The research and development of damage tolerant carbon fiber composites
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THE RESEARCH AND DEVELOPMENT OF DAMAGE TOLERANT CARBON FIBER COMPOSITES

A Record of Study

by

JOHN ARMANDO MIRANDA

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

DOCTOR OF ENGINEERING

December 1999

Major Subject: Engineering
College of Engineering

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ABSTRACT

The Research and Development of Damage Tolerant Carbon Fiber Composites.

(December 1999)

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Co-Chairs of the Advisory Committee: Dr. Hung-Jue Sue
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This record of study takes a first hand look at corporate research and development efforts to improve the damage tolerance of two unique composite materials used in high performance aerospace applications. The professional internship with The Dow Chemical Company - Dow/United Technologies joint venture describes the intern's involvement in developing patentable process technologies for interleave toughening of high temperature resins and their composites. The subsequent internship with Hexcel Corporation describes the intern's involvement in developing the damage tolerance of novel and existing honeycomb sandwich structure technologies.

Through the Doctor of Engineering professional internship experience this student exercised fundamental academic understanding and methods toward accomplishing the corporate objectives of the internship sponsors in a resource efficient and cost-effective manner. Also, the student gained tremendous autonomy through

exceptional training in working in focused team environments with highly trained engineers and scientists in achieving important corporate objectives.

DEDICATION

This record of study represents the end to an extraordinary collegiate experience signaling the arrival at another milestone in my life. In retrospect, this journey has presented many complex challenges, rewards and unbelievable quandaries. In passing this life landmark, I go forth a humble champion because of the will of the Lord Jesus Christ and his Holy Mother. So to you my adored Father and Holy Mother, through whom all things have been made possible, I offer this milestone.

In accomplishing this endeavor I was blessed to have had the unyielding support of an incredible family. To my virtuous, Lorena Grado, you have always been my inspiration. To my parents Leopoldo Armando, Jr., and Mary Dolores, and to my sisters Stephanie Marie and Marlo Antoinette thank you for your love and support.

To my grandparents Mr. and Mrs. Leopoldo Armando Miranda, Sr., and Mr. and Mrs. Juan Del Castillo, you have taught and demonstrated to me the value of courage, determination, integrity, love, sacrifice and faith in God. Because of the foundation you laid my life experiences have been a privilege.

ACKNOWLEDGEMENTS

I give my sincere gratitude to three people who have been instrumental in my success at Texas A&M. I thank Dr. Warren M. Heffington for altering my course in life by inspiring me to pursue graduate studies at the doctoral level. I thank Dr. Hung-Jue Sue for recruiting, advising and developing me for a rewarding career in polymer material science. I thank Dr. Karan Watson for her professional guidance and mentoring along the way to reaching my academic goals. To these three people I am indebted for they believed in my talents and character, and gave of themselves unselfishly so I could realize a dream.

I deeply appreciate the time, effort, and consideration expended on my behalf by the members of my advisory committee – Dr. Christian P. Burger, Dr. Walter L. Bradley, Dr. Leonard Bierman, and Dr. John L. Hogg. Moreover, I extend my appreciation to my internship supervisors, Dr. Dexter White and Mr. Bruce L. Burton, from The Dow Chemical Company, and Dr. Shaw Ming Lee, from Hexcel Corporation. I also acknowledge the special efforts of Teresa Wright from the College of Engineering.

I would like to acknowledge my advisors and mentors at The University of Texas at El Paso – Dr. Jack A. Dowdy, Dr. Juan M. Hererra, Dr. John Levosky, Dr. Donald A. Michie, Mr. Manny Pacillas, Dr. Stephen Riter, and Dr. Diana S. Natalicio.

Lastly, I would like to thank Texas A&M University and all those associated with it. From Bonfire to Muster to the Wrecking Crew and the Corps of Cadets, thanks for the memories. Gig ‘em Aggies.

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INTRODUCTION

The Doctor of Engineering Program at Texas A&M University is intended to prepare individuals for professional engineering activities in business, industry and the public sector. The Doctor of Engineering Program Graduate Program Manual states:

> As part of the degree requirements, each student will spend a minimum of one calendar year working under the supervision of a practicing engineer in industry, business or government. The objectives of the internship are two fold: (1) to enable the student to demonstrate and enhance his or her abilities to apply both knowledge and technical training by making an identifiable contribution in an area of practical concern to the organization or industry in which the internship is served, and (2) to enable the student to function in a non-academic environment in a position in which he or she will become aware of the employers approach to problems, in addition to those approaches of traditional engineering design or analysis. [1]

The internship was split into two and performed with two companies – The Dow Chemical Company and Hexcel Corporation. Dow Chemical was unable to keep its initial agreement for a year-long internship due to the sale of the Dow-United

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The style and format of this record of study follows that of the *Journal of Composite Materials*.

Technologies joint venture which was the division employing the intern. The initial internship proposal was prepared in December, 1997 and outlined the internship with Dow Chemical in Freeport, Texas. The internship with Dow Chemical occurred from January, 1998 to June, 1998. Due to Dow's initiative to focus on core business unit development the Dow-United Technologies division was sold, thus prompting a change in the internship sponsor to Hexcel Corporation. The proposal was subsequently amended and resubmitted in June, 1998 to cover the internship period from July, 1998 to December, 1998 with the Hexcel Corporation, Dublin, California. This record of study covers both internship periods.

This record of study is comprised of four sections including this introduction. The second section describes the internship with The Dow Chemical Company. It reviews the professional experiences, and the research and development of damage tolerant, high temperature epoxies and their composites. The third section is an overview of internship at the Hexcel Corporation. It reviews the professional experiences, and the development of damage tolerant, honeycomb core composites. An internship experience summary is presented as the last section.

The record of study does not contain information classified by The Dow Chemical Company, Hexcel Corporation, or the federal government. Additionally, the record of study represents the opinions and conclusions of the author, and does not reflect the official or unofficial views of any member of The Dow Chemical Company or Hexcel Corporation.

INTERNSHIP AT THE DOW CHEMICAL COMPANY

Introduction

The official internship period with the Dow-United Technologies joint venture of The Dow Chemical Company, in Freeport, Texas, commenced January 1, 1998, and was completed on June 31, 1998. The intern spent a co-op summer term (May, 1996 through August, 1996) and one calendar year (May, 1997 through June, 1998) in either an internship or internship-like setting at The Dow Chemical Company. During the intern's tenure he interacted with leading professional scientists and engineers involved in developing the next generation of damage tolerant, high temperature thermosetting resins and their carbon fiber composites for aerospace applications.

This division of Dow-United Technologies (Dow-UT) joint venture was focused on the research and development of novel composite technologies, and served as technical support for the manufacturing of tactical aircraft components. During the internship period, the corporate expectations of this research group were high and the work environment was fast paced. This division was pressed in meeting material development deadlines to bid on aerospace contracts utilizing the outcomes of this and other high performance composites research. The intern's efforts were instrumental to Dow-UT in meeting project expectations and deadlines.

Internship Supervision

The internship supervisor was Dexter White, Ph.D., P.E., who was the Senior Manager of Dow-UT, in Freeport. Dr. White currently serves as the Senior Manager of the Materials Research and Development Lab for The Dow Chemical Company. The immediate internship supervisor was Mr. Bruce L. Burton, a Research Leader in the Dow-UT joint venture group. Currently, Mr. Burton is employed by Huntsman Chemical in Austin, Texas. Dr. White and Mr. Bruce Burton's resumes are given in Appendix A, Resumes of Internship Supervisors.

As the Senior Manager of Dow-UT, Dr. White helped the intern develop professionally and technically. In maintaining a positive team-oriented environment, Dr. White enthusiastically promoted the intern's involvement in both the high temperature resin development project and the interleave toughening project discussed in this record of study.

This intern's development at Dow-UT was also guided by Mr. Burton. As the intern's immediate supervisor, he trained the intern in understanding the methods and implications of fundamental research in developing high performance thermosetting resins. He trained the intern in methods of resin characterization and resin rheology that can be applied to many polymeric resins. The intern as a result of this advanced training exercised great autonomy in managing projects and related tasks. In his mindfulness to the intern's professional development, Mr. Burton encouraged the intern to attend technical industry conferences, and professional development seminars and special topic seminars held at the company. Dr. White and Mr. Burton's commitment to this intern's technical and professional development made this internship an invaluable experience. Dr. White's summary of this internship is given in Appendix B, The Internship Supervisor's Final Report.

Internship Position

The internship position with Dow-UT focused on the development of damage tolerant, high performance resins and their composites. The intern was responsible for studying the damage tolerance of these inherently brittle resins via thermoplastic particulate interleaving in woven carbon fiber composites. The intern was involved in a joint effort between Dow-UT and Texas A&M (under the supervision of Dr. Hung-Jue Sue) aimed at developing patentable toughening technologies. The overall scope of this material development endeavor included manufacturing process development, resin characterization, morphological studies, and mechanical testing. The intern was primarily involved in developing novel manufacturing techniques, and performing mechanical testing for the development of thermoplastic interleaved composites.

Apart from the aforementioned interleave toughening studies, the intern was involved in developing high temperature resins and their composites for Dow-UT. This intern worked closely with his immediate supervisor in developing manufacturing processes, and characterizing resin rheology, thermal properties, and mechanical properties of novel high temperature resins and their composites. The intern was solely responsible for manufacturing these composites and developing vanguard processing techniques for a variety of novel resin formulations. This particular project involvement was continually challenging in meeting sometimes unrealistic material development deadlines. The efficient teamwork experience of being a member of a three-person material development team (Mr. Burton, Jim Bertram, Ph.D., and the intern) was integral in meeting project deadlines and overcoming project obstacles, and in contributing to the intern experience.

The intern was often given the opportunity to give project update presentations to colleagues and research leaders of Dow-UT. Throughout the internship period, the intern documented all material development research. All work related to high temperature resin development and thermoplastic interleave toughening of brittle matrix epoxies was thoroughly documented in Dow Chemical restricted data books.

This internship position allowed this student to witness and become functional in Dow Chemical's research "engine". Through the intern's varied project involvement, the experience allowed him the opportunity to observe the many corporate entities (i.e., R&D, Manufacturing, Marketing, and Testing) necessary in developing a novel material into a viable product. The opportunities that this student was afforded at Dow Chemical allowed him the experience to develop his leadership, professional and technical skills in a way unique to the Doctor of Engineering degree.

Internship Objectives

The internship objectives are described in a formal document dated July 1, 1997, which was prepared by the intern and endorsed by his committee (see Appendix C, Final Internship Objectives, Dow-United Technologies). They are summarized here. The Dow-UT corporate objective focused on developing patentable toughening technologies for resin transfer molding (RTM) applications to manufacture composites. This intern successfully obtained results aimed at meeting this corporate objective by studying the thermoplastic interleave toughening of brittle matrix carbon fiber composites. This toughening technology was being pursued to improve the damage tolerance of high temperature, thermoset resin composites used in high performance Dow-UT applications.

The intern's objective was targeted toward developing new methods of manufacturing interleaved composites and to evaluate the material properties of these composites. This intern was responsible in managing the internship project's progress toward meeting the corporate goals. These studies gave the intern insight in understanding the manufacturing process' influence on the toughening in interleaved composites. Finally, the intern researched the potential opportunities in patenting new toughening technologies related to this internship project. The corporate objective associated with developing this and other toughening technologies remains an ongoing concern.

Thermoplastic Interleaving Project

Background

Today there is an ever-increasing need for high temperature composites in aerospace, automotive, and industrial applications. These applications range from hypersonic vehicles, nose cones, engine components, power generators, tooling, and thermal protection. The application of these composites is continually under development. The major obstacle toward the application of these and many composite materials is their tendency to delaminate, which is the most predominant failure mechanism in composite structures. Delamination can be a result of manufacturing (i.e., residual stresses), service conditions (i.e., thermal cycling, moisture absorption), and impact damage. Improving the delamination resistance of continuous fiber composites is an ongoing concern in the composites industry. There have been many different approaches toward improving the delamination resistance of continuous fiber composites [2-10].

Recent research has shown that thermoplastic modification can improve the fracture toughness and impact damage resistance of relatively brittle epoxies [11-23]. The toughening approach used in this research is thermoplastic interleaving, in which thermoplastic particles remain as discrete phases in the resin-rich composite interlayer through the cure cycle (i.e., the particles do not dissolve). Improvement of the damage resistance of composite materials should enable a higher energy absorption capability and the use of higher allowable design strains in producing further weight savings compared to existing carbon fiber/epoxy systems [24]. This weight reduction in aerospace applications could lead to improved flight performance, increased payload (e.g., electronics and munitions), reduction in life cycle costs and improved survivability of the composite structure.

The general project approach to studying interleave toughening in brittle matrix composites commenced by studying the resin rheology (time-temperature-viscosity relationship), morphological issues of the modified resin, and the resin material properties [25-27]. When an understanding of the resin behavior was obtained then manufacture of the carbon reinforced composites began by developing a reproducible method to manufacture thermoplastic interlayed preforms. Once the preform manufacturing process was developed, manufacturing of the composites was performed via infusion molding. Infusion molding is a closed mold process in which a fixed amount of resin (i.e., resin charge) is infused into the preform during the cure cycle when the resin viscosity is low. After infusion molding, the toughened composites were prepared for testing. Mechanical tests performed included interlaminar fracture toughness, and compression after impact testing. The process-morphology-property relationship was also evaluated.

Project Tasks

The two thermoset systems used in these studies were bismaleimide (BMI) and epoxy/anhydride resin. The BMI, Cytec 5250-4RTM, is widely used in high performance composite applications. The resin exhibits a high glass transition temperature (approximately 260°C using the Dow-UT cure schedule), low moisture uptake, good dimensional stability, and can attain low viscosities for processing (i.e., autoclave, resin transfer or infusion molding). The epoxy/anhydride resin system used in this study is Dow Epoxy Novolac 438 (D.E.N.® 438)/Norbornene Anhydride. This resin system without reinforcement exhibits brittle fracture behavior (e.g., candy-like brittle fracture behavior). This cured resin system exhibits a high glass transition temperature (approximately 280°C), low moisture uptake, good dimensional stability, and can attain low viscosities for processing (i.e., resin transfer and infusion molding). This very brittle resin was chosen to accentuate the efficiency of the thermoplastic interleave toughening approach used in these studies. This method incorporates a thermoplastic particulate dispersion using a proprietary novel interlayering technique in preform manufacturing developed by the intern. These thermoplastic particulates are present as discrete domains in the resin-rich interlaminar regions of the composite throughout the cure cycle.

The two composite systems using BMI and epoxy/anhydride studied were interlayered with various engineering thermoplastics such as polyetherimide (PEI), polyphenylene oxide (PPO), and polyphenylene sulfide (PPS). The BMI composites were interleaved with 10-weight percent of PEI, and 10-weight percent and 20-weight percent of PPO (PEI and PPO from GE Plastics). The epoxy/anhydride composites were interleaved with 10-weight percent and 20-weight percent of PPO, and 10-weight percent and 20-weight percent of PPS (RYTON PPS from Phillips 66). These particles were placed in the interlayer between each of the woven plies. In calculating the average interlaminar distance using a Suppliers of Advanced Composite Material Association (SACMA) recommended method (SRM 10-88), the interlaminar resin weight was found and the thermoplastic particles were incorporated into the interlayer accordingly. For example, the terminology "10-weight percent" refers to the amount of thermoplastic particulate dispersed in the interlayer equivalent to ten percent of the resin weight estimated in the interlaminar region. The thermoplastics used in these composites were cryogenically ground and sifted with a 50-micrometer sieve.

Using a proprietary technique developed by the intern, the thermoplastic particles were strategically dispersed between the plies to promote effective toughening mechanisms. Specifically, not only were the thermoplastic particles dispersed between plies to promote the "particle crack bridging" mechanism and stop crack growth, but the particles by virtue of their size and placement also served as ply spacers [28-30]. These particles may create an interply distance possibly beneficial to improving the fracture toughness via formation of a larger crack tip damage zone.

The woven carbon fiber fabric used in these composites is an intermediate modulus, four harness satin, three thousand fibers per tow, and GP sized fabric (IM7-4HS-3k-GP) manufactured by BGF Industries. The composites after manufacture were

Mode I double-cantilever beam (G_{IC}) and Mode II end-notch flexure (G_{IIC}) toughness tested, and compression after impact (CAI) tested [31-41]. Rubber modification was not considered for these studies due to a resulting laminate stiffness reduction and thermal degradation concerns about the rubber phases at elevated temperature. High performance engineering thermoplastics by virtue of their elevated glass transition temperatures, low moisture uptake, high tensile modulus, low strain to failure, and dimensional stability while maintaining acceptable laminate stiffness were selected for these interleave studies.

Apart from the thermoplastics initially chosen for these studies, the intern researched other thermoplastic candidates for interleave toughening. For instance, RYTON PPS from Phillips 66 was chosen to interleaf toughen D.E.N.® 438/Norbornene Anhydride because of its high glass transition temperature, good chemical resistance, low moisture uptake and low strain to failure in comparison to the other thermoplastics used in this study. Also, a new amorphous polyimide (PI) from Mitsui Toatsu Fine Chemicals Incorporated, a Japanese polymer company, had exceptional properties but because of the internship interruption by the sale of the Dow-UT joint venture, this new polyimide could not be evaluated.

Manufacturing Process Development

The studies first focused on developing manufacturing processes that would produce high quality interleaved composites. The intern performed process related studies in resin transfer molding (RTM) and infusion molding. Dow-UT wanted to perform manufacture via RTM since the corporate objectives focused on developing patentable toughening technologies for this process. However, the expertise in manufacturing these types of composites needed to be developed. Thus, the intern performed a comparative study between RTM and infusion molding (a Dow patented manufacturing process) to select the best manufacturing process that would allow for the production of reproducible, high quality composites in a time efficient and cost effective manner. It was found, considering composite material yield, process preparation time, cost of production, and quality of the composite, that RTM was not the most feasible process for the manufacture of the composites to be used in these studies. On the other hand, the infusion molding process proved to be the most feasible process when considering the aforementioned issues.

Another objective of the intern was to develop process techniques to manufacture interleaved preforms. A preform is a preferentially oriented stack of carbon fiber plies that are sewn together to lock-in the configuration (e.g., quasi-isotropic or stacked). The preforms manufactured for these studies were either quasi-isotropic or stacked. The stacked preforms were comprised of carbon fiber plies that were placed and oriented with the warp tows aligned in the same direction. The quasi-isotropic preforms were constructed of carbon fiber plies that were placed in an approximating isotropic fashion by orienting the plies symmetrically in several directions (i.e., $[90, -45, 0, +45]_{2\text{Symmetric}}$) to make a 16 ply preform. In the case of "interleaved" preforms, thermoplastic particulate is dispersed between each of carbon fiber plies. A proprietary approach to applying these thermoplastics in a strategic manner was developed by this intern, which ultimately bolstered efforts to pursue a patent disclosure on a related technology.

Through this manufacturing process development initiative, the intern became intimately familiar with the nuances of RTM and infusion molding. This knowledge empowered the intern to effectively apply cycle time reduction principles toward streamlining the infusion molding process. The cycle time reduction of a one-man operation producing an infusion molded composite was reduced from one week per composite to one to two days per composite, depending on the resin cure schedule. Once the manufacturing processes were in place the manufacture of high quality thermoplastic interleaved composites commenced.

Thermoplastic Interleaving

Toughening studies began with the evaluation of BMI carbon fiber composites interleaved with PPO and PEI. Complementary studies about these interleaf toughened composite systems were performed on the modified resin morphology and fracture mechanisms by colleagues at Texas A&M [22]. This effort was important in comparing the toughening efficiency in the modified resin in relation to the interleaved composite. Before discussing the mechanical testing results, a brief summary of the morphological studies of 10-weight percent PEI, and 10-weight percent PPO modified BMI are reviewed. These studies offered important information that was considered by the intern toward designing the manufacturing processes (i.e., interleaving methods and cure schedule) that would promote effective toughening.

In this study, the morphological observations of the modified BMI gave insight as to the microstructure set-up within the composite interlayers. The morphological features of 10-weight percent PPO modified BMI revealed that the PPO particles did remain as discrete phases within the BMI. The PPO particles seemed to maintain the irregular shape exhibited just after cryogenic grinding. These particles exhibited strong interfacial adhesion, and by virtue of their shape also mechanically interlocked with the matrix. Figure 2.1 shows the fracture surface of the 10-weight percent PPO modified BMI resin. Plastic drawing of PPO particles is evident near the center of each particle. Also observed from the fracture surface is small-scale particle cavitation resulting from the triaxial stress state present at the crack tip.

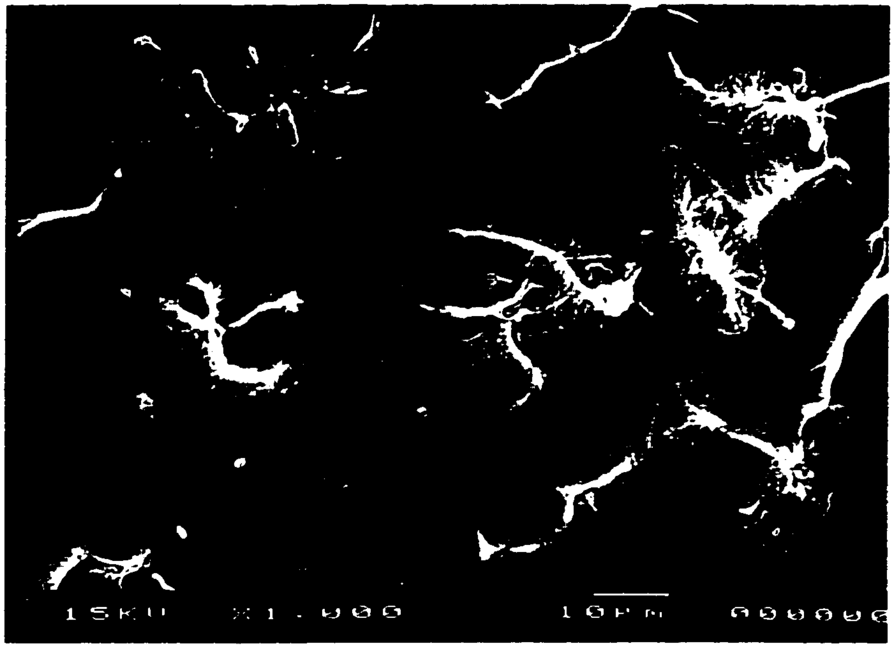


Figure 2.1. Fracture surface of 10-weight percent PPO-modified BMI 5250-4RTM. Plastic drawing of the PPO is observed.

It is generally recognized that highly crosslinked epoxies, like high temperature resins, do not readily undergo plastic deformation, and for these resins the particle crack bridging mechanism and stress induced plastic deformation can be the primary means of toughening. Therefore, strong interfacial adhesion is important for this particle crack bridging mechanism to operate in this and other brittle matrix resins. In Figure 2.2 transmission electron microscopy (TEM) and optical microscopy (OM) observations reveal that crazing in the PPO particle and crack tip blunting are other fracture mechanisms observed in this matrix system [22, 42]. The crack tip blunting observed in this system indicated that the BMI was capable of large plastic deformation in relation to highly cross-linked thermosets.

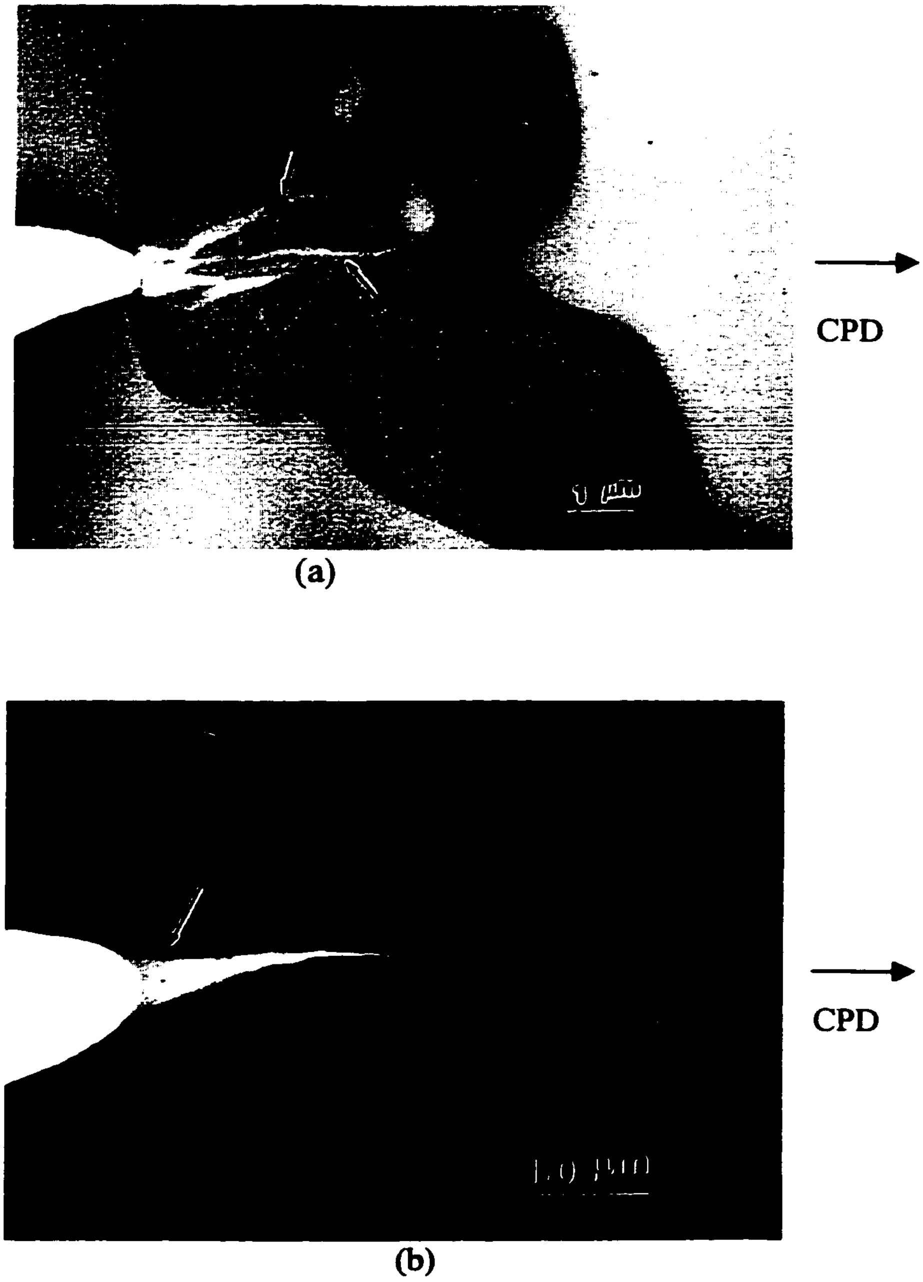


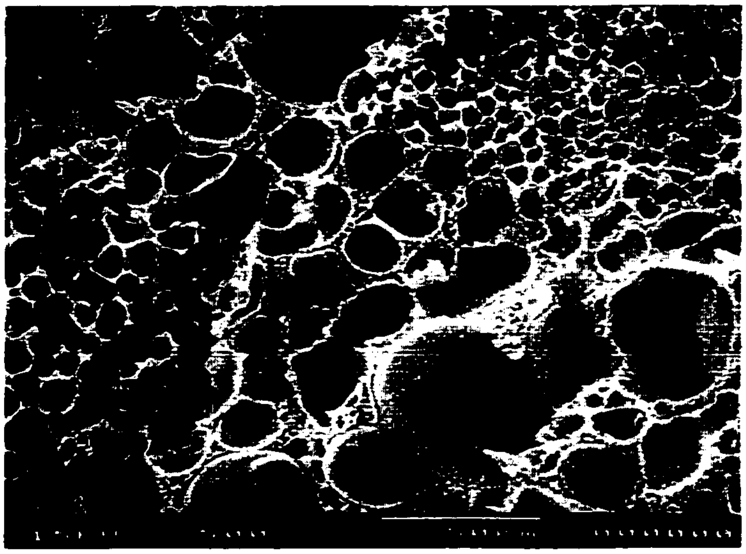
Figure 2.2. (a) TEM micrograph of 10-weight percent PPO-modified BMI 5250-4RTM taken at the crack tip damage zone of a double notch four point bend (DN-4PB) specimen. Crazes inside the PPO particle are evident at crack tip (see arrow). (b) Transmission electron microscopy micrograph of 10-weight percent PPO-modified BMI taken at the crack tip damage zone of DN-4PB specimen. Crack tip blunting is observed (see arrow). (CPD) Crack Propagation Direction. [22]

It remains however, that particle bridging via plastic deformation of the particle was the primary toughening mechanisms in absorbing fracture energy from the propagating delamination. Figure 2.3 shows particle crack bridging mechanism observed via TEM at the crack tip of a BMI/PPO double notch four point bend (DN-4PB) specimen.

Figure 2.3. TEM micrograph of BMI/PPO showing dilatation bands (see arrows) and microcrack nucleation at the crack tip damage zone of a DN-4PB specimen. The crack propagates from left to right. [22]

Due to the stress concentration at the crack tip the particles nucleate microcracks. These microcracks are manifested by the formation of dilatation bands at which the stresses becomes focused [43]. Consequently, this microcrack formation promotes particle bridging by which fracture energy is dissipated via the stretching and plastic deformation of the particle. In DN-4PB tests performed on the neat BMI resin dilatation bands were not observed at the crack tip damage zone. In determining the fracture toughness (G_{IC}) of this PPO-modified BMI system it was observed that there was an increase in fracture toughness in comparison to the unmodified BMI.

The PEI-modified BMI morphology was markedly different, as the PEI was miscible in BMI. In processing this toughened system it was important to realize that there were competing curing processes, for example, dissolving, BMI cure reaction, and phase separation. In light of the miscibility of the PEI into BMI, the approach to manufacturing the composite was to B-stage (i.e., pre-cure) the BMI matrix to promote a co-continuous phase. Without this pre-cure local phase inversion occurred leading to poor PEI dispersion and limited toughness improvement
(Figure 2.4 a).



(a)

(b)

***Figure 2.4. (a)** Local phase inversion in 10-weight percent PEI-modified BMI 5250-4RTM. **(b)** Co-continuous morphology of pre-cured 10-weight percent PEI-modified BMI 5250-4RTM.* [22]

When the BMI is pre-cured followed by the standard BMI cure, a co-continuous morphology is observed (see Figure 2.4 b). The co-continuous phase morphology was found to yield a higher G_{IC} value due to the large amount of ductile yielding of the PEI-rich phase, which is the main fracture energy release process in the BMI/PEI system. Table 2.1 summarizes the fracture toughness (G_{IC}) values for 10-weight percent thermoplastic modification of the BMI resin.

Table 2.1. Fracture toughness (G_{IC}) test results of 10-weight percent thermoplastic modified BMI 5250-4RTM.

Material System	G_{IC} (J/m^2)
BMI [a]	160
BMI/PEI [a]	350
BMI/PPO [b]	420

a. Cure schedule 185°C for 6 hours, and 220°C for 6 hours.
b. Pre-cure 120°C for 20 hours, 185°C for 6 hours, and 220°C for 6 hours.

From a material design standpoint much can be learned in studying the modified resin morphology and fracture behavior of a toughened composite. For instance, the integrity of interfacial adhesion of the components to one another can be observed, the morphological structure that best promotes damage tolerance can be optimized by cure schedule refinement, the fracture mechanisms involved in improving the fracture toughness can be identified, and the approximate amount of thermoplastic modification needed to enhance damage tolerance can be found, as well as the degree of dispersion of the modifier. These fundamental concerns, although not all-encompassing, are critical to improving toughened composite performance. Working with colleagues to understand these important issues gave the intern additional insight and understanding about composite manufacture and possible expectations in the composite performance.

Mechanical Testing

In studying the composite interlaminar fracture toughness (G_{IC} and G_{IIC}) and compression after impact strength, it was hypothesized that a correlation between G_{IIC} and CAI could be achieved. The fundamental concept of this correlation is based on the premise that the impact damage delaminations observed in low velocity impact tests is a Mode II dominated fracture phenomenon. This premise is derived from the mode II or shear loading condition under which delaminations propagate. Figure 2.5 illustrates the idea of the composite specimen bending during impact with characteristic interlaminar delaminations forming a cone-like fracture damage zone.

A stacked preform configuration was used initially in these studies because the orientation was the same specified in Dow-UT aerospace applications. The preform configuration was later changed to quasi-isotropic to allow for direct comparisons to literature and other company data.

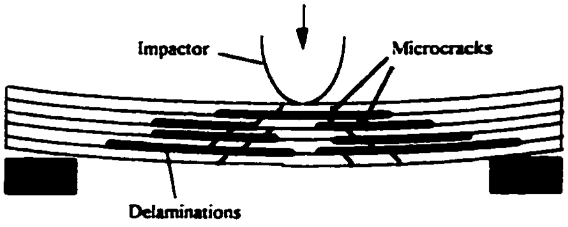


Figure 2.5. Illustration of out of plane impact for CAI specimens emphasizing a Mode II damage process.

In interlaminar fracture testing of the 10-weight percent PPO interleaved composites, it was found that there was an improvement in both G_{IC} and G_{IIC} interlaminar fracture toughness. The G_{IC} in the 10-weight percent PPO-modified composite system showed a 136% improvement in the fracture toughness compared to the unmodified BMI composite (Table 2.2).

Table 2.2. Mode I interlaminar fracture toughness of a 16 ply stacked, 10-weight percent thermoplastic interleaved BMI 5250-4RTM.

Material System	**G_{IC} (J/m^2)^a**	
BMI/ IM7	464.5±40.265	405.9
	356.42±22.06	
	396.88±50.02	
BMI/ PPO/ IM7	1179.8±177.46	1,057.2
	1064.59±314.17	
	927.29±138.07	
BMI/ PEI/ IM7	559.3±50.24	515.7
	525.57±43.180	
	462.5±24.74	

a. Normalized to 55% fiber volume.

The G_{IIC} in the 10-weight percent PPO also improved in comparison to the unmodified BMI by 43% (Table 2.3). Although the G_{IC} and in particular the G_{IIC} improved, there was not a statistically appreciable difference in the 10-weight percent PPO-modified BMI compression after impact strength (see Table 2.4, and compare Material System 1 with 3, and 4 with 5). It is interesting to note that the quasi-isotropic laminate did yield a higher CAI strength than the stacked laminate (see Table 2.4, and compare Material System 1 with4, and 3 with 5) [44, 45].

Table 2.3. Mode II interlaminar fracture toughness of 16 ply stacked, 10-weight percent thermoplastic interleaved BMI 5250-4RTM composites.

Material System	**G_{IIC} (J/m^2) ^{a}**
BMI/ IM7	1,637.9
BMI/ PPO/ IM7	2,634.4
BMI/ PEI/ IM7	1,598.1

a. Normalized to 55% fiber volume.

Table 2.4. Compression after impact results of 16 ply, 10-weight percent thermoplastic interleaved BMI 5250-4RTM composites.

Material System	**Damage Zone Size (cm.sq.)**	**Ultimate Compressive Strength (MPa)^a**	**Mean (MPa)**	**Standard Deviation**	**Coefficient of Variation**
1.) BMI/ IM7 Stacked	0.497	260.24	266.9	9.44	0.035
	0.510	273.59			
2.) BMI/ PEI/ IM7 Stacked	0.761	176.7	187.4	15.15	0.081
	0.710	198.12			
3.) BMI/ PPO/ IM7 Stacked	0.452	265.97	250.5	21.93	0.088
	0.471	234.95			
4.) BMI/ IM7 Quasi-Isotropic	0.368	313.82	313.5	0.52	0.002
	0.348	313.08			
5.) BMI/ PPO/ IM7 Quasi-Isotropic	0.329	307.89	311.2	4.70	0.015
	0.265	314.54			

a. Normalized to 55% fiber volume.

In testing the 10-weight percent PEI interleaved BMI it was found that there was minimal improvement in G_{IC} and a reduction in G_{IIC} interlaminar fracture toughness. The G_{IC} in the 10-weight percent PEI modified composite system showed a modest 23% improvement in the fracture toughness compared to the unmodified BMI composite (Table 2.2). Although the G_{IC} improved, the 10-weight percent PEI-modified BMI compression after impact strength was reduced significantly following the tendency of G_{IIC} (Table 2.3). The compression after impact strength of the 10-weight percent PEI-modified BMI was significantly reduced in comparison to the unmodified BMI composite (see Table 2.4, and compare Material System 1 with 2).

Once this round of testing was completed, a retrospective analysis of the CAI strengths was performed. In observing the significant improvements in fracture toughness of the BMI/PPO composites under Mode I and Mode II loading, it was hypothesized that the CAI would also be improved but this was not the case. It was determined that the BMI composite yielded a ductile matrix using the standard Dow-UT cure schedule. This deduction was substantiated based on the morphology and fracture mechanisms observed in the BMI/PPO resin studies that showed the BMI was capable of large plastic deformations as evidenced in crack tip blunting behavior. It was at this juncture in the project that the resin system to be used in continuing studies was changed to a brittle, high glass transition temperature epoxy. This change was made to accentuate the interleave toughening effectiveness that could be realized in high cross density thermosets in which the crack tip plastic deformation zone is small. The internship supervisor approached the intern to study the feasibility of selecting the new matrix resin for continuing research. The candidate resins for continuing research were MY 720, PR 500, and D.E.N.® 438/Norbornene Anhydride (epoxy/anhydride) developed for high temperature applications. PR 500 was not selected for continuing studies because of its ability to dissolve the candidate thermoplastics at cure temperatures. MY 720 posed potential problems concerning volatile degassing creating composite voids and causing safety concerns during processing. Therefore, the epoxy/anhydride system was selected for continuation in fracture toughness studies because of easier processability, chemical safety, and significantly lower cost.

The CAI testing results of the 10-weight percent PPO-modified epoxy/anhydride composite indicate that interleaving is effective in limiting impact damage severity (i.e., impact damage zone size) and improved the ultimate compressive strength of these composites (Table 2.5). Mean compressive strengths were 148.82 MPa for the untoughened composite and 160.89 MPa for the toughened composite (8% improvement). C-scans of the CAI specimens following impact showed that the PPO modified system sustained less damage than the untoughened composite specimens (Table 2.5).

Table 2.5. Compression after impact results of thermoplastic interleaved, 16 ply quasi-isotropic, D.E.N.® 438/Norbornene Anhydride.

Specimen No.	**Damage Zone Size (cm.sq.)**	**Ultimate Compressive Strength (MPa) [a]**	**Mean (MPa)**	**Standard Deviation**	**Coefficient of Variation**
Control					
1	0.845	146.586			
2	0.929	145.485	148.82	4.9	0.033
3	0.981	154.391			
10-weight percent PPO Interleaved					
1	0.755	165.040			
2	0.742	159.141	160.89	3.6	0.022
3	0.729	158.491			
20-weight percent PPO Interleaved					
1	0.723	165.034			
2	0.735	160.323	163.98	3.3	0.020
3	0.716	166.596			
20-weight percent PPS Interleaved					
1	0.665	166.136			
2	0.594	169.983	166.01	4.0	0.024
3	0.677	161.992			

a. Normalized to 55% fiber volume.

By visual inspection, it was observed that the impact damage is significantly greater in the non-interleaved system. Digital photos were taken of 10-weight percent PPO-modified CAI specimens prior to compression testing to illustrate this effect (Figure 2.6).

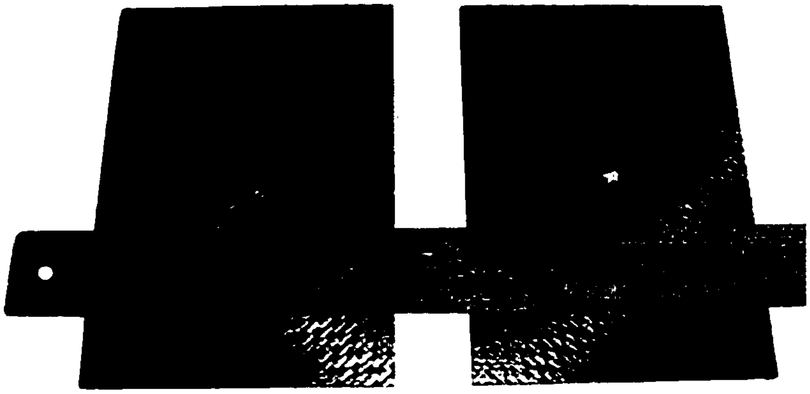


Figure 2.6. *Post impact specimens of D.E.N.® 438/Norbornene Anhydride. On the left is the 10-weight percent PPO-modified composite and to the right is the unmodified control.*

To determine if increasing the amount of interleaved PPO would improve CAI strength, additional panels were manufactured to attain 20-weight percent PPO in the preform interlayers. The CAI testing results (Table 2.5) indicate that the interleaved 20-weight percent PPO was effective in improving the ultimate compressive strength of the epoxy/anhydride system. The mean compressive strength for this system was 163.98 MPa, a 10.2% improvement above the control. Considering the CAI strength and standard deviation of the PPO modified epoxy/anhydride composite, it is likely that the limit of interleave toughening is around 20-weight percent for this system.

At this point, the internship objectives had been met but the intern wanted to pursue additional interleaved thermoplastic studies before departing to Hexcel Corporation to complete the Doctor of Engineering professional internship experience. Of the two high performance thermoplastics identified by the intern from Mitsui Toatsu (i.e., amorphous PI) and Phillips 66 (i.e., Ryton PPS), studies incorporating PPS in the epoxy/anhydride was pursued. The decision to interleave PPS was based on selecting a thermoplastic modifier close to the strain to failure of the brittle epoxy in order to promote the bridging particles to be drawn to failure thus utilizing their full energy absorbing capacity. The intern completed the manufacture and CAI testing of the 20-weight percent PPS-modified D.E.N.® 438/Norbornene Anhydride composite. It was found in using PPS that the impact damage zone size was reduced and compression after impact strength improved. The mean compressive strength for this system was 166 Mpa, an 11.5% improvement above the unmodified control (Table 2.5).

The results of these studies show that thermoplastic interleaving is effective in increasing the compression after impact strength of these epoxy/anhydride carbon fiber composites. The long-term goal of developing new toughening technologies for Dow-UT is an ongoing concern that will be pursued beyond the period of this internship. Furthermore, this study suggests that interleave toughening may not be necessary for these BMI composites cured according to the Dow-UT cure schedule and especially when the design criteria call for higher CAI strengths in this BMI.

Results

The internship objectives were met as new manufacturing methods were developed for infusion molded interleaved composites, and for fabrication of interleaved preforms. In addition, the intern evaluated the damage tolerance in BMI Cytec 5250-4RTM and D.E.N.® 438/Norbornene Anhydride thermoplastic interleaved composites. In meeting these internship project objectives, the project also supported the corporate objective of developing patentable toughening technologies for RTM applications. The information learned from manufacturing process development and fracture toughness testing supported developing patentable toughening technologies for Dow-UT.

In regard to evaluating the mechanical performance of thermoplastic interleaved composites, it was found that PPO is much more effective than PEI in increasing G_{IC} and G_{IIC} delamination toughness of BMI 5250-4RTM composites. In addition, PEI was found not to improve the CAI strength which was attributed to the poor adhesion and phase dispersion between PEI and BMI. It was also determined that PPO does not significantly improve the compression after impact strength of these woven carbon fiber BMI composites primarily because of the ductility of the cured matrix. Practically, for this BMI system, as a consequence of moderate improvements in G_{IIC} and CAI, and demonstrated neat resin ductile fracture behavior, interleave toughening may not be necessary.

When studying thermoplastic interleaving in D.E.N.® 438/Norbornene Anhydride carbon fiber composites, it was found that both PPS and PPO were effective in increasing the CAI strength. Specifically, PPS is more effective than PPO in toughening this brittle matrix composite. For these brittle epoxy/anhydride composites, thermoplastic interleaving was more effective in improving CAI and reducing the damage at impact. These findings support continuing efforts to toughen highly cross-linked, high temperature resins and their composites. Even with these modest improvements in CAI, thermoplastic interleaving can be a cost-effective method toward improving impact damage resistance. From this internship project research, it was learned that the special processing techniques developed from the manufacturing of these toughened composites may make interleaf toughening a practical and cost-effective approach.

INTERNSHIP AT HEXCEL CORPORATION

Introduction

The internship period with the Hexcel Corporation, in Dublin, California, commenced on July 1, 1998, and continued through February 28, 1999, although the official completion date was December 31, 1998. The intern was in the Research and Technology group, under the Supervision of Dr. Shaw Ming Lee, who is the Director of the Materials Science Group at Hexcel, responsible for Research and Technology (R&T). This internship focused on researching and developing both existing and novel honeycomb composite structure technologies for Hexcel.

The Hexcel Corporation is a global leader in the advanced composite materials industry. Hexcel manufactures a wide array of products from high performance fibers to prepregs, and honeycomb products to complete composite structures. Hexcel, being thrust into competitive global markets, has focused on executing a new paradigm, "Quality, high performance materials, at a low cost that are delivered on time" [46]. Currently, there is a revolution in the aerospace materials market similar to that experienced by the "Big Three" auto makers in Detroit, when the Japanese forced quality and performance at lower cost raising the global standards. Hexcel is steadfastly working to reorganize their global resources to target global customers and global products in order to gain and retain customers.

Internship Supervision

The internship supervisor was Dr. Shaw Ming Lee, who is responsible for managing large technical resources in support of Hexcel's current product lines and new product development initiatives. He also coordinates research efforts with manufacturing and marketing across all strategic business units, which include fibers, prepregs, honeycomb core, sandwich panels and composite structures (see Appendix A, Resume of Internship Supervisors).

Upon the intern's arrival at Hexcel, Dr. Lee set up a series of training and observation sessions, which included learning manufacturing details on the production of honeycomb cores, fabrication techniques of honeycomb structure composites, and continuous fiber resin impregnation methods. During the internship period, Dr. Lee supported this intern's efforts by encouraging investigation of all avenues necessary to obtain the project objectives. This leeway allowed the intern to interact with many professionals involved throughout Hexcel R&T. Once Dr. Lee gained confidence in the intern's abilities and understanding of the product concepts, he made it possible for the intern to participate in pertinent company discussions related to honeycomb product research. This opportunity revealed the inner workings of the company's material development process and related financial issues commonly discussed. Dr. Lee's support and encouragement throughout the internship was unwavering, which yielded positive outcomes from this internship experience. Dr. Lee's summary of this internship is given in Appendix B, The Internship Supervisor's Final Report.

Internship Position

The intern's position was that of Research Specialist. This may have been the formal description, but the intern operated much like a consultant. The intern was able to cross formal organizational lines to investigate by interviewing and forming business relationships with key personnel who were even remotely associated with the concerns of the impending project objectives. In addition, the intern's position allowed him to form unbiased opinions based solely on his research and select-information gathered from company personnel.

Dr. Lee called upon the intern frequently throughout the course of the internship to present research objectives, and current research findings and implications on behalf of the material research team to key company personnel, from marketing to manufacturing. The presentations were for upper management and to inform product development teams of the significance of the impending research. Frequently, project updates would be given to fellow colleagues from the resin development and the core manufacturing areas to allow for their input on research concerns.

In performing the tasks associated with the internship projects, the intern was also responsible for keeping record of the studies and observations made throughout the internship period.

Internship Objectives

The final internship objectives are described in a formal document dated August 20, 1998, and are endorsed by the interns committee (see Appendix C, Final Internship Objectives, Hexcel Corporation). They are summarized here. The Hexcel Corporation, during the period of this internship, was interested in researching two types of honeycomb core composites. One was the Nomex (i.e., Aramid fiber paper core) honeycomb core composites used for aircraft control surfaces, and the other was the aluminum honeycomb core panels that are used in secondary aircraft structure applications. For these research projects there were clear objectives to be attained during the internship, which will be described in detail in the following sections. The first project was the most time intensive and was named the Core-Skin Project, and dealt with understanding the composite structure and fracture mechanisms exhibited in drum peel tested Nomex honeycomb core composites. The second project dealt with understanding the fracture behavior of roller cart tested aluminum honeycomb core composites. These projects are described in the following sections.

The Core-Skin Project

Background

For the Core-Skin Project, the company objective is to develop a proprietary method to adhere damage tolerant facings to Nomex honeycomb core. The material development endeavor discussed here focuses on improving the drum peel strength of these novel composites by gaining an understanding of the fracture behavior, pertinent fabrication issues, the effect of fillet radius geometry between the core and skin, and interfacial adhesion. For the intended application, the drum peel strength is considered the most important material property in meeting the customer's design criteria. The existing honeycomb composite products incorporate a relatively brittle, unmodified resin matrix, which exhibit low drum peel strength in comparison to the new design criteria.

Therefore, in order to develop a higher drum peel strength, new company approaches to toughening the resin with thermoplastic and elastomer were investigated. Unlike thermoplastic interleaving (discussed in the previous section), in which the thermoplastic particles remain as discrete phases through cure, the modifiers used in this study solubilize in the resin matrix during cure. Developing these new modified resins and their prepregs resulted in higher drum peel strengths in honeycomb composites, which meet or exceed the customer's design criteria. The drum peel test strength results of both the thermoplastic and elastomer modified composites were at least tripled above the unmodified resin composites. The results of the drum peel tests, detailed morphological descriptions, and specific material properties will not be disclosed because the information is proprietary to Hexcel Corporation.

The assembly of the composites used in these studies consist of two facings, each comprised of two plies of resin impregnated (i.e., prepreg) woven carbon fiber, adhered to either side of the Nomex honeycomb core, thus forming a sandwich structure similar to that shown in Figure 3.1.

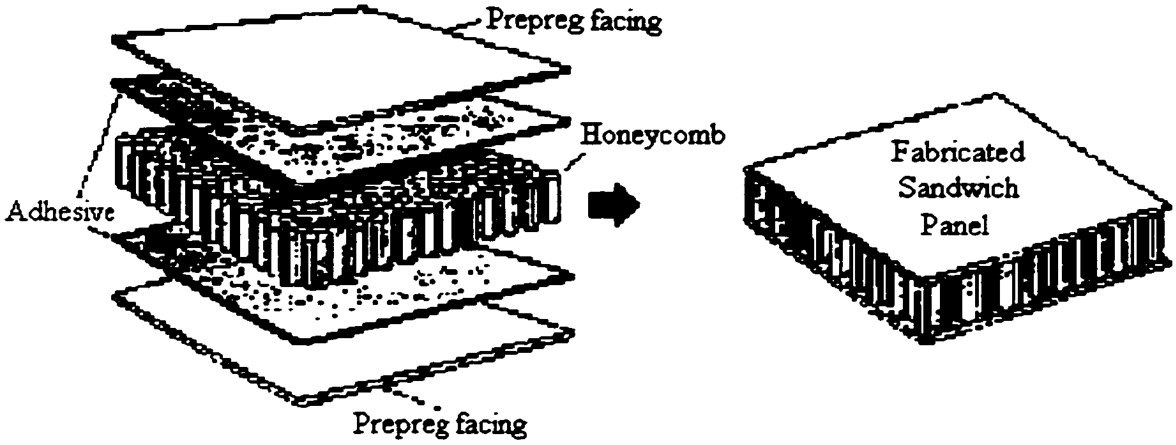


Figure 3.1. *Honeycomb core composite structure* [47].

The assembled composite structure is then sealed in an airtight vacuum bag, so a vacuum can be applied during the autoclave cure process (i.e., pressure cylinder with programmable thermal control). The nature of the autoclave process creates distinct differences in the composite structure (e.g., fillet formation) as a result of one skin being against the tool (i.e., a flat or contoured surface) and the other against the vacuum bag. The tool side skin has a smooth surface finish and is usually the exposed side when aesthetics and aerodynamics are a concern. On the other hand, the bag side skin has a surface that is irregular and rough. A honeycomb cell cross-section is shown Figure 3.2, which illustrates the contour resulting from the autoclave processing. The top surface is the bag side and the bottom surface is the toolside.

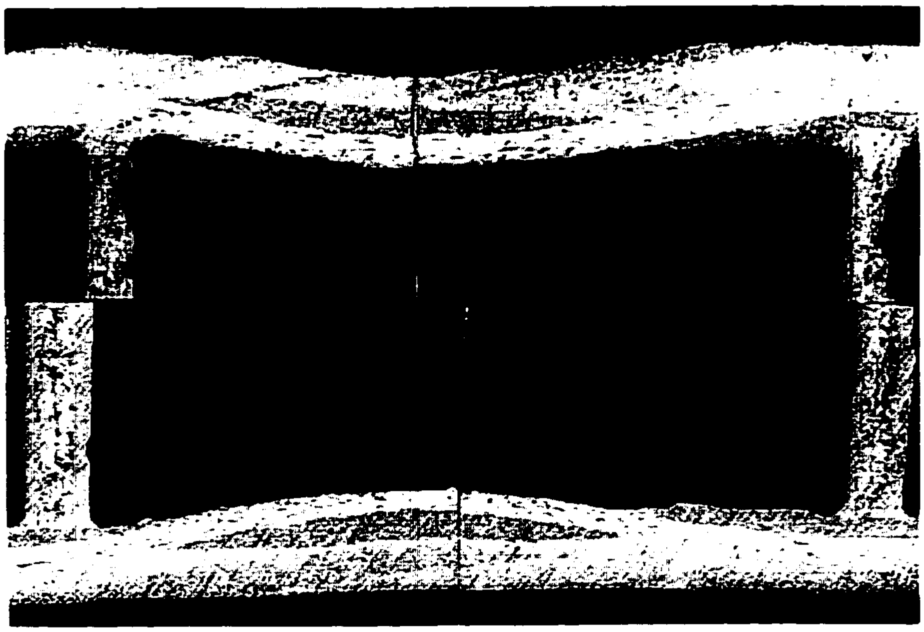


***Figure 3.2.** A cross-section of a Nomex, carbon fiber faced, honeycomb composite after autoclaving (magnification @ 20X).*

Once the panel is manufactured it is cut and prepared for drum peel testing [48]. Figure 3.3 shows an aluminum honeycomb core composite specimen prepared for drum peel testing. The tabs on the specimen are for clamping the specimen into the test fixture.

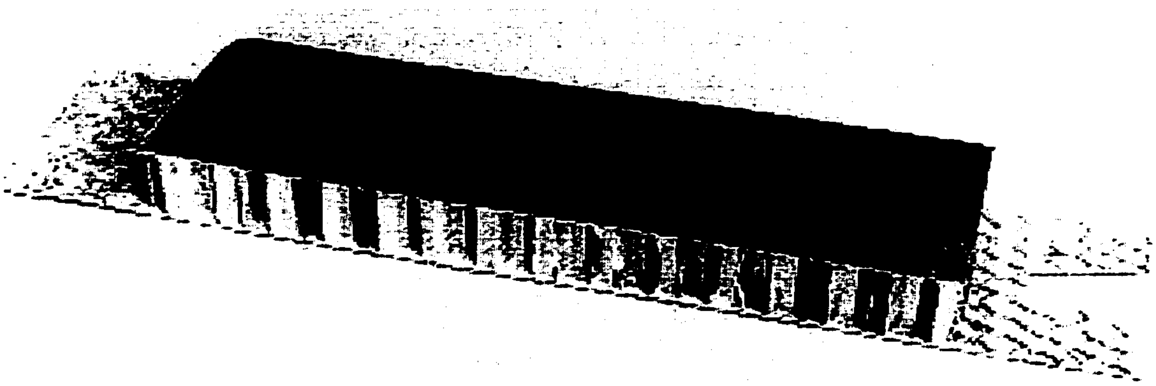


Figure 3.3. *Drum peel test specimen prepared from an aluminum honeycomb core panel* [47].

The specimen is then placed in a test fixture as shown in Figure 3.4. By pulling downward on the drum straps, the drum rotates and travels upward, thus, bending the secured facing around the drum and peeling the facing off the honeycomb core.

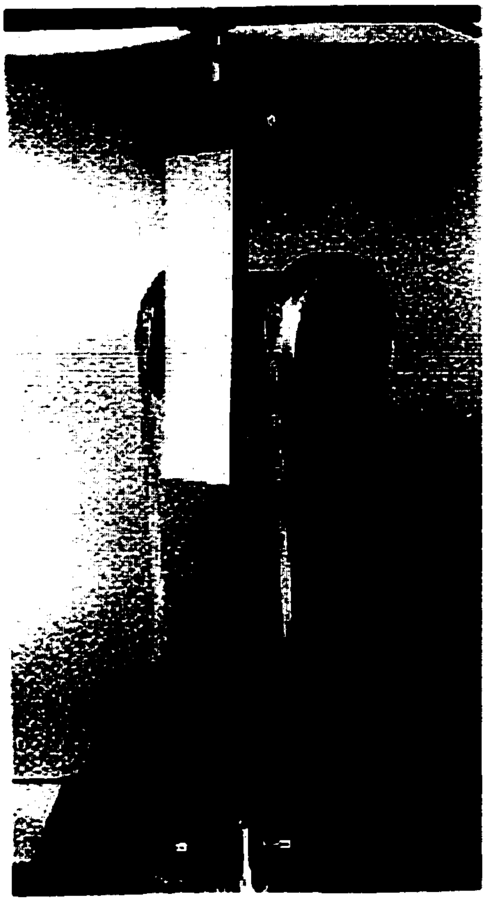


Figure 3.4. Drum peel test fixture with honeycomb composite affixed [47, 48].

Upon the intern's arrival at Hexcel the drum peel tests had been performed. The following sections describe the core-skin internship project.

Core-Skin Project Objectives

The principle objective of this internship project was to study the fracture behavior exhibited by these modified epoxy, Nomex honeycomb core composites after being subjected to a climbing drum peel test. Apart from this primary project objective, the intern also studied various material characteristics concerning composite consolidation, fillet radius geometry, and interfacial adhesion. The fundamental understanding gained in these studies will be used to design higher drum peel strength honeycomb core composites.

Initially the intern focused on learning as much as possible about Nomex honeycomb core with carbon fiber prepreg facing. This included reading company reports, which discussed different approaches aimed toward developing a higher drum peel strength composite. Existing company literature was reviewed that addressed such issues as resin toughness, as well as limited studies toward understanding the fracture mechanisms observed in the drum peel tests. These studies gave the intern a clear background about the company's efforts to develop toughened resins and their composites. In reviewing this literature, an evolution from brittle matrix resins to thermoplastic and elastomeric modified epoxies was revealed. The intern's studies complement this body of knowledge as the morphological influence on fracture behavior was clearly established, permitting the intern to evaluate the effectiveness of each matrix modifier for improving drum peel strength. Additionally, published literature was also studied concerning thermoplastic and elastomeric modification of epoxies, and their effects on resin and composite toughness [49-68].

In these studies the structural characteristics and fracture behaviors exhibited by the unmodified, elastomeric and thermoplastic modified epoxy prepreg skins were observed. These three epoxy types were used to impregnate the continuous woven carbon fiber prepreg used in studying these Nomex honeycomb composites. Therefore, the drum peel tests exhibited distinct differences in the fracture behavior and fracture mechanisms observed in these composite systems.

Project Tasks

The drum peel specimens used in this study were tested before the intern's arrival at Hexcel. In most cases the peeled skins were intact and adhered to the core. The observations regarding the fillet formation, and part consolidation were prepared from intact and undamaged portions of these specimens (i.e., areas where the skin had not been peeled away). Cross-sections from these undamaged regions were cut using a diamond blade table saw. The specimens were then set in a two-part epoxy and polished starting with 400 grit sanding paper and ending with a polishing cloth using an aqueous dispersion of 0.3 micron alumina powder. All polished samples were observed via optical microscopy (OM) to check the quality of the polish, and if not acceptable then additional polishing was performed. Once the specimens were polished, they were lightly gold sputter coated for scanning electron microscopy (SEM) analysis.

In studying the fracture behavior of these drum peel tested specimens, they were prepared so that the fracture surface on the honeycomb core could be observed. The honeycomb core specimens were cut using a diamond blade table saw and the fracture surface was cleared of debris with dry compressed air. Then these fracture surface specimens were lightly gold sputter coated and observed via SEM.

In studying the fracture behavior of these honeycomb sandwich composites the morphology was identified in order to better understand the observed fracture phenomenon. In identifying the morphology the intern gained tremendous understanding about the microstructure of the cured matrix, degree of dispersion of the modifiers and their affect on the preferred crack propagation path, and observed fracture mechanisms [69-71]. In this section the morphological features, observed fracture behavior and fracture mechanisms are discussed. Consolidation issues and interfacial adhesion observations made on these drum peel specimens follow these discussions.

Morphological Structure

The specimens prepared for the microstructure observations were cross-sections from unstressed and undamaged specimens that were set in epoxy, and polished. Then depending on the toughening modifier being observed an appropriate staining or etching technique was utilized. Then SEM micrographs were taken at the fillets where the morphology was likened to a neat resin observation. These observations gave insight to both the thermoplastic and elastomer modified epoxies morphology present in the skin facing. Etching the thermoplastic modified resin present in the fillet revealed a phase inverted microstructure and a limited degree of dispersion throughout the composite facings. Staining the elastomer-modified resin present in the fillet revealed a submicron domain size and a uniform degree of dispersion in the facing. These observations would prove critical to understanding the drum peel fracture behavior and the associated fracture mechanisms.

Because of SEM analysis, the morphological structure of the matrix resin, fillet, and the penetration of the toughening domains into the carbon fiber tows were clearly observed. The penetration of the matrix modifiers into the fiber tow bundles was key in understanding the fracture behavior observed in these drum peel specimens. Making these observations aided in the understanding and description of the fracture behavior evidenced in these composites.

Fracture Behavior

The observed fracture behavior and fracture mechanisms were directly influenced by the morphological characteristics exhibited in the composite skin and adhesive fillet. Upon first viewing the fracture surface features of the thermoplastic modified drum peel specimens one immediately notices the amount of resin and broken fiber remaining on the core (Figure 3.5).

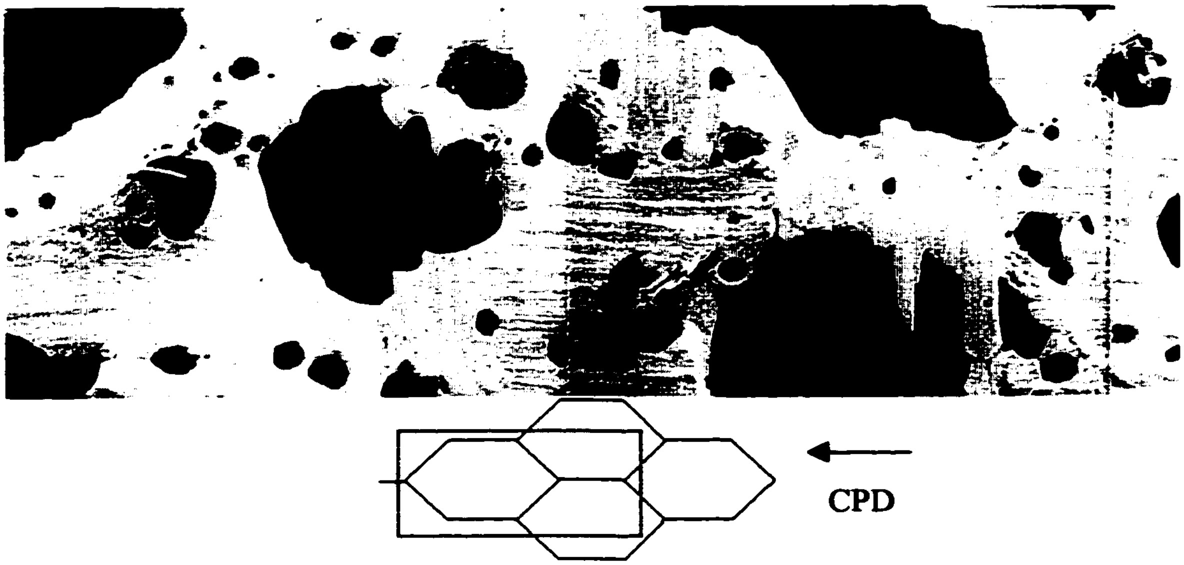


Figure 3.5. Fracture surface of thermoplastic modified epoxy and fractured carbon fiber remaining on the Nomex honeycomb core after drum peel testing (magnification @ 21.4X). (CPD) Crack propagation direction.

The SEM micrographs show that there are numerous carbon fibers remaining on the core in both longitudinal and transverse orientations to the crack propagation direction with limited thermoplastic morphological domains on the fracture surface. The limited dispersion of the thermoplastic phase on the fracture surface is consistent with the morphological observations made in the composite skin cross-section. These untoughened regions within the ply are less fracture resistant and this is evident in observing the brittle fracture features which appear as smooth, clean fractures on the on the surface (Figure 3.6).

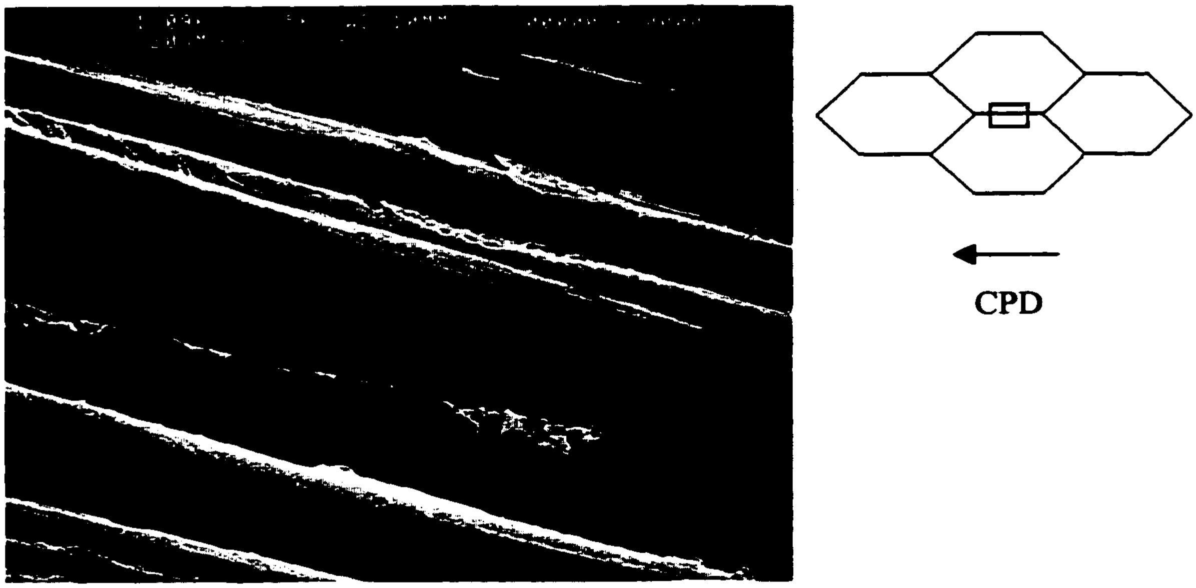


Figure 3.6. The drum peel fracture surface of the thermoplastic modified honeycomb composite where the carbon fibers are oriented parallel to the crack propagation direction. (CPD) Crack Propagation Direction.

Since the thermoplastic modified matrix of the fillet was tough (i.e., fracture resistant) the crack propagated through the less fracture resistant zones within the woven ply tows (i.e., intralaminar fracture path). This crack propagation behavior explains the numerous fiber fractures in both the longitudinal and transverse directions on the honeycomb fracture surface (Figure 3.7).

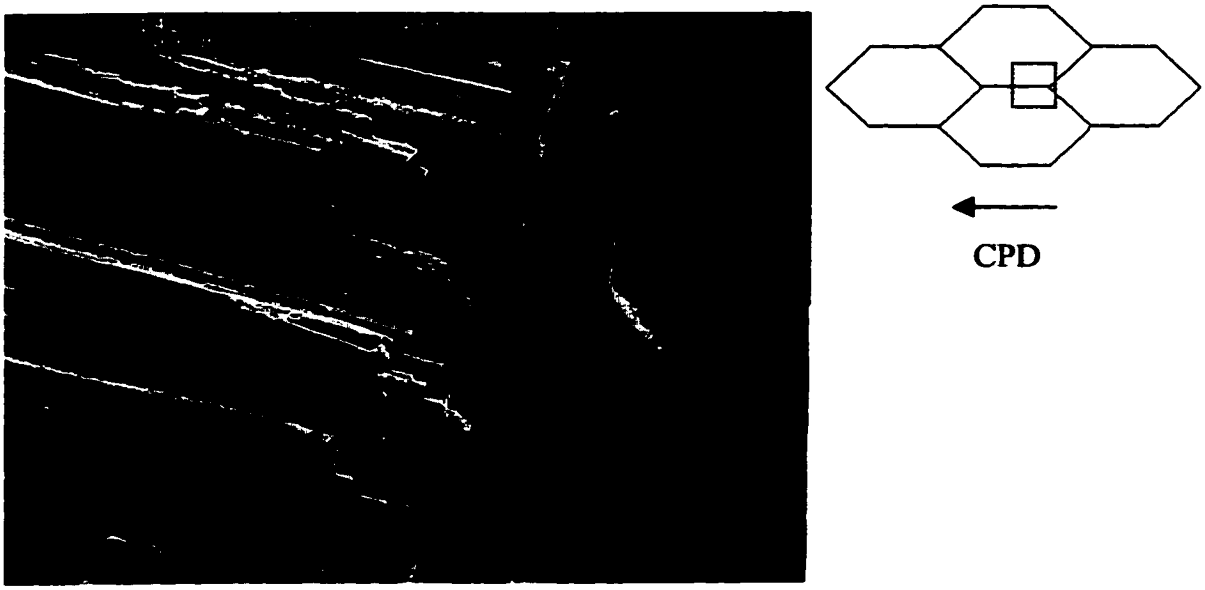


Figure 3.7. Fiber breakage characteristic of the thermoplastic modified epoxy system. (CPD) Crack Propagation Direction.

This thermoplastic modified epoxy system essentially promotes "fiber bridging" which has been shown to improve Mode I fracture toughness and may contribute to higher drum peel strengths. Fiber bridging, by requiring energy to break the carbon fibers, improves the fracture toughness [54].

Another fracture mechanism exhibited by this resin morphology is the plastic drawing and deformation of the thermoplastic in the phase-inverted regions (Figure 3.8).

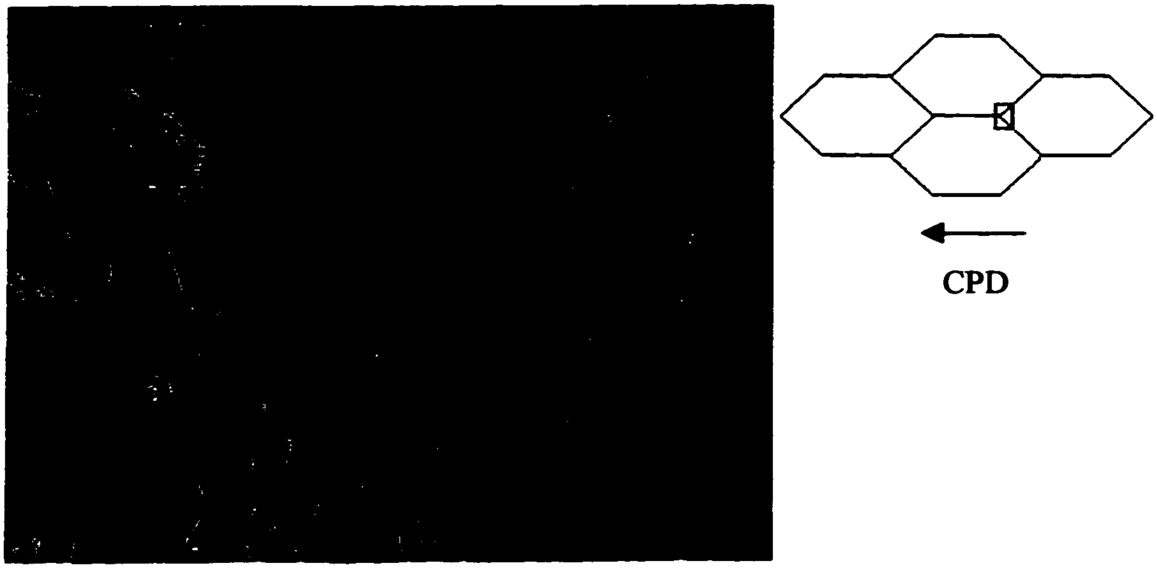


Figure 3.8. Toughening via plastic deformation of the thermoplastic phase. (CPD) Crack Propagation Direction.

In studying these drum peel cross-sections near the crack tip it was observed that cracking primarily occurred in the ply bonded to the core. As the crack propagates through the intraply, the crack deflects frequently with many microcracks stemming from the main crack. These crack deflections and additional crack branches create crack surface area, which in turn increases the fracture energy needed to propagate a crack.

In observing the elastomeric modified drum peel specimens one notices less fillet adhesive remaining on the core and fewer broken fibers in contrast to the thermoplastic modified honeycomb composites (Figure 3.9).

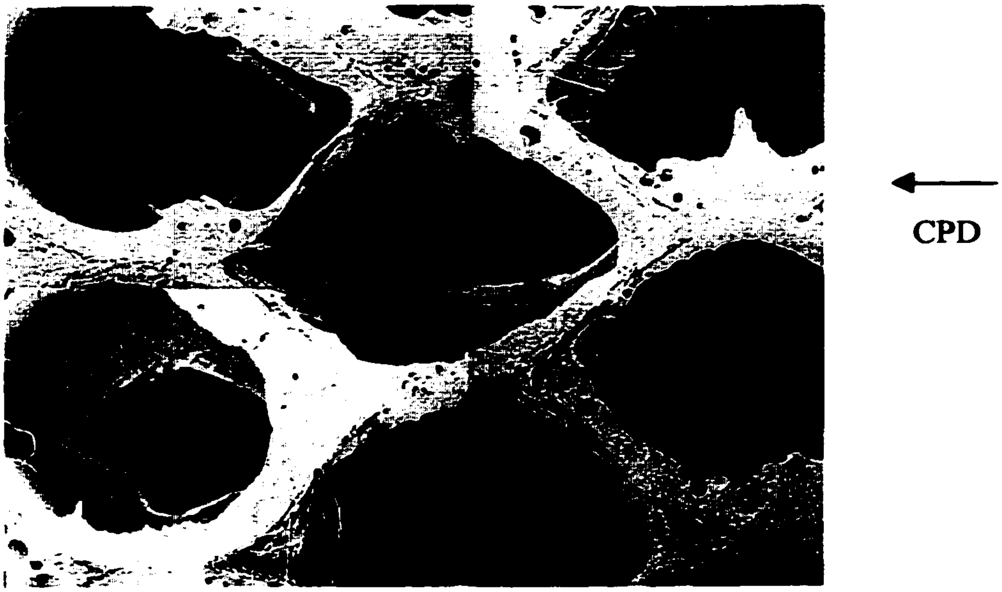


Figure 3.9. *Fracture surface of the elastomeric modified epoxy remaining on the Nomex honeycomb core after drum peel testing (magnification @ 21.4X). (CPD) Crack Propagation Direction.*

The fracture and separation of the skin from the core during the drum peel test is primarily a result of fillet fracture at the bond interface. Due to the fracture resistance and uniform dispersion of the elastomer modified resin, cracks rarely propagate into the carbon fiber ply adjacent to the core. By virtue of the disperse elastomer morphology, intraply fracture is deterred which promotes fracture at the adhesive fillet. Therefore, there are minimal amounts of broken carbon fibers on the fracture surface. Of the fibers remaining on the core most are from tows that were aligned perpendicular to the crack propagation direction.

The predominant features on the fracture surface of these drum peel tested specimens are the evidence of elastomer cavitation and matrix shear yielding seen as gross matrix plastic deformation [72-74]. Via SEM it was found that the elastomer domains from the unstressed to the cavitated state conservatively doubled in size (Figure 3.10).

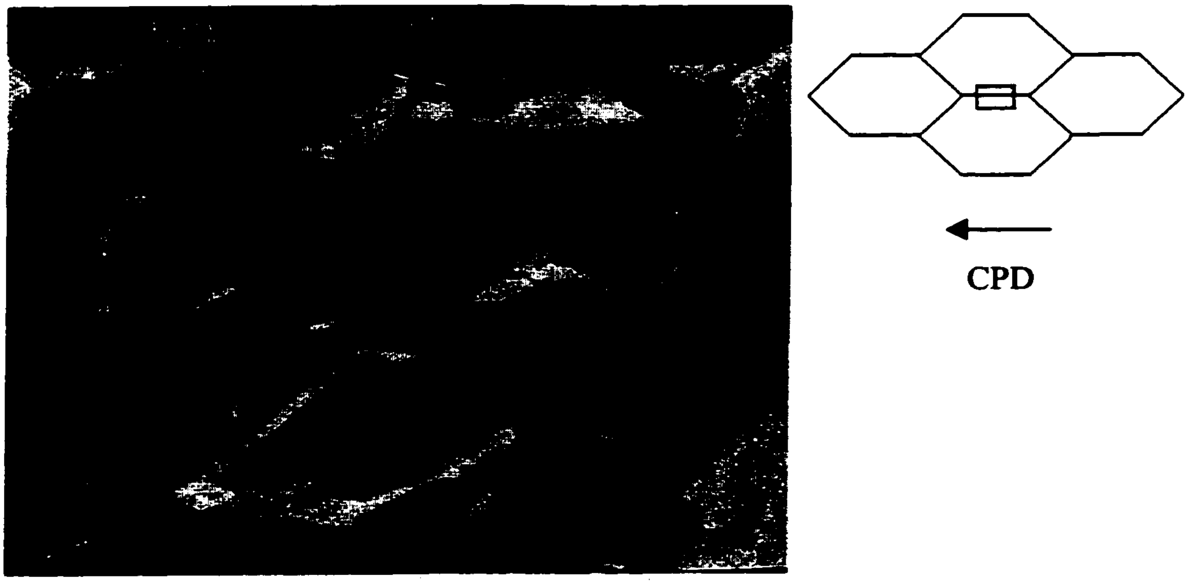


Figure 3.10. Representative fracture surface of elastomeric modified epoxy remaining on the Nomex honeycomb core after drum peel testing. (CPD) Crack Propagation Direction.

Structural Observations

Upon preparing the composite cross-sections from undamaged areas of the drum peeled specimens, observations regarding fillet radius geometry, composite consolidation, and morphological issues were studied. Fillet formation in these composites is formed when the viscosity of the adhesive is reduced, and both capillary and surface tension effects create a fillet (Figure 3.11). Using the SEM digitizer to take measurements of the fillet radius geometry it was shown that smaller fillets form on the bag side and larger fillets form on the tool side, yet the drum peel strength is higher on the bag side.

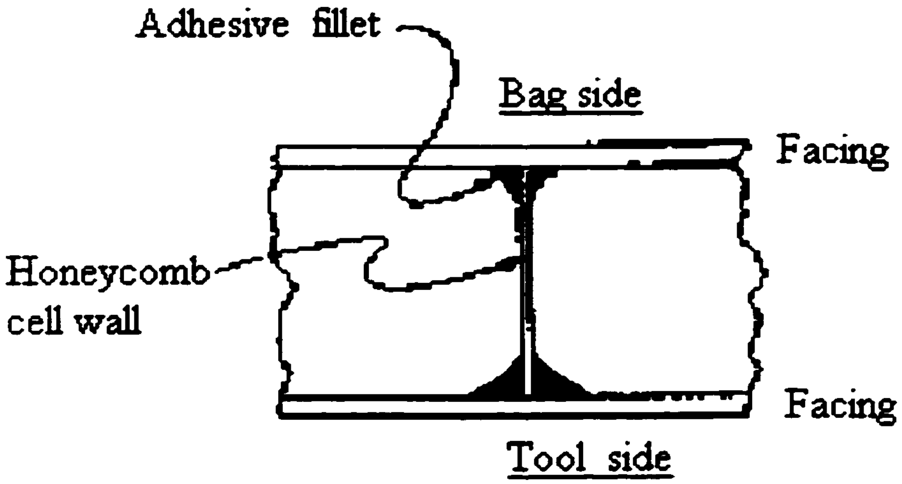


Figure 3.11. Cross-section of honeycomb structure emphasizing the adhesive fillet formation and size.

Previously, it was believed that larger fillets formed on the bag side, therefore, explaining the higher drum peel strength on this side. However, in making consolidation observations it was found that there was consistent void formation on the toolside. These toolside voids predictably form toward the center of each honeycomb cell where the consolidation pressure is the lowest. In Figure 3.2, the upper surface (bag side) contour is a result of the uniform autoclave pressure exerted on this face. Whereas, on the lower surface (toolside), the resultant force of the autoclave pressure is exerted onto the honeycomb cell walls, and transferred to the toolside skin to obtain good localized consolidation at the cell wall. Voids consistently form in the middle area of the bottom surface. The toolside skin only exhibits void free, local consolidation at the cell walls (given adequate local resin content), whereas the bag side experiences uniform autoclave pressure coupled with the vacuum bag pressure resulting in excellent consolidation (i.e., little to no void content). Whether on the bagside or toolside, fillets do ultimately form effectively to bond the facings to the core. The most significant observation resulting from studying the fillet geometry was that smaller fillets on the bag side yielded a higher drum peel strength than the toolside, which exhibits larger fillet radii. The intern deduced that the void content in composite skins was responsible in reducing the drum peel strength on the toolside. This deduction was bolstered by fracture behavior observations of the cross-section made in the intraply at the crack tip region of the drum peeled specimens.

A solution to reduce the void content on the tool side would be to lay-up the toolside skin and debulk. Debulking is a process in which vacuum bagging, and autoclaving consolidate a composite laminant (i.e., in this case the two plies of the toolside skin) in order to reduce voids. This additional step would evacuate any trapped air within the un-cured facing and residual volatiles by reducing the viscosity of the resin to allow any gases to escape.

In studying the interfacial adhesion of the resin systems used in this study, it was observed that these epoxies exhibited strong interfacial bonds with the carbon fibers. This was evidenced by substantial resin remaining on the fibers after fracture (Figure 3.12). In addition, the interfacial strength between the honeycomb cell walls and the adhesive fillet was strong as fractures mainly occurred at the fillet to skin adhesive interface.

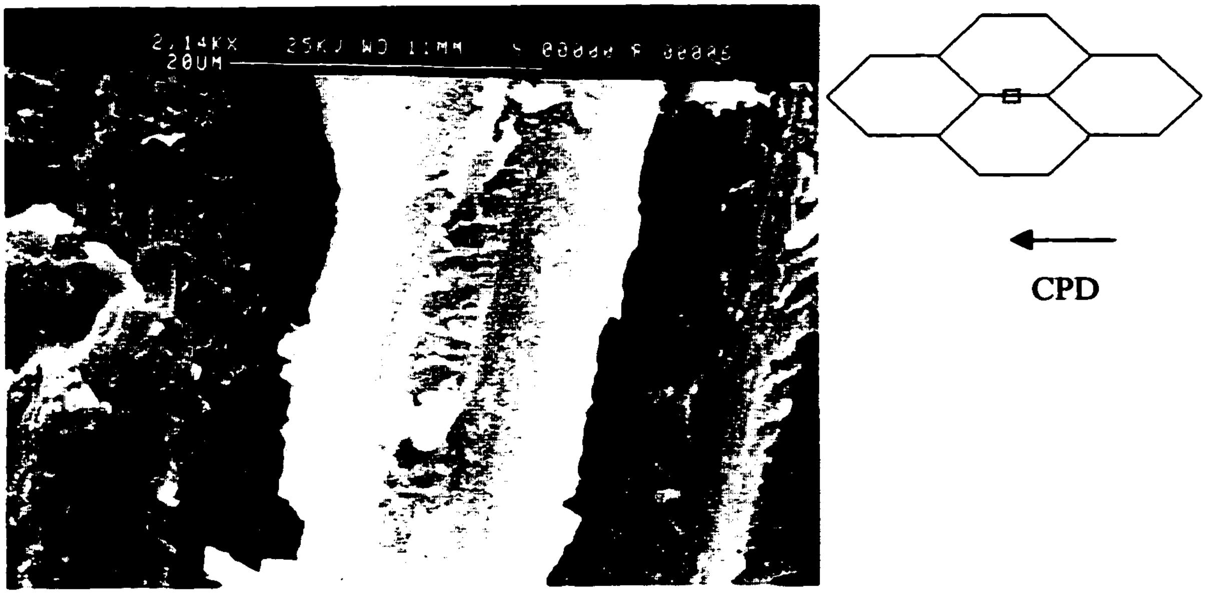


Figure 3.12. The amount of resin remaining on this carbon fiber after fracture is an example of good interfacial adhesion. (CPD) Crack Propagation Direction.

Results

These results imply that possibly other toughening approaches and technologies can be utilized in these composites to achieve higher drum peel strengths. This study suggests that tough fillets may force interlaminar fracture in a less fracture resistant facing. On the other hand, more fracture resistant facings promote fillet adhesive failure. In addition, these studies suggest that there is significance in the manner of carbon fiber prepreg lay-up and its orientation to the core that can improve drum peel strength.

Continuing Development

As a result of this work, it has been observed that there are benefits to the toughening approaches discussed here. In light of this work, studies are continuing to explore other novel toughening approaches, which can selectively promote beneficial fracture mechanisms.

This material research endeavor will require material studies of existing company technologies as well as developing novel toughening approaches. Once the best combination of resin and fiber type are found to create a composite which meets the customer's design criteria, then the next steps toward large scale production of damage tolerant honeycomb composites will be pursued.

Core-Skin Internship Project Summary

The internship objective was met as knowledge was gained about the fracture behavior and the fracture mechanisms exhibited by these modified epoxy, Nomex honeycomb core composites in drum peel testing. Also gained was knowledge in understanding the important relationship between morphological structure and its influence on drum peel fracture behavior. Using the knowledge gained through these studies, novel proprietary toughening approaches are being pursued to improve drum peel strength and other mechanical properties. The results of this study when compared to the drum peel test results of the specimens studied enhanced the understanding of the effectiveness of each toughening method. These approaches to toughening, and their effectiveness toward improving drum peel test strengths were not discussed here for proprietary reasons. In addition to meeting this project's internship objectives, knowledge was gained toward understanding the significance of fillet radius geometry and its effect on the drum peel strength. In these studies, it was determined that skin consolidation was an important factor that influences drum peel strength. Conversely, given the results of this study, the company can realize a competitive advantage by exercising technologies currently available to them.

The Roller Cart Project

Background

The roller cart project focuses on extending the roller cart fatigue life of Hexcel's aluminum honeycomb core composites. These materials are "sandwich" laminated composites consisting of two outer skins, each skin comprised of two plies of resin pre-impregnated (i.e., prepreg) unidirectional glass fiber reinforcement. These skins are then adhered to each side of a honeycomb core creating the composite sandwich structure (Figure 3.1).

The assembled composite is then placed in a heated press to consolidate the panel by curing the prepreg resin and the adhesive. As the viscosity of the adhesive is lowered both capillary and surface tension effects draw the adhesive resin toward and along the cell walls of the honeycomb creating an adhesive fillet. Once the panel completes the curing cycle in the press the panel is then trimmed, drilled and prepared for roller cart fatigue testing.

The roller cart fatigue test is a standardized test specified by Boeing in their BMS 4-17, 4-20 and 4-23 floor panel specifications, in which an edge supported honeycomb composite panel (21"×39") is bolted to a frame and subjected to a weighted and rotating three caster wheel apparatus (Figure 3.13). The edge supported aluminum honeycomb core panel under this rotating load experiences both global scale deflections and severe localized deflections with each pass of a caster wheel.

Figure 3.13. Roller cart test apparatus.

An inconsistency of the test itself is the subjective nature with which a failure is identified. A failure is loosely defined and includes such things as a panel that exhibits skin cracks to a panel that has frayed skins with exposed core. This is an important point because when a panel "fails", its number of cycles to fatigue are recorded and used as quality control information. For the panels used in this study a roller cart failure is defined as the onset of crack initiation which ranges from a surface whitening of the composite skin to a surface crack no bigger than two-tenths of an inch. It should be noted that roller cart testing, by coordinating with key testing personnel, was halted immediately upon the onset of failure damage. Recently, the “failure” criteria specification was revised. A failure is now defined as a permanent depression in the surface indicating subsurface core buckling or skin damage (top side or bottom side) exceeding five-tenths of an inch, whichever occurs first.

Roller Cart Project Objectives

The purpose of this project was to gain an understanding of the roller cart failure process of aluminum honeycomb core composites. The project concentrated on studying the dominant fracture initiation and the resulting fracture behavior. In addition to this primary project objective was to gain an understanding of the macroscopic structure formation (i.e., fillet formation and inherent features) of the composite. In applying the understanding gained in these studies, it is expected that the roller cart panel performance can be improved. In order to better distinguish the microstructure characteristics that influence panel performance, studies were focused on noting the differences between high (>120k rotational cycles), and low (<30k rotational cycles) cycle fatigue specimens. Various morphological characteristics via optical microscopy (OM) and scanning electron microscopy (SEM) were observed related to such issues as part consolidation, interfacial adhesion, crack propagation and crack initiation. These observations gave insight toward the possible solutions (e.g., related either to materials processing, manufacturing or mechanical testing) in manufacturing a quality, fatigue resistant, honeycomb composite product.

The first step toward accomplishing the project objective was focused on learning as much as possible about these aluminum honeycomb core composites. This included reading company reports that attempted to answer questions concerning the performance of these panels. The company reports addressed important issues regarding understanding the roller cart testing methods and the manufacturing processes to make these composite panels. Through literature review and interviewing company personnel much was learned, but it was particularly essential in this project to call on experience and academics to separate fact from bias [75,76]. This was important due to the fact that many key employees had many ideas and biases on improving roller cart performance. Due to the intern's prior experience at The Dow Chemical Company, he was well prepared to process the information and extract the critical issues necessary to attain the project objectives.

There were many theories as to why some roller cart tested panels would occasionally perform below the standard (i.e., fatigue cycles greater than or equal to

120k rotational cycles is passing for a roller cart test). These theories included resin distribution in the prepreg, part consolidation, part manufacturing processes, roller cart testing methods, resin chemistry, adhesive chemistry, and adhesive fillet formation. In addressing a few of these theories, the intern thoroughly evaluated the roller cart test and testing methods with employees to ensure that the panels used in this study were tested to specifications. There were no problems encountered with the roller cart test procedures at that time, except for inconsistent failure delineation. In addition, raw materials data relating to the manufacture of these resins used in manufacturing the panels in this study was obtained to research any correlation to roller cart performance. However, there was no distinguishable difference between the resin formulations of the various roller cart panels tested to panel performance. Attempting to identify the critical issues associated with these aforementioned concerns required interaction with many engineers, processing, and testing personnel outside of the intern's immediate research group. From these probing discussions, it became clear that there were many implications to this work that could affect and positively impact the quality in the manufacturing process from receiving the raw materials to preparing the final product. Investigation and discussion of these complex issues provided a sense of direction to the research that could answer many lingering questions.

Project Tasks

In the analysis of the tested panels, the first objective was to prepare microscopy specimens from relatively high cycle (i.e., >120k rotational cycles) and low cycle (i.e., <30k rotational cycles) fatigue panels. Some roller cart fatigue panels used in this study had previously been tested before the intern's arrival. The panels that were the least damaged (i.e., minor composite skin surface damage) were separated from those that had severe damage (i.e., broken and frayed composite skins, exposed or crushed honeycomb core). From these specimens it was believed that the cause of failure and possibly fracture initiation could be determined. However, shortly after beginning the cross-section observations it was clear that damage in some panels was too extensive to distinguish any one cause of fracture initiation. At this point the intern coordinated with key testing personnel to stop the roller cart test upon observing the onset of skin fracture or skin whitening. This skin whitening had been discussed with the key testing employees, and it was learned that damage initiated skin whitening (to be differentiated from inherent white visible surface streaks and striations) was usually a precursor to visible surface skin cracking.

Once the panels to be studied had been defined to meet the project goals, microscopy sample preparation began. The samples prepared from these roller cart tested specimens were cut from the bottom side of the roller cart panel just under and to the side of the roller cart caster wheel path. It should be noted that the damage to these panels occurred on the bottom side, where the topside was in contact with the weighted and rotating wheels of the roller cart apparatus. The bottom side is where most, if not all, of the damage was observed in the roller cart tested panels. The specimens were cut in both the transverse and longitudinal directions in order to make observations in both plies of the skin. The samples were cut using a diamond blade table saw. The specimens were potted in a two-part epoxy. A polishing method beginning with 400 grit sanding paper and ending with a polishing cloth using an aqueous dispersion of 0.3-micron alumina powder was utilized. All polished samples were observed via OM to check the quality of the polish and if not acceptable then additional polishing was performed.

Once a quality specimen had been prepared then macroscopic observations were made which helped in outlining the focus of SEM analysis. In performing OM (i.e., 1000X magnification and below) features such as voids, foreign debris, fillet formation and large scale cracking could be seen. Next, the same specimens used for OM were lightly gold sputter coated to prevent charging of the specimens. In performing the SEM analysis (i.e., 1000X magnification and above) microscopic features such as the following were observed; matrix/fiber interfacial adhesion, voids formation, foreign debris contamination, microcrack initiation, fracture face friction initiated microcracking and fillet radius measurements using a built-in SEM digitizer (Figure 3.14).

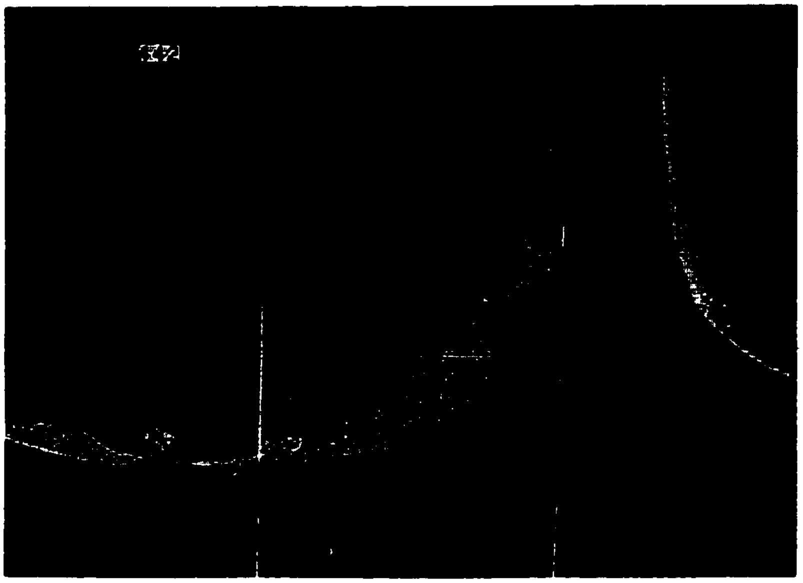


Figure 3.14. Cross-section of a fillet with vertical digitizing markers enabled.

Of the observations that were attempted on the composite cross-sections but were not effective was the effect of matrix modifiers on the crack propagation path. Etching or staining of this matrix resin proved unsuccessful due to the heavily modified resin matrix (i.e., fire retardants, fillers, and toughening additives).

Optical Microscopy

In studying the roller cart specimens via OM microscopy microcracks within the specimens were located, but the most noticeable feature observed was voids in the composite skins (Figure 3.15). It became systematically clear in OM observations that the high cycle roller cart tested panels had a lower void content, whereas the low cycle roller cart tested panels had a higher void content.

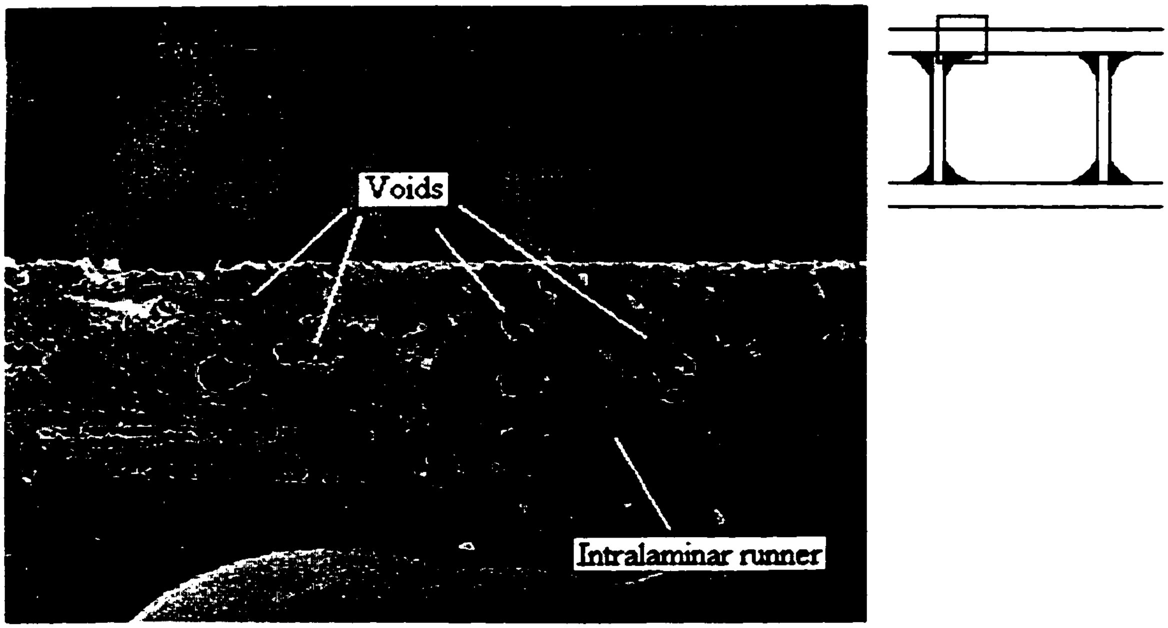


Figure 3.15. Aluminum honeycomb composite cross-section revealing inherent void formation.

Certainly, a relation between two-dimensional void area and roller cart cycles to failure could be made. In the intern's literature review it was found that this study had already been performed within Hexcel and there was no significant correlation found between void content and roller cart performance. However, it was evident to the intern that there was a relation between voids and performance in the present study, as these polished specimens were revealing. However, an explanation eventually would be found by making observations about these panels that had not yet been described. These key observations were made via SEM and will be discussed in the following section.

Locating the microcracks via OM was essential in understanding where the predominant crack initiation was occurring. At relatively low magnification (i.e., approximately 600X) a few honeycomb cells could be seen at one time. Figure 3.16 shows a honeycomb cell cross section with a matured crack along the cell wall. This crack appeared on the composite skin as a cloudy white aberration that was found via SEM to be a result of concentrated microcracking within the composite skin. Microcracking at the fillet/facing interface along the honeycomb cell consistently occurs at this locale of the fillet. As microcracking at these locales intensifies within the facing, a white cloudy effect is observed that is attributed to light scattering within the facing. This phenomenon is described in detail in the following section.

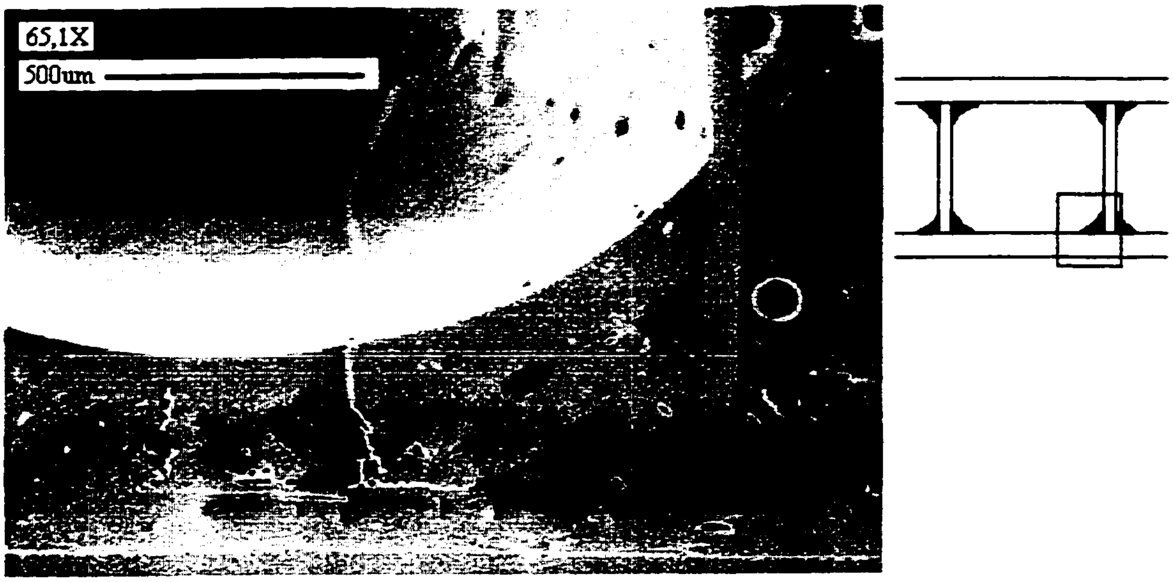


Figure 3.16. *Fatigue crack growth along a honeycomb cell wall. Along the crack length, microcracking into the facing creates cloudy white aberrations on the composite surface.*

Scanning Electron Microscopy

The specimens that were analyzed via SEM revealed structural characteristics and fracture behavior not previously identified in these composites. The following observed structural characteristics are those made on tested panels but the specimens were well removed from the roller cart caster path. In these specimens there was no testing induced damage observed. The observations that follow are those that would be expected to be seen in most newly manufactured panels (i.e., untested panels).

In manufacturing the honeycomb composite sandwich structure, an epoxy adhesive is used to adhere the composite skins to the honeycomb cores. As the epoxy adhesive is heated during the consolidation process the resulting drop in viscosity coupled with capillary and surface tension effects forms a fillet. These fillets form consistently at the walls of the honeycomb and the skin. The adhesive used for this bonding is supplied in a film form (containing a lightweight nylon fabric scrim as a carrier) on a release paper. The bond formed using this product does create a uniform fillet formation. The adhesive layer that is used to adhere the composite skins did not reveal any significant void formation. Using the prepared highly polished samples an SEM digitizer was used to measure the fillet radius formed at the interface. It was found that the fillet radius geometry was statistically similar for all specimens studied independent of the number of roller cart cycles. Therefore, it was concluded that fillet formation did not affect panel roller cart performance in the panels studied.

In using SEM the most noticeable inherent structural characteristics of these composites was the void content, void shape and void location. In observing these composite panels, one may notice short white streaks which usually span one to two honeycomb cell widths. The most noticeable of these streaks are those that appear inside the outer ply of the skin. These streaks via SEM were found to be elongated voids within each ply of the composite skin. These voids are oriented and elongated along the tow direction within both unidirectional plies. These voids, named intralaminar runners (i.e., within the woven ply), depending on the local resin content may disappear at the cell walls of the honeycomb structure due to the consolidation pressure exerted by the heated press during manufacture. Figure 3.17 shows an intralaminar runner located in the inner ply that is bonded to the core. This void was visible as a short white streak on the undamaged composite skin surface.

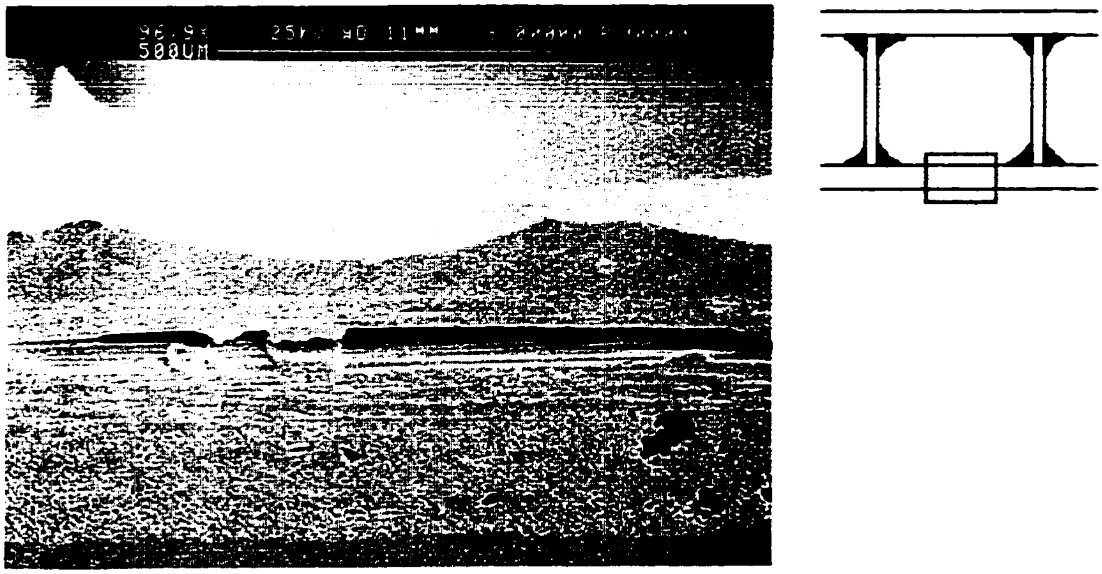


Figure 3.17. Cross section of an intra-laminar ply runner.

In observing these composite panels, one may notice striations (i.e. long streaks), which can be as long as six inches in length. These types of striations were identified as elongated voids within the interlaminar region (i.e., between plies). These voids, named interlaminar runners, depending on the local resin content may disappear and reappear over many cells of the honeycomb structure. This disappearing effect is due to the consolidation pressure exerted by the heated press during manufacture.

In Figure 3.18 an interlaminar runner cross-section is shown and a void clearly is observed at the interface of the two reinforcement plies. In this photo, the width of the void is shown, but the length of the surface visible void before sectioning was about four inches. Also present in this micrograph is an intralaminar void running transverse to the interlaminar void.

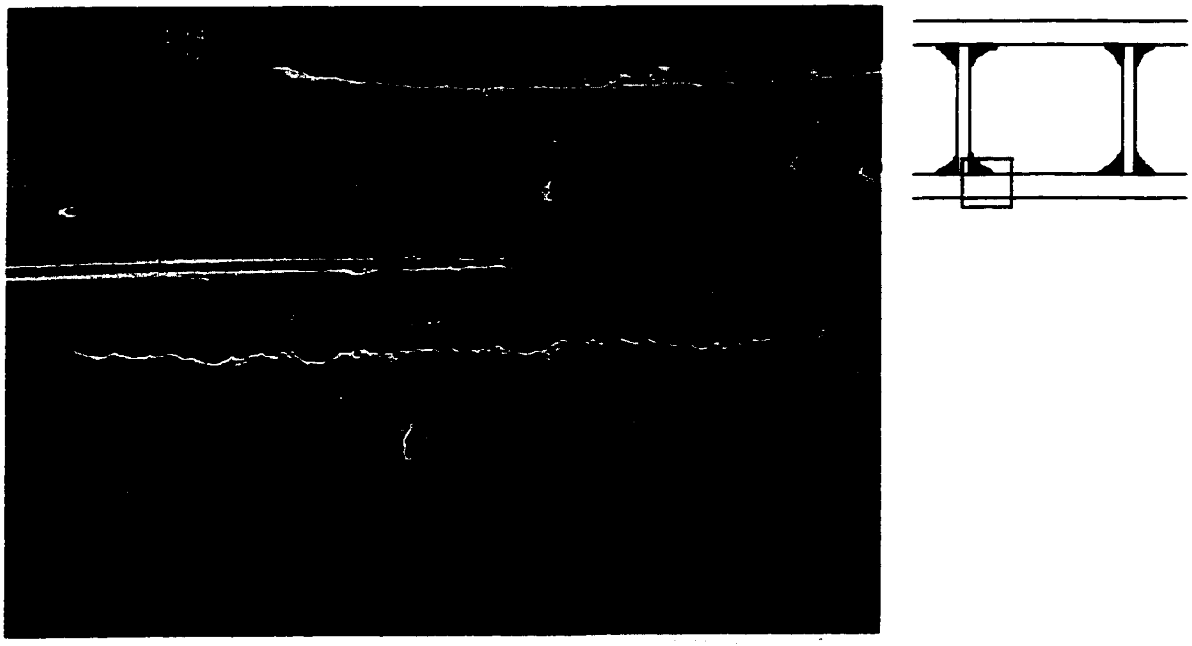


Figure 3.18. *A cross-section of an interlaminar runner with a width of approximately one millimeter.*

In a few instances, one may observe a panel that has surface grooves along the outer ply fiber direction (Figure 3.19). The grooves usually range in depth from a few microns to the entire outer ply thickness (approximately 400 microns). The panels with surface striations typically exhibit an extraordinary number of voids, and both interlaminar and intralaminar runners. This phenomenon is attributed to resin starvation in which the resin content in the prepreg is unusually low.

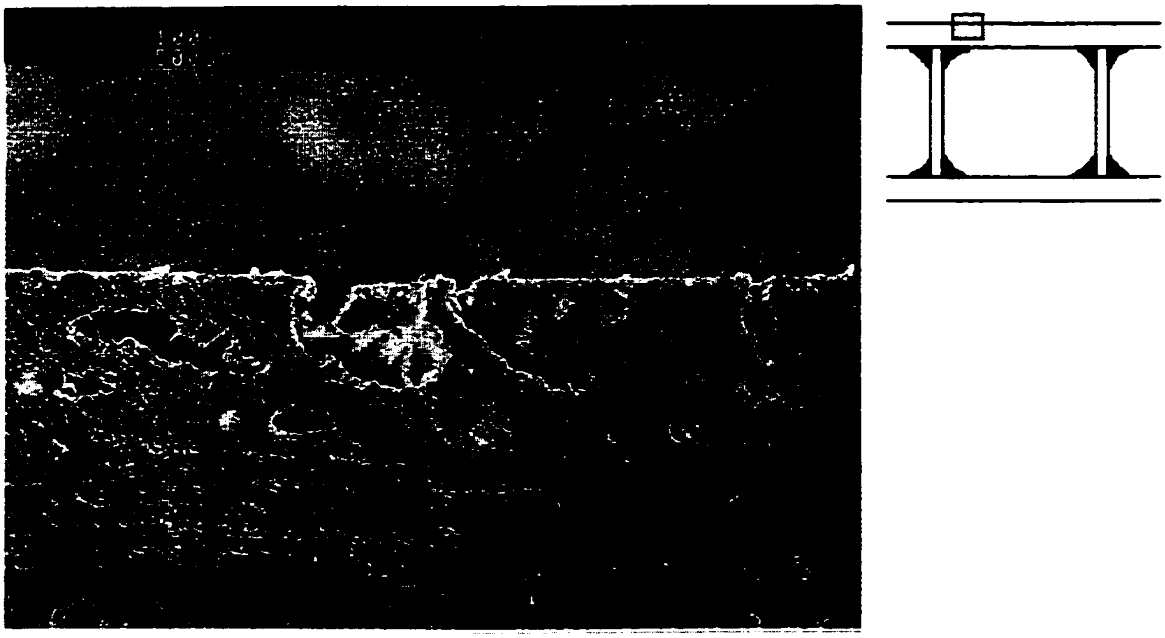


Figure 3.19. *Cross-section of a surface showing striations appearing as grooves on the composite surface and numerous voids present in the vicinity of the striation.*

It has also been observed that foreign debris introduced to the composite materials can cause void initiated microcracking by virtue of the poor bonding between the foreign debris and resin matrix. Figure 3.20 shows the cross sections of two incompatible objects debonding from the resin matrix. These objects may be nylon scrim fibers that are present in the adhesive film or other foreign debris. In either case, these objects are matrix anomalies that can initiate microcracking. Sources of debris may simply be from the assembly area, including common dirt and dust, or dust from cutting and shaping the honeycomb core.

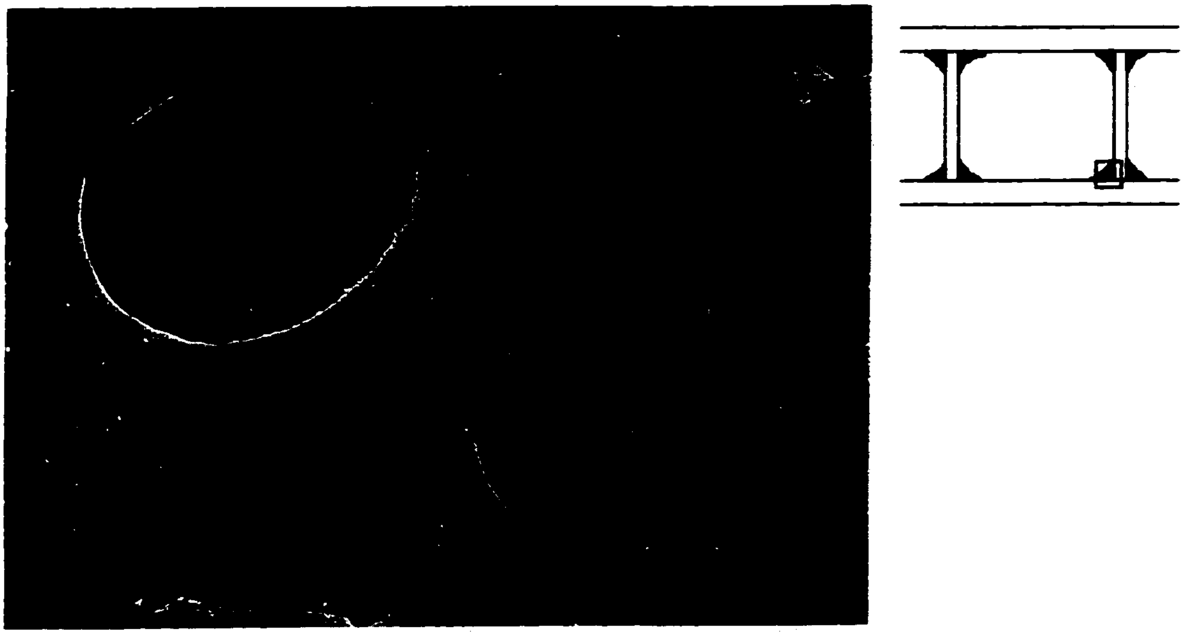


Figure 3.20. Debris present in the interlayer showing debonding occurring at the interface.

Fracture Behavior

In studying the roller cart specimens via SEM it was found that that fractures initiate predominately by resin/fiber de-bonding, foreign debris and void initiated microcracking which includes intralaminar and interlaminar voids. Of the panels observed in this study the void initiated microcracking was the predominant cause of panel failure. Given a panel with good consolidation (i.e., no voids) it is presumed that fatigue initiated fiber/matrix debonding would be the predominant cause of panel failure. This presumption is bolstered by observations in well-consolidated areas of the roller cart tested composites (>120k rotational cycles) where microcracks (a few microns in length) occur at the fiber/matrix interface. However, with relatively large (when compared to debond microcracks) and numerous voids initiating microcracks, this significantly reduces the amount of crack growth necessary to result in through thickness ply and skin cracking.

Voids, intralaminar and interlaminar runners, do promote microcracks. The intralaminar microcracks were observed to grow contained within each ply during the early stages of panel fracture failure. Since the panels used in this study were not subjected to testing after the appearance of "failure" most of the microcracks that appeared in the intralayers did not cross the resin-rich interlaminar region. In these specimens it was observed the crack propagation would deflect at the resin interlayer. However, it was also observed that in the presence of interlaminar runners these crack deflections at the interlayer would coalesce and create delaminations of the skin plies.

Debris in the interlayer and adhesive layer can initiate microcracks, because there is usually poor bonding between the debris and resin matrix. It is because of this weak interface that debris creates a void-like defect. It was observed that debonding occurs between the resin matrix and glass fibers even before any evidence of fracture can be seen on the composite surface. Debonding is a failure characteristic of all composites which can result from manufacturing processes (e.g., residual stresses, moisture absorption) or applied loads. The observed debonding is a characteristic of both high and low cycle roller cart tested panels. This debonding is observed as small microcracks at the fiber/matrix interface (Figure 3.21).

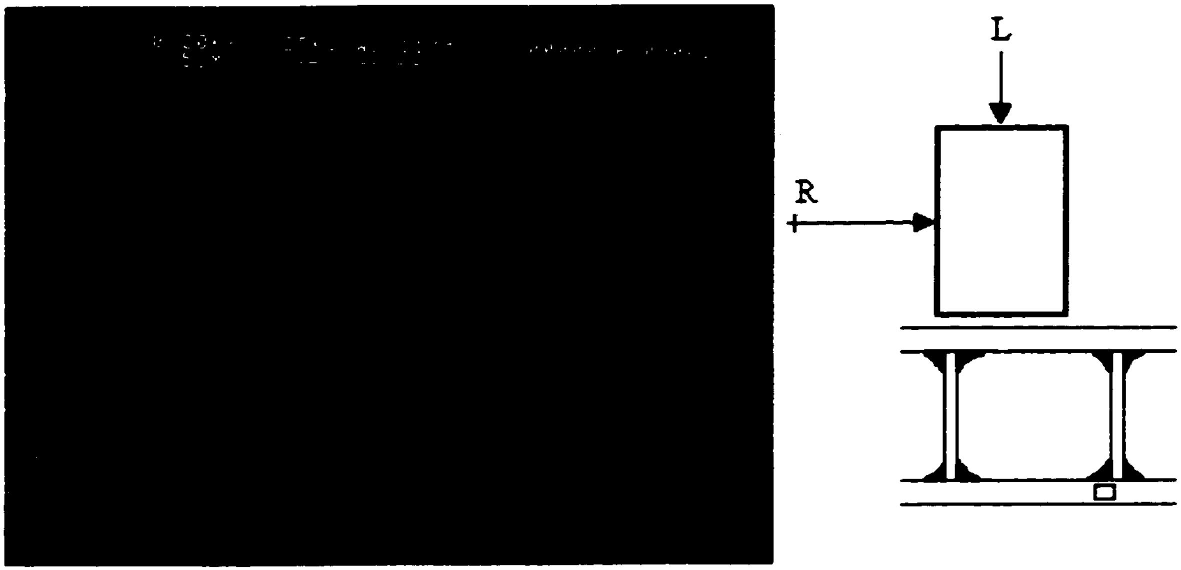


Figure 3.21. *Debonding occurring at the matrix/carbon fiber interface. The crack appearing from the top is from a debond fracture propagating from an adjacent fiber. (R) Radius of roller cart rotation, (L) Load applied by roller cart wheel.*

These microcracks grow and coalesce with adjacent fiber/matrix debonding locales and form larger cracks. It was apparent in these studies that there was good interfacial adhesion between the resin and carbon fiber.

It is rare that debonding is observed at the adhesive fillet and honeycomb cell wall interface. Debonding at this locale can be observed in panels that have been tested many cycles beyond initial observable fracture failure.

A fracture feature of matured microcracking is fracture face friction microcracking. As fatigue loading creates crack face friction, additional microcracks form along the crack face front breaking up the surrounding resin matrix (Figure 3.22). Fracture face friction microcracking is evident in the white-cloudy regions along honeycomb cell walls on the composite skin. This type of feature is an indication of extensive microcracking and is a precursor to through thickness skin cracking.

To get a sense of how much of the whitening could be attributed to stress whitening, some panel specimens were placed in an oven in an attempt to achieve material relaxation and possibly observe any disappearance of the whitening. Heating the specimens began at 20 degrees Celsius below the glass transition temperature and raised by 5 degrees per hour to 10 degrees above the glass transition temperature. Digital photos were taken before and after the procedure and there was a slight but not appreciable reduction in the whitening.

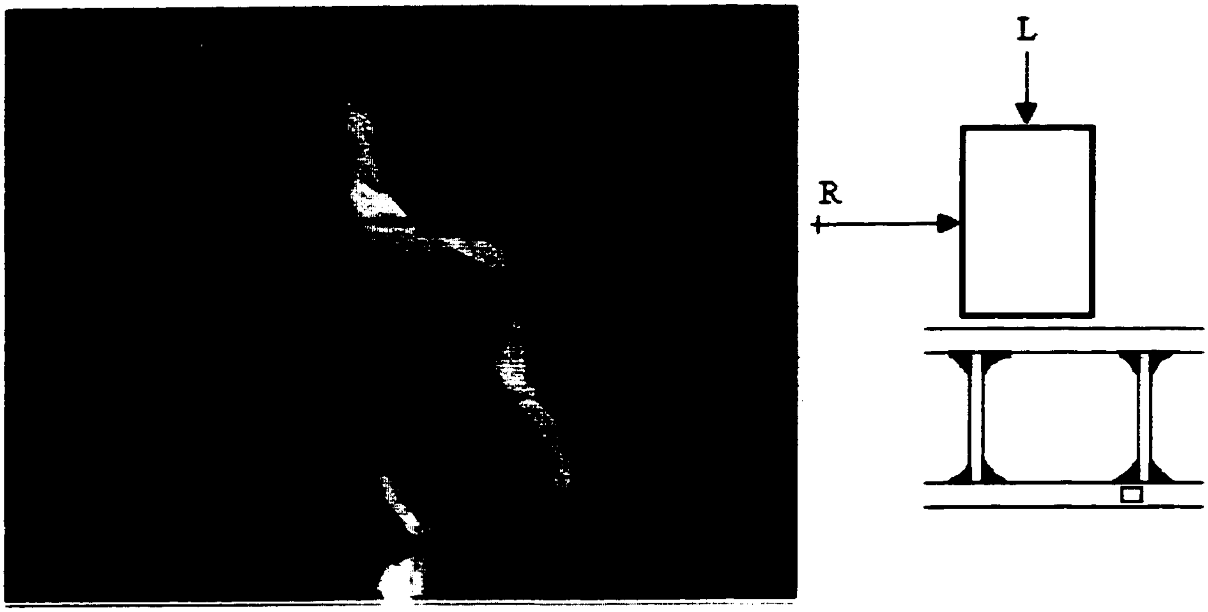


Figure 3.22. Fracture face friction along the fracture path. Note the smoothed mismatched fracture surfaces due to face friction. (R) Radius of roller cart rotation, (L) Load applied by roller cart wheel.

Therefore, it was determined that the white-cloudy effect was primarily due to light scattering because of microcracking (i.e., debonding and fracture face friction) and not stress whitening. The visibility of the streaks and striations, identified as voids (i.e., intralaminar and interlaminar runners) in the undamaged roller cart specimens, were also attributed to light scattering at these defects within the composite skin.

It should be clarified that the white aberrations appearing on the composite surface are caused either by voids, debonding, or fracture face friction and are indistinguishable when viewed on the composite surface (except to the trained eye). Thus, these features must be investigated via microscopy to identify the root cause.

General Failure Sequence

Several modes of initiation for microcracking have been demonstrated. An important observation is the sequence of crack propagation, which is helpful in understanding the fracture features observed on the composite skin during roller cart testing. The sequence of intraply failure generally occurs via the following process. First, a microcrack is initiated at a void (Figure 3.23) or a matrix/fiber debond (Figure 3.24).

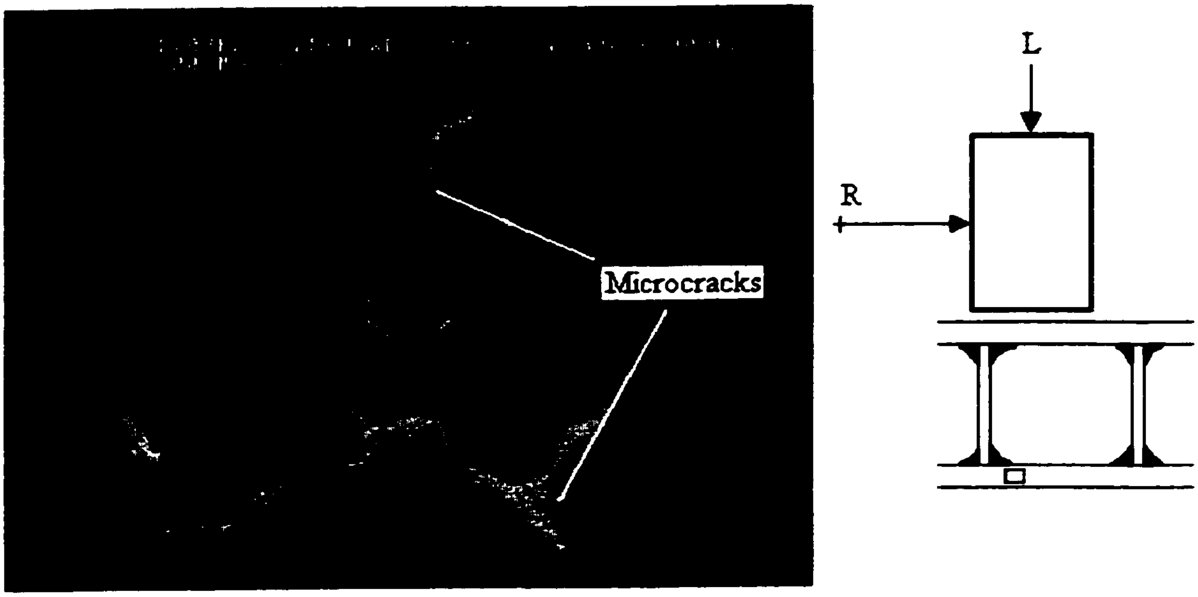


Figure 3.23. *Void initiated microcracking under fatigue load in roller cart testing. (R) Radius of roller cart rotation, (L) Load applied by roller cart wheel.*

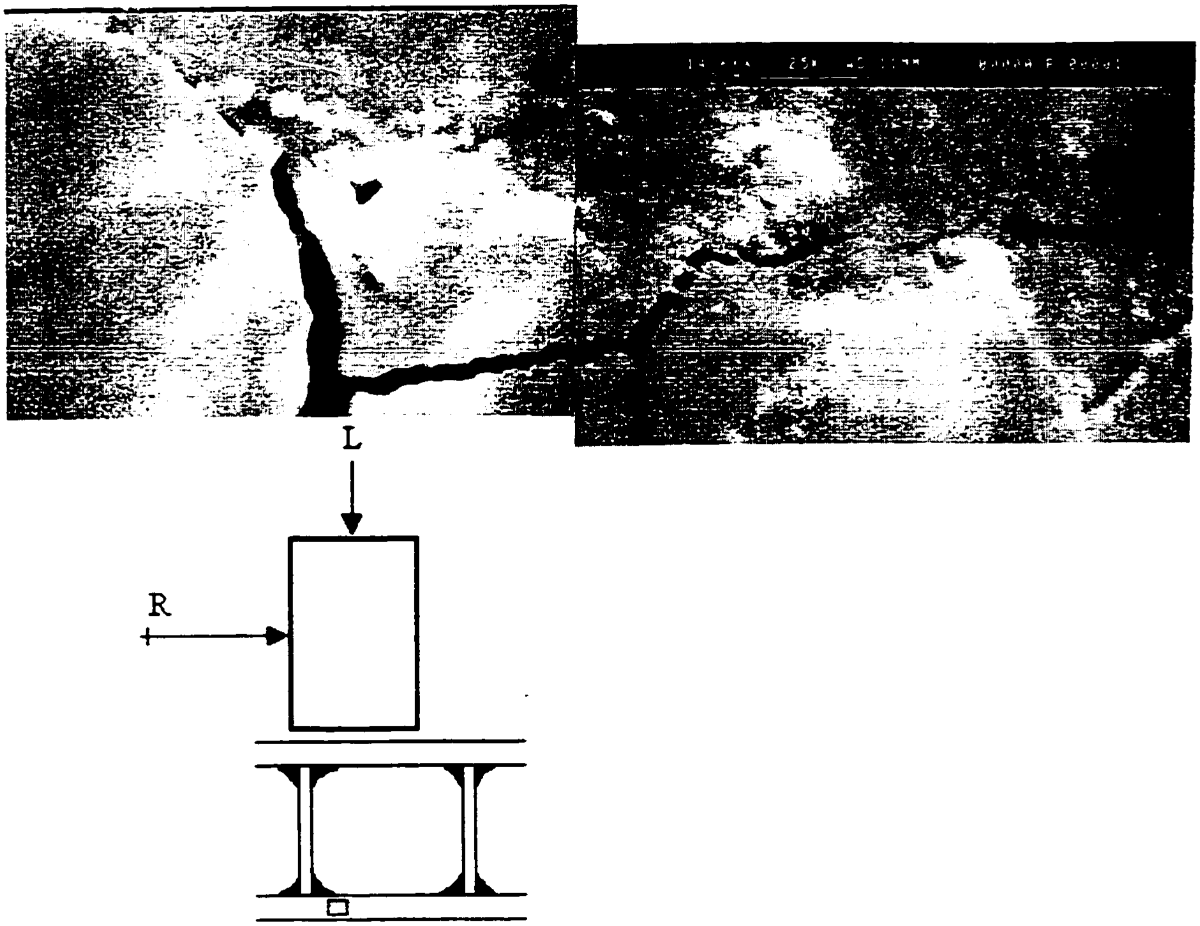


Figure 3.24. Debonding at the matrix/glass fiber interface. (R) Radius of roller cart rotation, (L) Load applied by roller cart wheel.

Second, the microcrack grows to a few microns toward a plane parallel to the applied load of the caster wheel (the damage is not yet visible). Third, the crack or debond grows to a few hundred microns and due to fracture face friction and crack bifurcation, the microcracking becomes visible as white clouding to the naked eye on the composite skin surface. This whitening has been attributed primarily to light scattering as a result of microcracking due to crack face friction and matrix/fiber debonding. Fourth, the mature crack then propagates and deflects within the ply (Figure 3.25).

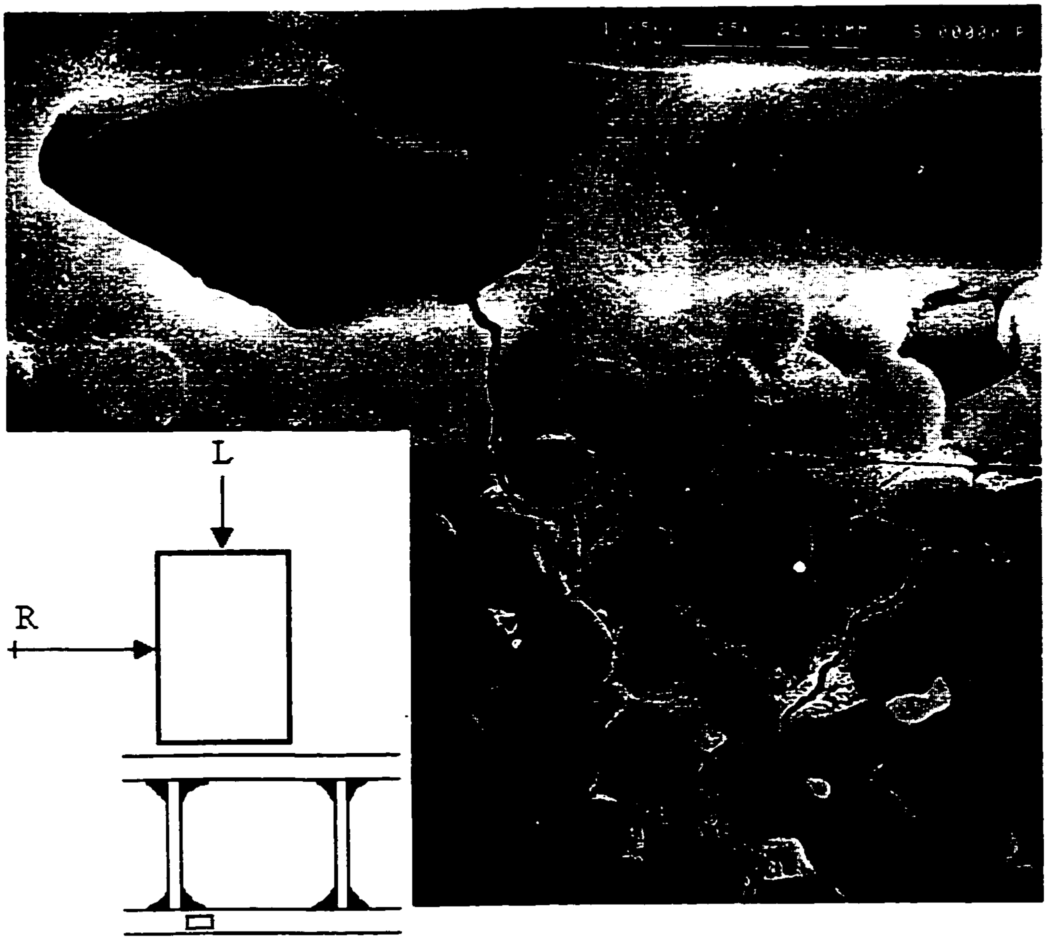


Figure 3.25. *Intraply fracture displaying crack deflections, and fiber/matrix debonding. The micrograph also reveals cracks propagating into the glass fibers indicating good interfacial adhesion. Since the microcracking is advanced it is not clear if the cracking initiated at the interlaminar void or matrix/fiber debond. (R) Radius of roller cart rotation, (L) Load applied by roller cart wheel.*

The microcracks initiating in the inner ply (i.e., nearest the core) of the skin propagate within the ply in line to the applied load and toward the interlayer. When the crack reaches the interlayer the crack will deflect at the interface (i.e., perpendicular to the applied load) creating a delamination (Figure 3.26).

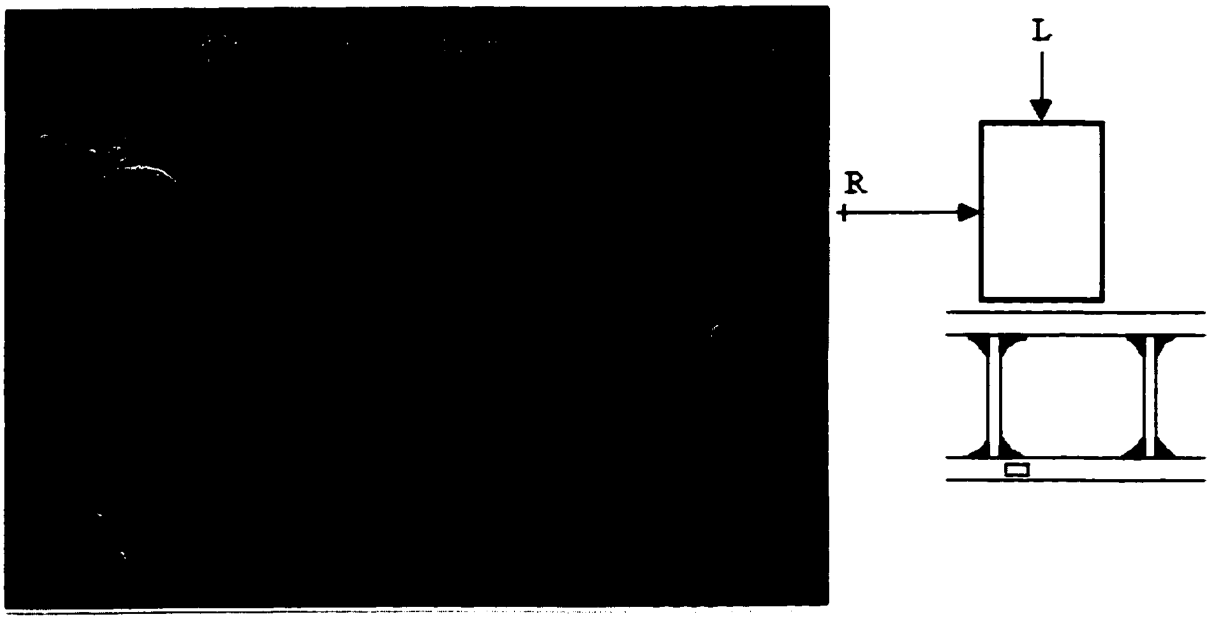


Figure 3.26. *Interlaminar crack deflection which gives rise to delamination of the facing plies. (R) Radius of roller cart rotation, (L) Load applied by roller cart wheel.*

The micro-cracks initiating in the intraply of the outer ply of the composite skin grow toward the outer surface before propagating to the interlayer. The mature crack will then crack deflect at the resin interlayer (i.e., perpendicular to the applied load) creating a delamination (Figure 3.27).

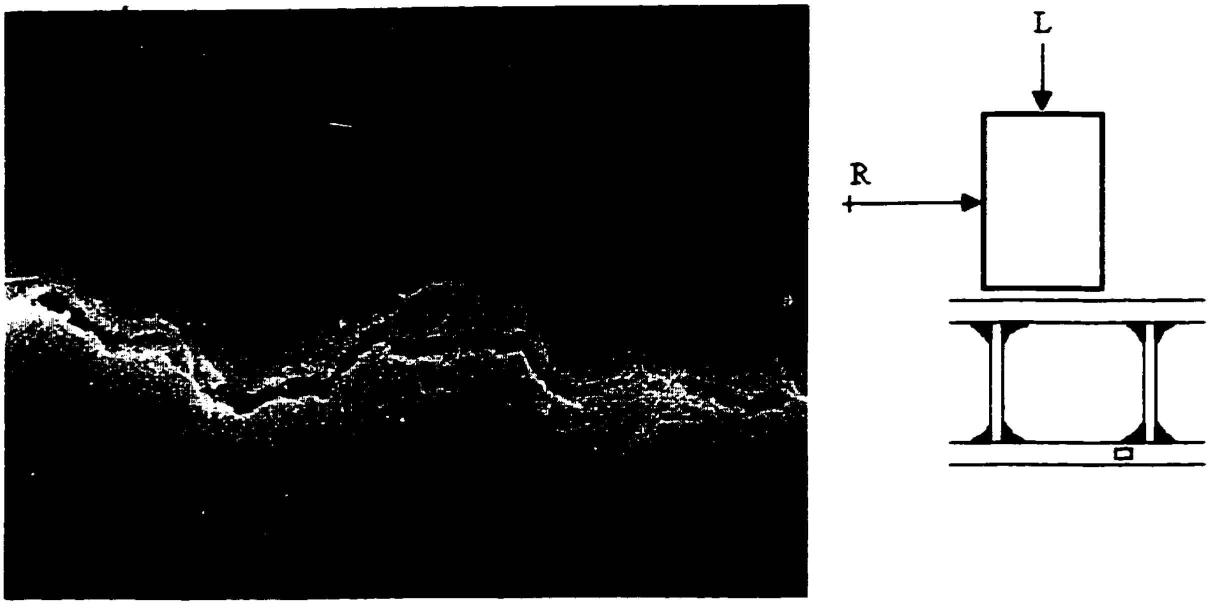


Figure 3.27. Delamination of the woven glass plies in the composite facings. (R) Radius of roller cart rotation, (L) Load applied by roller cart wheel.

When cracks from each of the plies meet at the interlayer then a through thickness crack is observed. Concerning the interlaminar runners and voids, they not only initiate microcracks and delaminations but also they promote large-scale delaminations when intraply microcracks meet the interlayer. Although the fiber/matrix interface was strong, the preferred crack propagation path was along this interface.

Results

This study suggests that resin formulation consistency and resin distribution are critical concerns in producing low void content composite skins for honeycomb structures. It also suggests that manufacturing well-consolidated (i.e., low void content) facings is essential to improving damage tolerance, fatigue life, and overall composite performance. Improving the fatigue life of these composites will reduce the customer's replacement cost and further increase customer satisfaction while meeting the new corporate paradigm.

This study further suggests that analysis of void volume versus roller cart fatigue cycles would best be studied using ultrasound or other suitable techniques (i.e., C-scan) to quantify the void volume per unit area. The results of this type of research could be used to predict the roller cart fatigue life of these panels. In addition, C-scanning techniques developed through this effort could be used for quality control purposes in monitoring the composite skin consolidation.

The implications of this research imply the importance of well-consolidated low void content skins in aluminum honeycomb core composites. The void content can be a result of raw materials quality, resin preparation, resin distribution or resin viscosity. Therefore, topics that were discussed throughout the internship in order to improve panel manufacturing and performance were quality control issues such as resin formulation consistency, quality of supplied raw materials, manufacturing process consistency, and developing a set of quality standards and procedures (e.g., ISO 9000). The intern suggested that in order to improve the manufacturing processes of these panels, a selected "team" of in-house professionals within the company would need to be assembled to resolve many of these complex issues. These in-house professionals would dedicate a portion of their time apart from their current responsibilities to address and solve these important issues. In *Organizing Genius*, by Warren G. Bennis, the importance of team formation in overcoming apparent insurmountable challenges is emphasized [77]. This type of team building is commonplace in today's lean workforce where companies cannot afford to hire entire teams of experts or even a few additional personnel. This type of team building has proven to be an extremely effective management technique proven by experiences at such companies as NASA, EXXON, Lockheed and The Dow Chemical Company.

Roller Cart Internship Project Summary

In achieving the internship project objective, knowledge was gained in understanding the fracture behavior of these epoxy/glass fiber honeycomb composites. In studying many high and low cycle roller cart panels the fracture behavior was characterized. OM and SEM were used to identify the structural characteristics and the causes of microcrack initiation. It was found that there are inherent structural characteristics such as intralaminar and interlaminar runners that are prevalent among low cycle and to a lesser degree in high cycle panels. In regard to fillet radius, it was found that there was not a distinguishable difference in roller cart performance in relation to the fillet geometry. The adhesive used to manufacture these panels forms consistent fillets between the composite skins and the honeycomb core.

Prior to the intern's arrival there was extensive work done in studying the void content via two-dimensional analysis of the composite cross sections. In these studies, it was observed that there was no relation between the number of cycles withstood before failure in the roller cart test and the void content. The observations made by this intern found that voids, especially elongated and continuous voids, should not be approximated as nearly symmetrical and concise. It is because of this observation that void volume per unit area versus roller cart fatigue life would be a much more practical comparison for predicting panel fatigue life.

INTERNSHIP EXPERIENCE SUMMARY

Through the professional internship experiences at The Dow Chemical Company and Hexcel Corporation, the objectives of the Doctor of Engineering internship have been met. The intern was afforded the opportunity to develop and exercise his leadership, engineering knowledge and creativity in addition to acquiring advanced technical training while focused on accomplishing the objectives of the internship sponsors. In addition, the intern observed and functioned in a non-academic setting to meet corporate objectives and apply the employer's problem solving approach. These professional internships revealed that the employer's approach to problem solving sometimes deviates from traditional engineering design and analysis, for example, when financial resources are strained, or time constraints call for abbreviated research and development plans. In such instances it is imperative to redefine the problem solving approach to consider only that work which is critically essential in making sound engineering decisions. These compromises, as experienced by the intern, proved to be effective in expediting the employer's demanding project schedules. This revealing experience occurred at both internship locations.

The internship experience with The Dow Chemical Company and Hexcel Corporation introduced this intern to the complex challenges encountered in new product development, from research to administrative concerns. The experience with Dow Chemical enriched this intern's technical and professional ability through continuous interaction and advanced training from experienced and highly trained scientists, engineers and managers. In working with Dr. White, Mr. Burton and Dr. Bertram this intern experienced efficient teamwork in meeting various project goals. Working with knowledgeable, motivated, and enthusiastic Dow personnel was a privilege and an unforgettable experience. This should not suggest that the work environment was calm because this was not always the case. When technical problems occurred or deadlines were rescheduled, the work environment became stressful, yet problem solving and achieving objectives continued, with continuing contribution by the intern. The entire professional internship experience including advanced technical training, applied academic knowledge, teamwork, and demonstrated leadership in a dynamic corporate environment was a true test and proof of the domain unique to this Doctor of Engineering internship experience.

The remainder of the Doctor of Engineering internship experience was performed at Hexcel Corporation. This internship provided a new set of challenges vital to the company. This experience was in support of a corporate directive to improve product performance to attain customer satisfaction. In researching and developing both existing and novel honeycomb composite technologies, the intern supported the corporate objectives. Upon the intern's arrival at Hexcel, he was briefed about the internship project objectives and took advantage of the training he received. He researched literature and company reports, and identified key personnel that could be utilized in support of the project objectives. The efficiency with which the projects were addressed is directly attributable to some of the training, team-building, and problem-solving skills previously acquired at Dow Chemical and Texas A&M. Achieving timely results in an abbreviated internship prompted the utilization of the this acquired skill set (see Appendix D, Papers and Patents). As the internship progressed further interactions with Dr. Lee and other employees continually gave insight into the company's problem solving capabilities and approach. In retrospect, the research and development initiative under the supervision by Dr. Lee were sound and similar in form to those experienced at Dow Chemical. However, Hexcel's corporate structure, in contrast to Dow Chemical, provided additional insights into communication and teamwork across business units, and corporate response to implementing change.

Finally, the internship experiences at The Dow Chemical Company and Hexcel Corporation served in partial fulfillment of the Doctor of Engineering requirements, and gave this intern an overview of two different corporate cultures, organizational structures, and approaches to problem solving. The internship assignments placed the intern in positions of responsibility where he could interact with professionals in meeting vital corporate objectives.

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APPENDIX A

RESUMES OF INTERNSHIP SUPERVISORS

Dexter White, Ph.D., P.E.

Polyolefins Research and Development

Education

B.S. in Chemical Engineering, The University of Texas at Austin, 1964
Ph.D. in Inorganic Chemistry, The University of Texas at Austin, 1968
12 hrs toward Master in Engineering, Texas A&M University, 1971-1976

Academic Experience

Postdoctoral Research, The University of Illinois at Urbana, 1968-1970

Industrial Experience

Dow Chemical Company, 1970 - Present
  Research Engineer, Polyglycol Research, 1970 - 1972
  Sr. Research Engineer, Toluene Diisocyanate Pilot Plant, 1972-1978
  Engineering Specialist, Ethylene Diamine Research, 1978-1980
  Group Leader, Ethyl Benzene/Styrene Research, 1980-1984
  Research Associate, Epoxy Resins TS&D, 1984-1992
  Manager, Dow-UT Support Group, 1992 - 1997
  Manager, Materials Research & Development Lab, 1997 – 1999
  Leader, Pilot Plants, Polyolefin R&D, 1999 - Present

Affiliations

Registered Professional Engineer #39560, State of Texas, 1975 - Present
Member, Society for the Advancement of Materials and Plastics Engineering

Publications (Internal)

95 Dow CRI technical reports - 1970 to Present
2 Technical Papers presented at TDI Conference - 1970 to Present

Publications (External)

6 Technical papers with A. H. Cowley, University of Texas at Austin, 1964-1968
2 Technical papers with R. S. Drago, University of Illinois at Urbana, 1968-1970
4 Technical papers presented at SAMPE National Conventions, 1984-1990
1 Short Course in Resin Transfer Molding, Atlantic Research University, 1987
14 U.S. and Foreign Patents on Dow Product and Process Technologies

Bruce L. Burton
3400 Ashmere Cove, Round Rock, Texas 78681-1002
Work phone: 512-483-0148 Home phone: 512-671-3011 blburton@io.com

1977 – 1999	**The Dow Chemical Company**	**Freeport, Texas**
1999 – Present	**Huntsman Petrochemical**	**Austin, Texas**

- I'm currently a research associate in the Surfactants, Performance Products, and EPS department of Huntsman working on amines for high solids and water-borne coatings. More than 22 years experience doing a broad scope of research and development. Very experienced with thermosetting polymer systems, such as epoxy, vinyl ester, cyanate, bismaleimide, urethane, and hybrid resin systems from fundamental research through scale-up and applications development
- Have considerable practical problem solving ability in a wide variety of areas, as well as highly technical understanding of polymer performance based on structure-property relationships. Understand performance trade-offs and how to tailor formulations for specific applications. All work is well documented (ca. 56 internal reports, 11 external papers) - excellent verbal and written communication skills. I have some German language ability.

Experience/Accomplishments:

- Synthesized and developed resins for cathodic electrodeposition (C.E.D.) coatings
- Led a project team developing high temperature RTM-able resins for aircraft engine applications.
- Toughened polymer development and basic research
- Environmental stability characterization and modeling
- Mechanical and thermal testing of neat resins and composites
- Have been an internal consultant in diverse areas e.g. toughening, high temperature performance, electrical potting compounds, static electricity
- Successful commercialization of Tactixâ695 epoxy resin, a tough, high Tg resin used for good hot-wet performance of carbon fiber composites.
- Resin Transfer Molding and Reaction Injection Molding (RTM & RIM)
- Particulate binders (tackifiers) for preform production

Education:

- M.S. in Chemistry (Analytical) - Texas A&M University, Aug. 1987
- B.S. in Chemistry - The University of Kansas, Jan. 1977

References:

Dr. Norm Johnston (757-864-4260) – NASA, Langley Research Center.
Dr. Mac Puckett (713-309-4117) – Lyondell Chemical, friend and former colleague.
Dr. Hung-Jue Sue (409-845-5024) – Assoc. Prof. of Mech. Eng., Texas A&M University.

Shaw Ming Lee

Hexcel Corporation, 11711 Dublin Blvd.,Dublin, CA 94568
Phone: (925)847-9500 x3246, Fax: (925)828-2277, e-mail: shaw.lee@hexcel.com

EDUCATION

Ph.D. in Mechanical Engineering, MIT, Cambridge, MA 1979.
M.S. in Engineering and Applied Science, Yale University, New Haven, CT, 1975.
B.S. in Mechanical Engineering, National Taiwan University, Taipei, Taiwan, 1972.

EXPERIENCE

1996-Present **Hexcel Corporation**, Dublin, CA
Director, Materials Science Group, R&T Dept.
In charge of a technical group to support Hexcel worldwide long range product development efforts by performing (a) research on product-related materials issues, (b) discovery of novel product concepts, and (c) testing and analytical supports. Responsibility areas cover all aerospace and industrial products: fibers, fabrics, prepreg, honeycomb core, sandwich panels and composite structures. Coordinate all research efforts with Marketing and Manufacturing across all Global Business Units (GBU's) within Hexcel. Direct collaborative technology development with outside firms or universities that offer potentially useful technologies.

1979-1996 **Ciba-Geigy Corporation, Composites Division**, Anaheim, CA
Held various R&D positions: Director of Materials Science (last), Manager of Materials Research, Senior Staff Scientist, Staff Scientist, Senior Research Engineer. Responsible for interdisciplinary research teams working on the scientific principles of developing composite products.

1978-1979 **Bell Telephone Laboratories,** North Andover, MA
Member of Technical Staff.
Performed R&D work on materials for telecommunication applications.

TEACHING EXPERIENCE

University of California, Los Angeles, CA (Part-time Teaching)
Taught a senior level Materials Science and Engineering Dept. course "Structure and Properties of Composite Materials" (Spring 1995). Lectured on "Advanced Materials and Structures: Composites" in UCLA Extension Short Course Program (Summer 1995). Covered the subject of Manufacturing of Composites in "Advanced Transportation Systems, Manufacturing Engineering" course of the Integrated Manufacturing Engineering Program at UCLA (Fall 1995).

APPENDIX B

INTERNSHIP SUPERVISORS' FINAL REPORTS

The Dow Chemical Company
Materials Research and Synthesis
Freeport, Texas 77541

Professor Hung-Jue Sue
Texas A&M University

Re: Review of John Miranda's Performance for the period, January 1 to June 1998

John Miranda worked in my Dow-UT Research Support group during his tenure at Dow. He had two projects, one concerned with studying how to toughen polymeric resins and a one concerned with making composite panels out of high temperature polymeric resins.

John's overall performance was superb. He is an extremely hard working individual and very concerned with doing a good job. He is a very capable hands-on type of individual with tremendous skills in the engineering and chemistry areas. I can recommend him to any future employer as a asset to their work force.

John's specific contributions to our program were two-fold. In the toughening project, he conducted lab preparations of the toughened resin using PPO as a toughening ingredient. He tried different loadings of the toughening ingredient by taking graphite preforms having different material loadings and making graphite composite panels using a novolac resin/anhydride hardener high temperature resin. John took these composite panels to Texas A&M and had them tested for improved resistance to impact (higher Compression after Impact, G1c and Short Beam Shear values).

In the high temperature resin project, he participated in molding graphite composite panels of polyimide resin. John prepared graphite preforms of various layup specifications, and setup the resin infusion mold on the Weyer press and molded under the desired conditions of temperature and pressure. He supervised the run, collected the data and helped his supervisor interpret it. John was instrumental in the solution of gasket problems that plagued this project. He also trained and documented Dow personnel on the entire molding process.

In summary, John was found to be a hard worker and a self-starter. It was possible to set goals for John and then let him conduct the research project as he saw fit. He was a model employee and will be welcome at Dow if he wants to return.

Regards

Dexter White
Area Manager
409-238-4578

HEXCEL Composites

April 9, 1999

Dr. Hung-Jue Sue
Texas A&M University
Department of Mechanical Engineering
College Station, TX 77843-3123

Dear Dr. Sue:

I am writing this letter to summarize my evaluation of John Miranda who was an intern under my direct supervision at Hexcel R&T from July 1998 to February 1999. His internship was as part of the requirements of the Doctor of Engineering Program at Texas A&M University.

John worked on two major research projects at Hexcel – roller cart and core/skin - as described in his Internship Objectives. One project had to do with a current product problem (roller cart) and the other was critical for next generation products (skin/core). John's accomplishments at Hexcel far exceeded my original expectation of simply gaining generic basic understandings. As it turned out, John was able to bring to these subjects unusual insights that have unanticipated but definitive product implications. I will discuss his work below with certain specific details withheld for proprietary reasons.

In the roller cart project, John was dealing with a problem involving many industrial practices (e.g., raw materials, manufacturing, and testing) that could contribute to the specific failing product performance. He had to sort out a lot of complex details to focus on the area most relevant to the problem. He then performed systematic microscopic analysis with fracture behavior consideration to identify many important failure mechanisms. What he found to be the most likely root cause was a unique type of microscopic manufacturing defect. Such a type of defect was very difficult to spot and never identified before (not obvious from 2-D view and only revealed by considering 3-D details). Its existence was entirely consistent with the material, processing and especially performance aspects of the products. John' s findings have allowed us to focus our current product improvement efforts on minimizing or eliminating the type of defect he identified.

On the subject of core/skin, John studied the microscopic mechanisms controlling core to skin bond strength. He had to first understand all the material, processing and testing factors related to the bond formation and fracture. He then proceeded with microscopic observations of the bond failure processes and fracture surfaces of many model systems. He was able to draw meaningful connections between the bond failure and the specific material approaches involved. As a result, several bond failure processes uniquely controlled by different approaches were identified. Some of these processes are highly favorable from the viewpoint of maximizing bond strength. From these observations, he deduced the possible material routes to take advantage of the desired failure mechanisms. Again, we are utilizing John's findings as an extremely useful direction for our current efforts to develop the next generation products.

John's excellent performance in his research work in my opinion was not coincidental. First of all, he worked very hard and was extremely persistent. During the course of his investigations, there were many occasions where he encountered obstacles or followed not so promising leads. He was always able to relentlessly push forward by learning from the setbacks and redirect himself accordingly. Second, he was highly objective. This was especially important for problems involving many aspects, and thus often conflicting views, of the industrial products he studied. He was able to clearly analyze and clarify the critical details before drawing any conclusion. Third, he had solid training in polymer science and mechanical behavior of materials. More importantly, he was able to effectively apply it to study the practical problems. The research approaches he took exemplify how to analyze the microscopic mechanisms controlling the macroscopic behavior of complicated material systems such as the advanced polymer composites.

It was my observation that John is a genuinely conscientious and self-motivated individual who needs little supervision to accomplish the tasks. He also has a pleasant personality and got along well with everyone here. For the relatively short period of time John spent with us, he made tremendous impact to our urgent projects far more than what we could expect. Based on my experience in the industry, people with his attitude and capabilities can usually excel in job no matter where they are. What he did at Hexcel also reflects the unique skills to bring basic understandings to practical problems. Such skills in my opinion are rare and highly valuable for the industry.

Best regards.

Sincerely,

Shaw M. Lee, Ph.D.
Director, Materials Science

APPENDIX C

FINAL INTERNSHIP OBJECTIVES

FINAL INTERNSHIP OBJECTIVES

Dow-United Technologies

INTRODUCTION

The final internship objectives described in this report are those accomplished by this Doctor of Engineering student during his tenure at The Dow Chemical Company's Dow-United Technologies (Dow-UT) division. The student successfully obtained industrial research results dealing with the toughening of brittle matrix carbon fiber composites by thermoplastic particle interlayering. Also, he visited Dow-UT headquarters in Wallingford, Connecticut to brief company managers about his project's progress, discuss expanding the scope of the project and discuss patent issues related to the project.

FINAL INTERNSHIP OBJECTIVES

The portion of the Doctor of Engineering internship spent at Dow-UT enabled this student to demonstrate and enhance both his professional abilities and technical knowledge. He successfully interacted with professional engineers, scientists and other professionals in a team effort to develop novel technologies such as the thermoplastic toughening of composites, the development of high temperature application resins and various other technologies creating new potential markets for Dow business.

The objectives focused on interacting with Dow-UT managers to meet their product needs regarding toughened composites. The intern was be responsible for managing the internship project's progress toward meeting business goals. Also, the intern was responsible for developing new methods to manufacture toughened carbon fiber preforms and for developing new techniques in the composite molding process. In addition, he evaluated the mechanical performance of toughened composites interlayered with various engineering thermoplastics. Finally, the intern researched the potential opportunities in patenting new toughening technologies related to this internship project.

In support of expanding new markets for Dow-UT business, this student was responsible for researching toughening technologies and informing the company about their patent stance regarding novel toughening applications. In addition, Dow-UT has expressed an interest in expanding the application of toughening project. Therefore, this intern will assist Dr. Hung-Jue Sue in developing a project plan and time-line to be used in estimating the necessary project resources needed to expand the scope of the project.

FINAL INTERNSHIP OBJECTIVES

Hexcel Corporation

INTRODUCTION

The final internship objectives described here are those to be accomplished by this Doctor of Engineering student during his tenure at the Hexcel Corporation. The principle objective of this internship will be to ascertain the relationship between the microstructure[1] of various honeycomb composite materials to mechanical performance.

In studying the micro-structure of Hexcel's honeycomb core composites it is expected that observed material characteristics (i.e., part consolidation, interfacial adhesion, fracture phenomenon, cycles to failure) will be identified, in some instances, as a consequence of material selection, material processing, composite manufacturing methods, mechanical testing, or any combination of these. Another important observation that will be made is the predominant cause (e.g., failures caused by voids, inadequate interfacial bonding, etc.) and systematic progression of the failures associated with their honeycomb core composites. The studies performed during this internship period will yield a fundamental understanding of honeycomb core composites that will lead toward producing quality, high performance[2], honeycomb composites.

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[1] The evaluation of the microstructure will be performed via bright-field optical microscopy and scanning electron microscopy.

[2] The term "high performance", refers to higher cycles to failure in roller cart testing of aluminum honeycomb core composites, and higher peel strengths in nylon paper honeycomb core composites.

FINAL INTERNSHIP OBJECTIVES

The two projects in which this intern has been asked to participate concerns two different types of honeycomb core composites. The first project described deals with improving the roller cart fatigue life of aluminum core honeycomb composites. The second project described deals with identifying the material structure characteristics and fracture behavior, which will contribute to the understanding in improving the climbing drum peel strength of nylon paper (i.e., trade named Nomex) honeycomb core composites.[3]

Roller Cart Fatigue Project

Background

The Roller Cart Fatigue Project focuses on extending the roller cart fatigue life (greater than 120,000 cycles) of Hexcel's aluminum honeycomb core composites.[4] These materials are "sandwich" type composites consisting of two outer skins, each skin consisting of two plies of resin pre-impregnated woven glass fiber reinforcement (i.e., prepreg). These skins are then adhered to each side of a honeycomb sheet (this becomes the "core" of the composite) creating the composite structure (Figure 1).[5]

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[3] Climbing drum peel test, ASTM D1781-15.06.

[4] The Roller Cart Test in not an ASTM or SACMA standard. This test has recently been standardized "in-house" by Hexcel.

[5] Figure taken from: Bitzer, Tom. Honeycomb Technology. 1st ed., Chapman & Hall, 1997.

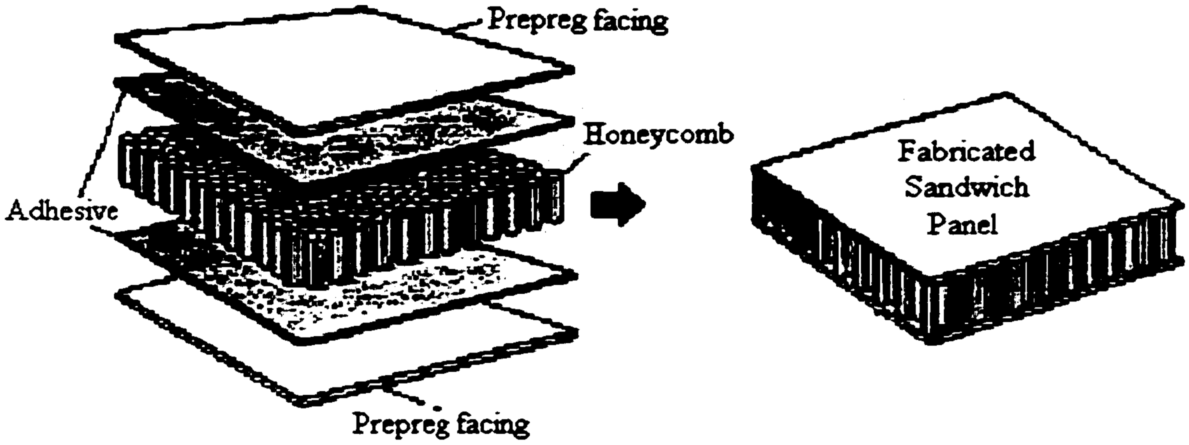

Figure 1. *Honeycomb core composite structure.*[6]

The assembled composite is then place flat in a heated press to cure the resin and adhesive. This manufacturing method promotes unique characteristics which effect the mechanical performance of the final composite structure. For example, there may be significant differences in the adhesive fillet formation between the prepreg and honeycomb core cell wall located at the top versus the bottom surfaces during press cure. Also, there could be differences in consolidation of the prepreg skins at the top versus the bottom surfaces. This type of investigation will be a part of understanding which characteristics lead to improved mechanical properties and fatigue life.

The roller cart fatigue test is a standardized test in which a bolted, edge supported honeycomb composite panel (21"×39") is subjected to a weighted, and rotating three caster wheel structure (see Figure 2). The aluminum honeycomb core panel supported

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[6] Figure taken from: Bitzer, Tom. Honeycomb Technology. 1st ed., Chapman & Hall, 1997.

under this rotating load, experiences both global scale deflections and severe localized deflections with each pass of a caster wheel.

Figure 2. *Roller Cart Test Apparatus.*

Throughout the past year Hexcel has observed test panels with the same, fiber reinforcement, prepreg resin chemistry, and prepreg to core adhesive perform inconsistently; exhibiting low, passing (i.e., >120Kcycles) and high cycles to failure. It should be noted that the roller cart test is considered to be a core failure test (i.e., the core generally failing in shear) but the roller cart tests, more often than in prior history, have been stopped due to inter-laminar delaminations and intra-laminar debonding, and not because of core failures.[7] It was believed that the test result variations were due to a variety of causes such as roller cart test setup, the relocation of a production line, and resin chemistry, just to name a few. At the beginning of the internship period, I was asked to identify the reasons these composites continued to exhibit sporadic roller cart test results. I proceeded to review roller cart test data from the previous year, and it was evident that the substandard roller cart fatigue life of the tested panels was steadily improving above the required number of cycles (i.e., greater than 120K cycles). Until very recently, low cycle fatigue results were experienced in "requalification" panels that were essential in assuring customers of a quality product. I decided to begin the studies by performing optical microscopy on the failed requalification panels and those panels successfully tested just week's prior. These panels used same resin chemistry, fiber reinforcement, fiber volume, adhesives and honeycomb core. In conjunction with evaluating material characteristics (via OM), an investigation into the possible correlation of raw materials used in resin formulations, to the sudden substandard roller cart performance of the requalification panel was undertaken. For instance, batch and lot numbers, and dates of manufacture from suppliers of the raw materials are currently being compiled for various panels of interest because not all panels are manufactured from the same prepared resin mix. In addition, to these panels I have began to study additional high (>200K cycles) and low (<30K cycles) cycle fatigue panels in order to better distinguish microstructure characteristics that can lead to poor panel performance. Thus far, I have been able to identify various characteristics via optical microscopy not

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[7] Inter-laminar delaminations and intra-laminar debonding constitute a roller cart test failure.

identified before dealing with issues such as part consolidation, interfacial adhesion, and crack initiation. These observations will give insight toward the possible solutions (e.g., related either materials processing, manufacturing or mechanical testing) in manufacturing a quality, fatigue resistant, honeycomb composite product.

To date this project has centered on 1.) studying and identifying pre-existing defects and fracture behavior of aluminum honeycomb core composites via optical microscopy (OM) and scanning electron microscopy (SEM) 2.) understanding and evaluating the roller cart test setup and testing procedure 3.) gathering raw material data related to the manufacture of selected honeycomb composite panels to evaluate any correlation to mechanical performance 4.) interviewing key personnel in order to understand which critical parameters (i.e., related to resin rheology, composite manufacture and prepreg processing) can be controlled in the manufacturing process to produce a consistent composite product. At this time many observations have been made via OM and SEM.

Final Objectives for Roller Cart Project

The principle objective of this project will be to ascertain the relationship between the microstructure of aluminum honeycomb core composites and the roller cart fatigue life by studying the characteristics of the the composites structure and fracture behavior is expected that various causes of the observed phenomena can be identified and addressed.

Core-Skin Project

Background

The Self-Adhesion Project focuses on using a proprietary method to adhere prepreg skins to nylon paper honeycomb core. The project initiative is customer driven because the customer does not want to apply a separate adhesive film as an additional step in their honeycomb composite assembly process (see Figure 1, note the separate adhesive film layer). Therefore, Hexcel is studying the possibility of offering customers a new prepreg product. I have been asked to study this honeycomb composite structure and its fracture behavior in order identify issues which may be related to improving the peel strength of these novel composites. This material development endeavor will include but will not be limited to studying fillet geometry between the core and skin, interfacial adhesion, fracture behavior and consolidation issues. The assembly of these composites is similar to that described in the background section of the roller cart fatigue project. The major difference in the manufacturing of these composites is that they are autoclaved (i.e., cured in a pressure cylinder at a specified temperature), whereas, the aluminum core described previously are heat pressed. The nature of the autoclave process creates distinct differences in the composite microstructure (e.g., fillet formation, consolidation) due to one skin being against the tool (e.g., contoured surface, or flat plate) and the other against a vacuum bag.

Final Objectives for the Self Adhesion Project

The principle objective of this project will be to ascertain the relationship between microstructure of nylon paper honeycomb core composites and the climbing drum peel strength. It is expected that various causes of the observed phenomena can be identified and addressed.

CLOSING

The past few weeks I have been on a steep learning curve. I have read internal study documents, company project progress reports, observed as many panel specimens as possible, and read a fellow colleagues book on honeycomb composites (Tom Bitzer). I have even surprised myself on how quickly I have "come online." Dr. Sue, I am always reminded of you stressing the fundamentals and because of that it has been easier to understand Hexcel's composite materials. Also, I had not realized just how much technical (e.g., polymer and composite processing, polymer flow and fracture), and working knowledge I had accumulated [at Dow Chemical]. Dr. Sue you also told me I would have the opportunity to observe, study, think and be creative; this has come true.

APPENDIX D

PAPERS AND PATENTS

PAPERS AND PATENTS

Publications

Miranda, John A. and Hung-Jue Sue. "Compression After Impact of Thermoplastic Interleave Toughened Brittle Epoxy/Woven Carbon Fiber Composites." *Journal of Composite Science and Technology.*

Derkowski, Brian, John A. Miranda, J.A. Avila, Shaw Ming Lee, Herbert M. Hsiao, and Hung-Jue Sue. "Morphology and Compression After Impact Strength Relationship in Rubber-Toughened Composites," *Journal of Composite Materials.*

Li, Yanmei, John A. Miranda, John Whitcomb, Hung-Jue Sue and Walter L. Bradley. "Study of Moisture Diffusion Behavior in Hybrid IM7/BMI Composites," *Proceedings of the Society of Plastics Engineers Annual Meeting 1999,* New York, NY, 45:3423-3427.

Wei, Guangxue, John A. Miranda and Hung-Jue Sue. "Morphology and Fracture Mechanisms in Thermoplastic-Modified BMI Resin," *Proceedings of the Society of Plastics Engineers Annual Meeting 1998*, Atlanta, GA, 44:1458-1460.

Patents

Patent Disclosure to improve composite toughness via novel manufacturing technique. (The Dow Chemical Company)

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