

KGK

KAUTSCHUK GUMMI KUNSTSTOFFE

KREISLAUFWIRTSCHAFT Kautschukrecycling und rCB

RUBBER New ACM Rubbers for Sealing and Fluid Transport

www.kgk-rubberpoint.de

Automation schafft Vorsprung

Zylinderkopfdichtungen
vollautomatisch produzieren, Seite 8



Hüthig

erfolgsmedien für experten

DKT IRC 2021

June 28 – July 1, Nuremberg, Germany

Neuer Termin: 27. – 30. Juni 2022

Internationale
Konferenz

Messe mit Produkt-
präsentationen

Attraktives
Rahmenprogramm

Science Campus

Educational
Symposium

Angebote für
Studierende

TPE Forum



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Bild: gesponsort von LWB Steinl

TITELSTORY
Automation schafft Vorsprung

Moderne Dieselmotoren sind auf ein hohes Niveau bei Effizienz, Leistung und Umweltfreundlichkeit getrimmt. Wichtige Komponenten in diesem High-Tech-Umfeld sind die Zylinderkopfdichtungen, wie sie bei Reinz in Neu-Ulm entwickelt und produziert werden. Es sind Metall/Gummi-Verbundteile, deren Gummikomponente die durch Spritzgießen montierten Dichtungen sind. Die Automatisierungskompetenz von LWB brachte hier den Vorsprung in der Produktion.

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Prof. Andreas Limper ist neuer Geschäftsführer der DKG

DEUTSCHE KAUSCHUK-GESELLSCHAFT

Prof. Andreas Limper hat zum 1. Januar 2021 die Geschäftsführung der Deutschen Kautschuk-Gesellschaft (DKG) übernommen. Er folgt in dieser Position auf Boris Engelhardt, der sich zukünftig auf seine Aufgaben als Hauptgeschäftsführer des Wirtschaftsverbandes der deutschen Kautschukindustrie (wdk) konzentriert. Der neue DKG-Geschäfts-



Bild: DKG

Prof. Andreas Limper ist neuer Geschäftsführer der DKG.

führer Professor Limper war bis 2019 CEO der HF Mixing Group und hat einen Lehrauftrag an der RWTH Aachen. Er ist in der Branche als Wissenschaftler und Unternehmer international anerkannt. Der Deutschen Kautschuk-Gesellschaft gehört Limper seit 1990 als Mitglied an, seit 2018 ist er auch Mitglied des DKG-Vorstandes. ■

www.dkg-rubber.de

DKT/IRC 2021 auf 2022 verschoben – Fachkonferenz online geplant

DEUTSCHE KAUSCHUK TAGUNG 2021

Die Deutsche Kautschuk Tagung 2021 wird auf das nächste Jahr verschoben. Die andauernden Einschränkungen durch die Covid 19-Pandemie haben den Veranstalter, die Deutsche Kautschuk-Gesellschaft veranlasst, die führende Messe für die kautschukverarbeitende Industrie in diesem Jahr abzusagen. Die Fachkonferenz soll wie geplant vom 28. Juni bis 1. Juli 2021 als Online-Event stattfinden. Alle drei Jahre veranstaltet die Deutsche Kautschuk-Gesellschaft e. V. (DKG) eine internationale Tagung in Nürnberg. Die nächste Deutsche Kautschuk Tagung DKT, gleichzeitig International Rubber Conference IRC, sollte vom 28. Juni bis 1. Juli 2021 wieder zum Treffpunkt der globalen Kautschuk- und Elastomerbranche mit mehr als 3.000 internationalen Gästen werden. Der sichere und er-



folgreiche Veranstaltungsbesuch aller Teilnehmenden, Vortragenden und Ausstellenden hat für den DKG-Vorstand hohe Priorität. Vor diesem Hintergrund und angesichts der Langfristigkeit der Auswirkungen der Pandemie wird die DKT IRC darum um ein Jahr, auf den 27. bis 30. Juni 2022, verschoben. Veranstaltungsort bleibt Nürnberg. Doch auch in diesem Jahr gibt es die Gelegenheit, sich in einer internationalen Konferenz über aktuelle Entwicklungen und neueste Ergebnisse der Elastomereforschung zu informieren. Ausgewählte Vorträge, wissenschaftliche Poster und Gelegenheiten zum Dialog mit anderen Teilnehmenden erwarten die Gäste des DKG Elastomer Symposiums, das vom 28. Juni bis 1. Juli 2021 als Online-Veranstaltung stattfindet. ■

www.dkt2021.de

KGK-Rubberpoint Contentletter geht an den Start

KGK-RUBBERPOINT Der KGK-Rubberpoint Contentletter liefert ab Februar einmal monatlich – Start ist am 23.02.2021 – aktuelle Infos aus der Kautschukbranche. Das Fachportal der Kautschukindustrie bietet nun einen E-Mail-Service an, der die Neuigkeiten und Veröffentlichungen direkt in Ihr Postfach liefert. Im digitalen Contentletter lesen Sie die Zusammenfassung aktueller Fachaufsätze aus Wissenschaft und Praxis, innovative Produkte und News der Kautschuk- und Kunststoffbranche. Keinen Tipp für die Praxis und kein brandneues Ergebnis der Kunststoff- und Kautschukforschung mehr verpassen und jetzt zum Content-



Jetzt Anmelden zum KGK-Rubberpoint Contentletter und keine Meldung mehr verpassen.

letter anmelden. Das Fachportal und die Fachzeitschrift bieten Informationen zu Kautschuk- Roh- und Zusatzstoffen, zu Silikonen und TPE. In Anwenderberichten werden aktuelle Verfahren und Anlagen zur Elastomerverarbeitung vorgestellt. Dazu finden User auf dem Portal neue Produktentwicklungen aus den Bereichen Rohstoffe, Maschinen und Anlagen, Software sowie Messtechnik und Qualitätssicherung. Für Abonnenten ist außerdem ein Zugriff zu exklusiven, wissenschaftlichen Ergebnissen aus der Kunststoff- und Kautschukforschung und eine Recherche im Archiv möglich. ■

www.kgk-rubberpoint.de/contentletter

Rehau-Firmengründer Helmut Wagner ist verstorben

REHAU Das Unternehmen Rehau trauert um seinen Firmengründer und Ehrenpräsidenten Helmut Wagner. Er starb am 24. Januar im Alter von 95 Jahren in der Schweiz im Kreis seiner Familie. Die Nachricht vom Tod des beliebten und bis zuletzt hoch engagierten Gründervaters bewegt Rehau-Mitarbeitende rund um den Globus. Seit 1948 hat Helmut Wagner die Welt der Kunststoffe geprägt und damit einer ganzen Branche wichtige Impulse gegeben. Mit der Firmengründung im oberfränkischen Rehau beginnt die Herstellung von Produkten aus polymerem Material und damit die bis heute anhaltende Erfolgsgeschichte eines Familienunternehmens, das nach wie vor stark geprägt ist von dem Pioniergeist des Firmengründers. Nach einer erfolgreichen Entwicklung des



Bild: Rehau

Helmut Wagner ist im Alter von 95 Jahren verstorben.

Unternehmens, über fünf Jahrzehnte, übergab Helmut Wagner im Jahr 2000 die Leitung der Firmengruppe an seine Söhne: Jobst Wagner wurde Präsident des Supervisory Board, Dr. Veit Wagner Vizepräsident. Inzwischen ist die dritte Generation an Board. 2015 ernannte der Rehauer Stadtrat Helmut Wagner zum Ehrenbürger. ■

www.rehau.com

Führungswechsel bei Barnes Molding Solutions

BARNES GROUP Zum 1. Januar 2021 hat Jessica Poliner bei der Barnes Group, Bristol, die Position als President Molding Solutions von Norbert Scheid übernommen. Scheid zog sich im Dezember 2020 aus dem operativen Geschäft zurück. Molding Solutions ist eine Geschäftseinheit innerhalb der Barnes Group, zu der auch Synventive, Thermoplay, Männer, Foboha, Priamus und Gammflux gehören. Mit über fünfzehn Jahren Führungserfahrung in global diversifizierten Industrieunternehmen übernimmt Jessica Poliner die Position als President Molding Solutions. Die studierte Juristin begann ihre Karriere als Anwältin. Zuletzt war Jessica Poliner General Manager & Vice President für die Geschäftsbereiche Marine, Rail und Air von Thermo King (Ingersoll Rand) und in diversen Füh-



Bild: Barnes

Jessica Poliner ist neuer Präsident Molding Solutions bei der Barnes Group.

rungspositionen bei Caterpillar tätig. Mit der Übernahme von Synventive kam Herr Scheid im August 2012 als Vice President and General Manager, European Operations bei Synventive zur Barnes Group. Zuletzt war er als President Molding Solutions tätig. Die Barnes Group bedankt sich für seine Führung und Unterstützung von Molding Solutions. ■

www.barnesgroupinc.de

Technologietage 2021 – Sechs Tage Technik zum Anfassen

ARBURG Um die Technologietage 2021 in Loßburg für geladene Gäste Coronaadäquat als physische Veranstaltung umzusetzen, hat sich Arburg dazu entschlossen, zusätzlich zur Verschiebung in den Juni das Event auf sechs Tage zu verlängern. Die Technologietage starten nun bereits am 7. Juni und dauern bis zum 12. Juni. „Nach der langen Zeit ohne Präsenzveranstaltungen wollen wir unseren Kunden im Juni 2021 wieder Innovationen und anspruchsvolle Anwendungen für die Kunststoffverarbeitung ‚live und in Farbe‘ bieten“, betont Juliane Hehl, die als geschäftsführende Gesellschafterin den Marketing-Bereich verantwortet. „Neben unseren hybriden und digitalen Events, die wir in den vergangenen Monaten erfolgreich vorangetrieben haben, ist und bleibt der persönliche Kontakt mit unseren Kunden äußerst wichtig. Deshalb tun wir alles dafür, dass unsere weltweit bekannten und geschätzten Technologietage in diesem Jahr ein erfolgreiches und spannendes physisches Event werden.“ Wegen der Corona-Situation wurde bereits im September 2020 beschlossen, das dies-



Bild: Arburg

jähriges Event vom traditionellen März-Termin auf den Frühsommer zu verschieben. Am bewährten Konzept „Technik zum Anfassen kombiniert mit persönlicher Betreuung“ ändert sich nichts. Die geladenen Gäste werden in diesem Jahr jedoch nach verbindlicher Voranmeldung in kleinen Gruppen persönlich betreut und zu definierten Timeslots durch das Unternehmen geführt. Dadurch ist sichergestellt, dass nicht zu viele Besucher gleichzeitig vor Ort sind. Das Unternehmen setzt bei den Planungen weiterhin darauf, dass sich die epi-

Auf den Technologie-Tagen 2019 war Corona noch kein Thema (Bild). In diesem Jahr werden die geladenen Gäste mittels eines neuen Besuchsmanagements durch das Unternehmen geführt.

demologische Lage, unterstützt durch die bereits begonnenen Impfungen, bis Juni 2021 zum Positiven ändert. Der hohen Verantwortung gegenüber Besuchern, Partnern und Mitarbeitern entsprechend, wird das bewährte Sicherheits- und Hygienekonzept konsequent beibehalten. Die weitläufigen Flächen des Stammhauses bieten ausreichend Platz, eine große Anzahl interessanter Exponate mit dem gebotenen Abstand zu präsentieren. Die Maschinen, Turnkey-Anlagen sowie digitalen Produkte und Services werden über das Kundencenter, das Prototyping Center, die Effizienz-Arena, das Schulungcenter und die Montagehallen verteilt. ■

www.arburg.com

Erholung der deutschen Wirtschaft vorerst gestoppt

IFO Der Ifo Geschäftsklima-Index ist im Januar auf 90,1 Punkte gefallen, nach 92,2 Punkten im Dezember. Die Unternehmer beurteilten ihre aktuelle Lage schlechter als im Vormonat, wobei der Industriesektor hier noch eine Ausnahme macht. Die Erwartungen für die nächsten Monate haben sich aber über alle Sektoren hinweg verdüstert. Im verarbeitenden Gewerbe ist der Geschäftsklima-Index nach zuletzt acht Anstiegen in Folge wieder gesunken. Dies war auf deutlich weniger optimistische Erwartungen der Industriefirmen zurückzuführen. Die aktuelle Lage bewerteten sie hingegen merklich besser. Die Kapazitätsauslastung stieg um 1,6 Prozentpunkte auf 81,8 Prozent. Sie liegt damit aber immer noch unter ihrem langfristigen Durchschnitt von 83,5 Prozent. Im Dienstleistungssektor ist der Geschäftsklima-Indikator

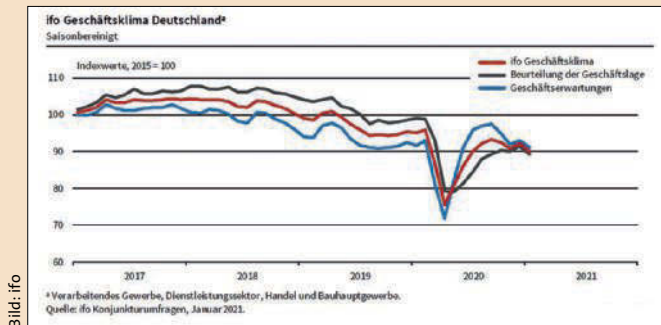


Bild: ifo

merklich gesunken. Die Unternehmen korrigierten ihre Einschätzungen zur aktuellen Lage nach unten. Zudem nahm der Pessimismus mit Blick auf die kommenden Monate deutlich zu. Der Auftragsbestand, insbesondere im Bereich Transport und Logistik, entwickelte sich schwächer. Im Handel ist der Geschäftsklimaindex eingebrochen. Der Indikator zur aktuellen Lage stürzte in

den negativen Bereich. Dies war der stärkste Rückgang seit April 2020. Auch die Erwartungen fielen deutlich pessimistischer aus. Die Stimmung verschlechterte sich, teilweise massiv, in nahezu allen Einzelhandelssparten.

Anders als im Dezember zeigten diesmal auch im Großhandel viele Indikatoren nach unten. Auch im Bauhauptgewerbe verschlechterte sich das Geschäftsklima. Die Einschätzungen zur aktuellen Lage fielen etwas schlechter aus. Zudem blickten mehr Unternehmer pessimistisch auf die kommenden Monate.

www.ifo.de

Prof. Hans-Josef Endres als Berater für SPE-Nachhaltigkeitspreis



Bild: Christian Wyrwa

Professor Hans-Josef Endres wird als Berater für die Bewertungskriterien und als Juror den Nachhaltigkeitspreis unterstützen.

SPE AUTOMOTIVE AWARD Im Sommer 2021 wird beim 20. SPE Automotive Award erstmalig ein neuer Preis verliehen: Der Sustainability-Award. Mit Professor

Hans-Josef Endres hat SPE Central Europe, Wolfsburg, einen Experten für Kreislaufwirtschaft und Nachhaltigkeitsbewertung in der Kunststoffbranche als Berater gewinnen können. Er wird gemeinsam mit den Verantwortlichen von SPE Central Europe die Bewertungskriterien ausarbeiten. Im Januar 2020 beim Jurorenmeeting vorgeschlagen, wird er 2021 beim SPE Automotive Award erstmals verliehen: der Sustainability-Award. Verliehen wird er an das Bauteil unter allen Einreichungen, das die beste ökologische Gesamtleistung aufweist. Damit will SPE Central Europe den Nachhaltigkeitsgedanken für alle Einreicher in den Fokus rücken und so umwelt- und ressourcenschonende Entwicklungen weiter fördern. Wichtige Kriterien für den Sustainability-Award werden sein, wie sich Bauteile, Technologien und Prozesse zur Bauteilherstellung durch eine verbesserte ökologische Gesamtbilanz darstellen. Dies beinhaltet sowohl nachhaltige Prozesslösungen als auch Kreislaufkonzepte und den Einsatz von umweltfreundlichen Werkstoffen, wie biobasierte Materialien, Recyclate oder nachhaltigere Leichtbaulösungen einschließlich eines verbesserten CO₂-Fußabdrucks. Selbstverständlich werden auch Design-for-Recycling-Ansätze und die Verwertungsstrategie am Ende des Produktlebens betrachtet. ■

www.se-k.de/spe-automotive-award

Stuttgarter Kunststoffkolloquium in digitaler Form



Bild: IKT

Die Teilnehmer des 27. Stuttgarter Kunststoffkolloquiums treffen sich Anfang März erstmals in digitaler Form.

IKT Das 27. Stuttgarter Kunststoffkolloquium findet erstmals vom 1. bis 4. März 2021, jeweils

nachmittags, in digitaler Form statt. Die Teilnehmer erwarten 44 Vorträge zu den neuesten Forschungsergebnissen der Universität Stuttgart in zwei parallelen Veranstaltungen. Besondere Highlights des 27. Stuttgarter Kunststoffkolloquiums sind die hochkarätigen Plenarvorträge „Additive Manufacturing in der Automobilindustrie“ Referent Martin Friedrich, Additive Manufacturing, Project Lead Research & Development Non Metal, BMW Group, sowie „Die Resilienz-Lücke: Lehren aus der Covid-19 Pandemie“ referiert von Prof. Dr. Dr. Ortwin Renn, wissenschaftlicher Direktor am Institut für Transformative Nachhaltigkeitsforschung (IASS) in Potsdam, Berater der Bundesregierung. Der dritte Tag wird von einer Podiumsdiskussion zum Thema „Chancen der Krise nutzen“ eingeleitet. Diskutieren werden Prof. Dr. Hanns-Peter Knaebel, Vorstandsvorsitzender der Röchling-Gruppe, Dr. Marco Wacker, Leiter des Geschäftsbereiches Augenschutz bei Uvex, Prof. Dr. Ernst Schmachtenberg, ehemaliger Präsident der RWTH Aachen, und Michael Weigelt, Geschäftsführer des GKV/TecPart. Bereits zum zweiten Mal werden im Rahmen des Stuttgarter Kunststoffkolloquiums die Wilfried-Enginger-Preise verliehen. ■

www.ikt.uni-stuttgart.de

60 Jahre Kunststoff-Zentrum SKZ

SKZ Das 1961 in Würzburg gegründete Kunststoff-Zentrum SKZ hat sich inzwischen zum hierzulande größten Kunststoff-Institut entwickelt. Zum 60. Geburtstag denkt das SKZ noch lange nicht daran, „in Rente“ zu gehen, sondern konzentriert sich weiterhin auf die innovativsten und zukunftsreichsten Entwicklungen rund um Kunststoffe. Heute zählt das SKZ-Netzwerk mehr als 400 Mitgliedsunternehmen mit all ihren Mitarbeitern – rund doppelt so viele wie noch vor zehn Jahren –, die sich schnell und unkompliziert über Online-Sessions sowie digitalen Foren und Plattformen unterhalten und so die wichtigsten Neuerungen austauschen können. Nicht geändert hat sich hingegen, dass immer noch der persönliche Austausch die zufriedenstellendste und präferierte Form ist. Sobald die Corona-Pandemie überwunden ist, wird das SKZ auch wieder intensiv auf den persönlichen Austausch auf Tagungen und in Kursen setzen. Institutsdirektor Prof.



Bild: SKZ

Dr. Martin Bastian ist dankbar für die erfolgreiche Geschichte des SKZ: „Mit Stolz und großer Dankbarkeit blicken wir auf die vergangenen Jahrzehnte zurück. Die stetige Unterstützung der Kunststoffindustrie und das außerordentliche Engagement unserer Mitarbeiter sind das Rezept für unseren Erfolg. Auch zukünftig wird das SKZ nicht davor zurückschrecken, neue Wege zu gehen und innovative Technologien einzusetzen.“ So stehen die im Bau befindlichen Erweiterungen in Würzburg, die Modellfabrik 2020 und das Trainingszentrum Qualitätswesen sowie der in diesem Jahr geplante Start der

Erweiterung des Standorts Selb mit dem Europäischen Zentrum für Dispersionstechnologien, stellvertretend für die Innovationskraft und für den Weitblick des Kunststoffzentrums in allen Bereichen der Kunststofftechnik und auch darüber hinaus. Das SKZ ist Mitglied der Zuse-Gemeinschaft. Diese ist ein Verbund unabhängiger, industrienahe Forschungseinrichtungen, die das Ziel verfolgen, die Leistungs- und Wettbewerbsfähigkeit der Industrie, insbesondere des Mittelstandes, durch Innovation und Vernetzung zu verbessern. ■

Vor 60 Jahren in Würzburg gegründet – das Kunststoffzentrum SKZ.

www.skz.de

Universal Robots mit weiteren deutschen Vertriebspartnern

UNIVERSAL ROBOTS (UR) Der Cobot-Hersteller Universal Robots (UR) gibt zwei neue Vertriebspartner für Deutschland bekannt: Hesse Vorrichtungen und Fertigungstechnik mit Sitz in Schloß Holte-Stukenbrock sowie A² Anlagenoptimierung & Anwendungstechnik, Stuttgart. Die beiden Unternehmen verstärken das Partnernetzwerk des dänischen Herstellers für kollaborative Robotik. Über 700 Partner zählt das globale UR-Netzwerk bereits. Ziel des Unternehmens ist, die Vorteile kollaborativer Robotik auch in kleine und mittlere Unternehmen zu tragen. Diesbezüglich sieht Andrea Albani, Regional Sales Director Westeuropa, das

Partnernetzwerk als wesentlichen Funktionsträger. Seit November 2020 ist Hesse Vorrichtungen und Fertigungstechnik offizieller Certified System Integrator von UR. Das Unternehmen mit Sitz im nordrhein-westfälischen Schloß Holte-Stukenbrock verfügt über 20 Jahre Erfahrung in den Bereichen Mechanik und Elektronikproduktion. Mit der Partnerschaft erweitert es sein Dienstleistungsangebot für Automatisierungs- und Fertigungstechnik. Der Stuttgarter Anbieter für Robotik- und CNC-Lösungen optimiert die Prozesse der Anwender nach dem Lean-Prinzip. Die Firma hat sich auf Robotikkonzepte spezialisiert. ■



Bild: Hesse

Universal Robots hat zwei neue Vertriebspartner in Deutschland.

www.universal-robots.com

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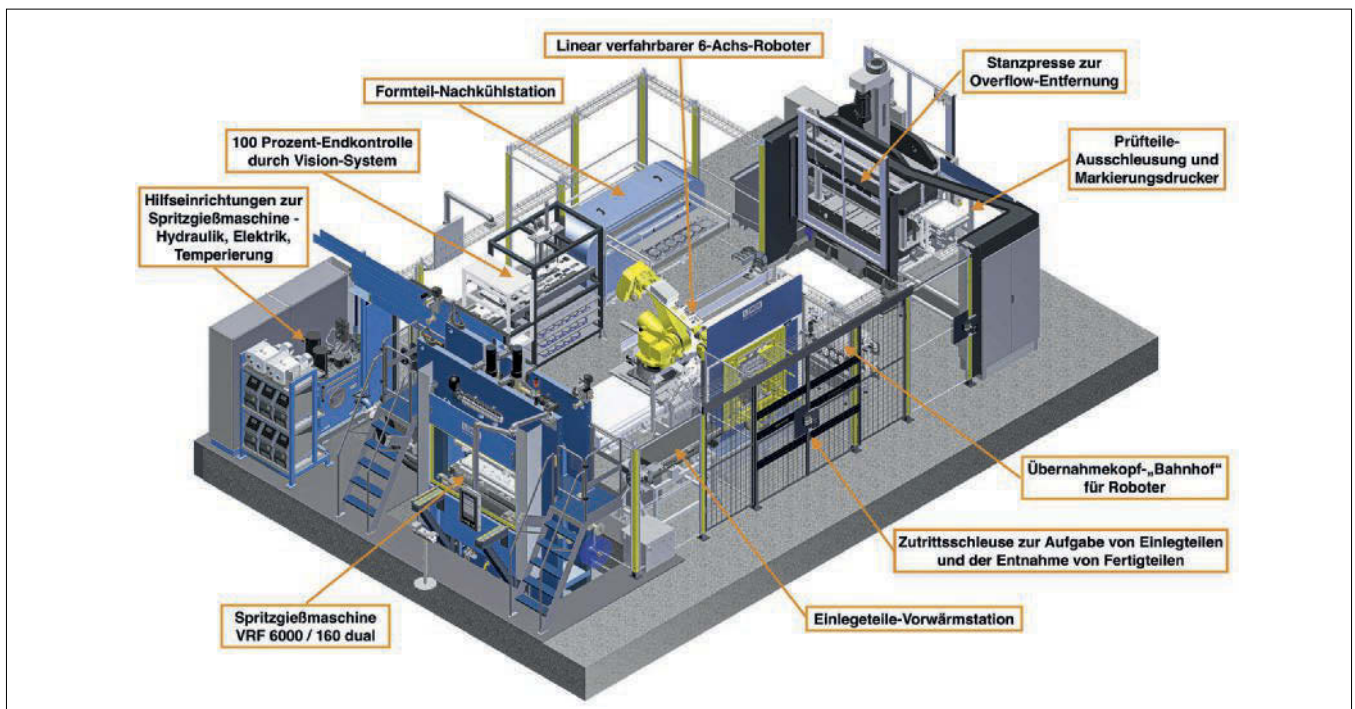


Bild: LWB Steinl

Die von Reinz und LWB-Steinl gemeinsam entwickelte Produktionszelle zur flexiblen Herstellung von Zylinderkopfdichtungen unterschiedlicher Größen automatisiert alle Arbeitsschritte der Metallteil-Manipulation vor und nach der Spritzgießmaschine inklusive der Nachkühlung, Entgratung und Qualitätskontrolle und -dokumentation.

Automation schafft Vorsprung

Zylinderkopfdichtungen vollautomatisch produzieren Moderne Dieselmotoren sind auf ein hohes Niveau bei Effizienz, Leistung und Umweltfreundlichkeit getrimmt. Wichtige Komponenten in diesem High-Tech-Umfeld sind die Zylinderkopfdichtungen, wie sie bei Reinz in Neu-Ulm entwickelt und produziert werden. Es sind Metall/Gummi-Verbundteile, deren Gummikomponente die durch Spritzgießen montierten Dichtungen sind. Die Automatisierungskompetenz von LWB brachte hier den Vorsprung in der Produktion.

Niedrige Treibstoffpreise, geringe Straßennutzungskosten und Fahrerlöhne und fallende Grenzen haben ab den 1990er-Jahren lange Lieferketten zwischen Hoch- und Niedriglohnländern begünstigt. Insbesondere für Produkte mit einem hohen Montageanteil. Niedrig entlohnte Handarbeit steht seitdem im Wettbewerb zu kostenintensiven Investitionen in Automatisierungen. Dass Lieferketten nicht nur kurzfristig, sondern auch über einen längeren Zeitraum reißen können, hat die Corona-Krise gezeigt.

Autor

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freier Fachjournalist,
Technokomm, Gmünd, Österreich
office@technokomm.at

Dies war aber nur der dramatische Höhepunkt in einem schon länger laufenden Umdenkprozess. Denn schärfere Transportgesetze und weitgehend flächendeckende Straßenmauten verteuerten bereits vor „Corona“ den Überlandtransport. Was die Kostenvorteile von manueller Niedriglohnarbeit schmelzen ließ und die Rückverlagerung samt Automatisierungsinvestitionen wieder attraktiv machte. Nicht zuletzt auch in der Gummiteile-Produktion, die aber wegen der Elastizität der Bauteile deutlich mehr Handhabungs-Know-how erfordert, als bei den deutlich formstabileren Kunststoffteilen.

Automatisierungskompetenz

Als die Reinz-Dichtungs-GmbH in Neu-Ulm, eine Tochterfirma der US-amerikanischen Dana-Gruppe, im Jahr 2018

nach einem Partner zur Konzeption einer hoch- bis vollautomatisierten Produktionszelle zur Spritzgießmontage von Gummidichtungen auf Zylinderkopfpfatten suchte, kam Gummi-Spritzgießmaschinenbauer LWB-Steinl aus Altdorf bei Landshut auf die Shortlist. Dazu Reinz-Projektleiter Mario Bauer: „Bei der Partnersuche wurde uns schnell klar, dass die Liste mit Firmen, die auf eine Expertise beim Handling gummielastischer Formteile in der angepeilten Komplexität verweisen konnten, tatsächlich „short“ sein würde. Letztendlich blieb nur LWB-Steinl übrig. Den Ausschlag dafür gab deren 2017 gegründetes Tochterunternehmen „LWB-Automation“ im baden-württembergischen Weinheim, deren Referenzen uns ins Gespräch kommen ließ. Und zu besprechen gab es genug. Denn



Bild: LWB Steinl

Eine der vier unterschiedlichen Zylinderkopfdichtungen für 5- und 6-Zylinder-LKW-Motoren, mit Abmessungen bis zu 1200 x 250 mm, die auf der Anlage produziert werden.



Bild: LWB Steinl

Hinter der Zutrittsschleuse befinden sich die Lagerrecks für Fertigteile (obere zwei Positionen) und die Übernahmeposition für die Einlege-
teile (darunter).

unser Plan war, bis zu 29 Dichtungen aus Fluor-Kautschuk an den Durchgangsöffnungen für Öl- und Kühlwasser von Zylinderkopf- Blechplatinen durch Spritzgießen zu montieren, und zwar alle auf einmal in einem Spritzgießzyklus. Doch bis es so weit war, gab es einiges zu entwickeln. Es wäre zu lange, die gesamte Evolutionsgeschichte des Projekts bis zur Produktionsreife nachzuerzählen. Nur eines: Es brauchte rund ein Jahr und mehrere Gesprächsrunden mit den Spezialisten des LWB-Maschinenbaus und der LWB-Automation, in denen wir uns schrittweise dem Vollautomatik-Konzept der Produktionszelle annäherten. Im Rückblick gesehen war es beeindruckend, wie mit jeder Gesprächsrunde die Produktionszelle vereinfacht und die Zykluszeit reduziert werden konnten. Letztendlich entschieden wir uns für einen 6-Achs-Roboter mit zusätzlicher Längsverfahrachse als zentralem Akteur. Durch ihn konnten alle Fertigungsoperationen von der Blechteil-Übernahme, dem

Einlegen in und das Entnehmen aus der Spritzgießmaschine, der nachfolgenden Entgratung durch Stanzen und die optische Endkontrolle zu einem Gesamtprozess integriert werden.“

Rahmengestell-Spritzgießmaschine für höchste Formstabilität

Der Entschluss des Projektteams, die großflächigen Zylinderkopfdichtungen in einem Spritzgießwerkzeug mit Multidüsen-Kaltkanal in einem Schuss mit allen Dichtungen zu versehen, hatte zwei wesentliche Auswirkungen auf die Maschinenauswahl. Einerseits war es die Forderung nach der größtmöglichen Gleichförmigkeit der Schließkräfteinleitung in die ausgeprägt rechteckigen Spritzgießwerkzeuge, andererseits die Notwendigkeit dem ausladenden Doppelgreifer des Roboters den nötigen Freiraum anbieten zu können. Ausladend deswegen, weil damit sowohl das Fertigteil entnommen und nur um einen kurzen Hubversatz weiter die Stahlplatine eingelegt wird.

Daraus abgeleitet fiel die Wahl auf eine Vertikal-Spritzgießmaschine mit Rahmen-Schließereinheit. Die Gründe dafür erläutert der technische Leiter von LWB-Steinl, Armin Stütz: „Durch die Rahmenschließereinheit, die wir als einziges Branchenunternehmen im Programm haben, konnten wir die Aufspannfläche bzw. die Heizplattengröße genau auf die Spritzgießwerkzeuge abstimmen. Mit einer Holm-Schließereinheit wären breitere Aufspannplatten zu akzeptieren gewesen und damit vergleichsweise höhere Durchbiegewerte, was angesichts der über die gesamte Breite der Stahlplatine verteilten Dichtungen inakzeptabel hohe Toleranzen bei der Dichtprofildicke und den Austriebsmengen zur Folge gehabt hätte. Nicht so bei der Rahmen-Schließereinheit. Deren Schweißkonstruktion in Kombination mit zwei Druckkissen-Schließmodulen ist insgesamt steifer und formstabiler, was eine höhere Planparallelität unter allen Lastzuständen gewährleistet.“



Der Stanzteil der Zylinderkopfdichtung wird mittels Roboter in die Schließereinheit eingelegt, mit Gummi umspritzt und anschließend entnommen. Der Roboter besitzt dafür einen Doppelgreifer.

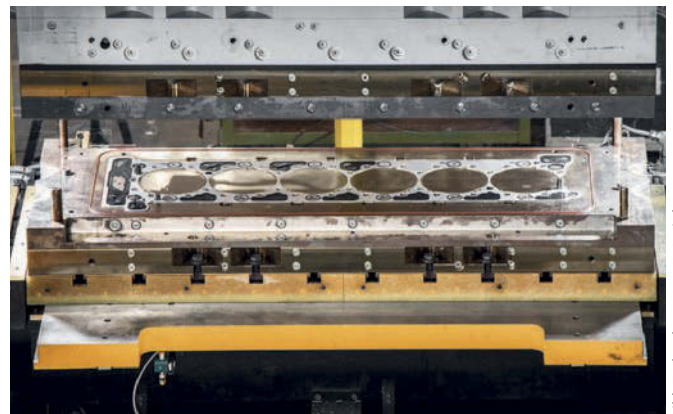


Bild: Technokomm, LWB Steinl



Bild: Technokomm

Nach der Entnahme aus der Spritzgießmaschine transferiert der Roboter den Verbundteil in eine Nachkühlstation und danach in die Overflow-Stanze (rechts der Mitte im Hintergrund).



Bild: Technokomm

In der Stanzstation werden die Overflow-Häute, wo vorhanden, gestanzt und nach unten abgeführt.

Am Ende des Definitionsprozesses stand eine Maschine mit der Typenbezeichnung LWB VRF 6000/160 dual mit 6000 kN Schließkraft. „Dual steht dabei für zwei von oben einspritzende 160 cm³-Klein-Spritzaggregate, die über zwei 8-fach Kaltkanal-Systeme eine Fluorkautschuk-Mischung in das Spritzgießwerkzeug einspritzen.“ Um rasch die Werkzeuge für die im Wechsel produzierten vier Größenvarianten der Zylinderkopfdichtungen umrüsten zu können, wurden sowohl die Spritzgießmaschine als auch die Stanzmaschine mit Werkzeugwechsellvorrichtungen ausgerüstet.

Vollautomatisierter Arbeitsablauf sichert hohe Produktqualität

Die gemeinsam konzipierte Produktionszelle fasst alle Arbeitsstationen in einer Rechteck-Konfiguration zusammen. Die Rechteck-Konfiguration ergab sich aus der Anzahl der Arbeitsstation, die größtenbedingt nicht im kreis- bzw. kugelförmigen Arbeitsbereich des zentralen 6-Achs-Industrieroboters Platz hatten. Die Lösung dafür war, den Ro-

boter auf eine Schienenbahn (7-te Achse) zu stellen und die einzelnen im Karree positionierten Stationen zykluszeitversetzt sequenziell anzufahren. Der Anfang und auch das Ende der Arbeitsfolge befinden sich in der Mitte einer Längsseite. Es sind die Übernahme- bzw. die Übergabepositionen für die Einlegeteile (Stanzteile) und die Fertigteile, die sich hinter einer Zugangschleuse für das Bedienpersonal befinden. Von dort übernimmt der Roboter die Einlegeteile, passiert dabei eine Dicken-Messstation, mit der ausgeschlossen wird, dass sich zwei durch Adhäsion zusammenklebende Teile auf dem Roboter greifer befinden. Der nächste Arbeitsschritt ist das Einlegen der Stahlplatte in eine Vorwärmstation und der anschließende Transfer in die Spritzgießmaschine.

Nach der Entnahme des Metall/Gummi Verbundteils aus der Spritzgießmaschine übergibt ihn der Roboter in eine Kühlstation, die sich diagonal gegenüber der Spritzgießmaschine befindet. Der nächste Arbeitsschritt findet in der daneben positionierten Stanzm-

schine statt, wo die Overflows im Zentralbereich der Dichtungen ausgestanzt und nach unten hin aus der Maschine abgeführt werden. Nach dem Stanzen übergibt der Roboter das Verbundteil an eine Kamera-Messstation, in der alle Dichtungsbereiche maßlich und auf Vollständigkeit geprüft werden. Auf den Gutbefund folgt als letzter Arbeitsschritt die Übergabe an das Fertigteil-Rack, womit sich der Kreis schließt. Alle Arbeitsschritte werden über eine zentrale Prozesssteuerung koordiniert und überwacht, alle Messergebnisse dokumentiert.

Zusammenarbeit führt zum Ziel

Nach der Projektierungsphase konnte im Dezember 2019 mit dem Bau begonnen werden. Im September 2020 war es dann schließlich soweit und die Anlage hat alle Abnahmetests erfolgreich absolviert und konnte ausgeliefert werden. Dazu noch Reinz-Projektleiter Mario Bauer: „Es war unser erstes Anlagenprojekt mit LWB-Steinl und daher ein Wagnis mit ungewissem Ausgang. Doch das Wagnis hat sich gelohnt, wie das Endergebnis eindrucksvoll belegt. Erfolgsentscheidend war insbesondere die während der gesamten Projektlaufzeit intensiv gelebte und über die Buchstaben des Auftrages hinaus gehende Zusammenarbeit. Ohne sie wäre das Automatisierungsprojekt das anfänglich befürchtete Wagnis mit ungewissem Ausgang gewesen.“

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Bild: Technokomm

Armin Stütz, der technische Leiter von LWB (links) und Mario Bauer, Projektleiter bei Reinz können mit berechtigter Genugtuung auf den Abschluss der zweijährigen Projektphase zurückblicken.

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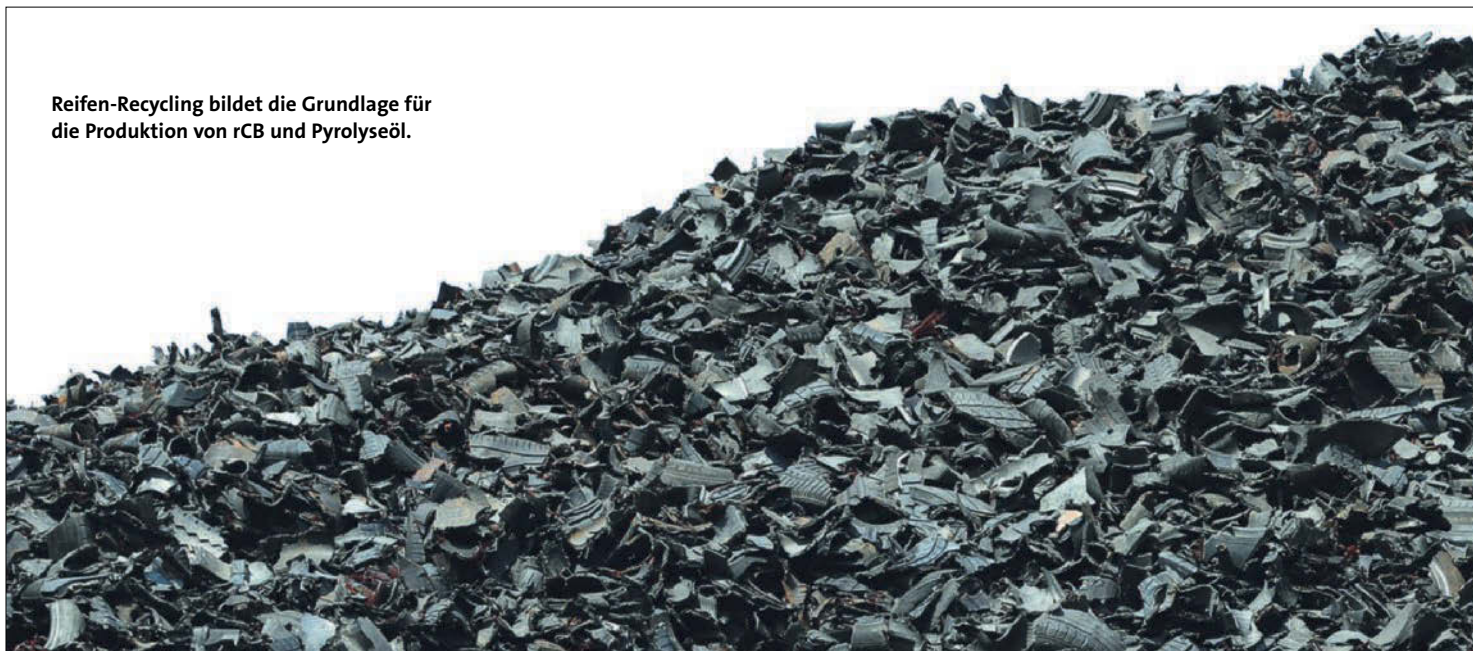


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Reifen-Recycling bildet die Grundlage für die Produktion von rCB und Pyrolyseöl.



Recovered Carbon Black - auf dem Weg zur Industrialisierung

Recycling von Reifen zu rCB und Pyrolyseöl Recovered Carbon Black ist ein recycelter Rohstoff, der im Jahr 2020 viel Presse bekam, nicht zuletzt Dank des großen Interesses der Reifenhersteller Michelin, Continental und Bridgestone, sowie des Chemiekonzerns BASF. Aber was ist recovered Carbon Black? Welches sind die Unterschiede zu Industrieruß? Und wie sieht es mit der Industrialisierung aus? Der Fachbeitrag erklärt Details zu diesem nachhaltigen Rohstoff.

Der Pyrolyseprozess ist ein thermischer Prozess, mit dem Biomasse und Polymerabfälle unter Ausschluss von Sauerstoff auf 250 - 750°C erhitzt werden und dabei in kleinere Moleküle gespalten werden. Beim Einsatz von PKW-Altreifen oder PKW-Altreifen-Granulat entsteht typischerweise ein nicht kondensierbares Kohlenwasserstoffgas (~10%), ein hocharomatisches Pyrolyseöl (~45%), Roh-Pyrolyseruß (~33%), sowie Recycling-Stahl (~12%).

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Recovered Carbon Black Grundlagen

Das Gas kann in der Pyrolyseanlage zur Wärme- oder Stromerzeugung verwendet werden. Das Öl kann sowohl thermisch als schweres Heizöl als auch stofflich als Rohstoff für Polymere und Industrieruß verwertet werden. Der Roh-Pyrolyseruß kann durch Vermahlung und Granulierung zu recovered Carbon Black aufbereitet werden. Recovered Carbon Black ist ein „grünes“ Substitut für Industrieruße, die in Gummianwendungen mechanische Verstärkung bieten und in Kunststoffen, Farben, Lacken und Druckfarben schwarze Färbung, UV-Schutz, thermische und elektrische Leitfähigkeit sowie Opazität erzeugen.

Was unterscheidet recovered Carbon Black (rCB) von Industrieruß? rCB ist der Festrückstand des Pyrolyseprozesses. Alle nichtflüchtigen Bestandteile

der Reifen gelangen in den Festrückstand: Kieselsäure, Zinksulfid und die Industrieruße der verschiedenen Reifenteile. Weiterhin können durch den Pyrolyseprozess verkockte Polymerreste als Kohlenstoff im rCB sein. Ein guter rCB-Produktionsprozess fängt mit einer Qualitätskontrolle des Rohstoffs an, den Altreifen. Altreifen haben je nach Typ und Marke eine unterschiedliche Zusammensetzung. Führende rCB-Produzenten messen daher die chemische Zusammensetzung eines jeden Reifens, der in den Produktionsprozess geht.

Industrie und Technik

Weltweit gibt es mehr als 200 aktive Firmen, die Technologie spezifisch zur Produktion von recovered Carbon Black entwickeln. Die meisten Firmen konzentrieren Ihre Entwicklung allerdings auf den Pyrolysereaktor, der die



Altreifen in Gas, Öl, Roh-Pyrolyseruß und Stahl zerlegt. Zur Aufbereitung des Roh-Pyrolyserußes werden meist Mühlen und Granulierer von bekannten Pulververarbeitungs-Firmen eingesetzt.

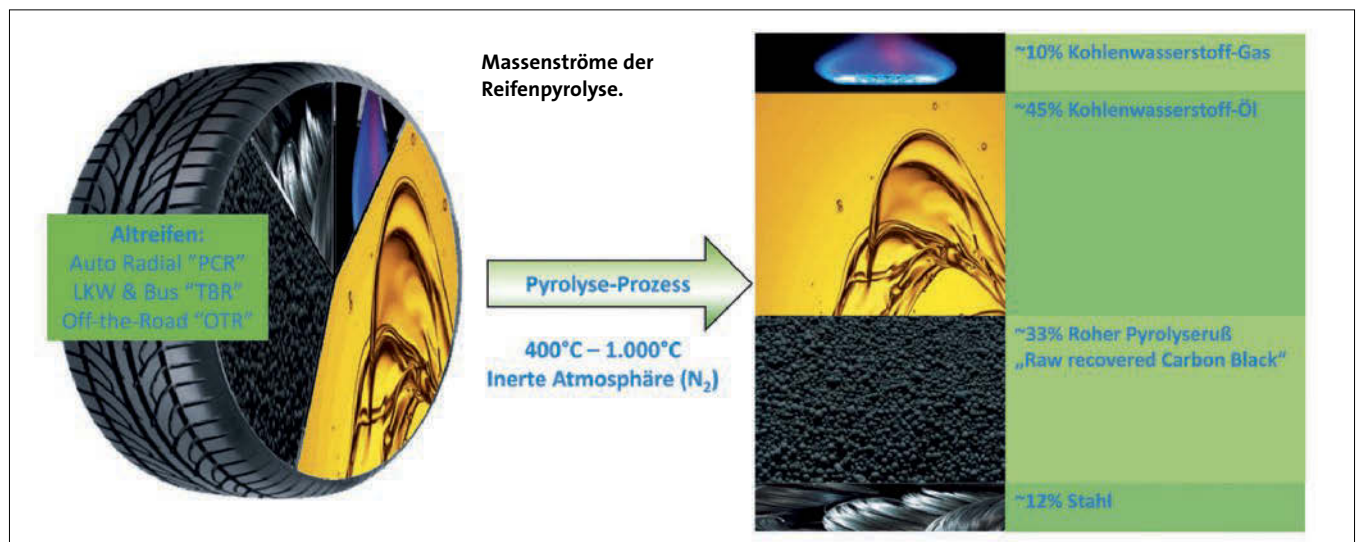
Grundsätzlich gibt es kontinuierliche und diskontinuierliche (Batch) Pyrolyse-Reaktoren. Kontinuierliche Reaktoren haben vor allem Vorteile und Skaleneffekte in Großanlagen, die mehr als 20.000 Jahrestonnen Altreifen verarbeiten. Eine weitere Klassifizierung kann durch die Art der Wärmeübertragung gemacht werden. Es kommen vorwiegend indirekte Wärmeübertragungssysteme zum Einsatz, wie zum Beispiel Gas-beheizte, Dampf-beheizte oder elektrisch beheizte Systeme. Die

direkte Wärmeübertragung mit Mikrowellen ist eine neuere und für recovered Carbon Black sehr interessante Entwicklung. Bei den kontinuierlichen Systemen kommen vor allem Drehrohröfen und Schneckenreaktoren, aber auch Bandförderer zum Einsatz.

Diskontinuierliche Produktion

Zu den Vorteilen der diskontinuierlichen Systeme gehört, dass ganze Reifen pyrolysiert werden können. Dies erspart die kostspielige mechanische Vorbereitung durch mehrstufige Schredder und Granulierer. Besonders attraktiv ist dies beim Recycling von großen Agri-, OTR- und Minen-LKW-Reifen. Auch kann durch die Verweilzeit, oft acht bis zwölf

Stunden, sehr gut eingestellt werden, dass der Roh-Pyrolyseruß fast keine flüchtigen organischen Bestandteile aus der Polymerersetzung enthält. Der besonders geringe Gehalt an polyzyklischen aromatischen Kohlenwasserstoffen (PAKs) ist ein Vorteil von rCB gegenüber normalem Furnace Industrieruß. Zu den Nachteilen diskontinuierlicher Systeme gehören die schwierige Skalierung sowie der typischerweise hohe Koksgehalt des Roh-Pyrolyserußes. Koks entsteht durch die Karbonisierung der an den Industrierußen gebundenen Gummipolymere und lagert sich auf den Industrierußen ab. Dadurch wird die Aktivität und Verstärkungswirkung der Industrierußen vermindert.





Farbsimulation von
RAL Schwarztönen
und rCB.

Kontinuierliche Anlagen

Zu den Vorteilen der kontinuierlichen Systeme gehört die bessere Skalierbarkeit. Neue recovered Carbon Black Projekte planen oft die Produktion von 10.000 bis 30.000 Jahrestonnen recovered Carbon Black. Hierfür sind kontinuierliche Pyrolysesysteme ideal. Die meisten kontinuierlichen Systeme benötigen eine Vorzerkleinerung der Altreifen auf ein Gummigranulat von 3 mm bis 2 cm. Einige Spezialkonstruktionen können allerdings auch zwei bis acht komplette Minenreifen aufnehmen, von denen jeder ein Gewicht von bis zu 5 Tonnen haben kann. Während die Koksbildung typischerweise geringer ist als bei diskontinuierlichen Systemen, ist es oft eine Herausforderung, die Polymerreste, die aus dem gebundenen Gummi entstehen, zu verdampfen.

Roh-Pyrolyseruß aus kontinuierlichen Systemen hat oft zunächst einen merklichen Gehalt an organischen flüchtigen Stoffen. Durch die Regelung der Temperatur, der Verweilzeit und der Größe des Gummigranulats kann aber auch in kontinuierlichen Systemen der Gehalt an flüchtigen organischen Stoffen inklusive PAKs minimiert werden. Sonderformen von kontinuierlichen Systemen sind zum Beispiel Vakuum-Pyrolysereaktoren und Mikrowellen-Systeme, die sich beide positiv auf die Qualität von recovered Carbon Black auswirken.

Recovered Carbon Black Anwendungen

Während in den Anfängen der Pyrolyseindustrie, in der Ölkrise vor 50 Jahren, vorwiegend das Pyrolyseöl von Interesse war, wurden in den 1980er Jahren erste Prototypen von recovered Carbon Black entwickelt. Die Bezeichnung war CBp, Abkürzung für carbon black pyrolytic, also Pyrolyseruß. In Vergleichen

mit Industrierußen wurde eine schwächere Verstärkung in Gummianwendungen festgestellt. Dies ist mit der Zusammensetzung des Pyrolyserußes erklärbar.

Als Festrückstand der Reifenpyrolyse besteht Pyrolyseruß aus der Summe aller in den verschiedenen Reifenteilen verwendeten Industrierußen. Dazu kommen Zinksulfid und, vor allem bei der Pyrolyse von Europäischen PKW-Reifen, Kieselsäure, die den Industrieruß in vielen Laufflächen-Anwendungen ersetzt hat. Prozessbedingt können auch noch flüchtige organische Bestandteile sowie Koks dazukommen.

Die vom Industrieruß unterschiedliche Zusammensetzung wirkt sich auf die Anwendungen des recovered Carbon Blacks aus. In Gummianwendungen kommt typischer rCB einem ASTM N772 beziehungsweise einem halb verstärkenden SRF Industrieruß ähnlich. Andere halb verstärkende Industrierußen wie ASTM N660 und ASTM N550 können je nach Einsatz teilweise ersetzt werden. Anwendern von recovered Carbon Black gefällt vor allem, dass es sich um ein 100% recyceltes Material handelt, dass die CO₂-Belastung um eine Größenordnung tiefer ist als beim Industrieruß, sowie dass rCB einen sehr niedrigen Gehalt an PAKs haben kann.

Pluspunkte für die Gummi-Kreislaufwirtschaft

Marktseitige Unterstützung bekommt der recovered Carbon Black vom Co-Produkt Pyrolyseöl, das ebenfalls eine hohe Nachfrage hat. Für das Projekt Chemcycle benötigt der BASF-Konzern große Mengen chemisch recycelter Rohstoffströme, um daraus wieder Polymere herzustellen, zum Beispiel Polyamid. Reifenpyrolyseöl ist hierfür besonders interessant, da es durch das in

den Reifen verwendete Naturgummi sogar einen Anteil Biomasse hat. Auch das EU-geförderte Projekt Blackcycle macht sich die Kreislaufführung zu Nutze. Das von Michelin geführte Projekt mit zwölf Partnern soll Altreifen-Gummipulver, recovered Carbon Black sowie aus Reifenpyrolyseöl bei Orion Engineered Carbons hergestellten nachhaltigen Industrieruß in die Reifenproduktion bei Michelin bringen.

Reifenhersteller nutzen rCB

Zu den heutigen Anwendern von recovered Carbon Black in der Reifenindustrie zählen zum Beispiel Bridgestone Americas, Continental und Michelin. Bridgestone Americas hat eine Beteiligung am amerikanischen rCB-Produzenten Delta Energy und hat rCB erfolgreich in Innerliner-Anwendungen getestet, bei denen das Lufthaltevermögen der Innerliner besser war als das von Innerlinern mit ASTM N660 Industrieruß. Continental hatte einen Liefervertrag für 10.000 Jahrestonnen mit dem rCB Produzenten Pyrolyx. Michelin hat in den Schwedischen rCB Produzenten Scandinavian Enviro Systems investiert und arbeitet ebenfalls mit der Deutschen Technologiefirma Pyrum Innovations im Rahmen des EU geförderten Blackcycle-Projektes. Sogar Pirelli, ein Reifenhersteller, der für seine Formel-1- und seine Motorsport-Reifen bekannt ist, interessiert sich für recovered Carbon Black.

rCB in Kunststoffcompounds

Auch in Kunststoff-Anwendungen hat rCB Einzug gehalten. Weltweit gibt es mindestens fünf Farbkonzentrat-Produzenten, die Masterbatches mit rCB anbieten, zum Beispiel Avient, Modern Dispersions, Ampacet, Cromex und Cabot Plastics. Cromex bietet sogar eine

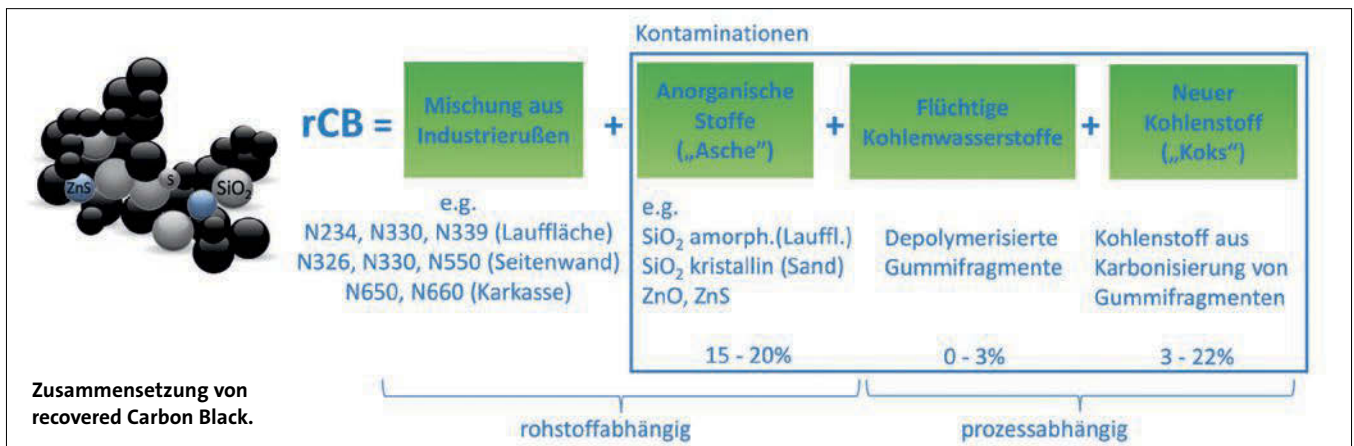


Bild: Wolfersdorff

volle Palette von Schwarzbatches in verschiedenen Trägerpolymeren wie PE, PP, PS, PA, PET und Universal Carrier an. Einschränkungen im Schwarzgrad bestehen für recovered Carbon Black durch den Gehalt an anorganischen Bestandteilen (oft auch Aschegehalt genannt). Dadurch ist rCB eher ein dunkles Graupigment als ein Schwarzpigment. Optimiert werden kann die Farbe durch eine Auswahl der Altreifen. LKW- und Agri-Reifen enthalten zum Beispiel weniger anorganische Stoffe als PKW-Reifen.

Zu den anspruchsvollen Anwendungen mit sehr hohen Qualitätsanforderungen an den rCB gehören Anwendungen in Farben und Lacken sowie als Batterie-Elektrodenmaterial. In Farben,

die heutzutage oft wasserbasiert sind, benötigen die von Natur aus hydrophobischen Industrieruße und auch der recovered Carbon Blacks eine aufwendige Nachbehandlung. Um als Elektrodenmaterial verwendet werden zu können, muss zum einen der Aschegehalt des rCB entfernt werden und auch eine Aktivierung bzw. Oberflächenvergrößerung vorgenommen werden.

Rohstoff-Kontrolle

Die Auswahl der Reifenart ist noch nicht ausreichend für eine gute Kontrolle der Qualitätskonsistenz. Eine Verbesserung bringt die thermo-gravimetrische Analyse (TGA) der Altreifen, mit der auf einen konsistenten Aschegehalt im rCB optimiert werden kann. Eine weitere Verbesserung bringt die vom Niederländischen rCB Produzenten Black Bear Carbon patentierte Technologie, mit Hilfe eines Online-Röntgendiffraktometers jeden Altreifen in seiner chemischen Zusammensetzung zu analysieren und die Reifen dann daraufhin gruppiert zu verarbeiten. Der amerikanische rCB Produzent Bolder Industries hat ein Qualitätssystem eingeführt, mit dem die Produkte in regelmäßigen Intervallen auf Ihre Konsistenz geprüft werden. Eine solch hohes Niveau der Qualitätskontrolle ist nötig, um Farbkonsistenz in Kunststoffen und färbenden Anwendungen zu erreichen und eine sichere Anwendung mit konsistenter Gummiverstärkung in Reifen.

Blick in die Zukunft des rCB-Markts

Fast jede Woche gibt es neue Pressemitteilungen über neue Reifenpyrolyse-Projekte. Aber die Verfügbarkeit von recovered Carbon Black ist in 2021 noch unzureichend im Vergleich zur Nachfrage. Mit dem Einstieg von gro-

ßen Firmen aus der Chemie- und Reifen-Branche wie BASF und Michelin ist nun die Hoffnung groß, dass sich nach 50 Jahren Entwicklungszeit Pyrolyseprozesse endlich mit nachgeschalteten Aufbereitungsprozessen für Öl und rCB auf ein industrielles Niveau etablieren.

Ein ganz wichtiger Meilenstein für die Vermarktung von recovered Carbon Black ist dabei die Definition von allgemein akzeptierten Qualitätsstandards und Testmethoden.

Die ASTM Arbeitsgruppe D36 arbeitet an solchen Standards seit 2016, hat aber bisher nur eine Nomenklatur-Norm herausgebracht. Die nächsten 2-3 Jahre versprechen, interessant für die Industrie zu werden, da viele rCB Produzenten planen, neue Werke in Betrieb zu nehmen!

Fachkongress zu rCB

Seit 2019 hat recovered Carbon Black auch eine eigene Konferenz, den Recovered Carbon Black Congress, der im Mai 2019 in Berlin von Wolfersdorff Consulting Berlin und Smithers zum ersten Mal veranstaltet wurde. Während im Jahr 2020 eine Online-Konferenz lief, gibt es Hoffnungen, dass der Recovered Carbon Black Congress im September 2021 wieder als Präsenzveranstaltung stattfinden kann. ■

1 PAK = (krebserregende) Polyzyklische Aromatische Kohlenwasserstoffe

2 EU PAK8 = Von der EU Kommission definierte Gruppe der acht wichtigsten PAKs: Benzo(a)pyren, Chrysen, Benz(a)anthracen, Benzo(b)fluoranthren, Benzo(k)fluoranthren, Benzo(g,h,i)perylen, Dibenz(a,h)anthracen und Indeno(1,2,3-c,d)pyren

KONTAKT

► Wolfersdorff Consulting, Berlin,
www.wolfersdorff.com

WEBINAR-ANGEBOT

Beratung zu rCB

Wolfersdorff Consulting Berlin berät globale rCB Produzenten, Investoren und große Reifenhersteller wie Michelin und Bridgestone. Für die Beratungsarbeit hat sich als sinnvoll herausgestellt, Qualitätskategorien zu definieren. Die Berliner Beratungsagentur hat daher drei vorläufige Qualitätskategorien definiert. Eine "rCB1" Qualität entspricht dem Stand der Technik für recovered Carbon Black, der durch Auswahl der Reifen, Prozesskontrolle und einer kontrollierten Aufbereitung erreicht werden kann. Dieser recovered Carbon Black ist auf kleiner als 10 µm vermahlen, verperlt und hat einen PAK-Gehalt von weniger als 8 ppm EU PAK82. "rCB2" entspricht einer zweitklassigen Qualität und "rCB3" einer nicht aufbereiteten Roh-Qualität, die in Gummi und Kunststoffen nicht einsetzbar ist. Webinar-Termine: 10.03. "Carbon Black in Asien", 10.06., 15.09., 10.11.2021

Schallschutzwände aus ELT-Recyclingmaterial sind witterungsbeständig, robust und können auch als Sichtschutz verwendet werden.



Bild: Conradi+Kaiser

INTERVIEW MIT STEPHAN RAU, WDK, ZUR INITIATIVE NEW LIFE

Reifenrecycling bietet Chancen

Neue Produkte aus Alt-Reifen Das Recycling von Kunststoffprodukten ist mittlerweile fester Bestandteil der wachsenden Kreislaufwirtschaft. Auch an der kautschukverarbeitenden Industrie geht diese Anforderung an den Wertstoffkreislauf nicht vorüber. Die Initiative New Life will Produkte aus Kautschuk-Rezyklat fördern und Stephan Rau, Technischer Geschäftsführer des wdk, erläutert im Interview die Bemühungen der Kautschukindustrie dazu.

Warum setzen Sie sich für die Kreislaufwirtschaft ein?

Stephan Rau: Die Wirtschaft hat – zusammen mit anderen Akteuren wie Politik und Gesellschaft – eine wichtige Aufgabe: Wir müssen Wege erarbeiten, mit begrenzten Ressourcen verantwortungsvoll umzugehen. Das Recyceln von Altreifen ist nahezu alternativlos. Recycling ist grundsätzlich eine wichtige Existenzgrundlage für zukünftige Generationen und schafft viele Arbeitsplätze. Langfristig werden Kreislaufwirtschaftsprodukte nicht mehr die Ausnahme darstellen, sondern die Regel sein müssen. Jeder gewonnene oder importierte Rohstoff muss so lange wie möglich eingesetzt werden, denn Deutschland ist rohstoffarm, aber sekundärrohstoffreich. Mit den Produkten der New Life Partner ist das möglich: Ob als Sportbelag, für Akustik-Lösungen, als Designobjekte, bei der Ladungssicherung oder im Bautenschutz. Produkte aus ELT (End-of-Life-Reifen) haben verschiedene Gesichter.

Auf einer Skala von 1 bis 100 – Wie weit sind wir mit den Bemühungen?

Rau: Das ist abhängig von dem jeweiligen Markt, den man betrachtet. Für den ELT-Markt sind wir unseres Erachtens auf dem richtigen Weg. Ich denke, wir haben den halben Weg hinter uns und liegen bei circa 50, denn es gibt gerade auf

politischer Seite noch viel zu tun. Dazu gehört, dass Rezyklate den Neuprodukten der baurechtlichen Zulassung gleichgestellt werden sollten – oder im Idealfall sogar bevorzugt! Was wir brauchen, ist ein klar definiertes Abfall-Ende-Szenario, am besten europaweit. Da sind uns übrigens andere Länder wie Italien und Portugal voraus.

Welche Anstrengungen unternimmt die Politik aktuell, um das Thema weiter auszubauen?

Rau: Die aktuelle Novellierung des Kreislaufwirtschaftsgesetzes ist genau die richtige Vorgehensweise. Für eine stärkere Verbreitung von Recycling-Produkten ist aber auch wichtig, dass nicht nur bei öffentlichen Ausschreibungen auf Nachhaltigkeit geachtet werden muss. Auch für private Bauprojekte sollte es Vorgaben geben, die den Einsatz nachhaltiger Materialien verlangen.

Sind die Anstrengungen ausreichend?

Rau: Nein, es liegt noch ein langer Weg vor uns. Das betrifft nicht nur die Politik, sondern auch die Gesellschaft. Grundsätzlich müssen wir ein Bewusstsein für echte Nachhaltigkeit schaffen und am Ende dieses Prozesses schließlich auch eine Handlungsveränderung herbeiführen. Jede und jeder Einzel-

ne muss sich angesprochen fühlen und sich für die Sache einsetzen: Im privaten Bereich, aber auch bei der Beschaffung am Arbeitsplatz und einem Engagement wie beispielsweise im Rahmen der Initiative New Life.

Was fehlt aus Ihrer Sicht noch, damit das Thema schneller vorankommt?

Rau: Meiner Meinung nach ist es unbedingt erforderlich, dass alle staatlichen Stellen ihre Verantwortung für eine Kreislaufwirtschaft ernst nehmen. Das Spannungsfeld zwischen wirtschaftlichen Interessen der Industrie und dem Verbraucherschutz wird von der gesamten Branche sehr ernst genommen. Es darf jedoch nicht passieren, dass Regularien aufgestellt werden, die die Fortentwicklung der Kreislaufwirtschaft behindern.

Wo sind die Hürden, um in Deutschland zu einer perfekten Kreislaufwirtschaft zu kommen?

Rau: Ich denke, dass Politik und Wirtschaft noch stärker an einem Strang ziehen müssen. Es dürfen keine unbegründeten Hindernisse durch Zulassungsbehörden aufgestellt werden. Stattdessen ist es mein Wunsch, gemeinsam den aktuellen Forschungsstand zu betrachten und Lösungen zu erarbeiten, die Nachhaltigkeit ermöglichen. Aktuell steht der Verbraucherschutz weit oben auf der Agenda der Politik, scheinbar aus dem Bauch heraus gibt es immer wieder neue Restriktionen. Das geht zu Lasten der Industrie, gefährdet Arbeitsplätze und untergräbt letztlich eine fortschrittliche Kreislaufwirtschaft. Dazu kommt, dass die Medien wenig Interesse an einer Richtigstellung haben und falsche Informationspolitik nahezu befeuern.

Was sind die Schwierigkeiten im Hinblick auf Kreislaufwirtschaft bei End-of-Life-Tyres (ELT)?

Rau: Tendenziell wird die Menge an Altreifen in Zukunft steigen, trotzdem müssen ELT-Produkte extrem lange Zulassungsverfahren durchlaufen. Ziel sollte es sein, diese Verfahren so kurz und pragmatisch wie möglich zu gestalten. Unternehmen, die innovative Lösungen zur Nutzung von ELT gefunden haben, sollten hierbei unterstützt werden. Darüber hinaus sollten Forschung und Entwicklung an Hochschulen, aber auch in Unternehmen gefördert werden. Schon jetzt sind die Investitionen in Forschung und Entwicklung zum Zweck des Gesundheitsschutzes im Vergleich zur Größe der Branche überdurchschnittlich hoch. Das sollte von der Politik gewürdigt und unterstützt werden.

Was wünschen Sie sich für die Zukunft?

Rau: Gesamtgesellschaftlich wünsche ich mir ein Bekenntnis dazu, dass Recycling und Recyclingprodukte unverzichtbar sind. Für meine Arbeit wünsche ich mir, dass wir es schaffen, die Zusammenarbeit zwischen Politik, Wirtschaft und Verbänden noch stärker auszubauen. Nur, wenn alle Interessensvertreter konstruktiv im ständigen Austausch stehen, kann ein praxisorientiertes Ergebnis erzielt werden. ■



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„Langfristig werden Kreislaufwirtschaftsprodukte nicht mehr die Ausnahme darstellen, sondern die Regel sein müssen.“

ktiv im ständigen Austausch stehen, kann ein praxisorientiertes Ergebnis erzielt werden. ■

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Bei der stofflichen Verwertung werden Reifen in ihre Bestandteile aufgeteilt. Das Granulat ist in verschiedenen Körnungen verfügbar.

Bild: Genan

Reichweitenoptimierung – Made in China

Thermomanagement von E-Fahrzeugen Die Elektromobilität erfordert die Entwicklung neuer Motorkomponenten. Der Einsatz von Kühlmittelsteuerventilen ermöglicht über ein gezieltes Wärmemanagement, Batteriestrom zu sparen und somit die Reichweite eines Elektrofahrzeugs zu erhöhen. Die Kombination von Materialeigenschaften und Verarbeitung waren bei der Auftragsvergabe eines chinesischen Automobilzulieferers ebenso ausschlaggebend, wie das umfangreiche Servicepaket des Werkstoffherstellers.

China ist der weltweit größte Fahrzeugmarkt. Im Jahr 2019 wurden dort über 25 Mio. Autos verkauft. Aktuell erholt sich dieser Markt ungewöhnlich schnell von der Corona-Krise: Der Absatz stieg im Juni gegenüber dem Vorjahresmonat um rund 11 Prozent auf 2,28 Mio. Fahrzeuge an. Fast alle in China verkauften Automobile werden auch dort hergestellt. Einer dieser Hersteller ist Zhejiang Geely Holding (ZGH), Hang-zhou, allgemein bekannt als Geely. Die ZGH Gruppe erzielte einen Gesamtabsatz von über 2.178 Mio. Fahrzeugen im Jahr 2019 (zum Vergleich: die Mercedes-Benz Car Group verkaufte im gleichen Zeitraum 2.385 Mio. Einheiten weltweit). Schon heute kooperieren Geely und Volvo bei der Umstellung auf elektrische Antriebe. Gleich mehrere Modelle basieren auf der gemeinsamen Plattform „Pure Electric Modular Architecture“ (PMA). Diese wird laut Geely die Forschungs- und Entwicklungszeiten verkürzen und die Beschaffungskosten für Elektrofahrzeuge senken, insbesondere dann, wenn Geely alle Daten für alle Autohersteller global kostenfrei zugänglich macht.

China hat mittlerweile international die strengsten Vorgaben für Fahrzeug-

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3-Wege-Kühlmittelsteuerventil mit zwangsentformten Anschlussstutzen mit Laser geschweißtem Gehäuse aus einer Prototypenabmusterung.

emissionen. Das Land der Mitte hat hier einen erheblichen Vorteil gegenüber anderen: Rund zwei Drittel der weltweiten Lithium-Ionen-Batterien werden in China hergestellt, und die EV-Produktionsstätten des Landes befinden sich in der Nähe der Quelle dieser Komponenten. Zhejiang Yinlun Machinery (Yinlun), Zhejiang, gehört zu den lokal ansässigen Firmen. Abnehmer sind Global Player wie Daimler, Cummins, Caterpillar und Ford. Eine Kooperation aus dem Jahr 2018 macht Yinlun zu einem der Hauptlieferanten für Elektrofahrzeuge von Geely. Im selben Jahr fanden auch die Angebotsanfragen für das hier vorgestellte Kühlmittelsteuerventil statt, wobei auch das PA9T Materialportfolio von Kuraray in Betracht gezogen wurde.

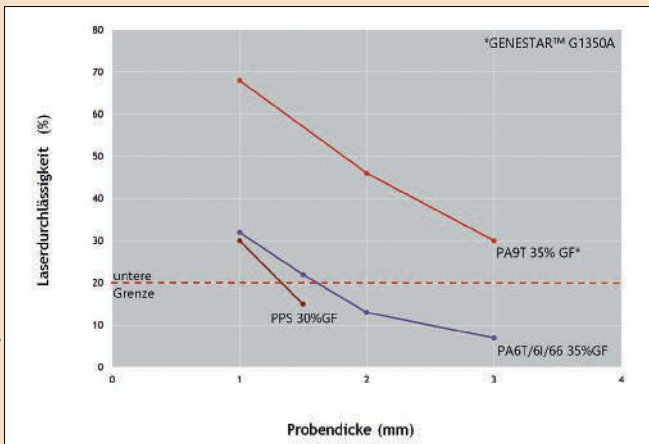
Effizientes Thermomanagement

Insbesondere bei E-Fahrzeugen kommt dem Thermomanagement eine Sonderstellung zu. Ein optimaler Temperaturbereich muss vorgehalten werden, um ein Elektroauto mit einem entsprechend hohen Wirkungsgrad zu betreiben. Deshalb sind Thermomanagementsysteme in E-Fahrzeugen komplexer ausgelegt als in herkömmlichen Fahrzeugen mit einem Verbrennungsmotor: Der Kühlkreislauf und der Kältekreislauf müssen optimal zusammenspielen. Kühlmittelsteuerventile nehmen hierzu entsprechende Befehle von der Steuereinheit entgegen, um den Kühlmittelfluss des Fahrzeugs präzise zu variieren und die gewünschte Temperatur gezielt beizubehalten. Hieraus ergeben sich aus Sicht der OEMs und TIERs die unterschiedlichsten Anforderungen:

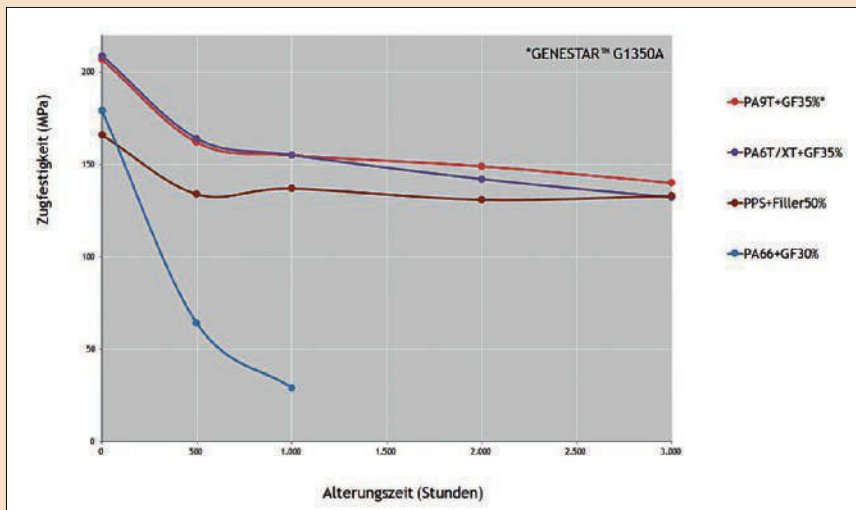
- Robustheit (neben einer Temperaturbeständigkeit von -40 bis 140 °C ist eine hervorragende chemische Beständigkeit gefordert);
- Langlebigkeit (die Dichtheit des Bauteils muss über die gesamte Lebensdauer gewährleistet sein);
- Effizienz: Ein geringer Druckverlust und schnelle Schaltzyklen müssen erreicht werden.

Speziell für den Fahrzeughersteller ist es wichtig zu wissen, ob ein solches Ventil für Verbrenner- und New Energy Vehicles-Umgebungen (NEV) anwendbar ist, und ob sich ein solches Bauteil für den Anbau direkt am Motor eignet. Weitere Anforderungen ergeben sich

Bild: alle Kuraray



Das PPA besitzt eine gute Lasertransparenz auf, wodurch es besonders für das Laserschweißen jenseits von Wandstärken von >1,5 mm geeignet ist.



Durchgeführt im Kuraray R&D Center Tsukuba, gemessen mit ISO A1 Prüfstäben in 50 % Kühlmittel (Toyota „genuine long life coolant“) und 50 % Wasser (die gezeigten Daten entsprechen typischen oder gemessenen Werten).

für die zu integrierende Elektronik und der geforderten Sicherheit. Ein leichter, kompakter und modularer Aufbau ist zwingend notwendig – hierzu gehören auch die verschiedensten Arten der Anbindungen. Um allen Anforderungen gerecht zu werden, war es unumgänglich, das Kühlmittelsteuerventil als Baukasten zu konzipieren.

Kühlmittelsteuerventil – quo vadis?

Auf dem Weg zu einem der führenden Elektroautohersteller hat auch Geely die Notwendigkeit leistungsstarker Kühlmittelsteuerventile erkannt. Der Einsatz solcher Ventile ermöglicht es, mit dem zugehörigen Wärmemanagementsystem, Batteriestrom zu sparen und die Reichweite des Fahrzeugs zu erhöhen. Die Kuraray hat in diesem Projekt ein umfangreiches Paket verschiedenster Dienstleistungen eingesetzt, um Yinlun bei Entwurf, Entwick-

lung, und vor allem bei Herstellung der Aktuatoren und der Ventiltechnologie zu unterstützen. Die Herausforderung bestand für alle Beteiligten darin, das Anforderungsprofil unter einem extremen Zeitdruck in eine Anwendung für einen Kühlkreislauf eines voll elektrisch betriebenen Fahrzeugs umzusetzen:

- Eine (Langzeit-)Kühlmittelbeständigkeit des Materials bei Temperaturen <130 °C,
- das Erreichen von mind. 50 % der Zugfestigkeit nach >2.000 Stunden,
- die Laserschweißbarkeit bei einer Wandstärke von >1,5 mm,
- eine Laserdurchlässigkeit des einzusetzenden Materials von mindestens 25 %, sowie
- erhöhte Anforderungen an die Stoßfestigkeit und Bruchdehnung.

Ein Vergleich der Kühlmittelbeständigkeit gängiger Materialien verdeutlicht das Dilemma: Das Kopf-an-Kopf-

Rennen der gemessenen Zugfestigkeiten über die Zeit lässt nicht eindeutig darauf schließen, welches Material hier in die engere Auswahl genommen werden muss. Im direkten Vergleich der Dichte zu einem PPS Material ist das Bauteil aus Genestar, einem langkettigen Polyphthalamid (PPA), bei gleicher Wandstärke um bis zu 15 % leichter. Schlussendlich war die Laserschweißbarkeit bei Wandstärken bis zu 1,7 mm der vorgeschlagenen Typen ausschlaggebend für den weiteren Verlauf dieser Anwendungsentwicklung.

Fertigung optimiert

Es wurden mehrere Füllstudien für das Gehäuse (Ober-/Unterteil), die Stutzen (Zwangsentformung) und dem Zahnradsatz durchgeführt. Optimale Anspritzpunkte der einzelnen Bauteile und die Laserschweißbarkeit der Gehäusebauteile herausgearbeitet. Ein besonderes Augenmerk lag auf den Verzug, der die Dichtigkeit des Kühlmittelsteuerventils maßgeblich beeinflusst. Weitere Ergebnisse der Untersuchungen ergaben Empfehlungen für Werkzeug- und Schmelzetemperaturen des Granulats. Basierend auf den vorliegenden technischen Daten wurden die Zahnfußspannungen der einzelnen Zahnräder mittels FEM berechnet, um eine Vorhersage für das Ermüden der Materialien zu treffen. Maßgebend war in diesem Zusammenhang auch die Vorgabe der Bauraumreduzierung. Der Nachweis der Laserschweißbarkeit und der geforderten Schweißnahtfestigkeit wurden bei zahlreichen Abmusterungen und Schweißversuchen im R&D Center Tsukuba, Japan erbracht. Die Ergebnisse sind in die Verarbeitungsempfehlungen eingeflossen, um einen reibungslosen Ablauf der Serienfertigung zu gewährleisten (ausgelegt auf eine Jahresproduktion von 1 Mio. Kühlmittelsteuerventile). Letztendlich wurden auch die Abmusterungen für die Prototypen, Vorserienteile bei den drei nominierten Spritzgießern von Yinlun unterstützt.

Durch die Zusammenarbeit der beteiligten Firmen ist es gelungen, Schnittstellen so aufeinander abzustimmen, dass das Kühlmittelsteuerventil individuell einsetzbar ist. Eine Weiterentwicklung des Baukastens ist nicht ausgeschlossen und birgt somit weitere interessante Herausforderungen. ■

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Maschinentechnik kombiniert mit dem Smart-Connect-4.U-Ecosystem vereinfacht die Produktion

AUTOMATISIERUNG Für eine effiziente Produktion steht der Kautschuk-Spritzgießmaschinenhersteller Desma mit seinem Smart-Connect-4.U-Programm. Das System vernetzt die Anlagen und bildet darüberhinaus eine Kommunikations- und Wissensplattform für die gesamte Geschäftsbeziehung. Die Desma gehörte zu den Vorreitern aus dem Maschinenbau für den Kautschuspritzguss, der konkrete Lösungen im Bereich der vernetzten Produktion – Industrie 4.0 – entwickelt hat. Inzwischen beeinflussen einige Megatrends die weitere Entwicklung der Automatisierung in der Produktion. Dazu gehören der demografische Wandel, die fortschreitende Digitalisierung, die immer knapper werdenden Rohstoffe und nicht zuletzt der sich immer stärker abzeichnende Klimawandel. Dies sind Herausforderungen, denen mit den richtigen Produkten begegnet werden kann. Zur Bewältigung der steigenden Komplexität hat sich die Desma auf den Weg zum maschinenbauenden Dienstleister, der sich in die Prozesse seiner Geschäftspartner integriert, gemacht. Dem Kautschukverarbeiter bietet das Unternehmen

neben der Maschine ein auf die Erhöhung seiner Wettbewerbsfähigkeit gerichtetes Wertversprechen und Leistungsangebot. Entscheidend dafür ist eine hohe Transparenz und Reproduzierbarkeit der Produktionsprozesse. Diese schaffen die digitalen Tools der Smart-Connect-4U-Produktfamilie, die Automations- und Verfahrenskompetenz, ein umfassendes Dienstleistungsangebot und das zur Geschäftsprozessintegration dienende Desma-Ecosystem, die Smart-Connect-4.U-Plattformlösung. Was vor drei Jahren mit einer Road-show zum Thema Industrie 4.0 und Maschinenvernetzung international präsentiert wurde, ist mittlerweile zu einer Plattform weiterentwickelt, die neben der reinen Maschinenvernetzung die gesamte Geschäftsbeziehung abbildet und den Kunden, der Desma sowie weiteren Geschäftspartnern auch als interne Kommunikations- und Wissensplattform zur Verfügung steht. Alle zwischenzeitlich weiterentwickelten Automatisierungsprodukte sind integriert und jetzt auch als insgesamt 12 Smart-Connect-4.U Einzelprodukte verfügbar. ■



Smart-Connect-4.U ist eine Kommunikationsplattform, mit der Maschinendaten ausgetauscht werden können, Fernwartung möglich ist und die viele andere digitale Tools enthält.



Bildquelle: Bild: Desma

Das System ermöglicht eine virtuelle Zusammenarbeit und zeigt alle Informationen in einer Plattform.

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Einschneckenextruder für Polyolefin-Rohre



Bild: Bausano

Die Rohrextruder ermöglichen aufgrund ihrer Schneckengeometrie eine hohe Maschinenleistung in kg/h und Polyolefine werden ohne übermäßige mechanische Beanspruchung verarbeitet.

E-GO. Bausano, Rivarolo Canavese, Italien, ist Hersteller maßgeschneiderter Extrusionslinien für die Herstellung von Polyolefin-Rohren. Die Einschnecken-technologie des E-Go ermöglicht die Herstellung von glatten, gewellten, mehrschichtigen oder einschichtigen, starren oder flexiblen Rohren, mit

einem Durchmesser, der von kleinen Größen bis hin zu großen Querschnitten variiert. Zudem gewährleistet er eine gute Homogenität der Schmelze von Kunststoffmaterialien wie PP, PP-R, PE, HDPE, LDPE, PE-X, PE-RT, PMMA, PC, PA und PU. Die Rohrextruder können hohe Maschinenleistung in kg/h erbringen und Polyolefine werden ohne übermäßige mechanische Beanspruchung verarbeitet. Darüber hinaus sind die spezielle Konstruktion sowohl der Schnecke als auch des Zylinders zusammen mit den Asynchronmotoren mit geringer Stromaufnahme eine Garantie für die Energieeffizienz. ■

KONTAKT

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Heiztunnel erwärmt starre Kunststoffrohre und -profile vor dem Schneiden

RS HEIZTUNNEL, MULTICUT

MC-NG Rolf Schlicht, Reinfeld, hat einen RS-Heiztunnel entwickelt. Harte Materialien haben teilweise die Eigenschaft, beim Schlagschnitt im kalten Zustand zu splintern oder Schneidspäne zu erzeugen. Durch das Erwärmen des Materials im Inline-Prozess wird ein sauberes und spanfreies Schnittbild erreicht. Damit entfällt eine teure Nachbearbeitung. Ideal funktioniert der Erwärmungstunnel in Verbindung mit dem RS-Multicut-Rotationsschneider MC-NG des Unternehmens. Die Vorwärmstrecken verfügen in dieser Anwendung über zwei Strahler mit je 3.000 Watt. Die Wärmezufuhr ist geschwindigkeitsabhängig geregelt. Zudem ist eine sehr schnelle Ein- und Ausschaltfunktion integriert, sodass die Wärmemengen prä-



Bild: Rolf Schlicht

Durch das Erwärmen des Materials im Inline-Prozess wird ein sauberes und spanfreies Schnittbild erreicht.

zise gesteuert werden können. Für verschiedene Polymertypen, Produktgrößen und Liniengeschwindigkeiten stehen unterschiedliche Wärmestufen zur Verfügung. ■

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Cut & Chip · OTR tire · weight loss · fiber · optimization · loss tangent

Cut and Chip properties of tire tread along with other physicals were examined for entire range of NR/SBR blends ratio, impacts of Sulphur/ Accelerator ratio, Silica as partial replacement of Carbon Black, Resins and specific short fiber. This also includes optimization of formulation selected using statistical technique with the help of JMP software by SAS. This study reveals that SBR based rubber compounds exhibit excellent Cut and Chip properties. Silica and resins have not shown any definite trend. Aramid and Basalt short fibers were found to increase modulus and stiffness of the tread compounds, but these slightly reduced the tensile strength and elongation at break and hence, marginally reduced Cut and Chip behavior.

“Cut and Chip” – Eigenschaften von Reifen-Kautschukmischungen

Cut and Chip · „Off-the-Road“-Reifen (OTR) · Gewichtsverlust · Fasern · Optimierung · Verlustfaktor

“Cut and Chip“-Verhalten von Reifenlaufflächen wurde zusammen mit anderen physikalischen Eigenschaften für die gesamte Breite an NR/SBR-Blends, bzgl. der Einflüsse von Schwefel/Beschleuniger-Verhältnis, Kieselsäure als teilweise Ersatz für Ruß, Harzen und spezifischen Kurzfasern untersucht. Dieses umfasst auch die Optimierung ausgewählter Rezepturen durch den Einsatz statist. Versuchsplanung mit Hilfe der JMP Software von SAS. Die Studie zeigt, dass SBR basierende Kautschukmischungen sich durch exzellente „Cut and Chip“-Eigenschaften auszeichnen. Kieselsäure und Harze haben keinen eindeutigen Trend gezeigt. Aramid- und Basalt-Kurzfasern erhöhen den Modul und die Steifigkeit von Laufflächenmischungen, aber reduzieren geringe Zugfestigkeit und Bruchdehnung sowie grenzwertig das „Cut and Chip“-Verhalten.

Figures and Tables: By a kind approval of the authors.

Cut and Chip Properties of Tire Rubber Compounds

Introduction

Tires, in off the road application, requires very specific product performance requirement as they are subjected to harsh conditions which may cause reduction in life of the product. Generally, in broader sense, tire life is noted from mileage ability of tread compound, it is addressed in terms of fatigue and abrasive wear in first stage while cutting and chipping in later stages [1].

Since Cut and Chip (C&C) property impacts product life through mileage efficiency, so recently this property has gained importance in tire industry. Numerous researchers addressed this phenomenon to characterize rubber compound on the basis of Cut and Chip resistance. Nah et al. examined Cut and Chip resistance of natural rubber and natural rubber-butadiene blends using B.F. Goodrich Cut and Chip tester. They also investigated field performance of selected compounds with test tires, and it was compared with laboratory test results [2]. Similarly, Radeck et al. explored carbon black filled natural rubber, styrene butadiene rubber and butadiene rubber using their laboratory testing method to characterize fracture arising due to Cut and Chip damage using cycling impact to surface of rubber sample and further quantified them using new physical parameter P (Cut and Chip propensity) [3]. Park et al. further investigated the physical properties of Natural rubber-styrene butadiene rubber blends and natural rubber-butadiene rubber blends with role of silica in carbon black base in different cure system. They addressed the effect of absolute values of 300% modulus on wear and Cut and Chip properties [4]. Scherbakov et al. focused on experimental methods and analytical methods to characterize Cut and Chip phenomena [1]. Recently, rubber industries are facing stiff challenges to fulfill stringent legislation to protect the environment, to reduce the emission and to achieve sustainable solutions. Filler plays vital role in imparting properties to rubber compound, new generation fillers like highly dispersible silica are useful to fulfill these objectives. Bhattacharyya et al. Bhattacharyya et al. analyzed the effect of highly

dispersible silica on physical properties, tearing energy and abrasion resistance on tire tread compounds [5].

On other hand, researchers laid emphasis to reinforce the rubber compounds with the short fibers. These short fiber reinforced rubber composites has become popular in industrial fields because of the processing advantages and increase in strength, stiffness, modulus and damping. The design of a short fiber reinforced rubber composite depends on several factors such as the aspect ratio of the fiber, control of fiber orientation & dispersion and existence of a strong interface between fiber and rubber [6, 7]. Chopped Aramid fibers in typical tire tread compound were found to improve Cut/Chip/Chunk resistance together with abrasion, hysteresis, heat buildup and other dynamic properties [8, 9].

In this work, the effect of fillers namely carbon black and silica on NR and SBR mixed batches were first investigated and the multi level optimization technique was applied to exclude those batches that showed little or insignificant impact on Cut and Chip (C&C) property. First, the impact on C&C property was analyzed using Aramid and Basalt fiber and later correlated with the dynamic property to investigate the nature and behavior of the Cut and Chip effect holistically.

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1 Recipe# of tread compound for phase 1 & 2																				
Material	A1	A2	A3	A4	B1	B2	B3	B4	C1	C2	C3	C4	D1	D2	D3	D4	E1	E2	E3	E4
NR	100	100	100	100	--	--	--	--	50	50	50	50	80	80	80	80	20	20	20	20
SBR 1502	--	--	--	--	100	100	100	100	50	50	50	50	20	20	20	20	80	80	80	80
Carbon Black	60	60	60	38	60	60	60	38	60	60	60	38	60	60	60	38	60	60	60	38
Silica	--	--	--	20	--	--	--	20	--	--	--	20	--	--	--	20	--	--	--	20
Resin	3	3	6	3	3	3	6	3	3	3	6	3	3	3	6	3	3	3	6	3
S*	1.2	0.7	1.2	1.22	1.2	0.7	1.2	1.2	1.2	0.7	1.22	1.22	1.2	0.7	1.21	1.22	1.2	0.7	1.22	1.20
CBS [†]	1.5	2	1.7	1.6	1.5	2	1.7	1.6	1.5	2	1.7	1.6	1.5	2	1.7	1.6	1.5	2	1.7	1.6
DPG [‡]	--	0.95	--	0.3	--	0.95	--	0.3	--	0.95	--	0.3	--	0.95	--	0.3	--	0.95	--	0.3

ZnO-4, Stearic acid-1.5, 6PPD-1 and processing oil-5 phr were used in each formulation *S: Sulphur †CBS : N-Cyclohexyl-2-benzothiazolesulfenamide ‡DPG:Diphenylguanidine

2 Recipe# of tread compound for phase 3						
C4/D1						
Ingredients	A1	A2	A3 (in phr ²)	A4	B1	B2
Aramid fiber	1	2	5	-	-	-
Basalt fiber	-	-	-	1	2	3

phr²: parts per hundred of rubber (in weight)

Experimental details

Materials

Elastomers used in this research were Natural Rubber (NR-RSS 2) and Styrene Butadiene Rubber SBR 1502. Other additives used were short fibers (Aramid and Basalt), antidegradants (6PPD), cure activators (ZnO & Stearic acid), cure accelerators (CBS, DPG) and curative sulfur. Table 1 provides the complete formulation for the rubber compound.

Sample preparations

Table 1 shows the complete recipe# for the phase-1 & phase-2 tread compounds. Phase-1 lists 100% Natural Rubber (NR) batches (A1-A4) and 100% Styrene Butadiene Rubber (SBR) batches (B1-B4). Phase-2 lists batches with different NR/SBR blend ratio. Batches D1 to D4 comprised

of 80/20 blend of NR/SBR, C1 to C4 comprised of 50/50 blend of NR/SBR, and E1 to E4 comprised of 20/80 blend of NR/SBR. Phase-3 was completely based on optimization study on phase-1 and phase-2 batches. Also, the effects of fibers on already optimized results from phase-3 were examined in detail as per recipe mentioned in Table 2.

The rubber compounds were prepared in 1.6L Banbury mixer (BR 1600), Farrel-Corporation. Three stages mixing were adopted for all the compounds presented in this work. For the master batch, first the polymer (NR/SBR) was fed into the mixer for 1 minute mastication, followed by addition of activators and other chemicals mentioned in Table 1. In the final stage, the master batch was mixed with the cure package for the sufficient time and was dumped at drop temperature in the range of 100 to 110°C. It was then

milled and sheeted out on a two-roll mill and was subjected to maturation for 24 hrs. prior to test. All samples were cured at 150°C for time equivalent to Tc90+10 and tested as per test method mentioned in Table 3.

Further, batch screening was carried out for optimization, and finally C4 & D1 compounds were selected to incorporate Aramid and Basalt fiber.

Results and discussion

1. Mechanical properties of phase-1 and phase-2 compounds

Mechanical properties for phase-1 and phase-2 compounds are shown in Tables 4 and 5 respectively. It is observed that 20 phr silica reinforced compounds like A4, C4, D4 and E4 show higher reinforcing effect than their carbon black filled compounds (reinforcing in terms of tensile strength x elongation at break).

As a result, these compounds show higher rebound resilience value. 100% NR filled compounds show higher 300% modulus, and tear strength than 100% SBR filled compounds. This effect gradually decreases as the NR content decreases in the formulation, which is due to characteristic feature of strain induced

3 Recipe of tread compound for phase 1 & 2			
Type of Test	ASTM	Equipments used	Conditions/Parameters
Tensile Test	ASTM D412	Instron testing machine	Crosshead speed of 500 mm/min
Tear Test	ASTM D624	Instron testing machine	Crosshead speed of 500 mm/min
Hardness	ASTM D2240	Multi-unit hardness tester	Shore A
Rebound resilience	ASTM D7121	RR Zwick Roell	100°C and 10 minutes heating of specimens.
Heat build-up	ASTM D623	Goodrich BFG Flexometer	Specimen preheat time: 30min, test time: 25min
Abrasion resistance	ASTM D5963	DIN Abrasion Tester	At room temperature.
Cut and Chip		Dynisco Cut and Chip analyzer	Circular rubber specimens (ø=50mm), rotation speed of 750 rpm, impact frequency = 1 Hz, and rotating time = 10 minutes
DMA	ASTM D5992	Metravib DMA+450	Planar shear mode G*, G', G'' and Tanδ @ dynamic strain: 5%, temperature: 60°C and frequency: 5Hz

4 Mechanical properties for phase 1 compounds									
Parameters	Unit	A1	A2	A3	A4	B1	B2	B3	B4
EB	%	530	532	517	524	635	661	667	669
M100	MPa	2.6	2.6	2.5	2.6	2.3	2.1	2.3	2.4
M300	MPa	14.4	13.3	13.6	14.1	10.6	8.0	9.9	9.8
Tensile strength	MPa	26.6	25.3	25.4	26.7	25.7	20.7	25.7	27.6
Tensile strength x EB	MPa	14098	13459	13131	13990	16319	13682	17141	18464
Hardness	Shore A	66	70	66	67	66	71	68	68
Tear strength	N/m	150.6	116.4	140.2	134.3	55.0	61.3	57.8	56.8
RR @100°C	%	51	42	49	58	46	40	46	49
ARI	%	93	81	92	97	127	131	120	123
C & Cwt. loss	%	7.43	7.58	7.82	7.78	6.23	6.64	6.61	7.19
G*	MPa	2.26	2.55	2.22	2.21	2.41	2.51	2.24	2.35
G'	MPa	2.19	2.46	2.14	2.15	2.31	2.40	2.14	2.28
G''	MPa	0.57	0.69	0.59	0.49	0.67	0.72	0.64	0.59
Tan δ	--	0.261	0.281	0.276	0.226	0.290	0.302	0.300	0.257

crystallization of NR. SBR enhances abrasion resistance index [3]. 100% SBR formulations show better abrasion resistance than 100% NR filled compounds; this effect gradually decreases as SBR content decreases.

2. Dynamic mechanical properties of phase-1 and phase-2 compounds

Dynamic mechanical properties were obtained by DMA and the results are laid down in Tables 4 and 5 for phase-1 and phase-2 respectively. Though, silica rein-

forced compounds show higher reinforcement and lower tan delta, these compounds don't have significant impact on Cut and Chip weight loss (%). It might be due to lower dynamic properties. SBR filled rubber compounds show higher dynamic property.

3. Effect of filler on Cut and Chip property

The C&C is influenced by the nature and type of filler used to reinforce the rubber matrix. The effect of carbon black and its

partial replacement with the silica in different polymer system differing in terms of blend ratio has been widely studied. NR and SBR recipe used here is already mentioned in Table 1. Figure 1 shows the relationship between the C&C weight loss (%) and the loss tangent property with same combination of filler for the different batches. It could be observed that on replacing CB partially with the silica, the loss tangent decreased with lower dynamic property. Weight loss as a measure of Cut and Chip could

5 Mechanical properties for phase 2 compounds													
Parameters	Unit	C1	C2	C3	C4	D1	D2	D3	D4	E1	E2	E3	E4
EB	%	548	615	564	580	578	643	576	571	540	638	568	584
M100	MPa	2.2	2.1	2.1	2.3	2.4	2.3	2.2	2.3	2.4	2.1	2.1	2.3
M300	MPa	12.1	9.7	10.6	10.7	12.1	10.3	12.0	12.5	11.7	9.2	10.8	10.6
Tensile strength	MPa	25.0	22.6	23.7	25.0	25.3	24.5	25.4	27.1	24.4	23.0	24.9	25.3
Tensile strength x EB	MPa	13700	13899	13367	14500	14623	15754	14630	15474	13176	14674	14143	14775
Hardness	Shore A	68	71	67	68	70	73	69	66	69	72	69	68
Tear strength	N/m	125.1	99.5	82.2	126.5	141.1	128.1	135.8	137.6	50.2	55.6	52.3	52.1
RR @100°C	%	49	42	48	53	50	41	49	56	51	45	50	54
ARI	%	110	102	106	111	99	86	95	102	115	116	116	118
C & Cwt. loss	%	7.54	7.45	7.79	7.88	7.84	7.69	8.08	8.36	7.26	7.58	7.1	7.65
G*	MPa	2.13	2.34	2.30	2.27	2.46	2.58	2.23	2.00	2.63	2.86	2.35	2.45
G'	MPa	2.06	2.23	2.20	2.21	2.36	2.46	2.14	1.96	2.52	2.74	2.27	2.38
G''	MPa	0.57	0.72	0.68	0.53	0.72	0.80	0.63	0.40	0.73	0.82	0.63	0.57
Tan δ	--	0.278	0.321	0.310	0.242	0.305	0.327	0.295	0.202	0.289	0.301	0.277	0.241

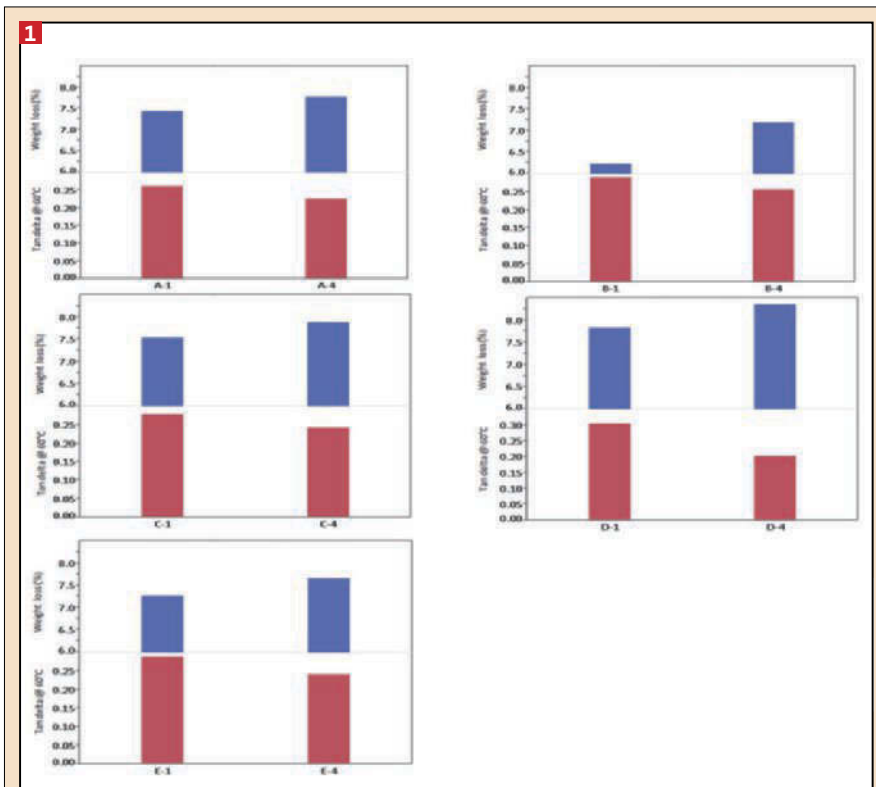


Fig. 1: Relationship between Cut and Chip property against loss tangent with same combination of filler for different NR/SBR blend.

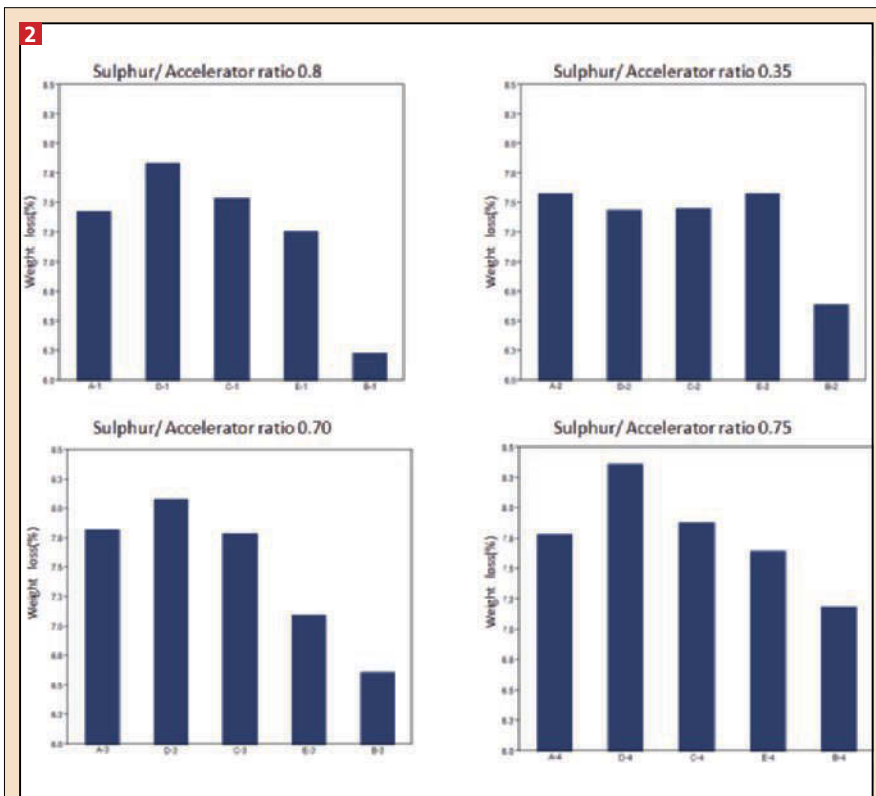


Fig. 2: Correlation of Cut and Chip property vs. NR/SBR blend ratio at different S/Accelerator ratio.

be correlated to loss tangent which showed a trend that as loss tangent gets reduced proving lower dynamic property, the weight loss increased leading to high propensity of cutting and chipping [10].

4. Effect of polymer blend systems on Cut and Chip property

The polymer types and their blends of a tire tread compounds help improve the wear characteristics significantly. At different level of Sulphur/Accelerator ratio, the weight loss was determined to understand C&C behavior. The study reveals the lowest weight loss in pure SBR batches at all levels of Sulphur and Accelerator ratio (See Figure 2).

As NR was partially replaced with the SBR gradually, the weight loss was found to increase. It was noticed that replacement up to 50phr of NR with the SBR did not improve the weight loss but as soon as it crossed 50 phr of SBR, SBR helped in significant improvement in Cut and Chip property. Thus, NR/SBR blend must be used for cutting resistance under low severity conditions.

Batch A1 marks a depth of cut as 721 μm corresponding to the weight loss of 7.43%, while batch B1 shows lower depth of cut as 284 μm corresponding to the significant reduction of weight loss (6.23%). Batch A2 shows a cut depth of 784 μm corresponding to the weight loss of 7.58% while batch B2 shows cut depth of 518 μm corresponding to the weight loss of 6.64% the loss tangent in batch B1 increased and C&C wt. loss decreased as compared to batch A1. Similar is the case in batch B2 as compared to batch A2. This shows a correlation between loss tangent and the C&C wt. loss, which was also confirmed by the smart zoom technology for Cut & Chip specimen which takes depth of cut into account as a measure of Cut and Chip property (See Figure 3).

5. Optimization steps to implement Basalt fiber and Aramid fiber in selected compounds

In order to optimize properties, a methodology was incorporated to screen out compounds from phase-1 and phase-2 to further incorporate Basalt and Aramid fiber in these compounds.

Procedure for optimization is as follows:-
a. Tabulate: Tabulation of all physical property.

b. Magnification: Correction factor is applied on individual property such that it gets magnified enough to be compared with each other.

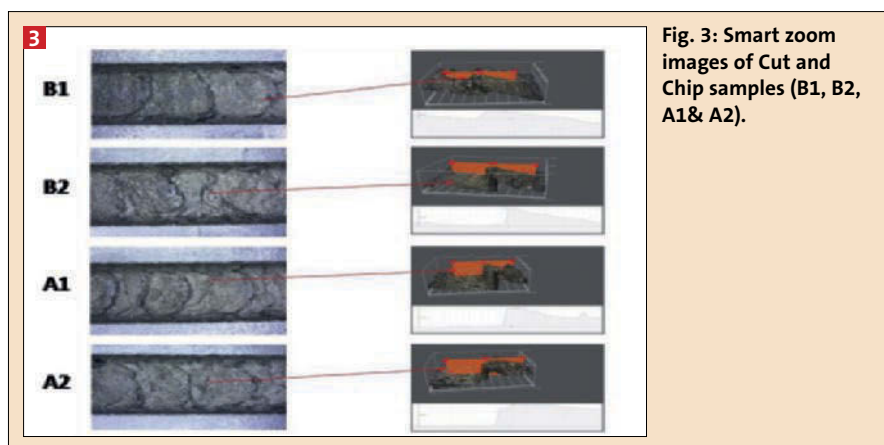


Fig. 3: Smart zoom images of Cut and Chip samples (B1, B2, A1& A2).

6. Mechanical and dynamic property of Basalt and Aramid fiber reinforced compounds.

Mechanical and dynamic properties are listed below in Tables 6 and 7. Fibers help to increase the modulus (at 300% strain) of the composite stretched in the longitudinal direction compared to the reference compounds D1 and C4.

Tensile strength and elongation at break for the fiber-reinforced composite are slightly lower than those for the reference compound. Reduction of tensile strength in low loading of short fibers in carbon black filled rubber was attributed to dilution of particulate fillers in these composites; however, the extent of reduction is not as much to imply weakness of the fiber reinforced composite as a tire tread compound. However, due to higher modulus of the composite stretched in longitudinal direction, the value of elongation at break is slightly lower in this case. Addition of short fibers increases the hardness of the composite relative to the reference compound.

c. Relative scaling: Relative % has been calculated with respect to sum of particular physical property.

d. Screening: By taking arbitrary % value screening is carried out for optimization.

Optimization activity is carried out using chart tool of JMP10 supplied by SAS. Labeling by percentage of total values is used for assigning relative scaling

(%). Physical property listed in Tables 3 and 4 is magnified by taking a suitable correction factor. After 1st optimization A1, A3, A4, C1, C2, C3, C4, D1, D2, D3 and D4 were selected. Further, 2nd optimization was applied to screen amongst the already selected batches from 1st optimization. Then, only compound D1 was selected after 2nd optimization.

6 Summarized physical properties of Basalt and Aramid fiber reinforced compounds

Compound	EB(%)	M300 (MPa)	Tensile Strength (MPa)	Hardness (Shore A)	Tear Strength (N/m)	Rebound resilience @100°C	Cut and Chip weight loss (%)	ARI (%)
C4	580	10.7	25.0	68	127.0	53	7.88	111
D1	578	12.1	25.3	70	141.0	50	7.84	99
C4_BA1	600	10.6	24.7	69	114.2	53	8.41	106
C4_BA2	575	10.8	23.7	69	114.8	52	8.98	105
C4_BA3	554	11.0	23.3	69	82.6	52	9.37	103
D1_BA1	554	12.9	25.2	69	132.5	50	8.21	89
D1_BA2	539	12.2	23.9	71	111.8	51	8.22	86
D1_BA3	551	11.8	23.8	70	126.2	52	8.75	85
C4_AR1	584	10.8	24.2	70	86.4	54	8.44	109
C4_AR2	559	10.8	23.4	72	75.2	51	8.63	104
C4_AR5	530	11.5	21.6	73	96.2	52	9.74	101
D1_AR1	531	13.6	24.7	72	122.2	50	7.94	93
D1_AR2	497	14.0	23.8	74	122.3	49	8.66	91
D1_AR5	463	14.7	22.3	76	109.2	49	9.41	86

7 Dynamic properties of Basalt and Aramid fiber reinforced compounds

Compound code	C4	C4_BA1	C4_BA2	C4_BA3	D1	D1_BA1	D1_BA2	D1_BA3
G*(MPa)	2.27	2.46	2.28	2.39	2.46	2.35	2.43	2.20
G'(MPa)	2.21	2.38	2.22	2.32	2.36	2.26	2.34	2.12
G''(MPa)	0.53	0.59	0.53	0.57	0.72	0.66	0.68	0.61
Tan δ	0.242	0.244	0.236	0.244	0.305	0.292	0.289	0.286
Compound code	C4	C4_AR	C4_AR2	C4_AR5	D1	D1_AR1	D1_AR2	D1_AR5
G*(MPa)	2.27	2.17	2.25	2.48	2.46	2.37	2.54	2.67
G'(MPa)	2.21	2.11	2.18	2.41	2.36	2.28	2.44	2.57
G''(MPa)	0.53	0.53	0.53	0.57	0.72	0.64	0.70	0.74
Tan δ	0.242	0.250	0.241	0.252	0.305	0.282	0.288	0.289

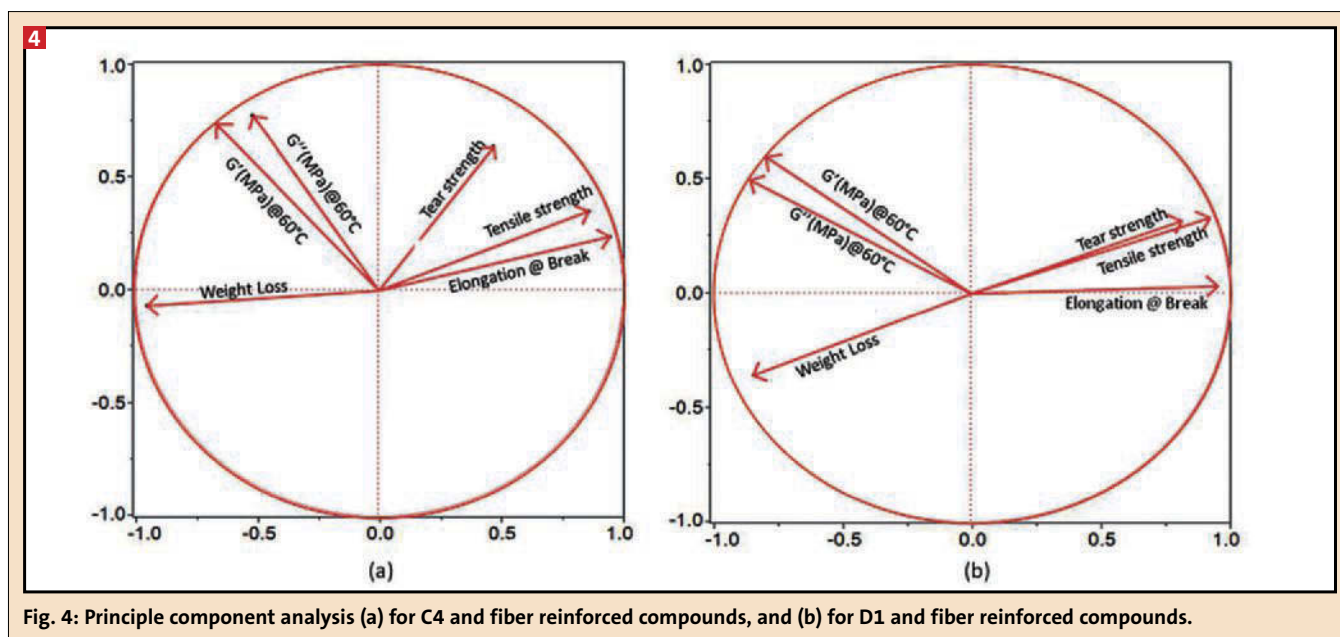


Fig. 4: Principle component analysis (a) for C4 and fiber reinforced compounds, and (b) for D1 and fiber reinforced compounds.

Figure 4 (a) & (b) shows correlation amongst various properties through principle component analysis. Basalt and Aramid fibers reinforced compounds reduce tensile strength and elongation slightly, which impacted negatively on Cut and Chip property. While it reduces energy dissipation anticipated by higher value of tan delta, due to which, in spite of higher dynamic property, gives higher Cut and Chip weight loss (%). Similarly, tear strength shows indirect relationship with Cut and Chip property.

■ The recommended polymer for off road tire tread compound should be NR, for excellent fatigue and failure properties under harsh condition and NR/SBR blend for cutting resistance under low severity conditions.

■ SBR based compounds showed better Cut and Chip resistance and abrasion resistance value under laboratory conditions as compared to NR based compounds.

■ Based on results of this study, replacing 20 phr of carbon black with silica has lowered the hysteresis energy of polymer compound, and hence there is significant reduction in heat buildup. Silica also offers significant advantages in some applications like lowering rolling resistance, but on other hand, it also affects the dynamic property, this may be reason for higher Cut and Chip weight loss.

■ It was shown that Aramid and Basalt short fibers can increase modulus and stiffness of the tread compound, but it

slightly reduces the tensile strength and elongation at break in a uniaxial tension test, which has insignificant effects on mechanical characteristics of tire tread compounds. It reduces energy dissipation anticipated by higher value of tan delta, due to which, in spite of higher dynamic property, gives higher Cut and Chip weight loss (%).

Conflicts of Interest

The authors declare no conflicts of interest in publication of this paper.

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References

- [1.] Scherbakov, M. and Gurvich M.R. A method of wear characterization under cut, chip and chunk conditions. *Journal of Elastomers and Plastics*. **35**, 73. (2003).
- [2.] Nah, C., Jo, B.W., Kaang, S. Cut and chip resistance of NR-BR blend compounds. *Journal of Applied Polymer Science*. **68**, 1537, (1998).
- [3.] Stoček, R., Mars, W.V., Kipscholl, R., Robertson, C.G. Characterization of cut and chip behavior for NR, SBR and BR compounds with an instrumented laboratory device. *Plastics, Rubber and Composites*. **48**, 14, (2019).
- [4.] Park, B.H., Chang, D.H., Song, H.S., Jung, I.T., Cho, C.T. Design Concept of Tread Compound for cutting and chipping re-

sistance of truck tyres on On/Off the roads. *Asian Journal of Chemistry*. **25** (9), 5208, (2013).

- [5.] Bhattacharyya, S., Lodha, V., Dasgupta, S., Mukhopadhyay, R., Guha, A., Sarkar, P., Saha, T., Bhowmick, A.K. Influence of highly dispersible silica filler on the physical properties, tearing energy, and abrasion resistance of tire tread compound. *Journal of Applied Polymer Science*. **136**, 47560, (2019).
- [6.] Norman, D.A., Robertson, R.E. The effect of fiber orientation on toughening of short fiber-reinforced polymers. *Journals of Applied Polymer Science*. **90**, 2740, (2003).
- [7.] Shun, F.S., Lauke, B. Effect of fiber length and fiber orientation distributions on the tensile strength of short-fiber reinforced polymers. *Composites Science and Technology*, **56**, 1179, (1996).
- [8.] Datta, R., and Pierik, S. C. J. Improving cut/chip/chunk resistance by using Sulfron 3000. *Kautschuk und Gummi, Kunststoffe*. **60** (6), 328, (2007).
- [9.] Kashani, M.R. Aramid-short-fiber reinforced rubber as a tire tread composite. *Journal of Applied Polymer Science*. **113**(2), 1355, (2009).
- [10.] Beatty, J.R., Miksch, B.J. A laboratory cutting and chipping tester for evaluating off-the road and heavy duty tire treads. *Rubber Chemistry and Technology*. **55**, 1531, (1982).

Polyacrylate elastomers · Heat resistance · low temperature properties · oil resistance

HyTemp polyacrylate elastomers (ACM) have a proven track record for use in demanding powertrain applications for both sealing and hose components. In this paper, the next-generation HyTemp HT-ACM technologies (Zeon) will be introduced, demonstrating how these new materials are simultaneously extending the boundaries for cold temperature capability toward -40° with improved heat resistance beyond 175°C, all-the-while maintaining mechanical integrity for sealing of advanced, low-viscosity transmission fluids. Long-term aging, with a focus on at-temperature service performance qualities, of HT-ACM will be presented.

Eine neue Generation von Polyacrylat-Elastomeren (ACM) für Dichtungsanwendungen und Flüssigkeitsmanagement in automobilen Getriebesystemen

Polyacrylat-Elastomere · Hitzebeständigkeit · Niedrigtemperatureigenschaften · Ölbeständigkeit

In dieser Publikation wird die nächste Generation der HyTemp HT-ACM Technologie vorgestellt, welche zeigt, wie diese neuen Materialien gleichzeitig die Grenzen der Tieftemperaturleistungsfähigkeit bis zu -40 °C und die Hitzebeständigkeit bis über 175 °C verbessern, während die mechanischen Eigenschaften von Dichtungen in niedrigviskosen Getriebeöleinsatz erweitert werden. In einem weiteren Schwerpunkt dieses Beitrags werden die Änderung der physikalischen Eigenschaften von HT-ACM nach Langzeitalterung - gemessen bei Anwendungstemperatur von 150 °C - im Vergleich zu anderen führenden Elastomeren für Dichtungen im Antriebsstrang vorgestellt. Dies ermöglicht den direkten Rückschluss auf die Materialeigenschaften unter den vorherrschenden Betriebsbedingungen und kann daher als ein realitätsnahes Szenario angesehen werden.

Figures and Tables:
By a kind approval of the authors.

Next-Generation Polymer Solutions for Sealing and Fluid Transport in High-Performance automotive Transmission Systems

In order to improve vehicle efficiency and reduce emissions, vehicle manufacturers are more frequently turning to charged air systems in order to boost power output of their engines. In addition to increasing power output, use of low-viscosity, synthetic engine oils, like 0W-20 weight, has now become an industry standard in order to further reduce power-robbing friction and drag in the engine. Now, some state-of-the-art transmission platforms, such as the Ford 10R80 and other 8 to 10-speed transmissions, are also specifying low and ultra-low viscosity fluids for reduced friction, which can be up to 40% lower viscosity than conventional transmission fluids.

While these efficiency-boosting measures have been effective in achieving their intended result of higher output and lower emission, they have unintentionally generated a more severe operating environment for the elastomeric components sealing and transporting these fluids. Increased engine power output and exhaust routing near the transmission can often create higher operating temperatures within the transmission; and, in the case of ultra-low viscosity fluids, chemical attack or fluid ingress into the elastomer may challenge the long-term integrity or sealing capacity of the elastomer seal, gasket, and hose components. Historically, the elastomeric materials most commonly used in transmission sealing components are fluorinated elastomer, such as FKM, and acrylic monomer-based elastomers, recognized by the ASTM designation of AEM (acrylic ethylene monomer) and ACM (acrylic monomer). Figure 1. provides a generalization of various polymers and their respective heat and oil resistance used in automotive applications. For the purposes of this paper, AEM and ACM will be compared as they are a similar class of elastomer and material cost.

Zeon is a global supplier of innovative elastomeric materials, many of which are engineered to serve in the automoti-

ve industry for use in engine and transmission fluid sealing and fluid management applications. Zeon's polyacrylic elastomer, known as HyTemp ACM, is a fully-saturated thermoset polymer derived from acrylic acid esters and is generally classified as a high-temperature, oil-resistant rubber with long-term service capability from -40 to 150°C, and up to 175°C for Zeon's latest polymer grades designated as 'HT-ACM'. Several examples of HyTemp HT-ACM can be viewed in Table 1, including Zeon's latest commercialized grade, HyTemp H570. H570 offers a broader service temperature range than other HT-ACM grades by providing excellent cold-temperature flexibility while maintaining long term heat aging resistance at 175°C with short-term capability beyond 190°C.

As mentioned earlier, AEM differs from ACM in that it is a co-polymer derived from both acrylic and ethylene monomers. Like ACM, the acrylic portion exhibits good heat and oil resistance while the ethylene portion of the AEM polymer backbone is attributed with providing good cold temperature properties and generally higher physical properties at room-temperature compared to ACM. The difference in polymer structures between ACM and AEM can be viewed in Figure 2.

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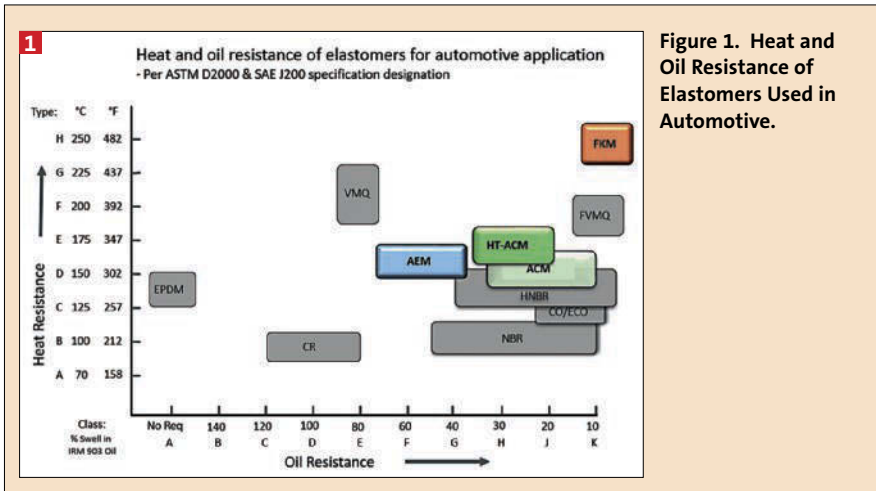
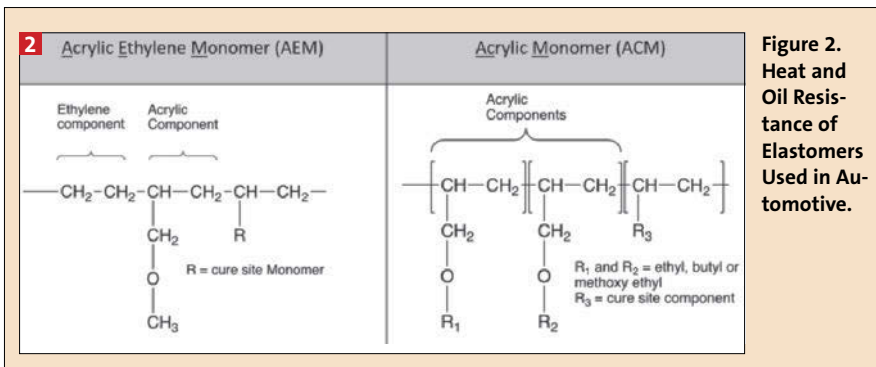


Table 1. Example HyTemp® HT-ACM Grades and Feature

HT-ACM Grade	Characteristic	Feature
AR12	Primary injection/transfer molding	Workhorse grade for seals, gasket, molded duct
AR12B	Primary injection/transfer molding	Fast curing grade of AR12 for Injection/Transfer molding
AR14	Ultra-low temperature flexibility	Use alone or blended with other HT-ACM to adjust low-temp flex
AR22	Increased mechanical strength	Use alone or blended with other HT-ACM to achieve higher tensile strength and adjust oil swell properties
H570	Broad temperature range	New grade that has excellent heat resist properties, plus low temperature flexibility
AR212HR	Primary extrusion grade	Workhorse grade for high heat and oil resistant hose and other extruded products



When automotive engineering teams are developing material specifications for polymers used in fluid sealing and transport, it is common for separate specifications, or material classes, to be written for either ACM or AEM. Oftentimes, the primary differentiator between the two materials specifications is the higher tensile strength or tear strength afforded by AEM at room-temperature, thus restricting consideration of ACM in some applications. In order to confirm a candidate material's suitability for an application, material specifications typically include a series of aging

conditions in hot air or fluids which the component will be exposed to, and in which the material must retain a certain level of physical properties to meet the specification. For example, the aging condition for a transmission sealing component is commonly 150°C in air or immersed in a specified fluid for 1008hrs; as noted in both Ford WSP-M2D432-A and GM GMW3083 specifications. While rubber compounds consisting of either ACM or AEM typically demonstrate good resistance to transmission fluids, it was found that, when measured at

service-temperatures, ACM provides similar or, in some cases superior, physical properties as compared to AEM. The compression set and cold temperature flexibility performance properties evaluated also demonstrate the capability of HT-ACM grades, particularly H570, to perform well in new, ultra-low viscosity transmission fluid sealing applications.

Experimental

Polymer Selection

The HyTemp HT-ACM family of polymers are all constructed of similar acrylic monomers and diamine cure chemistry that provide the basis for its excellent high heat and oil resistance characteristics. Each grade of HT-ACM has been specifically designed to exhibit key functional properties or meet specific technical needs. For example, AR14 (with a glass transition temperature of less than -40°) was designed to provide excellent low-temperature flexibility, but it can be susceptible to high volume swell in certain oils and diminished long-term heat resistance at 175°C. In contrast, AR12 exhibits excellent long-term heat resistance and exceptional oil resistance, but at the sacrifice of cold temperature service capability. Zeon's newest commercial HT-ACM grade, H570, is successful in achieving both long term heat aging resistance of 175°C and very good cold temperature performance with a Tg of -37°C. While there are many other grades of HT-ACM available for commercial use, the grades mentioned above were selected for use in this study due to their commonality for use in gaskets and seal applications, but also their contrasting nature in physical performance characteristics. The compound formulations used in this study are conventional recipes for ACM, only varying the levels of FEF black content in order to achieve finished test specimens with durometers of approximately 60, 70, and 80 shore A hardness. As mentioned, AEM was selected for comparison purposes to ACM as it is similar in chemical make up and cost, but also considered to be the most commonly used sealing material for transmissions in North America [1]). The grade used in this study is AEM-IP, as it promoted by the material manufacturer as having improved high-temperature aging resistance and higher physical properties at elevated temperature compared to standard grades of AEM, like AEM-G [2]).

2 Table 2. Raw materials and formulations of HT-ACM and AEM compounds

Compound Hardness (Sha)	60			70			80			60	70	80
Compound Base Polymer	AR12	H570	AR14	AR12	H570	AR14	AR12	H570	AR14	AEM-IP	AEM-IP	AEM-IP
Polymer	100	100	100	100	100	100	100	100	100	100	100	100
FEF Carbon Black	60	60	60	70	70	70	80	80	80	40	52	72
Steering Acid	1	1	1	1	1	1	1	1	1	1,5	1,5	1,5
Process Aid #1	0,5	0,5	0,5									1
Protection Agent	2	2	2	2	2	2	2	2	2	2	2	2
Ether-Ester Plasticizer										10	10	10
DBU Salt - Polymer bound	2	2	2	2	2	2	2	2	2			
DBU Salt - Powder										2	2	2
Process Aid #2	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5
Diamine Curative	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5	0,5	1,2	1,2	1,2
Total	166,50	166,50	166,50	176,50	176,50	176,50	186,50	186,50	186,50	158,20	170,20	190,20

3 Table 3: Test Materials and Original Physical Properties

Durometer range	60				70				80			
Compound Base Polymer	AR12	H570	AR14	AEM-IP	AR12	H570	AR14	AEM-IP	AR12	H570	AR14	AEM-IP
Hardness A, (pts)	64	64	58	58	73	72	70	69	77	76	74	81
Modulus @ 100 %, (MPa)	3.4	4.4	3.8	2.6	4.8	5.3	6.9	5.2	5.3	6.3	9.5	8.7
Tensile, (MPa)	8.7	10.9	10.4	18.3	9.5	9.8	11.1	18.2	9.1	10.1	11.5	18.6
Elongation, (%)	228	234	203	464	226	206	159	346	211	189	118	248
Tear Strength, Die B, (N/mm)	33	29	21	36	36	31	23	42	38	35	28	45

The compound recipes used for AEM-IP were identical to formulation recommendations as outlined in the material supplier's technical literature for AEM-IP grade, compounded for durometers of 60, 70, and 80 shore A hardness [3]). Table 2 shows the compound formulations used.

Sample preparation

The polymers used in the compounds prepared for this evaluation are outlined in Table 3. All compounds were made using a 1.5L internal mixer with intermeshing rotors. Per standard Zeon recommended process procedure, all mixes were prepared using an “upside-down” mix process and a two-pass method in order to obtain optimal dispersion and mix quality of the compounds. Following a 24hr rest period, the masterbatch then had curative ingredients added in a second pass (final) using a 20.3cm x 20.3 cm x 40.7 cm laboratory roller mill. All mixes were tested using an MDR (Moving Die Rheometer) for 20 minutes at 180°C to confirm cure state. Test sheets were then pressed using a standard ASTM 150mm x 150mm x 2mm, 4 cavity mold, with cure time of 15min at 180°C. Compression set buttons were pressed using ASTM D394 29.0mm x 12.5mm mold. All compounds were pressed consecutively within 48hr of mixing the second pass. Last, 24hr after molding, all sheets and compression set buttons underwent a post cure process for 4 hours at 175°C

Material Testing

This study investigates the aging characteristics of HT-ACM and AEM-IP in low viscosity and ultra-low viscosity automatic transmission fluids, as opposed to conventional DEXRON-VI (DEX-VI) fluid, due to their specification in later model

year transmission platforms. Mobil 1 Synthetic LV ATF HP, which is listed under GM part numbers 19417577 and 9986555, was used for the low viscosity ATF. The ultra-low viscosity ATF selected was Ford MERCON ULV, part number XT-12-QULV, which is specified for 2017 and

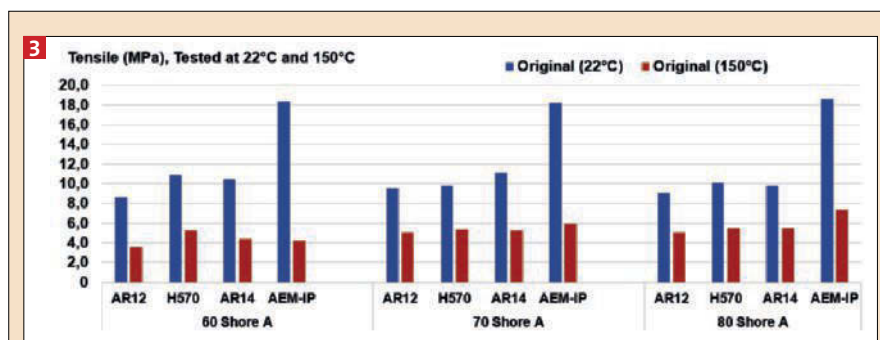


Figure 3. At-temperature Tensile of HT-ACM and AEM-IP.

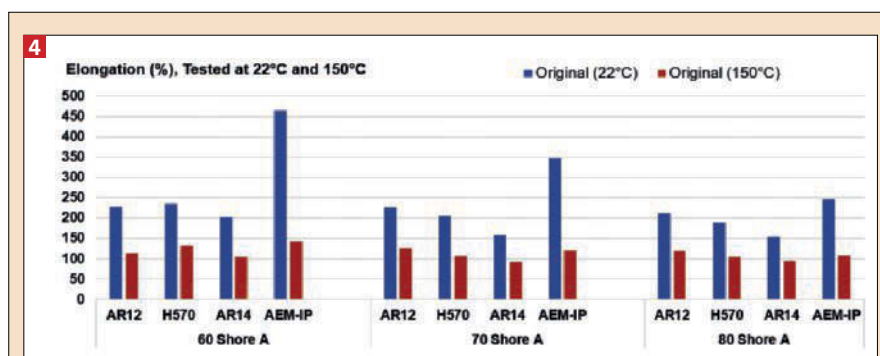


Figure 4. At-temperature Elongation of HT-ACM and AEM-IP.

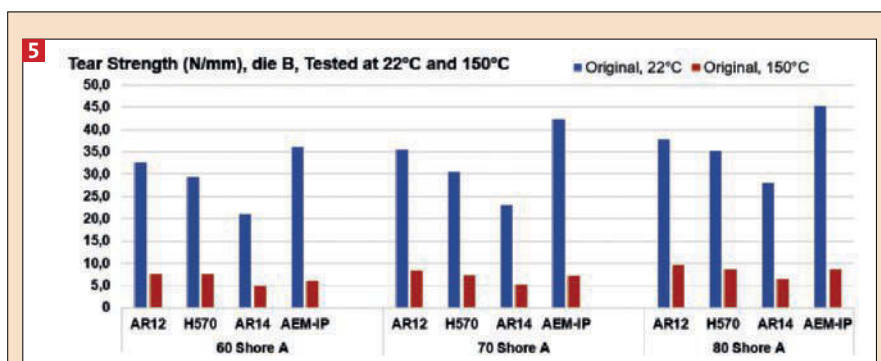


Figure 5. At-temperature Tear of HT-ACM and AEM-IP.

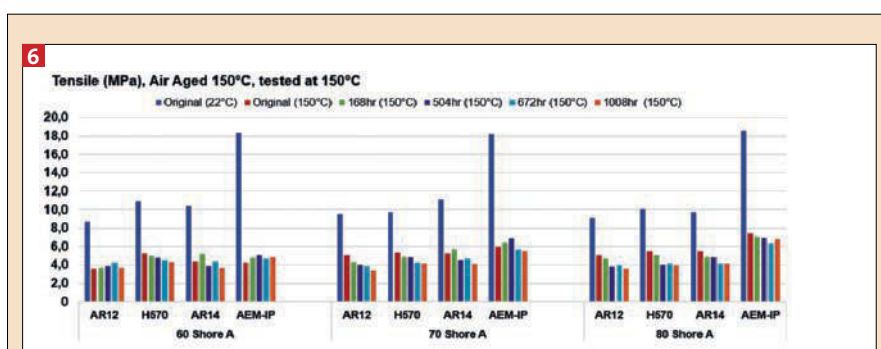


Figure 6. Tensile Strength, Air aged at 150°C, Tested at 150°C.

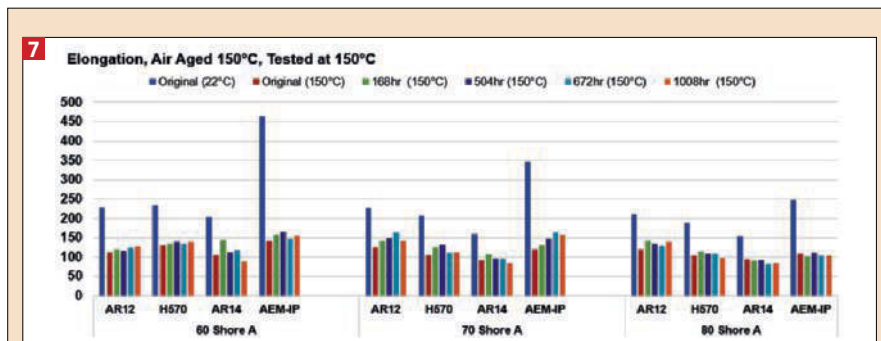


Figure 7. Elongation, Air aged at 150°C, Tested at 150°C.

later Ford F-150 and F-150 Raptor platforms using the 10-speed, 10R80 transmission. Standard hot air aging was also conducted.

The aging temperature chosen for all testing media was 150°C, mirroring the aging conditions, or upper service-temperature range, required in many material transmission sealing specifications. Following the completion of the heat aging period, the physical properties of the aged samples were evaluated to determine the level of properties retained and adherence to specification limits. Unlike most specification testing, which is performed at room-temperature (22°C), this evaluation differs in that the physical property measurements of ori-

ginal and aged samples were also performed at the aging temperature of 150°C. Testing at the elevated temperature demonstrates the materials' performance capabilities in actual operating environment of the application.

The physical properties, measured at a temperature of 150°C, include tensile, elongation, and tear strength. All tear testing utilized the ASTM 'die B' (nicked) test specimen in order to replicate 'worst-case' condition. The elevated temperature testing was performed using an Instron Model #5967 tensile tester with thermal test chamber installed. Specimens were tested following a minimum 15-minute conditioning period at 150°C, with a 3-minute soak peri-

od for each specimen once it was loaded into the Instron test fixture.

Since the new grade of HT-ACM, H570, was designed to improve cold temperature performance, material T_g, as well as TR10 after aging, were evaluated. Finally, compression set of the compounds was compared. Unless specified, all test methodology is based on ASTM standards. For all physical property testing conducted at-temperature (tensile, elongation, and tear resistance), the ultimate (actual) values of the tests were reported instead of %-change, as it was felt by author that the %-change value would not accurately portray a true comparison of the functional capabilities for the materials due to the drastic difference of AEM-IP's physical properties when comparing the room-temperature to hot-temperature results.

HEAT AND FLUID RESISTANCE

As mentioned earlier in this paper, AEM is commonly known to materials engineers to demonstrate significantly higher tensile and elongation properties than ACM at room temperature, and specifications for each material typically reflect this. However, when considering the demands of an application, the properties of a material should be considered at the operating temperature of the application. As seen in Figures 3 and 4, the original physical properties HT-ACM and AEM-IP were measured at room-temperature. The tensile of HT-ACM is roughly 9-11MPa with elongation between 150-250%, while AEM-IP demonstrates a higher tensile property of approximately 18MPa with an elongation of 250-400%. These values are consistent with the typical results expected of the respective polymer grades/families.

The materials were then measured at 150°C, and, as expected, all materials experienced a decrease in both tensile and elongation properties. However, it is noteworthy that the physical properties of AEM-IP were reduced disproportionately to HT-ACM, to the point that both material types were nearly equivalent. Of the lower durometer compounds, H570 retained the highest tensile strength, whereas the AEM-IP maintained higher tensile strength in the higher hardness compounds.

When measuring tear strength, the same phenomenon was observed in that the tear properties of all materials were significantly lower than when measured at room temperature (Figure 5). Although

the initial tear strength of AEM-IP was higher than all HT-ACM compounds, both AR12 and H570 maintained similar-to-higher tear strength than AEM-IP across all durometers when measured at high temperature. AR14 consistently demonstrated the lowest elongation and tear strength results; this is to be expected as the lower physical properties and heat aging characteristics of the material are a compromise for the material's ultra-low use-temperature capability.

AGED TESTING

As a class of materials, HT-ACM elastomers, are considered capable of long-term service (>1008hr) at 175°C in air and engine oil per SAE J2236 automotive standard, meaning that the materials must retain at least 50% of tensile and elongation properties after the aging period. Historical testing of HT-ACM and AEM-IP shows that both materials experience minor loss of properties when aged at 150°C, when tested at room temperature as most material specifications dictate. This study considers the impact on material physical properties of aging in air and LV/ULV ATF when tested at the aging temperature.

AIR AGED

Hot air aging of material compounds was conducted at 150°C, with specimens being tested at 168, 504, 672, and 1008 hours. Aged specimens were then tested at-temperature for tensile, elongation, and tear resistance. Figures 6 and 7 illustrate the shift of materials' original physical properties when tested at both room temperature and 150°C, followed by the aged test results. Similar to testing historically performed at room temperature, both HT-ACM and AEM-IP demonstrate good aged-property retention over the 6-week testing period. All materials maintained an ultimate elongation near or above 100% (a common indicator of a material maintaining good serviceability). The tear strength of both HT-ACM and AEM-IP remained closely matched after aging, across all three durometer ranges (Figure 8.).

FLUID AGED

ACM and HT-ACM have a long history of service in transmission applications using conventional DEXRON-VI ATF. This study evaluates the performance of HT-ACM in the latest-technology, synthetic low and ultra-low viscosity automatic transmission fluids. It is worth to note

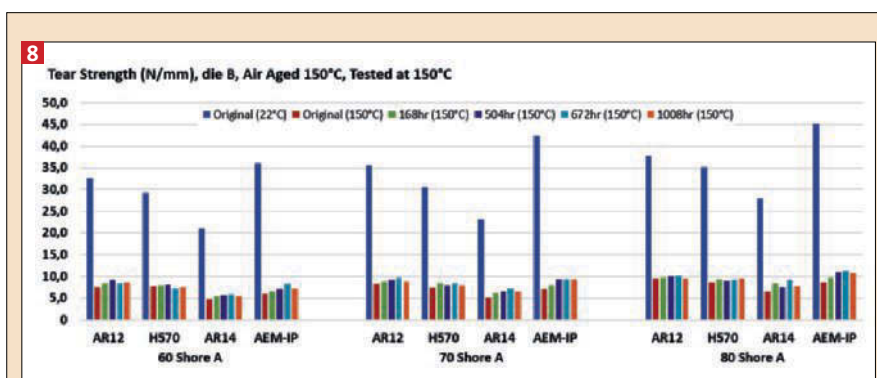


Figure 8. Tear Strength, Air aged at 150°C, Tested at 150°C.

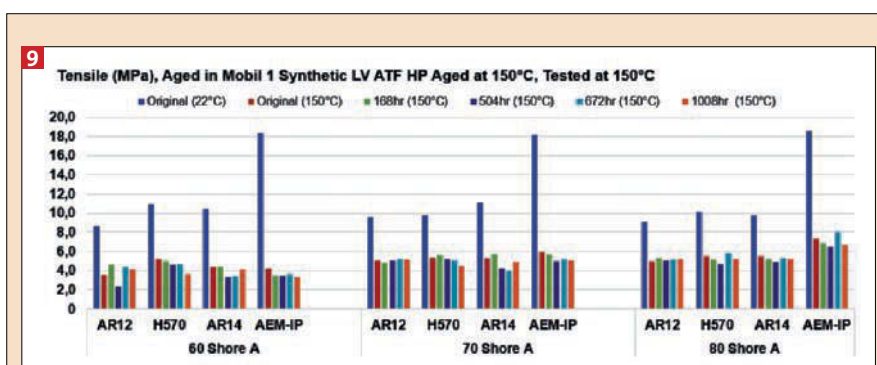


Figure 9. Tensile Strength, Aged in Mobil 1 Synthetic LV ATF HP at 150°C, Tested at 150°C.

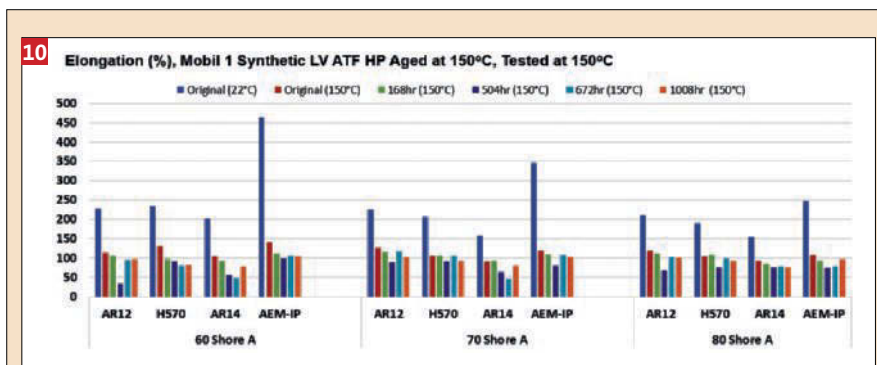


Figure 10. Elongation, Aged in Mobil 1 Synthetic LV ATF HP at 150°C, Tested at 150°C.

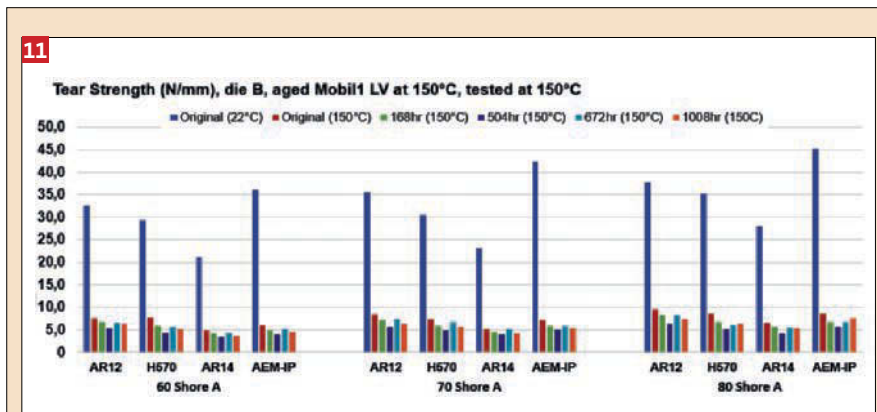


Figure 11. Tear Strength, Aged in Mobil 1 Synthetic LV ATF HP at 150°C, Tested at 150°C.

12

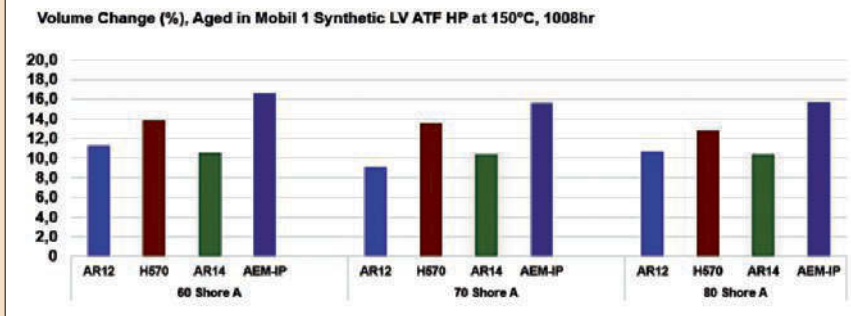


Figure 12. Volume Change, Aged in Mobil 1 Synthetic LV ATF HP at 150°C, Tested at 150°C.

13

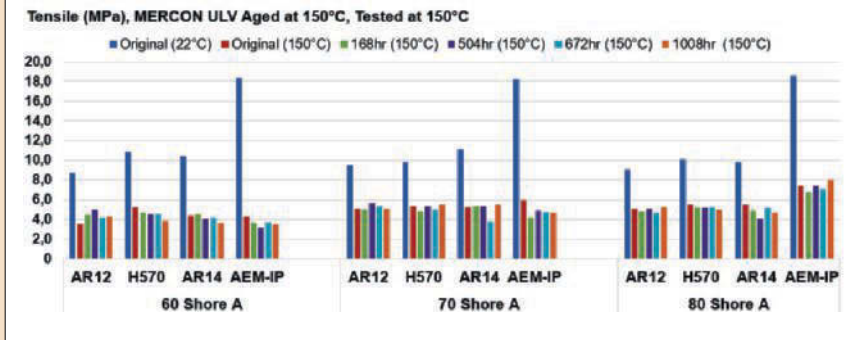


Figure 13. Tensile Strength, Aged in MERCON ULV at 150°C, Tested at 150°C.

14

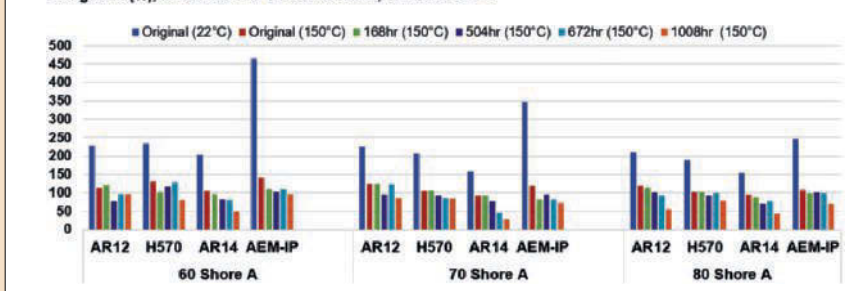


Figure 14. Elongation, Aged in MERCON ULV at 150°C, Tested at 150°C.

15

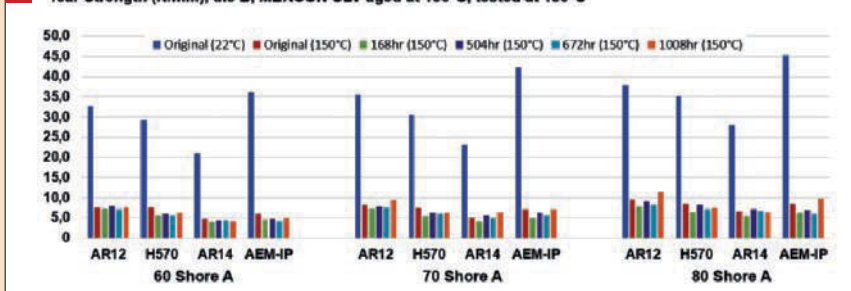


Figure 15. Tear Strength, Aged in MERCON ULV at 150°C, Tested at 150°C.

that no fluid changes were performed in the longer-term testing (>504hr) as many automotive materials testing protocol do not specify a fluid change. Additionally, many later-model transmissions platforms using fully-synthetic ATF are considered 'fill-for-life', where there is no scheduled service, or change, of the vehicle's transmission fluid.

First to be evaluated was the materials' resistance to Mobil 1 Synthetic LV ATF HP. In Figures 9 and 10, the tensile and elongation properties are shown for the original property values at room temperature as compared to 150°C for both the original and specimens aged in Mobil 1 low-viscosity ATF. Similar to the results for air aged results, AEM-IP generally demonstrates a slightly higher tensile than HT-ACM in the higher durometer range, whereas H570 retains highest tensile strength in the lower durometer compounds. With exception of AR14, elongation of all materials remains generally near 100%. After a slight initial drop between 0 and 168hr, the tear resistance of all materials remains remarkably stable for the duration of the aging period; with results of both HT-ACM and AEM-IP being very similar across the durometer ranges (Figure 11). Volume change of HT-ACM grades remained between 10-14% with H570 the having slightly higher swell than AR12 and AR14, and AEM-IP having a volume change of approximately 16% (Figure 12). Acceptable volume change for elastomers in transmission sealing applications is commonly 20-25%.

The second automatic transmission fluid used in this evaluation was Ford Motorcraft MERCON ULV (Ultra-low viscosity ATF). As with the low-viscosity fluid, the tensile strengths of HT-ACM and AEM-IP remained relatively stable during the 1008hr immersions test (Figure 13). However, as seen in Figure 14, the ultra-low viscosity fluid appears to be more aggressive, compared to the LV fluid, in that elongation properties of all HT-ACM and AEM-IP compounds trend downward toward the end of the aging period. The degradation of elongation is most clearly recognized in the AR14, which exhibits chronic loss of elongation over the entire aging period, in that it achieved only 30-50% actual elongation value after 1008hr. AR12, H570, and AEM-IP all maintain between 50-100% actual elongation. The tear strengths for all compounds mirror the stable tensile proper-

ties in that very little property loss is observed throughout aging period (Figure 15). Finally, despite the lower viscosity of the MERCON ULV fluid, volume change of both HT-ACM and AEM-IP was slightly lower, on average, as was observed when aged in the Mobil 1 LV fluid. Like in the Mobil 1 fluid, H570 exhibited 3-4% higher volume change than AR12 or AR14, but still considered low at ~13% (Figure 16). In considering historical data of H570 tested in DEXRON-VI (not shown), it is of note that the substantially lower viscosity, and assuming lower molecular weight, MERCON ULV fluid does not ingress into the polymer compound at a higher rate when compared to DEX-VI ATF; volume change of H570 in both ATF media is very similar at 13-15%.

COMPRESSION SET

In Figure 17, compression set values are shown for all three durometer compound groups aged at 150°C for 168hr in air, Mobil 1 LV ATF, and MERCON ULV ATF. Both HT-ACM and AEM-IP are known in the industry for automotive engine sealing applications and demonstrating good compression set values. This evaluation used ASTM Method B, large buttons with hot release. Compression set values for all compounds were similar, with H570 demonstrating the lowest compression set in majority of the tests. Figure 18 shows the results of the compression set tests of the 60 ShA compounds at 150 °C in air according to ISO 815-1, Method B (small test specimens, cold release) as well as according to VW PV 3307. It can be seen that all materials, HT-ACM as well as AEM-IP, show low compression set values and meet the requirements of the industry in terms of common specification limits and functionality.

LOW TEMPERATURE PERFORMANCE

Low temperature performance properties and flexibility of elastomers for automotive transmission applications are often critical since they are often further removed from a heat source such as the engine or exhaust. It is common for low temperature specifications for TR-10 (temperature retraction test) to be in the -25°C to -35°C range, and occasionally even down to -40°C in more severe specifications. Most specifications call out the cold temperature requirements on the original elastomer compound. In this study, both the compound Tg (glass transition temperature) and TR-10 were mea-

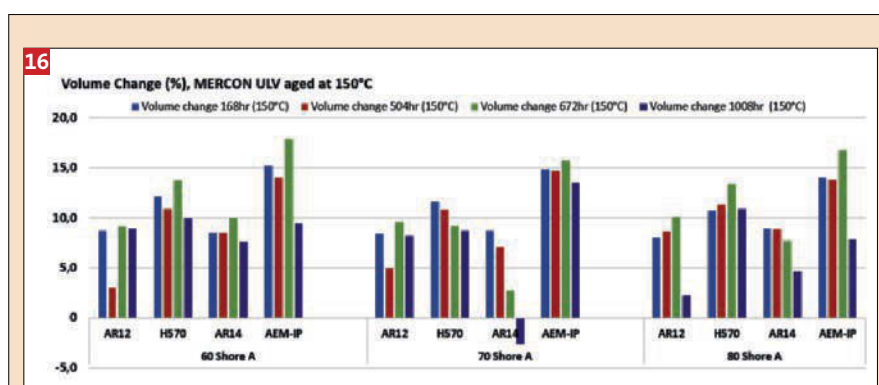


Figure 16. Volume Change, Aged in MERCON ULV at 150°C, Tested at 150°C.

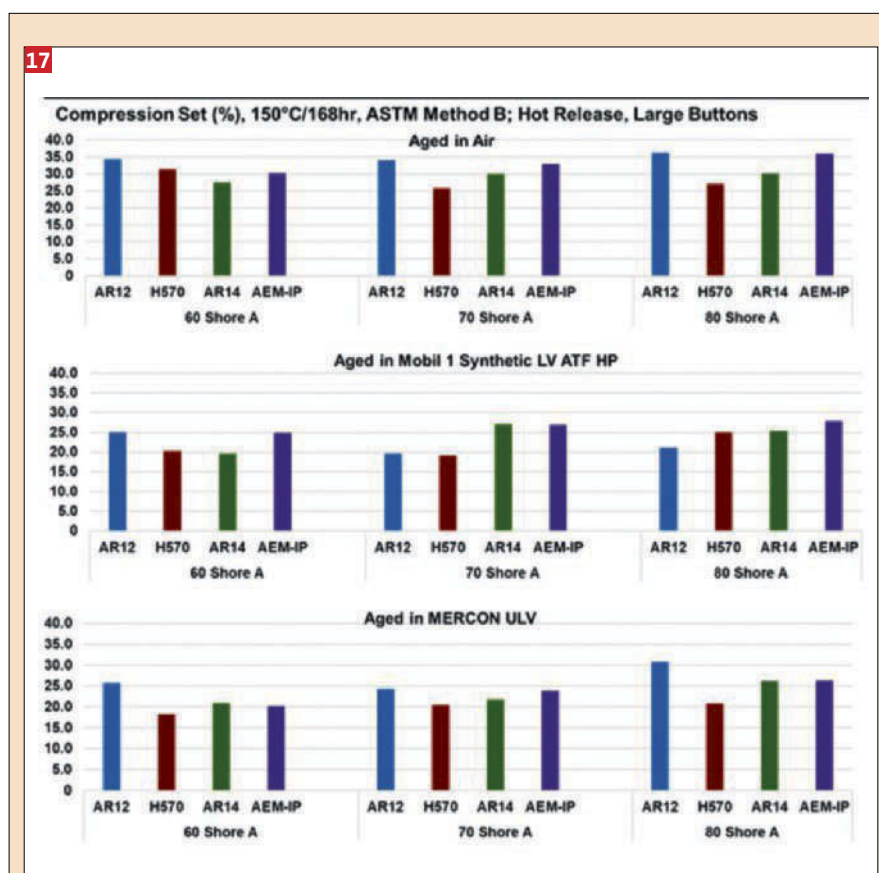


Figure 17. Compression Set, Aged in Air and ATF at 150°C, Tested at 150°C.

sured for each compound. As a follow up evaluation, TR-10 was also measured on samples aged at 150°C for 168hr in air and transmission fluid a to determine how elastomers' cold temperature properties may be impacted by the harsh operating environments within automotive transmissions.

As discussed earlier, the technical target for developing H570 was to improve cold temperature capabilities of HT-ACM while maintaining good physical proper-

ties and long-term, heat-aging performance. Seen in Figure 19, the Tg of H570 is reduced beyond that of AR12 by ~5°C, down to -37°C. In Figure 20, the TR-10 value of H570 is also improved over AR12 by approximately 5°C. Although heat aging in air tends to reduce the TR-10 value of all compounds by a few degrees, the elastomeric components are likely to be in constant or frequent contact with ATF fluid when used in fluid sealing or fluid management applications.

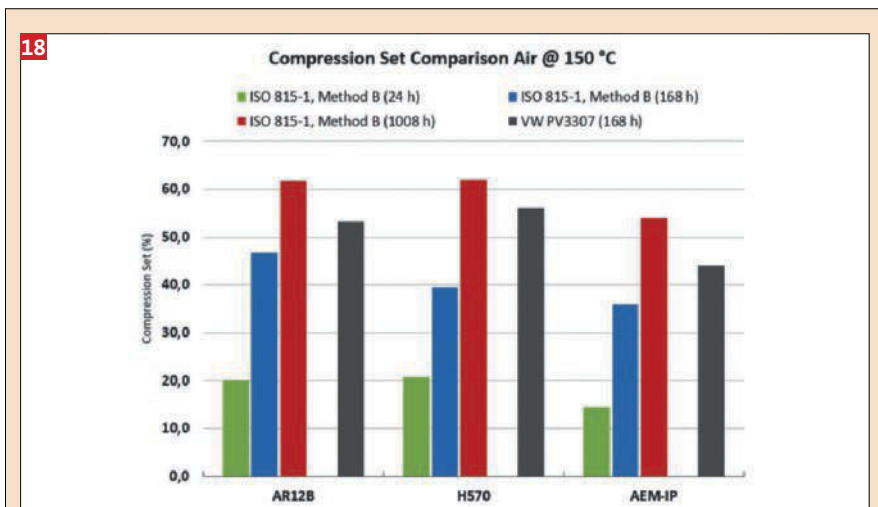


Figure 18: Compression set of HT-ACM and AEM-IP according to ISO 815-1, method B (cold release) and according to VW PV3307 after different measuring times in air at 150°C.

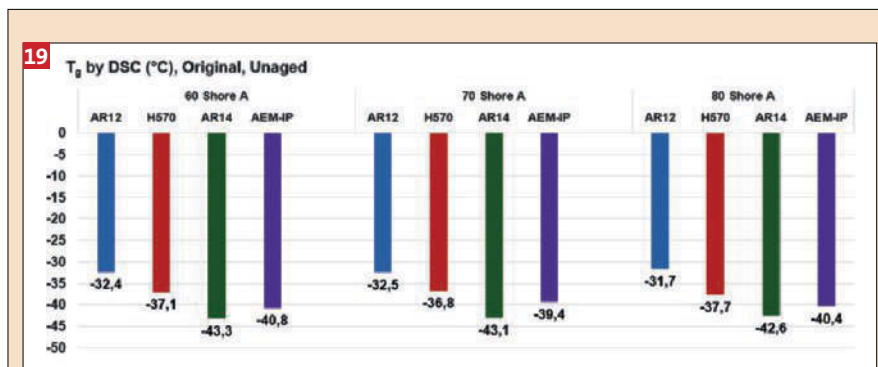


Figure 19. Tg (°C) by DSC.

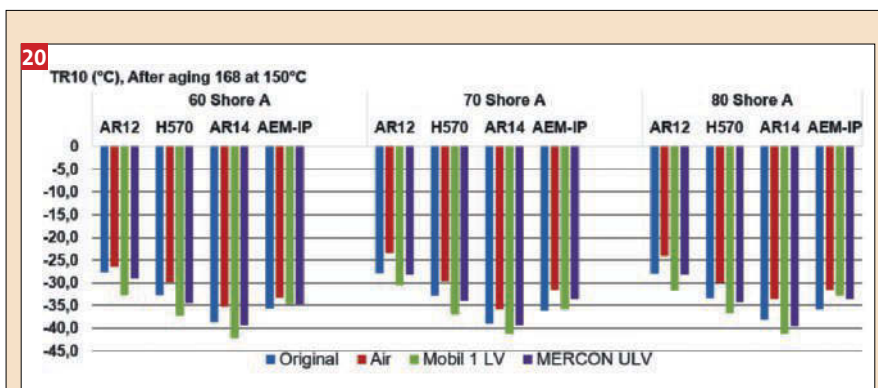


Figure 20. TR10 (°C), Aged in Air and ATF at 150°C, 168hr.

Data shows that, when aged in Mobil 1 LV or MERCON ULV ATF, the elastic recovery of H570 is improved to a TR-10 value of -35°C, similar to that of AEM-IP.

CONCLUSION

This study has presented an in-depth evaluation of HT-ACM, in comparison

with AEM elastomer, where the mechanical strength of HT-ACM was demonstrated under conditions expected within the operating environment of the latest automotive transmission technology. This study has also shown that, when tested at service temperatures, HT-ACM exhibits physical properties similar to

AEM materials. In addition, the newest HyTemp HT-ACM grade, H570, exhibited improved low-temperature service capability while providing excellent performance in high-heat operating conditions and ultra-low viscosity automatic transmission fluids.

Whether for a dynamic shaft seal, oil pan gasket, or transmission oil cooler hose, the HT-ACM grades offered by Zeon can perform well in demanding automotive transmission applications and serve as an effective, alternative material solution in applications considering use of the AEM family of elastomers.

ACKNOWLEDGEMENTS

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REFERENCES

- [1] Ewing, D. (2016). O-Rings and Seals for Automotive Transmission Fluid. Retrieved from <http://blog.parker.com/o-rings-and-seals-for-automotive-transmission-fluid>.
- [2] Paglia, P. (2010). New Vamac Ultra High Viscosity AEM Polymers With Extended Application Possibilities [PowerPoint presentation]. Retrieved from: <https://docplayer.net/85001176-New-vamac-ultra-high-viscosity-aem-polymers-with-extended-application-possibilities.html>
- [3] DuPont (2010). DuPont Vamac Ultra-IP, Technical Information – Rev. 2, July 2010. Retrieved from <https://www.dupont.com/content/dam/dupont/amer/us/en/transportation-industrial/public/documents/en/Vamac-Ultra-IP.pdf>

Recovered carbon black from waste tire pyrolysis · technology · reinforcement

The use of recovered carbon black from waste tire pyrolysis is not progressing. The potential reasons for this stagnation are discussed considering the latest investigations in this domain. What can be expected from the present achievements of the pyrolysis technology? The availability of active sites on the carbon black surface might be one of the major causes of the lack of performance in reinforcement of the recovered material. The indications in the carbon black literature are listed and discussed in view of potential new ways to improve the performance of the recovered material and close to some extent the carbon black cycle in the rubber industry.

Two technologies are pointed out. The first one is the use of low temperature plasma. The second one, looking more promising is the use of thermal plasma generating fullerenes and fullerene-like species on the surface.

Fortschritte in der Entwicklung von Rußen aus Altreifen - Stand und neue Wege zur Verbesserung der Leistung in Elastomeren

Ruß aus Altreifen · Pyrolyse von Altreifen · Verstärkung in Elastomeren · Hochtemperaturplasma · Niedertemperaturplasma

Der Gebrauch von Rußen aus Altreifenpyrolyse stagniert. Mögliche Gründe hierfür werden auf Basis der letzten Untersuchungen auf diesem Gebiet diskutiert.

Was kann man von dem heutigen Stand der Technik erwarten? Der Mangel an aktiven Funktionen an der Oberfläche dieser Ruße kann einer der Gründe sein. Die Rußliteratur listet eine Reihe von potentiellen Wegen die Oberflächengruppen der Ruße zu verbessern welche möglicherweise dazu helfen könnten, den Rußzyklus in der Gummiindustrie zu schließen. Zwei Verfahren werden diskutiert. Das erste Verfahren ist Niedertemperaturplasma, das zweite Verfahren ist Hochtemperaturplasma, welches Fullerene oder fullerenartige Strukturen an der Rußoberfläche erzeugt.

Figures and Tables: By a kind a approval of the authors.

Progress on recovered Carbon Black from Waste Tire Pyrolysis

Present status and potential improvements of their performance in rubber

Tire recycling is considered today as a must and waste tire pyrolysis is certainly for the time being one of the main technologies considered to achieve this goal. Many different research teams are working on this technology and in the meantime several plants have been constructed around the world, covering however only a very small fraction of the world waste tire recycling. The reason for this stagnation is the fact that recovered carbon black is not a valid candidate for carbon black replacement in order to close the product cycle. Recovered carbon black is practically not suitable for high quality use and only in a limited number of applications. It is also quite interesting to notice that no established carbon black producer has apparently shown any interest or need in investing in the, in principle, competitive product candidate and an ecological way to validate carbon black production.

We realize that since 2011, when we published the paper "Pyrolysis Carbon Black, an opportunity for the rubber industry [1] no significant progress in the performance of the recovered carbon has been made. Commercially some interesting developments have been reported; nevertheless a breakthrough and real closure of the material cycle cannot be expected till the recovered carbon black can assure a performing use in most carbon black applications. Work has been done in two domains:

- Changing the operating conditions of pyrolysis;
- Treating the recovered carbon black in order to reduce the impurity level (Martinez et al.) [2]

Those techniques, although bringing some small improvements, did not change the situation significantly. Recent publications and company presentations indicate some new attempts to change the surface of recovered carbon black. Cardona and al. [3] have made a summary and compiled many recovered carbon blacks from different sources. The table lists more than 20 carbon blacks, reporting their elemental composition and

major morphological characteristics. The variations we observe represent most probably mainly the difference in the waste tyre compositions used as raw material and also to some extent the difference in pyrolysis equipment and operating conditions. Pieter ter Haar [4] in a presentation of investigations executed at Pyrolox, reports the differences in analytical data and compounded performance of rCB from ASTM 3191 SBR compounds. It is quite interesting to notice the difference between lower and medium reinforcing carbon blacks like N-660 and N-330 on one hand and high reinforcing N-110. In the first case the drop in performance between virgin and recovered carbon black is visible in the stress strain value which could be due to a lower structure of the recovered material, while for N-110 the drop in performance between virgin and recovered carbon black is mainly visible in tensile strength, which is partially due to the higher modulus, but could be mainly due to poorer dispersion.

The results obtained by ter Haar on the pyrolysis of the ASTM3191-compounds containing the 3 carbon blacks may be considered as the base of the recovered carbon black problem.

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1 Green rubber /CB Pyrolysis		
	STSA	Weight gain %
N115	112	20,1
N330	76	15,3
N660	33	10,5
N990	10	7

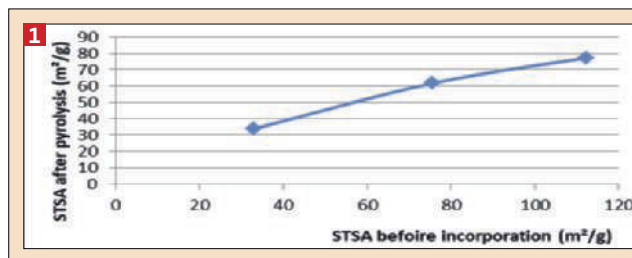


Figure 1. Specific surface area (STSA) before and after pyrolysis [4].

Table 1 Weight gain during pyrolysis relative to the carbon black content of the green rubber with different carbon blacks [4]

Compound pyrolysis generates carbon residues resulting in a deposit of relatively amorphous carbonaceous material and consequently in a weight gain of the recovered carbon over the original compound composition. This weight gain is a function of the aromaticity of the polymer. ASTM 3191 compound is based on SBR 1500 and as such quite aromatic. In the case of a waste tyre pyrolysis the rCB may also contain the pyrolysis residue of other organic ingredients like textile cords. Ter Haar shows that the weight gain is also a function of the specific surface area of the carbon black. The amount of surface deposit is proportional to the available surface area in a given volume of compound. This explains the reduction of the specific surface area due to the increase of particle size and the higher impact of this phenomenon for high surface area carbon blacks. (Figure 1)

Table 2 reports the rubber compounded data for the 3 furnace blacks and the corresponding recovered carbon blacks.

Table 2 Compounded properties in ASTM 3191 SBR formulation for N660, N330 and N110 for virgin carbon black and rCB [4]

It may be concluded that the main drop in performance observed on the rCB in comparison with the virgin carbon black occurs with fine particle furnace blacks? This would explain that a significantly higher performance cannot be

expected from rCB without further treatments.

Ter Haar investigates also the difference between rCB with low and high toluene extractables. The observed difference, although significant in tensile strength, is most probably related to the increase in modulus. The increase in modulus when the oily residues on rCB surface are reduced, might indicate that interstitial volumes are freed to some extent and consequently the void volume (structure) is increased. Void volume measurements may give more information as the Oil Adsorption Number (OAN), which is strongly affected by the wetting properties of the rCB and cannot be considered as a relevant property.

Summarizing the different aspects considered above and keeping in mind that only limited aspects of the reinforcement are reported in the literature, it may be concluded that:

- A. In the case of compounds without mineral fillers:
 1. The reinforcing properties of rCB in comparison with virgin furnace black are not very much reduced for coarse particle blacks and slightly affected in the case of semi-reinforcing carbon black;
 2. For higher reinforcing carbon black the reduction of the reinforcing performance is more pronounced.
- B. In the case of waste tyre pyrolysis:
 1. Effects mentioned in A. are added to the effect of mineral filler deposits;
 2. This means that the concentration of

rCB in a given compound containing +/- 20% mineral filler is actually only for 80% carbon black. The specific surface area of the carbon black, when recalculated from the average carbon black type and content in the original tire, can be situated between 60 and 70 m²/g.

We cannot expect a lot of improvement by playing with the pyrolysis operating conditions.

The elemental composition (ash) of the recovered carbon black can only be considered to a limited extent as an indication of its performance in rubber; the research should be focused primarily on the potential interaction of the recovered carbon black with the polymer.

Supposed that the performance of the recovered black is subject to the same parameters as for furnace blacks.

The reinforcement of carbon black in elastomers is determined by the interaction of the carbon black with the elastomer chains. Carbon black is mainly characterized by two morphological properties: Specific surface area (determined by the particle size) and structure.

Specific surface area:

For recovered carbon black the particle size is, or should be, if pyrolysis residues from the polymer and organic additives and especially mineral residues do not influence the particle size, the weighted average of the different carbon blacks present in the waste tyre used as raw material. We can however expect that the particle size has increased and conse-

1 Physical Rubber properties							
	N660	N660rCB	N330	N330 rCB	N115	N115 rCB	Reference rCB
UTS (Mpa)	20.7	20.3	23.2	20.4	27.4	20.2	18.9
E@B (%)	497	557	424	455	520	457	537
M100 (Mpa)	2.07	1.90	2.43	2.37	2.15	2.84	1.95
M300 (Mpa)	10.4	7.6	13.6	10.8	10.8	11.7	7.8
Shore A	62.0	61.5	66.0	65.0	65.5	67.5	62.0
Dynamic Properties RPA (60°C Amplitude sweep 1Hz 1-100% Strain)							
Delta g' (Kpa)	1008	1159	1586	1514	2154	1800	1205

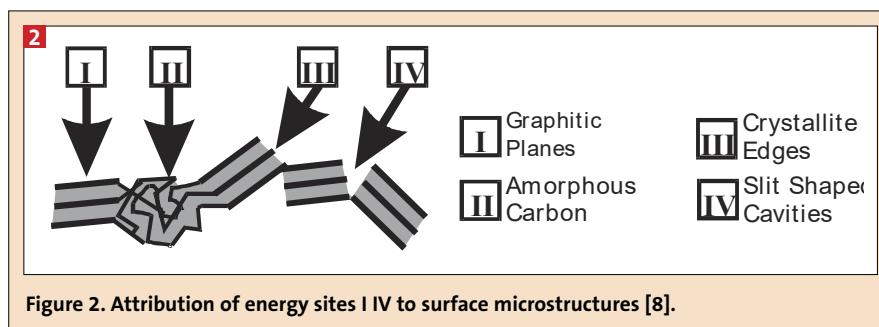
quently the specific surface area is reduced due to the pyrolysis residue which is covering the original particles. This carbonaceous residues resulting from the pyrolysis of organic parts of the tyre (other than carbon black) are a function of the aromaticity of the polymer and of the absolute surface of the carbon black in a given volume. The absolute surface area in a given volume of the compound is the surface area in the given volume in contact with the polymer. In addition to the carbonaceous deposit, mineral components present in the tyre compounds will deposit. The work of Pieter ter Haar [4] studying the residue for several carbon blacks after pyrolysis gives an estimation of the pyrolysis residue. Ter Haar confirmed the influence of absolute surface area. In the recipe, ASTM 3191, used by ter Haar, mineral residue is 4.75% and the carbonaceous residue represents +/-2% for the N-990; +/-7% for N-660; 10% for N-330 and going up to +/-15% for N-110. Carbon black surface acts as a catalyst on the deposition of carbon from a hydrocarbon environment. Carbon black is used industrially to increase the yield of hydrocarbon carbonisation. Consequently we can assume that the deposit of carbonaceous residue from the pyrolysis is mainly occurring at the carbon black particle surface. We can consider that the average particle diameter increases and consequently the specific surface area is reduced. This is confirmed in ter Haar's observations.

From ter Haar's investigations we can conclude that smaller particles have a noticeably more important growth of the specific surface area than larger particles. This means that we can expect a trunking of the particle size distribution and finally the tendency will be during waste tyre pyrolysis towards a recovered carbon black having the average particle size and specific surface area of a N-660.

This is in fact generally observed. This also indicates that the surface quality of a recovered carbon black is quite similar to the one of virgin furnace black.

Pyrolysis residue, as well as mineral residues, are also filling the interstices between the particles and the voids within the aggregates. The impact of silica residues is another subject to be investigated.

In order to have a better understanding, more fundamental investigations should be carried out on the surface of the carbon black. Techniques like TOFF-SIMS and Tunnelling Microscopy might be tech-



niques helpful to get a better view of the surface composition and of the surface arrangements of the carbon deposit.

Structure or void volume:

Structure, actually the volume of the voids within a carbon black and the interstitial volume, filled to a large extent in the tyre compound by the elastomer, has to be expected to be covered to a large extent during the pyrolysis process. Eventually new voids are created.

Experimental results by OAN indicate that structure remains more or less at the same level as could be expected from the carbon black mix. These results have however to be considered carefully, as the OAN test is quite dependent on the wetting process of the oil. More significant information would be obtained by void volume measurements.

Quite often specific surface area and structure are considered for virgin carbon black as the only determining parameters for the prediction of their performance. This interpretation is in the case of normal carbon black quite justified. Surface chemistry of a normal carbon black is well known as quality parameter; it is however quite constant within a given carbon black type with a defined surface area.

Ter Haar has studied the effect of oily hydrocarbon residues on the reinforcing performance of de recovered carbon black. In virgin carbon black oily residues are usually more considered as an environmental and health problem than related to performance. The difference in composition and the amount of oily residues on recovered carbon black and virgin carbon black would have to be investigated. In ter Haar's comparison the effect of the oil seems to be mainly related to a difference in structure, inducing a lower modulus for the oily material and consequently a higher tensile and ultimate elongation

Wang and Wolff [5] describe the tech-

nique of inverse gas chromatography to determine the surface energy of carbon black and discuss its significance for rubber reinforcement. Mainly two parameters are determined by this technique:

- γ_s^d the dispersive component of the surface energy describing the interaction between carbon surface and polymer;
- Γ^{SP} the specific component of surface energy.

Wang (6) and Wolff et al. [7] report the evolution of the dispersive component, determined by inverse chromatography, as a function of the specific surface area. The dispersive component is increasing linearly with the specific surface area. Similarly they report the bound rubber of an SBR compound of being in linear relationship with the dispersive component. High γ_s^d of the carbon black is imparting higher bound rubber.

These observations show that the fact that surface chemistry is usually not considered as a specific property of carbon black is justified due to its very constant relationship to the specific surface area. The dispersive component is a function of the crystallite dimension, which is a function of the curvature radius of the carbon particle: The smaller the particle, the smaller the crystallites on the surface and the higher is the concentration of active sites. In the case the normal furnace carbon black the surface generated in the reactor is rearranged to a quite graphite-like structure. Wang and al. showed that the dispersive component is a function of the average graphitic layer distance (L_c). This relationship is not existing when specific treatments are applied to the carbon black as for example graphitisation or surface treatment increasing the number and nature of the energetic sites on the surface.

Schröder [8] has studied (Figure 2) the various surface energy configurations which can be observed on carbon black surface.

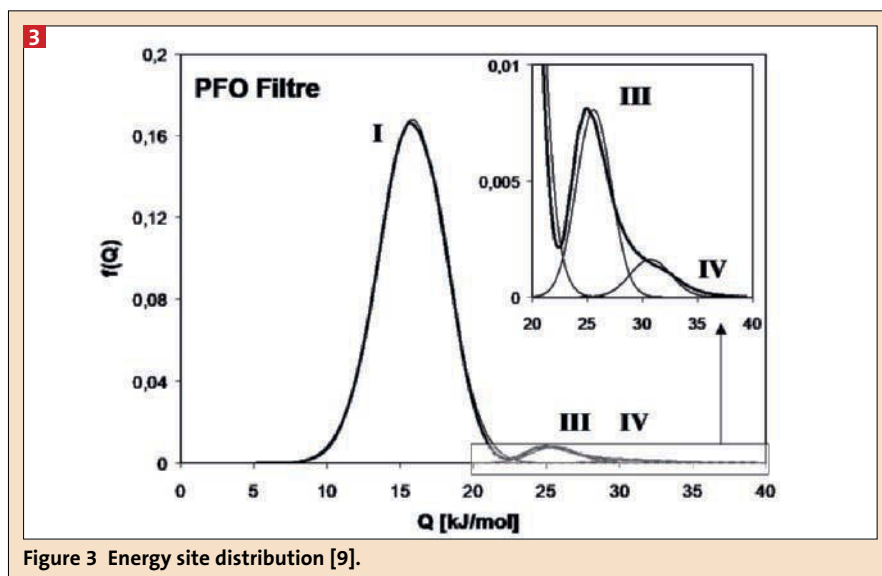


Figure 3 Energy site distribution [9].

Figure 3 [9] shows the distribution curve for energetic sites for a Thermal Plasma carbon black, which has also a similar shape as furnace black. Differences between plasma and furnace black grades are found in the high energy part with $Q \geq 20$ kJ/mol of the distributions. The considered plasma carbon black had been obtained at noticeably higher temperatures than furnace blacks and had consequently a larger graphitic surface.

For virgin carbon blacks the presence of energetic sites is proportional to the surface defects and graphite plan edges, which due to the curvature radius are proportional to the size of the graphitic plans. Donnet and Custodero [9] (9) have shown by Tunnelling Microscopy that the carbon black surface has a kind of graphitic scale arrangement. They also suggested that some fullerenic structures are present on the surface of the carbon black and could be the main explanation of the reinforcing properties of carbon black.

We can expect and it is quite obvious that the active sites from the original carbon black have been neutralized during the mixing of the tyre compound and have provided the reinforcement to the original tyre. The pyrolysis process may have evacuated some molecules which were adhering to the carbon black by physical and even chemical processes, the pyrolysis temperature is not high enough to graphitize the carbon black surface. The original carbon black surface is however not very reactive anymore, although some functional groups may remain on the surface. The surface activity of the carbonaceous deposit is

certainly not fully comparable to the one of the virgin carbon black.

It is as such very important to regenerate new functional groups able to undergo new interactions with the elastomer molecules or to remove inactive material. No impact from the particle size (specific surface area) can be expected as we are in the range of the average size, the mathematical average size of the blend of all carbon black types in the tyre.

Generation of active sites on the surface of the recovered carbon black

Several techniques to recreate a more reactive carbon black surface can be considered. In the present paper we consider two:

1. Low temperature plasma (Cold plasma)
2. Thermal plasma

1. Low temperature plasma

Atmospheric plasma or cold plasma has been investigated in the context of modification of the carbon black surface. The cold plasma could be generated by luminous discharges at reduced pressure ($1-10^3$ Pa), where free electrons, accelerated in an electric field, transfer their energy by collision to neutral gas molecules. This discharge can be produced at low frequency (60 Hz) between two electrodes or at high frequency without electrodes using different types of generators: by spark initiation (10-50 kHz), radio frequency (1.5-50 MHz) or microwaves (150-10000 MHz).

Two types of interactions exist:

- By energy transfer resulting in scission or excitement of chemical bonds;
- By transfer of chemical species.

In 1992 Weidong Wang [10] has been using a cold, high frequency plasma with a microwave generator. Two actions of the plasma can exist:

- a. Erosion of the carbon black surface due to the bombardment by ions, activated species, molecules...;
- b. Modification of the surface chemistry by active species (free radicals, ions...) or by reaction of the defects created by the reagents in the atmosphere.

Wang has been using different molecules: H_2 , Air, NH_3 and Ar. He has also combined the plasma treatment with a heat treatment at $900^\circ C$. He reported a noticeable increase of the dispersive component of the surface energy γ_s^d . This increase was mainly visible after the plasma treated carbon blacks were exposed to 4 hours heat treatment at $900^\circ C$.

The specific interaction parameter I_{sp} was also increased but to a lower extent.

As bound rubber is also strongly affected by the structure of the carbon black and considering that Weidong Wang observed the same effect on the same carbon black in a treated and untreated form, we can expect that the plasma treatments are an efficient tool to increase bound rubber due to better interaction between filler and polymer.

Recent work done by Millington, Bennett and Bradley [11] is using low temperature plasma to activate the surface of rCB. Ozone, oxygen and peroxide have been used as activated molecules, and important changes in the rubber properties have been observed.

Thermal plasma

The most significant improvement in the development of the recovered carbon black can be expected from thermal plasma.

Donnet et al [12] and Johnson et al [13] have shown that fullerenes are present in the toluene extract of furnace blacks. According to Cataldo [14, 15] the surface activity of carbon is largely determined by the presence of fullerenic structures on the carbon black surface. Fullerene-like structures would act as free radical acceptor sites where the rubber macro-radicals formed during mixing form chemical bonds between rubber and filler. These fullerenic structures would play a major role in bound rubber formation.

Many publications have reported the possibility to produce fullerenes using thermal plasma [16, 17]. The starting material is carbon black or other carbon material.

Mathew and al. [18] have also been able to show how fullerene structures can be used in compounds to significantly enhance the reactivity of a carbon black. Fullerenes are very reactive species and improve the interaction between carbon black and polymer. Probst et al [19] reported the effect of thermal plasma treatment of a N-330 carbon black. Mathew used an N-330 and Ensaco 250, a low reinforcing carbon black for his experiments and plasma-polymerized acetylene on its surface.

The fullerene soot was prepared by injecting the virgin carbon black into thermal plasma in an inert He/Ar atmosphere. Carbon from the surface was vaporized and recondensed as fullerene and fullerene structures.

After 1h plasma treatment with acetylene the virgin N-330 carbon black gave 1.05 weight percent coating, while the N-330 based fullerene soot gave 14% deposit. Similarly, the virgin Ensaco 250 gave after 1h plasma treatment with acetylene 2.4% deposit while the Ensaco based fullerene soot gave 17% deposit.

Schröder et al. [8, 9] show that only the sites II, III and IV are involved in the interaction with the polymer. Sites I energetic functions are graphitic and inert towards the polymer. For N-330 carbon black, only 10 % of the surface is involved in the reinforcement. Fullerene structures are very reactive; according to Sola et al. [20] these structures deviate from planarity and have very high electron affinity.

In March 2012 Probst and Löffler have run, with the help of the team of Fulchéri at the Centre Persee of Mines – Paris Tech (PSL Research University), in Sophia Antipolis a trial in a thermal plasma reactor with recovered carbon

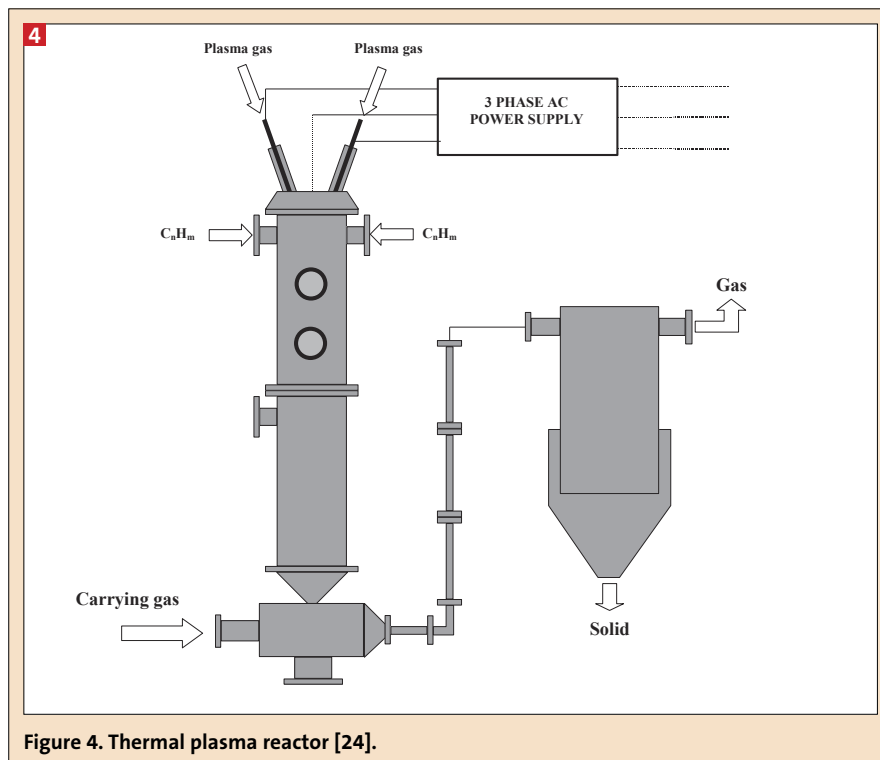


Figure 4. Thermal plasma reactor [24].

black from scrap tires. The trials were run successfully as the extract from the treated sample showed an intense red color, typical for the presence of a significant percentage of fullerenes. The plasma reactor was a 3-Phase AC reactor, the same reactor used in the production of fullerene material. The plasma power supply, as mentioned by Fabry F. et al [22] was 600 Hz, 0-400A, 263 kW maximum power. Each of the three phases was connected to the graphite electrodes of the 3-phase plasma torch located in the upper part of the reactor [23].

Solid recovered carbon black was injected in the continuous gas-phase synthesis chamber into the thermal arc plasma. The high temperature gas/solid aerosol was introduced through a quench system after having reached sufficiently low temperature in a filter system allowing the separation of the solid particles and the gas. The operating conditions for the

trials as reported by Fabry [24] were:

The recovered carbon black (Table 4) was partially vaporized when moving through the high temperature zone (+/- 4000 K). The vaporization rate is a function of the precursor particle size and the residence time in the high temperature zone. The vaporization can be described as an arc discharge, starting with the generation of small carbon species and finally followed by a quench and the condensation of the small species into fullerenes or/and as fullerene structures onto the remaining carbon black surface.

Conclusion

Waste tyre pyrolysis may not be able to close the product life cycle of carbon black as recovered carbon black by pyrolysis of scrap rubber/tyres cannot be expected to achieve reinforcing performances as those known for furnace black

3 Operating conditions on the thermal		
Property	Unit	Value
Average Power	kW	36
Average Current	A	180
Average Voltage	V	150
Plasma gas flowrate (N2)	Nm³/h	4
Carrier gas flowrate (N2)		4
Carbon black soot flowrate	Kg/h	0.8
Recirculation loop gas flowrate (N2)	Nm³/h	200

4 Analytical data of the recovered carbon black CT- R 6632 [1]		
BET S.A.	m²/g	69
CTAB S.A.	m²/g	62
OAN	ml/100g	106
COAN	ml/100g	84
Ash Content	%	14
Carbon Content	%	82
Oxygen Content	%	9
Sulfur Content	%	2.5

unless a way is found to increase its interaction with the polymer.

Surface treatments, which could regenerate active surface sites, might be an economical solution to achieve this goal to some extent. Two techniques are considered as promising:

1. Cold plasma
2. Thermal plasma

With both techniques the surface activity can be increased. Cold plasma is reported in literature as being able to increase the dispersive component of the surface energy of the recovered carbon black and consequently the bound rubber formation. Thermal plasma generates fullerenes and fullerene sites on the carbon surface and has been reported as significantly increasing the reactive performance of the treated carbon black.

Acknowledgement

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References

- [1] Probst N.; Loeffler M.; 180th Meeting of the Rubber Division of the ACS, Cleveland October 11-13, 2011.
- [2] Martínez JD1, Cardona-Urbe N2, Murillo R3, García T3, López JM3 ; Carbon black recovery from waste tyre pyrolysis by demineralization: Production and application in rubber compounding; *Waste Manag.* (2019 Feb 15;85:574-584. doi: 10.1016/j.wasman.2019.01.0.
- [3] Cardona N. , F Campuzano1 , M Betancur1 , L Jaramillo2 and J D Martínez1; Possibilities of carbon black recovery from waste tyre pyrolysis to be used as additive in rubber goods -a review; *IOP Conf. Series: Materials Science and Engineering* **437** (2018) 012.

- [4] Ter Haar P ; The effect of Carbonaceous residue on virgin Carbon black/eSBR mixtures in rCB; SMG London 2019.
- [5] Wang M.J. and Wolff S. ; Surface energy of carbon black ; ; Chapter 6; Carbon Black , second edition; Science and technology.
- [6] Wang, M.-J., Wolff, S., Donnet, J.-B., *Rubber Chem. Technol.*, **64**, 714 (1991).
- [7] Wolff, S., Wang, M.-J., Tan, E.-H. presented at the meeting of the rubber division, ACS, Detroit, Oct.8-11 1991.
- [8] Schroeder A., Ph.D. Thesis University of Hannover (2000).
- [9] Probst N., Grivei E., Fabry F., Fulchéri L., Flamant G., Bourrat X., Schroeder A., Quality and performance of carbon black from plasma process, Ohio, ACS Rubber Division Cleveland et 2001, Oct. 16-19.
- [10] Donnet J.B. ; Custodero E.; Carbon black surface studied by scanning tunneling microscopy; Chapter 5; Carbon Black , second edition; Science and technology.
- [11] Wang W. Thesis Mulhouse 1992.
- [12] Millington S., Bennett M., Bradley B. Surface Treatment of Recovered Carbon Black (SURE-CARB) Innovate UK Project 133639, Matsurf 27th February 2019.
- [13] Donnet, J.B. ; Wang, T.K. ; Wang, C.C. ; Monthieux, M. ; Johnson, M.P. ; et. al. Carbon Black and Fullerenes Part II : Precursor and Structure Identification. In: *KGK Kautschuk Gummi Kunststoffe*, Vol. **52**, 340 (1999).
- [14] Johnson, MP ; Locke, RW ; Donnet, JB ; Wang, TK, Wang, CC ; et. al. Carbon black and fullerenes: New discoveries in early formation mechanisms and nucleation. In: *Rubber Chemistry and Technology*, Vol. **73**, no. 5, 875 (2000).
- [15] Cataldo F. The impact of a fullerene-like concept in carbon black science; Volume **40**, Issue 2, February 2002, 157.
- [16] Cataldo F.; The role of fullerene-like structures in carbon black and their interaction with dienic rubber; Volume **8**, 2000 - Issue 1-2 Pages

105-112 | Received 23 Aug 1999, Published online: 23 Apr 2008.

- [17] L. Fulcheria, Y. Schwoba, F.F. abryb G. Flamantb L.F.P. Chibantec D.Laplazed, Fullerene production production in 3-phase AC plasma process; *Carbon* ;Volume **38**, Issue 6, 2000, 797.
- [18] VFrédéric Fabry, Jose Gonzales-Aguilar, Laurent Fulcheri, Nicolas Probst. Technologie plasma triphasée pour la synthèse de fullerenes en continu. 9ème colloque sur les arcs électriques - CAE IX, Mar 2009, Sophia Antipolis, France. 2 p. fffhal-00805356f.
- [19] T. Mathew, Rabin Datta, Wilma K. Dierkes, W.J. Ooij, Jacobus W.M. Noordermeer, N. Probst, T.M. Gruenberger, N. Probst ; Importance of fullerene active sites in surface modification of carbon black by plasma polymerization; *Carbon* ; Volume **47**, Issue 5, Ap.
- [20] Probst N. ; Fabry F. ; Gruenberger T. ; Grivei E. ; Modification of carbon black and graphite surface, Proceedings Deutsche Kautschuk Tagung, Nuernberg Germany.
- [21] Sola M. ; Mestres J., Duran N. ; Molecular size and Pyramidalisation: two keys for understanding the reactivity of fullerenes; *J. Phys. Chem.* 1995, **99**, 10752.
- [22] Frederic Fabry, Laurent Fulcheri. Synthesis of carbon blacks and fullerenes from carbonaceous wastes by 3-phase AC thermal plasma. 6th International Conference on Engineering for Waste and Biomass valorisation - WasteEng 2016, May 2016, Albi, France.
- [23] N. Probst1, E. Grivei1, F. Fabry1, L. Fulchéri2, G. Flamant3, X. Bourrat 4, A. Schröder5, Quality and Performance of Carbon Blacks from Plasma Process et *Rubber Chem Techn.* Volume 75, Issue 5 (November 2002).
- [24] Frederic Fabry, Laurent Fulcheri. Synthesis of carbon blacks and fullerenes from carbonaceous wastes by 3-phase AC thermal plasma. 6th International Conference on Engineering for Waste and Biomass valorisation - WasteEng 2016, May 2016, Albi, France.

Neuer Hitzestabilisator schließt Preis-Leistungs-Lücke zwischen Blends und Stabilisatoren auf Kupferbasis

PHENOLIC PLUS BRÜGGOLEN TP-H1803 Phenolic Plus Brüggolen TP-H1803 von Brüggemann, Heilbronn, ist ein neuer Hitzestabilisator für Polyamide, der die Leistung herkömmlicher Mischungen von sterisch gehinderten phenolischen Antioxidantien mit organischen Phosphiten deutlich übertrifft. Der Hitzestabilisator kann so-

mit die Preis-Leistungs-Lücke zwischen diesen Blends und Stabilisatoren auf Kupferbasis schließen. Im Vergleich zu Standardtypen aus sterisch gehinderten Phenolen und Phosphiten wird die Stabilisierung von Polyamiden auf Belastungen mit kurzzeitigen Temperaturspitzen bis 180 °C ergänzt. Darüber hinaus sind aufgrund der

höheren Effizienz niedrigere Dosierungen bei der Compounding möglich als bei herkömmlichen Stabilisatoren, um den gleichen Effekt zu erzielen. So konnte in Versuchen die Dosierung des Hitzestabilisators im Vergleich zu einem Standard-Stabilisatorpaket um 30 Prozent reduziert werden – bei gleicher Halbwertszeit der Zug-

festigkeit nach Hitzealterung bei 150 °C. Der Stabilisator wird in Form von staubfreien Pellets geliefert, die sowohl zum Compounding als auch zum Spritzgießen geeignet sind. ■

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• Silicone Rubber • Mechanical and
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In present work, the carbon hybrid nanofillers were used in room-temperature-vulcanized (RTV) silicone rubber as a rubber matrix. With an addition of 1 phr CNT in a CB composite, the compressive modulus increases from 5.17 MPa (3 phr CB-CNT hybrid) to 9.6 MPa (20 phr CB-CNT hybrid). Similarly, the tensile strength increases from 0.87 MPa (3 phr CB-CNT hybrid) to 2.29 MPa (20 phr CB-CNT hybrid). For flexible electronics applications, the resistance falls from 550 k Ω (3 phr CB-CNT hybrid) to 0.5 k Ω (20 phr CB-CNT hybrid). For the piezo-electric actuator, with increases in voltage from 2 kV to 8 kV for 3 phr CB+CNT and CB, the actuation displacement at 8 kV increases by 1.45 mm (CB+CNT) and 0.54 mm (CB).

**Gummiverbundwerkstoffe auf
Basis von CNT-CB-Hybrid und
Rußsystemen für industrielle
Anwendungen**

**Nano-Ruß • Kohlenstoff-Nanoröhrchen
• Silikon-Gummi • Mechanische und
elektrische Eigenschaften • Piezoelektrischer Aktuator**

In der vorliegenden Arbeit wurden die Kohlenstoff-Hybrid-Nanofüllstoffe in bei Raumtemperatur vulkanisiertem (RTV) Silikonkautschuk als Gummimatrix verwendet. Mit einer Zugabe von 1 phr CNT in einem CB-Verbundstoff erhöht sich der Druckmodul von 5,17 MPa (3 phr CB-CNT-Hybrid) auf 9,6 MPa (20 phr CB-CNT-Hybrid). In ähnlicher Weise steigt die Zugfestigkeit von 0,87 MPa (3 phr CB-CNT-Hybrid) auf 2,29 MPa (20 phr CB-CNT-Hybrid). Für flexible Elektronikanwendungen fällt der Widerstand von 550 k Ω (3 phr CB-CNT-Hybrid) auf 0,5 k Ω (20 phr CB-CNT-Hybrid). Für den piezoelektrischen Aktuator erhöht sich mit einer Spannungserhöhung von 2 kV auf 8 kV für 3 phr CB + CNT und CB die Betätigungsverschiebung bei 8 kV um 1,45 mm (CB + CNT) und 0,54 mm (CB).

Figures and Tables:
By a kind approval of the authors.

Rubber Composites based on CNT-CB hybrid and CB for industrial Applications

Introduction

The next generation soft electronic flexible devices need high stretchability, high flexibility, high deformity, and self-healing capability for actuators. Polymer nanocomposites based on new generation nanofillers such as CNT [1] and CNT hybrids [2] are promising candidates for this requirement. These nanofillers are the best alternative to traditional fillers such as CB, which requires a large amount of loading to reinforce a polymer matrix to obtain optimum properties [3]. For example, CNT at 2 phr leads to improved mechanical properties by 272%, which CB does not provide even at 20 phr loading in a silicone rubber matrix [4]. Thus, CNT are a valuable nanofiller that has promising reinforcing ability at low content in a rubber matrix.

Comprehensive studies show that nanofillers in single or hybrid forms are used in order to attain high properties [5,6]. Studies on graphene [7,8], CNT [7,9,10], and carbon black [5-7,10] show improved properties with the use of these nanofillers. The nanofillers have the ability to provide sufficient reinforcement and attain filler percolation threshold at low filler content in the rubber matrix [11]. The filler percolation threshold describes the formation of long-range continuous filler networks, and after its attainment, an exponential improvement was noticed [4, 5,12]. However, at high content of these nanofillers, there is a fall in the properties due to the formation of aggregates, especially for carbon nanofillers [13].

The formation of microstructure by filler particles with a polymer matrix plays an important role in determining the properties of nanocomposites. Fillers with hybrid forms are well known to exhibit a synergistic effect of higher properties than their use in single form at the same loading [5,14]. Investigations show that use of fillers in hybrid form is an innovative way to obtain higher properties without the use of high content [15]. Studies show that there is enhanced thermal conductivity in the composites with hybrid fillers [16]. Literature also

show that there are enhanced thermal and electrical properties for CNT-GnP hybrid filler in epoxy composites [17]. Research also shows that the use of silica/CB hybrid filler in natural rubber leads to improved properties in composites [18].

The properties also depend on the filler dispersion [19], polymer-filler interfacial interaction [20], filler networking in the polymer matrix [20, 21], polymer-filler compatibility [22], surface activity of the filler [23], filler morphology such as shape, particle size, and aspect ratio [23, 24], and type of rubber matrix [25]. Poor filler dispersion due to aggregation of filler particles during high filler loading leads to poor properties. Similarly, poor filler-polymer interaction leads to poor dispersion, and large differences in surface activity of filler-polymer lead to lo-

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wer properties. A filler particle with high aspect ratio helps in attaining the filler percolation threshold at lower filler loading. The aspect ratio plays an important role in determining the properties of the composites [26]. The polymer matrix can be a thermoplast, thermoset, or elastomer. The elastomer polymer matrix can be diene rubber, silicone rubber, or natural rubber. Among them, silicone rubber is the most promising due to easy processing, good properties, low viscosity, and high tensile strength.

There are various studies on the use of diene rubber or natural rubber in the preparation of polymer composites. However, there are less studies on the use of RTV silicone rubber as a polymer matrix reinforced with a hybrid filler system based on CNT and CB. Moreover, the use of hybrid filler as a cost-effective route in RTV silicone rubber for applications such as piezo-electric actuation has not been reported so far. Thus, in this work, we investigate the compressive and tensile mechanical properties and electromechanical electrical properties based on RTV silicone rubber and hybrid fillers such as CNT and CB. The CNT and CB are used in pristine form to avoid the use of hazardous chemical materials and to be useful for a large scale in industry. The improved properties were also transferred to a piezo-electric actuator.

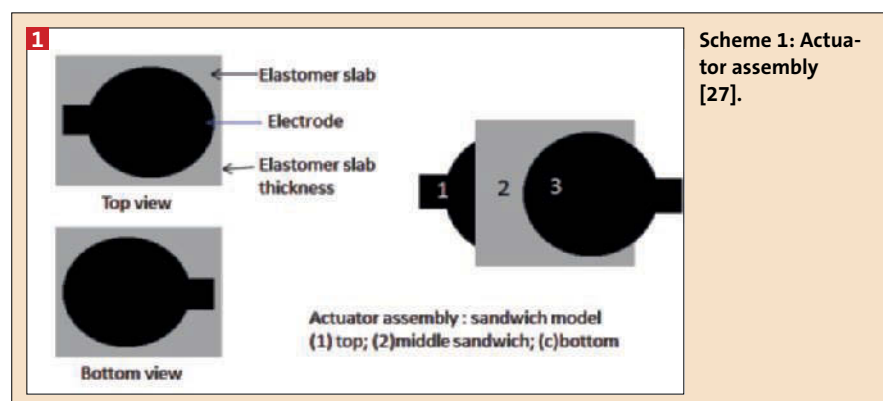
Experimental

Materials used for fabrication

Commercially available RTV silicone rubber (KE-441, Shin-Etsu Chemical Corporation Ltd.) was used as a rubber matrix. A hardener (CAT-RM, Shin-Etsu Chemical Corporation Ltd.) was used as a curing agent. Commercially available multi-wall CNT (CM-100, Hanwha Nanotech Corporation Ltd) and nano-carbon black (Conductex SC Ultra - Saehan Silichem Corporation Ltd) were used as reinforcing fillers in the silicone rubber.

Characterization techniques

Scanning electron microscopy (SEM; Hitachi) was used to study the morphology and dispersion of the CB and CNT particles in silicone rubber. X-ray diffraction (XRD, D8 Advance, Bruker) was used to study the crystal structure of the CB and CNT. The compressive and tensile strength was studied through a universal



testing machine (Lloyd instruments). The electrical properties were performed in ambient conditions using a digital multimeter (34401A; Agilent Technologies Inc).

Piezo-electric actuation measurements were performed using a laser sensor (Opto NCDT 1302, Micro-Epsilon Messtechnik). Composites based on CB and a CNT+CB hybrid were used as electrodes on either side of an elastomer slab made of 1 mm thick 3M elastomer. The electrode thickness was limited to 0.1 mm. The elastomer slab was sandwiched between the conductive electrodes based on nanocomposites filled with CB and CB, and CNT-CB hybrid [27].

Preparation of composites

The preparation of nanocomposites was initiated by spraying molds used in preparing samples with mold releasing agent and leaving them for 5-6 hours for drying. Then, the mixing of CB, CNT, and their hybrids was initiated with silicone rubber for 10-12 minutes. Afterwards, the composites were put in a vacuum for 30 minutes to remove air cavities in the silicone rubber. Then, 1.5 phr of hardener was added as a curing agent, mixed for 1 minute, and poured in a mold. The composites were kept for curing for 24 hours at room temperature and finally tested for mechanical, electrical, and actuation properties.

Results and discussion

Characterization of CNT and CB nanoparticles

The morphology of CNT was studied using SEM and is shown in Figure 1a. It was found that CNT exhibit 1-dimensional (1-D), tube-shaped morphology with different lengths and diameters. The formation of sp^2 hybridized nanotubes sug-

gests that engineering of the carbon allotropes is possible with different dimensions such as fullerene (0-D), carbon black (0-D), CNT (1-D), graphene (2-D), and graphite (3-D). The CNT were randomly distributed and dispersed in a highly interwoven state.

CNT were characterized with different lengths ranging from a few micrometers ($>5-7 \mu\text{m}$) to a few nanometers ($>500 \text{ nm}$). The diameter of these tubes was in the range of 12-15 nanometers. It is reported that the length, diameter, and orientation of CNT depends on the synthesis method [28,29]. The CNT core is hollow with multiple tubes wrapped concentrically and almost parallel to the CNT axis [30]. The pristine CNT are typically hydrophobic in nature, and in order to be functionalized, a hydrophilic organic group is required for providing attractive attraction [31].

Ball milling variables can affect the dispersion, interfacial structure, and dimension of the CNT. Insufficient milling affects the dispersion of CNT, and they are readily damaged. As the milling intensity is increased, the CNT are dispersed through plastic deformation [32].

CB has variable structure depending on the morphology from a few angstroms to the micrometers range. CB is widely used as a reinforcing filler for improving the mechanical, electrical, and thermal properties of composites. In general, the CB is a dimensionless aggregate of primary particles made of carbon. The morphology of CB is oval shaped with different aggregates of the CB particles forming a "grape-like" structure with a mean diameter of 25-30 nanometers. Some CB particles fuse together to form aggregates. The structure of CB becomes bigger as the number of aggregated particles increases. However, tracing out the regular outline of these CB aggregates is a challenge [33].

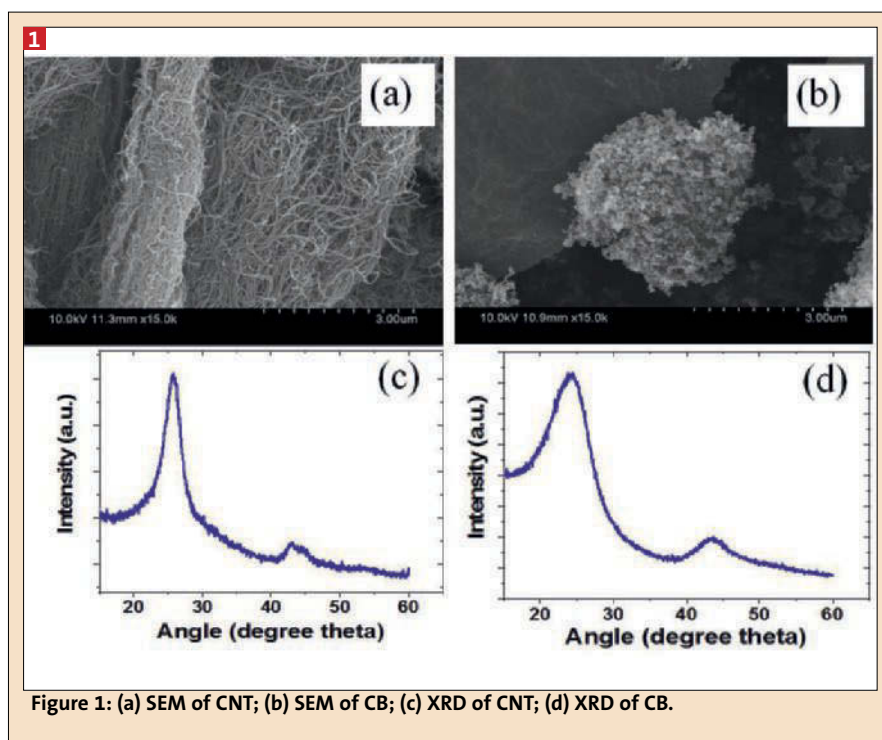


Figure 1: (a) SEM of CNT; (b) SEM of CB; (c) XRD of CNT; (d) XRD of CB.

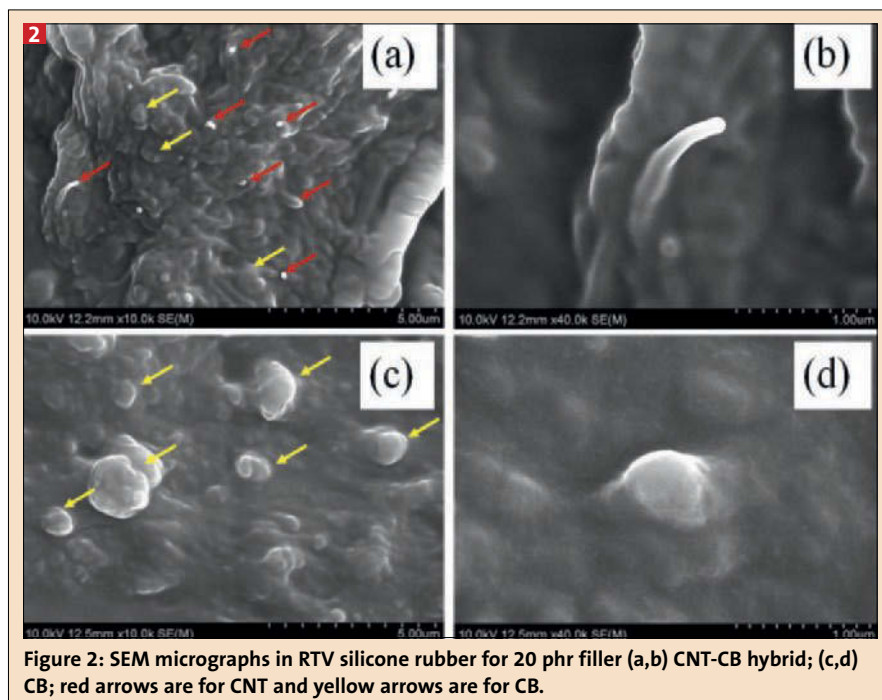


Figure 2: SEM micrographs in RTV silicone rubber for 20 phr filler (a,b) CNT-CB hybrid; (c,d) CB; red arrows are for CNT and yellow arrows are for CB.

With the bulk production of CNT for various industrial applications, extensive work has been done in studying the morphology and crystal structure of CNT. The crystalline state of CNT was studied through XRD and is shown in Figure 1c. There is a clear and sharp (002) peak at a 2θ value of 26.35° with an interlayer space (d_{002}) around 0.34 nanometers. However, the interlayer space between adjacent

sheets can be increased via an intercalant. The increased space depends upon the type of intercalant [34,35]. The (002) peak of CNT can be used to study the number of graphene layers wrapped concentrically ($N=40$ layers) in the CNT particles.

From Scherrer's equation, the size of the CNT is 13.5 nm, which is in agreement with the SEM micrographs. Another clear (101) peak at a 2θ value of

44.7° reveals crystalline phases in CNTs. The crystalline state of CB was also studied through XRD and is shown in Figure 1d. In CB, there is also a clear but broad (002) peak at a 2θ value of 24.1° that signifies the random arrangement of the graphitic layers in CB. XRD also reveals that an appreciable amount of amorphous carbon is absent in CNT and CB. Our XRD results of CNT and CB are in agreement with the published results in the literature [36,37].

Filler dispersion through SEM

The filler dispersion was studied as it is important factor which determines the properties of the composites [38]. The filler dispersion was studied through SEM and is shown in Figure 2. SEM micrographs show fine dispersion of the CNT-CB hybrid (Figures 2a-2b) and CB (Figures 2c-2d). SEM also shows that CNT and CB have a good interface with the silicone rubber matrix. SEM also confirms that CNT has better dispersion at the same loading than CB, where most of the matrix is unoccupied with the filler particles. This is due to the high aspect ratio of the CNT (65-70), their high surface area ($\sim 250 \text{ m}^2/\text{g}$) that has a higher interfacial area, and good polymer filler compatibility. It is reported that the filler dispersion and filler-polymer compatibility play a significant role in determining the properties of the vulcanizates [39]. Good dispersion and high polymer-filler interaction also justify the higher mechanical, electrical, and actuation properties, as shown in Figures 3-6.

Compressive mechanical properties

The compressive properties of CB and its hybrid with CNT were studied and are presented in Figure 3. The compressive profiles of stress and strain for CB filled composites are shown in Figure 3a, and CNT-CB hybrid composites are shown in Figure 3b. The results show that the compressive stress increases with increasing compressive strain from 0 to 35%. For example, the compressive stress at 1% compressive strain was 0.023 MPa (CNT-CB, 3 phr) and 0.019 MPa (CB, 3 phr) and increases to 1.99 MPa (CNT-CB, 20 phr) and 1.68 MPa (CB, 20 phr) at 35% compressive strain. Similarly, the compressive stress increases with increasing content of CNT-CB and CB in the silicone rubber matrix. This is attributed to the efficient filler networking [40,41], improved filler-filler interaction, and elasto-

mer-filler interaction [42].

The compressive modulus as a function of filler loading was studied and is shown in Figure 3c. It was found that the compressive modulus increases with increasing filler content with a maximum modulus for CNT-CB content of 20 than CB composites. For example, the compressive modulus was 5.17 MPa (CNT-CB, 3 phr) and 4.04 MPa (CB, 3 phr) and increases to 9.6 MPa (CNT-CB, 20 phr) and 7.9 MPa (CB, 20 phr). This can be attributed to the high aspect ratio of CNT, high interfacial area, and uniform dispersion of CNT in the rubber matrix [42]. Moreover, the compressive stress of the unfilled rubber increases with increasing compressive strain. This was attributed to increasing packing fraction of the polymer chains of silicone rubber with increasing compressive strain.

The reinforcing factor was studied as a function of filler loading for the CNT-CB hybrid and CB composites and is shown in Figure 3d. From the studies, it was found that the reinforcing factor increases with increasing filler content in the silicone rubber. For example, the reinforcing factor was 1.3 (CNT-CB, 3 phr) and 1.08 (CB, 3 phr) and increases to 2.6 (CNT-CB, 20 phr) and 2.13 (CB, 20 phr). It can also be interesting to note that the CNT-CB-based composites show the highest reinforcing factor at all loadings as expected. This is attributed to the high surface area, high aspect ratio [43,44], and high rate of stress transfer from polymer chains of the rubber matrix to CNT particle aggregates [45].

Tensile mechanical properties

The tensile stress-strain properties of the CNT-CB hybrid and CB were studied and are presented in Figure 4. The stress-strain profiles of CB composites are presented in Figure 4a, and the CNT-CB hybrid nanocomposites are shown in Figure 4b. It was found that the stress increases with increasing tensile strain until fracture strain for all nanocomposites. For example, the tensile stress increases with increasing filler content. It was found that the tensile strength increases with increasing filler content and was highest for CNT-CB composites. For example, the tensile strength was 0.87 MPa (CNT-CB, 3 phr) and 0.52 MPa (CB, 3 phr) and increases to 2.29 MPa (CNT-CB, 20 phr) and 1.69 MPa (CB, 20 phr). This can be attributed to the high aspect ratio of CNT that allows the formation of long-

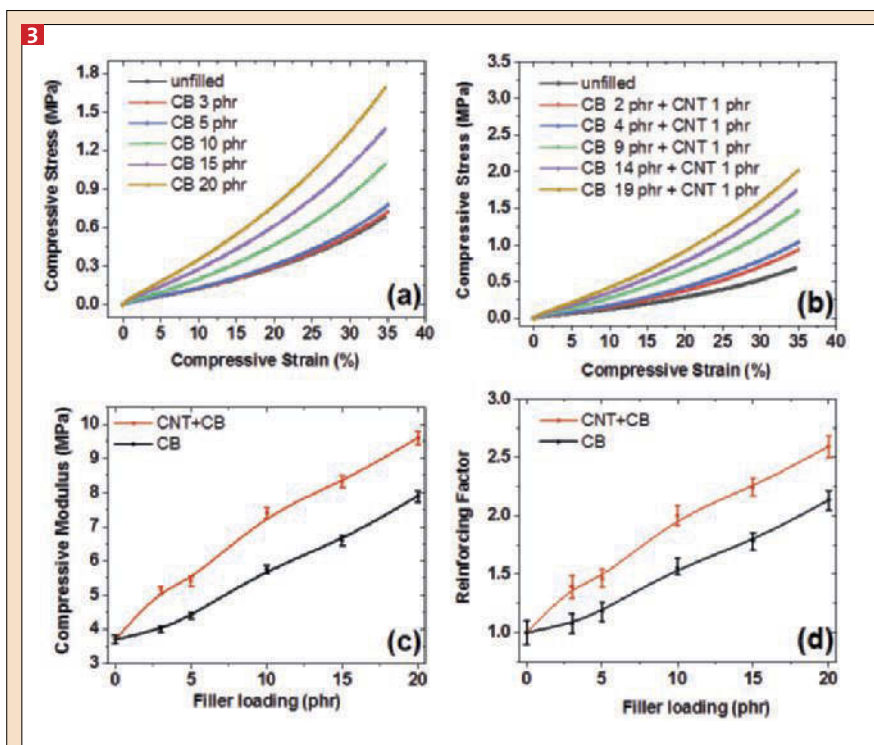


Figure 3: Compressive mechanical properties: (a) profiles of compressive stress as a function of compressive strain for CB composites; (b) profiles of compressive stress as a function of compressive strain for CB-CNT composites; (c) compressive modulus for CB and CB-CNT composites; (d) reinforcing factor for CB and CB-CNT composites.

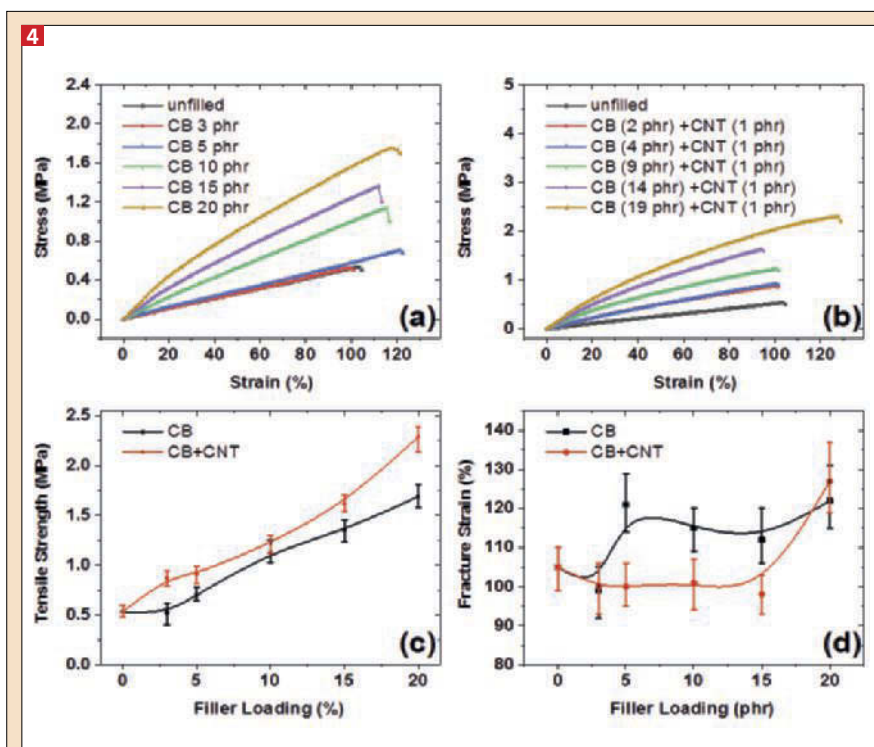


Figure 4: Tensile mechanical properties: (a) profiles of tensile stress as a function of tensile strain for CB composites; (b) profiles of tensile stress as a function of strain for CNT-CB composites; (c) Tensile strength for CB and CB-CNT composites; (d) Fracture strain for CB and CB-CNT composites.

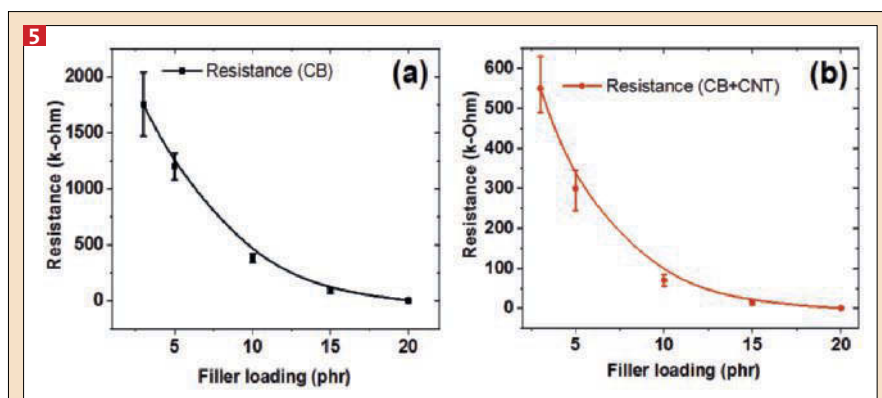


Figure 5: Electrical measurements: resistance as a function of filler content in rubber matrix (a) CB; (b) CB+CNT.

range filler networks at low filler content in the nanocomposites [46, 47].

The tensile strength was studied by changing the filler content of the composites and is shown in Figure 4c. It was found that the tensile strength increases with increasing filler content in the composites and was highest for CNT-CB composites at 20 phr. This was attributed to the higher reinforcing effect of the CNT due to their large length and CNT-matrix interface in the composites [48].

Figure 4d shows fracture strain increases with increasing filler content in the composite. It was found that the addition of filler make the composites stiffer and stretchable but brittle. That means with the addition of filler in the polymer matrix, the fracture strain decreases and then increases. For example, the fracture strain was 99 % (CNT-CB, 3 phr) and 100 % (CB, 3 phr) and increases to 127 % (CNT-CB, 20 phr) and 122 % (CB, 20 phr). This is due to the deformation of filler in polymer composites [49]. The fracture strain in CNT is also affected by vacancy defects, holes [50], and the filler percolation threshold [51].

Electrical properties

The resistance as a function of filler content was studied and is presented in Figures 5a and 5b. It was found that the electrical resistance falls as the content of filler increases in the composite. For example, the resistance was 550 k Ω (CNT-CB, 3 phr) and 1750 k Ω (CB, 3 phr) and decreases to 0.5 k Ω (CNT-CB, 20 phr) and 7 k Ω (CB, 20 phr). The decrease of resistance with increasing filler content is due to the formation of conductive filler networks in the nanocomposites [52,53]. It was found from the measurements that there is sharp drop in resis-

tance with the incorporation of CNT in CNT-CB hybrid composites. The high aspect ratio of CNT leads to formation of percolates networks at lower content (2 wt%), as described in the literature [54]. In another study, the formation of conductive percolates networks of CNT in composites was at below 5 phr [7].

Applications

Piezo-electric actuation

Piezo-electric actuators based on silicone rubber, the CNT-CB hybrid, and CB were studied and are presented in Figures 6a and 6b. The composites were used as electrode materials with a thickness of 0.1 mm, and the elastomer slab of the actuator was made up of 1 mm thick 3M silicone rubber. It was found that the mechanical actuation displacement increases with increasing input voltage and is the maximum for CNT-CB composites. For example, the actuation displacement for nanocomposites at 2kV was 0.15 mm (CNT-CB hybrid) and 0.1

mm (CB). It increases to 1.45 mm (CNT-CB hybrid) and 0.54 mm (CB) at 8 kV. It is therefore concluded that the actuation displacement depends on the type of material used, input voltage, thickness of the actuator electrode, and the actuator slab. For example, the actuation displacement rapidly increases with lower thickness of the electrode and elastomer slab, higher input voltage, and type of materials used in preparing the electrode and elastomer slab [27].

Conclusion

The work showed a cost effective new hybrid composite, its effect on properties, and applications such as piezo-electric actuation. The composites were found to exhibit high stretchability and flexibility to be useful for high actuation displacement. The morphological aspects, interfacial adhesion, and dispersion of the CB and CNT-CB particles in composites were studied through SEM micrographs. The compressive modulus was 9.6 MPa (20 phr CNT+CB) and 7.9 MPa (20 phr CB). It was found that the resistance falls to as low as 0.5 k Ω (CNT-CB, 20 phr) and 7 k Ω (CB, 20 phr). These improved properties were tested in real-time piezo-electric actuation. The piezo-electric actuation results showed that the actuation at 8 kV was 1.45 mm (CNT-CB, 3 phr) and 0.54 mm (CB, 3 phr). Thus, mechanical and electric measurements show high properties to achieve high actuation displacement.

Acknowledgements

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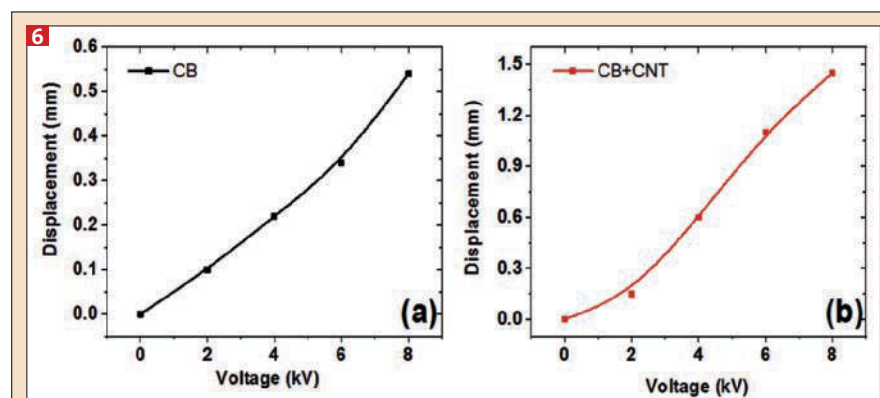


Figure 6: Actuation displacement as a function of voltage for 3 phr filler in electrode composite (a) CB- filled; (b) CB+CNT-filled rubber composites for flexible devices.

References

- [1] L. Bokobza, *Polymer*, **48** (17) (2007) 4907.
- [2] A. D. Moghadam, E. Omrani, P. L. Menezes, P. K. Rohatgi, *Compos. Part B: Eng.* **77** (2015) 402.
- [3] A. I. Medalia, *Rubber Chem. Technol.* **59** (3) (1986) 432.
- [4] V. Kumar, D.-J. Lee, *J. Appl. Polym. Sci.*, **134** (4), 44407 (1-9).
- [5] M. Galimberti, V. Kumar, M. Coombs, V. Cipolletti, S. Agnelli, S. Pandini, L. Conzatti, *Rubber Chem. Technol.* **87** (2) (2014) 197.
- [6] M. Galimberti, V. Cipolletti, S. Musto, S. Cioppa, G. Peli, M. Mauro, G. Guerra, S. Agnelli, T. Riccò, V. Kumar, *Rubber Chem. Technol.*, **87** (3) (2014) 417.
- [7] A. Das, G. R. Kasaliwal, R. Jurk, R. Boldt, D. Fischer, K. W. Stöckelhuber, G. Heinrich, *Compos. Sci. Technol.* **72** (16) (2012) 1961.
- [8] S. Stankovich, D. A. Dikin, G. H. B. Dommett, K. M. Kohlhaas, E. J. Zimney, E. A. Stach, R. D. Piner, S. B. T. Nguyen, R. S. Ruoff, *Nature*, **442**, (2006) 282.
- [9] V. Kumar, G. Lee, Monika, J. Choi, D.-J. Lee, *Polymer*, **190** (2020) 122221 (1-8).
- [10] V. Kumar, G. Lee, K. Singh, J. Choi, D.-J. Lee, *Sensors Actuators A: Phys.* **303** (2019) 111712 (1-9).
- [11] L. Karasek, B. Meissner, S. Asai, M. Sumita, *Polymer*, **28**, (1996) 121.
- [12] B. G. Soares, F. Gubbels, R. Jérôme, E. Vanlathem, R. Deltour, *Rubber Chem. Technol.* **70** (1) 1997, 60.
- [13] A. I. Medalia, *Rubber Chem. Technol.*, **47** (2) 1974, 411.
- [14] V. Kumar, J.Y. Lee, D.-J. Lee, *Polymer International*, **66** (3) 450.
- [15] Maurizio Galimberti Michele Coombs Pierluigi Riccio Theonis Riccò Simone Passera Stefano Pandini Lucia Conzatti Andrea Ravasio Incorporata Tritto, *Macromol. Mater. Eng.*, **298** (2) 2013, 241.
- [16] „G.-W. Lee, M. Park, J. Kim, J.-I. Lee, H. G. Yoon, *Compos. Part A: Appl. Sci. Manuf.*, **37**(5) (2006) 727.
- [17] A. Yu, P. Ramesh, X. Sun, E. Bekyarova, M. E. kis, R. C. Haddon, *Adv. Mater.*, **20** (24) (2008) 4740.
- [18] N. Rattanasom, T. Saowapark, C. Deeprasertkul, Reinforcement of natural rubber with silica/carbon black hybrid filler, *Polymer Testing*, **26** (3) 2007, 369.
- [19] H. Li, L. Yang, G. Weng, W. Xing, J. Wu, G. Huang, *J. Mater. Chem. A*, **3** (2015) 22385.
- [20] V. Kumar, R.R. Wu, D.-J. Lee, *Polymer Composites*, **40** (S1) E373-E382.
- [21] M. Kluppel, R. H. Schuster, G. Heinrich, *Rubber Chem. Technol.*, **70** (2) (1997) 243.
- [22] R. H. Schuster, H. M. Issel, V. Peterseim, *Rubber Chem. Technol.*, **69** (5) 1996, 769.
- [23] M. M. Mowes, F. Fleck, M. Kluppel, *Rubber Chem. Technol.*, **87** (1) (2014) 70.
- [24] K. Pal, R. Rajasekar, D. J. Kang, Z. Xi. Zhang, S. K. Pal, C. K. Das, J. K. Kim, *Mater. Design*, **31** (2) (2010) 677.
- [25] V. Kumar, X.W. Tang, S. C. Liu, D.-J. Lee, *Sensors and Actuators A: Physical*, **267**, 2017, 310.
- [26] M. L. Anderson, P. H. Mott, C. M. Roland, *Rubber Chem. Technol.*, **77** (2) 2004, 293.
- [27] V. Kumar, D.-J. Lee, *Polymers for Advanced Technologies*, **28** (12), 1842.
- [28] Zhu, H. W.; Xu, C. L.; Wu, D. H.; Wei, B. Q.; Vajtai, R.; Ajayan, P. M. *Science*, 2002, 296, 884.
- [29] Zhang, M.; Fang, S.; Zakhidov, A. A.; Lee, S. B.; Aliev, A. E.; Williams, C. D.; Atkinson, K. R.; Baughman, R. H. *Science*, 2005, 309, 1215.
- [30] D. Eder, A.H. Windle, *J. Mater. Chem.*, 2008, **18**, 2036.
- [31] M. S. Liu, M. C. C. Lin, I. T. Huang, C. C. Wang, *International Communications in Heat and Mass Transfer*, **32** (9) 2005, 1202.
- [32] H. J. Choi, J. H. Shin, D. H. Bae, *Composites Part A: Applied Science and Manufacturing*, **43** (7) 2012, 1061.
- [33] A.I. Medalia, *J Colloid Interf. Sci.*, **32** (1970) 115.
- [34] M.S. Dresselhaus & G. Dresselhaus *Advances in Physics*, **30** (2) 1981, 139.
- [35] M. S. Dresselhaus & G. Dresselhaus *Advances in Physics*, **51** (1) 2002, 1-186.
- [36] Bandow, S.; Asaka, S.; Saito, Y.; Rao, A. M.; Grigorian, L.; Richter, E.; Eklund, P. C. *Phys. Rev. Lett.* 1998, **80**, 3779.
- [37] Cao, A.; Xu, C.; Liang, J.; Wu, D.; Wei, B. *Chem. Phys. Lett.* 2001, **344**, 13.
- [38] J.Y. Lee, V. Kumar, X.W. Tang, D.-J. Lee, *Compos. Sci. Technol.*, **142**, 2017, 1-9.
- [39] L. Karásek, M. Sumita, *Journal of Materials Science*, 1996, **31** (2), 281.
- [40] Heinrich G., Klüppel M. *Advances in Polymer Science*, **160**, 2002, Springer, Berlin, Heidelberg.
- [41] „M.-J. Wang *Rubber Chem. Technol.*, **72** (2) (1999) 430.
- [42] J. Fröhlich, W. Niedermeier, H.-D. Luginland, *Composites Part A: Applied Science and Manufacturing*, **36** (4) 2005, 449.
- [43] J. Li P. C. Ma W. S. Chow C. K. To B. Z. Tang J.-K. Kim, *Adv. Funct. Mater.*, **17** (2007), 3207.
- [44] J.K.W. Sandler, J.E. Kirk, I.A. Kinloch, M.S.P. Shaffer, A.H. Windle, *Polymer*, **44** (2003) 5893.
- [45] L. Gong, I. A. Kinloch, R. J. Young I. Riaz, R. Jallil K. S. Novoselov, *Adv. Mater.*, **22** (24) (2010) 2694.
- [46] Lingbo Zhu, Jianwen Xu, Yonghao Xiu, Yangyang Sun, Dennis W. Hess, C.P. Wong, *Carbon*, **44** (2), 2006, 253.
- [47] H. Lorenz, J. Fritzsche, A. Das, K. W. Stöckelhuber, R. Jurk, G. Heinrich, M. Klüppel, *Compos. Sci. Technol.*, **69** (13) 2009, 2135.
- [48] H. Wan, F. Delale, L. Shen, *Mechanics Research Communications*, **32** (5) 2005, 481.
- [49] C. Bower, R. Rosen, L. Jin, J. Han, O. Zhou, *Appl. Phys. Lett.* **74**, 3317 (1999).
- [50] S. L. Mielke, D. Troya, S. Zhang, J.-L. Li, S. Xiao, R. Car, R. S. Ruoff, G. C. Schatz, T. Belytschko, *Chem. Phys. Lett.*, **390** (4-6) (2004) 413.
- [51] C. A. Martin, J.K.W. Sandler, M.S.P. Shaffer, M.-K. Schwarz, W. Bauhofer, K. Schulte, A.H. Windle, *Compos. Sci. Technol.*, **64** (15) 2004, 2309.
- [52] „F. Du, R. C. Scogna, W. Zhou, S. Brand, J. E. Fischer, K. I. Winey, *Macromolecules*, 2004, **37** (24) 9048.
- [53] I. Aliga, T. Skipa, D. Lellinger, P. Potschke, *Polymer*, **49** (16) 2008, 3524.
- [54] A. Das, K. W. Stöckelhuber, R. Jurk, M. Saphirnikova, J. Fritzsche, H. Lorenz, M. Klüppel, G. Heinrich, *Polymer*, **49** (24) 2008, 5276.

Mikroglasperlen als hochwertiger Füllstoff zur Verbesserung der physikalischen Eigenschaften von Kunststoffen

GLASFÜLLPERLEN Glasfüllperlen sind wichtige Füllstoffe von Kunststoffen. In der Kunststofftechnik werden die aus Kalk-Natron-Glasbruch hergestellten Glasfüllperlen Swarcoforce von Swarco, Amstetten, Österreich, zur Optimierung des Eigenschaftsprofils von Kunststoffen eingesetzt. Das Einarbeiten er-

folgt üblicherweise durch Compoundierung. Im Vordergrund der Verwendung von Glaskugeln für das Verstärken von Kunststoffen steht meist, dass die Steifigkeit erhöht, die Schrumpfung vermindert und die Oberflächenanmutung verbessert werden sollen. Bei sogenannten Hybridkombinatio-

nen von Glasfaser- und Glaskugolverstärkungen verbindet sich im Kunststoffcompound die höhere Steifigkeit der Glasfaser mit der Reduktion der Verzugsneigung durch die Glaskugolverstärkung. Dass Glasfüllperlen auch die Anforderungen mit ökologisch nachhaltigen Rohstoffen erfüllen, ist ein geschätzter Vor-

teil, da auch grüne Produkte verwirklicht werden können. Das zur Produktion verwendete Recyclingmaterial ist ausschließlich Verschnitt und Abfall aus Industrieglas mit hoher Qualität. ■

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T-Equal Channel Angular Extrusion · T-shaped angular domain · α -beveled punch · α -inclined punch · Computational Fluid Dynamics

The paper is focused on a Computational Fluid Dynamics (CFD)-based description of local flow peculiarities during T-Equal Channel Angular Extrusion (T-ECAE) of an α -inclined punch-driven viscous fluid through a T-shaped die, derived with a numerical solution of a boundary value problem for the vorticity equation. The range $0^\circ \leq \alpha \leq 45^\circ$ of technologically acceptable values for punch-tool inclination angles for the T-ECAE problem was visualized with 2D plots for computational flow-lines, absolute w -values of dimensionless flow velocities and absolute τ -values of dimensional tangential stresses.

Auf Fluid Mechanik basierende Untersuchung angeschrägter Extrusionsstempel unter Einsatz eines T-förmig gewinkelten Bereichs

T-Equal Channel Angular Extrusion · -Verfahren · T-förmig gewinkelter Bereich · α -angeschrägter Stempel · α -geneigter Stempel · Computational Fluid Dynamics

Die Arbeit ist fokussiert auf eine "Computational Fluid Dynamics (CFD)"-basierte Beschreibung von lokalen Fließbesonderheiten in der Anwendung des "T-Equal-Channel-Angular-Extrusion"-Verfahrens (ECAE) für ein viskoses Fluid, welches mit einem α - geneigten Stempel durch eine T-förmige Düse getrieben wird. Die hiermit verbundenen Werteprobleme für die Wirbelvektor Gleichung werden durch numerische Lösungen hergeleitet. Der Bereich von $0^\circ \leq \alpha \leq 45^\circ$ technologisch akzeptabler Werte für den Neigungswinkel des Stempelwerkzeugs für das T-ECAE Problem wurde durch 2 D-Plots für computergestützte Fließkurven, absolute w -Werte der dimensionslosen Fließgeschwindigkeiten und der absoluten w -Werte dimensionaler tangentialer Spannungen visualisiert.

Figures and Tables:
By a kind approval of the authors.

Fluid Mechanics-based Research into beveled Punch-assisted Extrusion through a T-shaped Angular Domain

1. Introduction

The technological problem of local flow of extruded material through a T-shaped die is an interesting technical and instructional problem of polymer processing (Figures 1 - 3).

This problem of T-extrusion was recently addressed in the work of Wei et al. (2015) from the upper bound theorem viewpoint [1]. Perig et al.'s (2010-2019) Equal Channel Angular Extrusion (ECAE)-related research efforts [2-6], [8-10], [13-21] were focused on physical simulation [2-6], [8-9], [13-19], upper bound analysis [2-3], finite element study [8], Lattice Boltzmann modeling [21] and computational fluid dynamics (CFD)-enhanced description [9-10], [13-20] of local flows of extruded materials of varying rheology through angular domains with different die geometries. The forming problems of α -bevel punch-assisted ECAE were addressed in the works of Vaitsekhovich et al. (2013) [7], Perig et al. (2015, 2018) [8-10], Nejadseyfi et al. (2016) [11] and Comănesci et al. (2017) [12].

However, the problem of α -inclined punch-assisted ECAE through the T-shaped die (Figures 1 - 3) has not been previously addressed in the known published works [1-21], which confirms the paper's actuality, timely nature and research originality.

2. Paper aims, scopes and prime novelty

The object of the research is the process of α -beveled (α -inclined) punch-tool-assisted local viscous flow of incompressible continuum through a T-shaped equal channel angular domain for a range of punch inclination angles $\alpha = \{0^\circ \dots 60^\circ\}$. The subject of the research is focused on the α -inclination dependent flow-shaped geometry of the macroscopic rotation zone with intensive material mixing during T-ECAE-forming. The research scope of the paper is focused on the CFD-enhanced determination of the rational value α^* of a beveled punch α -inclination angle for the best mixing of viscous ma-

terial within a T-ECAE die (Figures 1 - 3). The main research method is associated with the finite difference-based numerical solution of the Navier-Stokes CFD-equations in the vorticity (curl) transfer form. The prime novelty of the paper is that, for the first time, the boundary value problem for the α -beveled punch-driven viscous flow through a T-shaped ECAE domain was formulated, numerically solved and 2D visualized in Figures 1 - 3 as a CFD 2D problem for the vorticity equation by development and Object Pascal implementation of an author-proposed computer code.

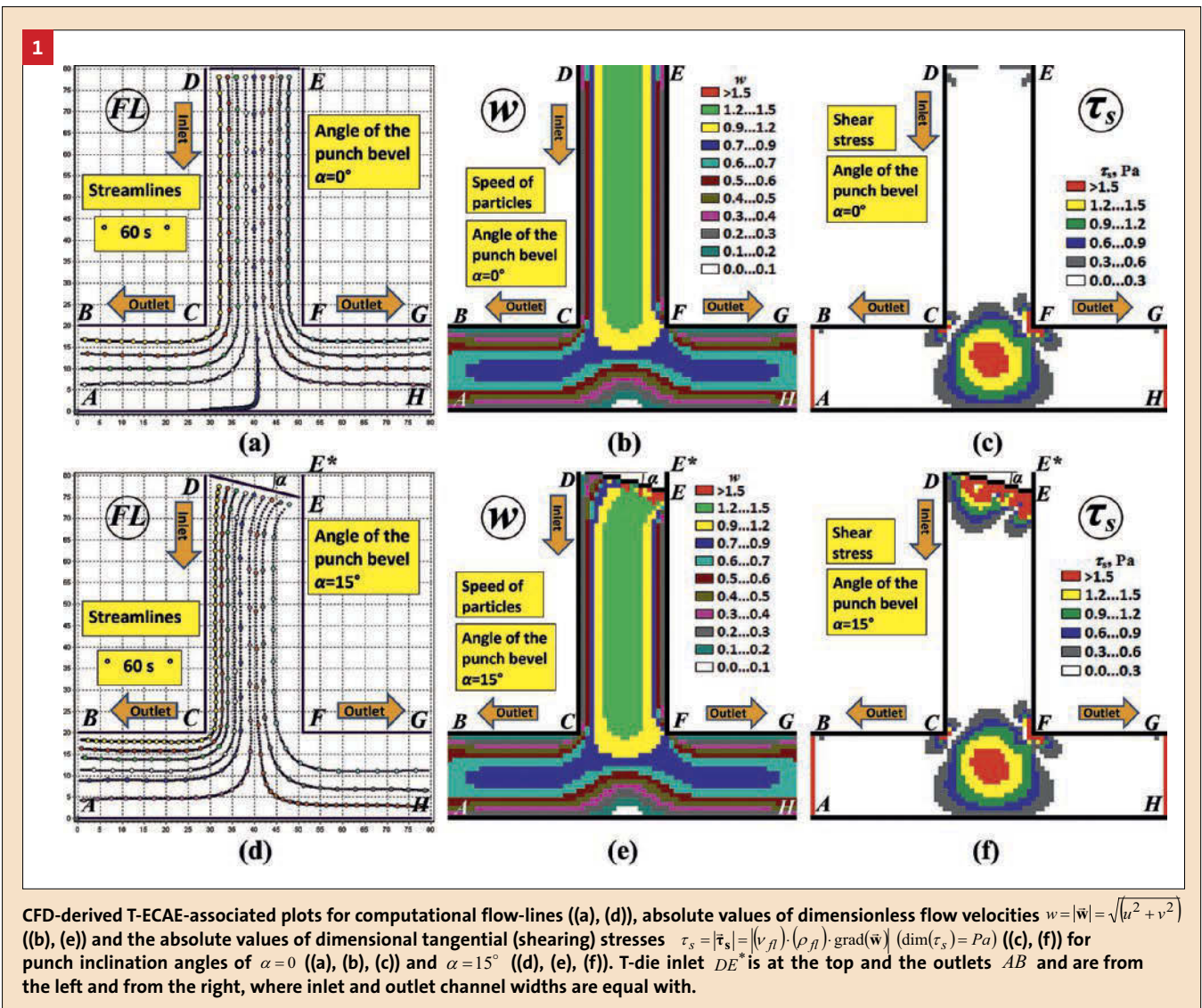
3. Discussion of research results and technical implications of the paper

The local flow of beveled (inclined) punch-driven viscous incompressible continuum (LFBIPdVIC) through a T-shaped angular domain (TsAD) is theoretically studied for a range of punch-tool inclination angles (PTIA) $\alpha = \{0^\circ \dots 60^\circ\}$ using the author-proposed and Object Pascal-implemented computer code for a Computational Fluid Dynamics (CFD)-based numerical solution of the corresponding boundary value problem for

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Curl (Vorticity) Transfer Equation (CVTE). The CFD-based study of (TsAD)-located (LFBIPdVIC) is based on the 2D visualization of the fields of full flow velocities (w -FFFV) in Figures 1((b), (e)); 2((b), (e)); 3(b) and tangential (shearing) stresses (τ -FTSS) in Figures 1((c), (f)); 2((c), (f)); 3(c). The geometric location of a two-central intensive material mixing zone (TCIMMZ) is determined as the first and the second (TsAD)-sub-regions in the (τ -FTSS) 2D-diagram with the largest values of $\tau \rightarrow \tau_{max}$ in Figures 1((c), (f)); 2((c), (f)); 3(c). It is visualized with (τ -FTSS) that the first center (TCIMMZ)₁ of (TCIMMZ) is geometrically localized below the inclined punch edge and the second center (TCIMMZ)₂ of (TCIMMZ) is employed in the die channel intersection central zone of (TsAD). It is found with (τ -FTSS) that (PTIA)-increase ($\alpha \uparrow$) results in a significant growth in the first area

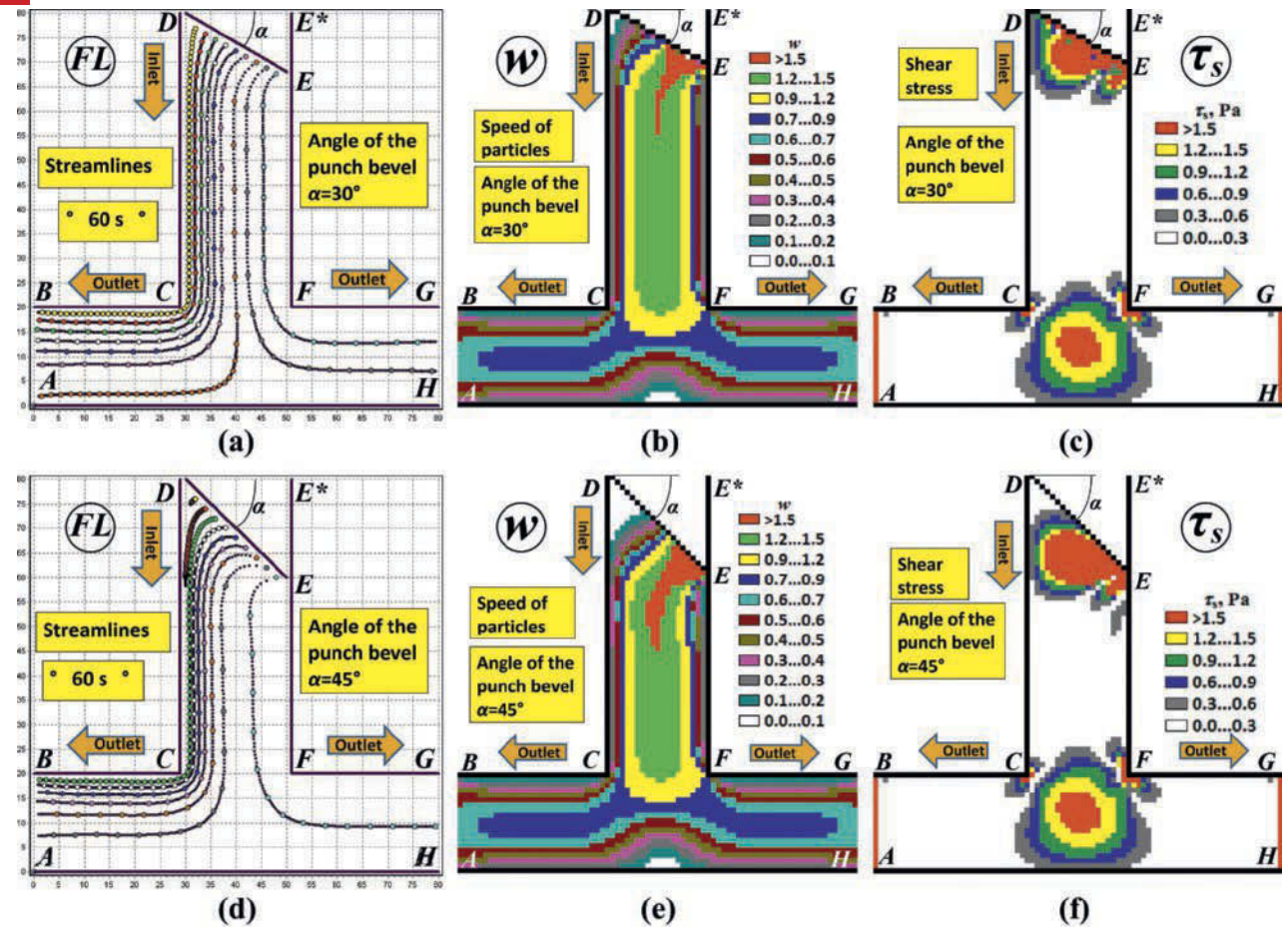
(Area_(TCIMMZ1) \uparrow): (($\alpha \uparrow$) \rightarrow (Area_(TCIMMZ1) \uparrow)). It is also found with (τ -FTSS) that (PTIA)-increase ($\alpha \uparrow$) does not have a substantial effect on the second area (Area_(TCIMMZ2) \approx const) of the second mixing zone (TCIMMZ)₂. The geometric location of the material dead (stagnation) zone (MDSZ) is determined as the third (TsAD)-sub-region in the (w -FFFV) 2D-diagram with a zero-level of $w = 0$ in Figures 1((b), (e)); 2((b), (e)); 3(b). It is found with (w -FFFV) that (PTIA)-increase ($\alpha \uparrow$) results in a slight growth in the area (Area_(MDSZ) \uparrow) of (MDSZ): (($\alpha \uparrow$) \rightarrow (Area_(MDSZ) \uparrow)).

4. Conclusions

The paper is the research into local flow and material mixing during beveled punch-assisted equal channel extrusion of viscous fluid through a T-shaped angular domain for a range of punch-tool inclination angles (Figures 1 - 3). The

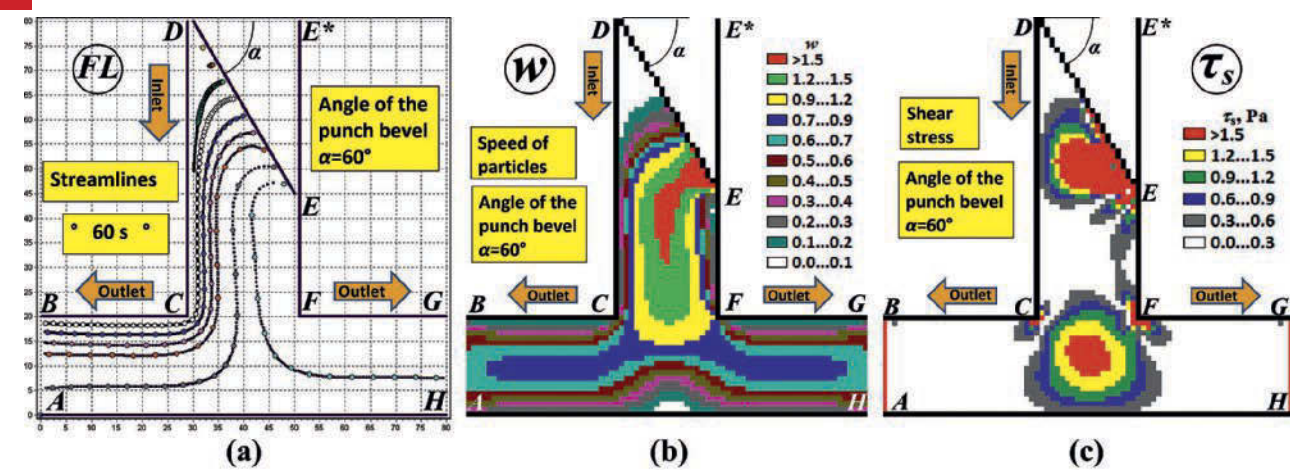
main CFD-supported research result of the paper is that the maximum technologically-acceptable value of α -beveled punch inclination angle for the best mixing of extruded material within T-ECAE die is $\alpha_{max} = 45^\circ$ in Figure 2((d), (e), (f)). Moreover, a further increase in α -values results in the unwanted formation of an extra-large area of viscous material dead zone (of white color) in the vicinity of the acute angle $\angle(CDE)$ in Figure 3, plotted for $\alpha = 60^\circ$. The main technical implication is that the author-proposed numerical solution of the vorticity equation essentially broadens the rheological ideas of applied engineering specialists and technical instructors in the field of materials science and engineering concerning the geometric peculiarities of α -beveled punch-tool-driven local flow of viscous continuum through a T-shaped angular domain in Figures 1 - 3.

2



CFD-derived T-ECAE-associated plots for computational flow-lines ((a), (d)), absolute values of dimensionless flow velocities $w = |\vec{w}| = \sqrt{u^2 + v^2}$ ((b), (e)) and the absolute values of dimensional tangential (shearing) stresses $\tau_s = |\vec{\tau}_s| = (v_{fl}) \cdot (\rho_{fl}) \cdot \text{grad}(\vec{w})$ ($\text{dim}(\tau_s) = \text{Pa}$) ((c), (f)) for punch DE^* inclination angles $\alpha = 30^\circ$ of ((a), (b), (c)) and $\alpha = 45^\circ$ ((d), (e), (f)). T-die inlet DE^* is at the top and the outlets AB and HG are from the left and from the right, where inlet and outlet channel widths are equal with.

3



CFD-derived T-ECAE-associated plots for computational flow-lines (a), absolute values of dimensionless flow velocities $w = |\vec{w}| = \sqrt{u^2 + v^2}$ (b) and the absolute values of dimensional tangential (shearing) stresses $\tau_s = |\vec{\tau}_s| = (v_{fl}) \cdot (\rho_{fl}) \cdot \text{grad}(\vec{w})$ ($\text{dim}(\tau_s) = \text{Pa}$) (c) for punch inclination angle of $\alpha = 60^\circ$ ((a), (b), (c)). T-die inlet DE^* is at the top and the outlets AB and HG are from the left and from the right, where inlet and outlet channel widths are equal with $a_{DE^*} = a_{CF} = a_{AB} = a_{HG}$

References

- [1] K. Wei, P. Liu, Z. Ma, W. Wei, I.V. Alexandrov, J. Hu, *Acta Metall. Slovaca* 21 (2015) 4, doi:10.12776/ams.v21i1.539.
- [2] A.V. Perig, *Mater. Res.-Ibero-Am. J. Mater.* 17 (2014) 1226, doi:10.1590/1516-1439.268114.
- [3] A. Perig, *Mater. Res.-Ibero-Am. J. Mater.* 18 (2015) 628, doi:10.1590/1516-1439.004215.
- [4] A.V. Perig, *J. Mater. Educ.* 39 (2017) 193.
- [5] A.V. Perig, I.S. Galan, *Lett. Mater.-Pis Ma O Materialakh* 7 (2017) 209, doi:10.22226/2410-3535-2017-3-209-217.
- [6] A.V. Perig, *Int. J. Contin. Eng. Educ. Life-Long Learn.* 29 (2019) 182, doi:10.1504/IJCE-ELL.2019.101039.
- [7] S.M. Vaitsekhovich, V.M. Mikhalevich, V.A. Kraevskii, *Powder Metall. Met. Ceram.* 52 (2013) 132, doi:10.1007/s11106-013-9505-y.
- [8] A.V. Perig, A.F. Tarasov, I.G. Zhibankov, S.N. Romanko, *Mater. Manuf. Process.* 30 (2015) 222, doi:10.1080/10426914.2013.832299.
- [9] A.V. Perig, N.N. Golodenko, *Mech. Sci.* 6 (2015) 41, doi:10.5194/ms-6-41-2015.
- [10] A.V. Perig, N.N. Golodenko, *Acta Metall. Slovaca* 24 (2018) 306, doi:10.12776/ams.v24i4.1147.
- [11] O. Nejadseyfi, A. Shokuhfar, S. Sadeghi, *Mater. Sci. Eng. A-Struct. Mater. Prop. Microstruct. Process.* 651 (2016) 461, doi:10.1016/j.msea.2015.08.050.
- [12] R.I. Comăneci, D. Nedelcu, L.G. Bujoreanu, *AIP Conf. Proc.* 1896 (2017) 200004, doi:10.1063/1.5008241.
- [13] A.V. Perig, A.M. Laptev, N.N. Golodenko, Y.A. Erfort, E.A. Bondarenko, *Mater. Sci. Eng. A-Struct. Mater. Prop. Microstruct. Process.* 527 (2010) 3769, doi:10.1016/j.msea.2010.03.043.
- [14] A.V. Perig, N.N. Golodenko, *Chem. Eng. Commun.* 201 (2014) 1221, doi:10.1080/00986445.2014.894509.
- [15] A.V. Perig, N.N. Golodenko, *Int. J. Adv. Manuf. Technol.* 74 (2014) 943, doi:10.1007/s00170-014-5827-2.
- [16] A.V. Perig, N.N. Golodenko, *Mater. Res.-Ibero-Am. J. Mater.* 19 (2016) 602, doi:10.1590/1980-5373-MR-2016-0013.
- [17] A.V. Perig, N.N. Golodenko, *Mater. Res. Express.* 3 (2016) 115301, doi:10.1088/2053-1591/3/11/115301.
- [18] A.V. Perig, N.N. Golodenko, *Adv. Mater. Sci. Eng.* 2017 (2017) 7015282, doi:10.1155/2017/7015282.
- [19] A.V. Perig, N.N. Golodenko, *AIMS Mater. Sci.* 4 (2017) 1240, doi:10.3934/mat-science.2017.6.1240.
- [20] A.V. Perig, N.N. Golodenko, *Adv. Mater. Process. Technol.* 5 (2019) 617, doi:10.1080/2374068X.2019.1666502.
- [21] D.S. Svyetlichnyy, A.V. Perig, L. Lach, R. Straka, A. Svyetlichnyy, *Int. J. Contin. Eng. Educ. Life-Long Learn.* 29 (2019) 151, doi:10.1504/IJCE-ELL.2019.101044.

Verschlussstopfen für gebohrte Kühlkreisläufe



Bild: Hasco

Die neuen Verschlussstopfen mit Viton-O-Ringen sind druckfest bis 10 bar, temperaturbeständig bis zu 180 °C und weisen eine sehr gute Korrosionsbeständigkeit auf.

und Umlenken des Temperiermediums innerhalb des Temperierkreislaufts. Sie lassen sich einfach montieren und demonstrieren und sind somit wieder verwendbar. Die Verschlussstopfen werden bei der Montage in die Bohrung geschoben und an vorgesehenen Stellen durch Spreizen eines O-Rings fixiert. Eine Befestigungsschraube mit Torx-Geometrie ermöglicht dabei ein einfaches Positionieren der Verschlussstopfen. Das Torx-Profil gewährleistet eine optimale und sichere Kraftübertragung der Drehmomente, ohne das Innenprofil zu beschädigen.

KONTAKT

Hasco, Lüdenscheid,
www.hasco.com

Z9425/... Kühlkreisläufe in Spritzgießwerkzeugen bestehen oft aus mehreren Bohrungen, die fertigungsbedingt nur gerade verlaufen können. Es werden möglichst konturnah mehrere sich kreuzende Bohrungen eingebracht. Um einen effektiven Durchlauf des Kühlmediums zu gewährleisten, werden die Bohrungen durch Verschluss- und Umlenkstopfen an bestimmten Stellen verschlossen, wodurch ein entsprechender Kühlkreislauf entsteht. Die neuen Verschlussstopfen Z9425/... von Hasco, Lüdenscheid, eignen sich sowohl zum Verschließen von Temperierbohrungen nach außen hin als auch zum Verteilen

Schmelzuführung ohne Ecken und Kanten mit Standardumlenkern

UMLENKTECHNIK Mittels einer ausgeklügelten Verteilertechnik, fließtechnisch optimierter Umlenker und hochwertig bearbeiteter Konturstopfen werden Füllvorgänge positiv beeinflusst und es wird ein gleichmäßigeres Fließverhalten erreicht. Günther Heisskanaltechnik, Frankenberg, bietet neben einer rheologisch und thermisch optimal ausgelegten Verteilertechnik neue, technisch ausgeklügelte Umlenkeinheiten für eine optimale Schmelzuführung an. Die neuen, geteilten Standardumlenker bieten fließtechnisch viele Vorteile, wie etwa hochgenaue Oberflächen der schmelzführenden Kanäle. Zudem haben sie den praktischen Vorteil, dass man sie bei Verschleiß oder zur Reinigung leicht und schnell wechseln

kann. Neben Umlenkern sind Stopfen zum Verschließen der hochwertig gebohrten Verteilerkanäle von entscheidender Bedeutung. Hierbei wird auf ein umfassendes Stopfen-Sortiment zurückgegriffen. Schräg- oder Konturstopfen lenken die Schmelze durch ihre Oberflächegeometrie sanfter um und sorgen trotzdem für einen guten Zugang zum Schmelzkanal im Reinigungsfall. ■

KONTAKT

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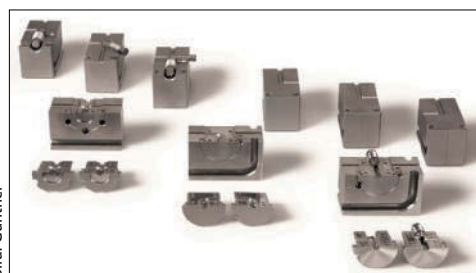


Bild: Günther

Die neuen, geteilten Standardumlenker bieten fließtechnisch viele Vorteile, wie etwa hochgenaue Oberflächen der schmelzführenden Kanäle.

Rubber-bonded magnets · magnetic characterisation · mechanical characterisation · hydrogenated acrylonitrile butadiene rubber · acrylonitrile butadiene rubber

The mechanical and magnetic properties of hard ferrite filled rubbers are investigated. Different compounds based on acrylonitrile butadiene rubber (NBR) and hydrogenated acrylonitrile butadiene rubber (HNBR) were manufactured and analysed. The magnetic fillers OP-71 from Dow and FA-700 from Toda Kogyo were used. The compounds were mixed in an internal mixer and subsequently processed in a roller mill. The hard ferrite particles were straightened by the rolling process. This effect is known as mechanical orientation. The vulcanisation occurred with and without magnetic field using a heating press. The cured materials with and without magnetic field are designated as mechanical-magnetic oriented and mechanically oriented respectively. The effect of anisotropy on the magnetic properties was investigated.

Magnetische und mechanische Charakterisierung von kautschuk-gebundenen harten Ferrit-Kompositen

Elastomergebundene Magnete magnetische Charakterisierung · mechanische Charakterisierung · hydrierter Acrylnitril-Butadien-Kautschuk · Acrylnitril-Butadien-Kautschuk

In dieser Veröffentlichung werden die mechanischen und magnetischen Eigenschaften von hartferritgefüllten Kautschuken untersucht. Hierzu wurden verschiedene Mischungen auf Basis von Acrylnitril-Butadien-Kautschuk und hydriertem Acrylnitril-Butadien-Kautschuk hergestellt und analysiert. Zum Einsatz kamen die magnetischen Füllstoffe OP-71 und FA-700. Die Mischungen wurden in einem Innenmischer gemischt und anschließend auf einer Walze verarbeitet. Die Hartferritpartikel wurden durch den Walzprozess gerichtet, was als mechanische Orientierung bezeichnet wird. Die Vulkanisation erfolgte mit und ohne Magnetfeld unter Verwendung einer Heizpresse. Die ausgehärteten Materialien mit und ohne Magnetfeld werden als mechanisch-magnetisch orientiert bzw. mechanisch orientiert bezeichnet.

Figures and Tables:
By a kind approval of the authors.

Magnetic and mechanical Characterisation of Rubber bonded hard Ferrites Composites

Introduction

Rubber-bonded hard ferrites composites are produced by mixing magnetic particles into rubber [1,2]. Similar to the plastic-bonded magnetic composites, these materials have a high geometrical freedom and high integration potential. The rubber-bonded magnets are vulcanized on coated insert component. By using the proper adhesion promoter, the bonding between the insert and the rubber layer is ensured. The manufactured components are used in sensor technology and drive technology [3,4,5]. The encoders for rotation speed measurement in the power train of the automatic transmissions are made of rubber-bonded materials. Despite the long cycle times for the manufacturing of rubber bonded magnets, the material cannot be replaced by the plastic bonded magnets in some specific applications, as some rubbers have better thermal and chemical resistance. In addition, the rubber bounded magnets have a better homogeneous particle distribution than the plastic bounded magnets. The homogeneity of the magnetic filler distribution in compounds greatly affects the accuracy of the manufactured products. The magnetic properties such as remanence, coercivity or energy product are the main criteria for the field of application of the manufactured products. The coercivity H_c is the magnetic field strength required to bring the polarisation back to zero in a ferromagnetic material which has been magnetised to saturation. The remanence B_r is the remaining magnetisation in a magnetic material at zero field strength, after it has been magnetised to saturation in a closed path. The greatest possible energy product $(B \cdot H)_{max}$ describes the highest energy density that can be achieved with a material. Figure (1) shows a hysteresis loop of a magnetic material. B_s describes the saturation of magnetisation and μ_i is the initial permeability.

Experiment

Sample preparation

The compounds were prepared by mixing NBR with FA-700 and HNBR with

OP-71 in an internal mixer and then kneaded on the roller mill. The hard ferrite powders OP-71 and the FA-700 were supplied from Dow and Toda respectively. The properties of both fillers are listed in table (1).

The magnetic fillers FA-700 and OP-71 are hard ferrites, which have a hexagonal crystal grid structure. The ferrimagnetic materials are mostly composed of strontium ferrite $SrO \cdot 6Fe_2O_3$ and barium ferrite $BaO \cdot 6Fe_2O_3$. The barium ferrites ($BaO \cdot 6Fe_2O_3$) has a lower crystal anisotropy than strontium ferrites ($SrO \cdot 6Fe_2O_3$). This manifests itself in lower coercivity. The FA-700 particles are on average coarser than the OP-71 particles. The pressed FA-700 powder has a slightly higher density than the OP-71 powder. The magnetic values cannot be compared with each other, as the data comes from pressed and powdery state. The hard ferrite particles have a plate shape. This form produces good adhesion between the particles and the polymer chains. In addition, the elongated shape can simplify the mechanical orientation. Figure (2) shows the particle distribution of OP-71 in an elastomer mixture.

NBR and HNBR were used due to their good thermal and media resistance. Table (2) provides an overview of the composition of the different compounds.

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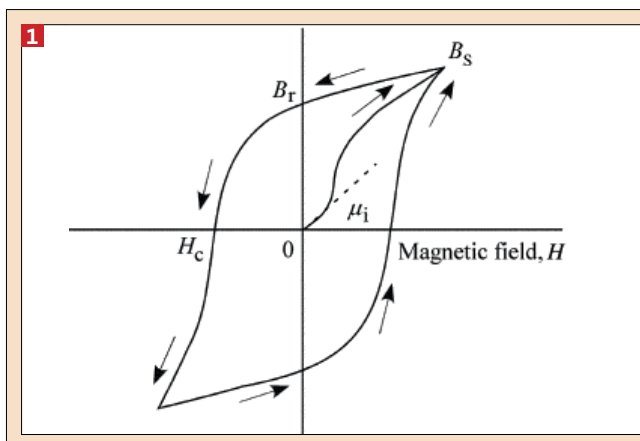


Figure 1: Hysteresis loop of a magnetic material [6].

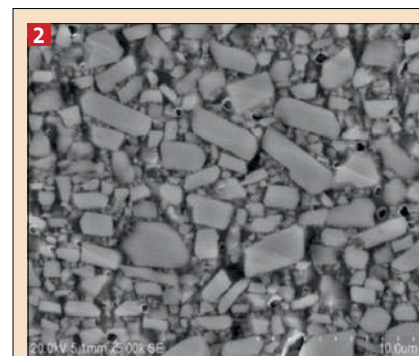


Figure 2: Cross section view of the OP-71 filled rubber compound [9].



Figure 3: Schematic of the curing setup for the preparation of magnetic-mechanically oriented samples during vulcanisation in an electrical heating press [10].

field in the cavity at the room temperature is about 0.3 T.

Mechanical characterisation

The density, hardness Shore A, elongation at break and tensile strength of the prepared samples were analysed. The test samples were punched out from a vulcanized plate. 6 mm and 2 mm plates were produced by compression moulding at a temperature of 180°C. The hardness Shore A was measured on a vulcanized 6 mm disk. The tensile strength and the elongation at break were measured on a uniaxial test machine using the S-2 tensile specimen.

Magnetic characterisation

The magnetic properties were determined on the Permagraph of the company Steingroever. The sample is clamped between two adjustable pole shoes of an electromagnet. The magnetic field is generated by the two coils. The metallic frame between the two pole shoes forms the magnetic inference. The induction and polarization of the sample is detected by an arranged measuring coil. The pole shoes are moved vertically until the sample and poles are on contact. Next, the excitation voltage is continuously increased.

By picking up the polarization and induction of the magnet using a rewind, the hysteresis loop is created. The magnetic characteristic values are determined from the second quadrant of the hysteresis loop. The measured curve in the second quadrant is known as “the demagnetization curve”. The remanence (B_r), the coercivity (H_c) and the energy product ($(BH)_{max}$) are calculated from this measured curve.

Results and discussion

Although the hard ferrites have weaker

The prepared samples were produced by the compression moulding process. To investigate the influence of the magnetic field on the orientation of the particles, samples at the German institute of rubber technology (DIK) were cured in

the presence of an external magnetic field. Figure (3) shows the curing setup to produce the pressed magnetic-mechanically oriented samples.

The magnetic field was generated by two square magnetic coils. The magnetic

1 Properties of the investigated hard ferrite powder [7, 8]

	Average particle diameter (APD) [μm]	Compressed density ρ_c [g/cm^3]	Remanence B_r [mT]	Coercivity H_c [kA/m]
FA-700	1.30±0.15	3.44±0.05	283±7	251±24
OP-71	1.25±0.15	3.35±0.1	195±7	203±12

2 Composition of the investigated compounds

Composites	Rubber	Crosslinking system	Hard ferrite type	Mass fraction of hard ferrite [m%]	Density [g/cm^3]
1	NBR	Sulphur	FA-700	23.8	1.36
2	NBR	Sulphur	FA-700	38.5	1.60
3	NBR	Sulphur	FA-700	48.5	1.78
4	NBR	Sulphur	FA-700	55.6	1.92
5	NBR	Sulphur	FA-700	80	2.95
6	NBR	Sulphur	FA-700	84	3.21
7	NBR	Sulphur	FA-700	86	3.39
8	NBR	Sulphur	FA-700	89	3.63
9	HNBR	Peroxide	OP-71	80	2.88
10	HNBR	Peroxide	OP-71	84	3.01
11	HNBR	Peroxide	OP-71	86	3.16

magnetic performance than the rare earth magnets, they are still frequently used because they have good electrical resistance, good chemical resistance and are significantly cheaper. In addition, the adhesion between elastomer and hard ferrite is much better than between rare earth magnets and elastomers.

The mixtures 1 to 4 are low filled and are hardly used for industrial applications because the magnetic performance is insufficient. The remaining mixtures produce very good magnetic performance due to the high filling level. The NBR mixtures were sulphur crosslinked while the HNBR mixtures were peroxide crosslinked. The influence of the crosslinking system on the heating time and the mechanical properties is not analysed in the context of this study

By mixing the hard ferrite particles in rubber, the density of the compound is higher than the density of common elastomer mixtures. Figure (5) shows the density and the hardness of the compounds depending on the filler content.

The curves show a steady density increase with increasing magnetic filler content. With the same filler content, the density values of the HNBR mixture are lower than the NBR mixture, since the density of the magnetic filler FA-700 is about 2.5% higher than the OP-71. Up to hard ferrite mass fraction of 80%, the hardness of the filled NBR compounds increases nearly linear. The increase of the hard ferrite mass content over 80% leads to a strong growth of the hardness. The hardness of the filled HNBR compounds is steadily increasing with increasing hard ferrite mass content.

Figure (6) shows the elongation at break and the tensile strength of the studied compounds depending on the mass fraction of the hard ferrite.

Up to a mass fraction of 55.6% hard ferrite, the elongation at break of the NBR mixture hardly changes. The elongation at break of these mixtures is at about 400%. Increasing the mass fraction of the hard ferrite from 55.6% to 80% decreases the elongation at break from 400% to 300%. From 80% mass fraction of the hard ferrite, the elongation at break sharply decreases with increasing mass content. In the case of HNBR mixtures, the elongation at break shows no clear tendency. The elongation at break of the HNBR mixtures is higher than the NBR mixtures. Up to a hard fer-



Figure 4: Permagraph of company Steingroever [11].

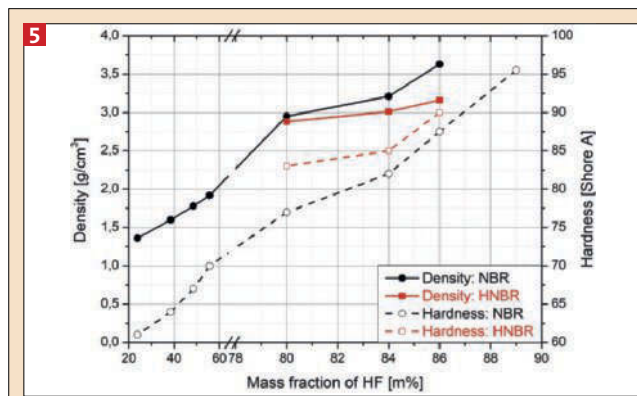


Figure 5: Density and Hardness of the composites as a function of mass fraction of hard ferrite.

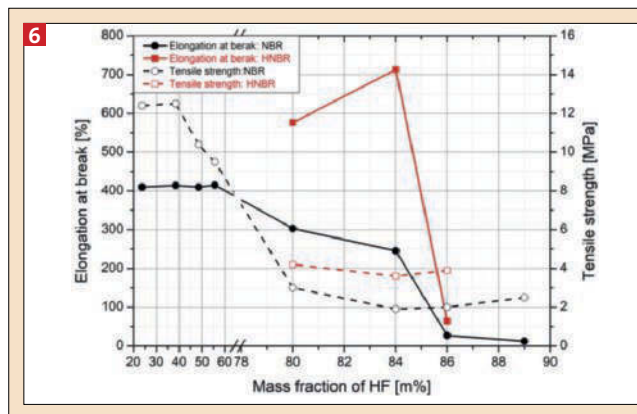


Figure 6: Elongation at break and Tensile strength as a function of mass fraction of hard ferrite.

rite mass fraction of 38.5%, the tensile strength of the NBR compounds is approx. 12 MPa. Between 38.5% and 84% mass fraction of hard ferrite the tensile strength decreases with increasing filler content. Between 84% and 89% mass fraction of hard ferrite the tensile strength increases with increasing filler content. The HNBR compounds show similar tendencies to the NBR mixtures. The tensile strength values of the HNBR mixtures are higher than the NBR mixtures.

Figure (7) shows the remanence and the coercivity of the cured samples without a magnetic field as a function of the filler content.

Increasing the filler content increase the remanence of the analysed compounds. For most industrial applications, only the mixtures with a mass fraction of hard ferrite above 80% are interesting. The particles are orientated by compression moulding. The Form anisotropy can be identified by the good magnetic values. The increase in the filler content leads to a decrease in the coercivity. This behaviour is caused by the mixing process. Increasing the amount of filler in the mixture causes more damage in the network filler structure. Figure (8) shows the remanence and the coercivity of mechanical oriented and mechanical-magnetic oriented NBR mixtures

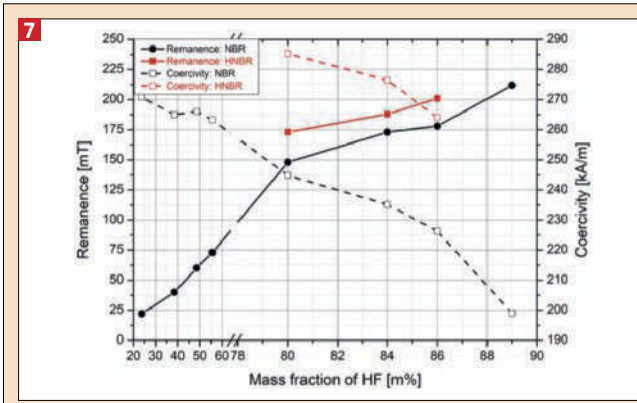


Figure 7: Remanence and the coercivity of the mechanical oriented NBR & HNBR-composites as a function of mass fraction of hard ferrite.

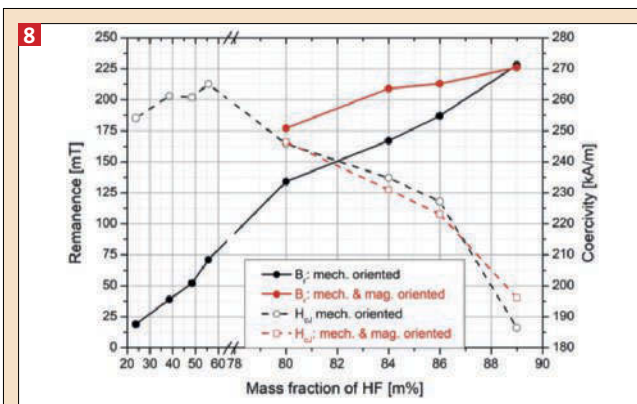


Figure 8: Remanence and the coercivity of the mechanical and mechanical-magnetic oriented NBR-composites as a function of mass fraction of hard ferrite.

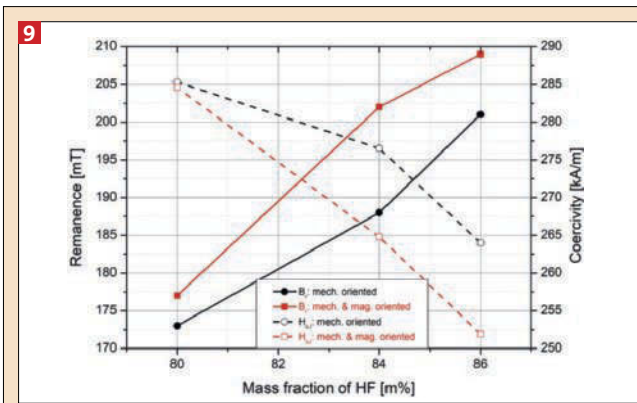


Figure 9: Comparison of the remanence and the coercivity between the mechanical and mechanical magnetically oriented HNBR-composites as a function of mass fraction of hard ferrite.

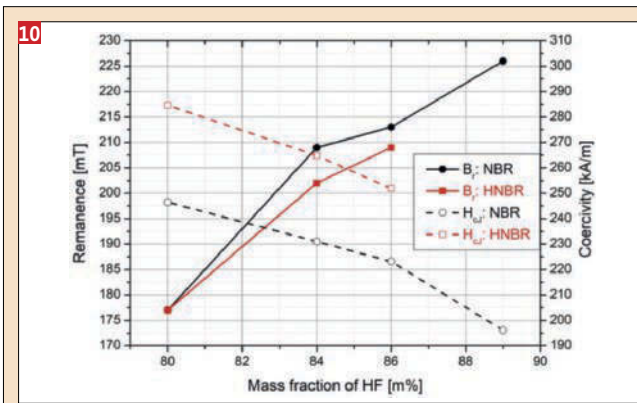


Figure 10: Comparison of the remanence and the coercivity between the mechanical-magnetically oriented NBR and HNBR-composites as a function of mass fraction of hard ferrite.

The mechanical oriented samples show high magnetic values. The remanence of the mechanical-magnetically oriented samples is higher than the mechanical oriented samples. The differences in remanence becomes smaller as the filler content increases. With a filler content of 89%, there are hardly any differences between the mechanical and mechanical-magnetic oriented samples. The coercivity shows no differences, since the samples come from the same mixtures and the pressing process causes no damage of the network filler structure. Figure (9) shows the remanence and the coercivity of mechanical and mechanical-magnetically oriented HNBR-Mixtures.

The remanence of the mechanical magnetically oriented samples is higher than the mechanical oriented samples. The differences in remanence was between 4 and 14 mT. The magnetic orientation of OP-71 causes a very slight increase in remanence compared to FA-700 (Figure (8)). The differences in coercivity are below 6%. These differences can be neglected because the samples originated from the same mixture. Figure (10) shows the remanence and the coercivity of the mechanical-magnetically oriented NBR and HNBR mixtures.

The mixtures show similar magnetic values. The NBR mixtures with the filler FA-700 show higher remanence values. The coercivity decreases with increasing filler content. The coercivity curve of the NBR compounds is below the coercivity curve of the HNBR compounds. These differences can only be induced by the mixing process.

Conclusion

In these studies, mixtures with various base polymers and various magnetic fillers were magnetically and mechanically investigated. In the magnetic investigations, the influence of the form anisotropy and the switched magnetic field on the orientation and thus on the magnetic values was investigated. By increasing the hard ferrite filler content, the elongation at break and tensile strength of the mixtures decreases. The remanence increases with increasing filler content. During the calendaring and pressing process by more than half of the particles present in the mixture are oriented. This orientation is called mechanical orientation or form anisotropy. If a magnetic field is switched on during the vulcanization, the rema-

nence values increase. The increase in remanence due to a magnetic field depends very much on the field strength in the cavity. Presumably, the measured field strength of 300 mT in the cavity was insufficient to align all particles. The coercivity decreases with increasing filler content. This effect is due to the resulting friction between the particles during mixing. The more filler there is in the mixture, the higher the friction between the particles. This friction leads to damage to the particles, which leads directly to a decrease in the coercivity. Vulcanization with no magnetic field has little effect on the coercivity, as the magnetic field does not cause any additional damage to the particles.

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REFERENCES

- [1] Makled M. H., Matsui T.: Magnetic and mechanical characterisation of natural rubber coprecipitated barium ferrite composites at high loading, *Plastics, Rubber and Composites* Vol **38**, 297, 2009.
- [2] Przybylski M., Ślusarek B., Bednarczyk T., Chmiel G.: Magnetic and mechanical properties of strontium ferrite and Nd-Fe-B rubber bonded Permanent magnets, *Acta physica Polonica*, Vol **136** (2019) No.5, 685.
- [3] Schumacher H.: Elastomergebundene Magnetsysteme, *Kunststoffgebundene Dauermagnete*, **165**, 2004.
- [4] Schliesch T.: Kunststoffgebundene Dauermagnete für Antrieb und Sensorik in Kleinmoto-

ren, GMM-IMM Fachtagung, Maing 2001.

- [5] Drummer D., Eimeke S.: Prozessbedingte Vorteile, *Plastverarbeiter* **55**, 88, 2004.
- [6] Umakoshi Y., Yasuda H.Y.: Nondestruction Lifetime Prediction by Magnetic Measurements, *Encyclopedia of Materials: Science and Technology*, 2004.
- [7] Toda Kogyo Corp.: Product data sheet, FA-700
- [8] DOWA F-TEC Co., Ltd.: Product data sheet, P-71
- [9] DOWA F-TEC Co., Ltd.
- [10] Aloui S.: Herstellung, Charakterisierung und mikromechanische Modellierung von magneto-sensitiven Elastomeren, *Doktorarbeit*, 2018
- [11] Wilmes A.: Einfluss des Füllstoffes auf die Verarbeitungs- und Endigenschaften des Compounds, *Technikerarbeit*, 2017

Möglichkeiten der PCR-Einfärbung

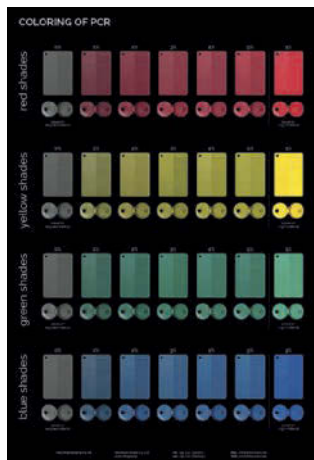


Bild: Finke

Durch individuelle Anpassung von Färbemitteln lassen sich PCR-Materialien in vielen attraktiven Farbtönen einfärben.

SCHLMASTERBATCH FÜR PCR

Die Einfärbung von PCR-Polyolefinen stellt eine große Herausforderung dar. Nicht alles, was sich mit Neumaterial realisieren lässt, ist auch auf gleiche Weise mit PCR-Material umsetzbar. Grund dafür ist vor allem die Eigenfärbung. Durch individuelle Anpassung von Färbemitteln lässt es sich dennoch in vielen attraktiven Farb-

tönen einfärben. Finke, Wuppertal, macht nun am Beispiel eines Polyolefins erfahrbar, was sich bei der Einfärbung von PCR-Material mit Masterbatches erreichen lässt. Dazu wurde ein PCR-Polyolefin, das eine graue deckende Eigenfärbung aufweist, mit einem Farbmaterbatch eingefärbt und zu Spritzgussplaketten verarbeitet. Dabei wurden jeweils ein Rot-, ein Gelb-, ein Grün- und ein Blauton in verschiedenen Masterbatchkonzentrationen eingesetzt, um die Farbwirkung zu demonstrieren. Schon eine Masterbatchkonzentration von 1 Prozent zeigt selbst im dunklen Rezyklat eine deutliche Farbwirkung. Der Vergleich mit einem Neuwareplättchen, das mit dem gleichen Masterbatch bei gleicher Konzentration eingefärbt wurde, offenbart den Unterschied, der sich aus der Eigenfärbung der PCR ergibt. ■

KONTAKT

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Neue flammwidrige thermoplastische Verbundwerkstoffe mit Polyamid 6-Matrix

TEPEX DYNALITE Die endlosfaserverstärkten thermoplastischen Composites der Marke Tepex von Lanxess, Köln, zeigen eine hohe inhärente Flammwidrigkeit. Sie bestehen bereits ohne Flammschutzadditive viele, für typische Anwendungen vorgeschriebene Flammschutztests. In einigen Einsatzfällen ist allerdings oft eine V-0-Einstufung im UL-94-Brandtest obligatorisch. Für solche Fälle hat das Unternehmen drei neue halogenfrei flammgeschützte Varianten mit Polyamid 6-Matrix entwickelt. Tepex Dynalite 102fr-RG600 (x)/ 47% ist mit Rovinggeweben aus Glasfasern verstärkt. Tepex Dynalite 102fr-FG290 zielt auf Anwendungen im Elektro- und Elektronikbereich. Mit einer Verstärkung aus Glasfeingewebe ergibt es hochwertige, gut zu lackierende Oberflächen. Tepex Dynalite 202fr ist mit Carbonfasern verstärkt und für mechanisch extrem beanspruchte Bauteile vorgesehen, wie etwa hochfeste Elektronikgehäuse. Die V-0-



Bildquelle: Lanxess

Brandtest an einem Modellträger. Das schwarze Trägerprofil besteht aus einem der neuen Typen, die Verrippung aus einem orangenen, ebenfalls halogenfrei flammgeschützten Polyamid 6. Die Flammen breiten sich nicht aus, sondern erlöschen nach Entfernen des Brenners, unabhängig davon, ob der Träger flächig oder an den Kanten beflammt wird.

Einstufung nach UL 94 bezieht sich auf Probekörperdicken von 0,5 bis 3,5 Millimeter. Alle Flammschutzpakete entsprechen der EU-Richtlinie Rohs sowie der europäischen Reach-Verordnung. ■

KONTAKT

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Mechanochemical Wear · Oxidation · Hydrogenated styrene-butadiene-rubber

Effect of hydrogenation on the styrene-butadiene rubber (SBR) in terms of the mechanochemical wear behavior was studied focusing on the mechanisms of the mechanochemical reactions during the wear process. The wear test performed in the air and in a nitrogen gas atmosphere indicated that the oxygen molecules accelerated the mechanochemical wear. Oxidation and molecular chain scission during the wear process in the presence of oxygen was proved by Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy and Gel Permeation Chromatography (GPC). The comparison of the wear test results and the analysis of wear particles for the SBR and hydrogenated SBR (H-SBR) compounds indicated that the H-SBR was less sensitive to the mechanochemical reaction, even in the presence of oxygen.

Der Einfluss der Hydrierung von Styrol-Butadien-Kautschuk (SBR) auf das mechanochemische Verschleißverhalten

Mechanochemischer hydrierter Verschleiß · Oxidation · Hydrierter SBR
Der Einfluss der Hydrierung auf Styrol-Butadien-Kautschuk (SBR) wurde im Hinblick auf das mechanochemische Verschleißverhalten untersucht, wobei die Mechanismen mechanochemischer Reaktionen während des Verschleißprozesses im Mittelpunkt standen. Es wurde gezeigt, dass Sauerstoffmoleküle den mechanochemischen Verschleiß beschleunigten. Die Oxidation und die Spaltung von Molekülketten während des Verschleißprozesses wurden mit Röntgen-Nahkanten-Absorptions-Spektroskopie (Near Edge X-ray Absorption Fine Structure NEXAFS Spectroscopy) und Gelpermeationschromatographie (GPC) analysiert. Der Vergleich der Verschleißtestergebnisse und die Analyse der Verschleißpartikel für die SBR- und hydrierten SBR (H-SBR) -Gummischungen zeigten, dass der H-SBR selbst in der Luft mit Sauerstoff weniger empfindlich gegenüber der mechanochemischen Reaktion war.

Figures and Tables: By a kind approval of the authors.

Effect of Molecular Structure on the mechanochemical Wear Behavior of hydrogenated and conventional SBR Rubbers

1. Introduction

Along with the innovative changes in the vehicle usage such as autonomous vehicle and shared driving, for example, supporting the safety driving by keeping the initial tire performance for long term will be increasingly important. In general, tire performance gradually changes due to the decrease of tread depth and the degradation of rubber materials [1]. The shallow tread grooves of worn tires could easily induce aquaplaning, which arises from the difficulty of drainage of a water film from the contact patch. The elastic modulus of the tread compound may increase as a result of degradation of the tread rubber compound, caused by oxidation and loss of plasticizer. The increase in the elastic modulus leads to a reduction of a real contact area between the pavement and the tread surface and, as a consequence, reduces the grip performance of the tire. In addition, higher elastic modulus results in less wear performance, because the tread compound becomes brittle. Therefore, the grip performance in the initial state can be kept for long term by improving the wear performance in terms of rubber degradation.

The effect of degradation on the rubber wear performance has been rarely reported for tire materials. There are some studies regarding the thermal aging effect on the wear performance for nitrile butadiene rubber which is widely used in industries as O-ring, oil seal and so on [2, 3]. However, in order to elucidate the detailed mechanism of the rubber wear performance change in response to the degradation process, it is essential to investigate the attribute of chemical reactions and changes of the molecular structure during the degradation process. In addition, only bulk mechanical properties such as hardness and tensile strength have been commonly analyzed with respect to the effect of degradation on the rubber wear performance while the wear phenomenon occurs at the surface.

Rubber wear can be mainly categorized into three processes as abrasive

wear, fatigue wear, and smearing wear. The last one is induced by the mechanochemical reaction and defined as mechanochemical wear in the present study [4]. The abrasive wear occurs on substrates consist of very sharp edges on the surfaces, which cut the rubber fragments away from the rubber surface. The fatigue wear is caused by crack propagation on blunt substrates. Periodic wave patterns, known as abrasion pattern, can be observed, in particular, on the surface of worn rubber samples which were undergone the fatigue wear. The smearing wear is attributed to local large deformations and high temperatures at the con-

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tact patch. Large deformation triggers molecular chain scission and generates polymer free radicals [5]. Then, the free radicals easily react with oxygen molecules at high temperatures; these processes are called the mechanochemical reaction. As the molecular degradation proceeds at the rubber surface, a sticky layer, composed of degraded polymers, forms on the rubber surface. The formed sticky layer is finally torn off due to the subsequent contact with the substrate. The mechanisms of the abrasive wear and the fatigue wear have been relatively well investigated by some researchers [4, 7-9]. On the other hand, however, there is a relatively less number of reports on the mechanochemical wear. For example, Gent and Pulford performed fundamental studies with a blade equipped to their handmade wear tester [10]. The wear mechanisms of the wear on asphalt pavement or concrete are significantly important for tire industries as a practical case. According to the Persson's theory [15, 16], at an asperity contact region, the rubber surface is subject to large deformation and high, so-called, the flash temperature, which is a result of the frictional heat buildup. Molecular scission potentially occurs due to the large deformations and high temperatures. Therefore, the sticky layer would be formed on the rubber surface under tire operational conditions on the asphalt and concrete. The formation of the sticky layer would affect the surface rubber properties, resulting in changes of wear behavior.

It is natural to assume that oxygen or ozone molecules are playing an important role on the mechanochemical reaction. The effect of the oxygen molecules against the wear mechanism was studied by Gent and Pulford [10]. They reported that the wear rate in an inert gas atmosphere is much lesser than that in the presence of oxygen. From these former researches, it is clarified that oxidation of rubber molecules is related to the mechanochemical wear. However, an analysis on chemical reactions in the molecular scale has not been performed due to the complicated reaction pathway. The oxidation of molecular chains would cause the molecular chain scission as a part of molecular degradation processes. It is known that unsaturated polymers are more likely to oxidize. Thus, a reduced number of the double bonds results in an improvement of the resistance against oxidation. The influence of

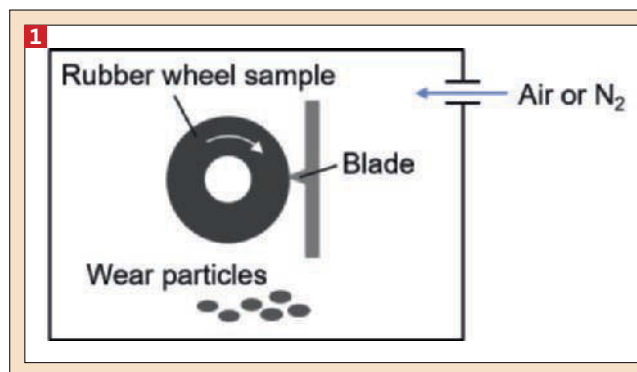


Figure 1: The wear test machine employed in the study. The test machine is installed in an environmental chamber, capable of conducting wear tests in the air or nitrogen gas atmosphere.

the number of unsaturated double bonds on the oxidation has been investigated for hydrogenated polymers [17, 18]. Hydrogenated polymers show a high resistant to not only oxidation [18] but also wear phenomena [19-21]. However, the effect of hydrogenation on the mechanochemical wear in terms of number of unsaturated double bonds has not been reported yet.

In the present study, the mechanism of the mechanochemical reaction and the effect of molecular structure on the mechanochemical wear were investigated for a different level of hydrogenation of styrene-butadiene-rubber (SBR). This kind of study belongs to efforts to improve the wear performance of tires by reducing mechanochemical reactions, which occur at tread rubber surfaces.

2. Experimental

2.1 Materials

Silica filled rubber vulcanizates were prepared using SBR or hydrogenated SBR (H-SBR), both of which are supplied by JSR corporation, Japan. The hydrogenation ratio of H-SBR was optimized for rubber compounding. The mixing process consisted of two steps: (i) 100 parts per hundred rubber (phr) of SBR or H-SBR were mixed with 2.0 phr of stearic acid and 2.0 phr of zinc oxide within a Banbury mixer at 140 °C for 3 minutes. For the filled samples, 60 phr of silica (Ultrasil VN3, Evonik, Germany) and 5.2 phr of silane coupling agent (Bis(triethoxysilylpropyl)polysulfide) were added. (ii) SBR or H-SBR compound was mixed with 1.6 phr of sulfur and 4.0 phr of two kinds of accelerator (N-tert-butyl-2-benzothiazolesulfenamide and 1,3-Diphenylguanidine) by a two roll mixing mill at 80 °C. The obtained green rubber compounds were vulcanized to wheel shaped samples for wear test using a heating press at 170 °C. The diameter of the rubber wheel was 80 mm and the width was 18 mm.

2.2 Wear Test

In order to investigate the wear mechanism at a contact patch, wear particles generated by a home-made wear test machine were collected and analyzed. Figure 1 illustrates the wear test machine. A wheel shaped sample was pushed on a blade with load of 6,5 N and revolved at a constant speed of 60 rpm, which corresponded to a sliding speed of 0.9 km/h. We referred the similar investigation of Gent and Pulford that the wear rate changes in inert gas and this change would be correlated with chemical reaction [10]. The wear test machine was installed in an environmental chamber, which has a transparent acrylic window, and the atmosphere in the chamber was replaced by high purity nitrogen gas. The nitrogen concentration in the chamber was monitored by Nitrogen Concentration Meter (AJX-N2B, AS ONE Corporation, Japan) and was adjusted to be higher than 98%. Wear particles during the wear test were collected with an aluminum container set beneath the blade. At the end of the wear test, the weight loss of the rubber wheel was measured to evaluate the wear rate.

2.3 Structural Analysis

Gel Permeation Chromatography (GPC) measurement was carried out to determine the molecular weight of the wear particles. The collected wear particles and bulk rubber as a reference were dissolved in tetrahydrofuran (THF) immediately after the collection of the particles because the solubility of wear particles in solvent is likely to decrease with increasing an interval time between the particle formation and the solubility test [13]. The extracts from the wear particles and bulk rubber in THF were used for the GPC analysis. The used columns were two Shodex GPC LF-804 (8.0 mmI.D. X 300 mm) and one Waters Styragel Column HR 0.5 (50Å, 5 µm, 7.8 mm X 300

mm) (Showa Denko, Japan). Injection volume was 20 μL and column temperature was 40°C.

Near Edge X-Ray Absorption Fine Structure (NEXAFS) measurements were performed to investigate wear particles on an atomic scale. The data were acquired at the beamline BL17 of SAGA Light Source in Japan, using a grating monochromator. The C K-edge (275~320eV) NEXAFS signals were observed from the wear particle and bulk rubber sample mounted in an ultra-high vacuum chamber. A thin film, the thickness of which is 0.2mm, is prepared for rubber particles and bulk rubber before NEXAFS measurement.

2.4 Quantum Chemical Calculation

Quantum Chemical Calculations (QCC) were implemented to investigate the effect of molecular structure on the strength of the polymer chain scission against mechanical tension and the reactivity with oxygen molecule. The mechanical strength of rubber can be evaluated by the dissociation energy of the carbon-carbon bonds, which is calculated with density-functional theory (DFT). All the DFT calculations in this study were performed using the Becke-Lee-Yang-Parr functional in conjunction with the double numerical polarized basis set, as

implemented in DMol³ [22]. The reactivity with oxygen is demonstrated by conducting molecular dynamics (MD) simulations based on density-functional tight-binding (DFTB) method [23]. The third order of DFTB with the 3ob Slater-Koster parameters was used, and the MD (canonical ensemble) simulations were run at 300 K. BIOVIA Materials Studio [24] was used to perform the calculations and to generate the graphical results.

3. Results and Discussion

3.1 Quantum Chemical Calculation for Investigating Effect of Molecular Structure on Mechanical Strength and Reactivity with Oxygen

Degradation of rubber molecules by the mechanochemical reaction would proceed as a result of the oxidation of rubber molecules following the chain scission by the mechanical force. Therefore, we investigated the effect of types of chemical bonding, between carbon-carbon single bonds and carbon-carbon double bonds on the strength against mechanical chain scission and the reactivity of polymer free radicals with oxygen molecules using the QCC.

Figure 2 (a) shows the bonding energy as a function of the bonding distance between neighbor atoms for the mono-

mer repeating unit of a SBR molecule and a H-SBR molecule. We modeled the difference between them as the difference of bond type; the SBR and the H-SBR consists of a pair of butadiene and styrene, and a pair of butane, as a hydrogenated butadiene, and styrene, respectively. The bonding energy was evaluated for each chemical bond as a potential minimum depth while the bond is stretched until molecular chain scission. The weakest bond was determined based on the bonding energy. The weakest bonding energy for the H-SBR and the SBR was compared as an index of mechanical strength. We focused on the bond energy potential curve of the weakest bond in the SBR, as a reference state. We also analyzed the bond energy potential curve for the H-SBR at the chemical bond, corresponding to the weakest bond in the SBR model. The bonding energy potential curve for the H-SBR molecule was steeper than that of the SBR molecule. A lower minimum energy means higher bonding energy. The bonding energy of the single bond neighboring a double bond becomes weak because of the allylic resonance. The QCC results revealed that the chemical bonds for the H-SBR molecule have nearly an identical energy level without particular weaker bonds, while the SBR molecule has weak chemical bonds which are attributed to the double bonds. This result indicates that the H-SBR molecule is highly resistant to mechanical scission.

The QCC was also implemented to investigate the reactivity of polymer free radicals with the oxygen molecules. Oxidation of a polymer chain was studied with the QCC in such a way that oxygen molecules are approaching to the radical of the polymer chain, as a molecular model after a chain scission. As shown in Figure 2 (b), polymer free radicals of the SBR tended to react with oxygen molecules after chain scission. On the other hand, the polymer free radical of the H-SBR tended to recombine with the other free radical end rather than reacting with the oxygen molecules. The SBR forms more stable polymer free radicals compared with the H-SBR due to the allyl radical resonance. Thus, due to the longer lifetime of free radicals for the SBR, there is enough time to react with the oxygen molecules. In contrast, the lifetime of free radicals for the H-SBR is too short to encounter with the oxygen molecules and immediately bonds the other end of a free radical. As a result, a recombination

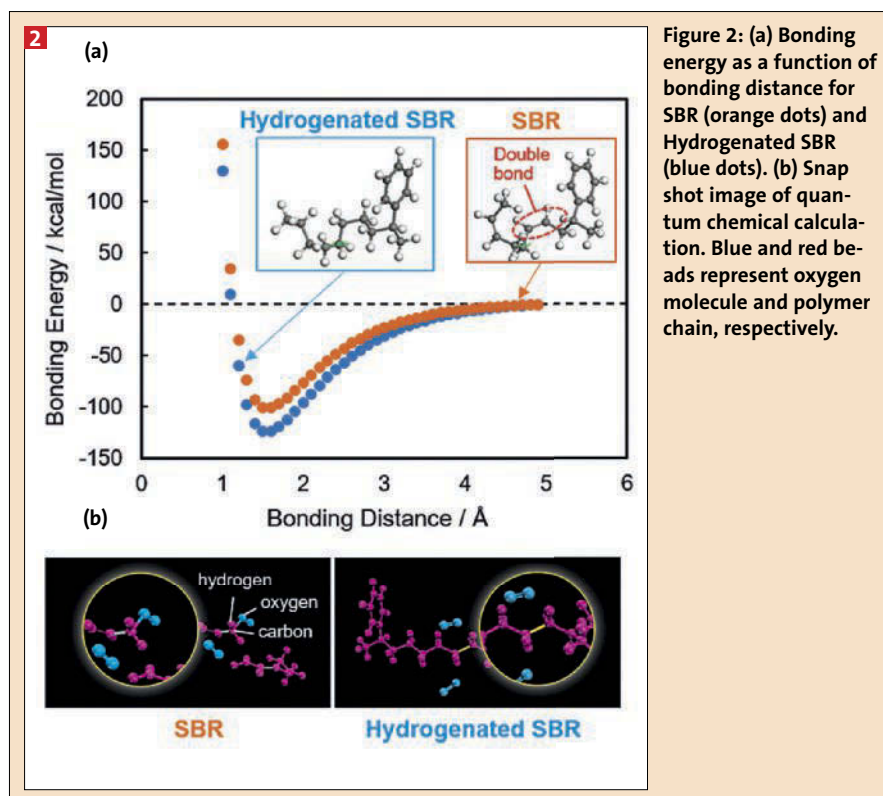


Figure 2: (a) Bonding energy as a function of bonding distance for SBR (orange dots) and Hydrogenated SBR (blue dots). (b) Snap shot image of quantum chemical calculation. Blue and red beads represent oxygen molecule and polymer chain, respectively.

on of two polymer free radicals instantaneously occurred. This calculation result indicates that the H-SBR molecule is insensitive to oxidation as compared to the non-hydrogenated SBR molecule.

3.2 Effect of Oxygen on Rubber Wear Behavior

In general, the molecular degradation is caused by oxidation in combination with heat, UV and so on. It is a plausible hypothesis that the oxygen is considered as an essential part of the wear behavior throughout the mechanochemical reaction, due to the mechanical deformation of the rubber and heat. We performed the wear tests with different oxygen levels to verify the effect of oxidation on the wear mechanism. In order to reduce the oxygen level, the air in the chamber, in which the wear test machine is installed, is replaced by the nitrogen gas. The wear test is performed within the chamber. Figure 3 (a) shows the results of wear tests for the SBR and H-SBR compounds in the air and nitrogen gas atmosphere. For the SBR compound, the wear rate in the nitrogen gas atmosphere was nearly half of the amount of the corresponding wear test in the air. Since the attribute of mechanochemical reaction can be negligible in the nitrogen gas atmosphere, the wear mechanism observed in the nitrogen gas atmosphere would be mainly attributed to the fatigue wear, dominated by the mechanical crack propagation. A ridge pattern was observed on the worn surface of the rubber wheel after testing in the nitrogen gas atmosphere. The presence of ridge patterns also indicates that the observed wear, in the nitrogen gas atmosphere, proceeded mainly through the mechanical crack propagation. Therefore, the difference of the wear rates between the tests in the air and in nitrogen gas atmosphere can be explained by the mechanochemical reactions. Here, it is revealed that nearly half amount of wear in the air is due to the mechanochemical reaction.

The attribute of the mechanochemical reaction can be also found in the difference of appearance of the wear particles. Figure 3 (b) and (c) show the pictures of the wear particles in the air and nitrogen gas atmosphere. The size of the wear particles in air is larger than that in the nitrogen gas atmosphere. The difference of the sizes can be explained by a growing process of the wear particles. The wear particles produced in the air

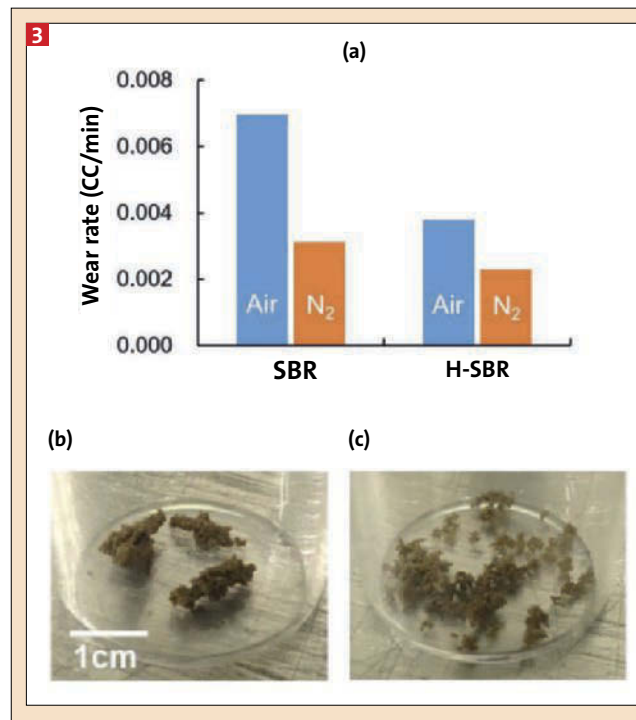


Figure 3: Wear rate of rubber wheel in the air and in nitrogen gas atmosphere for the SBR and H-SBR compounds (a). Pictures of wear particles of the SBR compound in the air (b) and nitrogen gas atmosphere (c), respectively. Wear particles in the air were sticky, and wear particles in nitrogen gas were powdery. The mean size of wear particles in the air is larger than that in nitrogen gas atmosphere.

are sticky and they merge into larger particles during the growing process, whereas the primary particle size produced in the nitrogen gas atmosphere is nearly constant due to a lack of stickiness. The stickiness of the surface is attributed to the degraded rubber molecules, mainly due to the molecular chain scission by the oxidation, indicating a higher attribute of the mechanochemical reaction in the air.

3.3 Effect of Molecular Structure on Mechanochemical Wear

In order to clarify the relationship between the molecular structure and wear mechanism, the wear rate in the different atmospheres is compared between the SBR and H-SBR compounds. The results for the H-SBR compound is also given in Figure 3 (a). The attribute of mechanochemical reaction can be evaluated as the difference of wear rate in the air and nitrogen gas atmosphere. The difference for the H-SBR compound was 40% of the corresponding value of the SBR compound. A smaller difference of the wear rate corresponds to the less attribute from the mechanochemical reaction to the total wear rate. It is concluded that the wear rate for the H-SBR compound proceeds mainly as crack propagation but not as mechanochemical wear. Here we can discuss the effect of molecular structure on the degree of mechanochemical wear in terms of the

number of double bonds. It is noted that total number of double bonds for the H-SBR molecule is much less than that for the SBR molecule.

The wear rate for the H-SBR compound in the air was almost half of the SBR compound while the wear rates in the nitrogen gas atmosphere were on comparable levels. Thus, the difference of the wear rate in the air was mainly attributed to the corresponding level of the mechanochemical wear. This result suggested that the H-SBR compound shows better wear performance due to the less intensive mechanochemical reactions. In the case of the nitrogen gas atmosphere, we expect similar levels of the wear rates for the two cases of the SBR and H-SBR. However, in reality, we find slight differences as shown in Figure 3 (a). Obviously, this slight difference between the wear rates of the H-SBR and SBR compounds in the nitrogen gas atmosphere is reflecting the different microstructures of the investigated polymers and the resulting impact on the mechanical properties of the final compounds. Generally, the fatigue wear behavior is thought to strongly depend on mechanical properties [4, 7-9]. We note that the value of elongation at break of the H-SBR compound was 1.8 times higher than that of the SBR compound. Therefore, the H-SBR compound would show slight lower fatigue wear rate than the SBR compound.

	SBR compound	H-SBR compound
A_N/A_A	0.248	0.466

Table 1: The area ratio (A_N/A_A) of fraction 1 in GPC chart obtained from wear particles of the SBR and H-SBR compounds. The lower the ratio, the larger the difference of the amount of low molecular weight component measured from wear particles between in the air and in nitrogen gas atmosphere.

3.4 Effect of Oxygen on Molecular Weight Distribution of Wear Particles

It is reported that the wear particles, generated by the mechanochemical wear, contain lower molecular weight components as compared to the original polymer before the vulcanization [13]. Thus, the amount of low molecular weight components is assumed to depend on the oxygen level. If the molecular chain scission is accelerated by the presence of oxygen molecules, the amount of low molecular weight components in the nitrogen gas atmosphere would be less than that in the air. Figure 4 shows the GPC chart obtained for the extracts from the original rubber and wear particles of the SBR compound. The wear particles are collected after the wear test running for 1 hour in the air and nitrogen gas atmosphere. The GPC chart revealed that the molecular distribution for each sample, which are the original and wear particles in the air or nitrogen gas atmosphere, consist of two fractions; the fraction 1 corresponds to the molecular weight 50,000 g/mol and the fraction 2 is around 700 g/mol. Comparing the GPC chart of the bulk rubber with those of the wear particles, we can find that the amounts of fraction 1 significantly increases through the wear process, while the amounts of fraction 2 is almost identical in all the samples. Therefore, fraction 1 and fraction 2 would be mainly attributed to low molecular weight polymers generated by the wear process and the

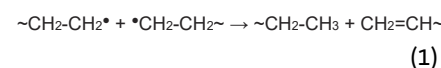
bulk rubber component such as a process oil and accelerator, respectively. The amount of each molecular fraction can be quantitatively evaluated by the area of the GPC chart. The area of the fraction 1 for wear particle in the nitrogen gas atmosphere was smaller than that for the wear particle in the air. This means, at a higher oxygen level, the lower molecular component is larger than that in the nitrogen gas atmosphere. Therefore, the result indicated that oxygen accelerates the degradation of rubber molecule during the wear process, as we assumed.

In order to compare the effect of double bond on the mechanochemical reaction between the SBR and H-SBR, the areas of the GPC charts in the air and nitrogen gas atmosphere are evaluated. Table 1 shows the A_N/A_A of the fraction 1 obtained from the wear particles of the SBR compound and H-SBR compound, where the quantities, A_N and A_A , represent areas of the fraction 1 in the nitrogen gas atmosphere and in the air, respectively. Higher A_N/A_A ratios means higher resistance against the mechanochemical reaction. The A_N/A_A ratio for the H-SBR compound was higher than that for the SBR compound. This result indicated that H-SBR molecules were less affected by the oxygen on the degradation during the wear process. The results about the lower wear rate for the H-SBR compared to the SBR compound (Figure 3 (a)) and, correspondingly, the lower molecular fraction for the H-SBR compound as

shown in table 1, indicated that the formation of lower molecular components increases the wear rate. In addition, the decrease in the amount of carbon-carbon double bonds of the polymer molecules would lead to the reduction of the degradation, resulting in the improvement of mechanochemical wear, vice versa.

3.5 Effect of Molecular Structure on Reactivity with Oxygen

In order to investigate the degradation mechanism during the wear process in a molecular scale, NEXAFS was carried out for the wear particles of the SBR and H-SBR compounds using a synchrotron radiation source. Figure 5 shows the changes in the number of specific functional groups in the SBR and H-SBR compounds after the formation of the wear particles. We focused on the number of carbonyl groups, which represents the degree of the oxidation and reflects the number of the molecular chain scissions. The ordinate represents the number of the functional groups, such as carbon-carbon double bond and so on. A positive value means an increasing number of the functional groups. As shown in Figure 5, the number of carbon-carbon double bonds in the SBR compound decreased during the wear process. It is suggested that the oxidation of the carbon-carbon double bonds through an ozonide formation may result in the reduction in the number of the double bonds. On the other hand, the carbon-carbon double bonds in the H-SBR compound slightly increased. The difference between the H-SBR and SBR compounds could be explained by the difference of free radical reaction pathways. In the case of the SBR it is easy to react with the oxygen molecules after a formation of free radicals as mentioned above, on the basis of the molecular simulation. Contrary, in the case of the H-SBR compound, free radical formation after a molecular scission is likely to induce a recombination or disproportionation which causes an increase of the double bonds as follows:



In terms of the oxidation, the increase in the number of the carbonyl groups after the wear process for the H-SBR is smaller than that for the SBR compound. This result directly revealed that the H-SBR molecule was less reactive with oxygen than the SBR molecules.

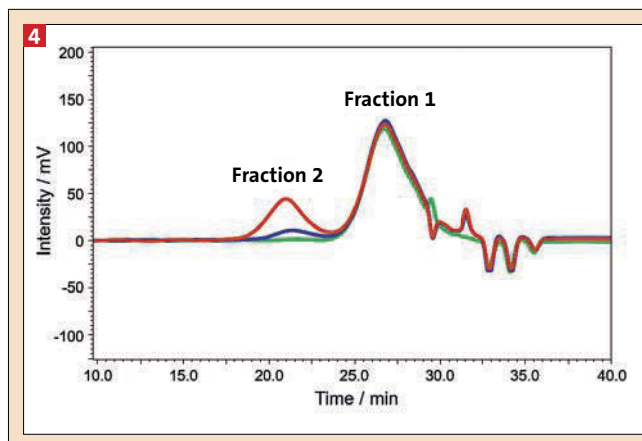


Figure 4: GPC chart obtained for bulk rubber (green line), wear particles in the air (orange line), and wear particles in nitrogen gas atmosphere (blue line) of the SBR compound. Fraction 1 corresponds to low molecular weight SBR and fraction 2 corresponds to low molecular weight additives such as process oil.

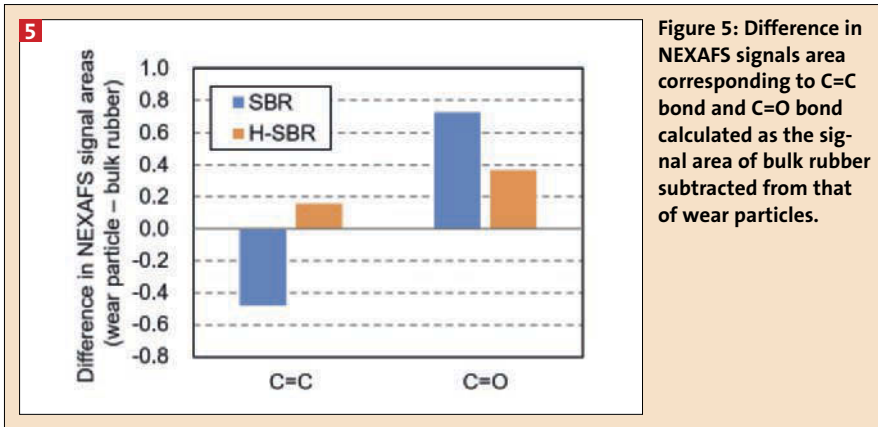


Figure 5: Difference in NEXAFS signals area corresponding to C=C bond and C=O bond calculated as the signal area of bulk rubber subtracted from that of wear particles.

4. Conclusion

In order to improve the wear performance by reducing the mechanochemical reaction, which occurs at a tire tread surface during a wear process, the effect of oxygen on the mechanochemical reaction and the effect of molecular structure on the mechanochemical wear were studied. GPC analysis and NEXAFS results for the wear particles generated in the different oxygen levels revealed that the oxygen molecules accelerate the degradation of rubber molecule during the wear process. The wear test for the SBR compound performed in the air and nitrogen gas atmosphere indicated that the mechanochemical wear dominates more than half of the total wear amount in the air. The effect of oxygen on the mechanochemical reactions was much smaller for the H-SBR molecule, whose carbon-carbon double bonds were highly hydrogenated. The wear amount of the H-SBR compound is about half of the wear amount of the non-hydrogenated SBR compound. When designing tire tread

compounds with polymer molecules having less amount of carbon-carbon double bonds, a further upgrading of wear performance is expected through the control of mechanochemical reactions.

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6. References

- [1] K. R. S. Wright, T. R. Botha, P. S. Els, *J. Terramechanics*, **84**, **21** (2019).
- [2] C. L. Dong, C. Q. Yuan, X. Q. Bai, X. P. Yan, Z. Peng, *Wear*, **322**, **226** (2015).
- [3] B. Jiang, X. Jia, Z. Wang, T. Wang, F. Guo, Y. Wang, *Tribol. Lett.*, **67**, **86** (2019).
- [4] B. N. J. Persson, *J. Phys.: Condens. Matter*, **21**, **485001** (2009).
- [5] A. Ahagon, Y. Kirino, *Rubber Chem. Technol.*, **79**, **641** (2006).
- [6] K. A. Grosch, A. Schallamach, *Transaction of the Institution of the Rubber Industry*, **41**, **80** (1965). B.
- [7] A.G. Thomas, *J. Polym. Sci., Symp.*, **48**, **145** (1974).
- [8] Y. Fukahori, H. Yamazaki, *Wear*, **171**, **195** (1994).
- [9] A. Emami, S. Khaleghian, *Tribol. Int.*, **136**, **487** (2019).
- [10] A. N. Gent, C. T. R. Pulford, *J. Appl. Polym. Sci.*, **28**, **943** (1983).
- [11] C. T. R. Pulford, *J. Appl. Polym. Sci.*, **28**, **709** (1983).
- [12] A. P. Rudakov, E. V. Kuvshinskii, *Rubber Chem. Technol.*, **37**, **291** (1964).
- [13] Wu, Guangchang, Ph.D Thesis, Queen Mary University of London, 2016.
- [14] A. Ahagon, Y. Kirino, *Rubber Chem. Technol.*, **80**, **265** (2007).
- [15] B. N. J. Persson, *J. Phys.: Condens. Matter*, **18**, **7789-7823** (2006).
- [16] B. Lorenz, B.N.J. Persson, G. Fortunato, M. Giustiniano and F. Baldoni, *J. Phys.: Condens. Matter*, **25**, **095007** (2013).
- [17] M. D. Sarkar, P. G. Mukunda, P. P. De, A. K. Bhowmick, *Rubber Chem. Technol.*, **70**, **855** (1997).
- [18] D. K. Parker, R. F. Roberts, H. W. Schiessl, *Rubber Chem. Technol.*, **67**, **288** (1994).
- [19] P. Thavamani, A. K. Bhowmick, *J. Mater. Sci.*, **28**, **1351** (1993).
- [20] E. Padenko, P. Berki, B. Wetzels, J. Karger-Kocsis, *J. Reinf. Plast. Compos.*, **35**, **81**, (2016).
- [21] S. Inoue, T. Nishio, *J. Appl. Polym. Sci.*, **103**, **3957** (2007).
- [22] B. Delley, *J. Chem. Phys.*, **92**, **508** (1990).
- [23] Th. Frauenheim et al., *J. Phys. Cond. Matter*, **14**, **3015** (2002).
- [24] Ver. 2018, BIOVIA Materials Studio, San Diego, CA, USA.

Neues Portfolio an Wire-and-Cable-Compounds

WIRE & CABLE Hexpol, Malmö, Schweden, hat ein umfassendes Portfolio an Wire-and-Cable-Verbindungen, basierend auf Hochleistungsmaterialien wie VMQ, fortschrittlichen Kautschuktechnologien, Additiven und TPE, entwickelt. Das Portfolio umfasst Silikonkautschukmaterialien, CPE-, EPDM-, NBR + PVC-, CR-Kautschukmischungen, Pigment- und Additiv-Masterbatch sowie Thermoplast-, EVA-, TPE- und TPV-Technologien. Typische Eigen-

schaften sind halogenfreie Flammhemmung, geringer Rauchgehalt und geringe Toxizität. Die Materialien sind Rohs-, SVHC- und Reach-konform mit Qualitäten, die den internationalen Kabelnormen wie EN50363 oder IEC 60811 entsprechen. Kabelfüllmasse, Isolierung und Ummantelungsmaterialien gehören zum Portfolio. Zu den Anwendungsbereichen gehören Räume mit teuren oder sensiblen Geräten in Krankenhäusern, Flughäfen,

Zug- und Verkehrsknotenpunkten, Kommunikationsbörsen, Stromerzeugungsanlagen und Offshore-Plattformen. Mit verbesserten thermomechanischen Eigenschaften stehen für die Telekommunikation wieder Qualitäten zur Verfügung und elektrische Kabel von Hoch- zu Niederspannung. ■

KONTAKT

Hexpol, Malmö, Schweden,
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Bild: Hexpol

Die neuen Compounds basieren auf Hochleistungsmaterialien wie VMQ, fortschrittlichen Kautschuktechnologien, Additiven und TPE.

Nonlinear rheology · FT Rheology, recovered carbon black · pyrolysis · surface area · filler structure

Colloidal properties of different carbon black (CB) grades represented by their structure and specific surface area were characterized before and after pyrolysis. Additionally, rubber compounds produced with these fillers were investigated by Fourier Transform (FT) Rheology, since the nonlinear, rheological behavior of filled elastomers under specific conditions is associated with the filler-polymer interaction. For comparison, some commercially available, recovered carbon blacks (rCB) were analyzed. While pyrolysis only slightly affects the structure and the specific surface area of carbon blacks, its influence on the nonlinear material behavior is significant. Different rCB grades can be differentiated by FT Rheology with a high level of sensitivity.

Fourier Transform Rheologie – ein innovatives Werkzeug für die Charakterisierung von rückgewonnenem Ruß

Nichtlineare Rheologie · FT-Rheologie · Ruß · Pyrolyse · Füllstoffoberfläche · Füllstoffstruktur

Kolloidale Eigenschaften verschiedener Ruße wurden anhand ihrer Struktur und ihrer spezifischen Oberfläche vor und nach der Pyrolyse untersucht. Zusätzlich wurden Kautschukmischungen, die mit diesen Füllstoffen hergestellt wurden, mittels FT-Rheologie analysiert, da das nichtlineare, rheologische Verhalten der Mischungen unter bestimmten Bedingungen mit der Füllstoff-Polymer-Wechselwirkung in Zusammenhang steht. Zum Vergleich wurden kommerziell verfügbare, wiedergewonnene Ruße (rCB) analysiert. Während die Pyrolyse nur geringfügig die Struktur und spezifische Oberfläche der Ruße beeinflusst, ist ihr Einfluss auf die Nichtlinearität des Materialverhaltens signifikant. Unterschiedliche rCBs lassen sich durch die FT-Rheologie mit hoher Sensitivität differenzieren.

Figures and Tables:
By a kind approval of the authors

Fourier Transform Rheology – an innovative Tool for the Characterization of recovered Carbon Black

Introduction

In recent years, the accelerated growth in the world's population and the global economy has led to a rapid development of the automobile industry and a remarkable increase of vehicle numbers worldwide resulting in environmental discussions and public concerns over End of Life Tire (ELT) disposal. Today, nearly 1.5 billion tires are being disposed of each year and half of them are discarded in landfills without any treatment [1, 2]. The disposal of waste tires has become a global problem due to their large volume, durability, mixed composition induced by the presence of multiple components and the potentially strong environmental impact as some components or degradation products are toxic [3 - 5]. In addition to that, there is a high demand of increasing the share of sustainable and recycled materials in the automobile industry driven by customers and potential future legislation. Therefore, there is a mandatory need for sustainable recycling technologies to reuse or reclaim materials from tires. Recycling technologies for tires can be divided into the three categories of: civil engineering, energy recovery and material recovery [6].

In most cases, material recovery technologies have been based on the so-called Life Cycle Assessment (LCA) in recent years [7, 8]. The pyrolysis of ELT is a process that does not only combine environmental challenges, but also allows the recovery of valuable products like oils and carbon fillers for elastomeric materials [9]. Pyrolysis is a thermal decomposition method that comprises irreversible chemical reactions created by heat in an inert, oxygen free atmosphere leading to the rupture of the molecule structure and the subsequent degrading of the material [10, 11].

Filler materials such as rCB generated from this process are known to consist of the original carbon black, carbonaceous deposits and inorganic rubber compounding ingredients [9]. Currently, vir-

gin carbon black (CB) is mainly used as a reinforcing filler in rubber products. A high energy input and the use of fossil fuels are necessary to produce CB, which inadvertently leads to a high output of carbon dioxide.

While colloidal properties indicate that rCB should be a reinforcing filler, its in-rubber performance is only semi-reinforcing. This incongruence has been associated with the formation of carbonaceous residues during the pyrolysis process [9]. However, there are just a few works dedicated to the characterization of rCB [9, 12, 13]. For the application of rCB in new rubber compounds, it is important to have simple and accessible test methods to characterize the rCB in detail and to set precise specifications.

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Therefore, in this research paper, colloidal properties of carbon black are analyzed and compared to the influence of carbon black on the nonlinear behavior of a standardized compound using Fourier Transform Rheology with the aim to propose a sensitive test method to evaluate the reinforcing capabilities of commercially available rCB.

Experimental Section

Materials

Seven different grades of carbon black (CB) classified according to ASTM D1765 [14] were used to elucidate both their colloidal properties and their influence on the nonlinearity of a standardized rubber compound: N121, N220, N326, N330, N339, N550, N660. Additionally, these carbon blacks were recovered from the compound via lab pyrolysis and compared to four industrially recovered carbon blacks (rCB1, rCB2, rCB3, rCB4) produced from ELTs via a conventional pyrolysis process. The standard carbon blacks recovered by lab pyrolysis are identified with the ending “_P”, e.g. N121_P. The standardized compound corresponds to the one in ASTM D3192 [15] (see Table 1).

The standardized compound was mixed in a two-step mixing process with a 2 liters internal mixer (Harburg-Freudenberger, Germany) using a tangential two-wing rotor in analogy to ASTM D3192 [15]. In the first step, all ingredients but the vulcanization system were mixed for 7 min. After adding the vulcanization system in the second step, the compound was mixed for 3 more minutes.

To recover the carbon blacks, the experimental compounds were vulcanized for 30 minutes at 140 °C and subsequently pyrolyzed. The pyrolysis was conducted in a compact horizontal split tube furnace (Carbolite Gero GmbH & Co. KG, Germany). The furnace was previously rinsed with nitrogen for 1 h. Afterwards, the samples were pyrolyzed for 2 h at 550 °C and cooled to room temperature in a nitrogen atmosphere.

Experimental methods

As representative of colloidal properties of carbon black, the filler structure and the specific surface area were selected.

In order to obtain structure parameters, void volume as a function of pressure was measured using the Void Volume Tester (Brabender GmbH & Co. KG, Germany). The void-volume test procedure

1 Detailed recipe of the test compound in analogy to ASTM D3192 [15]	
Ingredient	Amount [phr ^c]
Natural Rubber	100
Carbon Black	50
DTPD ^a	1
6PPD ^b	1,5
TMQ ^c	1
Wax	2,5
Zinc Oxide	3
Stearic Acid	2
TBBS ^d	0,75
Sulfur	1,4

a N,N'-Di-(p-tolyl)-p-phenylenediamine
 b N'-phenyl-p-phenylenediamine
 c poly(1,2-dihydro-2,2,4-trimethyl-quinoline)
 d N-t-Butyl-2-Benzothiazole Sulfenamide
 e parts per hundred rubber, non-SI unit

has been standardized in ASTM D7854 [16] and defined as the measure of the intra-aggregate void space or occluded volume within the primary structure of carbon black, and therefore, is an innovative descriptor of the filler structure. The result reported is the void volume value given in [cm³/100 g] at pressures of 50 MPa, 75 MPa and 100 MPa and is based on the geometrical mean of the applied and transmitted pressures. [16, 17]. In this work, the void volume at 100 MPa is used as structure descriptor.

The specific surface area was determined by nitrogen adsorption in analogy to ASTM D6556 [18] with a BET-analyser BELSORP-mini II (BEL Japan Inc., Japan).

Rheological measurements were carried out on an SIS V50 Rubber Process Analyzer (Scarabaeus GmbH, Germany, now: TA Instruments, USA). Strain sweeps in a range from $\gamma = 0.1$ to 1.2 were performed at a temperature of 80 °C and a frequency of 0.2 Hz. The stress data were analyzed by Fourier transformation, and the ratio of the 3rd harmonic to the 1st harmonic, $I_{3/1}$, was plotted as a function of the strain amplitude. As stated by Schwab et al. [19, 20], the influence of the CB fillers on the nonlinearity of rubber can be observed in a range of the strain amplitude from $\gamma = 0.2$ to 0.4. Therefore, the evaluation has been preferably performed at the strain amplitude of $\gamma = 0.32$.

In rheology, the viscoelastic properties of uncured rubber are typically characterized by oscillatory shear. The material response is linear viscoelastic for small

oscillatory shear amplitudes (SAOS) and nonlinear viscoelastic at large oscillatory shear amplitudes (LAOS) [21]. Rubber compounds filled with carbon black already show nonlinear viscoelastic behavior at relatively low strains [22]. The decrease of the shear modulus with increasing strain amplitude, known as Payne effect, is a common effect of nonlinear behavior [23]. It is the consequence of the breakdown of the filler-filler network structure in the rubber compound at large strain amplitudes [24, 25].

FT Rheology can be used to analyze and quantify the nonlinear viscoelastic behavior of polymers [21]. Applying an oscillatory strain $\gamma(t) = \gamma_0 e^{i(\omega_0 t)}$ results in a time-dependent stress response $\sigma(t) = \sigma(t + \frac{2\pi}{\omega_0})$. The stress response is periodic, but it is only a single sinusoidal signal if the material behavior is linear viscoelastic [19, 26]. If the material behavior is nonlinear viscoelastic, the shear modulus G^* becomes a function of the applied strain. In this case, G^* can be described by a Taylor series of the strain with even exponents (Equation 1). The precondition for this procedure is that the material is isotropic, like rubber, and the applied strain is symmetric, as in an oscillatory shear deformation [20, 27].

$$\sigma(t) = [G_0 + G_1\gamma(t)^2 + G_2\gamma(t)^4 + \dots]\gamma(t) \quad (1)$$

$$= G_0\gamma_0 e^{i(\omega_0 t)} + G_1\gamma_0^3 e^{i(3\omega_0 t)} + G_2\gamma_0^5 e^{i(5\omega_0 t)} + \dots \quad (2)$$

where the moduli G_0, G_1, \dots are complex numbers.

If the material's behavior is nonlinear, the intensity spectrum of the Fourier transform of $\sigma(t)$ shows peaks at ω_0 and the odd multiples $n\omega_0$ with $n = 3, 5, 7, \dots$ according to Equation 2 [19].

The intensity of the 3rd harmonic $I(3\omega_0)$ is the most intense for a specific strain amplitude γ at the frequency ω_0 . For generating a useful nonlinearity parameter, $I(3\omega_0)$ is normalized to the intensity of the 1st harmonic $I(\omega_0)$, as a relative third harmonic contribution.

$$I_{3/1} \equiv \frac{I(3\omega_0)}{I(\omega_0)} \quad (3)$$

This normalization allows a comparison between different measurements. It can also compensate for systematic experimental deviations [19]. $I_{3/1}$ describes the nonlinear viscoelastic behavior of a rubber material. [20, 28]

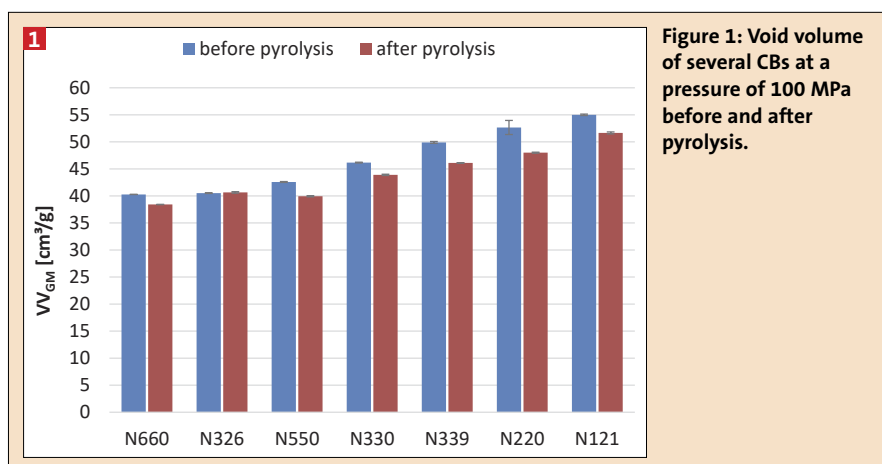


Figure 1: Void volume of several CBs at a pressure of 100 MPa before and after pyrolysis.

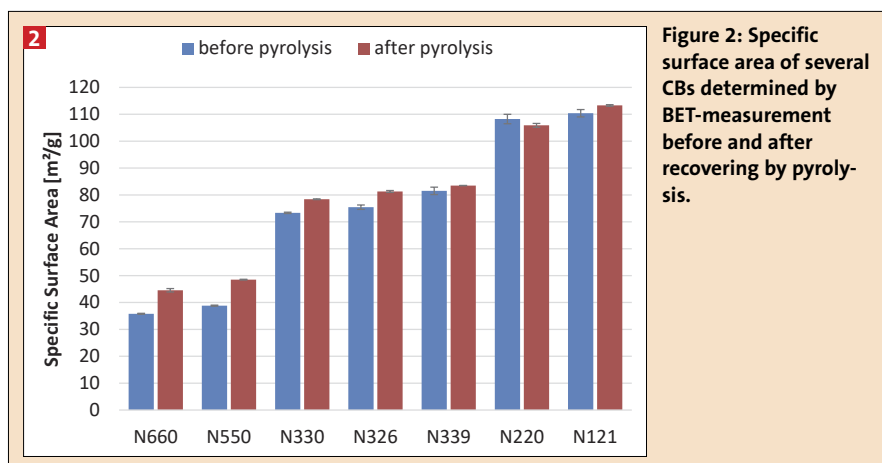


Figure 2: Specific surface area of several CBs determined by BET-measurement before and after recovering by pyrolysis.

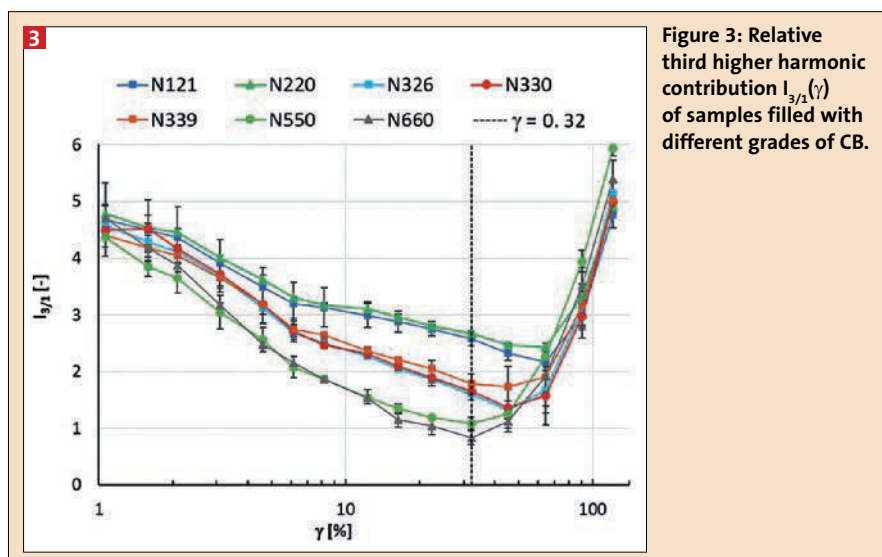


Figure 3: Relative third higher harmonic contribution $I_{3/1}(\gamma)$ of samples filled with different grades of CB.

Results and Discussion

Structure and specific surface area

The original structure and the specific surface area of several carbon blacks were investigated and compared to the structure and specific surface area after recovering them via pyrolysis.

Figure 1 shows the influence of pyrolysis on the structure of carbon black, as represented by the void-volume. The structure of all carbon black types is reduced by around 5 % after pyrolysis. This reduction of the structure is not caused by pyrolysis, but probably triggered by

the mixing process. During mixing, shear forces can slightly affect the structure of the CB aggregates, especially in compounds with high amounts of filler [29].

Figure 2 shows that the specific surface area is slightly influenced by pyrolysis. The increase of the specific surface area is probably due to ashes, vulcanization agents and the agents used to pelletize the CB. During pyrolysis, they can be decomposed, and therefore, seemingly increase the surface area of the fillers. The specific surface area of the CB itself is not affected by pyrolysis. [30, 31].

The methods of evaluation discussed above are known to describe the performance of carbon black taking into account structure and specific surface area, but they do not differentiate between virgin and pyrolyzed carbon blacks. On the other hand, pyrolysis is used to produce rCB. Nevertheless, colloidal parameters of rCB are not related to performance, as observed in the case of the pyrolysis of standard carbon blacks [30].

Fourier Transform Rheology Before pyrolysis

The compounds investigated before and after pyrolysis show a different nonlinear, rheological behavior, especially in the range of a strain amplitude from $\gamma = 0.2$ to 0.4 as shown in Figure 3. The only difference between these compounds is the CB grade used. At strain amplitudes lower than $\gamma = 0.1$, the signal-to-noise ratio is predominant [19, 20]. Above a strain amplitude of $\gamma = 0.5$, the nonlinearity of the polymer prevails [19], so that all compounds investigated nearly show the same curve progression at high levels of strain. In order to analyze the effect of carbon black on the nonlinear behavior of the compounds, only the normalized third harmonic $I_{3/1}$ at a strain amplitude of $\gamma = 0.32$ will be considered in the following. For purposes of simplicity, this value is called $I_{3/1}(\gamma = 0.32)$.

$I_{3/1}(\gamma = 0.32)$ of the samples filled with the CB grades concedes the classification into three groups of CB: N121 and N220 form the first group, N326, N330 and N339 the second one and N550 and N660 the third one. The nonlinearity parameter $I_{3/1}$ decreases from the first to the third group. As expected, the nonlinear behavior of the compound coincides with the well-known reinforcing capabilities of these carbon blacks. This fact has been attributed to the interactions between the filler and the polymer which result in both a higher level of

nonlinearity and a stronger reinforcement of the rubber compound.

In Figure 4 (left), the values of $I_{3/1}(\gamma = 0.32)$ for samples filled with different grades of CB are plotted against the specific surface area of the CB used in the sample. On a logarithmic scale, there is a linear correlation between the nonlinear viscoelastic behavior and the specific surface area. In Figure 4 (right), $I_{3/1}(\gamma = 0.32)$ is plotted against the void volume at a pressure of 100 MPa representing the structure of the CB grade. In analogy, a linear trend is evident on a logarithmic scale between the nonlinear viscoelastic behavior of the compound and the structure of the CB.

As already known, the specific surface area and the structure of a CB can be used to predict the reinforcing effect of carbon black in a compound. This fact can be confirmed by the analogous, nonlinear viscoelastic behavior of the corresponding rubber compound.

After pyrolysis

Pyrolysis does not substantially affect neither the specific surface area nor the structure of carbon black. In contrast, the nonlinear viscoelastic behavior of the compounds is significantly reduced after recovering carbon black from vulcanized rubber via lab pyrolysis. Figure 5 shows $I_{3/1}(\gamma = 0.32)$ for the compounds using virgin CB compared to those using the same CB grade recovered via lab pyrolysis. The decrease of $I_{3/1}(\gamma = 0.32)$ is significant: after pyrolysis, all CB grades investigated show a reduction of the induced nonlinear viscoelastic behavior on the compound.

On the left side, Figure 6 shows $I_{3/1}(\gamma = 0.32)$ of the samples filled with different grades of CB recovered via lab pyrolysis plotted against their specific surface area. After pyrolysis, the nonlinear viscoelastic behavior is significantly reduced, but the corresponding specific surface area has hardly changed. The changes in nonlinearity are not reflected by the specific surface area. The linear trend before pyrolysis has been kept in the chart for orientation purposes only.

The right side of Figure 6 shows $I_{3/1}(\gamma = 0.32)$ plotted against the structure of the CB grades recovered via lab pyrolysis. After pyrolysis, the nonlinearity is significantly reduced while the void volume of the carbon blacks is only slightly shifted to lower values due to the repeated compound mixing after lab pyrolysis. Again, the linear trend be-

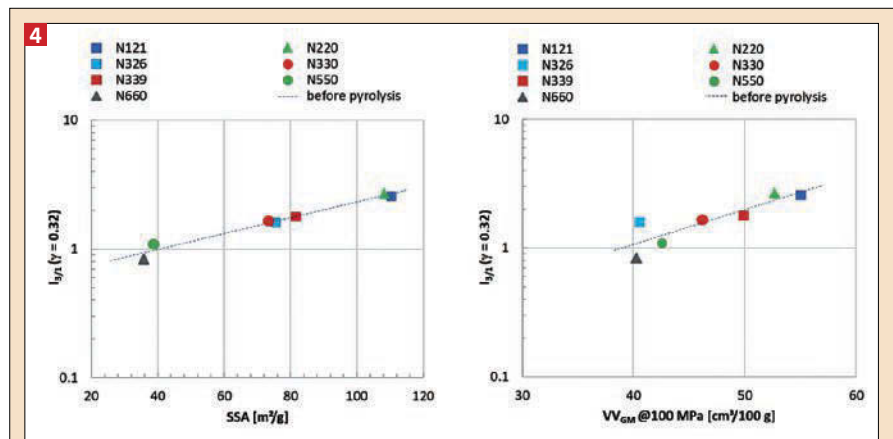


Figure 4: Nonlinearity parameter $I_{3/1}(\gamma = 0.32)$ of samples filled with different grades of CB against their specific surface area (left) and their structure (right), respectively.

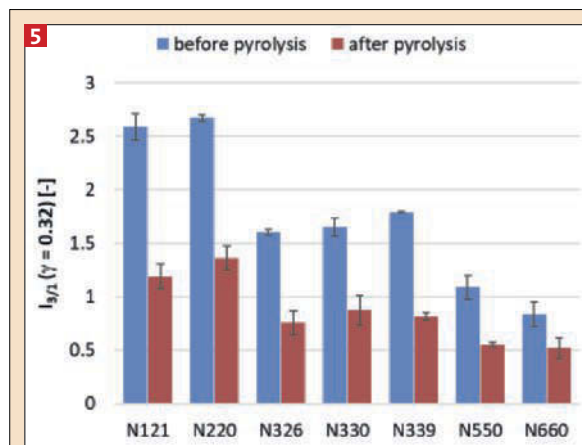


Figure 5: Nonlinearity parameter $I_{3/1}(\gamma = 0.32)$ of samples filled with different grades of CB before and after recovering by pyrolysis.

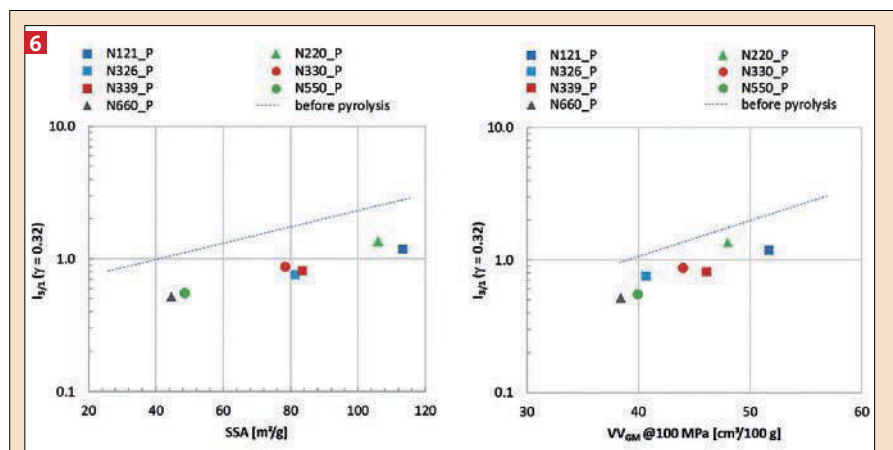


Figure 6: Nonlinearity parameter $I_{3/1}(\gamma = 0.32)$ of samples filled with different grades of CB recovered via pyrolysis against their specific surface area (left) and their structure (right), respectively. The linear trends from Fig. 4 (before pyrolysis) are included for comparison.

fore pyrolysis has been included in the chart as reference.

The colloidal properties of the carbon blacks recovered via lab pyrolysis do not reflect the changes in nonlinear behavior of the respective compounds, as they are

not indicative of reinforcing properties of pyrolyzed CB either, as suggested by Norris et al. [12]

Presumably, carbonaceous deposits cause the reduction of $I_{3/1}$. Roy et al. [31, 32] proved that carbonaceous deposits

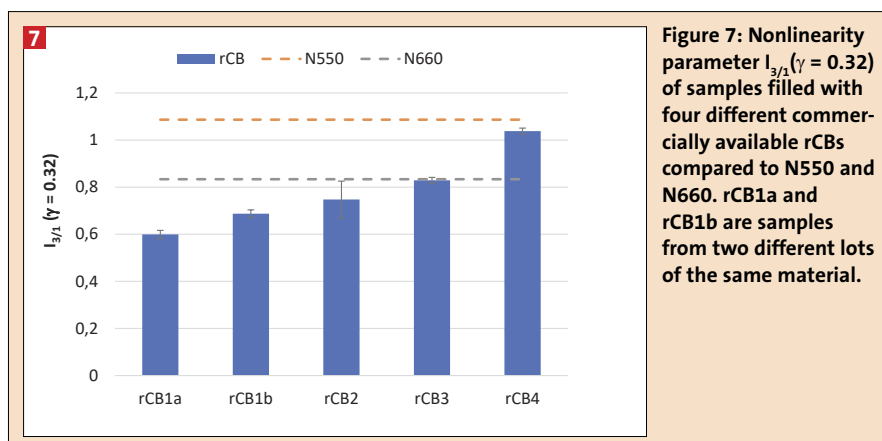


Figure 7: Nonlinearity parameter $I_{3/1} (\gamma = 0.32)$ of samples filled with four different commercially available rCBs compared to N550 and N660. rCB1a and rCB1b are samples from two different lots of the same material.

are formed on the CB surface during pyrolysis. They smooth the CB surface and cover a portion of the energetically active sites on the surface.

Recovered Carbon Black

For comparison, rubber compounds containing commercially available rCBs based on the same recipe shown in Tab. 1 were also investigated. In contrast to the CBs recovered via lab pyrolysis, they consist of a mixture of different carbon black grades and inorganic ashes depending on the specific feed stock. Figure 7 shows $I_{3/1} (\gamma = 0.32)$ for rubber compounds containing four different commercially available rCBs. Two different lots of the material rCB1 were analyzed (rCB1a, rCB1b).

Differences between the rCBs become evident by FT Rheology. It is expected that the different levels of nonlinear behavior of the rubber compounds correlate with a different level of interaction between the fillers and the polymers. The reason for these differences might be found in the composition of the feed stock and the conditions used to produce the specific rCB, like temperature and pressure during pyrolysis, which might affect the formation of carbonaceous residues [31]. Higher amounts of carbonaceous residues probably reduce $I_{3/1} (\gamma = 0.32)$ by hindering filler-polymer interaction. The differences between the two batches of the same rCB1 in Fig. 7 are probably caused by variations of the feed stock composition.

As reference values, Figure 7 also shows $I_{3/1} (\gamma = 0.32)$ of compounds with virgin N550 and N660. These are the virgin carbon blacks with the lowest influence on the nonlinear behavior of the compound investigated here. The influence of most rCBs on nonlinearity is even

lower than the one of virgin N660. Only rCB4 reaches a value of $I_{3/1} (\gamma = 0.32)$ comparable to the one of N550.

Because of the high sensitivity of FT Rheology, quantifying slight variations between different rCBs becomes accessible and assessments of performance regarding the partial replacement of virgin carbon blacks might become possible.

Conclusions

Different CB grades were recovered from compounds via lab pyrolysis and compared to their virgin state. The structure and specific surface area of the CBs before and after pyrolysis have been studied in this work. Additionally, their influence on the nonlinear behavior of a standardized compound has been investigated by FT Rheology and compared to commercially available rCBs.

The pyrolysis of carbon black only has a negligible influence on its specific surface area and structure. In contrast, the influence of the pyrolyzed CB on the nonlinear behavior of the compound is clearly affected: the $I_{3/1} (\gamma = 0.32)$ values decrease after pyrolysis. Carbonaceous residues which hinder the filler-polymer interaction are probably responsible for the decrease in nonlinearity.

FT Rheology is a highly sensitive, suitable technique to evaluate the reinforcing capabilities of rCBs independently of the conventional techniques used for the characterization of colloidal properties of carbon black.

References

[1] Thomas, B. S. u. Gupta, R. C.: A comprehensive review on the applications of waste tire rubber in cement concrete. *Renewable and Sustainable Energy Reviews* **54** (2016), 1323.

[2] Gómez-Hernández, R., Panecatí-Bernal, Y. u. Méndez-Rojas, M. Á.: High yield and simple one-step production of carbon black nanoparticles from waste tires. *Heliyon* **5** (2019) 7, e02139.

[3] Liu, H., Wang, X., Jiao, Y. u. Sha, T.: Experimental Investigation of the Mechanical and Durability Properties of Crumb Rubber Concrete. *Materials* (Basel, Switzerland) **9** (2016) 3, 172.

[4] Bockstal, L., Berchem, T., Schmetz, Q. u. Richel, A.: Devulcanisation and reclaiming of tires and rubber by physical and chemical processes: A review. *Journal of Cleaner Production* **236** (2019) 117574.

[5] WBCSD u. Tire Industry Project: Global ELT Management – a global state of knowledge on regulation, management systems, impacts of recovery and technologies. 2019.

[6] Tillman, A.-M.: Significance of decision-making for LCA methodology. *Environmental Impact Assessment Review* **20** (2000) 1, 113.

[7] Corti, A. u. Lombardi, L.: End life tyres: Alternative final disposal processes compared by LCA. *Energy* **29** (2004) 12, 2089.

[8] Hoang, A. T., Nguyen, T. H. u. Nguyen, H. P.: Scrap tire pyrolysis as a potential strategy for waste management pathway: a review. *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects* (2020), 1.

[9] Darmstadt, H., Roy, C. u. Kaliaguine, S.: Characterization of pyrolytic carbon blacks from commercial tire pyrolysis plants. *Carbon* **33** (1995) 10, S. 1449.

[10] Antoniou, N. u. Zabaniotou, A.: Features of an efficient and environmentally attractive used tyres pyrolysis with energy and material recovery. *Renewable and Sustainable Energy Reviews* **20** (2013), 539.

[11] dos Santos, R. G., Rocha, C. L., Felipe, F. L. S., Cezario, F. T., Correia, P. J. u. Rezaei-Gomari, S.: Tire waste management: an overview from chemical compounding to the pyrolysis-derived fuels. *Journal of Material Cycles and Waste Management* **22** (2020) 3, 628.

[12] Norris, C. J., Hale, M. u. Bennett, M.: Pyrolytic carbon: factors controlling in-rubber performance. *Plastics, Rubber and Composites* **43** (2014) 8, S. 245.

[13] Cataldo, F.: On the characterisation of carbon black from tire pyrolysis. *Fullerenes Nanotubes and Carbon Nanostructures* **28** (2020) 5, 368.

[14] ASTM D1765. Classification System for Carbon Blacks Used in Rubber Products. ASTM International, West Conshohocken, PA. doi: 10.1520/D1765-19

- [15] ASTM D 3192. Test Methods for Carbon Black Evaluation in NR (Natural Rubber). ASTM International, West Conshohocken, PA. doi: 10.1520/D3192-09R19.
- [16] ASTM D 7854. Test Method for Carbon Black-Void Volume at Mean Pressure. ASTM International, West Conshohocken, PA. doi: 10.1520/D7854-18C.
- [17] Grunert, F., Wehmeier, A. u. Blume, A.: New Insights into the Morphology of Silica and Carbon Black Based on Their Different Dispersion Behavior. *Polymers* **12** (2020) 3.
- [18] ASTM D 6556. Test Method for Carbon Black Total and External Surface Area by Nitrogen Adsorption. ASTM International, West Conshohocken, PA. doi: 10.1520/D6556-19A.
- [19] Schwab, L., Hojdis, N., Lacayo, J. u. Wilhelm, M.: Fourier-Transform Rheology of Unvulcanized, Carbon Black Filled Styrene Butadiene Rubber. *Macromolecular Materials and Engineering* **301** (2016) **4**, 457.
- [20] Schwab, L. „Fourier transform rheology of complex, filled rubber materials“, Dissertation, Karlsruher Instituts für Technologie, 2016.
- [21] Hyun, K., Wilhelm, M., Klein, C. O., Cho, K. S., Nam, J. G., Ahn, K. H., Lee, S. J., Ewoldt, R. H. u. McKinley, G. H.: A review of non-linear oscillatory shear tests: Analysis and application of large amplitude oscillatory shear (LAOS). *Progress in Polymer Science* **36** (2011) **12**, 1697.
- [22] Leblanc, J. L.: Simplified modeling calculations to enlighten the mechanical properties (modulus) of carbon black filled diene rubber compounds. *Journal of Applied Polymer Science* **122** (2011) **1**, 599.
- [23] Ziegler, J. „Beeinflussung der Polymer-Füllstoff-Wechselwirkung durch Oberflächenmodifizierung von Füllstoffen“, Dissertation, Universität Hannover, 2004
- [24] Pan, Y. u. Zhong, Z.: A viscoelastic constitutive modeling of rubber-like materials with the Payne effect. *Applied Mathematical Modelling* **50** (2017), 621.
- [25] Donnet, J.-B. u. Custodero, E. “Reinforcement of Elastomers by Particulate Fillers”. In: Erman, B., Mark, J. E. u. Roland, C. M. (Hrsg.): *The science and technology of rubber*. Amsterdam: Elsevier Acad. Press 2013, 383.
- [26] Dötsch, T., Pollard, M. u. Wilhelm, M.: Kinetics of isothermal crystallization in isotactic polypropylene monitored with rheology and Fourier-transform rheology. *Journal of Physics: Condensed Matter* **15** (2003) **11**, 923.
- [27] Wilhelm, M., Maring, D. u. Spiess, H.-W.: Fourier-transform rheology. *Rheologica Acta* **37** (1998) **4**, 399.
- [28] Mattes, K. „Methoden zur Charakterisierung des nichtlinear viskoelastischen Verhaltens von Polymerschmelzen“, Dissertation, Universität Freiburg, 2007.
- [29] Geberth, E. „Analyse der Füllstoffkopplung von gefüllten Elastomeren unter mechanischer Belastung“, Dissertation, Universität Hannover, 2013.
- [30] Moulin, L., Da Silva, S., Bounaceur, A., Herblot, M. u. Soudais, Y.: Assessment of Recovered Carbon Black Obtained by Waste Tires Steam Water Thermolysis: An Industrial Application. *Waste and Biomass Valorization* **8** (2017) **8**, 2757.
- [31] Roy, C., Chaala, A. u. Darmstadt, H.: The vacuum pyrolysis of used tires. *Journal of Analytical and Applied Pyrolysis* **51** (1999) **1-2**, 201.
- [32] Roy, C. u. Darmstadt, H.: Carbon blacks recovered from rubber waste by vacuum pyrolysis - comparison with commercial grades. *Plastics, Rubber and Composites Processing and Applications* **27** (1998) **7**, 341.

Erstes 10-Zoll-Android-Tablet für den Ex-Bereich

TAB-EX PRO Ecom Instruments, Assamstadt, erweitert mit dem Tab-Ex Pro die bestehende Tab-Ex-Serie und damit das Angebot an mobilen Endgeräten für den Ex-Bereich. Mit seinem 10-Zoll-Bildschirm ist das Tablet für optisch anspruchsvolle Anwendungen und das Anzeigen webbasierter Inhalte besonders geeignet. Zudem gelingt Anwendern damit ein leichter Übergang vom Industrieinsatz ins Büro und zurück. Das Tablet ist trotz des großen Bildschirms und des robusten Aufbaus leicht und handlich gehalten. Dem Tablet dient mit dem Galaxy Tab Active Pro erneut ein Samsung-Tablet als Basis – mit Features wie Google AR Core, Gesichtserkennung oder dem Qualcomm SDM670 Octa-Core 64bit 2.0 GHz, 1.7 GHz Prozessor. Samsung Knox



Das Tablet ist seit September 2020 mit NEC-/CEC-Zulassung und seit Oktober 2020 mit ATEX-/IECEx-Zulassung erhältlich.

und Android 9 (Android 10 ist geplant) garantieren eine hohe Daten- und Gerätesicherheit. Mithilfe der Samsung-DeX-Funktion können Anwender das Tablet an einen großen Bildschirm und eine Tastatur für eine Desktop-Variante anschließen. So wird aus dem Tablet für den Feldeinsatz schnell

ein Gerät für das Büro. Zusätzlich ermöglicht das Tablet Anwendungen für den Alleinarbeiterschutz, die besonders für den Industrieinsatz notwendig sind. Das Tablet ist mit einer programmierbaren Taste für einen schnellen Alarm ausgestattet. Die für die Zone 2/22 und Division 2 ausgelegten Mikro-

fon- und Lautsprecheranschlüsse gewährleisten eine reibungslose Kommunikation. Pogo-Pin-Aufladung und ein leistungsstarker, austauschbarer Akku mit einer Kapazität von 7.400 mAh für eine Laufzeit von bis zu 15 Stunden sorgen für eine lange Einsatzzeit im Gelände. Mit passender Peripherie wie unterschiedlichen Headsets, Lautsprechern und Mikrofonvorrichtungen oder einer Smartwatch erweitern Anwender die Funktionalität. Es ist seit September 2020 mit NEC-/CEC-Zulassung und seit Oktober mit ATEX-/IECEx-Zulassung erhältlich. Damit ist es das weltweit erste 10-Zoll-Android-Tablet für den Ex-Bereich. ■

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