August 9, 2005

Dr. Christopher C Ibeh, Director

2005 PSU-CNCMM REU/RET

Professor, Plastics Engineering Technology

Pittsburg State University

RE: **Properties of Nanocomposites Impregnated Aramid Papers**

Dear Dr. Ibeh:

I am very pleased to inform you that I have completed the above stated project.

The main objectives of this report is to inform you that I have accomplished the mechanical test (Tensile testing) of the above project and also the flammability test on the Nomex paper. This report evaluates the procedure and principles of the experiment, along with results of my research work.

I hope this report will meet with your approval. If you have any other information that you would like to see from the experimental, I'll supply it for you upon your request. If you have any questions or comments, I can be reached at bernzioki@yahoo.com

Sincerely,

Bernice Nzioki

Properties of Nanocomposites Impregnated Aramid Papers

July 27, 2005

By

Bernice Nzioki

Plastics Engineering Technology

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Submitted to:

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Advisor

Dr. Andrey Beyle

Table of Contents

Abstract

The main objective of this research was to study the mechanical load carrying capacity, prevent permeability and develop better thermal insulation of both Kevlar and Nomex paper by impregnating the papers with polymers and adding Nanoparticles.The use on nanoparticles in combination with polymeric materials to create plastics nanocomposites is a current trend. The infusion of these nanoparticles into the polymeric matrix is typically carried out through sonication but in this project Mechanical mixing was the main mixing procedure .Kevlar and Nomex are synthetic (Man-made) material known as a polymer. A polymer is a chain made of many similar molecular groups bonded together called monomers. A single Kevlar or Nomex polymer chain could have anywhere from five to a million segments bonded together.

Test bars for this experiment with and without nanoclay were obtained through hand lay-up and vacuum assist forming. The clay percentage used was limited to 4%.

The results obtained from this experiment indicate that Nomex has a greater tensile strength than Kevlar. Pure Nomex had a tensile strength of 15.17 ksi while pure Kevlar yielded 2.34 ksi.Burn tests was not carried out for Kevlar because the samples available were not adequate for the bunt test to be performed.

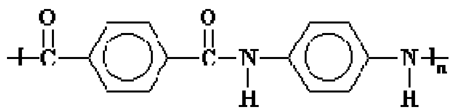
Introduction

Polymer nanocomposites were developed in the late 1980s in both commercial research organizations and academic laboratories. The first company to commercialize these nanocomposites was Toyota, which used nanocomposite parts in one of its popular car models for several years. Following Toyota's lead, a number of other companies also began investigating nanocomposites. Since then the interest increased because they produced improved properties, such as barrier properties, tensile strength, modulus, and heat and flame resistance. Modified layered silicates as fillers are dispersed within a polymer matrix and new extraordinary properties are obtained. Two types of clay can be incorporated into the formulation to produce the nanocomposite.

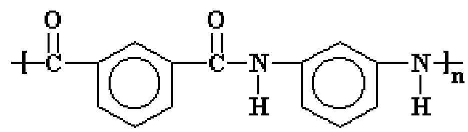
Natural clay is called montmorillonite, and synthetic clay is called fluorehectorite. Through nanocomposite formation the thermal stability and flame retardancy of polymers are also improved. The flame retardancy of layered silicate nanocomposites is based on Char formation and its structure. These types of materials also have excellent chemical resistance. The purpose of this study is to examine the effects of different amounts of Clay % by weight in the Aramid nanocomposite. The student will be examining and discussing the results of flammability resistance using a burn test. Mechanical properties of the nanocomposites will also be examined in order to determine what type of applications this material will best be suited for. These mechanical tests will be conducted on the Instron tensile testing machine.

Kevlar was first synthesized in 1964 by Stephanie Kwolek at the Dupont laboratories in Wilmington, Delaware in the United States. KEVLAR® is a compound that was invented in the early 1960's by Stephanie Kwolek. The compound that she created was predicted to be five times stronger than steel and weighed far less. She patented the compound in 1966. She worked in Dupont's research lab as soon as it opened in 1950. Kwolek's job was to find new and stronger polymers. She created polymers by synthesis and one day created a polymer in a test tube that was a cloudy solution, but Dupont wasn't interested in a non-clear polymer solution.

Kwolek insisted on spinning, which would cause the Kevlar in the solution to become separated from the solution. Her insistency returned an incredibly useful compound. This compound would soon find many uses and would save thousands of lives. KEVLAR® is created through a process called interfacial polymerization which uses the process of synthesis at very low temperatures.



In Kevlar the aromatic groups are all linked into the backbone chain through the 1 and 4 positions. This is called *para*- linkage.



In Nomex the aromatic groups are all linked into the backbone chain through the 1 and 3 positions. This is called *meta*- linkage.

Literature Review

The ballistic impact characteristics of Kevlar® woven fabrics impregnated with a colloidal shear thickening fluid

Young S. Lee[1], E. D. Wetzel[2] and N. J. Wagner

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This study reports the ballistic penetration performance of a composite material composed of woven Kevlar® fabric impregnated with a colloidal shear thickening fluid (silica particles (450 nm) dispersed in ethylene glycol). The impregnated Kevlar fabric yields a flexible, yet penetration resistant composite material. Fragment simulation projectile (FSP) ballistic penetration measurements at ~244 m/s have been performed to demonstrate the efficacy of the novel composite material.

The results demonstrate a significant enhancement in ballistic penetration resistance due to the addition of shear thickening fluid to the fabric, without any loss in material flexibility. Furthermore, under these ballistic test conditions, the impregnated fabric targets perform equivalently to neat fabric targets of equal areal density, while offering significantly less thickness and more material flexibility. The enhancement in ballistic performance is shown to be associated with the shear thickening response, and possible mechanisms of fabric-fluid interaction during ballistic impact are identified.

Nomex paper T-411 impregnated with adhesive varnish and its application Tian Jianzhong   Wang Jianhe
Nanjing Turbine & Electr. Machinery Group Co. Ltd.;

The authors show that adhesive Nomex paper 411 has operated for a long time in many rotor coils of generators. It is of stable quality, they are going to push this product as a new insulating material to the market. Nomex insulating paper 411 is a non-rolled smooth paper, this kind of paper is of heat shrinkage and easy moisture absorption type.

The paper will be extended both crosswise and longitudinally, so, it must be treated by heat-shrinkage before use, so as to avoid quality problems by shrinkage in application. Mechanical performance of non-rolled smooth paper Nomex 411 is not as good as 410, so the ideal applied state for adhesive Nomex paper is to treat it by heating extrusion and fixing in the structure, for instance, insulation between turns of magnetic pole coils in a synchronizer, so that it improves the mechanical strength

Aramid nanocomposites Properties

Typical properties include:

- Usually yellow in appearance
- Low density
- High strength
- Good impact resistance
- Good abrasion resistance
- Good chemical resistance
- Good resistance to thermal degradation
- Compressive strength similar to E-glass fibers
- Some grades of aramid fiber can degrade when exposed to ultraviolet light

Methodology

Equipment used

1. Vacuum assist Former Blue M
2. Digital scale METTLER AE 100
3. Universal Testing Machine,Instron Model 4467
4. Paint brushes(hand lay up)
5. 12” x 12” steel metal plates
6. A pair of scissors

Materials used

1. Nomex and Kevlar paper-(UON)
2. Fiber glass PU based resin
3. Nanocor 1.33M
4. MAS epoxy Resin #30-002
5. MAS Epoxy Hardener #30-532
6. Alcohol
7. Acetone (C_3H_6O), Acros Organics, CAS#67-64-1, Lot#B0506911, UN1090, viscosity 0.32 mPa s @ 20^oC
8. MEKPO catalyst

Experimental

Permeability samples

Nomex and Kevlar honeycombs are fire retardant, flexible, lightweight, and have good impact resistance. They offer the best strength to weight ratio of the core materials. Nomex 3T412 and Kevlar 2.8N636 aramid papers from DuPont were coated in 4 trials with unsaturated polyester mixed with 1.5% of Methyl Ethyl Ketone Peroxide (MEKPO), also 5% by wt. of acetone was added as a diluent. Resin was applied with brush or rollers to both sides of test samples and cured at room temperature in press between sheets of nylon films and metal plates or in vacuum-bag between peel plies. Helium permeability tests were performed on a specially built set-up with gaseous helium as a permeant using Alcatel ASM 142, an universal helium leak detector with usable helium sensitivity in the 10^{-6} Pa·cm^3/s range. The helium was pumped with stable pressure 6.9 and 34.5 MPa. The composite sample was a circular disk of 2.54 cm diameter with thickness measured in 4 points.

Tensile Testing Sample Preparation

1. Cut out the Kevlar and nomex papers to 10x10 inches from the big rolls
2. Weigh each piece of paper and record the weight
3. Cover the metal plates with FPO paper and tape the paper to the back of the metal plates with heat resistant tape
4. Place the cut Kevlar or Nomex paper on one of the plates ready for brushing
5. Weigh fiber glass resin (four times the weight of the paper)
6. Weigh 2% MEKPO catalyst
7. Using a stirring stick mix the catalyst and the resin.
8. Pour adequate amount of the mixture on to the paper surface and brush it on.
9. Flip the paper to the backside and repeat brushing pouring some more resin until all the paper is all covered with the resin
10. Place the other metal plate on top of the paper.
11. Wrap the metal plates with the bleeder cloth.
12. Envelop the plates in a nylon bag and seal it and apply vacuum
13. Put the vacuum bag into the oven at 200^{0}F for 1 hour

Results

After the test samples were prepared test bars were cut from each composition of the nanocomposites, a burn tests was performed to determine the flame resistance of the Prepared samples and tensile testing was done to determine the mechanical properties .The test specimens consisted of the following:

- pure Kevlar
- Kevlar paper impregnated with UP resin
- Kevlar impregnated with UP resin and 1gram of alcohol
- Kevlar impregnated with UP resin and 2 PHR of nanoclay
- Pure Nomex paper
- Nomex paper impregnated with UP resin
- Nomex paper impregnated with UP resin and 2 PHR nanoclay
- Nomex paper impregnated with UP resin and 4 PHR nanoclay

Horizontal test was performed these standards have certain criteria for each type of test, with the vertical having more detail. The horizontal test (94HB) shall not have a burning rate exceeding 1.5 inches per minute over a 3.0 inch span. The horizontal test showed that all of the different nanocomposites passed the 94 HB standards. For this test, all of the specimens showed excellent burn resistance, with the 2PHR clay content showing the best results. A Horizontal Burn Test was also conducted and the specimens would not burn to the designated marks, so all passed the 94 HB standards.

Table 1: Vertical Burn Test, Nomex

Material	Standards	Extinguishing time	Observations
Pure Nomex		41 sec.	Black smoke, charred
UP resin Impregnated Nomex		1 min 24sec.	Black smoke, charred
2 PHR		1min 32sec.	Black smoke, charred
4 PHR		1 min 51sec	Black smoke, charred

Table 2 Tensile results Kevlar

Material	Max load(lbf)	Tensile Stress@max load(ksi)	Tensile strain @max load (%)	Modulus (ksi)
Pure Kevlar	10.46	0.17	7.27	2.34
UP impregnated Kevlar	31.73	0.50	9.67	5.17
1g alcohol UP impregnated Kevlar	29.47	0.52	10.78	4.82

Table 3 :Tensile Results Nomex

Material	Max load(lbf)	Tensile Stress@max load(ksi)	Tensile strain @max load (%)	Modulus (ksi)
Pure Nomex	20.45	0.33	2.19	15.17
UP impregnated Nomex	42.04	0.67	1.79	37.43
2 PHR	31.73	0.51	4.13	12.35
4 PHR	53.31	0.85	1.74	48.85

Discussion of Results

Nomex T412 has low permeability without the resin coating, while Kevlar N635 paper is saturable and porous. As a consequence, resin will penetrate Kevlar paper, giving a non-

Porous material. Samples of Kevlar uncoated and coated in Trial 2 were very permeable to air and did not hold the vacuum during the test.

From the tensile test results Pure Nomex had the higher tensile strength of 15.18 ksi than Pure Kevlar that had a Tensile strength of 2.34 ksi.the results obtained on Kevlar were not consisted, the results show that addition of 1gram of alcohol to the matrix lowered the tensile strength of the samples.

Nomex 4% clay had the greatest Tensile strength of 48.85Ksi indicating that in order to obtain the greatest use of the Nomex n tensile strength 4% clay addition would be the suitable amount to use. 2% of nanoclay had lower tensile strength (12.35 ksi) than pure Nomex paper(15.17 ksi) .

The burn test showed that with increase in the % of nanoclay the paper took longer time to burn but all charred and yielded black smoke.

Conclusion

From the results obtained, the flame resistance of a material can really determine what type of applications they are suitable for. The aerospace and military industries could really benefit from various types of nanocomposite structures because they deal with fire and flame type situations. The more a material can resist flames, smoke, and damage, the better. Sometimes an improvement in one property can result in a drop in another, such as this experiment resistance. This study demonstrates the effects of nanoparticles on Kevlar and Nomex tensile strength and Flammability. From the above results a conclusion can be drawn in that addition of 4% nanoclay to the matrix increases the tensile strength of the paper. Addition of nanoparticles to Nomex paper increases the time the material takes to completely burn.

Acknowledgement

- Dr. Ibeh Christopher C, Director, CNCMM
- Andrey Beyle - Advisor
- Nsikakabasi Akpan
- Jennifer Muoghalu
- Praneetha Sanibasker

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Appendix

APPLICATIONS

POLICE, EMT, MILITARY USES and, FIREFIGHTER

These types of vests are used for tactical police units, such as S.W.A.T. Team units Nomex, a similar fiber to Kevlar is used as a fire retardant and protect firefighters from high temperatures. Military helmets today are much lighter and much stronger than helmets of the past. Helmets of the past consisted of metal. As you know metal can get heavy, especially after long durations of time. Kevlar is also five time stronger than metal. The new Kevlar helmets help keep our troops safe, and do not cause fatigue on their heads. Combat vehicles use composite panels made of Kevlar liners mounted to the inside of the vehicles structure. With the combination of other metals or ceramics, they greatly increase the survivability for the crew members. Kevlar does not melt or shrink when exposed to extreme heat. It is also extremely resistant to cuts, punctures, and chemical substances.

Many new canoe makers make their canoes entirely out of Kevlar. With Kevlar's resistance to puncture, it is a perfect marriage for water recreationists who don't want the hustle of bailing water out from a punctured boat. Some new tire manufactures are creating tires from Kevlar composite materials to form as "puncture proof" tires. The old fashion way of having puncture proof tires was to have solid rubber tires. The drawback from solid rubber tires was their incredible weight. Now with the new Kevlar technology the puncture proof tire idea can come to life and still be practical.

www.kevlar.com