e.V. (Germany)**

Metal–Plastic Hybrids, Rigid Flexible Composites and inflexible composites with an elastic interlayer are used as important components in lightweight constructions in many cases. The application of these composites is limited by the bond strength, i.e. the adhesion between the different components. The bond strength can be estimated under different modes of load. Recently established methods for the characterization of a flexible component on a stiff component as well as a flexible interlayer between stiff components were presented and discussed with respect to the mechanical (stiffness) and geometrical (thickness) parameters of the flexible component.

Keywords: Rigid flexible composites, Metal–TPU hybrid, Elastic interface layer bond strength, Peel test shear test, Energy release rate, Shear strength, G_{lc} , G_{ext} , G_{adh} .

Literature

- [1] Bräuer, M., Lönnecke, K., Lauke, B., Schneider, K.: Peel test for practical application: Determination of the bond strength of two component rigid flexible composites. KGK Kautschuk Gummi Kunststoffe 70 (2017) 22–26.
- [2] Bräuer, M., Lehmann†, D., Lönnecke, K., Reichert, V., Schneider K.: Metal–plastic-composites with an elastic intermediate layer: Production by means of press-injection moulding process or sandwich-injection moulding. KGK Kautschuk Gummi Kunststoffe 73 (2020) 24–30.

Effects of UV aging, water storage and recycling on mechanical properties of biogenic wood–plastic composites from polyamide 11

P. Hirsch^{1,2}, T. Theumer¹;

¹ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ² Merseburg University of Applied Sciences (Germany)

Biogenic wood–plastic composites from bio-based polymers and wood fibers (bio-WPC) provide an improved sustainability and carbon footprint compared to conventional composites. Within this study, the weather stability of a bio-WPC made of bio-based polyamide 11 (PA11) and 30 wt.% of beech wood particles (BP) was evaluated. For this purpose, injection molded test specimens were exposed to UV weathering according to ISO 4892 and water storage according to EN 317 and the resulting mechanical properties after 8, 16, 32 and 64 days of weathering were determined in the tensile test according to ISO 527. It was found that UV weathering had no significant effects on the modulus of elasticity and tensile strength of the bio-WPC. With increasing weathering time, however, a slight trend towards embrittlement of the material was found, which was reflected in the decreasing characteristic values of the elongation at break. A white/gray coloration of the material was observed with increasing weathering duration. It was also found that the water absorbed by the hydrophilic beech wood particles and by the PA11 matrix during water storage acts as a plasticizer and results in a decrease in stiffness and strength as well as an increase in the elongation at break. The second focus of this study was the evaluation of the mechanical properties after recycling. For this purpose, injection molded test specimens were crushed with a laboratory impact mill and injection molded again to standard test specimens. The resulting mechanical properties were evaluated by tensile test according to ISO 527 after conditioning according to ISO 1110. It was found that the recycling leads to a reduction of the modulus of elasticity and tensile strength by about 20 % compared to the original bio-WPC, while the elongation at break increases slightly. This can be attributed to a mechanical and thermo-oxidative degradation of the beech wood particles but also of the PA11 through recycling by crushing with a hammer mill and renewed injection molding. A corresponding drop in stiffness and strength is typical of recycled composite materials. Overall, the recycled bio-WPC therefore has a good level of mechanical properties after recycling.

Ultramicrotomy of polymers at its best: Ultra sonic

C. Mayrhofer¹, H. Plank^{1,2}, H. Gnägi³, I. Letofsky-Papst^{1,2};

¹ Graz Centre for Electron Microscopy (Austria), ² Institute of Electron Microscopy and Nanoanalysis, Graz University of Technology (Austria), ³ Diatome Ltd, Nidau (Switzerland)

The patented ultra sonic [1, 2] knife allows the cutting of ultrathin sections free of compression as well as absolutely flat block-faces. Also, the elimination of cyclic compression and relaxation during slicing results in much more homogeneous block-face surfaces for AFM, as well as in thinner sections for TEM investigations (slice thickness <20 nm) [3]. Excellent results are achieved with rigid polymers such as PS, PMMA, ABS, HIPS, modified PP and blends of various kind at room temperature; for soft polymers outstanding results in TEM and AFM analyses were achieved by using the cryo sonic that is currently being developed by Diatome (Figure 1).

Felmi-ZFE designed optimal holders to measure samples in the microscopes without rec-lamping the sample after microtomy.

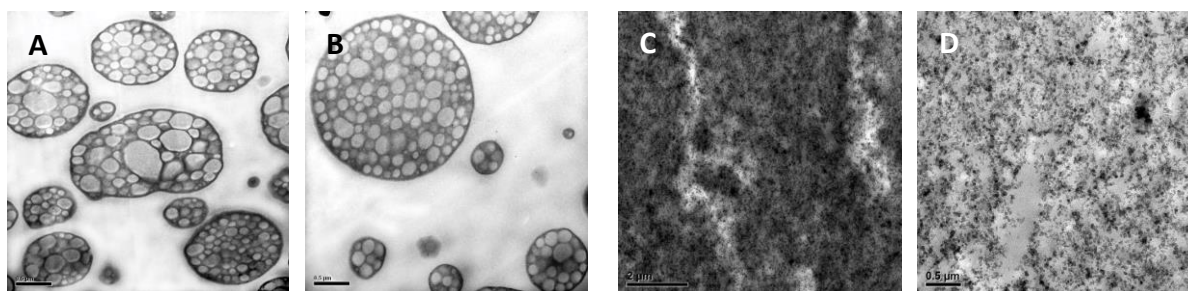


Figure 1: Left: Room temperature: Comparison HIPS without (A) and with oscillation (B). Right: Cryo: Comparison vulcanizates histo cryo (C) and cryo sonic (D).

Literature

- [1] Studer, D., Gnaegi, H.: Minimal compression of ultrathin sections with use of an oscillating diamond knife. *Journal of Microscopy* 197 (2000) 94–100.
- [2] Michler, G. H., Lebek, W.: *Ultramikrotomie in der Materialforschung*. Carl Hanser Verlag, München (2004).
- [3] Mayrhofer, C., Zankel, A., Plank, H., Kothleitner, G.: Tips and tricks in ultramicrotomy. *Microscopy Conference 2013 (MC13)*, Regensburg (25.–30.08.2013), In: *Instrumentation and Methods. Materials Science* (2013) 299–300.

Morphology and degradation behavior of ternary blends comprising PLA, PBAT and natural fibers

P. Gautam^{1,2,3}, K. Shrestha³, J. Giri^{1,4}, R. Adhikari^{1,3};

¹ Central Department of Chemistry, Tribhuvan University (TU), Kathmandu (Nepal), ² School of Engineering, Pokhara University, Lekhnath, Kaski, Pokhara (Nepal), ³ Research Centre for Applied Science and Technology (RECAST), TU, Kathmandu (Nepal), ⁴ Tri-Chandra Multiple Campus, TU, Ghantaghar, Kathmandu (Nepal)

Every year 350–400 million tons of plastics are being produced for various applications and thus polymers have become indispensable part of human life. However, the resistance towards degradation and poor recyclability have become the major hurdles for the waste produced after their end use. Therefore, biobased eco-friendly natural fibres reinforced degradable polymer composites are one of the alternatives used for green practices [1–3]. In this work, biodegradable copolymer polymer blends comprising polylactic acid (PLA) and poly(butylene adipate-co-terephthalate (PBAT) are compounded in different proportions with carpentry wastes (Khayer dust) with suitable chemical treatments. The developed composites were then exposed to various environmental conditions namely; water, cultivated soil, and compost soil for a period up to six months. After exposure, the composites were visually inspected to find signs of crack formation, discoloration and percentage weight loss that indicated the onset of biodegradation [1–5]. In the present investigation, the soil bacteria and fungi were isolated from plastic contaminated soil sample. The bacteria identified were *Bacillus subtilis* and the fungi identified were *Aspergillus niger*. The tests performed were Gram's staining and biochemical analysis for bacteria and macroscopic and microscopic Lacto phenol cotton blue test for fungi.

Literature

- [1] Kim, H.-S., Kim, H.-J., Lee, J.-W., Choi, I.-G.: Biodegradability of bio-flour filled biodegradable poly(butylene succinate) bio-composites in natural and compost soil. *Polym. Degrad. Stab.* 91 (2006) 1117–1127.
- [2] Giri, J., Lach, R., Grellmann, W., Susan, M. A. B. H., Saiter, J.-M., Henning, S., Katiyar, V., Adhikari, R.: Compostable composites of wheat stalk micro- and nanocrystalline cellulose and poly(butylene adipate-co-terephthalate): Surface properties and degradation behavior. *J. Appl. Polym. Sci.* 136 (2019) 48149–48160.
- [3] Rudnik, E.: *Compostable Polymer Materials*. 2nd Ed., Elsevier Publication, Cambridge/USA (2019).
- [4] Tille, P. M.: *Bailey & Scott's Diagnostic Microbiology*. 13th Ed., LabMedicine, Oxford University Press, Oxford (2013).
- [5] Alo, M. N., Egbule, U. C. C., Orji, J. O., Aneke, C. J.: Microbiological analysis of soil from Onu-Ebonyi contaminated with inorganic fertilizer. *Am. J. Infect. Dis.* 1 (2013) 70–73.

Investigation of the mechanical behavior of partially electron beam cross-linked polyamide

M. Busch, T. Theumer, I. Jahn, A. Krombholz;

Fraunhofer Institute for Microstructure of Materials and Systems (IMWS),
Halle (Germany)

The cross-linking of plastic moulded parts by electron irradiation is used already for a long time to improve their properties for various practical applications. For applications with high impact toughness requirements it is state-of-the-art to blend the polyamide-compound with toughness modifiers, e. g. grafted polyolefins with low glass transition temperature [1]. Increasingly, reactor special products are synthesized as the product INFUSE of the Dow Chemical company, which consists of a combination of PE-HD blocks and ethylene–octene copolymer with tailor-made modifier properties (Figure 1).

Due to their rubber-like behaviour the toughness modifiers usually perform considerably lower heat distortion temperatures compared to the polyamide. To improve this, an electron beam radiation cross-linking of impact resistant modified polyamide is carried out in such a way that only the toughness modifiers are cross-linked while the polyamide matrix is preferably not affected by the radiation. The influences of the used toughness modifiers, of cross-linking additives and of the radiation conditions on the properties were systematically investigated.

It could be demonstrated that a moderate increase of the heat distortion temperature by maintained toughness could be achieved, but although a cross-linking of the polyamide did not prevail the processability dropped down drastically.

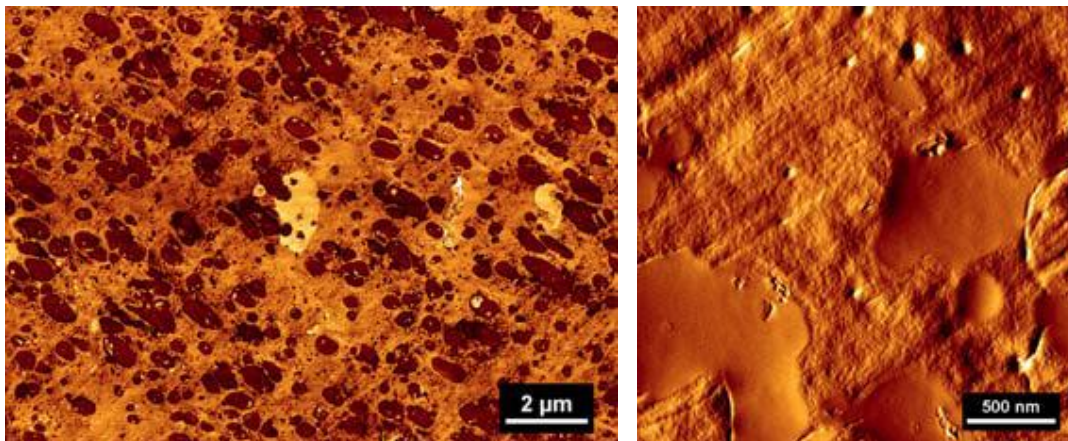


Figure 1: Distribution of toughness modifier in polyamide by atomic force microscopy.

Literature

- [1] Tomova, D.: Ternäre Polymerblends auf Polyamidbasis mit funktionalisierten Elastomeren. Martin-Luther-Universität Halle-Wittenberg, Dissertation, Shaker Verlag, Aachen (2001).

Different contributions to dissipation in NR and NR–BR blend composites filled with carbon black particles

S. M. Nagaraja^{1,2}, M. Beiner^{1,2};

¹ Fraunhofer Institute for Microstructure of Materials and Systems (IMWS), Halle (Germany),
Marin-Luther-Universität Halle-Wittenberg, Naturwissenschaftliche Fakultät II, Halle (Germany)

Mechanical properties characterized by dynamic mechanical analysis of NR and NR–BR blend composites filled with carbon black particles are presented in this poster. The focus is to understand the molecular origin to dissipation in rubber nanocomposite. The loss modulus (G'') data dependent on dynamic shear strain amplitude (γ) is systematically studied by varying the carbon black content and measurement temperature. The modified Kraus equation approximates the experimental data $G''(\gamma)$ adequately and identify three different contributions to dissipation [1]. Two among the three contributions are related to filler network formed above the percolation threshold of carbon black loading and are temperature dependent. This result further suggests the earlier conclusion derived from reinforcement that immobilized rubber formed due to physical adsorption of rubber segments on the filler particles acts as a bridge between the filler particles/aggregates in a percolated filler network. The physical state of these bridges is glassy and it softens sequentially at temperatures several 10 K above the bulk rubber glass temperature (T_g) inducing the viscoelastic nature to the filler network [2]. There are the three identified contributions to dissipation in the light of this physical picture. (i) At small strain amplitude the dissipation contributions $\Delta G''_D$ due to deformation of intact glassy bridges in filler network do dominate, (ii) dissipation at the intermediate strain amplitude $\Delta G''_F$ is mainly due to breaking of glassy bridges, and (iii) contributions to dissipation which are filler network independent remain at very large strain amplitudes [1]. The consistency of this physical picture with experimental findings further suggest that that chemical composition of the glassy bridge and topology of the filler network have a strong influence on the dissipative properties of nanocomposites [3, 4].

Literature

- [1] Nagaraja, S. M., Mujtaba, A., Beiner, M.: Quantification of different contributions to dissipation in elastomer nanoparticle composites. *Polymer* 111 (2017) 48–52.
- [2] Mujtaba, A., Keller, M., Ilisch, S., Radusch, H.-J., Thurn-Albrecht, T., Saalwächter, K., Beiner, M.: Mechanical properties and cross-link density of styrene–butadiene model composites containing fillers with bimodal particle-size distribution. *Macromolecules* 45 (2012) 6504–6515.
- [3] Aguiar da Silva, C., Nagaraja, S. M., Weydert, M., Beiner, M.: Diblock-copolymer-based composites for tire-tread applications with improved filler network topology. *ACS Appl. Nano Mater.* 1 (2018) 1003–1008.
- [4] Nagaraja, S. M., Mujtaba, A., Beiner, M.: Filler network contribution to reinforcement and dissipation: Natural rubber vs natural rubber–butadiene blends filled with carbon-black (under submission).

Continuous reinforced polyamide composite materials with unidirectional fiber orientation

B. Tillner¹, S. Lehmann², I. Jahn¹, J. Celevics¹, M. Zscheyge¹;

¹ Fraunhofer Institute for Microstructure of Materials and Systems IMWS, Halle (Germany), ² DOMO Engineering Plastics GmbH, Leuna (Germany)

Fiber reinforced composites especially with continuous unidirectional fiber orientation (UD tapes) offer an extraordinary lightweight potential due to the combination of the high anisotropic material with a low material density. These UD tapes consist of a thermoplastic matrix polymer and continuous fiber reinforcements. An efficient way to produce large amounts of tape material with high quality standards is the direct melt impregnation. Due to the aim to utilize the UD tapes for high load bearing applications there is a need to create high fiber contents in combination with optimized fiber matrix compatibility. To reach the goal of highest utilization of the fiber reinforcement inside the UD tape a complete infiltration of thermoplastic melt inside the fiber bundles is necessary.

In the present study an optimization on the direct melt impregnation of glass fiber-polyamide UD tapes is shown. The modular setup of the impregnation tool can give the insight view of the dependencies of the impregnation time and the acting forces on the fiber matrix system. Further investigation points are the matrix material and the influence on the quality of the UD tapes. The main developments are the wetting behavior of the fibers by influencing the melt viscosity and the surface tension of the matrix and the interaction between fibers and matrix, which directly correlates with the strength properties of the material. Furthermore an optimization of the process parameters was done to reach increased fiber content of 50 vol% inside the UD tape and improve the process efficiency.

The second part of this research project focuses on the development and manufacturing of tape based lightweight structures [1]. This contains on the one hand, the research on automatable and reproducible processes for realizing high performance components via hybrid injection molding. This economical process enables in cycles times of 1 minute the thermoforming of the tape laminates and the functionalization with injection molding material. On the other hand, a tape based lightweight structure need a reliable design. This includes a detailed material characterization and deep material understanding as well as specific fiber composite material modelling for the structure design including characteristics of the thermoplastic matrix material.

Literature

- [1] Zscheyge, M., Michel, P., Jahn, I.: Bauteile in thermoplastischer UD Endlosfaserverstärkung für die Automobilindustrie. In: 4. Leichtbau Tagung, Fraunhofer Allianz Leichtbau, Oberhausen (11./12.02.2015).

Entwicklung eines automatisierten Druck-Schmelz-Analysenverfahrens (DSV) für Kunststoffe und Kunststoffgemische zur Ableitung qualitativer und quantitativer Aussagen hinsichtlich der Stoffbeschaffenheit und Verarbeitungsfähigkeit

**T. Wagner¹, M. Neul², N. Teuscher¹, M. Wießner¹, A. Krombholz¹,
R. Schlimper¹;**

**¹ Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS,
Halle (Germany), ² Lubey AG, Halle (Germany)**

Um der Herausforderung des Wandels von einer Durchflusswirtschaft zur nachhaltigen und umweltverträglichen Kreislaufwirtschaft gerecht werden zu können, kann auf ein effizientes Recycling von Wertstoffen im Allgemeinen und von Kunststoffabfällen im Besonderen nicht verzichtet werden. In den derzeitigen Wertschöpfungsketten vom Kunststoffabfall bis zum Sekundärprodukt treten grundsätzliche Probleme bei der Analyse der stofflichen Zusammensetzung und des Zustandes der Abfälle und ihrer Verarbeitungsstufen auf. Meist wissen weder die Lieferanten noch die Empfänger, um welche Stoffe es sich genau handelt und kennen deren Zusammensetzung nicht. Bisher übliche Analysenverfahren erweisen sich zunehmend als unzureichend, zu aufwendig und zu langsam oder werden aus Kostengründen gar nicht durchgeführt. Zudem sind die durch die europäische Abfallgesetzgebung zu erwartenden höheren Anforderungen an das Kunststoffrecycling so hoch und die neuesten Erkenntnisse der Umweltanalytik, wie z.B. zur Mikroplastik in den Meeren und in der Nahrungskette bis zum Menschen so alarmierend, dass hier großer Handlungsbedarf besteht. So lassen sich die geforderten höheren Quoten des werkstoff- und rohstoffbezogenen Recyclings und die Minimierung der thermischen Verwertung in Müllverbrennungsanlagen und der Deponierung mittel- und langfristig nur erreichen, wenn das wertmindernde Down-Recycling minimiert und die Erfassung und Sortierung der Kunststoffabfälle verbessert werden. Ziel muss es sein, die Quoten des werkstoff- und des rohstofflichen Recyclings zu erhöhen.

Dies und die ebenso wichtige, optimale Zuordnung der Kunststoffabfälle zu den vorhandenen Recyclingverfahren erfordern ein digitales System zur Bewertung von Abfällen, das auf der selektiven, schnellen und robusten Analytik für die jeweils im Abfall vorkommenden Kunststoffe und Kunststoffkomposite sowie ihrer Verteilung beruht.

Im aktuell laufenden Projekt wird deshalb vom Fraunhofer IMWS und der Lubey AG ein Druck-Schmelz-Analysenverfahren (DSV) und die dazugehörige servicetechnische Peripherie entwickelt. Dazu werden Kunststoffrezyklate zu Folien verpresst und diese mittels IR-Spektroskopie charakterisiert. Mit einer automatisierten Spektrenauswertung kann die Zusammensetzung und Qualität des Kunststoffgemischs ermittelt werden.

Multifunktionales Beschichtungssystem auf der Basis pflanzenölbasierter kationischer Tenside und Lignine

**T. Theumer;
Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS,
Halle (Deutschland)**

Fracture mechanics investigations of unidirectionally fibre-reinforced polymer tapes

R. Lach¹, L. Castro Key², B. Langer^{1,2}, W. Grellmann¹, B. Tillner³, N. Teuscher³, S. Celevics³, I. Jahn³, P. Michel^{2,3};

¹ Polymer Service GmbH Merseburg (Germany), ² Hochschule Merseburg, Fachbereich Ingenieur- und Naturwissenschaften (Germany), ³ Fraunhofer Institut für Mikrostruktur von Werkstoffen und Systemen, Halle (Germany)

Thin polymer tapes reinforced with unidirectionally oriented (UD) continuous fibres form the basis for innovative components used in light weight constructions. Both a UD-tape production facility was developed within the laboratory scale and the UD tapes manufactured with this facility were procedurally as well as material-specifically investigated. Based on the reference adjustment previously obtained by optimization processes of the laboratory-scale impregnating tool the influence of variation of selected processing parameters on the mechanical and fracture mechanics properties of the UD tapes were investigated. Due to the pseudo-ductile stick-slip crack propagation behaviour of the UD tapes cross to the fibre orientation both the essential-work-of-fracture (EWF) approach and the method of Vu-Khanh are comparably useful to quantify the crack initiation toughness. While both methods provide nearly the same material parameters and similar material ranking, the methods cannot be applied in fibre orientation. Irrespective of whether a clear influence of selected processing parameters (all other parameters were the same than a reference tape) on the crack initiation resistance was found. The fibre-matrix adhesion quantifiable by the crack initiation toughness in fibre orientation also affects the crack initiation toughness cross to the fibre orientation (Fig. 1a), because fibre pull-out is the main energy-dissipative failure mechanism inside the UD tapes. The line roughness determined by profilometer-supported 3D surface scans, especially the highly processing-dependent roughness data in fibre orientation, can be used as a fast and effective instrument for quality assessment of the UD tapes. This roughness data correlates with the crack initiation toughness in fibre orientation (Fig. 1b) and can be used to trigger morphology-property relationships. Acknowledgement: Federal Ministry of Education and Research (BMBF), FHprofUnt 2015 (HerDeLaUD, grant: 13FH056PX5); Investitionsbank Sachsen-Anhalt (ModCF, grant: 2004/00059).

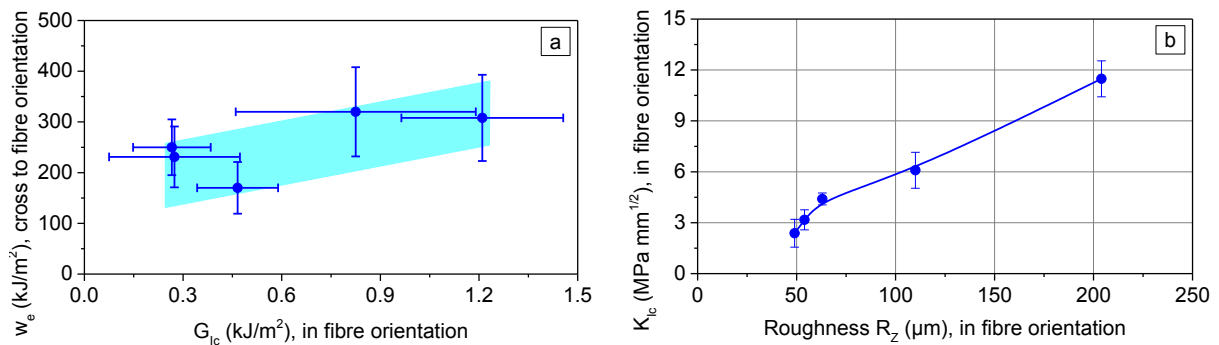


Figure 1: Crack initiation toughness cross (w_e) and in (G_{ic}) fibre orientation (a); crack initiation toughness (K_{ic}) and roughness R_z both in fibre orientation (b).

Vergleichende Lebenszyklusanalyse von zwei Faserverbund-Konstruktionsweisen für ein Light Electric Vehicle

P. Beckert¹, A. Ortwein², S. Wüstenhagen¹;

¹ Fraunhofer-Institut für Mikrostruktur von Werkstoffen und Systemen IMWS, Halle (Deutschland), ² Hochschule Merseburg, Fachbereich Ingenieur- und Naturwissenschaften (Deutschland)

Im Rahmen einer Bachelorarbeit wurde die Lebenszyklusanalyse eines Light Electric Vehicle durchgeführt. Der Schwerpunkt lag auf der Materialauswahl für die Herstellung des Fahrzeugs, wobei verschiedene Bauweisen verglichen wurden. Diese unterscheiden sich vor allem durch ihren Anteil an glasfaserverstärktem Kunststoff, naturfaserverstärktem Kunststoff und Stahl. Insbesondere die Bauweisen mit glasfaserverstärktem Kunststoff wurden detailliert analysiert, was möglich war, da die meisten Daten von den Kooperationspartnern zur Verfügung gestellt wurden und diese Verbundwerkstoffe derzeit auch von ihnen produziert werden.

Als Allokationsmethode wurde die Systemerweiterung und -substitution verwendet, da das Fahrzeug möglichst kreislauffähig ausgelegt werden soll und diese Allokation daher am geeignetsten ist.

Die Bewertung wurde mit dem deutschen Strommix durchgeführt und in einer Sensitivitätsanalyse zusätzlich mit anderen Strommixen verglichen. Es zeigte sich, dass trotz des geringen elektrischen Energieverbrauchs des Fahrzeugs die elektrische Energiebereitstellung über den gesamten Lebenszyklus die größten Auswirkungen aufweist.

Die Substitution von erdölbasierten Kunststoffen oder energieintensiv hergestellten Glasfasern durch nachwachsende Rohstoffe hat einen ökologischen Vorteil. Beim aktuellen Strommix gilt es aber vor allem, den Stromverbrauch zu senken, was primär durch die Reduzierung der Masse erreicht werden kann. Für die ökologische Gesamtbilanz macht es derzeit noch wenig Unterschied, ob nachwachsende Rohstoffe anstatt fossiler Rohstoffe eingesetzt werden. Dies wird sich jedoch mit zunehmendem Anteil der erneuerbaren Energien am Strommix ändern.

Keywords: Naturfaserverstärkte Kunststoffe, NFK, Faserverstärkte Kunststoffe, FVK, Life Cycle Assessment, LCA, Light Electric Vehicle, LEV.

Influence of TDAE oil on silica–rubber interactions in SBR/BR homopolymer and binary blends

S. Sökmen¹, K. Oßwald¹, S. Ilisch², K. Reincke¹;

¹ Polymer Service GmbH Merseburg (Germany), ² Trinseo Deutschland GmbH, Schkopau (Germany)

Material properties are influenced by various material factors. Processing aids such as plasticizers are one of the most known and studied factor which influence mechanical and dynamic mechanical properties of products. However, any detailed study on plasticizer influence on rubber–filler structures and their interactions is not present in the literature. In this study, the influence of treated distillate aromatic extract (TDAE) oil on interactions between silica and rubber chains and material properties were investigated. Rubber–filler and filler–filler interactions of uncured and cured silica-filled styrene–butadiene rubber (SBR) and butadiene rubber (BR) homopolymer mixtures and SBR/BR blends were characterized by using rubber layer L concept and dynamic mechanical analysis. The influence of TDAE oil content on material properties were observed by investigation of tensile properties, Shore A hardness and crack growth behaviour.

Evaluation of the fracture behavior of PMMA materials with respect to environmental stress cracking

J. Posch¹, K. Oßwald¹, B. Langer^{1,2}, K. Reincke¹;

¹ Polymer Service GmbH Merseburg (PSM) (Germany),

² Hochschule Merseburg (University of Applied Sciences) (Germany)

The formation of cracks in thermoplastics under influence of hostile chemical environments and stress, also known as “Environmental Stress Cracking” (ESC), is one major cause for the failure of plastic components (estimated as approximately 15 %) [1]. The most severely affected thermoplastics are amorphous polymers such as poly(methyl methacrylate) (PMMA). In theory, PMMA behaves very brittle below the glass transition temperature (approximately 105 °C, depending on the method of determination [2]) with very high notch sensitivity. In practice, it is modified by means of the addition of plasticizers or blending with other polymers, such that it may even show ductile behavior. In this work, two PMMA materials were investigated which are practically used as surface layer in decor films for window profiles. The overall goal was to find a method (or combination of methods) to characterize the PMMA materials and assess their ESC susceptibility with low requirements on equipment and time. For this purpose, the materials were investigated by different tests and methods, including the tensile test and the Instrumented Charpy Impact Test (ICIT) in dependency of notch radius and thermo-oxidative aging time. Furthermore, the materials were examined using the bent strip method (according to DIN EN ISO 22088-3: Determination of resistance to environmental stress cracking (ESC) — Part 3: Bent strip method) with ethanol of different concentrations and the bending test as indicator test. Lastly, a fracture mechanics approach was applied to generate crack propagation curves from the quasi-static bending test. The investigation has shown that both materials exhibit tough mechanical behavior with distinct yield points and high notch sensitivity, only with a low dependency on the thermo-oxidative aging they were exposed to. Applying the bent strip method resulted in almost immediate material failure at high ethanol concentrations but revealed that some of the specimen which did not fail became tougher. This toughening behavior was also seen by the generated crack propagation curves from the quasi-static bending test.

Literature

- [1] Yan, C., Zhang, J., Han, J., Wang, X., Guan, Z., Zhang, L., Liu, C., Shen, C.: Improvement of environmental stress cracking resistance of polycarbonate by silicone coating. Polym. Test. 60 (2017) 6–11.
- [2] Bandrup, J., Immergut, E. H., Grulke, E. A., Abe, A., Bloch, D. R. (Eds.): Polymer Handbook. 4th Edition, John Wiley & Sons, New York (2003), p. VI/203, 204.

Adjusting PI-PS-based multigraft copolymers properties via blending

R. Schlegel¹, H. Budde², U. Wendler², N. Mahmood³, S. Henning¹, M. Beiner^{1,3};
¹ Fraunhofer Institut für Mikrostruktur von Werkstoffen und Systemen IMWS, Halle (Germany), ² Fraunhofer Pilotanlagenzentrum PAZ, Schkopau (Germany), ³ Martin-Luther-Universität Halle-Wittenberg, Naturwissenschaftliche Fakultät II, Halle (Germany)

The property profile of polyisoprene(PI)–polystyrene(PS) based tetrafunctional multigraft copolymers with about 18–22 wt-% PS synthesized on technical scale (100 g – 1 kg) shows tensile strength values of ca. 16–17 MPa, strain at break values of approximately 2000 % and ultimate toughness values of up to 70 N/mm³. These properties are significantly controlled by the number of PS₂PI repeating units (or number of branch points β) in the macromolecules. The main objective of this study is to optimize the processing of the PI-PS-based multigraft copolymers through blending strategy without compromising the mechanical performance. To facilitate a fine tuning of the materials properties in practice, it was studied to what extend such tendencies could be achieved using multigraft copolymers with high (e.g. $\beta_1 = 6.0$) and low (e.g. $\beta_2 = 2.0$) number of branch points. The blends of the multigraft copolymers were prepared via solution casting. Their mechanical characterization was carried out by tensile tests as well as rheological measurements and structural information was obtained by small angle X-ray scattering (SAXS). In addition, selected blend systems were investigated by imaging methods such as atomic force / transmission electron microscopy (AFM/TEM). Typical feature in mechanical properties is an increase of the tensile strength by increasing the amount of a component with high β -values. The observed trends in mechanical performance show that the strain at break and ultimate toughness pass through a maximum with $\beta = 3.3$ –4.1. The AFM/TEM investigations on blends reveal a common microphase separated morphology. Worm-like PS-domains in a PI-matrix are observed. The average Bragg-spacing are in the range from 40 nm to 25 nm in the investigated multigraft copolymers depending on blend composition. It was found that MGCP-MGCP blends are showing similar characteristics as observed for pure as-synthesized MGCP. If an average $\beta_{\text{blend,avg}}$ value is calculated for MGCP–MGCP-blends (β_1, β_2), quite similar trends to unblended MGCP in structural and mechanical data are observed. The continuous change of the Bragg-spacings with blend composition indicates that the maxima in the mechanical data are of molecular origin and not related to the phase morphology due to microphase separation.

Influence of the antioxidant type on the mechanical and viscoelastic properties of silica-filled NR and SBR vulcanizates after thermo-oxidative aging

**O. Aghazada, K. Oßwald, K. Reincke;
Polymer Service GmbH Merseburg (Germany)**

Elastomers and their components lose their useful properties due to polymer chain degradation under various environmental conditions. In particular, most elastomers can have their strength reduced by oxidation in oxygen-containing atmospheres, and the effect is further enhanced when exposed to high temperatures. A low amount of oxygen (about 1 %) absorbed by a natural or synthetic rubber has a negative effect on its physical properties [1]. Furthermore, in many industrial automotive applications, electrical insulations, etc., the evolution towards higher operating temperatures has allowed the use of more thermally stable elastomers that are more resistant to oxidation. If chain scission dominates during ageing, the elastomer can either become softer and sticky, leading to a decrease in tensile strength and hardness, and an increase or decrease in elongation at break, depending on the extent of degradation. This is often observed in unfilled natural rubber vulcanizates. The vulcanizate hardens and becomes brittle when post-crosslinking dominates during ageing, leading to an increase in stress–strain properties and hardness, but a decrease in elongation at break. This is the case with unfilled styrene–butadiene rubber vulcanizates.

In the project, silica-reinforced compounds based on styrene–butadiene rubber (SBR), natural rubber (NR) were produced. The influence of variation of an antioxidant type on the thermo-oxidative ageing behavior was investigated.

The mixtures were prepared in an internal laboratory mixer. After the compounding process, the crosslinking kinetics was investigated to determine the crosslinking time at a temperature of 160 °C. The thermo-oxidative ageing was carried out in an air-circulation oven at 80 °C. Samples were taken at different aging times and the mechanical, and viscoelastic properties were characterized. For this purpose, the stress–strain properties were analyzed in quasi-static tensile test. Furthermore, the hardness test was conducted and the dynamic-mechanical properties were investigated using the Rubber Process Analyzer (RPA).

Literature

- [1] Mcdonel, E. T., Shelton, J. R.: Effect of curing system on rubber oxidation and physical degradation. J. Chem. Eng. Data 4 (1959) 360–366.

Katalytische Depolymerisation von polyolefinischen Kunststoffabfällen

M. Seitz, S. Schröter;
University of Applied Science Merseburg (Germany)

Polyolefinreiche Kunststoffabfälle können durch katalytische Spaltung wieder in Monomere überführt werden. Durch einfache Aufreinigungsverfahren können Störstoffe abgetrennt werden und die Pyrolyseprodukte direkt in die Produktaufbereitung eines Steamcrackers gelangen.

In einer Kreislaufwirtschaft müssen Verfahren wie Depolymerisation und Pyrolyse zur Erzeugung von Monomeren und anderen Chemikalien aus Kunststoffabfällen (chemisches Recycling) etabliert werden [1, 2]. Insbesondere Polyolefine als Hauptanteil des Kunststoffabfalls können zu Ölen gespalten werden, die als Ausgangsmaterial für Naphtha-Cracker dienen.

Die katalytische Spaltung von Polyolefinen mit sauren Katalysatoren ermöglicht hohe Gasausbeuten mit einem Propen- und Butenengehalt von über 60 %, bei Temperaturen von 350 – 400 °C. Dieses Gas kann durch Absorptionsprozesse von Störkomponenten wie Chlorwasserstoff und Ammoniak, die u.a. aus PVC- und Polyamidanteilen des Kunststoffabfalls stammen, gereinigt werden.

Damit die katalytische Spaltung von Mischkunststoffen zu den Zielprodukten gelingt, wurden deshalb Trägermaterialien so mit sauren Katalysatoren beladen, dass in einem Pyrolysedrehrohr kaum Abrieb stattfindet und der Katalysator nach dem Crackprozess leicht von Fremdstoffen wie Pigmenten und Füllstoffen abgetrennt und regeneriert werden kann. Die Auswahl geeigneter Katalysatoren und die wesentlichen Einflussgrößen auf den Prozess sowie der Einfluss von Störkomponenten wurden in einem Semibatch-Drehrohr unter Inertbedingungen bei Normaldruck untersucht. Als Hauptstörkomponenten im Kunststoffabfall wurden Polyamide, Polyester und PVC identifiziert [3]. Polyamid deaktiviert dabei besonders die äußere Kristallitoberfläche der Katalysatoren. Die Zielproduktausbeute bleibt jedoch im Wesentlichen unbeeinflusst. Das bedeutet, dass durch die Wahl geeigneter Katalysatoren und Betriebsbedingungen trotz problematischer Eingangsstoffe eine stabile Gaszusammensetzung und damit die Erzeugung von Propen und Butenen direkt aus dem Pyrolyseprozess möglich sind.

Literatur

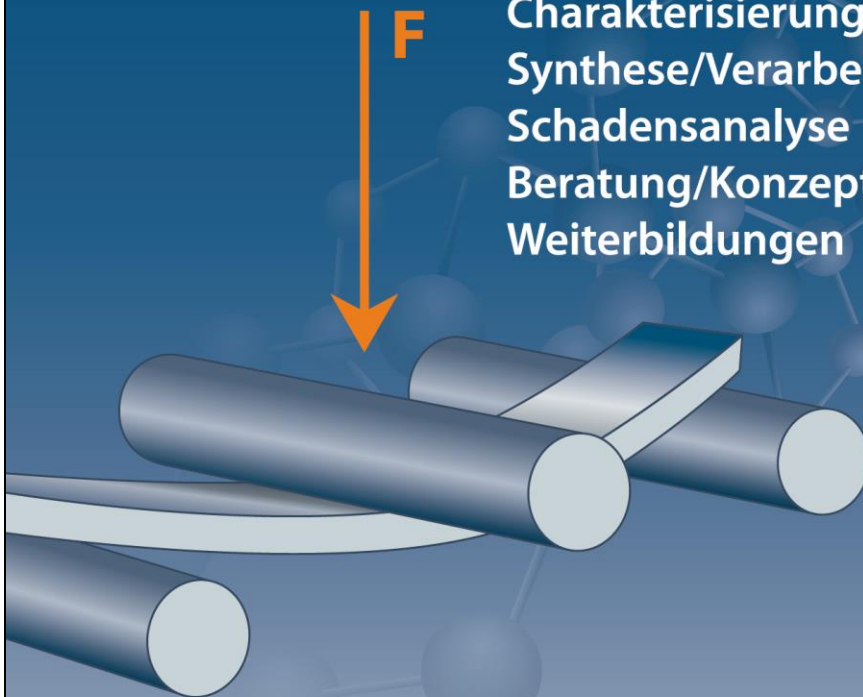
- [1] Joas, F., Witecka, W., Lenck, T., Peter, F., Seiler, F., Samadi, S., Schneider, C., Holtz, G., Kobiela, G., Lechtenböhrer, S., Dinges, K., Steinbacher, K., Schröder, J., Sach, T., Schimmel, M., Kliem, C., Altmann, M., Lehnert, W., Finke, J., Yilmaz, Y.: Klimaneutrale Industrie. Schlüsseltechnologien und Politikoptionen für Stahl, Chemie und Zement. Agora Energiewende & Wuppertal Institut für Klima, Umwelt, Energie, Berlin (2019).
- [2] Geres, R., Kohn, A., Lenz, S., Ausfelder, F., Bazzanella, A. M., Möller, A.: ROADMAP CHEMIE 2050. Auf dem Weg zu einer treibhausgas-neutralen chemischen Industrie in Deutschland. DECHEMA Gesellschaft für Chemische Technik und Biotechnologie e. V. & FutureCamp Climate GmbH, Frankfurt München (2019).
- [3] Seitz, M., Schröter, S., Thamm, D., Engelhardt, A., Klapproth, J., Klätte, M., Cepus, V.: Thermo chemical depolymerisation technologies to recover olefins. In: Reprints of the DGMK-Conference Circular Economy – A Fresh View on Petrochemistry (October 9–11, 2019, Dresden).



Polymer Service GmbH Merseburg

An-Institut an der Hochschule Merseburg

Kunststoffprüfung
Charakterisierung/Analytik
Synthese/Verarbeitung
Schadensanalyse
Beratung/Konzeption
Weiterbildungen



Polymer Service GmbH Merseburg
Eberhard-Leibnitz-Str. 2, Gebäude 131
D-06217 Merseburg

Telefon: +49 (0) 3461 46 2895
Telefax: +49 (0) 3461 46 2592
E-Mail: info@psm-merseburg.de
www.psm-merseburg.de



Förderung der Tagung



ISBN 978-3-00-069372-4