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WINTER 2021

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Official Newsletter of the SPE Composites Division

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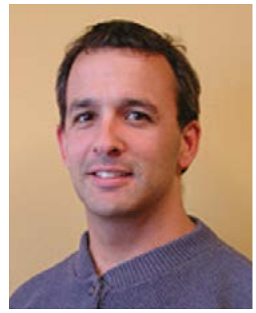

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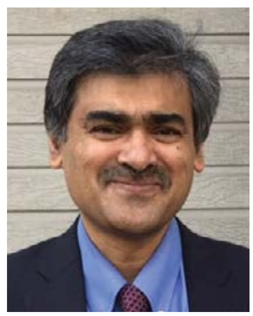

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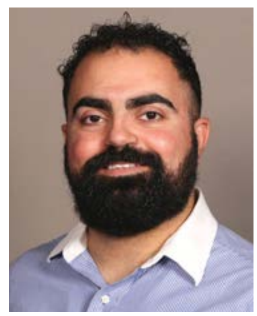

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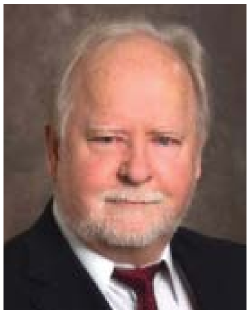

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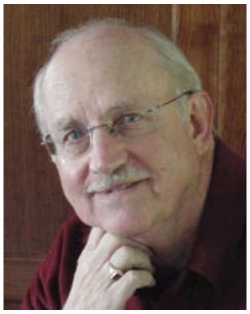

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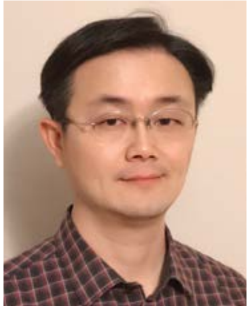

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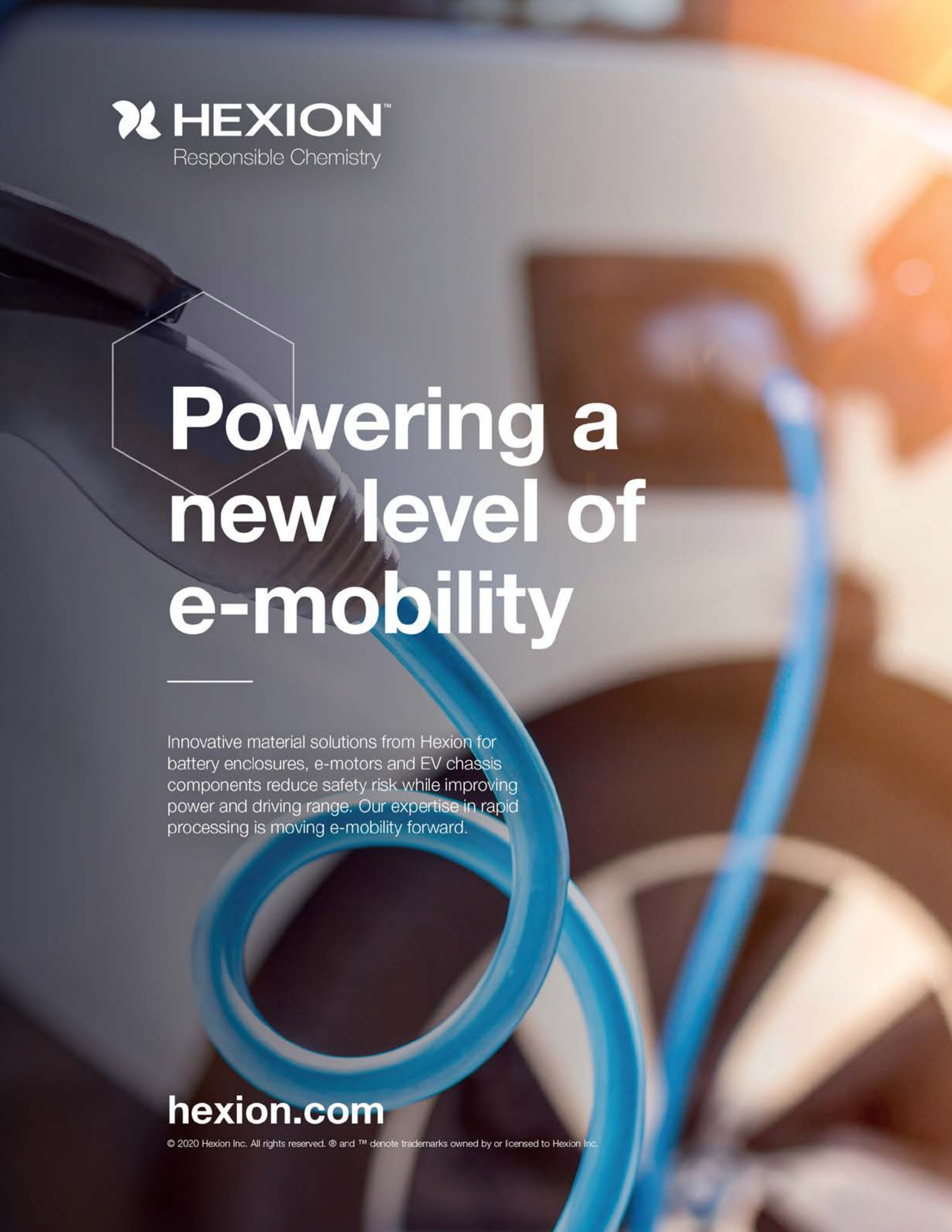
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This Issue:

Award Winning Paper

Influence of Soy Hull Based Biocarbon and Graphene Nanoplatelets on the Performance of Polypropylene Biocomposites for Automotive Applications

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Abstract

Polypropylene (PP) shows wide usage in the automotive industry, owing to its strength, chemical resistance, and processing ease. The movement away from petroleum-dependence has led to research into sustainable fillers to partly replace the PP matrix, with biocarbon (BioC) being of particular interest. However, this filler commonly diminishes mechanical strength, and to address this the compatibilizer maleic anhydride grafted polypropylene (MA-PP), as well as the nanomaterial graphene nanoplatelets (GnPs), were incorporated as a second filler. It was observed that the optimal formulation was PP/17%BioC/3%MA-PP/3%GnP, which led to mechanical and thermal properties beyond that of neat PP. Flexural strength and modulus were raised by around 28 and 59%, respectively, owing to the stiffness of the filler particles and restriction of chain mobility. This restriction in chain mobility had the added benefit of enhancing the heat deflection temperature by 28%, as well as the dimensional stability, as observed through a decrease in the coefficient of linear thermal expansion by around 17% as compared to neat PP. Overall, these biocomposites provide a direction to the improvement of PP properties while simultaneously decreasing its petroleum content, making them ideal for automotive applications.

Introduction

Polypropylene (PP) is widely used in automotive applications due to its chemical and heat resistance, low density, and comparatively strong mechanical properties, with usage in dashboards, bumpers, and hoods [1]. However, PP is a petroleum-based polymer that is typically either discarded at landfills or incinerated and as such contributing to the global issue of climate change [2]. A variety of publications have analyzed the effect of biocarbon (BioC) addition as a biofiller to a PP matrix due to its cost-effectiveness, low density compared to conventional fillers (glass fiber and talc), and ability to increase the biocontent of composites in a petroleum-dominated field [3]. It has been found that high-temperature pyrolysis of waste feedstock to produce BioC is more compatible with a PP matrix, but lower temperatures have improved yield and thus are more realistic for industrial applications [4,5]. This biofiller commonly leads to drops in mechanical strength, but its high thermal stability can be seen to enhance the thermal properties of PP composites [4,6].

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To account for worsened adhesion between the PP matrix and BioC, the compatibilizer maleic anhydride-grafted-polypropylene (MA-PP) has been found to be efficient [7,8]. To further restore any lost mechanical strength of the composites, incorporation of nanofillers like graphene nanoplatelets (GnPs) have received increasing attention [9-11]. GnPs lack the presence of functional groups, making them compatible with non-polar PP matrices, as demonstrated through a study by Jun et al. [9]. This study found that smaller particle diameters (< 15 μm) had improved dispersion within the matrix, leading to stronger stress transfer interactions. However, this nanomaterial often suffers from agglomeration due to strong π-π and van der Waals interactions that serves to limit its effectiveness [10].

This study is based on our recent work, which was the first study to characterize PP composites containing both BioC and GnPs [11]. The goal of this research was to develop cost-effective composites at an elevated bio content that enhanced the mechanical and thermomechanical properties beyond that of neat PP. It is expected that the hybridization of BioC's thermal stability and the mechanical strength of GnPs have the potential to accomplish this. A brief characterization of the soy hull BioC has been performed, along with the morphological (SEM and TEM), mechanical (flexural properties), and thermomechanical characterization of the fabricated composites.

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Experimental

The process of biocomposite fabrication has been outlined in Scheme 1. The PP (grade PP1350N) was purchased directly from Pinnacle Polymers (USA), while MA-PP (Fusabond P353) was purchased from Dupont (USA), and the GnPs (grade M5) were purchased from XG Sciences Inc. (Lansing, MI, USA). The biocarbon powder was prepared through pyrolysis of soy hull (supplied by Nieuwland Feed, Drayton, ON, Canada) at 500oC with a heating rate of 7.5 °C/min and residence time of 1 h in a GLO Carbolite 10/11-1G pyrolyzer with an inert nitrogen atmosphere. Following pyrolysis, the biocarbon was collected and ball-milled for a total of 2 hr. Materials apart from the hydrophobic PP pellets were dried prior to pyrolysis, and then mixed according to the specified composite formulation. This material then underwent extrusion and injection using a Microcompounder DSM (Netherland) at 180oC to fabricate flexural bars conforming to ASTM D790 standards. These fabricated composites then underwent mechanical and thermomechanical testing to examine their material properties. Thermogravimetric analysis coupled to Fourier transform infrared spectroscopy (TGA-FTIR) was applied under inert gas (N2 gas) with a heating rate of 20 ml/min for soy hull powder, using a TA 5500 connected to a FTIR Nicolet iS20 from Thermoscientific, USA. Scanning electron microscopy (SEM) of the cryofracture samples was accomplished using a SEM Desktop Phenom ProX Microscope (Netherlands) at 15 kV. Transmission

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electron microscopy (TEM) of the hybrid composite was analyzed using a TEM 2010F Joel Microscope at 200kV. Heat deflection temperature (HDT) measurements of the composite samples were performed with a load 0.455 MPa using TA Instruments (TA Q800) according to ASTM 648 standard. The coefficient of linear thermal expansion (CLTE) of the PP composite samples were analyzed in the flow direction (FD) with a force 0.05 N and heating rate of 5 oC.min-1 using TA Instruments (TA Q400).

Results and Discussion

TGA-FTIR analysis of Soy hull gases

To determine the environmental impact of the pyrolyzed BioC for industrial-scale applications, TGA-FTIR analysis was conducted to examine the release of volatile compounds. The gas products start to evolve at around 253oC and finished at around 600oC, as observed in Figure 1. The major volatile shifts identified included water/alcohols, hydrocarbons, carbon monoxide, carbonyl compounds, ether compounds, and carbon dioxide. The strongest absorption peak correlated to carbon dioxide, which constituted over 64% of the released volatiles during the pyrolysis process. There were other minor contributions by carbonyl compounds (~15%), ether compounds (~8%) and water/alcohols (~6%). A promising aspect for the usage of soy hull feedstock is that carbon monoxide emissions from the pyrolysis process are very low (~1%).

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Scheme 1: Preparation of PP hybrid biocomposites.

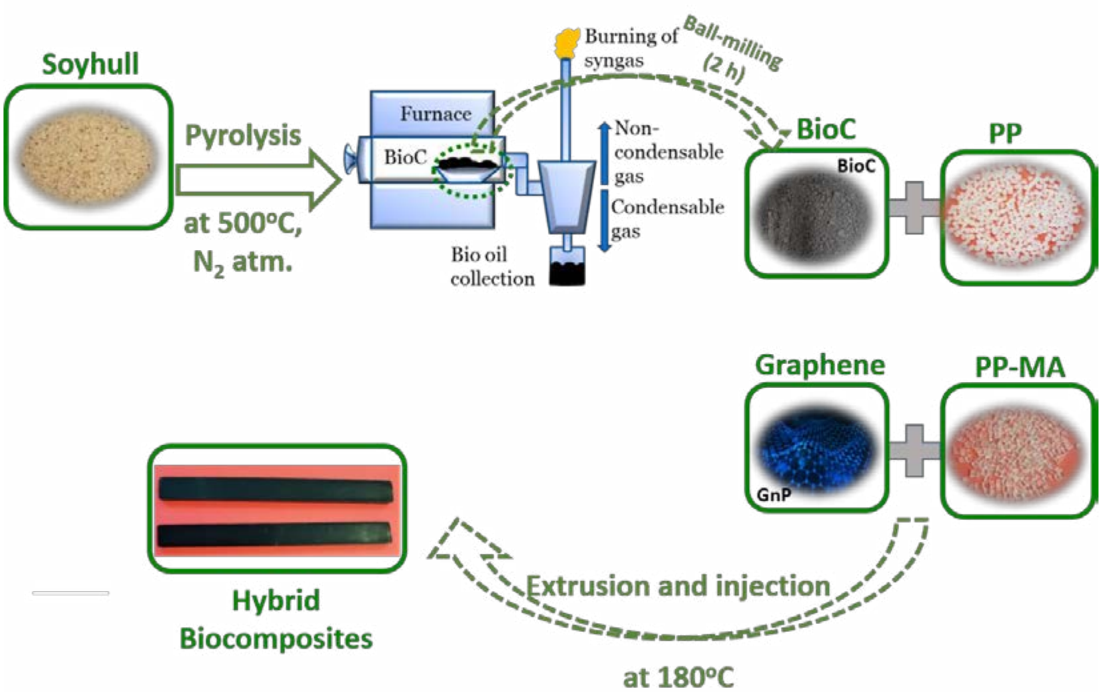

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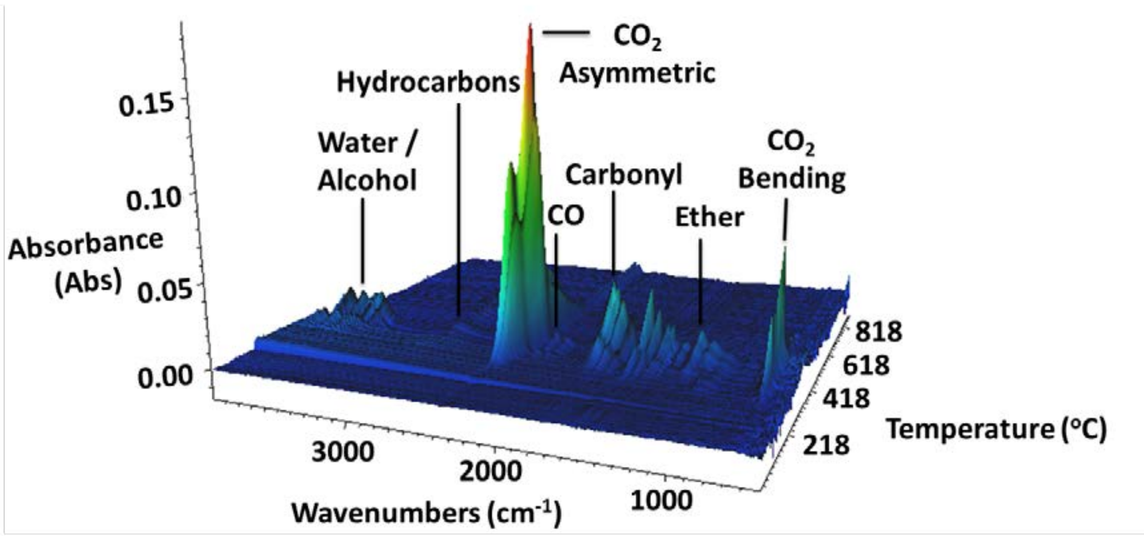
Figure 1: TGA-FTIR *generated soy hull 3D surface plot, with relevant released volatile compounds indicated* [11].



Composite Morphology

To examine the effect of filler addition on a PP matrix, scanning electron microscopy (SEM) analysis was employed to image the cryo-fracture interface. As expected, based on previous studies, the addition of 20 wt.% BioC in Figure 2a showed clear signs of phase separation and post-fracture particle pull-outs. This arose due to an absence of interactions between the polar BioC filler and non-polar polymeric matrix, and was partly recovered via compatibilizer incorporation, as identified in Figure 2b. It was determined based on mechanical and thermomechanical characterization that the optimal GnP incorporation was at 3 wt.%, with a composite formulation of PP/BioC/MA-PP/GnPs at loadings of 77/17/3/3 wt.%. This composite is visible in Figure 2c, and due to the 6-8 nm thickness of individual GnP layers, the identified graphene layers constitute a multitude of stacks that appeared poorly dispersed within the PP matrix. This agglomeration was attributed to intermolecular forces and residual moisture content within the nanomaterial. However, due to the nanoscale of this filler, it is more appropriately characterized through transmission electron microscopy (TEM), as in Figure 2d. This further reinforced the outcomes seen through SEM imaging, where agglomeration of nanoplatelets were recognized.

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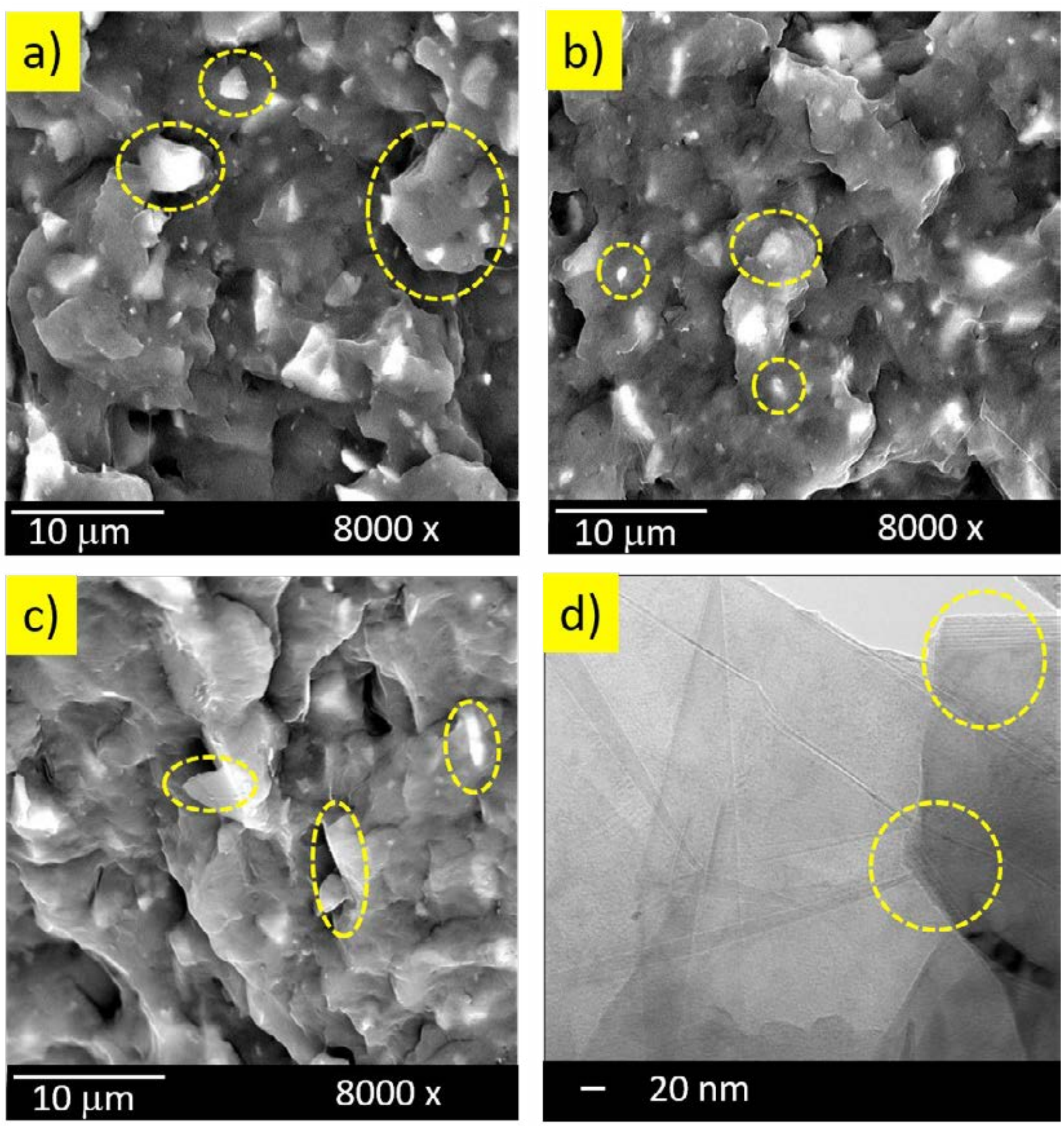
Figure 2: SEM micrographs of cryo-fractured composites: a) PP/20%BioC, *b)* PP/20%BioC/3%MA-PP *and c)* PP/17%BioC/3%MA-PP3%GnP. *d)* TEM *micrographs of* PP/17%BioC/3%MA-PP3%GnP [11].



Flexural Properties

Flexural properties of neat PP and the fabricated biocomposites can be seen in Figure 3. Surprisingly, it was observed that the addition of BioC led to an improved flexural strength, unlike tensile strength as observed in a previous study [11]. Compatibilizer addition led to an increase in flexural strength due to improved bonding within the matrix, which can resist elastic deformation. Even a minor addition of GnPs led to a comparatively large increase in strength. These improvements in flexural strength may be due to the stiffness of both the GnP sheets and BioC particles. BioC had the benefit of being well dispersed within the matrix, and its particles aided deformation resistance while under load. In comparison, natural GnPs are

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far stiffer than BioC due to their two-dimensional hexagonal lattice structure, meaning even small incorporations serve to enhance mechanical properties [12]. The modulus also saw improvement through filler addition, as the stiff particles restricted mobility of the PP matrix. BioC and GnP have both been seen to have this effect on PP, while MA-PP had little effect on modulus [13,14]. As seen, the fabricated biocomposites have a flexural strength and modulus that has been improved by up to 28 and 59% as compared to neat PP, respectively.

Figure 3: Flexural properties of PP *hybrid biocomposites*

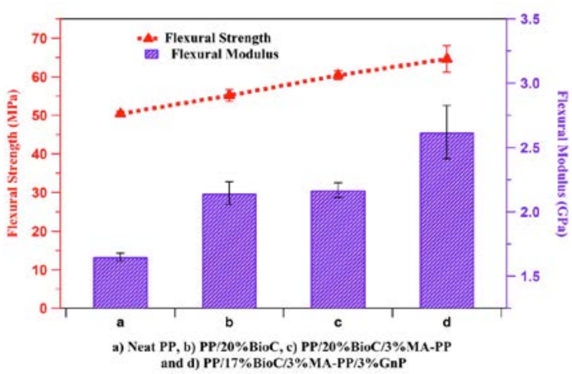
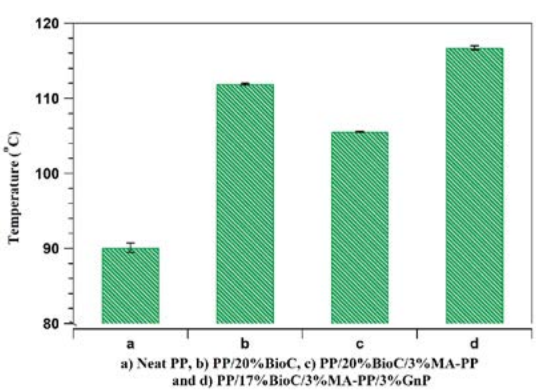


Figure 4: HDT *of* PP *hybrid biocomposites*.



Thermomechanical Characterization

Heat Deflection Temperature (HDT)

HDT, being a description of heat resistance, is an important property for automotive composites as thermal stability must be maintained over extended use in warm environments. The HDT values of all fabricated biocomposites can be observed in Figure 4, and neat PP saw a large increase by 21oC through BioC addition. This derives through PP chain mobility restrictions as deformation is thus hindered, and this effect has been observed elsewhere [6,14,15]. The rubbery nature of MA-PP led to a subsequent decrease in HDT, but was improved through further chain restrictions from GnP addition. This is a very promising property of the biocomposites, with the optimal formulation leading to a 28% increase in HDT.

CLTE Analysis

CLTE is another property that indicates dimensional stability of the composites, as it describes how material size changes at varying temperatures. A high CLTE is not desirable in automotive applications. There are two major regions of the CLTE, being glassy (measured from -30 to 30oC) and rubbery (-30 to 100oC) states. As seen in Figure 5, BioC addition (20 wt.%) resulted in a reduction in the CLTE value in the rubbery state by 18.5% as compared to neat PP, which underwent another 7.6% decrease upon GnP inclusion. It can be concluded from this that the well-dispersed BioC particles served to hinder polymeric movement, which supports HDT data and corresponds to findings by other studies [1,13,15]. While not having a major impact in the rubbery state, MA-PP resulted in a minor reduction in CLTE in the glassy state, which was attributed to improvements in bonding between the biofiller BioC and the matrix. GnPs have a naturally low coeffi-

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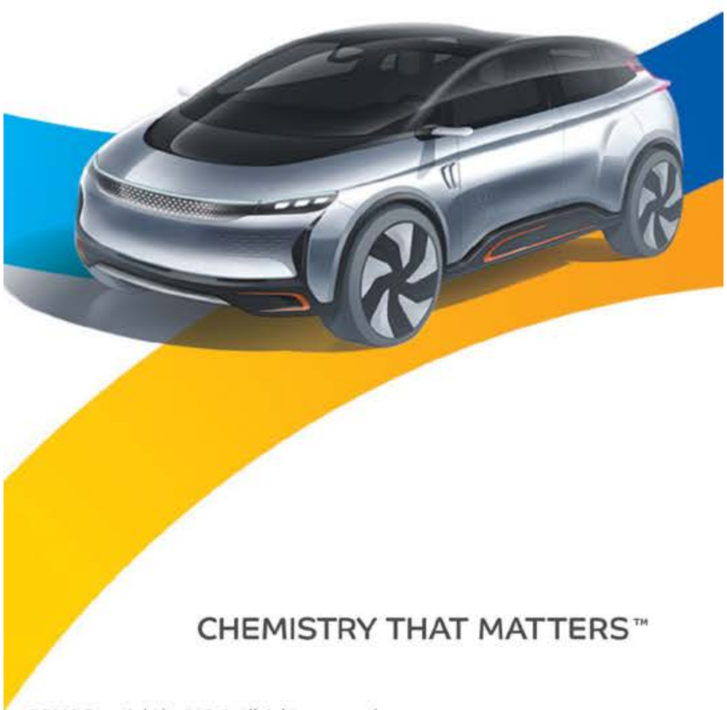
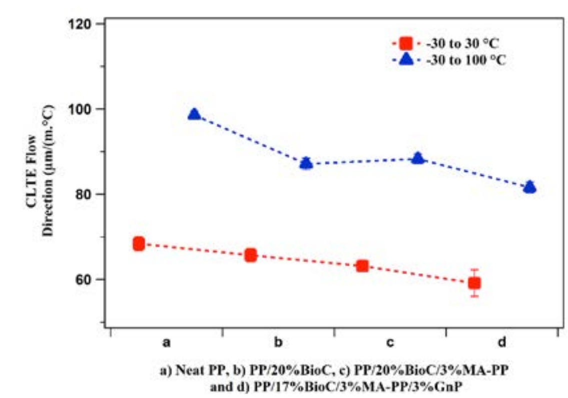


cient of thermal expansion and a high surface area, which serves to explain the improvements it had on CLTE [14]. The optimal biocomposite had a CLTE that was reduced by 25% as compared to neat PP, indicating that it has a higher dimensional stability.

Summary and Next Steps

BioC and GnP was successfully incorporated into a PP matrix to form biocomposites, with an optimal formulation of PP/17%BioC/3%MA-PP/3%GnP. Poor interaction between the biofiller and PP matrix was improved through compatibilizer incorporation, while GnPs showed signs of agglomeration due to intermolecular forces. Despite this, the biocomposites still saw significant increases in both mechanical and thermomechanical proper-

Figure 5: CLTE of the PP hybrid biocomposites in the flow direction.



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ties. Flexural strength and modulus improved beyond neat PP by 28 and 59%, owing to the stiffness of both BioC and GnP. The chain restrictions imparted through filler addition led to a subsequent improvement in HDT by up to 28%, as well as a stronger dimensional stability as measured through CLTE analysis. These biocomposites show great promise for automotive applications, as they have improved mechanical strength, as well as thermal stability for heat-intensive applications at an elevated biocontent. Based on the results obtained, these composites would benefit through future study on GnP dispersion methods. Agglomeration partially limited the benefits GnPs can provide on both mechanical and thermal properties, and proper dispersion could lead to superior biocomposites.

Acknowledgements

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Board Meeting Minutes June 16, 2020

By: John P. Busel

SPE Composites Division Board of Directors Meeting

Tuesday, June 16, 2020

12:00 PM – 1:30 PM Eastern US

Conference Call

1. Welcome

- Ian Swentek called the meeting to order at 12:06 pm.
- John Busel conducted the Roll Call.

2. Administrative

- Ian Swentek reviewed the last meeting minutes. Enamul Haque moved to accept the last meeting minutes of March 17, 2020 - Conference Call as written. The motion was seconded. No discussion. Motion passed.
- Ian Swentek reviewed the action items from the last meeting. See Attachment 1 for status. He asked those who complete their action items or if there are problems, please contact Ian Swentek directly.
- Ian Swentek reviewed the work and changes to the vision statement. John Busel moved to approve the proposed vision and mission statement as written. Alex Kravchenko seconded. No discussion. Motion passed. Ian Swentek will coordinate with the website chair and update the bylaws for sharing at the next meeting.

3. Treasurers Report

- Tim Johnson reviewed the distributed current financial report and proposed FY 20/21 Budget. The budget was prepared because revenue from ACCE would be breakeven. It was reported that the steering committee is looking at several options including to hold ACCE virtually. It was agreed that assuming revenue neutral for ACCE is a good starting point. Each budget line item was reviewed.
- The Board discussed the budget line items. Jim Griffing moved to accept the FY 20/21 budget as proposed by the Treasurer Tim Johnson. Fred Deans seconded. Motion passed.
- Ian Swentek acknowledged the work of Tim Johnson as Composites Division Treasurer, but he would like to step down by September. He asked for volunteers to help during the transition. Ian Swentek will contact each board member directly to solicit a new treasurer. It was suggested that hiring an outside consultant should be considered.

4. ACCE Support

- Ian Swentek reported that a letter was sent to the Automotive Division regarding the position of the Composites Division with ACCE. The letter was well received. The 2021 program will have a joint leadership and co-chair for ACCE. The Board will need to determine who will be the Composites Division representative for ACCE. Ian Swentek reported it is almost certain that ACCE will become a virtual event. Dale Brosius reported that the Implications are being evaluated by the conference steering committee. The members added additional information regarding issues being discussed for the ACCE event. Sponsorships for the changed event will be an important consideration. It was noted that technical presentations might be smaller than planned if the event goes virtual. It was suggested that there is the potential for each division to absorb expenses if there is not enough income. Dale Brosius will distribute an overview of the changed event for consideration by material suppliers.

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Board Meeting Minutes continued...

5. Committee Reports

- Audit: Ian Swentek reported that the committee was very satisfied with the investigation of the Division financials. Ian Swentek shared 3 recommendations for Board consideration. One, we need a co-treasurer to learn the process during the transition to a new treasurer. Two, ensure alignment between our budget, bank statements, and tax forms, specifically how the investment is reported. Three, need to capture the standard practice in the form of a procedure document. This will provide additional security and consistency with the finances. The ComDiv Policy Manual stipulates the Audit committee perform the audit annually, but this action was not done for several years. Michael Connelly moved to accept the audit report as presented. Alex Kravchenko seconded. Motion passed.
- Newsletter: Pritam Das reported there are some decisions the Board needs to make to improve newsletter sponsorship. He referenced his committee report distributed prior to the meeting and reviewed the options.
  - o Distribute the newsletter to a new audience – Contact was made the Automotive and Thermoset Divisions. Automotive was receptive to redistribution. The process might be reciprocal with the Division.

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This Issue:

Board Meeting Minutes continued...

o Showcase sponsor logos.

o Eblast sponsor's events and new product launches to members. – The Board agreed that if these were educational webinars, that is better than a product launch. This would be one eblast per year.

o Develop a new mode of distribution with creation of a QR code to allow for more efficient distribution. More work is needed.

- Awards: Hicham Ghossein reported that sponsorships for the Educator of the Year Award is not resolved. He reviewed the process of the SPE Foundation and funding for the Jackie Rehkopf Scholarship. He recommended no changes in Division support at this time. It was pointed out that the record of contributions provided by the Composites Division needs to be corrected and reflected that the $6K comes from the Composites Division. Hicham Ghossein to prepare a 1-page overview of all the awards that could be distributed to a contact list.
- Membership: Ray Boeman reported that the division has slightly under 500 members. The category breakdown has not changed much since the March 2020 report. It was suggested that there might be SPE members that are not connected to the Composites Division. John Busel volunteered to help with the membership committee.

6. Wrap Up

- Ian Swentek will add those reports not reviewed during this meeting will be added to the next scheduled meeting.
- Ian Swentek will compile the Action Items and will be distributed with the minutes.
- The Next Meeting is scheduled for September 2020 as a conference call. Date and time to be determined.

7. Adjourn

- Ian Swentek adjourned the meeting at 1:32 pm.

Respectfully submitted,
John P. Busel
Secretary/Chair-Elect, SPE Composites
Division Board of Directors

Attendees

OFFICERS:
Ian Swentek, Chair
Tim Johnson, Treasurer
John P. Busel, Secretary/Chair-Elect
Dale Brosius, Councilor
Ray Boeman, Past Chair
DIRECTORS:
Dan Buckley
Rich Caruso
Michael Connolly
Pritam Das
Fred Deans
Hicham Ghossein
John Gillespie
Jim Griffing
Enamul Haque
Alex Kravchenko
Christoph Kuhn
Marcos Pantoja
Andy Rich
Antoine Rios
Khaled Shahwan
Shankar Srinivasan
Uday Vaidya

This Issue:

How To Learn Mold Design Virtually

Davide Masato, Stephen Johnston

– Department of Plastics Engineering, University of Massachusetts Lowell

The Plastics Engineering program at the University of Massachusetts Lowell has been teaching mold design and engineering to undergraduate students for over 60 years. The Mold Engineering class is one of the most unique and appreciated classes in the curriculum. Over the 13 weeks of a typical semester, teams of 5-7 students (~8 teams per semester) usually have the opportunity to design a plastic part, analyze and design the mold, manufacture soft prototype tooling, and ultimately produce parts by injection molding. Students are usually required to run all the machines, under the supervision of instructors and teaching assistants, so they can learn how mold manufacturing processes constrain the design of plastic parts. Examples of projects and molds manufactured by students include UMass Lowell-themed gadgets, toys, and many new fun and useful parts. The hallmark mold design project has been a significant part of the class for over a decade, and the quality of parts produced by students has increased to the point that our Student Chapter of the Society of Plastics Engineers (SPE) now manufactures and sells them at the UMass Lowell Bookstore with profits supporting student activities. Moreover, according to the recent ABET feedback, the Mold Engineering project was confirmed to be the premiere design experience in the undergraduate curriculum.

This year the unexpected arrival of the COVID-19 pandemic in mid-March forced us to revisit our class program and objectives. Similar to other academic courses, the class became virtual. This meant redesigning an intensive hands-on class into one that could be taught and taken from our home offices while maintaining academic rigor and continuing to meet critical student learning objectives. The timing of the pandemic meant that students, who were completing tooling split designs and starting CNC programming, could not move forward with machining, assembly, and molding. Instead, their projects became virtual learning experiences. Here we take the opportunity to share ideas implemented in our teaching to introduce young engineers to the world of injection mold design…virtually.

When the pandemic arrived, teaching a hands-on design class and making it a significant virtual experience for students posed many challenges. The teaching staff had to work hard to pivot the class, with one week's notice, into a rigorous and interactive design experience. The hands-on component of the Mold Design class is so important and unique, representing the opportunity for plastics engineering undergraduates to blossom as design engineers. Understanding we could not replicate that exact experience, we built up other elements of students' evolution as designers. We decided to pivot the class to focus on: 1) detailed mold design with supporting hand calculations, 2) further and more advanced CAE analysis, and 3) CAM generation with fully virtual validation. Changing the requirements and expectations for the team projects pushed students to perform engineering analyses of their prototype molds and use state-of-the-art CAD, CAE, CAM, software for quick design iterations, and effective reporting. Further, the use of hand calculations and verification of

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This Issue:

Mold Design Virtually continued...

simulation results reinforced understanding of the design details. This happened while still working in teams and having to meet numerous deadlines and toll-gates in the design process.

Through this pandemic, we have learned new approaches to teaching mold design that we would not have had the opportunity to explore under normal circumstances. The virtual delivery of lectures and lab activities through video recording technology allowed us to provide students the same practical demonstrations and real-world examples that we would typically use in class. This included video lectures on theory, examples of real molds and components, and demonstrations of machining operations. Similarly, software tools typically taught in a computer lab were instead taught in a series of application-specific video tutorials.

Overall, virtually teaching a subject that relies on significant laboratory experiences such as mold machining, polishing, assembly, and molding has certainly proven to be a challenge. Instructors and students were challenged to pivot into a new format that placed greater responsibility on everyone involved. The response from the students was outstanding, and they demonstrated their ability to meet deadlines, communicate professionally, maintain a connection with team members, and deliver virtual technical presentations. This has been remarkable, considering the responsibility and discipline required by virtual learning.

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Sponsor the Newsletter

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7" x 10", 8.5" x 11" $1,250

Contact: Teri Chouinard CBC, APR
SPE Composites Division
Sponsorship Chair
C/O Intuit Group, Inc.
phone 248.701.8003
Teri@IntuitGroup.com

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Please provide Ads as High Resolution PDF files
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- **Also reach 1,070 SPE Composites Division Linked In Members 3 times a year and an additional 11,492 COMPOSITES Linked In Members Secondary group where the link to the newsletter is posted year-round**
- **Reach many more as a sponsorship also includes your logo on our website, www.composites.4speorg with a link to your company**
- **Maximize your exposure to the customers & the trade**
- **Stay informed on the latest composites activity**

Increase your presence on the web leading to more sales by sponsoring our Electronic Newsletter which is published on the SPE Composites Division Website and emailed to all Division Members (1,000 approx.) 3 times annually. Rates include 3 issues (not on calendar basis - published approx. Nov/Dec, Mar/April, July/August). All ads include a link to your website increasing your exposure on the worldwide web exponentially. Sponsorship also includes your logo ad with a link to your website on www.composites.4speorg further increasing your presence on the Web as a Leader in Composites Technology.

This Issue:

Mold Design Virtually continued...

Given the circumstances, the overall experience was positive and productive, and aspects of the virtual class will be incorporated into future on-campus offerings. For example, our new archive of video lab tutorials could be used to prepare students for on-campus labs so they arrive more prepared to discuss their team's mold designs. However, this new virtual experience does not replace the hands-on experience that we want our students to have in a mold design class. As instructors and passionate mold designers, we cannot wait to be back in labs with the class, helping everyone succeed with their projects. We want our students to smell the cutting fluid as the CNC machine manufactures their inserts, feel the grit as they polish their inserts to a shine, hear the cadence of the molding machine running on an automatic cycle, and see their finished products in their own hands. The satisfaction that students show when, at the end of the semester, they can hand off their unique parts to the rest of the class is unique and irreplaceable. The main learning experience is manufacturing original plastic parts using mold inserts that they have created as a team from scratch. The expansion of virtual or online education, which is a timely topic, will continue to open up new opportunities for improvement; however, it is always going to be a different learning experience.

The Mold Design teaching team takes this opportunity to acknowledge the support of Dassault System, Autodesk, and Mastercam, for the continuous support with educational licenses of state-of-the-art CAD, CAE, and CAM, software. The contribution of these companies to the education of Plastics Engineers at UMass Lowell continues to make the difference. The delivery of the virtual class was made possible by the use of Zoom for online meetings, and of Camtasia for video editing of the lecture recordings.

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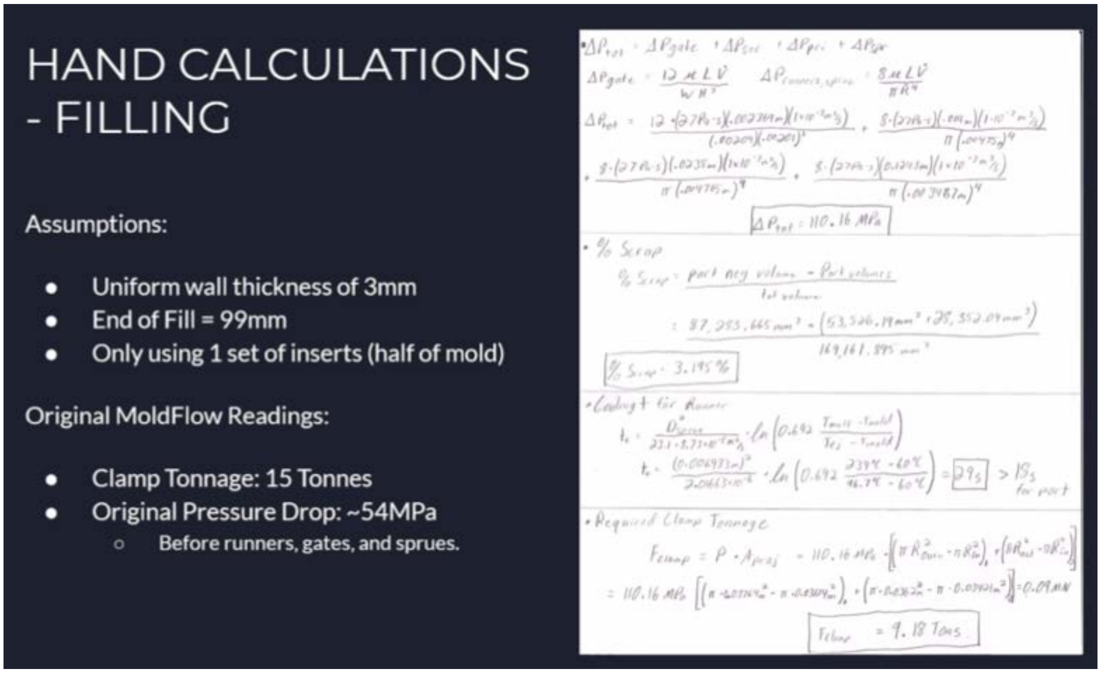
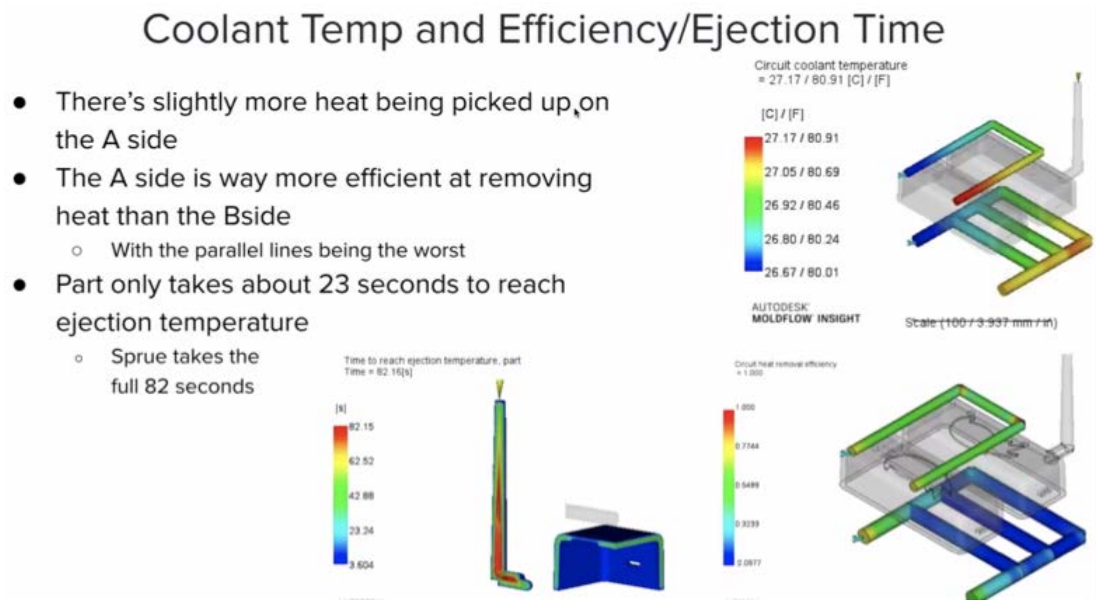
Example of supporting calculations to validate mold design. CREDIT: Popsicle Maker team.



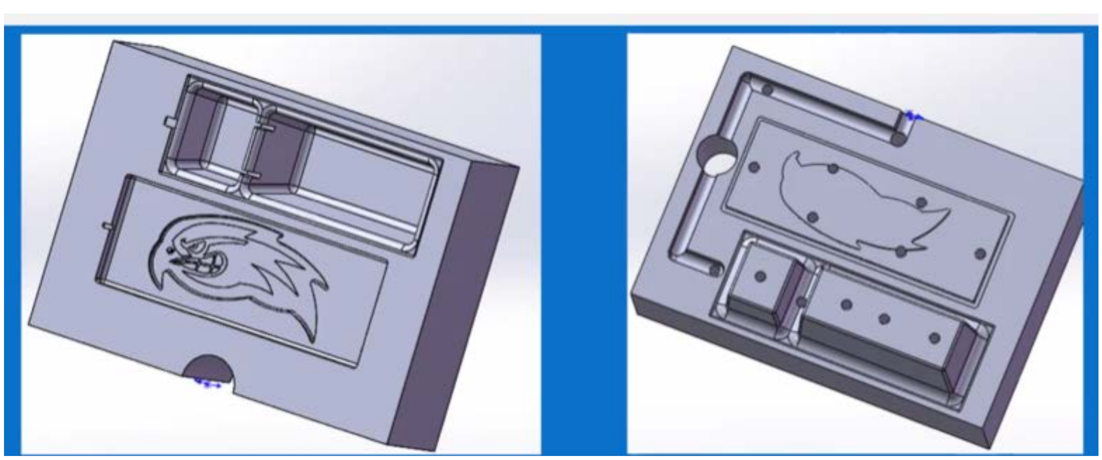
This Issue:

Mold Design Virtually continued...

Example of CAE analysis of the cooling system design. CREDIT: Popsicle Maker Team



Example of core and cavity inserts with runner layout, runner shut-off, and ejector pin locations. CREDIT: Dunkaroos Team

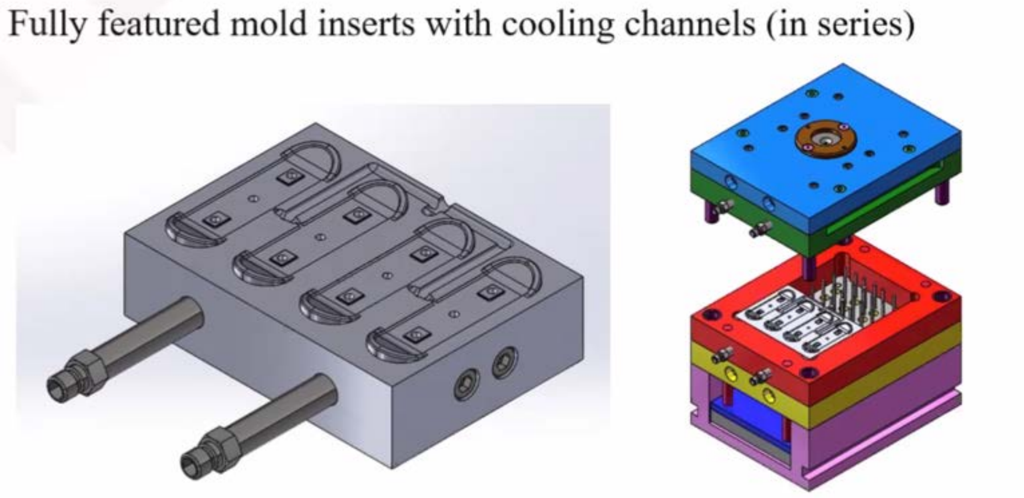


This Issue:

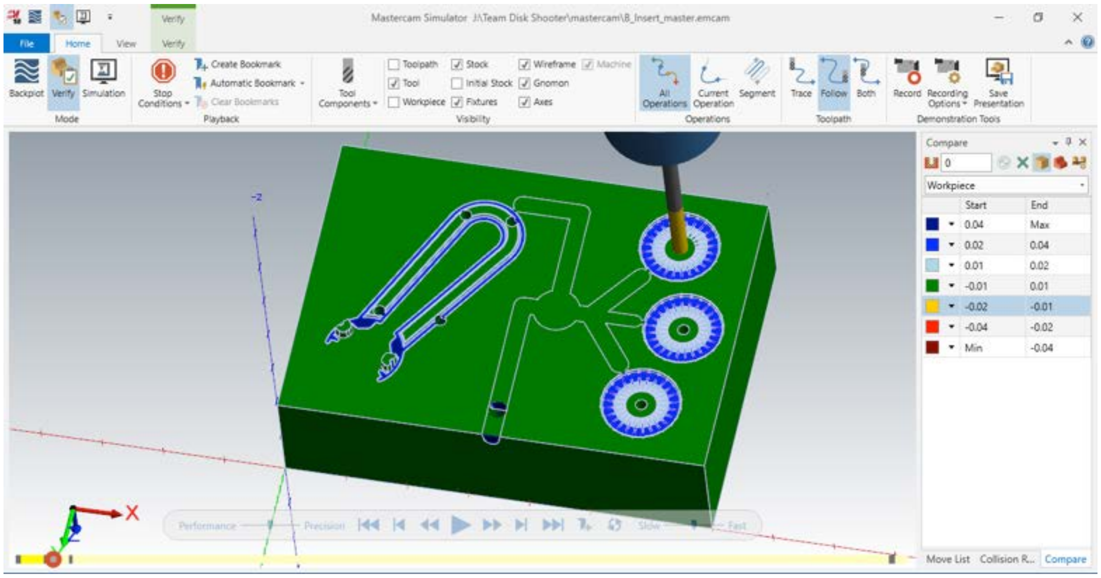
- BOD Listings
- Award Winning Paper
- Board Meeting Minutes
- Virtual Mold Design

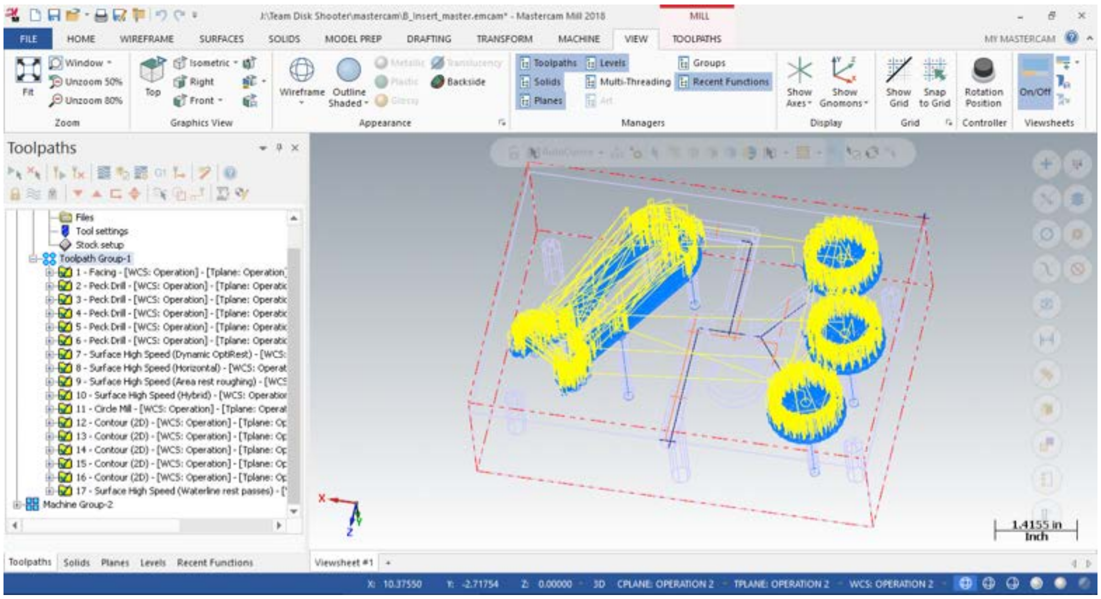
Mold Design Virtually continued...

Example of designed multi-cavity mold insert and mold base. CREDIT: RowdyBoard Team



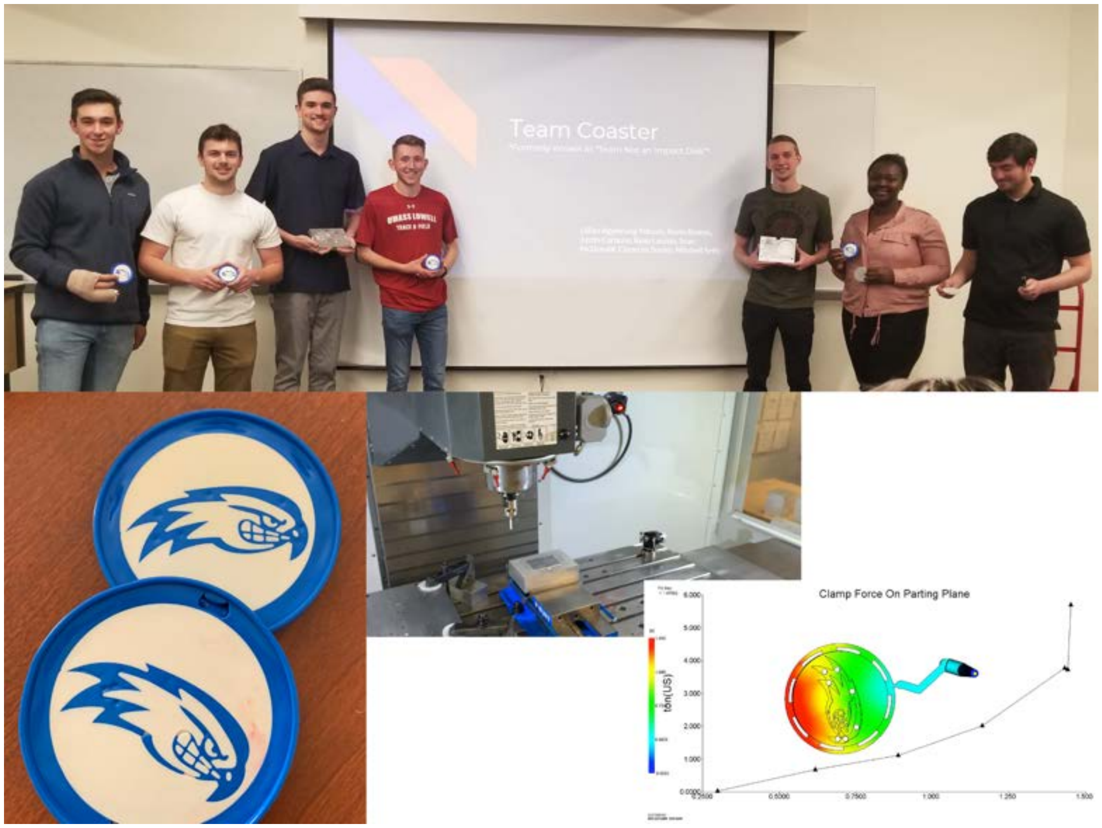
Examples of material check for the generated CAM program. CREDIT: Disc Launcher Team





Mold Design Virtually continued...

Examples of student projects in a normal year.



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This Issue: