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2. ULUSLARARASI KATILIMLI POLİMERİK KOMPOZİTLER SEMPOZYUM-SERGİ VE PROJE PAZARI

2nd POLYMERIC COMPOSITES SYMPOSIUM-EXHIBITION AND BROKERAGE EVENT (INTERNATIONAL PARTICIPATION)

symposium exhibition and brokerage event

(international participation)

POLYMERIC composites

26-28 Kasım / *November* 2010

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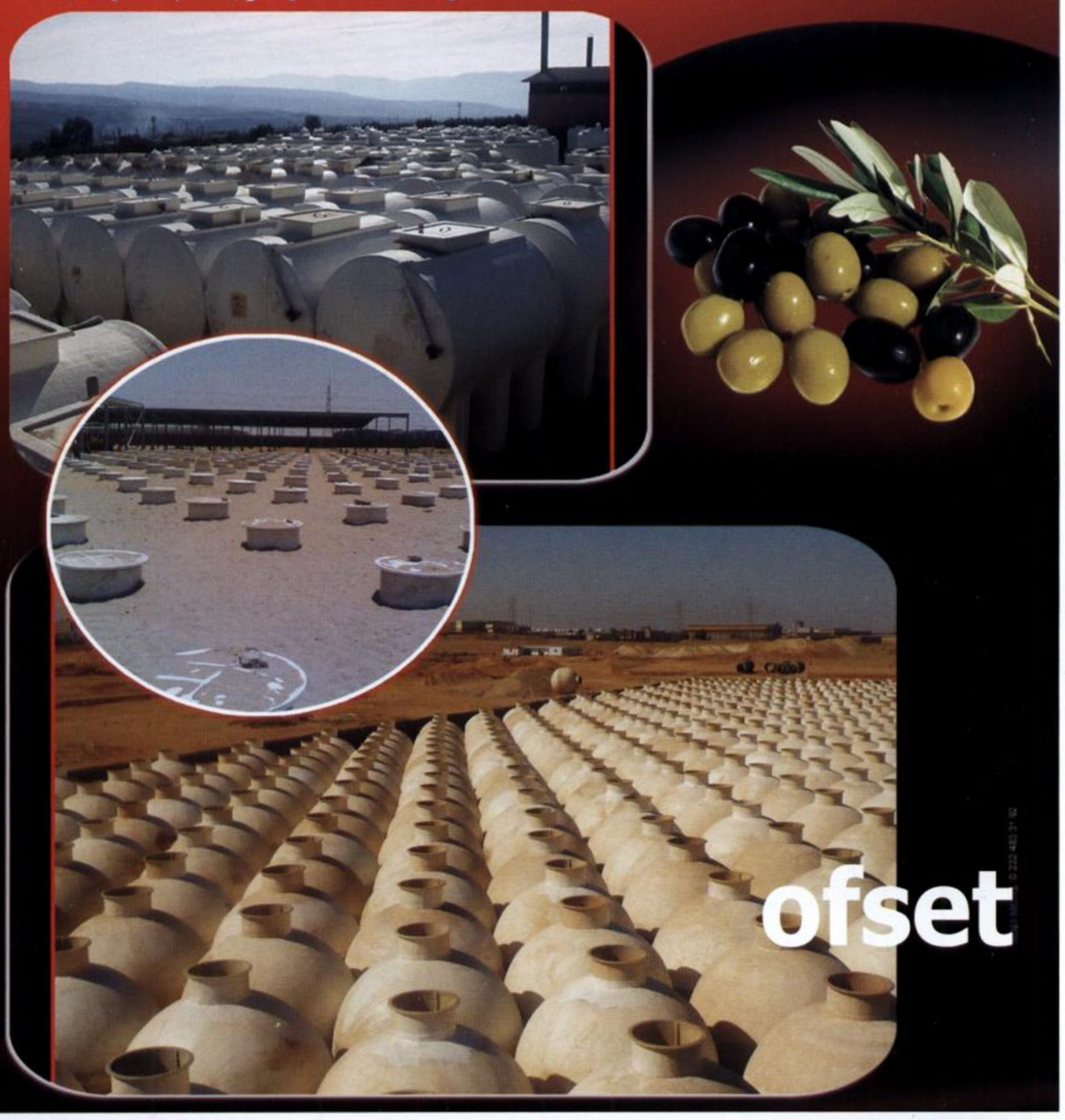
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''We are grateful to the public organizations and following companies for their contribution to the ''2nd Polymeric Composites Symposium-Exhibition and Brokerage Event (International Participation)'' İzmir, November, 2010 organized by UCTEA Chamber of Chemical Engineers.''

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ÖNSÖZ

Daha önceki önsözlerimizde de belirttiğimiz gibi bilimin ve buna bağlı olarak teknolojinin gelişmesinin, ülkelerin, toplumun ve yaşamın daha iyiye gitmesinde etkisi büyüktür. 21. yy'da öne çıkan yeni alanlardan biri, malzemeye istenilen özellikleri kazandıran polimerler ve oluşturdukları kompozit malzemelerdir. Isıl ve elektriksel iletkenlikler metallere benzer biçim de geliştirildiği gibi yalıtkanlık da istenilen düzeyde sağlanabilmekte, gereğinde daha hafif, fiziksel ve kimyasal etkilere, korozyona, darbelere dayanıklı esnek ya da sert yapıda olabilmekte ve bu nedenle yaşamın gereksinim duyduğu sağlık, tarım, uzay ve havacılık, denizcilik, ulaşım, inşaat, otomotiv, enerji, analizler gibi hemen her alanda uygulanabilmektedir.

Üretimde ve kullanımda standartlaşma, çevresel etkiler gibi, sorunların daha iyiye gitmesi, daha yeni bilgilerin paylaşılması, ortak çözüm üretilmesi, bu tür bilimsel ve teknik toplantıların yapılmasıyla bir ölçüde sağlanabilmektedir.

Bu nedenlerle TMMOB Kimya Mühendisleri Odası tüm kurum ve kuruluşlara eşit yakınlıkta ve hiçbir ticari amaç gütmeden, ilgili olduğu alanlarda etkinlikler düzenleyerek, bilimsel ve teknolojik birikimi olan üniversite ve araştırma kurumları ile üretici, sanayici ve uygulayıcının deneyimlerini aynı ortamda buluşturup, farklı bakış açılarının yeni ufuklar açmasını, sorunlara çözüm üretilmesini dolayısıyla ülke ve toplum yararını sağlamaya katkılı olmayı amaçlamaktadır.

Bu amaç doğrultusunda gerek araştırma kurumlarında gerekse sanayide yer alan üyeleriyle dayanışma içinde onların önerilerini alarak, etkinlikler yapmaktadır. Dört yıl önce polimerik kompozitler alanının önemini vurgulayan ve böyle bir etkinliğin yapılması gereğini öneren ve başlatılmasında ve sürdürülmesinde katkı sağlayan üyemiz Sn. Hasan Ömer'e bir kez daha teşekkür ederiz. Aynı biçimde uluslar arası katılımlı olarak gerçekleşen 2. Etkinlikte de Sayın İsmail Darcan, Sayın İsmet Çakar'a ve Sayın Hasan Ömer'e katkıları için teşekkür ederiz. Katılımcıların anketlerde yer alan önerilerine dayalı olarak etkinliğin iki yılda bir yapılması isteğiyle bu yıl da (26-28 Kasım 2010) gerçekleştirilecek etkinliğin uluslararası katılımlı biçimde yapılması konuya evrensel bir bakış açısı kazandırması ve bu yıl ilk defa düzenlenecek olan proje pazarı etkinliği nedeniyle ayrı bir önem taşımaktadır.

Etkinliğin yapılması için büyük bir özveriyle katkıda bulunan sponsor, sergi alanında yer alan ve reklam vererek katkıda bulunan kuruluşlara, bilimin ve teknolojinin, sanayinin ilerlemesini amaç edinen TÜBİTAK, İZMİR BÜYÜKŞEHİR BELEDİYESİ, EGE BÖLGE SANAYİ ODASI ve İZMİR TİCARET ODASI gibi devlet kuruluşlarına katkıları için şükranlarımızı sunarız.

Etkinliğin önemli ve vazgeçilmez niteliği olan, bilimsel ve teknolojik katkıyı sunan yurtdışı ve ülkemizdeki üniversitelerden ve araştırma kurumlarından, sanayiden katılan uzman ve araştırmacılara da özellikle teşekkür ederiz.

Sergi alanında ise ticari amaç taşımadan, sempozyum konularının görsel anlamda vurgulanmasını sağlayan ve etkinliğe destek verme amacındaki duyarlı kuruluşlar yer almakta ve bu yolla yeni ürünlerini, uygulamalarını tanıtma ve ilgililerle buluşma olanağı bulmaktadırlar. Sergi ziyaretine bilgi ve görgü arttırmayı amaçlayan ve ilgilenen herkes davetlidir.

"2. Uluslararası Katılımlı Polimerik Kompozitler Sempozyum-Sergi ve Brokekerage Event" etkinliğinin gerçekleşmesinde; değerli katkılar veren tüm kurum ve kuruluşlara, çağrılı konuşma yapan, sözlü ya da poster sunumları ile kongrenin bilimsel ve teknolojik yönünü nitelikli kılan değerli uzman ve katılımcılara, desteklerini esirgemeyen Düzenleme Kurulu ve Bilimsel Kurul üyelerine, Kimya Mühendisleri Odası ve Ege Bölge Şubesi Yönetim Kurulu üyelerine, yoğun emek ve çabaları ile etkinliği hazırlayan sekreteryaya, oda personeline ve katkıda bulunan üyelerimize teşekkürlerimizi sunarız.

Düzenleme ve Yürütme Kurulu

INTRODUCTION

As we mentioned before, the developments of the science and consequently the technology have a big effect on the improvements of the countries, the society and the life itself. One of the in front new areas in 21st century is the polymers which give the required specifications to the material and the composite materials formed by these polymers. The thermal and electrical conductivity of the materials can be improved as in metals and the required insulation can also be provided. In case of a need, the lighter and resilient materials to corrosion and to chemical and physical effects can be produced. The structure of these materials can be elastic or solid; there fore they are applicable almost in all areas like medical, agricultural, space and aeronautics, nautical, transportation, construction, automotive, energy and analytical technologies.

The achievements of the scientific and technical meetings like this one are providing the standardizations in the production and consumption, the environmental improvements, the new information sharing and the mutual solutions up to certain levels.

Because of all these above mentioned reasons, Union of Chambers of Turkish Engineers and Architects (UCTEA) Chamber of Chemical Engineers (CCE) is aiming to contribute towards the benefit of the country and the community by organizing the related activities, standing with all the organizations on the same level without any commercial concern. In these activities, the universities and the research institutions with their accumulation of scientific and technological knowledge and the producers, the industrialists and the practitioners with their experiences are coming together at the same place and creating new horizons and solutions with their different views.

In line with the object, the Chamber of Chemical Engineers' organizations are built and achieved with the cooperation and the various proposals of the members from the research institutes and the industrial organizations. At this point, we would like to thank once again to our member Mr. Hasan Ömer who emphasized the importance and sustainability of the polymeric composites sector and proposed an activity like this one and contributed for the start up four years ago. And we tkank to Mr. İsmail Darcan, İsmet Çakar and Mr. Hasan Ömer for their supports for the 2nd symposium (Polymeric Composite Symposium-exhibition and brokerage event with international participant). Base on the proposals indicated in the participants' questioners for an activity on every two years periodically, the international characteristic of this year's activity from November 26th to 28th, 2010 has a special importance because of this universal angle of vision and also breaking event which will be held this year for the first time.

We are grateful to firms who took a place in the exhibition and put an advertisement and to the public organizations like THE SCIENTIFIC AND TECHNOLOGICAL RESEARCH COUNCIL OF TURKEY (TÜBİTAK), İZMİR GRAND MUNICIPALITY, AEGEAN REGION CHAMBER OF INDUSTRY (EBSO) who aims for the advancement of the science, technology and industry, for their valuable contribution.

We would like to thank especially to the experts and researchers coming from universities, research institutes and industries of our country and abroad, for their scientific and technological contributions.

The polymeric composite applications and the subjects of regulations will be debated and the lectures will be given by the experts during the workshops and there will be practice of applications for some of the subjects. The participants will have a certificate.

In order to support this activity; the organizations are taking place in the exhibition which gives a visual emphasis to the subjects of symposium and by doing so, they have the opportunity to present their new products and applications as well as to meet with the interested parties. All the interested parties with educational and experimental purposes are invited to visit the exhibition.

We would like to thank to all of the organizations and institutes for their valuable contributions, to the invited speakers, to the estimable experts and participants, to the members of organizing and scientific committees for their support, to the members of the board of directors of Chamber of Chemical Engineers and Aegean Branch, to the secretariat who prepared this activity with their hard work and a lot of effort, to the personnel of the chamber and to all of our members who have been contributed for the achievement of "Polymeric Composites Symposium-Exhibition and Brokerage Event (International Participation)" activity.

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ÇAĞRILI KONUŞMACILAR/ *INVITED LECTURERS*

Ana Konuşmacılar/ *Keynote Speakers*

Amar Kumar Mohanty, Prof. Dr.	Kanada-Guelph Üniversitesi / *Canada-University of Guelph*
M. Cengiz Altan, Prof. Dr.	Amerika-Oklahoma Üniversitesi / USA-The University of Oklahoma
S. A. R. Hashmi	Hindistan-Gelişmiş Malzemeler ve Prosesler Araştırma Enstitüsü / *India- Advanced Materials & Processes Research Institute*
Yuri Korshak, Prof. Dr.	Rusya Mendelev Üniversitesi / *Russia- Mendeleyev University for Chemical Technology of Russia*

Davetli Konuşmacılar / *Invited Speakers*

Alexander Bismarck, Prof. Dr.	*Imperial College London*
Andreas Leuteritz, Dr.	Leibniz Institute of Polymer Research Dresden
A.M.Guliyev, Prof. Dr.	Institute of Polymer Materials of Azerbaijan National Academy of Sciences

Aref CEVAHİR, Dr.	Cam Elyaf Sanayii A.Ş. / *Glass Fiber Industry Inc.*
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Fulya Aktaş	AKSA Akrilik Kimya San. ve Tic. A.Ş.
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Mustafa Cüneyt Gezen	TMMOB Kimya Mühendisleri Odası / *UCTEA Chamber of Chemical Engineers*
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BİLDİRİLER

ANA KONUŞMACILAR /

KEYNOTE SPEAKERS

RECENT ADVANCES IN FABRICATION OF HIGH-PERFORMANCE COMPOSITES:

VACUUM ASSISTED RESIN TRANSFER MOLDING OF MICRO AND NANOCOMPOSITE LAMINATES

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Abstract

The first part of this paper investigates the effect of tooling and process parameters such as the length of distribution media used in vacuum assisted resin transfer molding (VARTM) of composite laminates. To achieve this goal, carbon fiber reinforced, epoxy laminates are fabricated by using various lengths of distribution media and their mechanical properties are characterized. It is shown that for relatively thinner laminates, extending the distribution media degrades the flexural properties by as much as 14%, possibly due to air pockets entrapped during through-the-thickness impregnation of the fibrous fabric.

In the second part, improvements due to small amounts of multi-walled carbon nanotubes (MWNTs) dispersed in the composite laminates are presented. In addition, effects of different nanotube functionalization and morphology are characterized via scanning electron microscopy and optical microscopy. To achieve adequate nanotube dispersion in the epoxy resin, both tip sonication and mechanical mixing have been used. The effect of sonication time on the dispersion of nanotubes is reported by monitoring the temporal changes in the nanotube cluster size. Even at volume fractions less than 1%, almost 10% improvements in flexural properties is observed. Extensive void formations are reported for laminates containing MWNTs, possibly preventing greater improvements in mechanical properties.

Introduction

Developing low-cost fabricating methods for high-performance, multi-functional composites has been one of the primary issues facing composites industry for several decades. During the last decade, various research initiatives and developments in nanomaterials have led to significant advances in fabrication methods and the materials used in high-performance polymeric composites [1,2]. Within this framework, various new processing concepts are used to improve the performance of micro and nanocomposite laminates [3,4]. A commonly-used fabrication method for medium to large, high-performance composite products is vacuum assisted resin transfer molding (VARTM). VARTM is an open-mold process, where the reinforcing fabric is placed in a vacuum bag and impregnated by a low-viscosity thermosetting resin. The reinforcement is often made of continuous carbon or glass fibers that are woven to provide bidirectional reinforcement. Unlike resin transfer molding and compression molding processes that use a metallic, closed-mold, the tooling and equipment cost for VARTM is much lower. Using a sealed vacuum bag and impregnating the fabric using vacuum pressure lead to slow impregnation and, for large parts, the vacuum level may not be sufficient to complete the impregnation. To alleviate these challenges, various versions of the process have been proposed, and in most cases, the use of a distribution media is suggested. The distribution media is a highly porous plastic mesh that is placed on top of the woven fibers. It acts as a lower resistance path for the resin and facilitates quick resin distribution further downstream.

Another way to improve the properties of VARTM composite parts is to introduce nanomaterials into the resin. Among the commercially available nanomaterials, various types of nanotubes or nanoclays have been most popular. Due to their lower density and high mechanical and thermal properties, carbon nantotubes are more attractive for high-performance applications where the primary reinforcement is supplied by woven graphite fibers. Even a small weight percentage of carbon nanotubes mixed with the resin may potentially improve the overall properties of the laminate [5-7]. Mixing and properly dispersing nanotubes with various types of thermosetting resins has been challenging. Mechanical mixing or sonication have been used as the primary means of introducing nanotubes, where the nanotube clusters are expected to break down and disperse throughout the resin. The improvements in the laminate properties are likely to be correlated with the degree of dispersion achieved. Thus, a detailed assessment of dispersion state and the effectiveness of sonication need to be investigated for a particular pair of nanomaterial and resin. Another important aspect of improving the properties of nanocomposite laminates is to achieve favorable interfacial bonding between the nanomaterials and the resin. To achieve adequate dispersion and bonding, several types of functionalization have been proposed [8]. It is well-known that the surface modification of the nanotubes and their functionalization influence the uniformity of dispersion, entanglement of nanotubes, and interfacial bonding strength.

In this article, we address the three primary issues mentioned above, i.e., (i) the effect of the use of distribution media during impregnation in VARTM processes; (ii) the effect of sonication time on the dispersion of different types of functionalized multi-walled carbon nanotubes and; (iii) the effect of different nanotubes on the mechanical properties of micro and nanocomposite laminates fabricated by VARTM method.

Experimental Studies

Effects of distribution media in vacuum assisted resin transfer molding (VARTM) was studied by varying its length. Laminates with dimensions 88.9mm x 152.4mm x 1.2mm were fabricated with minimum possible distribution media as well as distribution media extending to ¼, ½ and ¾ of the laminates' length. A total of 8 laminates (two for each case) were fabricated to characterize the effects of distribution media. The schematic representation of the layup prepared for VARTM process is shown in Figure 1. The vacuum bag was prepared by placing a layer of release film, 6 layers of woven carbon fabric (Cytec T650/35, plain weave, 3K), a layer of peel ply and desired length of distribution media over an aluminum tool plate. The layup was then enclosed in a vacuum bag and placed in an oven for infusion. The resin (Epon 862) and the cure agent (Cure Agent W) were mixed by a mechanical mixer at 250 rpm before infusion. Before infusion started, the vacuum bag and the plate were preheated to 121°C. The infusion was carried out at vacuum pressures of 26• to 27• Hg and often took 10-12 minutes to complete. Following the completion of the infusion process, the temperature is ramped at a rate of 5°C/min to the recommended cure temperature of 177°C. The layup is held at 177°C for 90 min before started to cool down at room temperature.

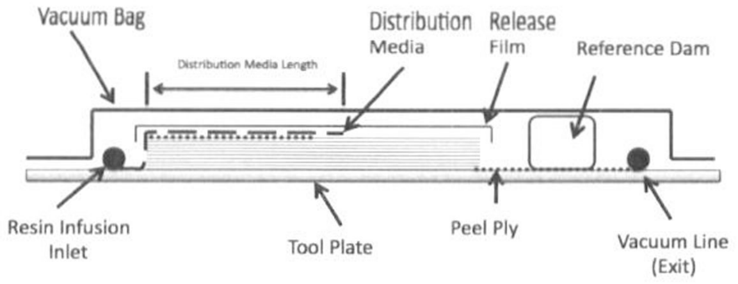

Figure 1. Schematic representation of VARTM process.

In addition to the effects of distribution media, effects of multi-walled carbon nanotube (MWNT) functionalization and morphology on the mechanical properties were investigated by fabricating laminates comprising 0.5wt.% (i) non-functionalized MWNT, (ii) -COOH functionalized, short MWNT, and (iii) -COOH functionalized, long MWNT. The MWNTs are purchased from Nanostructured and Amorphous Materials, Inc. Lengths of nanotubes were reported to be between 0.5 to 2□m for -COOH functionalized, short MWNTs and 10 to 30□m for -COOH functionalized, long MWNTs. Two laminates for each nanotube type were fabricated making a total of 6 laminates. MWNTs are mixed into the resin before mixing with the cure agent and sonicated for 2h. During the 2h sonication samples of resin are collected on microscope slides at 15min intervals. The average cluster area was obtained on each type of MWNTs. These laminates are fabricated using minimum amount of dispersion media possible.

Microscopic void analysis was performed to study general void structures in composite samples. A sample from each laminate was embedded into quick cure acrylic resin and polished with 600, 1500 and 2000 grit sand papers sequentially and studied at 200X magnification on a MEIJI metallurgical microscope. Same microscope is utilized at 100X magnification to study the resin samples collected during sonication. Using ImageTool software package, the images captured from resin samples are converted to binary images and characterized in automated mode to determine area fraction of MWNT clusters.

Microstructure of MWNT prior to dispersion is studied via scanning electron microscopy using a JSM-880 SEM by JEOL. MWNT samples as received from the supplier were placed on a copper tape and studied at various magnifications.

Effects of distribution media length and MWNT type on the mechanical properties were characterized in three-point bending with a span of 31.75mm. For the average sample thickness of 1.2 mm, a span of 31.75 yields span to depth ratios of more than 26 which comfortably satisfies ASTM's span-to-thickness threshold of 16 for flexural failure as stated in ASTM D790.

Results And Discussion

The effects of the length of distribution media on mechanical properties of carbon/epoxy composites is studied by placing minimum length of distribution media to form the baseline and over the ¼, ½ and ¾ of the 88.9mm length of the laminates. Two laminates for each case are fabricated and a total of 30 samples for mechanical testing are prepared. Figure 2 indicates the flexural strength and flexural stiffness of the laminates with different lengths of dispersion media. It is observed that as the distribution media length is increased to ¾ of the length of the laminate, the flexural strength and stiffness degraded by more than 14 and 12%, respectively. This reduction is believed to result from formation of microscopic voids as extended distribution media favors through-the-thickness infusion over longitudinal one-dimensional impregnation. As the laminates being fabricated are relatively thin (6-plies resulting in an average thickness of 1.2 mm) longer distribution media races the resin first on the top surface and forces it to infuse in the through-the-thickness direction. As the flow progressing along the length of the laminate and through the thickness meet, air pockets are entrapped forming the microscopic voids that later degrade the mechanical properties. Thus, for the given laminate thickness, it is identified that the use of minimum possible distribution media along the laminate to be fabricated provides optimum mechanical properties. Therefore, this baseline case is used when laminates for characterizing effects of different MWNTs were fabricated.

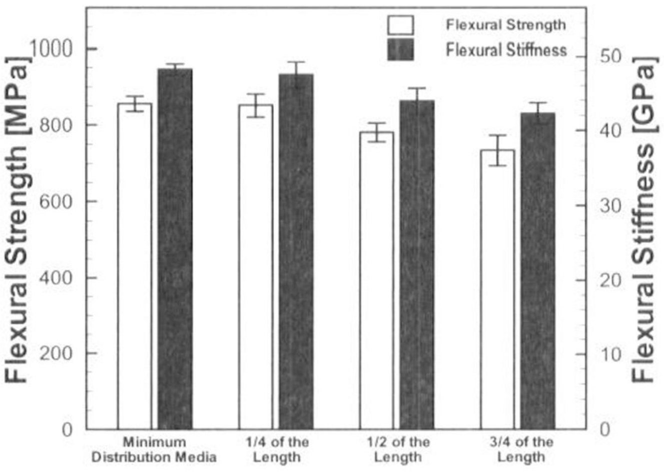

Figure 2. Effects of length of distribution media on the flexural properties of the composite laminate.

Figure 3 demonstrates typical void formations in a composite sample. Voids as large as 150 microns are possible when longer dispersion media is used. These voids are mostly observed in the resin rich inter-layer regions.

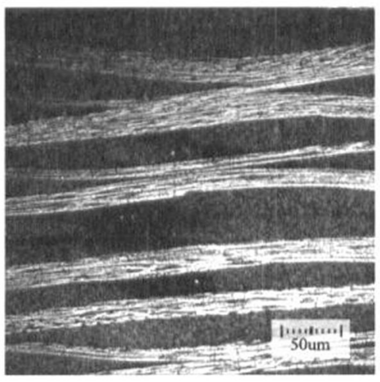

Figure 3. Microstructure of the through-the-thickness surface of a laminate depicting multiple voids (Image captured at 200X).

Scanning electron microscopy studies of the MWNT clearly identify different morphologies of the nanotubes used. Figure 4 shows non-functionalized, short MWNTs with -COOH functionalization and longer MWNTs with -COOH functionalization. It is observed that the non-functionalized MWNT used were more loosely entangled and were larger in diameter compared to its -COOH functionalized counterparts. When the short and the long functionalized MWNTs were compared, it is observed that as claimed by the supplier, their lengths were significantly different. However, both samples displayed similar characteristics of entanglement. Overcoming the high degree of entanglement commonly observed in MWNTs is the primary problem in achieving an effective dispersion within the resin before infusion.

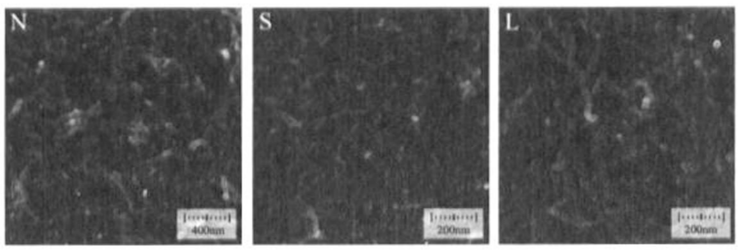

Figure 4. Scanning electron micrographs of three types of carbon nanotubes studied. N: Non-functionalized, S: -COOH functionalized, short MWNTs and L: -COOH functionalized, long MWNTs.

Although more entangled than the non-functionalized MWNTs, -COOH functionalized MWNTs displayed better dispersion characteristics as proven by the optical micrographs of the resin during mixing. Figure 5 shows images of the resin/MWNT suspension before and after sonication for the three types of nanotubes studied. It is clear that after 2h sonication, the average cluster area reduced for all of the nanotube types. However, non-functionalized MWNT sample contains considerably larger clusters that are larger than 50 microns. Figure 6 shows the evolution of average cluster size with time during sonication. The initial average cluster area for non-functionalized, functionalized short MWNTs and functionalized long MWNTs samples were 96.3, 18.15 and 32 pm^2, respectively. At the end of the 2h sonication period, these values dropped to 38.6, 14.3, and 20.4 pm^2. Although the highest drop is observed in non-functionalized MWNT with 60%, the final cluster area observed by the optical microscope (i.e., 38.6 pm^2) is significantly larger and is expected to degrade mechanical properties as well as result in significant filtering during the infusion. The smallest final cluster size is observed in the functionalized short MWNTs with 14.3pm^2. It can also be deduced from Figure 6 that the average cluster size approaches an asymptote within approximately 30min. Sonication beyond this time frame seems to have little effect in breaking down clusters further.

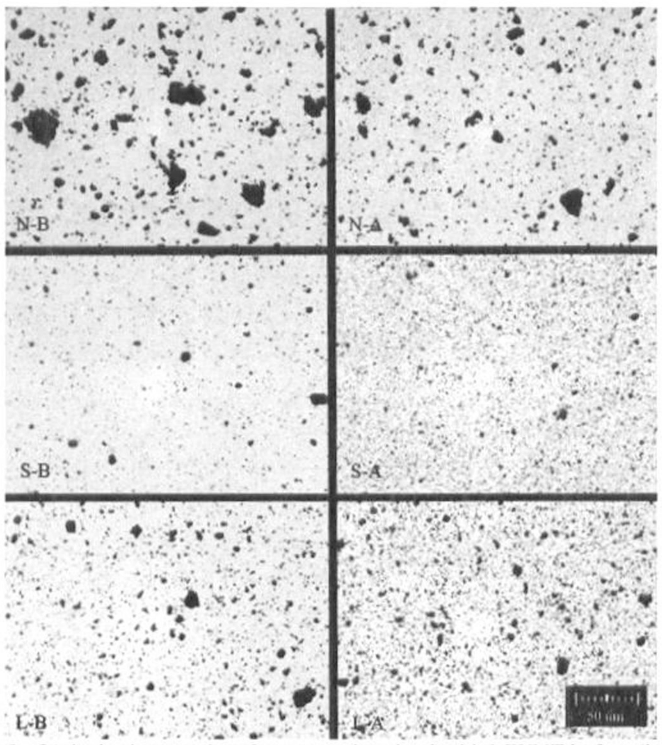

Figure 5. Optical micrographs of epoxy resin mixed with MWNT before (left) and after (right) sonication. N: Non-functionalized, S: -COOH functionalized, short MWNT and L: -COOH functionalized, long MWNT.

The mechanical properties of the laminates fabricated using different types of MWNTs were significantly different. Figure 7 shows the flexural strength and flexural stiffness of the laminates fabricated using the three types of MWNTs used. A total of 30 samples from two laminates fabricated are tested for each case. However, visual observations revealed localized voids that have degraded the properties. Thus, to eliminate the effects of voids and only resolve the effects of MWNTs, average of top 5 flexural strength and stiffness values are presented in Figure 7. To compare the effects of MWNT, mechanical properties of laminates fabricated using neat resin with baseline distribution media from Figure 2 is carried over to Figure 7. It can be seen from Figure 7 that addition of MWNT into the resin affects the properties considerably. Possibly due to incomplete dispersion,

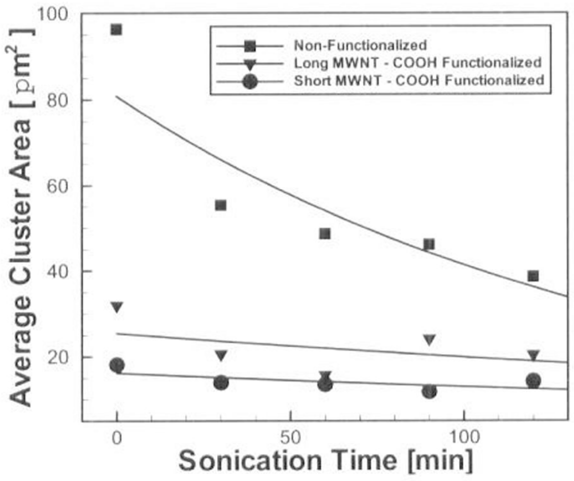

Figure 6. Evolution of the MWNT average cluster area as the sonication progresses.

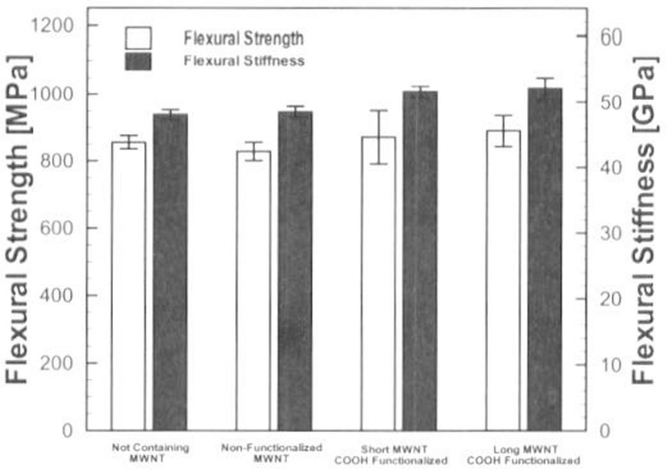

Figure 7. Flexural properties of carbon/epoxy laminates comprising different types of MWNTs.

laminates containing non-functionalized MWNTs displayed a slight reduction in flexural strength. On the other hand the flexural stiffness remained constant. However, addition of -COOH functionalized MWNTs improved both the strength and stiffness considerably. Addition of short functionalized MWNTs resulted in 2 and 7.3% improvements in the flexural strength and stiffness, respectively. Similar improvements of 4% and 8.3% in flexural strength and stiffness were observed in laminates incorporating long functionalized MWNTs. Clearly, the improvement in dispersion observed in Figures 5 and 6 with functionalized MWNTs manifested itself as improvements in mechanical properties as well.

Conclusion

The length of distribution media used to facilitate resin impregnation was shown to have a considerable effect on the mechanical properties of the fabricated composite laminates. The distribution media, placed at the top of the laminate, led to higher void content throughout the laminate, thus reduced both flexural strength and stiffness. The reduction in properties was observed to gradually increase as the length of the distribution media was increased, and reached a reduction level of more than 10%. Thus, in VARTM processes, fabricating composite laminates by utilizing a minimum amount of distribution media should be preferred. Effect of sonication on the dispersion characteristics of three different types of MWNTs was obtained as a function of time. With the help of sonication, the cluster sizes seen on optical micrographs were reduced significantly in all types of MWNTs. However, -COOH functionalized MWNTs dispersed much better compared to non-functionalized MWNTs in the poxy resin used. 0.5wt.% of -COOH functionalized MWNTs yielded improvement in both stiffness and strength of the fabricated nanocomposite laminates. In particular, almost 10% stiffness improvement is obtained in laminates that contain -COOH functionalized, long MWNTs.

Acknowledgement

Funding for this research is provided by a grant from AFOSR Grant# FA9550-10-1-0031.

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ELECTROCONDUCTIVE POLYMER COMPOSITES FOR BIOMEDICAL APPLICATION

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Abstract

This paper presents a small review of electroconductive polymer composites for biomedical purposes. Unlike well-known polymer composites consisting of a polymer matrix and a filler (loading agent), prepared usually by mixing components with a proper method, the electroconductive polymer composites for biomedical application embody rather fine and complicated systems made by very special and different procedures. Moreover they could not be defined as composites in the usual sense of the word. However these articles are true composites constructed often on a molecular level. During the past decade a series of publications have appeared describing application of certain polymers with conjugated double bonds such as polypyrrole, polyaniline and polythiophene; this field continuously expands to give many interesting and promising outcomes.

Introduction

Polymers containing a system of conjugated double bonds in backbones or polyconjugated polymers, such as polyacetylene, were initially disclosed as semiconductors possessing high electrical resistivity. Later it was discovered that these polymers can be easily transformed into conductive metallic state by doping with electron-donating (n-type), or electron-accepting (p-type) reactants. Despite the high conductivity achieved for such polymers practical application of the latter is still vague due to some disadvantages. In a pristine form those substances are prepared usually as infusible barely soluble deeply colored powders, which is a great difference compared to usual polymers. However, a new group of PCP obtained by oxidative polymerization of some heterocycles has attracted attention due to ease of preparation and excellent stability under atmospheric conditions. During the past decade a bunch of publication has appeared describing the possible application of some polymers with conjugated double bonds such as polypyrrole, polyaniline and polythiophene. These polymers are rather highly conductive in the doped state and show reasonably low biological activity. They are very accessible and may be prepared in a simple procedure through oxidative polymerization of appropriate monomers to yield polymers stable to oxygen and water media. Their ability to change electrical resistance by absorbing various chemicals became the basis for developing sensors for detecting inorganic and organic substances including those specific for biology (e.g., glucose, urea, DNA). Immobilization of bio-molecules by grafting onto surface of electroconductive polymers provides another interesting and prominent way for substrate modification, as well as drug controlled release stimulated by applied electric current and attempts to construct models of artificial muscles. Actually, all these arrays represent electroconductive polymer composites of conjugated polymers used for biomedical application.

Preparation And Basic Properties Of Some Conductive Polymers

Inherently conductive polymers are a group of polymers containing a system of extended conjugated double bonds. The term "inherently" means that these polymers show high electric conductivity due to their electronic structure other than conductive fillers. These polymers reveal rather high electrical resistivity at room temperature (10^{13}–10^{16} Ohm·cm) typical for usual dielectrics. However, they may be turned into conductive materials by treatment with small amounts of some electron-donating (n-type) or electron-withdrawing (p-type) substances (the so-called "dopants") with conductivity values in the range of 10^{-5} to 10^{5} S/cm. Due to this high electric conductivity these polymers were named "synthetic metals". Polyacetylene $(-CH=CH-)_n$, the first carbochain polymer built up form alternating single and double carbon-carbon bonds, was synthesized by several methods [1–3]. Comprehensive description of the polymer is given in the book [Chen]. Despite its high conductivity in doped state (as high as 10^5 S/cm), polyacetylene has low stability to oxidation and poor mechanical properties that together with complicated synthetic procedure makes its practical application impossible.

Another group of heterochain conducting polymers (HCP) was developed by oxidation polymerization of some aromatic and heterocyclic substances, such as aniline, pyrrole, thiophene, and their derivatives. These polymers have reasonable electrical conductivity. Thus, conductivity for polyaniline was found to be in the range of 10^{-4}–10^{1} S/cm, for polypyrrole – $7 \cdot 10 - 2 \cdot 10^{2}$ S/cm, and for polythiophene – 10^{-2}–10^{2} S/cm. In contrast to polyacetylene, these polymers can be easily prepared under ambient conditions in water or organic media by chemical or electrochemical oxidation. They are stable in atmosphere under ambient conditions and reveal rather high electrical conductivity in the doped state. However, these polymers are deeply colored from dark blue and green to black that does not make them much attractive. Besides, they have limited solubility and poor processing ability compared to standard polymers commonly used for biomedical application. However, during past years a bunch of papers has been published describing some evident and new fields of application for such heterochain conductive polymers (HCP).

Biosensing Devices

The first and evident application for HCP is biosensors construction. Indeed, these polymers are able to interact reversibly with various chemical substances which is accompanied by a remarkable change in their conductivity in the range of $10^6 - 10^8$ orders. Therefore, a small amount of some substances can be detected. Thus traces of ammonia can be detected by using HCP-based sensor devices. For example, ammonia reacts with polypyrrole in the sensing element resulting in sharp decrease of electric resistance that can be monitored by any suitable method. After washing with dry nitrogen the resistance can be totally recovered [4]. Nitrate ions NO_3^- and carbon monoxide were also detected with polypyrrole and polyaniline-based sensors, respectively [5, 6]. This principle property of HCP was used for creation of sensors for biological application. Such biosensor should contain a HCP sensing element including bio-molecule and a transducer. Interaction of a bio-substrate with bio-receptor results in the change in conductivity of conducting polymer layer that can be scanned by a transducer. Thus, an amperometric biosensor for uric acid determination was prepared from urease immobilized in polyaniline-polypyrrole film [7]. Actually, such sensing devices represent a composite system consisting of several elements. Electronic tongue proposed in [8] was able to distinguish salt, sweet, bitter and acidic solutions. It was composed of six different sensing units: a bare interdigitated electrode and interdigitated electrodes coated with stearic acid, a polyaniline oligomer, polypyrrole and mixtures (1:1, w/w) of 16-mer/stearic acid and polypyrrole/stearic acid.

Some organic substrates participating in standard metabolism should also be mentioned, which were determined with biosensors including enzymes: glucose (glucose oxidase) and urea (urease) – polypyrrole-based sensors [9, 10], cholesterol (cholesterol oxidase/esterase) – polyaniline-based sensor [11], DNA – polypyrrole-based sensors [12].

Tissue Engineering

Evidently, all conductive polymers have rather rigid backbone macromolecules that create some problems with their mechanical properties even for biomedical application. Composite materials may help to solve this problem, at least partially. A composite material was synthesized consisting of polyvinyl alcohol and polypyrrole and a carrier with high surface but low conductivity was obtained suitable for cell adhesion [13]. Some various biomolecules have been entrapped, such as adenosine 5'-triphosphate [14–16] and nerve growth factor [17] in polypyrrole and other conductive polymers for both drug delivery and tissue engineering applications. In particular, co-entrapment of nerve growth factor and dextransulfate, followed by the controlled release of the former via polypyrrole reduction [17].

Another approach to bio-composites of HCPs and biopolymers is a complex of polyglutamic acid with polypyrrole described in [18]. This functionalization technique binds the bioactive molecules at the surface, but simultaneously reduces conductivity. This novel technique is advantageous in that a single material can be used to easily incorporate a range of different biomolecules including uncharged ones.

Beside that, it was found [19] that peptides can modify surface of polypyrrole with the arginine–glycine–aspartic acid peptide and promote cell adhesion in serum-free media, whereas no cell adhesion was seen on unmodified surfaces. Advantageously this surface modification technique will not affect conductivity and could be used to modify polypyrrole with a range of different biomolecules that do not need to be charged.

Neural probes are multichannel electrode arrays that facilitate the functional stimulation and recording of neurons in the peripheral and central nervous system. For long-term implantations, surface modification is necessary for maintaining the stable connection between electrodes and neurons. The conductive polymer polypyrrole and synthetic peptide were co-deposited on the electrode surface by electrochemical polymerization. Coated probes were implanted into brain of guinea pigs for periods of 1, 2 and 3 weeks. Recording tests were performed and the impedance was monitored. The explanted probes and tissue were examined by immunocytochemical studies. Significantly more neurofilament positive staining was found on the coated electrode which indicated that the coatings had established strong connections with the neuronal structure in vivo [20].

In another approach, cellulose fibers were used as a substrate for preparation of a composite material with polypyrrole [21] by adsorption polymerization; a special technique should be used for creating a high specific surface area that is important for close contacts with neurons [22].

Perhaps the main limitation of conductive polymer composites for in vivo applications is their inherent inability to degrade.

Conducting Polymers For Drug Release Regulation And Artificial Muscles

The doping-redoping process for heterochain conducting polymers is accompanied by visible volume change that may be used under appropriate conditions for drug storage and subsequent drug release. Thus, the release of heparin from polyvinyl acetate hydrogels grafted onto polypyrrole films was stimulated by applying electrical potential and an accelerated release of heparin from the hydrogel was observed under electrical stimulation of polypyrrole [23].

Nanotubes from poly-3,4-ethylenedioxythiophene were prepared (~100nm in diameter) and loaded with dexamethasone. The nanotubes released the drug in a controlled fashion upon electrical stimulation, probably as a consequence of expansion/reduction of polymer cavities produced by the expulsion of anions [24].

The change in volume and linear dimension under electrical stimulation of heterochain conducting polymers can be used for model development of artificial muscles. A triple layer composite of conducting polypyrrole film separated with a polymer insulator was constructed. By applying electrical potential one could observe bending of the layered sample comparable to the reaction of natural muscles [25, 26]. Various composites and composites of conductive polymers with carbon nanotubes showed ability to function as actuators and the composite with nanotubes increased the electromechanical effect [27]. These types of devices are promising as actuators for many biomedical applications, such as steerable catheters for minimally invasive surgery [28], micro pumps [29, 30], blood vessel connectors and micro valves for urinary incontinence [31].

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FLOW PATTERN OF LCP/PET BLENDS AT SOLID AND MOLTEN STATES OF LCP

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Abstract

Liquid crystalline polymers (LCP) are high performance engineering materials with high strength, stiffness, chemical resistance, and low linear thermal expansion coefficient. LCP consists of repeating stiff mesogenic (liquid crystalline) monomer units those are incorporated either in the main-chain or in the side chain of a polymer backbone. The liquid crystalline polymer used in this study was co-polyester of p-hydroxybenzoic acid (HBA) and 2-6-hydroxynaphtoic acid (HNA), Vectra A 950 of Polyplastics Co.Japan. It was blended with polyethylene terephthalate (PET) in different proportions. Polyethylene terephthalate was a commercial product 1206 of Unitika Co., Japan The rheological properties of these blends have been reported under different conditions. The studies related to the different physical states of LCP in the blend i.e. the solid state at 265°C and the melt state at 285°C provide interesting information. The PET was in molten state at both the temperatures. The flow behavior of immiscible polymer blends is more complex due to the competition between deformation, breakup, coalesce of dispersed phase and viscoelastic nature of phases. The viscosity of the blends showed its dependence on shear rate, temperature and the composition. A maxima was observed in the viscosity versus composition plot and its intensity was highly shear dependent. First normal stress difference (N_1) has also been examined and the increased value of N_1 with shear- rate was explained assuming a tendency of asymmetric particles to rotate under velocity gradient of suspending medium.

Introduction

Liquid crystalline polymers (LCP) are high performance engineering materials. The mesogenic phase such as nematic units enables LCP to orient along the flow direction during processing and contributes significantly in reducing the viscosity of melt. Blending with thermoplastics further broaden the scope of LCP that functions as process aid as well as a reinforcement[1-2].

The studies related to LCP/PET blend are generally concentrated around studying the mechanism and conditions of lowering viscosities, formation of LCP fibrils etc. but the high viscosity of LCP/PET blends than those of constituent polymers is discussed rarely [3,4]. The shear rate dependence of viscosity and first-normal-stress-difference (N_1) of LCP/PET blends at two different temperatures each one at below and above melting temperature of LCP with varying blend composition are discussed here. The increased value of N_1 with shear- rate is explained assuming a tendency of asymmetric particles to rotate under velocity gradient of suspending medium.

Experimental

Materials and Methods

Vectra A 950 of Poly-plastics Co. Japan was co-polyester of p-hydroxy-benzoic acid (HBA) and 2-6-hydroxynaphtoic acid (HNA). Polyethylene terephthalate (PET) was a commercial product 1206 of Unitika Co., Japan. Both polymers were dried in an oven at 90°C for 14 hours and then at 150°C for 4 hours before processing. The composition of LCP and PET was varied as 10, 20, 40, 50, 60, 70, 80, 90 and 100 volume percent of LCP in LCP/PET blend. An elastic melt extruder was used for blending at 285°C. The elastic melt extruder works on the Weissenberg principle [5]. The blended material was quenched in water at 12°C and was further cut to 5 mm length. The pellets were dried in an air-circulating oven at 75°C for 12 hours. The weighed amount of pellets was compressed between the hot plates for 3 minutes under 5 MPa pressure at 265°C. The mould was then cooled immediately under the same pressure by circulating water at 12°C and sheets were prepared. These sheets were cut to disc shape for tests. These discs were tested on a cone plate type rheometer (radius, R = 1.25 cm), from Nihon Rheology Ki-Ki Co. Ltd.

Shear stress v_{12}, viscosity k and first normal stress difference, $\mathbf{N_1}$ were calculated using following relations;

$$v_{12} = 3T / 2sR^2 \qquad (1)$$

$$k = v_{12} / \dot{j}' \qquad (2)$$

$$N_1 = 2F / sR^2 \qquad (3)$$

where torque T and thrust F acting on cone were measured at different shear rates $\dot{j}'$

The complex viscosity, ÉK*Éwas calculated from storage modulus G′ and loss modulus G″ values at two different temperatures as per following relation [3]

$$ÉK^*É = [(G'/z)^2 + (G''/z)^2]^{1/2}$$

Results and Discussion

Shear viscosity

In the plots, not shown here, between the viscosity of LCP/PET blends and composition of blend at 265 and 285°C, at different shear rates, the viscosity increased with increasing LCP content in the blend. The viscosity showed high sensitivity with shear rate, particularly at low shear rates, it increased sharply with the change in composition of blend. A maxima was observed in LCP/PET:: 90/10 blend which indicates immiscibility of components . Shear rate sensitivity is high amongst the sample containing more than 50% of LCP in the blend. Shear-thinning phenomenon was observed and and was strongly composition dependent in this case.

Viscosity of PET is least amongst all other blends as well as LCP and decreased with increased shear rates. Viscosity increased with addition of LCP in PET up to 90 % of LCP in PET. Interestingly, viscosity of LCP is lower as compared to LCP/PET blends having higher volume fraction of LCP than PET. Due to the competition between deformation, breakup, coalesce of dispersed phase and viscoelastic nature of phases the flow behavior of immiscible polymer blends is very complex. The imposed deformation is very less and chances of fibrillation at 285°C are negligible. Hence it may be assumed that LCP remained in droplet form distributed in PET matrix showing high viscosity of system. On increasing shear rate these droplets were deformed in flow direction and shear thinning was observed.

First normal stress difference

Normal stress developed during shear flow of a viscoelastic fluid implies finite elastic strain developed in the fluid. These stresses have their origin in elasticity of liquids. The normal stress difference is expressed in terms of principal stresses.

When N_1 was plotted against shear rate at two different temperatures 265°C and 285°C, N_1 increased with content of LCP in the blends and with shear rate. N_1 is expected to decreases with increased loading of filler in a polymer matrix because of reduced recoverable elastic strain in the system. A possible mechanism to explain experimental observation is given below. The dispersed particle in a sheared flow experiences a torque due to the differential velocities of layers of suspending medium. This torque tries to rotate the particle on it's own axis. The tendency of rotation of particle disturbs the flow. An increase in asymmetry of particles would disturb the flowing layers and a normal force perpendicular to flow direction develops. This exerts a normal force on layers of suspending medium due to the shape of particle. This force is expected to be proportional to number of particles and the asymmetry of particles.

Conclusions

High shear viscosity was observed in blends having more than 50% of LCP than that of either LCP or PET at low shear rates. LCP was found more shear sensitive as compared to PET. A maxima was observed in viscosity versus composition plot. First normal stress difference decreased with LCP content in the blend at 265°C whereas at 285°C this trend was opposite. The value of N_1 increased with shear- rate as well as with shear stress indicating increase in recoverable elastic strain with shear.

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BIOPLASTICS AND GREEN NANO-BIOCOMPOSITES: PAST, PRESENT AND FUTURE

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The unpredictable price of crude oil, reduced land-fill space and escalating environmental threats are daily headlines. The government's push for green products, consumers' desire, and energy conservation are some of the key factors that drive research towards the development of renewable resource-based natural and green composite materials. Biobased economy is challenging to agriculture, forestry, academia, government and industry.

The incorporation of bio-resources, e.g. crop-derived green plastics and plant derived biofibres (natural fibres) into composite materials are gaining prime importance in designing and engineering green composites. Biocomposites derived from natural fibers and traditional polymers like polypropylene and unsaturated polyesters resins have been developed for automotive parts and building structures. Renewable resource based bioplastics like polylactic acid (PLA), polyhydroxyalkanoates (PHAs), biobased polytrimethylene terephthalate (PTT), cellulosic plastics and vegetable oils derived bioresins need value-added and diverse applications to compete with the fossil fuel derived plastics. Through reactive blends, composites and nanocomposites new biobased materials are under constant development.

Natural fibres are lighter, less expensive, have superior specific strength, require comparatively less energy to produce, are good for the environment, biodegradable and have superior sound abatement characteristics as compared to synthetic glass fibres. All of these attributes are quite favourable, especially in the automotive sector where even a fractional weight saving can make a significant contribution to energy savings with reduced gasoline consumption and with added advantages of ecofriendliness. It is true that natural fibres are comparatively hydrophilic and less thermally stable as compared to glass fibers. However, the recent developments of natural fibre technologies overcome these disadvantages if used intelligently. Hybrid and intelligently engineered green composites are going to be the major drivers for sustainable developments. Besides agricultural natural fibers like kenaf, jute, flax, industrial hemp, sisal and henequen as well as inexpensive biomasses such as wheat straw, rice stalks, corn stovers, grasses, soy stalks and lignin (the byproducts from pulp and paper and lingo-cellulosic ethanol industries) have great potential for use in sustainable biobased composite materials.

Natural fibre reinforcements in conjunction with nanotechnology are poised to create major breakthroughs. Green nanocomposites that are derived from bio-based polymers and bio-based nanoparticles tend to be viewed as the next generation of materials for innovative industrial uses.

This presentation will highlight the current status, opportunities and challenges of bioplastics, natural fibre composites and nano-enhanced bicomposites (nano-biocomposites) for uses in car parts, consumer goods and sustainable packaging.

Acknowledgements: Ontario Ministry of Agriculture, Food and Rural Affairs (OMAFRA)-New Directions Research Programs; University of Guelph-OMAFRA (Bioeconomy for Industrial Uses); Natural Sciences and Engineering Research Council of Canada (NSERC)-Discovery Grant; and Auto21.

ANALYSIS, DESIGN AND OPTIMIZATION OF STRUCTURAL COMPOSITES: TRENDS AND CHALLENGES

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Abstract

In this paper, the overall computer-based design process of structural composites is recalled. Optimization and failure are the two trends and challenges discussed here. Some optimization methods recently developed and new ideas for solving the ply distribution and the stacking sequence problems are discussed. The modeling and analysis of composite failure, and especially delamination, is also addressed and numerical methods developed to solve this problem are compared. Even though advanced numerical techniques exist for the analysis and optimization of composites, research is still necessary to improve the modeling of so complex materials.

1. Introduction

Nowadays composite materials are used extensively and their design for advanced applications is accomplished using computers and numerical tools. This typically involves three disciplines. The first one, called Computer Aided Design (CAD), aims to define the overall geometry of the part, and the regions of laminates with their stacking sequence. It is linked to Computer Aided Manufacturing (CAM), which provides specific capabilities for the manufacturing processes simulation. These tools are used to determine the accurate fiber orientations and the deformation of the plies during draping. The last discipline, called Computer Aided Engineering (CAE), is used to analyze the structural integrity of the composite structure when subjected to the expected loads.

The regions of laminates with their detailed stacking sequence are determined in the analysis phase, and further reported by the designer in the CAD model for a draping simulation. The selection of the optimal stacking sequence and the ply distribution in the structure is a challenge. Since the problem is combinatorial, specific parameterizations of the model and adapted numerical optimization methods must be used.

In most cases, the finite element method is used for the analysis of the structural composites, especially for complex geometries. Composite structures exhibiting non linear material behaviors, large displacements and instabilities under the in-service loads can be analyzed nowadays. One particularity of structural composite components, when compared with metallic parts, is delamination. This specific fracture process can be modeled and analyzed with fracture mechanics and the cohesive elements approach. Even if interesting results have been obtained for the simulation of inter-laminar crack propagation in laminated composites, assessing damage tolerance of such structures remains a challenging topic.

In this paper, the overall design process of structural composites is recalled. Some optimization methods recently developed and new ideas for solving the ply distribution problem are then discussed. Finally, the problem of delamination is addressed and numerical methods are compared. The computations are conducted with the SAMCEF finite element code and the BOSS Quattro optimization tool box (www.samtech.com).

2. The computer-aided design process of composite structures

The design process includes three main disciplines [1]. First, Computer Aided Design (CAD) is used for the definition of geometry, laminate and plies properties, i.e. the stacking sequence and the plies location. Typically, the designer assigns composite properties inside zones delimited by structural members such as stiffeners. Plies are defined on the structure and can intersect inside specific zones, providing in each zone laminates of different thickness and fiber orientations (Figure 1). The designer can apply specific tools to estimate the deviation of the fiber orientation resulting from the draping or the true fiber trajectories when AFP techniques are used. In this case, CAD is linked to Computer Aided Manufacturing (CAM) software.

In the Computer Aided Engineering (CAE) phase, numerical tools are used to assess the structural integrity of the composites. For simple geometries, analytical methods based on the classical lamination theory are used. To obtain more accurate results, the finite element method is preferred. Both approaches can be combined to study real-life structures [2]. These tools are used to determine the general structural properties of the composites, i.e. mass, vibration frequencies, stiffness and buckling responses regarding the applied loads. Local criteria are also taken into account to assess the possible inter and intra-laminar ply failure [3]. Thanks to the capabilities of the CAD-CAM software, the analyses can be conducted with the true fiber orientations and properties. During this design stage, optimization is often used to identify the optimal ply distribution over the structure and the stacking sequence in each laminate [1,2,4]. This information is then reported to the designer in his CAD environment and possibly processed by the CAM software in order to validate the design with the manufacturing restrictions.

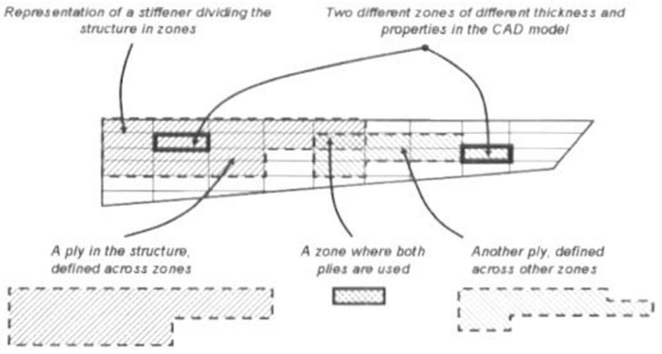


Figure 1. Zones and ply definition in a composite structure: CAD representation

The design process is clearly iterative, alternating CAD, CAM and CAE steps. When only CAD and CAE are used, a preliminary design is addressed. When CAM is included in the loop, manufacturing constraints are taken into account in a design-for-manufacturing approach [5-8].

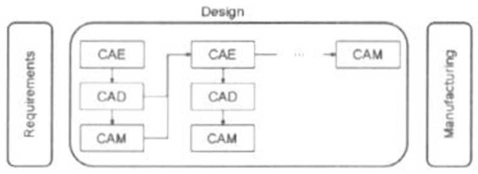


Figure 2. The iterative design process of composite structures

3. Optimal design of composite structures

The interest for structural optimization of composites has grown over the last decade, since primary aero-structures are now made of such specific materials (see the Boeing 787 and Airbus A350 aircrafts). The goal of structural optimization is to provide lightweight and safe designs [4,9]. The general formulation of an optimization problem is written in (1). It includes one objective function $g_0(\mathbf{x})$ to be minimized and m constraints $g_j(\mathbf{x})$. These functions depend on n design variables $\mathbf{x} @ \sim x_i @ i @ 1,\ldots,n¢$, which are the parameters whose values are varied to find an optimal solution. At the optimum, each constraint must be lower than or equal to a given value $\bar{g}_j$. Side constraints are added to the problem, providing lower and upper limits on the design variable values, representing physical or manufacturing restrictions.

$$\min_{x} g_0(\mathbf{x}) \quad \textbf{s.t.} \quad g_j(\mathbf{x}) \, f \, \bar{g}_j ,\ j @ 1,\ldots,m \quad \textbf{and} \quad \underline{x}_i \, f \, x_i \, f \, \bar{x}_i ,i @ 1,\ldots n \tag{1}$$

The functions g_j in the problem (1) generally are non linear. They can be global and impact the whole structure, as is the case for the structural stiffness, the buckling loads or the vibration frequencies. They can also be local, and so defined in each ply (Tsai-Wu and Tsai-Hill criteria). The solution of problem (1) is obtained iteratively. At each iteration, a structural analysis is carried out, the results of which feed the optimizer which provides new values for the design variables. Several optimization methods exist and can be used to solve the problem (1). Gradient-based methods, such as the Mathematical Programming methods [10] and the Sequential Convex Programming methods [11] use the first order derivatives of the structural functions. Zero order methods, such as the Genetic Algorithms [12] or the Surrogate Based Optimization methods [13], use only the function values. The latter require a large number of structural analyses, but can be used directly when the gradients are not available. A lot of formulations can be defined for the optimal design of composites [4,9]. In this paper, the identification of the optimal plies in the structure and the problem of optimal fiber orientation are addressed.

3.1. Optimal fiber orientations and ply distribution

Most of the time, conventional laminates are used in aeronautics and the fiber orientations are limited to 0°, 45°, -45° and 90° plies. Although finding the optimal orientations is a problem which is discrete by nature, it can be written in terms of continuous design variables. This formulation is appealing since reliable optimization algorithms able to manage large scale problems with continuous variables exist [14]. In this approach [15], the constitutive matrix **Q** of each ply k is replaced by a linear combination of the constitutive matrix $\mathbf{Q}(\square\square)$ of the 4 candidate plies, as in (2). The goal is to determine which w_i is equal to 1 at the solution, while the others become 0.

$$\mathbf{Q}^{(k)} @ w_1\mathbf{Q}^{(k)}(0\cdot) \;.\; w_2\mathbf{Q}^{(k)}(45\cdot) \;.\; w_3\mathbf{Q}^{(k)}(90\cdot) \;.\; w_4\mathbf{Q}^{(k)}(045\cdot) \qquad (2)$$

This principle is illustrated in Figure 3. The structure is divided in zones where the optimal fiber orientations are sought. The solution is provided in Figure 4 [16]. For the details on the evaluation of the w_i coefficients in (2), see [15].

In order to relate the optimization phase to the design phase, and to define the plies based on analysis requirements, this kind of formulation can be adapted to the optimal distribution of the plies in the structure (Figure 1). Predefined plies covering the structure can be proposed by the designer, as illustrated in Figure 5, creating a store of candidate plies.

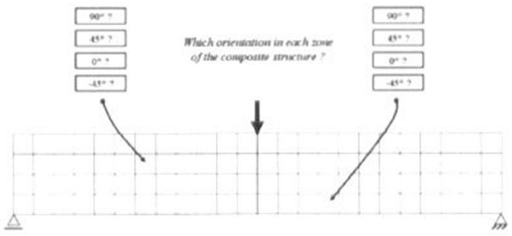


Figure 3. Optimal fiber orientation using conventional plies: problem statement

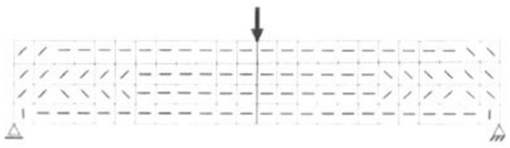


Figure 4. Optimal fiber orientation using conventional plies: solution

The presence or absence of each ply and the corresponding optimal fiber orientation can then be obtained by the analyst with an optimization approach. It is interesting to note that the blending requirement is satisfied with this formulation.

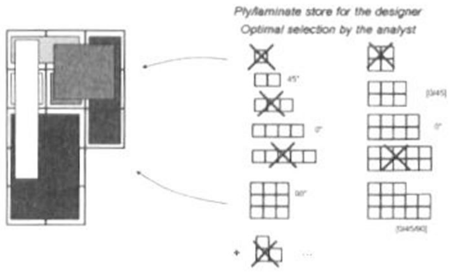


Figure 5. Plies store for the design and analysis phases

The presence or absence in the final design of each candidate ply *k* of the store is identified with a topology design variable, named □$^{(k)}$. Such variables are used to determine whether or not a ply of the store will be present in the solution. As it was done in (2) for the constitutive material stiffness, the ply thickness can be parameterized with respect to the thickness of the 4 candidate materials corresponding to plies oriented at 0°, -45°, 45° and 90°. These variables are used to determine the fiber orientation in the existing plies at the solution. The ply parameterization writes:

$$t^{(k)} = \frac{1}{P(k)}\, t^{q} \left\{ \sum_{i=1}^{4} w_i^{(k)} t_i^{(k)} \right\} \tag{3}$$

As it is the case in topology optimization [15], an exponent q is used in (3) in order to avoid a mixture of the candidate plies at the solution.

This new approach is tested on a simple example, illustrated in Figure 6. The structure is divided in 4 regions, and 16 candidate plies are defined in the store. Their number provides their location in the stacking sequence. The goal is to select the relevant plies and to determine their optimal orientation, in a structure of maximum stiffness. The problem includes 48 design variables: 16 topology design variables (one for each candidate ply), and 32 variables for the selection of the optimal fiber orientation (2 design variables for each of the 16 plies, see [15]). A constraint imposes that 8 plies must remain in the structure at the solution.

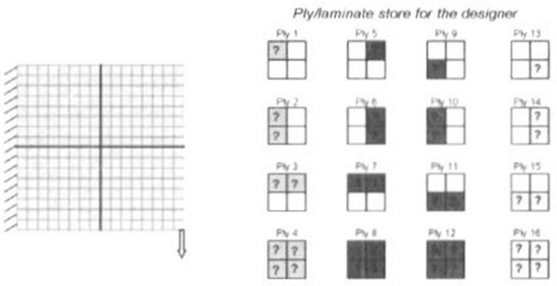


Figure 6. Test case: candidate plies with unknown orientation

The solution is provided in Figure 7. It is seen that large plies with fibers oriented at -45° are present in the structure, together with 0° plies at the fixed edge and 90° plies in the load direction, what is relevant with the physics of the problem. The solution is obtained in 11 iterations with the specific gradient-based optimizer described in [14]. Applications of this formulation to large size structures, such as the one of

Figure 1, will be undertaken in a near future. See [17] for another possible formulation not presented in this paper. Research is still very active in this field but a general solution procedure not yet identified.

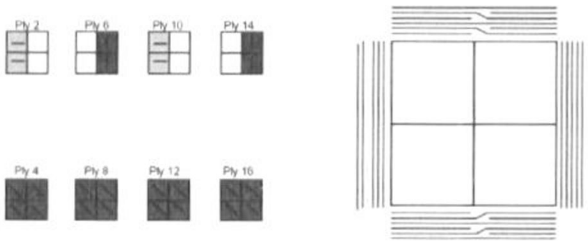


Figure 7. Solution of the test case: selected plies and their optimal orientation

3.2. Optimal stacking sequence

When working with conventional orientations (0°, 45°, -45°, 90°), specific design rules must be satisfied by the optimal stacking sequence. These rules specify that, e.g. there must be at most 4 successive (superposed) plies with the same angle, the maximum gap between two adjacent (superposed) plies is 45° and a minimum percentage of each fiber orientation must exist at the solution. Such constraints can be expressed in terms of the coefficients w_i used in (2) and (3), as explained in [18], and included in (1).

4. Modeling the failure of composites

Another key issue in the design of composites is to assess their damage tolerance. Efficient numerical methods are now available to study the inter and intra-laminar failure of laminated composite structures [19]. One particularity of structural composite parts, when compared with metal, is delamination. This specific fracture mode can be modeled and analyzed with fracture mechanics and the cohesive elements approach [20]. In the fracture mechanics approach, the Virtual Crack Closure Technique (VCCT) or the Virtual Crack Extension (VCE) method can be used [20,21]. Both methods aim to compute the energy release rates by modes, G_I, G_{II} and G_{III}, at the crack front. Once obtained, these values can be included in a criterion and compared to the critical fracture toughness G_{IC}, G_{IIC} and G_{IIIC} in order to determine if the crack is dangerous, the propagation load and to potentially simulate the crack propagation. Results obtained with VCE and VCCT in the computation of the energy release rates are provided in Figure 8 for a SLB specimen. On the other hand, in the cohesive elements approach, a specific material law is assigned to a thin layer of finite elements modeling the interface between two plies (Figure 9). For a certain range of solicitation in the interface, the material behavior remains elastic. Beyond this limit, a damage appears in the interface, which can grow up to the final de-cohesion of the adjacent plies, simulating an inter-laminar crack propagation. This method is very efficient and certainly more reliable than VCE and VCCT when multi-delaminated problems are studied [20]. However, the characterization of the material properties needed to feed the material law of Figure 9 is challenging.

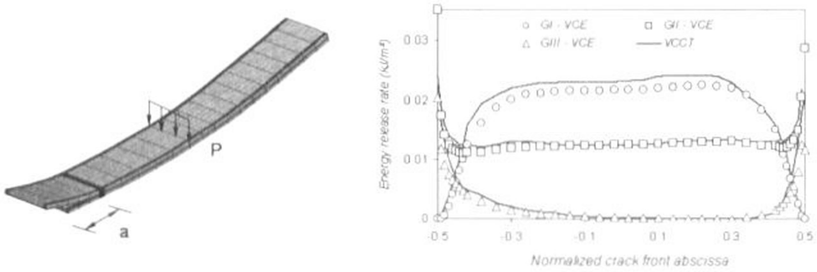


Figure 8. Comparison of VCE and VCCT

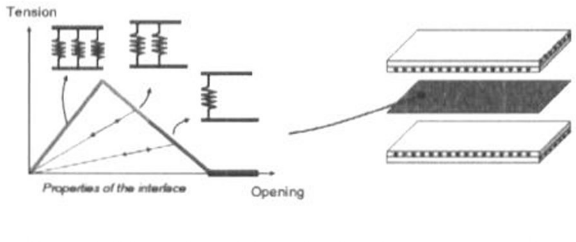
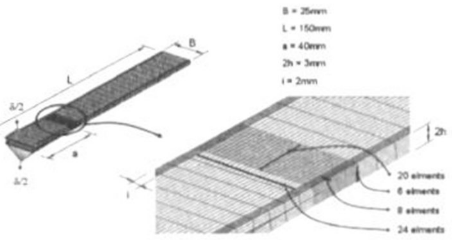


Figure 9. Illustration of the cohesive elements approach

The simulation of crack propagation with a pure fracture mechanics approach (VCCT and VCE) remains difficult to manage, requires a very fine mesh in the vicinity of the crack and can sometimes provide inaccurate results. This is demonstrated with the DCB specimen modeled in Figure 10. Oscillations can appear in the load-opening curve for the implementation of the VCCT method described in [21]. For the VCE [19], a more stable crack propagation is obtained, which is quite close to the analytical solution. When the cohesive elements approach is used, the non linearities are more present, and the solution depends on the material properties assigned to the interface.

5. Conclusions

The overall computer-based design process of structural composites has been reminded. This process is iterative and includes CAD-CAM-CAE steps. Attention has been focused on the CAE stage of the design process. New ideas for solving the ply distribution problem, as well as the stacking sequence optimization problem have been discussed. The modeling of composite failure, and especially delamination, is also presented and numerical methods developed to solve it are compared.



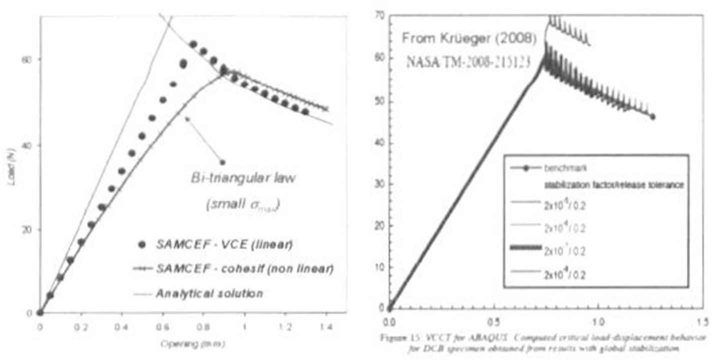


Figure 10. Results obtained for the crack propagation in the DCB test case

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