

FAPSIG Newsletter — Issue 13

August 2017



Call FOR PAPERS—ANTEC 18

• 2017 ANTEC WRAP UP

Special points of interest:

- DR. MYER EZRIN BEST PAPER WINNER
- FAPSIG STUDENT AWARD WINNER
- ANNOUNCEMENTS
- FAPSIG BOARD MEETING MINUTES

Letter from the Chair — Brian Ralston

Letter from the Chair:

Summer greetings from the Failure Analysis and Prevention SIG (FAPSIG)! Thanks to all who attended and participated in FAPSIG's ANTEC sessions in Anaheim this past May. We look forward to your continued involvement and support.

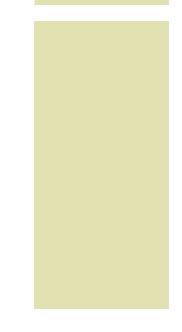
It's hard to believe that it's August already and autumn is just around the corner. Along with the leaves changing color, cool crisp nights, and football, autumn is also the season for drafting papers for ANTEC 2018! As always, FAPSIG is soliciting papers related to the prevention and analysis of plastics-related failures. ANTEC is a great opportunity to learn, and to share what you know, about anticipating, preventing, and analyzing failures. So if you have an idea for a paper, I urge you to make it a reality and submit it to FAPSIG for ANTEC 2018.

In addition to paper presentations, FAPSIG is aiming to put together sessions geared towards an introduction to failure analysis for young/new engineers. If you have any questions or want to get involved, don't hesitate to contact Todd Menna, our new FAPSIG TPC (and SPE Counselor) for 2018-2019 (todd.menna@element.com).

I am also proud to share that 2018 will be the second year for the FAPSIG Student Award. My hope is that this award will further encourage graduate and undergraduate students to become involved in SPE. Please join me in spreading the word and encouraging any interested undergraduate and graduate students to apply for this \$2,000 award.

Warm regards, Brian Ralston Cambridge Polymer Group FAPSIG Chair





FAPSIG Newsletter Editor: Paul Gramann, The Madison Group



ANTEC 2018 (May 7-10, 2018)

Call for Papers/Presentations

Failure Analysis and Prevention Special Interest Group

The Failure Analysis and Prevention Special Interest Group (FAPSIG) is soliciting papers and presentations for the next ANTEC conference, which will be held alongside the National Plastics Expo (NPE), May 7-10, 2018 in Orlando, Florida.

The rules for submissions are different than in years past. In addition to formal papers, ANTEC will now allow presentations to be submitted without the need of a formal paper submission. Presentations will still be subjected to the peer review process and must be submitted, in full, by the submission deadline.

Many aspects of product and process development, material selection, manufacturing, and end-use/service life testing fall under the umbrella of failure analysis and prevention. Papers/presentations can be submitted addressing failure analysis and prevention related to plastic and composite materials, including automotive, consumer products, medical, sporting goods, construction, piping, aerospace, additive manufacturing, etc.

Topics for which FAPSIG is soliciting papers include (but are not limited to):

- Identifying and demonstrating practical solutions to prevent or mitigate common industrial problems (e.g., adhesion, failures, chemical resistance, and environmental stress cracking)
- Case studies on failure prevention in product design and manufacturing
- Case studies illustrating the use of complimentary tools/techniques to determine the root cause of plastic failures
- Use of new methodologies in performing plastic product failure analysis
- Use of non-destructive methodologies (e.g., CT scanning, finite element analysis) to identify and prevent failures in plastic components
- Fracture behavior of polymers and the role of fractography in understanding plastic product failures
- Failure analysis and lifetime prediction of plastic pipe and fittings
- Failure of medical implants, including designed failures (e.g., resorbables)
- Prevention of plastic product failures with use of accelerated aging and/or service lifetime prediction methodology

Paper Submission Deadline: January 2018

Questions? Contact: Todd Menna FAPSIG Technical Program Chair E-mail: todd.menna@element.com Phone: (262) 901-0533

ANTEC 2017 Wrap Up

FAPSIG offered two well-attended sessions at ANTEC 2017 in Anaheim. We partnered with the Pipes and Fittings SIG for a joint session Monday to present papers related to Preventing Failure through Simulation and Characterization of Failure Mechanisms. We held a session presenting several case studies and an interactive Failure Analysis expert panel discussion on Wednesday morning. A panel of invited experts answered a variety of questions on polymer characterization methods and provided advice on troubleshooting plastics-related failures. Thanks to Don Duvall of ESI and Dr. James (Jim) Rancourt of Polymer Solutions for sharing their time and expertise.

Congratulations to Farzana Ansari (lead author/presenter), Christopher Lyons, Ryan Siskey, Suresh Donthu, and Steve MacLean of Exponent, Inc. for winning the Dr. Myer Ezrin Best Paper Award for their paper "*Mechanical Characterization and Fractography of PC, ABS and PMMA - A Comparison of Tensile, Impact and ESC Fracture Surfaces*". A copy is included at the end of this newsletter.

FAPSIG would also like to thank our sponsors for supporting and funding our ANTEC activities: Element, Engineering Systems, Inc., Exponent, The Madison Group, and Polymer Solutions. Because of our sponsors we were able to sponsor student travel, establish the new FAPSIG Student Award, and continue our annual tradition of handing out \$5 Starbucks gift cards to thank attendees for their interest and participation in FAPSIG sessions. If your company is interested in becoming a sponsor for ANTEC 2018, please contact Jeff Jansen (jeff@madisongroup.com).

Overall, the FAPSIG sessions at ANTEC 2017 were a success and we look forward to ANTEC 2018 in Orlando.

Brian Ralston, FAPSIG Technical Program Chair, ANTEC 2016-2017



Element New Berlin · www.element.com

FAPSIG Student Award Winner: Sebastian Goris from UW-Madison

We are pleased to announce Sebastian Goris from the University of Wisconsin-Madison as the winner of the first annual FAPSIG Student Award. Sebastian's research is titled. "Process-Induced Fiber Migration in Injection Molding of Long Glass Fiber-Reinforced Thermoplastics and the Impact on Failure Analysis." His research deals with predicting the variation of fiber concentration throughout the part, which will serve to improve the prediction of stresses and failures. The summary of his research is given on the next page.

The FAPSIG Student Award is a two-part award. For the first part, Sebastian was presented with \$500 at ANTEC 2017. To receive the second part of the award (\$1,500), the research described in the winning application must be submitted as a paper, accepted and presented at ANTEC 2018.

The FAPSIG Student Award winner is selected based on a 1-page summary of their research. The summary must be written by the student and submitted in early March. The winner is announced by email via a valid university email account. To be eligible for the second part of the award, the applicant is expected to complete the proposed research as a university student and to be enrolled at the time the paper is submitted to the following year's ANTEC.

Mark your calendar so next year you can submit your research or please distribute this information to students you may know.





www.chemir.com

Process-Induced Fiber Migration in Injection Molding of Long Glass Fiber-Reinforced Thermoplastics and the Impact on Failure Analysis

Summary of research submitted by Sebastian Goris, University of Wisconsin—Madison. Recipient of the 2017 SPE Failure Analysis and Prevention Scholarship

Long fiber-reinforced Lothermoplastics (LFTs) have gained significant importance in the transportation industries due to their favorable material properties, lower manufacturing costs and superior lightweight characteristics. However, the configuration of the reinforcing fibers is significantly changed throughout the entire production process, reflected in the form of fiber attrition, fiber alignment, fiber jamming and fiber matrix separation. The process-induced variation of the fiber microstructure within the molded part introduces heterogeneity to the mechanical properties and adds significant complexity to finite element analysis (FEA) modeling and lifetime prediction of LFT parts. The challenges of controlling and predicting this heterogeneity and its impact on the mechanical performance limits the potential of LFT for lightweight applications. In particular, the underlying physics of the process-induced change in fiber concentration have been not fully understood vet and is not addressed in the structural analysis as all modeling approaches assume a uniform fiber concentration throughout the molded part. My research focuses on the experimental evaluation of fiber matrix separation in LFT injection molding and its impact on the mechanical performance of the finished part. Results of an experimental study suggest a substantial agglomeration of fibers in the core-layer of an injection molded part with up to 40% more fibers, as illustrated in Figure 1. As part of my research project, I will complete a DoE of injection molded plates at varying fiber concentration (5%wt. to 60%wt.) to generate a comprehensive set of experimental data. The fiber configuration (orientation, length, and concentration) are measured using novel measurement concepts, including micro computed-tomography (µCT) and image processing. Additionally, tensile tests and flexural tests will be performed of coupons extracted from the molded parts. The results of this experimental study will answer which damage mechanisms are contributing to failure of the LFT material and how it can be correlated to the observed fiber agglomeration in the core layer. In the next step, my goal is to study if local damage initiation and overall failure prediction can be accurately modeled via finite element analysis (MultiMechTM coupled with ANSYS®) if the fiber concentration gradient is accounted for. This part of my work will specifically include a comparison of the predicted failure with the simplifying assumption of a uniform fiber concentration. Of particular interest will be the numerical failure analysis focusing on the correlation between fiber orientation and fiber concentration since the first experimental results suggest that both microstructural properties have a correlated core-shell distribution through the thickness.

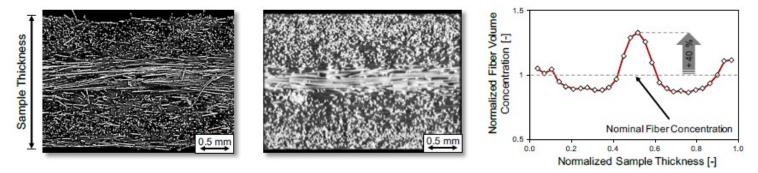


Figure 1. Illustration of the observed fiber concentration gradient through the thickness of an injection molded plaque (40%wt. glass fiber-reinforced polypropylene): SEM image of the fracture surface of a tested tensile specimen (left), 2D cross-sectional view from a μ CT scan (center) and measured fiber volume (normalized) through the thickness (right).

Ultimately, this work will answer the question of whether the simplifying assumptions of a uniform fiber concentration in injection molded parts is permissible or not for FEA modeling and lifetime prediction of LFT components. As a result, it may be possible to achieve a more reliable prediction of the structural performance of LFT parts if the actual variation in fiber concentration is accounted for in the design process. The outcome will contribute to a better understanding of the process-microstructure-property relationship and might help to exploit the full potential of this material class.

FAPSIG Meeting Minutes, May 9, 2017

Attendees:

- Jennifer Hoffman Chair
- Brian Ralston Technical Program Chair (TPC)
- Todd Menna TPC-in-Training (acting Secretary)
- Javier Cruz Education
- Don Duvall Memberships
- Antoine Rios Activities
- Sue Mantell Webmaster

Absent

- Jose Perez Secretary
- Steve MacLean Treasurer
- Jeff Jansen Sponsorships
- Michael Hayes Social Networking
- Paul Gramann Newsletter

Others

- Dale Edwards
- Brian Hauser
- Maureen Reitman

Board (Hoffman):

- Minutes approved from 2016 meeting, no changes requested. A quorum was deemed to be present.
- Board position changes:
 - Jennifer Hoffman to vacate Chair position and become Past Chair, Brian Ralston to take position as Chair for 2017-2019.
 - Brian Ralston to vacate TPC position, Todd Menna to take position as TPC for 2017-2019.
 - Jose Perez to take TPC-in Training position for 2017-2019.
 - Brian Ralston to vacate Best Paper Award position. Jennifer Hoffman to take position as Best Paper Award for 2017-2018.
 - Jose Perez to vacate Secretary position. Position now vacant.
 - All other Board positions to remain the same through 2017-2018.
- Positions open to replacement: Newsletter, Sponsorships
- Decision made to dissolve the Social Networking position and merge it with Activities. Antoine will remain the Activities Chair.
- Hoffman to update/develop "Communication Plan," the goal of which is to set dates for various activities to be performed (newsletter goals, request for sponsors, paper solicitations, paper due dates, email blasts, etc.). To work with others as necessary.
 - More resources are available through SPE such as 2016-2017-Leadership-Checklist.doc", "Communication-Planning-Template.xls", "Goals-Workplan-Template.doc"

Finance (Hoffman):

- Hoffman handled Finance due to absence of MacLean.
- As of 12/31/2016, balance was \$6,242.42.
- As of 05/08/2017, balance was \$11,607.37.
- This amount does not take into consideration the \$500 Student Award or the \$750 student travel fund donation.
- A discussion was held regarding whether to continue purchasing Starbucks gift cards to be handed out after FAPSIG presentations by sponsors. No decision was made.
 - Too many gift cards, not enough talks;
 - Some sponsors end up taking cards home instead handing them out;
 - o Is there something better we can do with this money?

Newsletter (Hoffman):

- We only had one newsletter over the last calendar year. Sponsorship requests state that we publish one 3-4 times a year. We need a more frequent, expected publication date.
- Proposal to publish the newsletter in June/July to summarize ANTEC, call for papers for next ANTEC, advertise scholarships, call for sponsors for next ANTEC, general information. Second newsletter to be published in January/February to announce ANTEC sessions/program, call for sponsors, general information.
- Hoffman to follow-up with Newsletter

TPC Update (Ralston):

- Total of eleven FAPSIG papers for 2017 ANTEC. We had five in 2016. One invited speaker, Jim Rancourt from Polymer Solutions.
- Although not advertised for 2017 ANTEC, a decision was made in 2016 by the SPE ANTEC Technical Program Committee to allow presentations to be submitted instead of full papers. Presentations would still need to be submitted by the paper deadline (roughly two months before ANTEC, set by ANTEC TPC) and peer reviewed prior to acceptance. Presentations would be preserved and included in the conference proceedings. Invited speakers are not required to submit a paper or presentation. This information needs to be considered when soliciting papers for 2018 ANTEC.
- Begin soliciting papers for 2018 ANTEC ASAP.
 - Start advertising in the new summer newsletter.
 - Email blasts.
- Ideas for 2018 ANTEC:
 - Approach equipment manufacturers regarding the use/role of XXX (e.g., FTIR, GC/MS, spectroscopy, SEM, mechanical testing, etc.) as a tool in failure analysis.
 - Goal is not to have these be commercial in nature, i.e., not an advertisement for a specific manufacturer or instrument.
 - Menna approached several manufacturers present at ANTEC. All are interested and already have canned presentations available. Stressed noncommercial nature.
 - Session regarding the status of the teaching of failure analysis in academia. What does academia teach, and how? This would need to be an in-depth research study, and possibly coordinated with several professors from across the nation.

FAPSIG Meeting Minutes, May 9, 2017—Continued

Tutorial session, "Intro to Failure Analysis," at 2018 ANTEC.

Best Paper Award (Ralston):

- Review process appears to be working. Of the eleven papers, no two papers had the same three reviewers. Award criteria will continue to be based upon paper as well as presentation scores (weighted 67/33 paper/presentation).
- Award Winner (announced during Wednesday's paper session) was Farsana Ansari et al. from Exponent.

Sponsorships (Hoffman)

- Five sponsors for 2017 ANTEC: Polymer Solutions, ESI, Element, Exponent, Madison Group. These are basically the same sponsors as the last few years. How can we get more/others?
 - Consider test equipment OEMs?
- FAPSIG sponsored the ANTEC Reception on Monday night for \$1,000. Not worth the money as we had a meager sign and no official table. Not recommended to sponsor next year's reception unless significant changes occur.
- Request for sponsorships for 2017 ANTEC was less than two weeks before ANTEC started. Payments were due within days of initial request.
- Some companies have still not been billed for their 2016 sponsorship.
- Can we start the search earlier in the year so that we have the opportunity to obtain more sponsors?
 - Newsletter announcements
 - Email blast
- Should we include in the newsletter the list of items FAPSIG sponsors?

Education (Cruz)

- Cruz will interface with the TPC and the Chair regarding potential tutorial sessions geared towards younger engineers/scientists for 2018 ANTEC.
 - Cruz to contact the SPE Next Generation Advisory Board (NGAB) for access to members, ideas on how to contact them, etc.
- Consider hosting a free webinar through SPE regarding failure analysis.
 - Webinar is not free to host, and FAPSIG would have to sponsor it. Cost to be determined. Mantel to contact SPE to learn more.

Activities (Rios)

- Student Award was in its inaugural year. Only two applicants. One was clearly better than the other. Winner was Sebastian Goris from the Polymer Engineering Center at the University of Wisconsin. Half of the award money will be given now. This student will be expected to present his research at a future ANTEC in order to receive the second half of the award money.
- Advertising the award
 - o Create a flyer to advertise the Student Award during next year's poster session?
 - Advertise the award in the newsletter.
 - Advertise the award on the slides between presentations in the sessions.
 - Have the moderator mention the award between presentations in the sessions.
 - Advertise the award on the FAPSIG website.
 - Have more frequent email blasts. Set the blasts timing such that there is a reminder two weeks before the application is due.
 - Continue to discuss the Award with academia. All are encouraged to send out the Award notification to academic contacts.
 - Send email blast to all FAPSIG members with a .edu email address.
 - Work with NGAB?

Membership (Duvall)

- From the publically available membership data available on SPE website: SPE overall has less than 12,000 members. Over 75% are Professional members. FAPSIG has 1,203 members.
 - Membership is declining, but FAPSIG has the second largest membership of all Divisions or SIGs.
- Can we determine a breakdown of memberships by "Sector?" Sectors to be defined by FAPSIG, but would define the industry category of the member, e.g., resin supplier, consultant, manufacture type, etc.
 - Can we further define by geographic location?
 - What does FAPSIG's membership look like by Sector? Can we do an analysis of it to define how, when, why or membership changes through the year? For example, it was noted that the Philadelphia Section tied their declining membership to the death rate of their senior members.
 - Information can be used to target potential sponsors, guide for technical content, recruiting members, recruiting Board members.
- There is no additional cost to an SPE membership to add a SIG. Do current or new
 members know this, or do they think they have to pay extra? Are we taking advantage of
 this? If not, how can we?

Announcements

The Madison Group is Pleased to Welcome Patrick Mabry.



The Madison Group has announced the addition of Patrick Mabry to its engineering team. Patrick earned his M.S. in Mechanical Engineering in 2015 from the University of Wisconsin – Madison's Polymer Engineering Center and joined the Madison Group in May of 2017. His primary responsibility is performing Moldflow simulation for injection and compression molded parts. Patrick's background is rooted in the design and processing of composite materials, these skills having been developed working for Trek Bicycle Corporation and HEAD Sports. Through his previous work he gained knowledge of lean manufacturing techniques, mold design, and composite part design.

"We are pleased to have Patrick join our growing team of engineers at The Madison Group. He brings excellent composite manufacturing knowledge that will

bolster our capabilities to The Madison Group," said Bruce Davis (CEO).



Element Materials Technology New Berlin Hires Clayton Zortman.

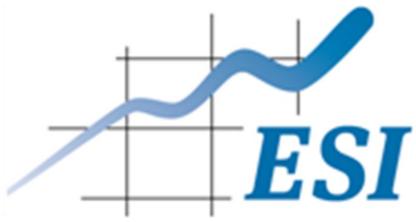
Clayton Zortman has recently joined the chemical analysis team at Element Materials Technology New Berlin as a Chemist in our materials analysis laboratory. Clay is an analytical chemist with experience in polymeric and materials chemistry and analysis. His most recent position was with Rehrig Pacific, where he served as a Quality Assurance Technician assisting with production and quality control. Clay obtained his Bachelor's degree in Chemistry from University of Wisconsin – Parkside, with minors in Mathematics and Physics.



Dr. Donald E. Duvall Named SPE Honored Service Member

Anaheim, CA – May 7, 2017. The Failure Analysis and Prevention SIG is pleased to announce that Dr. Donald E. Duvall has been named an Honored Service Member. According to the SPE Bylaws, to be elected an Honored Service Member, a candidate shall have demonstrated long-term, outstanding service to, and support of the Society and its objectives. Dr. Duvall received this award in part for his many contributions to the plastics industry and the Society of Plastics Engineers, including holding leadership roles in the Engineering Properties and Structure Division (EPSDIV), the Failure Analysis and Prevention SIG (FAPSIG), and the Plastic Pipe & Fittings SIG. Dr. Duvall was nominated by the Plastic Pipe & Fittings SIG, and additional referrals were submitted by Paul Gramann (FAPSIG) and Murali Rajagopalan (EPSDIV).





www.esi-website.com

2017 Failure Analysis and Prevention SIG 2017 ANTEC Sponsors

On behalf of the Failure Analysis and Prevention Special Interest Group of SPE I would like to thank our 2017 ANTEC sponsors.

Element Materials Technology ESI Exponent Polymer Solutions The Madison Group

Because of their generosity we are able to forward our mission of plastics education. Directly, the sponsorships were used to fund the newly created FAPSIG Student Award, for students involved in research related to failure analysis and prevention, and the annual Dr. Myer Ezrin Best Paper Award.

If you are interested in sponsoring FAPSIG at the 2018 ANTEC, please contact Jeff Jansen at jeff@ madisongroup.com

SPE Failure Analysis and Prevention SIG Board Members

Brian Ralston, Ph.D. Chair Cambridge Polymer Group 56 Roland St₄ Suite 310 Boston, MA 02129 brian.ralston@campoly.com

Jennifer L. Hoffman, Ph.D. Past–Chair/Best Paper Chair

AirXpanders, Inc. 1047 Elwell Ct. Palo Alto, CA 94303 P: 650-390-9015 jhoffman@airxpanders.com

Steve MacLean, Ph.D. Treasurer

Exponent, Inc. 17000 Science Drive, Suite 200 Bowie, MD 20715 Work Phone: (585)387-0327 smaclean@exponent.com

Todd J. Menna, Ph.D. TPC

Element Materials Technology 3200 South 166th Street New Berlin, WI 53151 todd.menna@element.com

Jose Perez

TPC in Training GE Healthcare jose.miguel.perez@ge.com

Paul J. Gramann, Ph.D.

Newsletter Editor The Madison Group 2615 Research Park Dr. Madison, WI 53711-4951 Work Phone: (608) 231-1907 paul@madisongroup.com Javier Cruz, Ph.D. Education Comm. Chair The Madison Group 2615 Research Park Dr Madison, WI 53711-4951 Work Phone: (608) 231-1907 javier@madisongroup.com

Jeffrey A. Jansen Sponsorship Chair Engineering Manager The Madison Group 2615 Research Park Dr Madison, WI 53711-4951 Work Phone: (608) 231-1907 jeff@madisongroup.com

Michael Hayes, Ph.D. LinkedIn Chair

Engineering Systems Inc 6190 Regency Parkway Norcross, GA 30071 Work Phone: (678)990-3280 mdhayes@esi-atl.com

Prof. Susan Mantell

Website Coordinator University of Minnesota ME 11A Minneapolis, MN 55455 Ph: 612-625-1324 smantell@umn.edu

Antoine Rios, Ph.D. Activities Chair

The Madison Group 2615 Research Park Dr Madison, WI 53711-4951 Work Phone: (608) 231-1907 antoine@madisongroup.com

Donald E Duvall, Ph.D. Membership Comm. Chair Engineering Systems Inc 3851 Exchange Ave

Aurora, IL 60504 Work Phone: (630) 851-4566 deduvall@esi-il.com

Dale B Edwards

Director Engineering Systems, Inc. 3851 Exchange Avenue Aurora, IL 60504 Work Phone: (630) 851-4566 dbedwards@esi-il.com

Andy R. Shah Director

Engineering Systems, Inc. 3851 Exchange Avenue Aurora, IL 60504 Work Phone: (630) 851-4566 arshah@esi-il.com

2017 FAPSIG Dr. Myer Ezrin Best Paper Award Recipient

MECHANICAL CHARACTERIZATION AND FRACTOGRAPHY OF PC, ABS AND PMMA – A COMPARISON OF TENSILE, IMPACT AND ESC FRACTURE SURFACES

Farzana Ansari, Ph.D., Exponent, Inc., Menlo Park, CA Christopher Lyons, Ph.D., Exponent, Inc., Menlo Park, CA Ryan Siskey, M.S., Exponent, Inc., Philadelphia, PA Suresh Donthu, Ph.D., P.E., Exponent, Inc., Menlo Park, CA Steven MacLean,

Abstract

This work presents an effort to document and describe fracture surfaces for three commercially available amorphous polymers (PC, PMMA and ABS) each subjected to tension, impact and environmental stress cracking (ESC). We present mechanical properties as well as microscopic characterization at low and high magnification to distinguish between slow tensile loading, fast impact loading, and environmentally assisted creep failure mechanisms. Chemical surface analysis of select fracture surfaces was also performed to evaluate its utility as a failure analysis technique for identifying ESC failure. The fractographic atlas presented herein serves to assist others in identifying topographical fracture surface features and crack growth mechanisms of failed plastic components, and more accurately distinguish between pure mechanical failure and ESC-generated fracture, where possible.

Introduction

The need for more cost- and energy-efficient materials in modern engineered products has necessitated a shift towards the use of load-bearing polymeric materials in a variety of applications across multiple industries. With this transition comes an increased need for understanding how such load-bearing polymers inevitably fail, especially with respect to cracking and fracture. Fractography is a useful tool for discerning the loading mode and environment that the polymer has experienced during failure. Examination at different magnifications can be combined with chemical analysis tools to understand the nature of initiation and propagation mechanisms that contributed to component failure.

Deciphering the differences between tensile, impact and environmentally-assisted stress fracture surfaces of plastics requires a nuanced understanding that is distinct from classical fractography used for metals, glasses and ceramics. While fractography guides are available for polymers [1-8], they often lack necessary details on the conditions of failure (e.g. loading mode, environment) or composition and processing of the polymer in question, which makes it difficult to use them for failure analysis of field failures.

This work presents a systematic fractographic evaluation of three commonly-used amorphous polymers under controlled stress conditions: tension, impact and environmental stress cracking (ESC). In reality, these three modes can often work in combination to create a fractured surface. For example, it is not uncommon for slow crack growth mechanisms, such as creep, fatigue and ESC, to transition to stress overload fracture once the advancing crack propagates to a critical length. However, evaluating each mode in isolation can greatly facilitate the creation of a "road map" to crack origination and root cause. The evaluation of ESC is particularly important for amorphous polymers, which are notoriously more susceptible to this failure mode due to their higher free volume and unordered molecular structure when compared to semi-crystalline polymer counterparts, [9]. ESC fracture surfaces can differ in morphology depending on applied stress/strain, the chemical composition and concentration of the ESC agent. In this work, we present a comparison of ESC failure in three materials exposed to two strain levels and solvent types. Ultimately, this work seeks to establish an atlas of polymeric fracture surface damage under controlled loading and environmental conditions to facilitate failure analysis efforts.

Materials and Methods

All three polymers evaluated were obtained in resin form and molded into final specimen geometries according to the manufacturers' recommended processing conditions. Materials evaluated included polycarbonate (PC) (Lupoy®, 1201-10), polymethylmethacrylate (PMMA) (LG Chem, IH 830), and acrylonitrile butadiene styrene (ABS) (LG Chem, HI-121).

Tensile Testing:

ASTM D638 [10] Type I dog bones with a thickness of 3.15 mm were prepared for tensile testing. Tensile testing was performed in ambient conditions at a crosshead displacement rate of 5 mm/min (strain rate: 0.1 min⁻¹), while recording engineering stress and strain. All testing was performed on a Zwick Roell Z010 (Ulm, Germany) load frame with a 10 kN load cell. Strain was measured using a Zwick BTC-ExMacro .001 (self-supporting) video extensometer (Ulm, Germany). The tensile testing was performed on five replicates per polymer type.

Izod Impact Testing:

Single-edge notched Izod impact specimens were molded in accordance with ASTM D256 [11] dimensions, with a thickness of 3.15 mm. Testing was performed in ambient conditions on an Izod impact tester equipped with a pendulum having a nominal total energy of 21 Joules. The impact strengths (Izod energy / cross sectional area) of five specimens for each material were recorded.

ESC Testing:

ESC testing of each polymer type was conducted using a custom-made constant strain jig. Bars 3.15 x 10 x 50 mm in size were subjected to a constant bending tensile strain of approximately 1.5%. Specimens of each material were exposed to either ambient (air), Bis (2ethylhexyl)phthalate (DEHP), or isopropyl alcohol (IPA). The ESC agents were applied to the tensile surface of each sample. Time-to-failure and failure mode observations (crazing, cracking and/or complete fracture) were monitored using time-lapse photography. Fractography of ESC specimens was performed only on those that exhibited complete breakage.

Fractography:

Fracture surfaces from each loading mode were examined using a Keyence optical microscope (Osaka, Japan) under direct and indirect lighting. Scanning electron microscopy (SEM) was performed using a FEI Versa 3D DualBeam (Hillsboro, Oregon) on the same fracture surfaces in both secondary and backscatter mode in low vacuum at 10 kV. The fracture surfaces were not coated for SEM examination. Fouriertransform infrared (FTIR) spectroscopy was performed on fracture surfaces of ESC and tensile specimens (control) to determine the detectability of residual ESC agents several days after fracture.

Results and Discussion

Mechanical Properties:

Tensile testing results are shown in **Figure 1** and **Table 1**. PC exhibited the highest ductility and moderate tensile strength, while ABS showed the lowest strength. PMMA showed the highest tensile strength and least amount of ductility as evidenced by the lowest elongation to break value. Strain hardening was also evident at high strains, likely due to chain alignment and stretching [5,6].

Impact data for the three polymers is summarized in **Table 2**. PC demonstrated the highest impact strength, followed by ABS and PMMA, which is consistent with tensile test results. Both PC and ABS exhibited a hinge failure mode, consistent with relatively higher ductility compared with PMMA, which consistently demonstrated a clean break. The differences in yield strength between the three polymers were statistically significant (p<0.05).

The high impact toughness in PC measured in this study appears to be greater than reported in the literature [12], which could be due to the small specimen thickness used in this study.

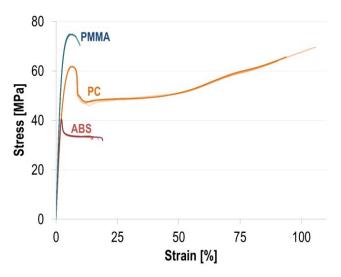


Figure 1 | Engineering stress-strain data from the three amorphous polymers evaluation.

Table 1 Tensile	properties	extracted	from	stress-strain	
data for all three materials evaluated.					

	Yield Strength [MPa]	Yield Strain [%]	UTS [MPa]	Elongation at Break [%]	Modulus [MPa]
PC	61.8	6.5	61.8	97	2380
	± 0.1	± 0.1	± 0.1	± δ	± 35
ABS	40.4	2.2	40.4	17	2412
	± 0.2	± 0.0	± 0.2	± 2	± 11
PMMA	74.7	6.2	74.8	7	3405
	± 0.4	± 0.1	± 0.1	± 2	±86

 Table 2 | Izod notched impact properties for PC, ABS and PMMA.

	Charpy Impact Strength [kJ/m2]	Failure Mode
PC	93 ± 4	Hinge
ABS	30 ± 2	Hinge
PMMA	2 ± 1	Clean Break

ESC Testing:

ESC results are summarized in **Table 3**. Among the three polymers, PMMA exhibited the shortest time to failure for both DEHP and IPA, followed by ABS and then PC. Interestingly, though crazing occurred relatively rapidly in ABS and PC samples treated with IPA, complete failure did not occur, possibly either due to evaporation of the IPA from the sample surface, or strain relief in the sample over time due to extensive crazing. Specimens exposed to ambient air did not exhibit any cracking or fracture after one week of testing.

Sample	Strain %	ESC Agent	Time to First Signs of Crazing (min)	Time to Failure (min)
ABS	1.5	DEHP IPA	140 60	660 N/A*
PC	1.5	DEHP IPA	380 60	760 N/A*
PMMA	1.5	DEHP IPA	N/A Immediate	100 5

*Crazing observed, but complete failure did not occur after 1 week of testing

Fractography:

Optical microscopy of representative fracture surfaces from each material and loading mode are shown in **Figure 2**. Gross necking was observed on PC and ABS specimens loaded under tension or impact, while no necking was seen on PMMA specimens or on any ESC samples. The behavior of PMMA is consistent with the low ductility evidenced from mechanical testing. Both impact and tensile loading of PMMA samples generated little, if any, ductility, despite the difference in strain rate exhibited by both loading modes and the introduction of a notch for impact testing. ABS, on the other hand, demonstrated a ductile-brittle transition at room temperature, with significant deformation under slower strain (tensile loading) compared to high strain (impact).

A single fracture origin is observed on tensile specimens for all materials, while multiple origins are apparent on ESC surfaces. Furthermore, unlike tensile and impact specimens, ESC fracture surfaces contain multiple morphological features including glossy, smooth regions intertwined with sharp ledges, multiple fracture origins on neighboring planes, discontinuous growth bands, and/or surface fibrillation due to craze rupture.

Figure 3 shows SEM imaging of the fracture surfaces of PC samples at different magnifications. No cavitation or fibrillation is observed under the three loading modes at room temperature. Instead, macroscale linear features and smooth crack transitions are observed, which, in combination with gross necking seen in Figure 2, are consistent with the relatively high toughness and impact strength exhibited by this material. Significant necking of the impact specimen is likely due to its small thickness combined with PC's high intrinsic toughness. Plane stress conditions at the specimen surface lead to enhanced bulk specimen yielding, which encourages ductile crack growth. Interestingly, under high magnification (Figure 3), the fracture surface adjacent to the notch and at the center of the specimen (where plane stress conditions are minimized) do not show features indicative of ductility. This is consistent with the PC fracture surfaces for thicker specimens previously reported in the literature [12].

Unlike ABS and PC, the fracture origin of PMMA tensile specimens displays a very smooth surface texture adjacent to the initiation point, which then transitions into a series of concentric rib markings located approximately 400 μ m from the origin (**Figure 5**). This is followed by fast, multi-planar fracture that resulted in fragmentation of PMMA under tensile loading, consistent with this polymer having relatively low ductility compared to ABS and PC. The concentric rib markings seen on tensile samples were also seen on

ESC Notch Notch Notch MPACT TENSILE PC ABS **PMMA**

Figure 2 | Optical microscopy of representative fracture surfaces for each material and loading mode.

Fracture origins on tensile specimens are labeled with white solid arrows. Notches on impact specimens are oriented on top and impact direction is shown by the white dashed arrows. ESC fracture surfaces are shown for DEHP-treated samples, and are oriented such that the tensile surface is on the right side of the image.

impact specimens, though with significantly less spacing between each band (**Figure 6**). Still, both tensile and impact specimens exhibited variable spacing between rib markings, with each concentric band growing further apart downstream from the origin. Alternatively, as seen in **Figure 6C**, discontinuous growth bands (DGBs) seen on the ESC PMMA specimen were more uniformly distributed, with no noticeable change in spacing.

ESC fracture surfaces exhibited strong similarities between the three materials; however, subtle differences in surface morphology could be seen when comparing different regions of the same specimen or ESC agents. ABS shows smooth surfaces separated by ridges near an origin, as well as multiple fracture planes, while surface fibrillation due to craze rupture appears farther away from the origin (**Figure 7**). In general, both solvents demonstrated some degree of ESC failure for all three polymers, consistent with their expected solubility in each material. Hansen solubility parameters for IPA and DEHP were 11.5 $(cal/cm^3)^{1/2}$ and 7.9 $(cal/cm^3)^{1/2}$, respectively, close in range to solubility parameters for PMMA (9.6 $(cal/cm^3)^{1/2}$), PC (10.5 $(cal/cm^3)^{1/2}$) and ABS (9.39 $(cal/cm^3)^{1/2})$ [10,13]. FTIR analysis of all ESC samples successfully led to identification of DEHP on all fracture surfaces (results for the ABS ESC sample are shown in Figure 8). FTIR data was collected several weeks after fracture when surfaces appeared residue free, demonstrating its potential effectiveness as a failure analysis tool. However, the success of FTIR analysis does depend on the ESC solvent, in particular, its vapor pressure and chemical structure. This was seen with analysis of PMMA ESC specimens exposed to IPA, in which FTIR analysis did not detect any residual solvent on the surface. Chemical surface detection of ESC agents depends on both the volatility of the solvent as well as its affinity (or lack thereof) for the polymer material. In other words, a negative finding from chemical analysis does not necessarily imply that ESC was not the failure mechanism; fractographic analysis and a thorough understanding of the end-use environment become crucial in isolating ESC as the root cause. Chemical analysis can further validate that failure mode.

Conclusions

The morphology of fracture surfaces depends on multiple parameters, including loading mode, fundamental material properties and environment (solvent type). Evaluation of tensile, impact and ESC failure of PC, ABS and PMMA revealed similarities and differences that can be leveraged in analysis of failed components in the field.

- All ESC fractures exhibited consistent surface features: multiple origins, often occurring on multiple adjacent planes, a lack of macro- or microductility, and a smooth, glossy appearance near the origin.
- Solvent type and applied load did influence the degree and rate of ESC failure.
- Similarities between tensile and impact specimens were seen for PMMA specimens, which exhibited lowest toughness and ductility, while ABS specimens demonstrated a remarkable shift from ductile to brittle behavior between the two loading modes.
- The differences in low and high magnification imaging of PC impact specimens revealed the importance of utilizing both optical and scanning electron microscopy in interpreting fracture surfaces, especially where variable thickness can be involved.

• Chemical analysis of ESC fracture surfaces can be useful in identifying the ESC agent and further validate this failure mode. . However, the detection of the ESC agents depends on their volatility and affinity to the polymer. A lack of conclusive results from chemical analysis does not necessarily negate ESC as the primary cause of failure.

Given that failure of polymeric components is often multifactorial, our study demonstrates the utility of controlled, bench-scale testing in isolating relevant parameters to provide a robust road-map to determining the root cause(s) of failure.

Acknowledgements

We would like to acknowledge Chris Pederson of Exponent for his assistance with all scanning electron microscopy presented this study, Intertek PTLI for performing the tensile evaluation of all materials, and Maureen Reitman and Brad James for their guidance on study design and direction. References

- Hayes, M., D. Edwards, and A. Shah. *Fractography* in *Failure Analysis of Polymers*. William Andrew, 2015.
- 2. Greenhalgh, E. Failure analysis and fractography of polymer composites. Elsevier, 2009.
- 3. Engel, L. An Atlas of Polymer Damage: Surface Examination by Scanning Electron Microscope. Prentice-Hall, 1981.
- Ezrin, M. *Plastics Failure Guide*. 2nd Ed. Carl Hanser Verlag GmbH & Co., 2013.
- 5. Hertzberg, R. W., & Manson, J. A. (1980). *Fatigue of engineering plastics*. Academic press.
- 6. Lampman, S. (2003). *Characterization and failure analysis of plastics*. ASM International.
- Roulin-Moloney, A. C. (Ed.). (1989). Fractography and failure mechanisms of polymers and composites. Elsevier applied science.
- 8. Schiers, J. Composition and Failure Analysis of *Polymers*. John Wiley & Sons Inc 2000.
- 9. Wright, D.C. *Environmental Stress Cracking of Plastics*. RAPRE Technology Ltd. 1996.
- 10. ASTM D683-14, Standard Test Method for Tensile Properties of Plastics
- ASTM D256-10e1, Standard Test Methods for Determining the Izod Pendulum Impact Resistance of Plastics
- Ralston, B. E., et al. (2013). Fractographic examination of failures in polycarbonate and polyoxymethylene due to impact, tensile, fatigue and creep mechanisms. Proceedings, ANTEC 2013, Society of Plastics Engineers, Cincinnati, OH.
- 13. Hansen, C. M. (2007) *Hansen solubility parameters:a user's handbook.* CRC press.

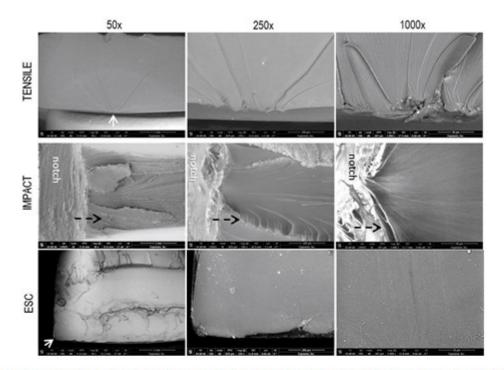


Figure 3 | SEM images of the PC fracture surfaces at different magnifications for the three loading modes. Images were taken near an origin. White solid arrow points to fracture origin. Black dashed lines indicate crack growth direction on impact specimens.

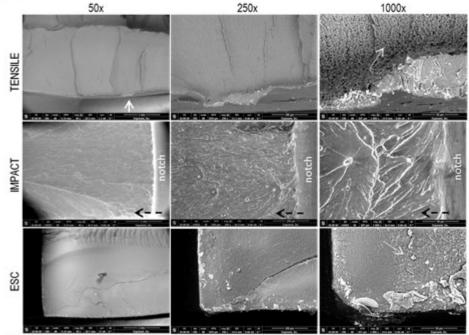


Figure 4 | The SEM images of ABS fracture surfaces at different magnifications for the three loading modes. Images were taken near an origin. White solid arrow points to fracture origin. Dotted white lines point to worm-like features indicative of craze rupture. Black dashed lines indicate crack growth direction on impact specimens.

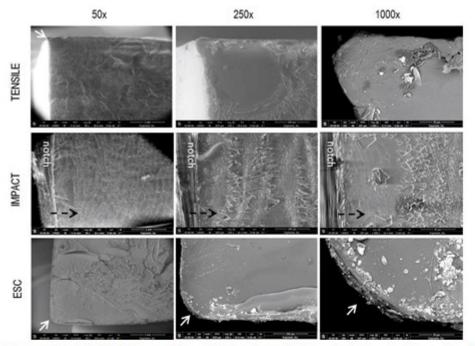


Figure 5 | SEM images of the PMMA fracture surfaces at different magnifications for the three loading modes. Images were taken near an origin. White solid arrow points to fracture origin. Black dashed lines indicate crack growth direction on impact specimens.

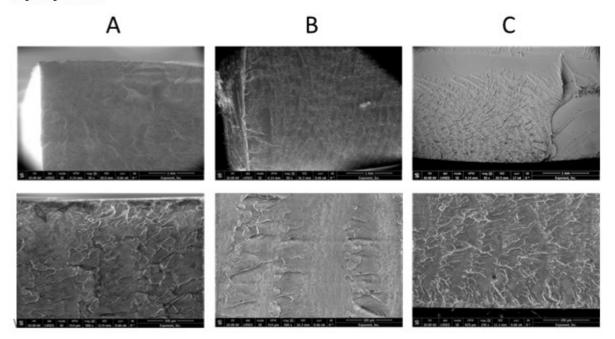


Figure 6 | Low (top row) and high (bottom row) magnification imaging of the concentric banding on (A) tensile, (B) impact and (C) ESC fracture surfaces of PMMA.

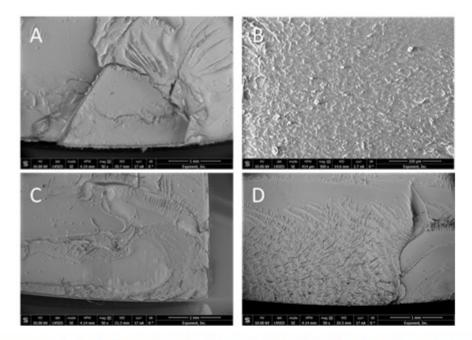


Figure 7 | SEM images highlighting the differences in surface morphology exhibited on ESC fracture surfaces. (A) ABS fracture surface near an origin. (B) ABS fracture surface further away from a crack origin. (C) PMMA fracture surface after exposure to 1.5% DEHP. (D) PMMA fracture surface exposed at 1.5% isopropyl alcohol.

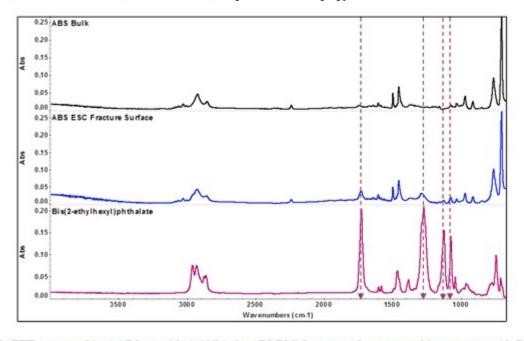


Figure 8 | FTIR spectra of (top) ABS material, (middle) the ABS ESC fracture surface generated by treatment with DEHP, taken several weeks after failure occurred, and (bottom) an authentic spectrum of DEHP. Characteristic peaks associated with DEHP near 1740, 1280, 1130, and 1090 cm⁻¹ were observed on the ABS fracture surface.