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14. ABSTRACT The objectives of this research are: (a) To demonstrate the feasibility of processing novel transparent interpenetrating polymer networks by combining stiff and compliant polymeric constituents and (b) To mechanically characterize them under impact loading conditions. On the material processing front, novel polymer systems termed Molecular Interpenetrated Polymer Composites (MIPC) comprising of two or more networks are partially interlaced at the molecular scale to produce sheet stock. By manipulating the reaction kinetics MIPCs are tailored to possess good optical transparency as well as improved mechanical characteristics. On the mechanical					
15. SUBJECT TERMS transparent armor, interpenetrating polymer networks, graft polymers, dynamic fracture, impact failure, optical metrology, visualization and quantification					
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a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU			19b. TELEPHONE NUMBER 334-844-3327

Report Title

Final Report: Molecular Interpenetrated Polymer Composites (MIPC) for High-Strain Rate Applications: Development and Characterization of Novel Lightweight Transparent Materials

ABSTRACT

The objectives of this research are: (a) To demonstrate the feasibility of processing novel transparent interpenetrating polymer networks by combining stiff and compliant polymeric constituents and (b) To mechanically characterize them under impact loading conditions. On the material processing front, novel polymer systems termed Molecular Interpenetrated Polymer Composites (MIPC) comprising of two or more networks are partially interlaced at the molecular scale to produce sheet stock. By manipulating the reaction kinetics MIPCs are tailored to possess good optical transparency as well as improved mechanical characteristics. On the mechanical characterization front, high-strain rate failure characterization of MIPCs is critical for armor applications. Hence techniques capable of characterizing MIPCs by exploiting their optical transparency would be of great value. Accordingly, a novel, non-contact, full-field deformation measurement method to investigate fracture and failure of monolithic and layered transparent sheets under stress-wave loading conditions is developed.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
10/07/2016	17 R.K.A. Pasumathy, H.V. Tippur. Mechanical and Optical Characterization of a Tissue Surrogate Polymer Gel, Polymer Testing, (): 219. doi:
10/07/2016	19 R. Ballesterro, B. M. Sundaram, H. V. Tippur, M. L. Auad. Sequential graft-Interpenetrating polymer networks based on polyurethane and acrylic/ester copolymers, Express Polymer Letters, (): 204. doi:
TOTAL:	2

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

1. ‘Visualization and quantification of dynamic crack penetration vs. branching at a weak interface in a brittle bilayer,’ H. V. Tippur, Proceedings of the symposium on Mechanics of Materials Across Nano to Geological Time and Length Scale, Brown University, Providence, RI, Sept 2016.
2. ‘Dynamic Crack Growth in Monolithic and Bi Layered PMMA,’ H. V. Tippur, and B. M. Sundaram, Proceedings of the 17th International Conference on Experimental Mechanics (ICEM’17), Rhodes, Greece, 2016.
3. ‘Crack Path Selection During Dynamic Crack Growth Past A Perpendicularly Oriented Interface: An Experimental Study Using Digital Gradient Sensing,’ B. M. Sundaram* and H. V. Tippur, ASME-IMECE, Houston, TX, 2015.
4. ‘Dynamic Penetration and Bifurcation of a Crack at an Interface in a Transparent Bi-Layer: Visualization and Quantification,’ B. M. Sundaram* and H. V. Tippur, 52nd SES Annual Conference, College Station, TX, 2015.
5. ‘Digital Gradient Sensing: A Novel Optical Method for Investigating Transient Deformations in Transparent and Reflective Solids,’ H. V. Tippur [A. S. Kobayashi Award Lecture], International Conference on Computational & Experimental Engineering and Sciences (ICCES’14), Changwon, Korea, June 2014.
6. ‘A digital gradient sensor and its applications to study fracture and failure of materials,’ ASME-IMECE, San Diego, CA, November, 2013.
7. ‘Fracture and Impact Energy Absorption Characteristics of PMMA-PU Transparent Interpenetrating Polymer Networks,’ K. C. Jajam, S. A. Bird, M. L. Auad and H. V. Tippur, 50th SES Annual Technical Meeting and ASME-AMD Summer Meeting, Providence, RI, July 2013.
8. Keynote Presentation: ‘Visualization and Quantification Transient Deformations in Transparent Armor Materials Using a Full-field Digital Gradient Sensor’, H. V. Tippur, SEM Annual Conference, Lombard, IL, June 2013.
9. “Sequential graft-Interpenetrating polymer networks of polyurethane and acrylic/vinyl ester based copolymers,” R. A. B. Mendez, B. M. Sundaram and H. V. Tippur, M. L. Auad American Chemical Society National Meeting. Dallas, Texas, March 2014. Oral presentation.

Number of Presentations: 9.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

<u>Received</u>	<u>Paper</u>
02/16/2016	14 B.M. Sundaram, H.V. Tippur. Dynamic Crack Propagation in Layered Transparent Materials Studied using Digital Gradient Sensing Method, SEM Annual Conference. 08-JUN-15, . . ,
08/25/2014	6 Balamurugan M. Sundaram, Hareesh V. Tippur. Dynamic Crack Propagation in Layered Transparent Materials Studied using Digital Gradient Sensing Method , Society for Experimental Mechanics Annual Conference. 06-MAR-14, . . ,
TOTAL:	2

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

<u>Received</u>	<u>Paper</u>
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02/16/2016	15.00	B.M. Sundaram, H.V. Tippur. A DIGITAL GRADIENT SENSOR FOR INVESTIGATING DYNAMIC FRACTURE OF TRASPARENT LAYERED MATERIALS, 16th Int Conference on Experimental Mechanics (ICEM16). 08-JUL-14, . : ,
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TOTAL: 1

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Manuscripts:

Books

<u>Received</u>	<u>Book</u>
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TOTAL:

Received

Book Chapter

TOTAL:

Patents Submitted

PROVISIONAL US PATENT APPLICATION NO.: 62/029,100, TITLE: "Graft Interpenetrated Polymer Networks (G-IPN)," INVENTORS: ~~M. L. Auad, H. V. Tippur, R. A.B. Mendez and B. M. Sundaram, 2014.~~

Patents Awarded

Awards

Tippur:

1. F. G. Tatnall Award, Society for Experimental Mechanics, 2016
2. Orr Best Paper Award, ASME-Materials Division, November 2014
3. A. S. Kobayashi Award, 2014 ICCES, Changwon, Korea, July 2014
4. Keynote Speaker, Int. Conf. Experimental Mechanics 2013, Bangkok, Thailand, Nov 2013
5. Keynote Speaker, SEM Annual Conference, June 2013

Auad:

1. 2015 Women of Distinction, Faculty Leadership Awards, Auburn university Women's resource Center.
2. Gold award for graduate technical paper/oral competition. for paper "Sequential graft-Interpenetrating polymer networks of polyurethane and acrylic/vinyl ester based copolymers", R. A. B. Mendez, B. M. Sundaram and H. V. Tippur, M. L. Auad. Society of Hispanic Professional Engineering Conference. Detroit, Michigan, November 6, 2014.
3. Creative Mentorship Award for Undergraduate Advising, Auburn University, 2015

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
B. M. Sundaram	1.00	
R. A. B. Mendez	1.00	
FTE Equivalent:	2.00	
Total Number:	2	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
Hareesh V. Tippur	0.07	
Maria L. Auad	0.13	
FTE Equivalent:	0.20	
Total Number:	2	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Mathew Halvorson	0.03	
FTE Equivalent:	0.03	
Total Number:	1	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 1.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 1.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 1.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

Names of Personnel receiving masters degrees

<u>NAME</u>
R.A.B. Mendez
A. Jain
Total Number:

Names of personnel receiving PHDs

<u>NAME</u>
Total Number:

Names of other research staff

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See Attachment

Technology Transfer

- Some of the results pertaining to DGS methodology were communicated to Dr. Tusit Weerasooriya (RDRL-WMP-B).
- Dr. Constantine (Costas) Fountzoulas at ARL has a strong interest in modeling some of the fracture behaviors observed by the PI. A conference presentation that included aspects of this research was made during 2015 SEM Annual Conference held at Costa Mesa, CA.
- The PI was invited to ARL in Sept 2014 to discuss potential advantages of DGS for transparent armor materials research. Since then, there has been an expressed interest to utilize DGS to study (a) cavitation in gels (Dr. Sikhanda Satapathy, Soldier Protection Branch, ARL) (b) crack growth in polymers and ceramics (Dr. Chris Meredith, Impact Physics Branch, ARL)

**Final Report – Grant # W911NF1210317 (Proposal No. 60172EG)
(Reporting Period: September 2012 – August 2016)**

**Molecular Interpenetrated Polymer Composites (MIPC) for High-Strain Rate Applications: Development and Characterization of
Novel Lightweight Transparent Materials**

Hareesh Tippur (PI), Department of Mechanical Engineering
Maria Auad (co-PI), Department of Chemical Engineering
Auburn University, AL 36849

Objective

The main objectives of this research are: (a) To demonstrate the feasibility of processing novel transparent interpenetrating polymer networks by combining stiff and compliant polymeric constituents and (b) To mechanically characterize them under impact loading conditions. On the material processing front, novel polymer systems termed Molecular Interpenetrated Polymer Composites (MIPC) comprising of two or more networks are partially interlaced at the molecular scale to produce sheet stock. By manipulating the reaction kinetics MIPCs are tailored to possess good optical transparency as well as improved mechanical characteristics. On the mechanical characterization front, high-strain rate failure characterization of MIPCs is critical for armor applications. Hence techniques capable of characterizing MIPCs by exploiting their optical transparency would be of great value. Accordingly, a novel, non-contact, full-field deformation measurement method to investigate fracture and failure of monolithic and layered transparent sheets under stress-wave loading is developed.

Approach

The feasibility of processing transparent “interpenetrating polymer networks” (IPNs) by combining stiff (acrylic) and tough (polyurethane) constituents in different proportions, both polymerized simultaneously in the presence of each other, was previously demonstrated by the investigators. By controlling the reaction kinetics, phase separation issues were overcome in order to preserve optical transparency of the IPN. However, due to difficulties with scaling up material processing to produce samples with testable scale, an epoxy-based material system cured with an acrylate co-monomer in combination with a compliant polyurethane phase was developed in the current work. These novel polymer networks, called MIPCs, can be swelled in solvents without dissolving and possess good creep and flow behavior. The presence of one rubbery phase and one glassy phase produce a synergistic effect leading to impact resistant characteristics. In the first component of this research, the reaction kinetics, weight fraction of the components, and molecular weight of the constituents are among the issues investigated.

In the second facet of this research, high strain-rate failure behavior of transparent materials in the monolithic and layered configurations is studied under impact loading using full-field optical methods and high-speed imaging techniques. A new optical technique based on the digital image correlation method and the elasto-optic effect, exhibited by transparent solids, has been developed for studying MIPCs alongside currently fielded transparent materials. This new methodology complements existing experimental mechanics methods for material characterization at elevated rates of loading.

Relevance to Army

Impact-resistant transparent materials are in high demand in a wide variety of military and civilian applications. Examples include protective visors such as eyewear and face shields, transparent armor, blast resistant windows, vehicular windshields, aircraft canopies, explosive ordinance disposal shields, etc. Among the polymers used in these applications polycarbonate (PC) and poly(methyl methacrylate) (PMMA) have been the most widely used lightweight transparent materials for blast and shock protection over the past several decades. This research examined the feasibility of alternative materials and methods to characterize them under high-strain rate loading conditions. This research will also help train research personnel on these topics of significant engineering relevance.

Accomplishments

- The feasibility of producing tough and highly transparent materials by synthesizing MIPCs (graft-IPNs) has been demonstrated. They were synthesized using an elastomeric polyurethane phase and a stiff acrylate-base copolymer phase. A high degree of transparency (85%) is achieved in the materials by successfully generating crosslink points between the stiff and ductile phases. The generation of chemical bonds between the two networks minimized phase separation as validated by TEM and SEM images and dynamic mechanical analysis (DMA). The MIPCs also showed an increase of quasi-static fracture toughness, by as much as ~150%, when compared to commercially procured PMMA.
- In order to further improve the fracture toughness, a free-radical terpolymerization approach was used for synthesizing MIPCs. Here, the stiff phase was chemically modified by adding a third monomer. Quasi-static fracture tests showed approximately 25% increase when compared to the previous MIPC system and over 300% when compare to commercially procured PMMA. The DMA showed that an increase in the stiffness of the samples. This is a highly desirable result since an increase in the stiffness of the system generally leads to lower fracture toughness and vice versa.
- To mechanically characterize transparent planar solids, a new digital image correlation based optical method used in conjunction with elasto-optic principles called the ‘Digital Gradient Sensing’ (DGS) has been developed. The method can accurately measure small angular deflections ($\sim 10^{-5}$ radians) of light rays related to the local state-of-stress as two orthogonal in-plane stress gradients. The method has been extended to mechanical characterization of transparent materials under impact loading conditions.
- DGS has been used to study fracture mechanics of transparent acrylics under static and dynamic loading conditions. Edge notched sheets of PMMA have been studied under symmetric three-point bending and one-point impact loading conditions. Full-field stress gradient fields near mode-I cracks, before and after crack initiation, have been mapped using DGS in two orthogonal directions (normal to and along the crack) to extract stress intensity factor histories.
- DGS has been used next to investigate the crack growth behavior in layered transparencies. PMMA bilayers prepared using acrylic adhesives to examine the interaction of a dynamically growing mode-I crack with the adhesive layer. The primary interest was to examine

the role of adhesive strength on fracture path selection within the interface and in the second layer. The interface caused the crack to branch and then trigger globally symmetric and locally mixed-mode growth into the second layer. Optical measurements suggest that a weaker interface dissipated more fracture energy relative to the stronger counterpart along with a significant crack speed reduction.

- Further studies on bi-layered transparent sheets with a weak interface, normal to the crack path in the first layer, was undertaken by varying the location of the interface relative to the initial crack tip. By doing so, different crack speeds and hence stress intensity factors at impingement on the interface were created. Experiments show that the in-coming crack penetrates the interface without branching in some configurations whereas varied amounts of configurationally symmetric interfacial growth along the interface before penetration into the second layer (as two separate mixed-mode cracks) with global symmetry occur in others.
- DGS was used to evaluate the dynamic fracture performance of MIPC sheets of different amounts of copolymer to PU ratios ranging from 60:40 to 80:20. Due to difficulties in processing sufficiently large sheets for dynamic fracture studies only a few experiments were successful. These limited results show that MIPCs offer enhanced dynamic fracture response; for example, 75:25 graft-IPN showed a 100% improvement in dynamic crack initiation toughness when compared to the commercially procured PMMA.

Collaborations and Technology Transfer

- Some of the results pertaining to DGS methodology were communicated to Dr. Tusit Weerasooriya (RDRL-WMP-B).
- Dr. Constantine (Costas) Fountzoulas at ARL has a strong interest in modeling some of the fracture behaviors observed by the PI. A conference presentation that included aspects of this research was made during 2015 SEM Annual Conference held at Costa Mesa, CA.
- The PI was invited to ARL in Sept 2014 to discuss potential advantages of DGS for transparent armor materials research. Since then, there has been an expressed interest to utilize DGS to study (a) cavitation in gels (Dr. Sikhanda Satapathy, Soldier Protection Branch, ARL) (b) crack growth in polymers and ceramics (Dr. Chris Meredith, Impact Physics Branch, ARL)

Resulting Journal Publications

- ‘Dynamics of Crack Penetration vs. Branching at a Weak Interface: An Experimental Study,’ B. M. Sundaram and H. V. Tippur, Journal of the Mechanics and Physics of Solids, Vol. 96, pp 312-332, 2016.
- ‘Mechanical and Optical Characterization of a Tissue Surrogate Polymer Gel,’ R.K.A. Pasumarthy and H.V. Tippur, Polymer Testing, Vol. 55, pp 219-229, 2016.
- ‘Dynamic Fracture of Bi-Layered Transparent Sheets: Optical Investigation of Crack-Interface Interactions and Crack Branching using Digital Gradient Sensing Method,’ B. M. Sundaram and H. V. Tippur, Experimental Mechanics, Vol. 56, pp 37-57, 2016.

- “Sequential graft-Interpenetrating polymer networks based on polyurethane and acrylic/ester copolymers”, R. Ballesterro, B. M. Sundaram, H. V. Tippur, M. L. Auad, Express Polymer Letters, Volume 10 Issue: 3 Pages: 204-215 2016.
- ‘Non-destructive Evaluation of Transparent Sheets using a Full-Field Digital Gradient Sensor,’ C. Periasamy and H. V. Tippur, NDT & E International, Vol. 54, pp 103-106, 2013.
- ‘A Full-Field Reflection-Mode Digital Gradient Sensing Method for Measuring Orthogonal Slopes and Curvatures of Thin Structures,’ C. Periasamy and H. V. Tippur, Measurement Science and Technology, Vol. 24, Paper # 025202, (9 pp), 2013.
- ‘Measurement of Orthogonal Stress Gradients Due to Impact Load on a Transparent Sheet using Digital Gradient Sensing Method’, C. Periasamy and H. V. Tippur, Experimental Mechanics, Vol. 53, No. 1, pp 97-111, 2013.
- ‘Measurement of Crack-tip and Punch-tip Transient Deformations and Stress Intensity Factors using Digital Gradient Sensing Technique’, C. Periasamy and H. V. Tippur, Engineering Fracture Mechanics, Vol. 98, pp185-199, 2013.

Graduate Students Involved During Reporting Period

- Ricardo A. B. Mendez – graduate student (M.S.) in the Polymer and Fiber Engineering Department (October 2012-Dec 2015)
- Amith Jain – graduate student (M.S.) in Mechanical Engineering Department (October 2013-January 2015)
- Balamurugan M. Sundaram – graduate student (Ph.D.) in the Mechanical Engineering Department (October 2012-present)

Undergraduate Students Involved During Reporting Period

Mathew Halvorson – undergraduate student, Polymer and Fiber Engineering department (August 2014- present)

Awards, Honors and Appointments

- H. V. Tippur has been appointed as a member of the Executive Committee of the Society for Experimental Mechanics for 2015-2017.
- Gold award for graduate technical paper/oral competition. Submitted paper "Sequential graft-Interpenetrating polymer networks of polyurethane and acrylic/vinyl ester based copolymers", R. A. B. Mendez, B. M. Sundaram and H. V. Tippur, M. L. Auad. Society of Hispanic Professional Engineering Conference. Detroit, Michigan, November 6, 2014.
- Dr. M. L. Auad, 2015 Women of Distinction, Faculty Leadership Award, Auburn University.

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- A diagram showing a network of nodes (black dots) and edges (solid black lines). The network is partially enclosed by a dashed blue line. One node is highlighted with a red circle.

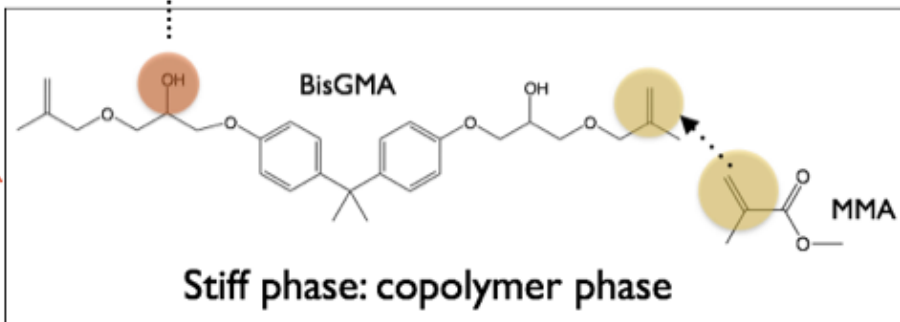


Figure 1. Schematic representation of the chemistry used for generating MIPCs. Here, the vinyl ester resin (BisGMA) is capable of undergoing (a) a step growth polymerization with the isocyanate group present in the ductile phase and (b) chain growth polymerization with the MMA monomer in the stiff phase.

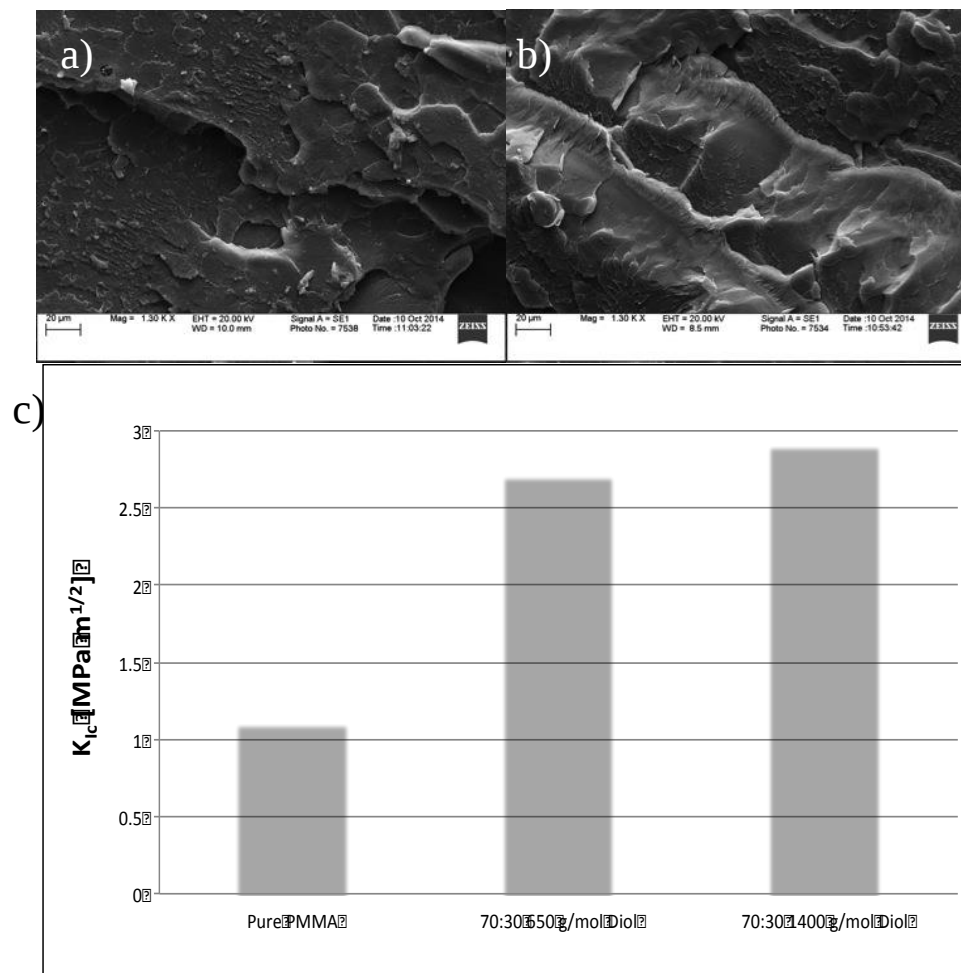


Figure 2: SEM images of MIPCs with a stiff to ductile phase using a 1400 g/mol Diol with a 70:30 and 90:10 stiff/ductile phase weight ratio, (c) chart comparing the crack initiation toughness between PMMA and the aforementioned MIPCs. Evidently no distinct domains are present in the TEM images resulting in good optical transparency. Further the MIPCs show substantial increase in crack initiation toughness relative to neat PMMA without significant loss of stiffness (not shown).



Figure 3. A sample MIPC produced at Auburn University showing excellent optical transparency. The target/logo is located ~50 mm away from the sample plane demonstrating superior optical clarity achieved.

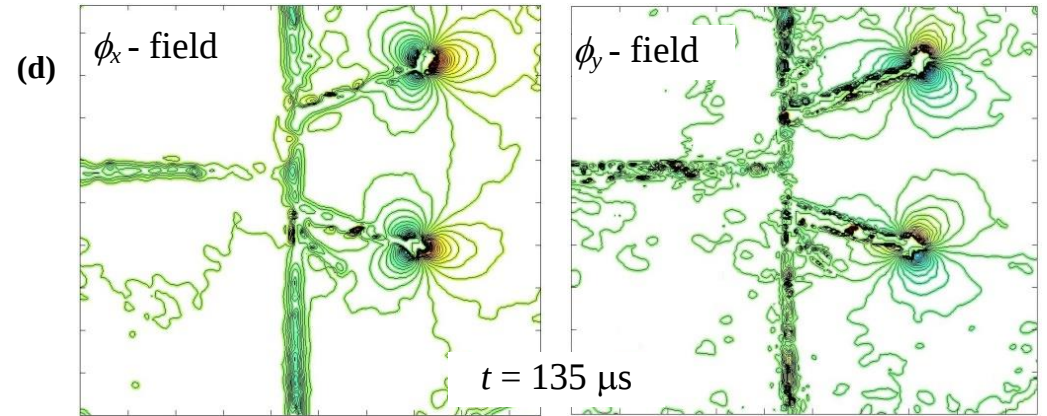
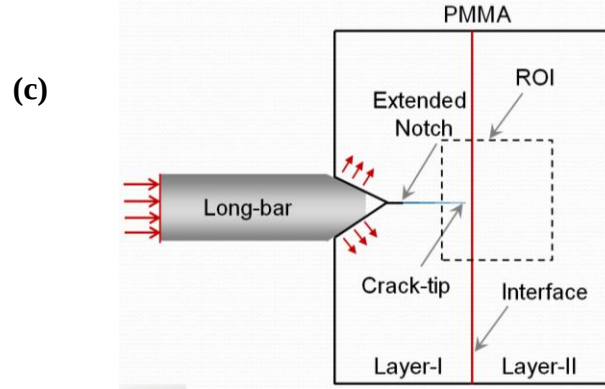
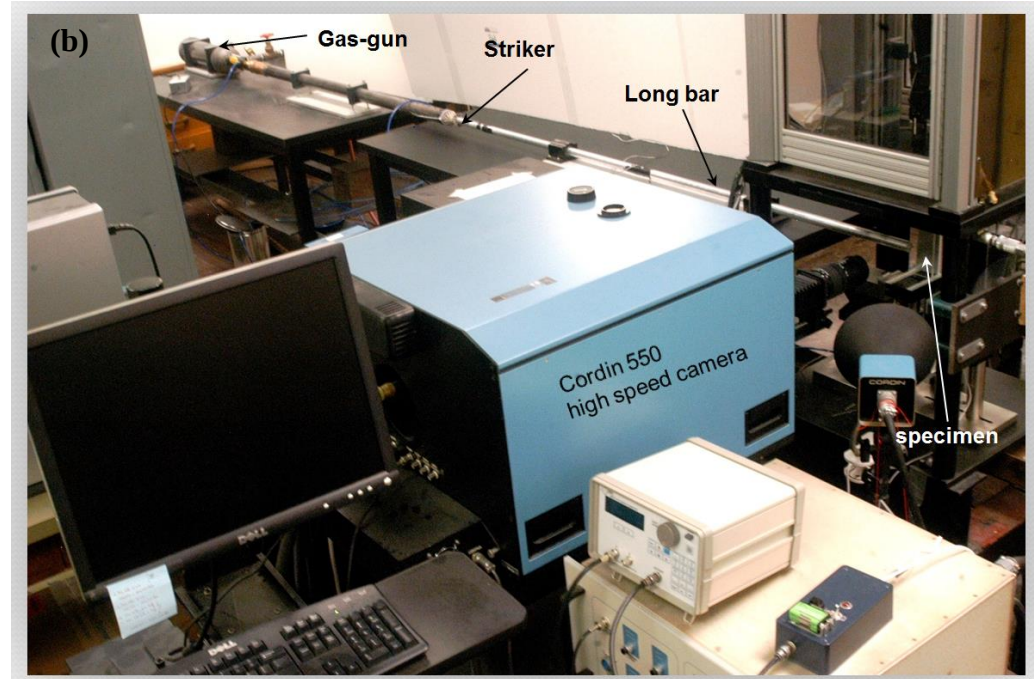
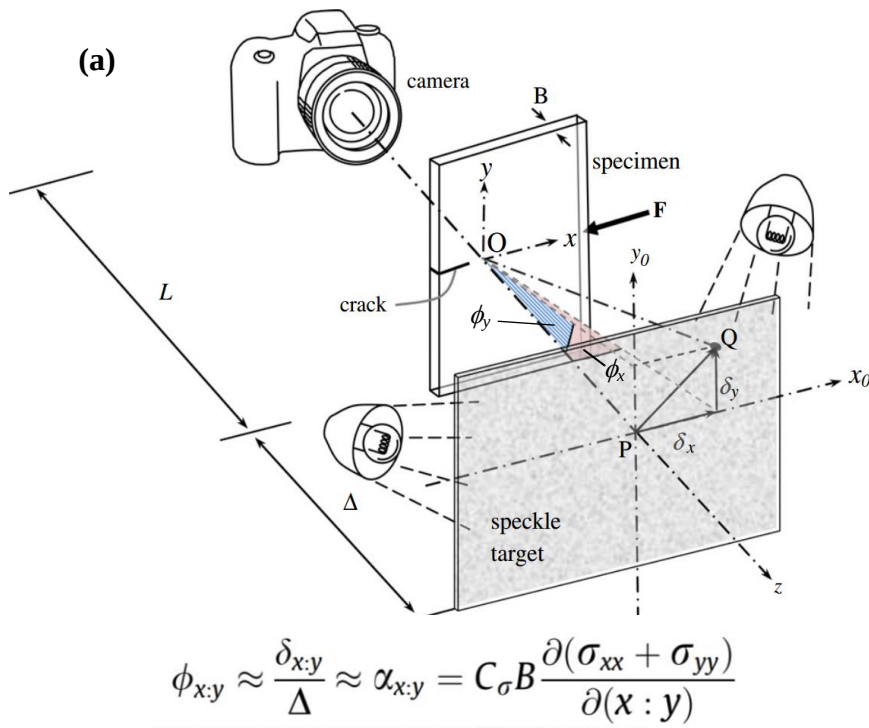


Figure 4: (a) Schematic of the experimental setup for implementing DGS methodology to measure ϕ_x and ϕ_y in the region of interest, (b) Photography of the DGS apparatus used to implement DGS using high-speed photography and long-bar impacting device, (c) Schematic of the specimen configuration used to study dynamic fracture of bi-layered transparent sheets with an interface normal to crack growth direction (crack growth from left to right), (d) Measured orthogonal angular deflections (proportional to in-plane stress gradients) at a time instant when the crack is propagating as a branched mixed-mode crack in layer-2.

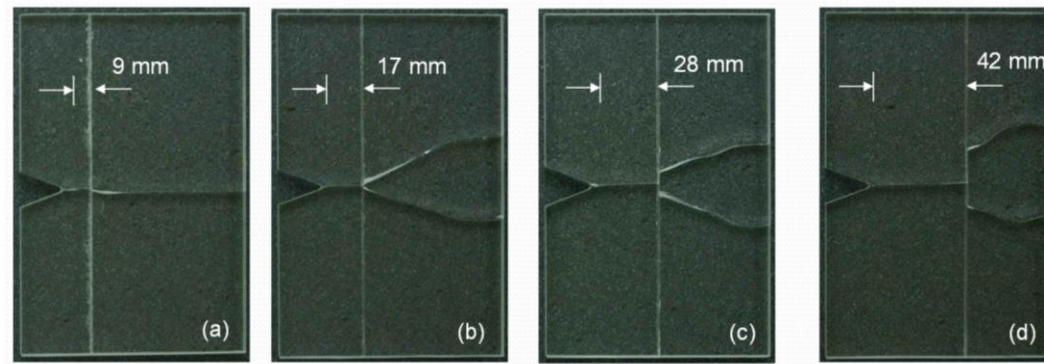


Figure 5: Effect of a weak interface location in the sheet on the crack growth behavior. Crack branching is greatly affected by the location in four different cases studied.

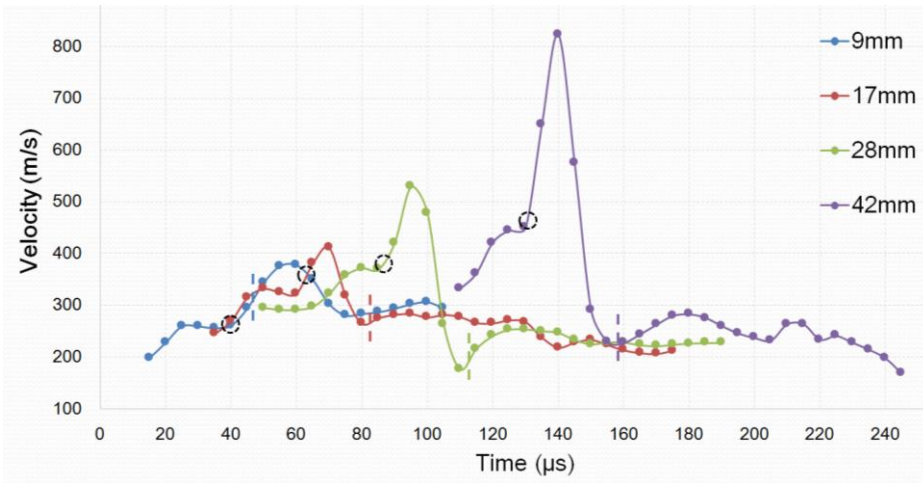


Figure 6: Crack velocity histories in four the different interface location cases. Broken circles represent crack speeds before the dynamic impingement on the interface; dotted lines correspond to speed of upper crack branch immediately after exiting the interface.

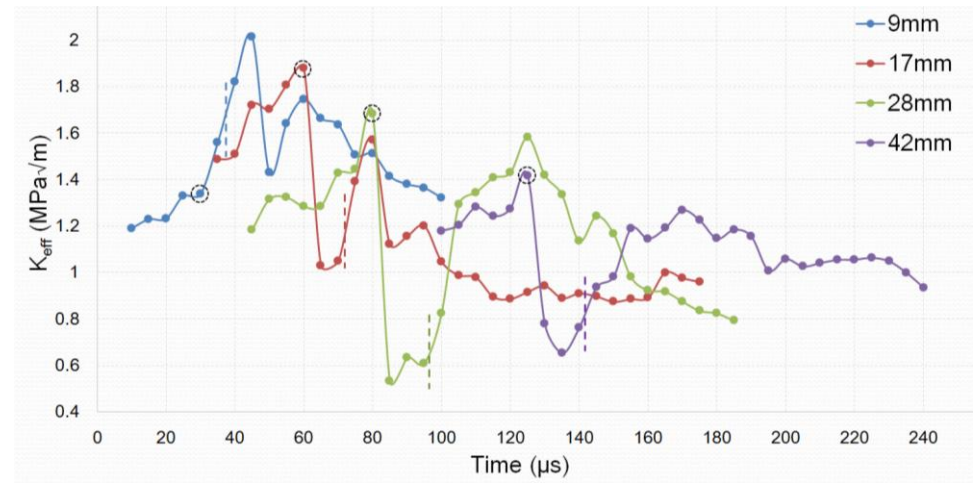


Figure 7: Stress intensity factor histories in the four different interface location cases. Broken circles represent effective stress intensity factor before the dynamic impingement on the interface; dotted lines correspond to the effective stress intensity factor of upper crack branch immediately after exiting the interface.

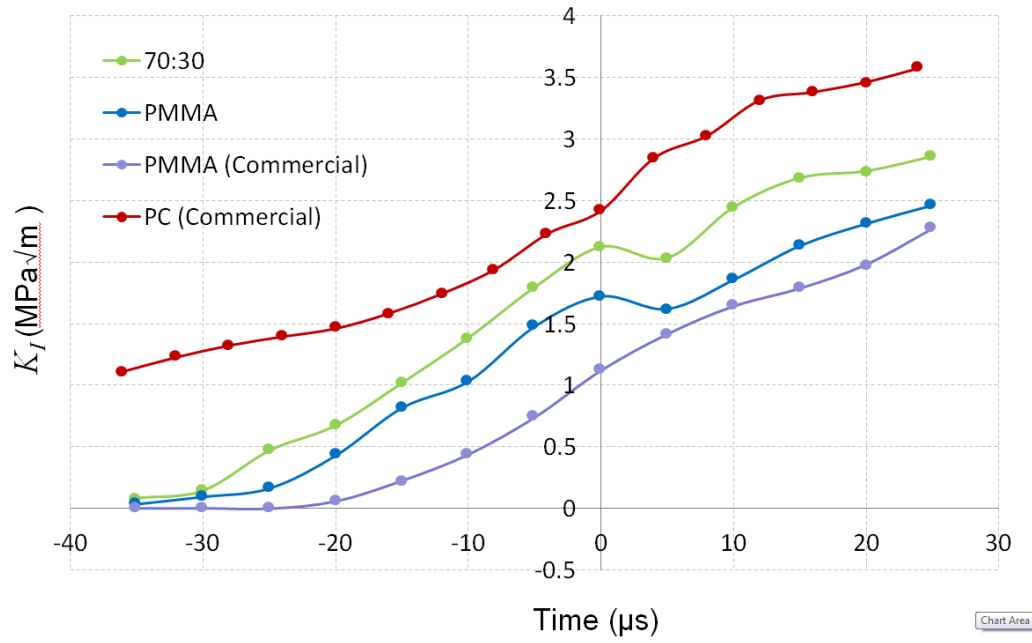


Figure 8: Dynamic stress intensity factor histories MIPC (70:30), PMMA and PC extracted using DGS. Relative to commercially procured PMMA, MIPC shows ~100% improvement in K_I at crack initiation ($t = 0$). Commercial polycarbonate is currently being targeted by other MIPC formulations.