Research and Education at the Center for Nanocomposites and Multifunctional Materials [CNCMM], Pittsburg State University

Christopher C. Ibeh[1,2], Monika Bubacz[2], Andrey Beyle[2], Stefano Bietto[2], Stan Scoville[2], Dilip Paul[3], Charles Blatchley[3]
1 – Professor, Department of Engineering Technology, PSU
2 – CNCMM, PSU
3 – Professor, Departments of Physics and Chemistry, PSU, Pittsburg, KS

Abstract

The Center for Nanocomposites and Multifunctional Materials [CNCMM] at Pittsburg State University, is an inter-institutional and multidisciplinary effort to implement the ONR-sponsored "Multifunctional Materials for Naval Structures" project. CNCMM's partners include the University of New Orleans, North Carolina A & T University and several companies. CNCMM's interdisciplinary efforts comprise of faculty and personnel from several departments and colleges at PSU, and provide a critically needed mechanism for expertly coordinated efforts of basic, applied and innovative research and education in the areas of nanocomposites and multifunctional materials for naval structures and homeland security. Specifically, activities of this project and the Center for Nanocomposites and Multifunctional Materials (CNCMM) are in five focus areas: fundamental and applied research on radar transparent, flame retardant, corrosion resistant, blast resistant/impact, and self-repairing advanced structural materials; nanoengineered sensors and innovative sensor technology; database development for nanocomposites and multifunctional materials; service as clearinghouse for academiagovernment-industry linkage and exchange of information on new technologies resulting from center's activities; and facilitation of a modern workforce and new industries via technology transfer. CNCMM provides a viable means of enhancing Kansas' relatively new nanocomposites industry by establishing an excellent research and education center for nanocomposites and multifunctional materials with focus on naval structures and homeland security issues.

Introduction

A major priority of CNCMM's effort is in the area of "Blast Damage Protection of Naval & Aero Structures and Components under Collision or Battle Conditions." Also of importance is the design of material structures for multifunctional characteristics. Hence the emphasis on the study of flame resistant materials; CNCMM acquired a "state-of-the art" Cone Calorimeter, FTT model for this thrust. The flammability resistance parameter is paramount in the design, development, formulation and study of blast resistantmaterials and structures when taken into consideration the fact that fire is inherently associated with blast conditions. Blasts predominantly manifest as shock waves. Shock wave effect[1][2], travels away from the explosion faster than the speed of sound. The arrival of a shock front at a location causes a sudden rise in the ambient pressure or overpressure. Associated with the overpressure are simultaneous pressures or dynamic pressures created by the blast winds. Figure I vividly depicts the rapid decay of overpressure and dynamic pressure from shock wave effect. The impact of shock wave on an object results in the absorption of much of the impact energy, and the amount absorbed determines whether the impacted object will be ignited. Hence the need for flammability resistance in blast resistant-materials and structures. In keeping with its stated objectives, the Center for Nanocomposites and Multifunctional Materials [CNCMM] is developing a multi-institutional and interdisciplinary infrastructure for designing and creating new nano-systems capable of meeting the future materials challenges that the Navy and related industries are facing. Ultimately, the essence is to develop intellectual capital via collaborative and cooperative inter-disciplinary research, education and knowledge transfer. CNCMM research efforts are complemented by its education and workforce development thrusts. CNCMM plans to develop an interdisciplinary, graduate level materials science and technology program with emphases in nanocomposites, computer proficiency and nanotechnology entrepreneurship.

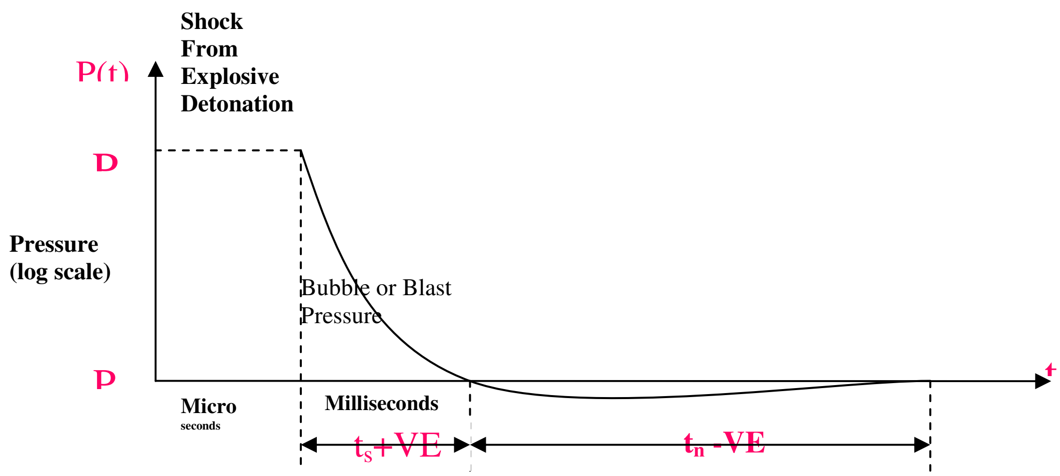


Figure I: Underwater Shock and Blast Pressures

The objectives and activities of the "Multifunctional Materials For Naval Structures" project and the Center for Nanocomposites and Multifunctional Materials (CNCMM) are in five focus areas: i. fundamental and applied research on blast resistant, radar transparent, flame retardant, corrosion resistant, and self-repairing advanced structural materials, ii. nano-engineered sensors and innovative sensor technology, iii. database development for nanocomposites and multifunctional materials, iv. service as clearinghouse for academia-government-industry linkage and exchange of information on new technologies resulting from center's activities, and v. facilitation of a modern workforce and new industries via technology transfer . Developments and innovations in these five focus areas are of vital importance to the shipbuilding, national defense, homeland security and Kansas industries.

CNCMM'S Research

CNCMM's research efforts is in three major areas:

1. Energy Dissipative, Blast Resistant Polymeric Sandwich Structures for Naval Applications, 2. Nanoparticles Synthesis via the Modified Sol-Gel Method, and 3. Development of Nano-engineered Sensors.

1. Energy Absorbing, Blast Resistant Polymeric Sandwich Structures

Problem Statement

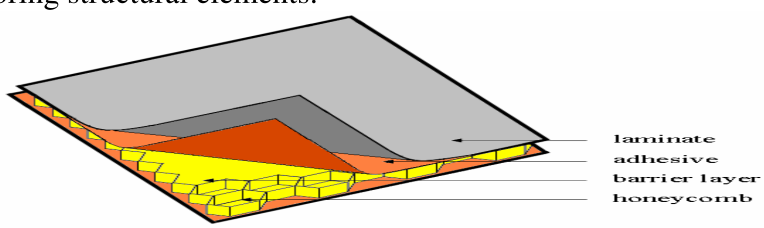
Naval and defense industries' interest in the development of structures that enable safety of personnel and systems in extreme attack threats is a current trend. As such one of the current top priorities of researchers, scientists, engineers and technologists is the creation of structures able to sustain heavy loads derived from blast. These blast resistant structures must withstand high stresses produced by shock waves, sudden increase in static pressure and ballistic impacts. Blast wave (dynamic pressure) and overpressure are usually modeled as distributed loads and concern both structural design and single panel stiffness. When a structure incurs overpressure, panels and beams are forced to bend to equilibrate the pressure gradient between external and internal conditions, with the possible consequence of catastrophic collapse. Rigid materials with high flexural stiffness can contribute positively to the overall safety of the structure in this case. Another dangerous hazard produced by blast is high speed flying objects of various dimensions, which can sensibly weaken the structure, perforate the external shell or locally induce buckling. In this scenario, new materials able to absorb high energy impacts have a fundamental role. Polymeric composite materials have been studied for years showing excellent impact characteristics; these polymeric and foam materials, in general, exhibit impact energy absorption and dissipation properties. Bulletproof structures development efforts indicate that high static strength of materials do not translate to high dynamic resistance. Only combinations of high strength (but brittle) materials, such as ceramic and strong metals, with high-stiffness and high-strength, viscoelastic polymeric fibers give good effects. The combination of damping effect from blast/impact energy absorption, overall structural stiffness and strength, and flammability resistance is the key in the design and fabrication of blast resistant structures.

Blast Resistant Polymeric Sandwich Structures

Polymeric sandwich structures provide a suitable solution for resisting impact loads and bending. Sandwich structures are popular due to their high bending stiffness per unit of mass [specific stiffness]. However, their resistance to transversal loads and shear loads is relatively low. Transversal loads due to blasts are high, and classical sandwich structures become very vulnerable. The ehancement of transversal static and dynamic resistance of sandwich structures is one of the main goals of this research effort. A new concept of high resistance sandwich structures include new design of face sheets and core materials, where Kevlar® honeycomb and viscoelastic foams are incorporated to form incredibly responsive structures. In addition, a new class of reinforcement fibers made of ultra high molecular weight polyethylene (UHMWPE) show extremely promising properties in terms of energy dissipation. Kevlar® fibers are light (about 1.3-1.4 g/cm^3), have good combination of high strength, high stiffness, and they have non-linear creep, which takes place only when stresses are high. This creep provides energy dissipation at impact loading. UHMWPE fibers have low density (about 0.95-0.98 g/cm^3), high specific strength and stiffness. However, their properties decrease rapidly with increase in temperature, and these fibers tend to experience a more linear creep at relatively low static loads. Mechanical and physical properties of UHMWPE fibers are comparable with commonly used reinforcement, such as carbon and glass fibers. To improve the bonding between this class of fibers and resin matrix systems, such as epoxy, polyester and vinyl ester, a surface treatment is usually required. Other advantages are resistance to water/moisture related effects and it is light weight relative to water, which make the use of UHMWPE fibers a material of choice in marine industry.

Material Configurations and Design -- Figure II: Conceptual, Basic Design of Blast Resistant Sandwich Material

The blast resistant material is a sandwich structure with honeycomb core. The facesheets are composites made of Marine epoxy resin [unsaturated polyester resin, vinyl ester for cost-effectiveness, and polyimides for strength and flammability resistance] with addition of nanoparticles (nanoclay, SiC, CNF), with glass and aramid fibers reinforcements. The core is of aramid/or carbon honeycomb filled with viscoelastic foam and/or polymeric nanocomposites. Different concentration of aramid fibers, nanoparticles and glass fibers are currently being tested. A gel spinning process for the manufacture low cost UHMWPE fibers is being initiated at CNCMM, and the properties will be evaluated. High blast resistance can be achieved if material and structure have high stiffness, high strength, high ability of energy dissipation and high resistance to crack propagation (especially to major cracks). This combination of properties is typically not available in one material, and it is necessary to combine several materials. Effective combination of materials depends on architecture of combined structure. Proposed hierarchical structures include 3-D and 2-D reinforcement, hierarchical core, and reinforced bond. The purpose of 3-D elements is to provide resistance to major crack propagation, the purpose of 2-D elements is to stop dynamic movement of structure after achieving some allowable displacements, and the purpose of a hierarchical core is to dissipate main blast energy and to redistribute damped loads on neighboring structural elements.



Experimental and Technical Approach

Processing and fabrication involve validation of each process technology such as materials and components cutting technology [laser, water jet, diamond knife etc], fabrication technology [compression molding, VARTM, VAHLUP] parameters (processing window). Characterization efforts involve morphology determination [AFM, TEM, DSC, etc], static and dynamic mechanical measurements of stress-strain properties with emphases on structural stiffness, dimensional stability, toughness etc.[universal testing machine], and flammability resistance [cone calorimetry (FTT), horizontal and vertical burn tests].

Results and Discussion of Results

Preliminary results indicate that nanocomposites-filled honeycombs have very good potential as sandwich cores for enhanced mechanical properties [Figures II and III] and are very good prospects for energy dissipation and blast mitigation. With respect to low density foam-filled honeycombs, the summation rules for strength and for Young's modulus apply. At higher density of foam, this rule does not apply, and a sharp synergistic effect is observed. This synergy occurs as the foam carries part of the total compressive load in addition to other mechanical functions such as: i. support of the walls of honeycombs as an elastic foundation and increasing their critical buckling load, and (ii). formation of quasi-monolithic layers on the walls of honeycombs, and

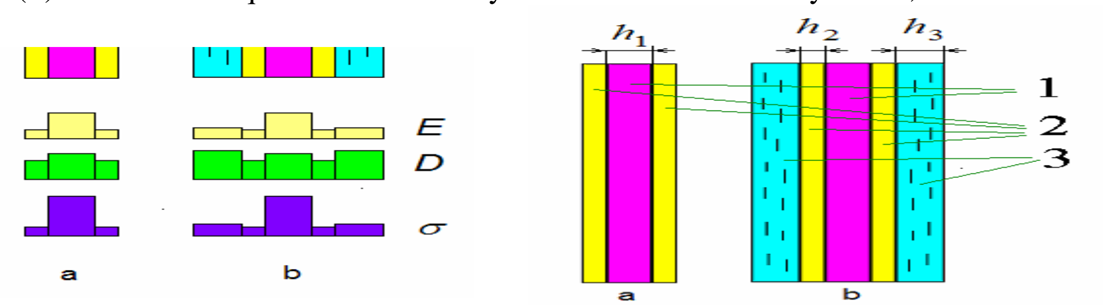


Figure III: Schematic Structure Honeycomb Wall: (a) Stabilized Wall of Conventional Honeycomb; (b). CNCMM Modified Honeycomb Wall[1][2];1= Impregnated Aramid Paper; 2 = Excess of Polymer; 3 = Additional Layer from Nano-filled Polymer; E = Distribution of Young's Modulus; D = Bending Stiffness; σ = Strength through the Wall Thickness of Honeycombs.

conversion of the walls' structure to a sandwich-like structure with increase in bending stiffness and resistance to buckling of the walls. Modeling of the elastic supporting effect by foam shows that the compressive strength of the foam-filled honeycombs can be estimated according to the formula[4]:

$$\left|\left\langle \Pi_{3(-)}^{(h)} \right\rangle\right| = K \frac{\pi^2 D_2}{b^2 h^{(w)}} c \quad \ldots\ldots\ldots\ldots (1)$$

Where b is the size of the honeycomb cell, $h^{(w)}$ is the height of the honeycomb wall (thickness of the core), D_2 is the honeycomb wall's bending stiffness in the plane perpendicular to the core and to the wall, K is a coefficient that depends on the interaction

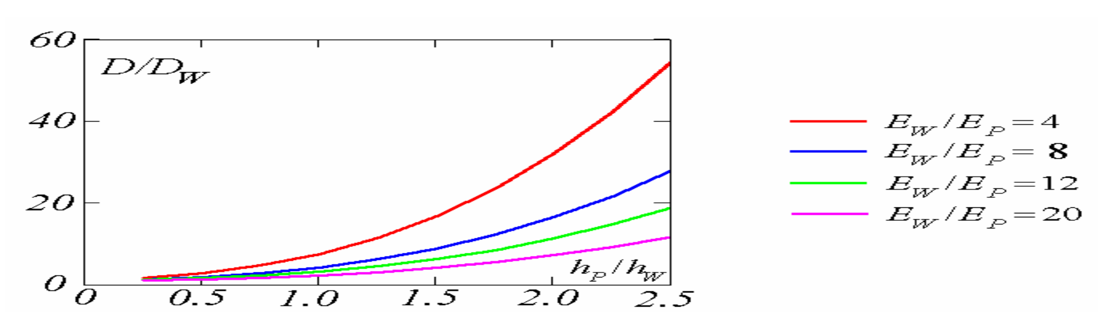


Figure IV: Dependencies of Relative Bending Stiffness of Honeycomb Wall on Ratio of Thickness of Quasi-monolithic Polymer Layer to Wall Thickness for Different Ratios of Young's Modulus of Wall Material to Polymer's Young modulus[1][2].

Property\Material	***Honeycomb***	***Honeycomb with foam*** / ***Foam 2***	***Honeycomb with foam*** / ***Foam 4***	***Honeycomb with foam*** / ***Foam 10***
Density (kg/m^3)	94.2 (2.83%)	138.4 (2.21%) / 50 (13.4%)	161.7 (2.34%) / 68.9 (5.32%)	204.7 (5.84%) / 90.9 (5.94%)
Compressive strength (MPa)	4.55 (6.45%)	4.90 (7.0%) / 0.345 (26.5%)	4.96 (10.0%) / 0.483 (23.0%)	7.03 (5.0%) / 0.90 (8.0%)
Young modulus (MPa)	39.8 (2.52%)	45.3 (7.0%) / 2.42 (35.6%)	48.6 (12.0%) / 2.63 (41.0%)	101.7 (8.0%) / 13.5 (32%)

Table 1: Mechanical Properties of Honeycombs, Foams, and Foam-filled Honeycombs[1][2] with the Coefficient of Variation for each Property in Bracket.

with the neighboring wall and on the Young modulus of the foam; *c* is the volume part occupied by the walls of the honeycombs. Successful project implementation[3][4] involves validation of materials' morphological and mechanical characteristics with respect to compressive, tensile and shear stress-strain profiles with respect to Figure IV.

2. Nanoparticles Synthesis via the Modified Sol-Gel [Aerogel] Method

The objectives of the "Nanoparticles Synthesis Program (NSP)" are to synthesize and characterize nanonoparticles by a wide selection of metal precursors and proper solvent removal techniques with emphasis on the preparation of high surface area and highly reactive nanoparticles. This effort complements the "Energy Dissipative, Blast Resistant Polymeric Sandwich Structures for Naval Applications" research project.

Technical Approach

Synthesized nanoparticles are characterized by different physical and chemical methods such as powder x-ray diffraction, surface area measurements, transmission electron microscopy, and hydroxyl/carbonate species determination via FT-IR. Drying and calcination play an important role in affecting the physical and chemical properties of nanoparticles, hence controlled dehydroxylation and calcination will be carried out using a combination of FT-IR and mass spectroscopic techniques. The photochemical reactivity of these semiconductor mnanoparyticles will be investigated at a molecular level using infrared and mass spectroscopic techniques. Initially, simple molecules with different functional groups will be chosen and their reactivity towards these semiconductors will be observed by careful monitoring of their vibrational frequencies and different mass fragments. The plan is to extend these ideas to complex molecules which are often used as surrogates of chemical or biological warfare[10][11].

Results & Discussion of Results

Table II Shows Higher Surface Area for In-Lab AP Nanoparticles.Reactions of acetaldehyde on 2 mixed metal oxide nanoparticle surfaces that were studied using in-situ FTIR and mass spectroscopy. Mixed metal oxides, 2.5%Sb-2%Cr-SrTiO3 and 2%Cr-SrTiO3 produced in the labs of Prof. K. Klabunde, KSU are found to absorb acetaldehyde via irreversible mechanisms involving surface hydroxyl groups. The Nanoparticles Synthesis Program (NSP) techniques are currently being refined such that significant modifications of the physical and chemical properties of the nanoparticles can be achieved. This will facilitate formulation and preparation of nanocomposites with desired properties. The thermal and photochemical reactitivity of these nanomaterials will be expanded to more compounds with environmental concerns and of DOD's interest. A significant molecular level understanding of synthesis of nanomaterial along with their thermal and photochemical reactions with compounds of interest will be achieved. A Parr reactor is being acquired to facilitate in-lab synthesis of these nanoparticles at PSU.

3. Development of Nano-engineered Sensors via Fast Luminescent Silicon.

This research effort involves the use of mesoporous silica materials, in film form which contain stabilized clusters of silicon atoms with a size of 2 nanometers or less and, because of their thin size, may be embedded into matrix composites without causing

- **Table II: Textural Properties of Mixed Metal Oxide Nanoparticles**

$SrTiO_3$ Sample	Crystallite Size (nm)	Surface Area (m^2/g)	Total Pore Volume (cc/g)	Avg Pore Size (d), Å
CM-$SrTiO_3$ S	145	1.0	0.003	93
NCM-$SrTiO_3$	25	17	0.12	290
$SrTiO_3$ (methanol)	25	82	0.58	280
$SrTiO_3$ (ethanol)	8	159	0.62	150
AP-$SrTiO_3$ (isopropanol)	20	121	0.59	190

*** CM – commercial; NCM – commercial nanosized, AP – aerogel process samples**

significant effects to the elastic modulus or strength of the matrix. They are capable of electroluminescence when the appropriate current or voltage is applied, emitting fast photons with nanosecond lifetimes [12]. Electroluminescence, as well as photoluminescence, has been demonstrated in many devices by the use of porous silicon, which can reach 5% quantum efficiency, but has serious problems with respect to fragility, short degradation times and integration into microelectronic circuitry [13]. Also the large size of the silicon units in porous silicon(2-50 nanometers) produce emission times in the millisecond to microsecond range, which is too slow for use in LED's [14]. However by producing silicon clusters of 2 nanometers or less, significantly different electronic properties are achieved, in terms of significantly faster photons for photoluminescence, electroluminescence and photoelectronic applications.

Technical Approach

This research effort produces silicon nanoclusters of 2 nm or less which would, in terms of porous solids, place them between microporous (pore width up to 2nm) and mesoporous (pore width from 2 to 50nm). Techniques employed involve dehydration at 100-150^{o} C.for 5 hours followed by addition of disilane and then chemical vacuum deposition onto the mesoporous film at 100-140 ^{o}C for 40 hours for the production of silicon nanoclusters [12]. The silicon nanoclusters are overlaid by an optically transparent electrode of a thin gold layer and placed onto a metal electrode, and appropriate millivolt levels of electricity are applied to test for electroluminescence. Subsequently, nanosensors will be embedded in the matrix of nanocomposites being produced for improved blast resistance. It will then be tested, as the nanocomposite is stressed or heated and its use as a nanosensor for detecting damage in hulls of ships or subs, and to kevlar or some material similar to military body armor .

CNCMM Education and Workforce Development Activities

Objectives

CNCMM's education activities enhance the materials science and technology knowledge base of PSU. The student participants of this project are provided the opportunity to develop academically by making available at the graduate level courses in the materials area with emphasis on nanocomposites and multifunctional materials. During the first two years of this project, a committee of dedicated PSU faculty worked on the development of a materials science and technology curriculum and program. This involved a thorough survey and evaluation of available materials science and technology programs nationwide. The committee's work identified already available, eligible faculty and courses at PSU. New courses are being developed in identified deficient areas. The materials science and technology program committee is researching the requirements for viability of the program at PSU.

Approach

A committee of dedicated PSU faculty, the graduate curriculum committee [GCC] is working on the development of a materials science and technology curriculum and program."

Program Timeline
2005-06

1. documentation of the results of the survey and evaluation of "materials science and technology" programs nationwide,

2. identification and documentation of already available, eligible faculty and courses at PSU,
3. development of recruitment and selection protocol for prospective students of the graduate "materials science and technology" program.

2006-07
1. design of a graduate "materials science and technology" curriculum,
2. designation of courses that can be used for "nanocomposites" emphasis by project-supported graduate students in the MS and MET programs, and
3. development of a viable strategy for securing authorization for a graduate program in "materials science and technology."

2007-08
1. develop a viable strategy for securing authorization for a graduate program in "materials science and technology."
2. attainment of authorization for a graduate "materials science and technology" program,
3. recruitment and selection of graduate students,
4. develop a strategy to sustain funding for the center for nanocomposites and multifunctional materials (CNCMM), and the graduate "materials science and technology" program, and
5. provision for adequate faculty level to staff the graduate "materials science and technology" program,

2008-10
1. implementation of the "materials science and technology" program,
2. sustainability of efforts for funding of the Center for Nanocomposites and Multifunctional Materials (CNCMM), and the graduate "materials science and technology" program, and
3. development of a strategy for enhancing career opportunities for graduates of the "materials science and technology" program.

Results and Discussion of Results

Concurrently, CNCMM is implementing several research and education programs involving students at various levels of academia. The REACH-RS [Research and Education Academy for Coaching/Mentoring High School – Rising Stars] program invites talented and interested K-12 students to participate in a 2-week long summer program. By mentoring "high school—rising stars" in the science, mathematics, engineering and technology (SMET) of materials/nanocomposites and processing research, CNCMM hopes to encourage them to choose careers in the areas of materials and processing. "Materials and processing" are common grounds for most industries, and the bases for productivity. "Nanomaterials and nanotechnology" are current trends.

REACH-RS is a hands-on oriented program, and provides the students preview and introduction to college, improvement of communication and inter-personal skills, acquisition of technical proficiency in the areas of materials and processing, awareness of the impact of nanotechnology and nanocomposites in industry and society, development of simple, cost-effective laboratory experiments (teachers), enhancement of research and development skills, and development of entrepreneurial skills.

Several undergraduate and graduate students are sponsored year-long by CNCMM, and are required to participate in CNCMM's research, education, ethics, entrepreneurship, assessment and dissemination (RE^3AD) program[26].

Conclusions

CNCMM is a viable research and education unit at Pittsburg State University that is positioned to enhance Kansas' relatively new nanocomposites industry by establishing an excellent research and education center for nanocomposites and multifunctional materials with focus on naval structures and homeland security issues.

CNCMM will have a significant impact on Kansas' economy mainly through its collaborations and partnerships with companies in Kansas or companies that have affiliations in Kansas, and also through its undergraduate/graduate nanocomposites educational and training initiatives.

CNCMM's major priority effort is in the area of "Blast/Ballistic Damage Protection of Naval & Aero Structures and Components under Collision or Battle Conditions." This effort is complemented by the flammability resistance studies that have been initiated by CNCMM, and its academia-industry collaborations and partnerships.

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