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Nanomaterials at Zyvex:
From Conception to Consumer Products in Less Than Three Years

John Randall, Ph.D.
Chief Technical Officer
Zyvex Corporation

jrandall@zyvex.com

Outline

- Zyvex Introduction
- Zyvex’s CNT Technology
- Commercialization of Nanotechnology

Corporate Introduction

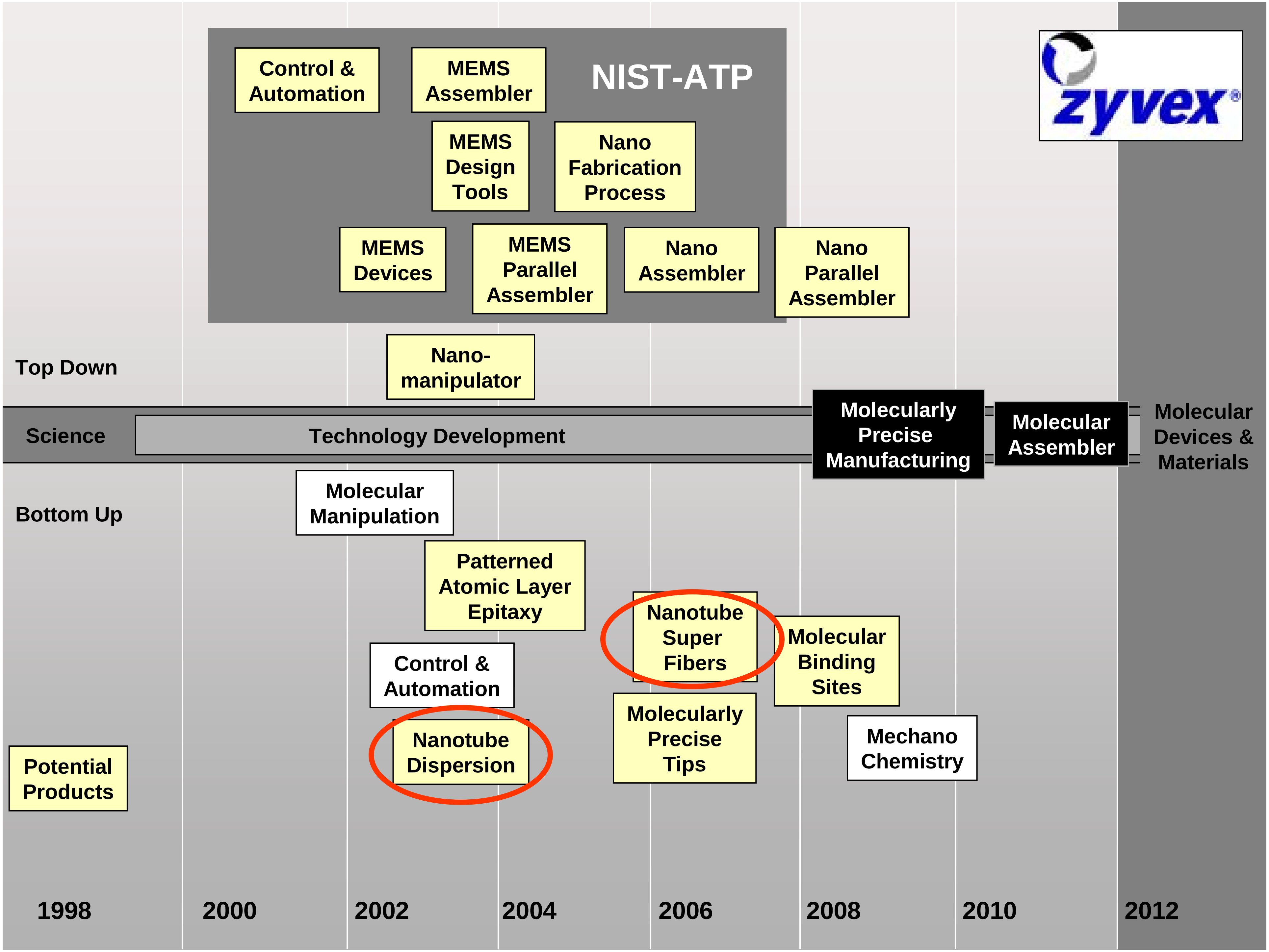
- Privately held company; Founded in 1997
- Located in Richardson, Texas
- Detailed Five-Year
  Strategic Business and Marketing plans
- Detailed Technology Roadmap
- Aggressive IP strategy
- Extensive list of scientific and business publications

Resources

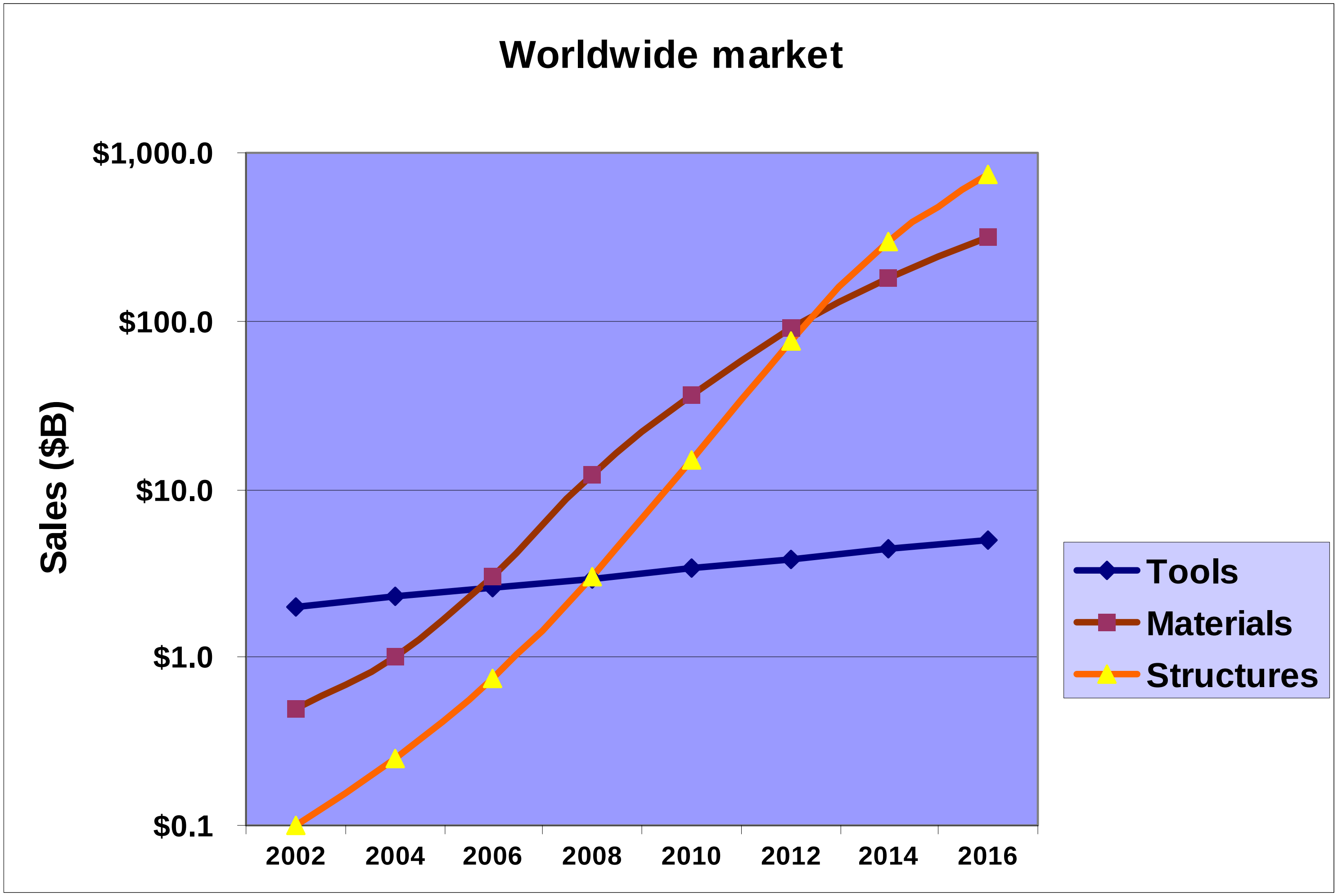
- **Headcount: 61 and increasing**
  - 17 Ph.D.s
  - Engineers, scientists, technical management
- **44,000 sq. ft. facility**
- **Class 1000 clean room**
- **Several fully equipped laboratories**
- **CNC-equipped machine shop**
- **Equipment: SEMs, TEMs, AFMs, UHV-STM, numerous lasers, MEMS motion analyzer, electrical testing station**

Our Vision

To be the leading worldwide supplier of tools, products, and services that enable adaptable, affordable, and molecularly precise manufacturing.


NIST-ATP
Control & Automation
MEMS Assembler
MEMS Design Tools
Nano Fabrication Process
MEMS Devices
MEMS Parallel Assembler
Nano Assembler
Nano Parallel Assembler
zyvex
Top Down
Nano-manipulator
Science
Technology Development
Molecularly Precise Manufacturing
Molecular Assembler
Molecular Devices & Materials
Bottom Up
Molecular Manipulation
Patterned Atomic Layer Epitaxy
Control & Automation
Nanotube Super Fibers
Molecular Binding Sites
Nanotube Dispersion
Molecularly Precise Tips
Mechano Chemistry
Potential Products
1998
2000
2002
2004
2006
2008
2010
2012

Tools, Materials, & Structures



Products

- Tools - NanoWorks™
  - Nanomanipulators
    - S100 (SEM), F100 (FIB), A100, L100
  - Accessories
    - NanoEffector™ Probes and Microgrippers

- Materials -NanoSolve™
  - Additives
  - Bulk Material Concentrates
  - Finished Goods

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Customers

Partial List

EASTON

BOEING

Texas Instruments

DARPA

intel

Dow

hp

FIAT

RICE UNIVERSITY

AMD

Honeywell

TEXAS SCOTTISH RITE HOSPITAL

massachusetts institute of technology

Schick

IBM

Harvard University

NPL National Physical Laboratory

HARRIS

Raytheon

Texas A&M UNIVERSITY

UNIVERSITY OF MARYLAND

Sandia National Laboratories

PENNSTATE

NIST National Institute of Standards and Technology

CYPRESS

NJIT NEW JERSEY INSTITUTE OF TECHNOLOGY

BAE SYSTEMS

UTD

UNIVERSITY of LOUISVILLE

BROWN UNIVERSITY

energy.gov U.S. DEPARTMENT OF ENERGY

Princeton University

tsmc

FLORIDA STATE UNIVERSITY 1851

Boston Scientific

Drexel UNIVERSITY

Alliances

Partial List

FROST & SULLIVAN

NASA

Honeywell

RICE UNIVERSITY

ARKEMA

KEITHLEY

COVENTOR

TEXAS SCOTTISH RITE HOSPITAL

Kellogg School of Management

SouthWest NanoTechnologies

MichiganTech

HITACHI Inspire the Next

ASI

MITSloan MANAGEMENT

Washington University Saint Louis

Texas A&M UNIVERSITY

Pyrograf Products Inc.

XEI Scientific

Thomas Swan & Co

UNIVERSITY OF MICHIGAN

Carnegie Mellon

THE UNIVERSITY OF TEXAS AT AUSTIN

UTD

BSAC Berkeley Sensor & Actuator Center

UNIVERSITY of VIRGINIA

NORTHWESTERN UNIVERSITY

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Press

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Forbes

abc WORLD NEWS TONIGHT WITH PETER JENNINGS

Discovery CHANNEL

NATIONAL GEOGRAPHIC

USA TODAY

c|net

TLC LIFEUNSCRIPTED

SCIENTIFIC AMERICAN

The New York Times

smalltimes BIG NEWS IN SMALL TECH.

electronic design

FOX 4

EETIMES

IEEE SPECTRUM

HISTORY CHANNEL.COM THE BEST SEARCH IN HISTORY

Dallas Business Journal

D

The Dallas Morning News

Government Outreach


U.S. Senator George Allen


21st Century Nanotechnology R&D Act


Presidential Advisor Karl Rove


Vice President Dick Cheney

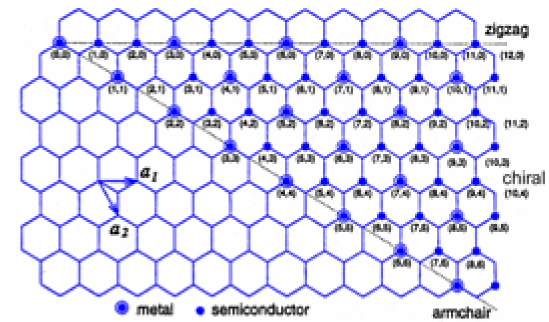

President George W. Bush


Senate Majority Leader Bill Frist

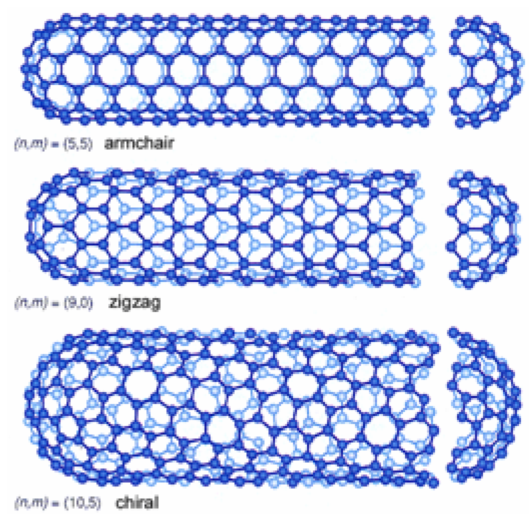
Acknowledgements

- Dr. Hassan Ait-Haddou
- Gopinath Balasumramaniyam
- Dr. Mark Banash
- Robert Folaron
- Tanja Kmecko
- Dr. Pritesh Patel
- Dr. Nagesh Potluri
- Marni Rutkofsky
- Dr. George Skidmore

CNT Background



Graphite



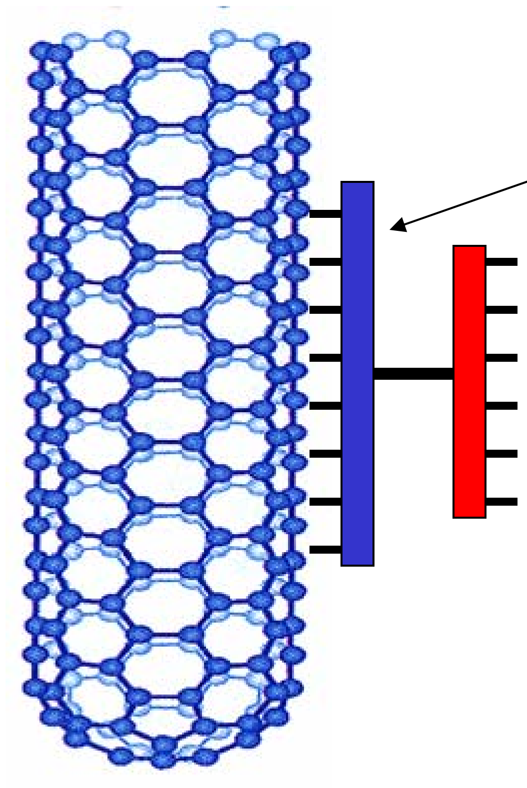
Single-Walled Carbon Nanotubes

Multi-Walled Carbon Nanotubes

Carbon Nanotubes

- Remarkable properties
  - — 20 times the strength of steel, 1/6th the density
  - — Better electrical conductivity than copper
  - — Thermal conductivity comparable to diamond
- What is the catch?
  - — Hydrophobic
  - — Insoluble
  - — Forms aggregates

Core Competence - Functionalizing



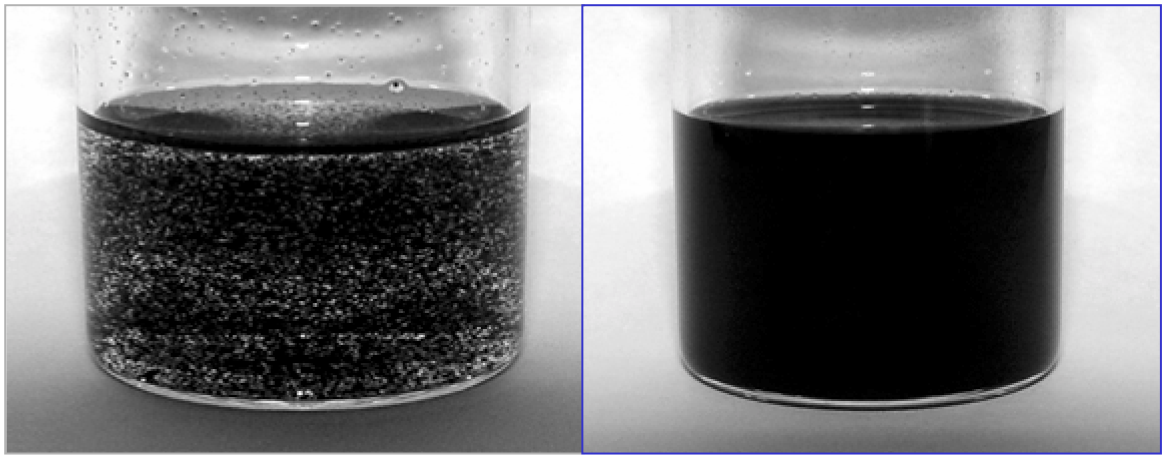
Functionalization technology

- **Two distinct functions:**
  - Non-damaging binding
  - Customizable
- **Binding applicable to CNMs (SWNTs, MWNTs, CNFs)**
- **Functionality may be customized for different applications:**
  - Dispersion in solvents
  - Adhesion to composites
  - Other chemical functionality

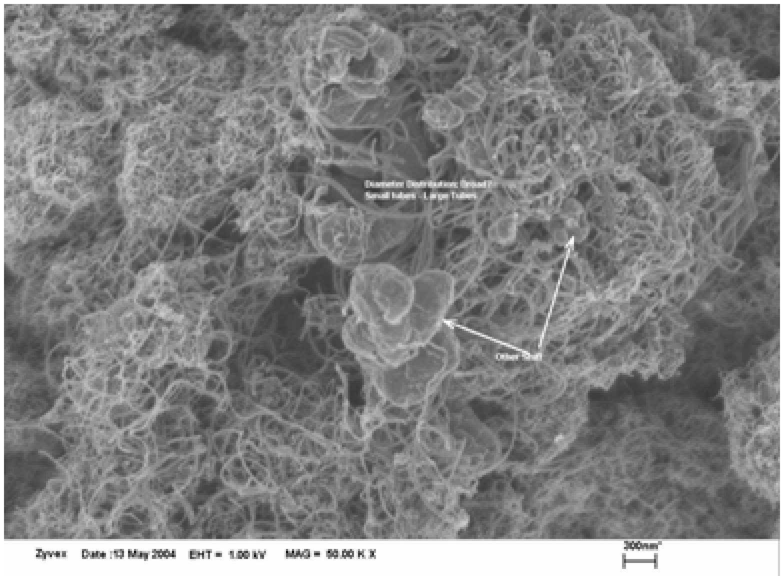
Current Capabilities

- Functionalize and solubilize CNMs
  - Solvents
    - Chloroform, chlorobenzene, methylene chloride, tetrachloroethylene, dichloroethane, THF, toluene, xylene, MEK, Isopropanol
  - Water
- Dispersion in composite matrices:
  - Thermoplastics and Thermosets

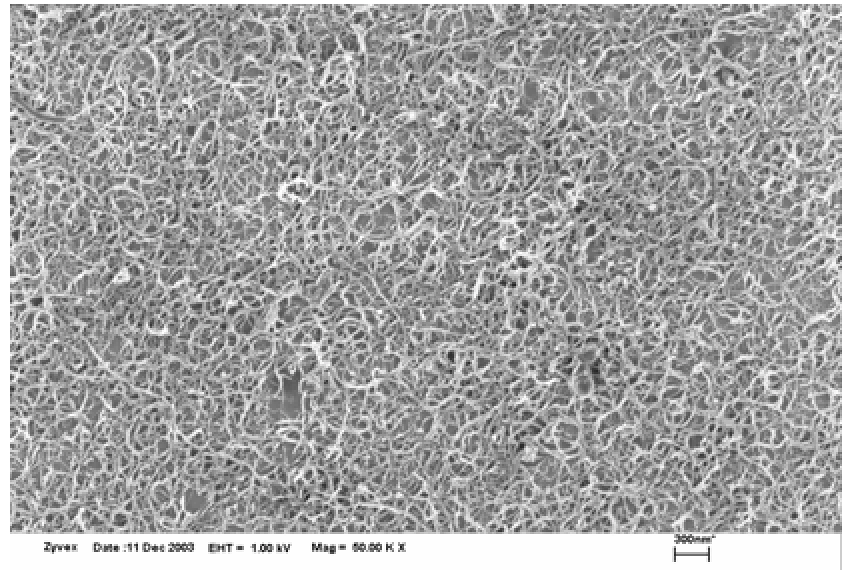
Poor vs. Good Dispersion/PU



Exfoliation & Dispersion with Kentera™



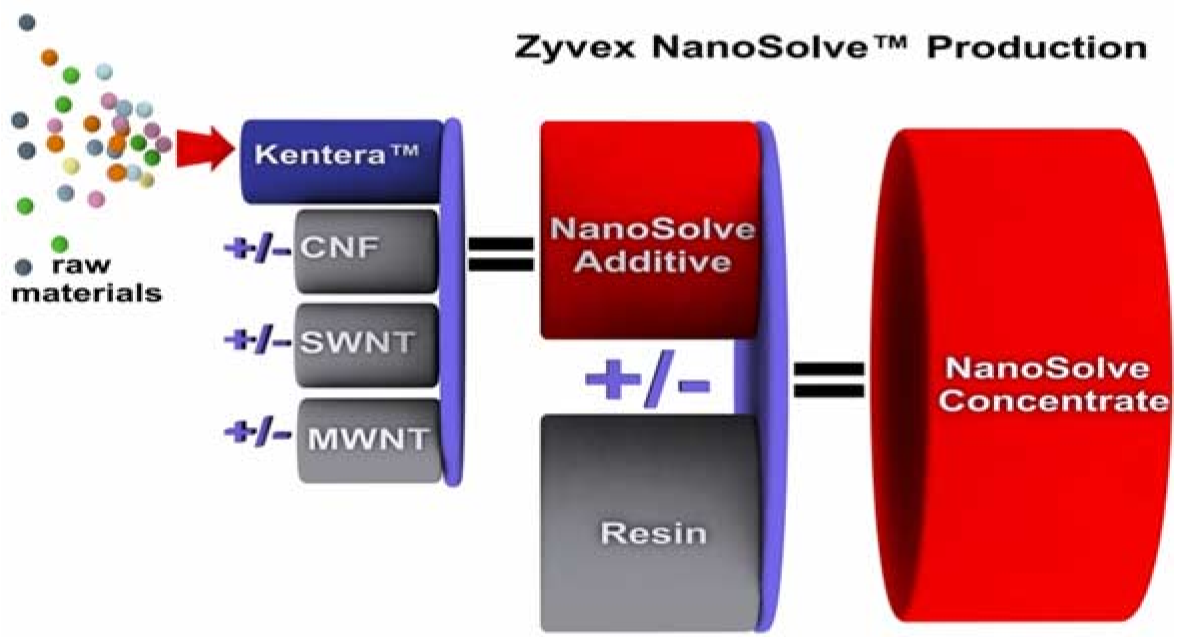
Raw Tubes



Kentera Treated Tubes

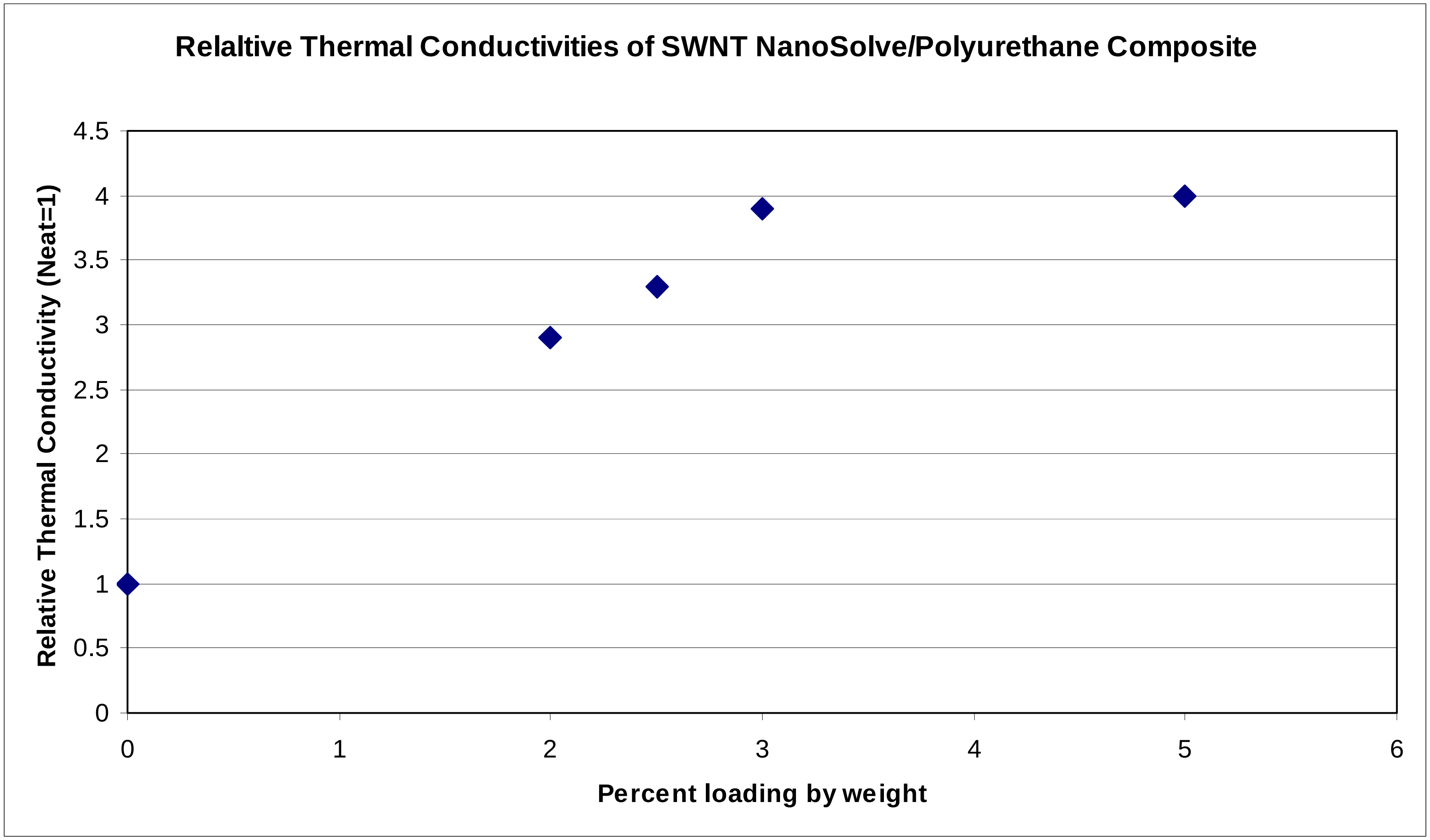
- 50KX Magnification
- Equal Concentrations
- Mutli-wall CNTs

Zyvex Nanosolve™

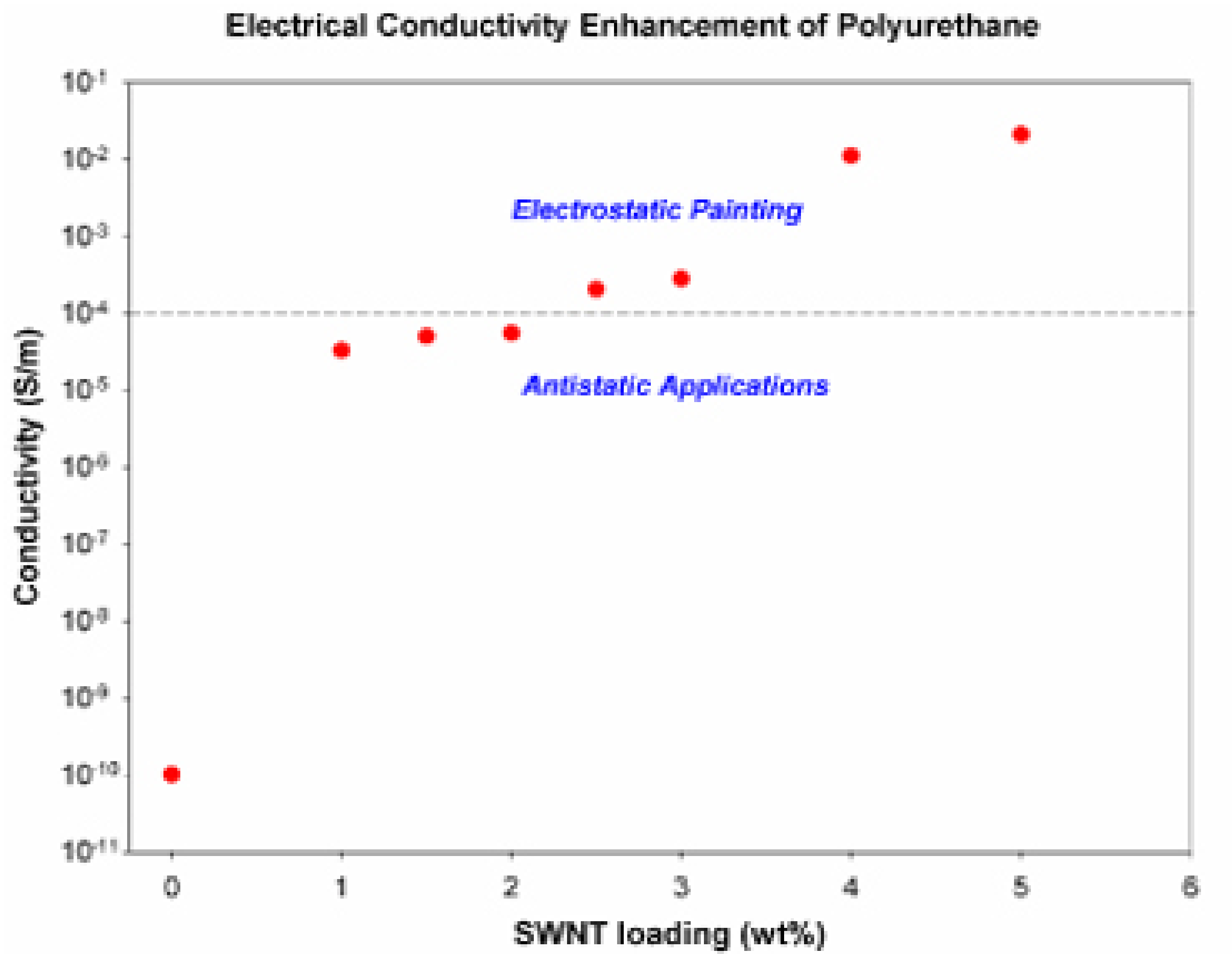


Thermal Conductivity

CNTs have extremely high thermal conductivities > 2000 W/m- degree K for some SWNTs

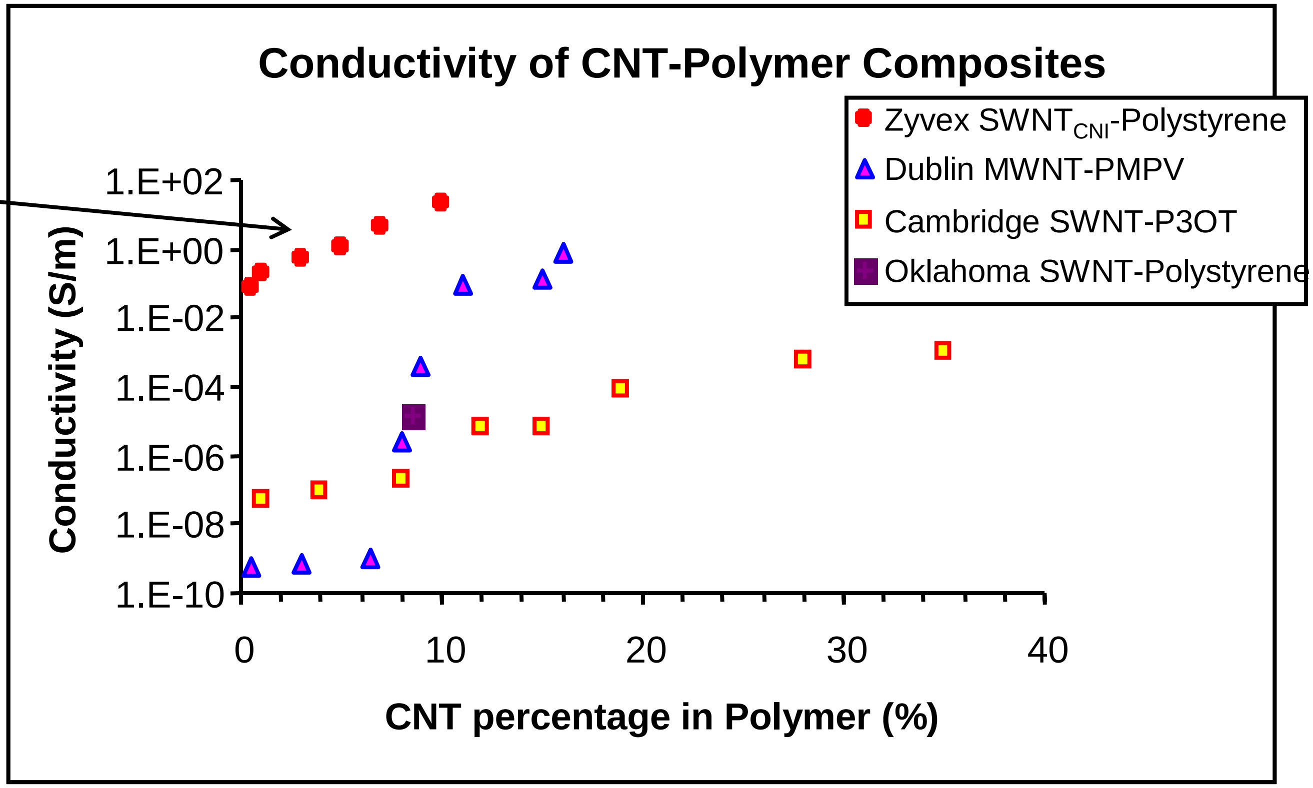


Electrical Conductivity of Polyurethane



Electrical Conductivity

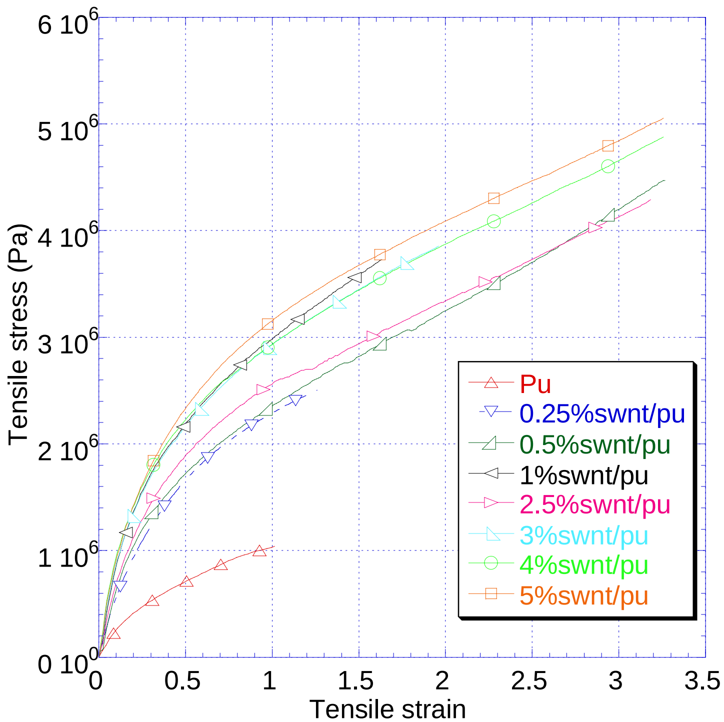
Conductivity as an indicator for CNT dispersion



Dispersed SWNTs significantly increase the electrical conductivity

SWNT/ Polyurethane Tensile Modulus, Yield Stress, and Fracture Stress

- Solids Analyzer RSA-3. at 0.25%, 0.5%, 1%, 2.5%, 3%, 4%, 5% wt percent to the pre-polymer. 5%SWNT/PU are increased by 298% and 183% respectively compared to the neat PU



Composition	Tensile modulus of elasticity (MPa)	Yield stress (Pa)	Fracture stress (MPa)	Strain at the max stress (%)
Polyurethane	2.8863	210700	1.04	101.44
0.25%swnt/PU	6.6538	332690	2.5036	126
0.5%swnt/PU	8.4849	398790	4.47	326.63+
1% swnt/PU	9.249	406956	4.29	318
2.5%swnt/PU	10.563	528150	3.73	163.39
3% swnt/PU	11.359	545232	3.8526	196.27
4%swnt/PU	11.495	586245	4.8785	325.65+
5%swnt/PU	11.485	597220	5.054	325.65+

Data courtesy of Prof. Nandika D'Souza Univ. of North Texas

Polycarbonate Property Enhancements

- Polycarbonate – Mechanical Properties of Cast Films (Lexan 101) at 3wt% MWNT Loading
  - 100% Improvement in tensile strength (~66MPa vs. ~32MPa)
  - 200%+ Improvement in Young's Modulus (~1.8GPa vs. .7GPa)
  - One order of magnitude - Improvement in Elongation to Break (65% vs. 10%)
- Polycarbonate – Electrical Properties of Cast Films (Lexan 101) at 1wt% CNT Loading
  - 5 S/m SWNT
  - 0.8 S/m MWNT

Epoxy Case Study
Property Enhancements

- Epoxy – Epon 828 with only 1 wt% CNT loading in uni-directional carbon fiber laminates
  - 5-10% improvement in tensile strength, modulus, short beam shear, flex modulus
  - 13-15% improvement in 3 point bend flex strength

Hierarchical Composites Compromising Continuous Carbon Nanotube Composite Fibers in a Nanotube-Reinforced Matrix

NASA SBIR Phase II (599k)

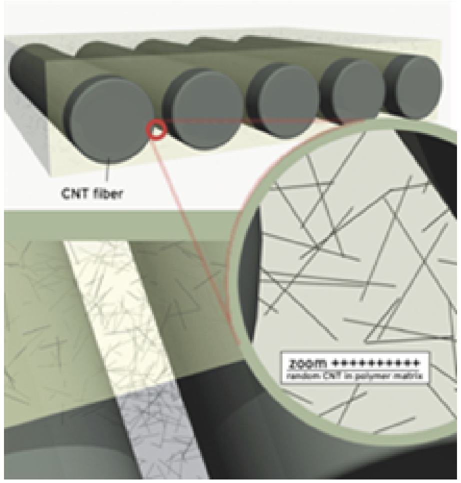
PI: Gopi Balasubramaniyam,, Zyvex Corporation

Identification and Significance of Innovation

The Zyvex/UTD team will develop technology to fabricate hierarchical composites of continuous Carbon Nanotube (CNT) composite fibers in a CNT-reinforced matrix.

The program builds on recent breakthroughs:
• Zyvex's CNT functionalization approach that dramatically increases CNT solubility without degrading CNT properties. This has resulted in high-strength CNT-reinforced polymer matrices.
• UTD's continuous CNT composite fibers.

These approaches, combined here for the first time, will result in mechanical properties exceeding those of current carbon fiber composites.



Conceptual Drawing of Hierarchical CNT Composite-in-Composite

Technical Objective and Work Plan

Technical Objective:
We intend to make ultra-high-performance composites that utilize CNTs on two structural levels: continuous CNT composite fibers will reinforce a matrix that is itself a CNT composite.

Work Plan:
1. Develop strong continuous CNT composite fibers by optimizing UTD's fiber spinning technology.
2. Develop CNT-reinforced polymer matrices using Zyvex-functionalized single wall nanotubes.
3. Combine these high strength continuous fibers (CNT fibers, carbon fibers etc.) and improved matrices to obtain ultra-high-strength composites.

NASA and non-NASA Applications

• Lightweight, multifunctional structural components for aerospace vehicles, armor, helmets, and fabrics for soldier uniforms
• Multifunctional structural components for the space station
• Advanced materials for fabrics and coatings used in space suits and other space applications
• Coatings and bonding agents for high-value components and equipment
• Composites for satellite armor
• Materials for thermal management
• Advanced materials for medical applications (prosthesis, artificial joints, splints)
• Structural components for high-value civilian transportation
• Materials for professional sports and leisure equipment such as yachts, race cars, golf clubs, fishing rods, and tennis equipment
• Advanced flywheels capable of significantly higher rotational speed

Multifunctional Carbon Nanotube/Polyethylene Complex Composite for Space Radiation Shielding

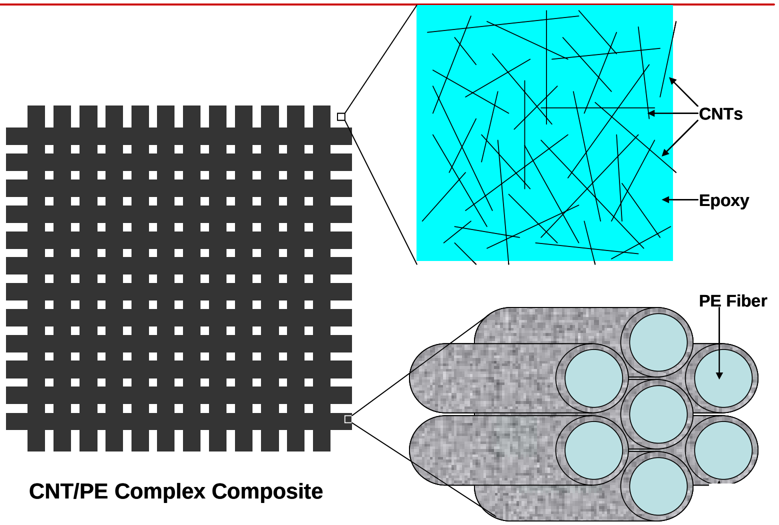
NASA SBIR Phase I ($97K)

Identification and Significance of Innovation

Polyethylene (PE) has been identified at NASA as a promising radiation shielding material against galactic cosmic rays and solar energetic particles. Carbon nanotubes (CNTs) are recognized as the ultimate carbon fibers for high performance, multifunctional composites.

The Zyvex team will develop a multifunctional CNT/PE complex composite that utilize continuous PE fibers will reinforce a matrix that is itself a CNT/epoxy composite.

The program builds on a recent breakthrough:
Zyvex's CNT functionalization approach that dramatically increases CNT solubility without degrading CNT properties. This has resulted in high-strength, high electrically conductive CNT-reinforced polymer composites.



CNT/PE Complex Composite

Technical Objective and Work Plan

Technical Objective:
We intend to make a multifunctional CNT/PE complex composite with high radiation shielding performance, high mechanical strength, high electrical conductivity, and improved thermal stability.

Work Plan:
1. Develop an electrically conductive PE fiber fabric.
2. Produce a CNT-reinforced epoxy matrix.
3. Investigate the radiation shielding effectiveness of CNT/PE complex composite against space radiation.
4. Measure the physical properties of CNT/PE complex composite before and after the radiation exposure.

NASA and non-NASA Applications

• Lightweight, multifunctional structural components for aerospace transportation vehicles, and liquid hydrogen tanks
• Multifunctional structural components for the space structures
• Advanced materials for fabrics and coatings used in space suits and other space applications
• Components and coatings for deep space power systems, and in-space manufacturing and repairing
• Coatings and bonding agents for high-value components and equipment
• Composites for satellite armor
• Space and aerospace crafts and habitats for commercial space travel
• Components for particle accelerators and nuclear reactors
• Radioactive chemical, biological and nuclear waste containment vessels
• Materials for professional sports and leisure equipment such as yachts, race cars, golf clubs, fishing rods, and tennis equipment
• Lightweight, multifunctional materials for soldier uniforms, armor, and helmets

DARPA Multifunctional Next Generation Carbon Nanotube Super Fibers

PI: Hassan Ait-Haddou, Ph.D., Zyvex Corporation

TECHNICAL OBJECTIVES

- Zyvex will develop ultra-high strength and multi-functional carbon nanotube (CNT) continuous spun fibers.
- By applying Zyvex's independently developed CNT functionalizing technology, we will develop the fundamental material that will enable an entire new generation of super-strong, multi-functional, composite materials.

APPLICATIONS

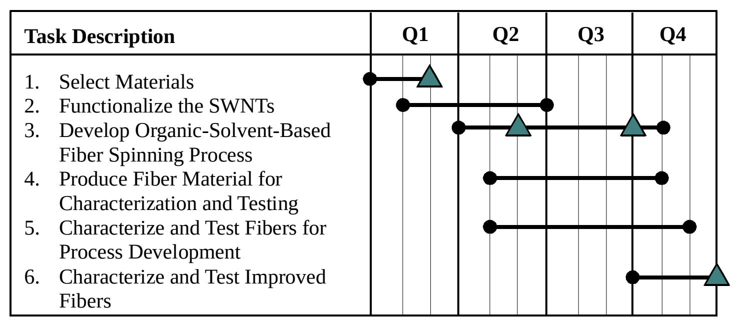
Concept Representation of a Reconfigurable Array of Tethered Airships

•Woven fabrics and tethers will enable large reconfigurable arrays of moderate sized airships
•Super strong fibers with high thermal conductivity can also be woven into multi-functional fabrics that provide both ballistic protection and thermal management
•Enabling material for a low cost system to de-orbit satellites

TECHNICAL APPROACH

- The principal outcome of this seedling program will be development of an organic solvent based CNT fiber spinning process by incorporating our functionalizing technology that overcomes some severe limitations associated with CNT suspensions in aqueous solutions.
- Our demonstrated excellent dispersion and high concentration solutions in organic solvents will not only produce better uniformity in the spun fibers, but also opens up the choice of potential binders to virtually any polymer material.

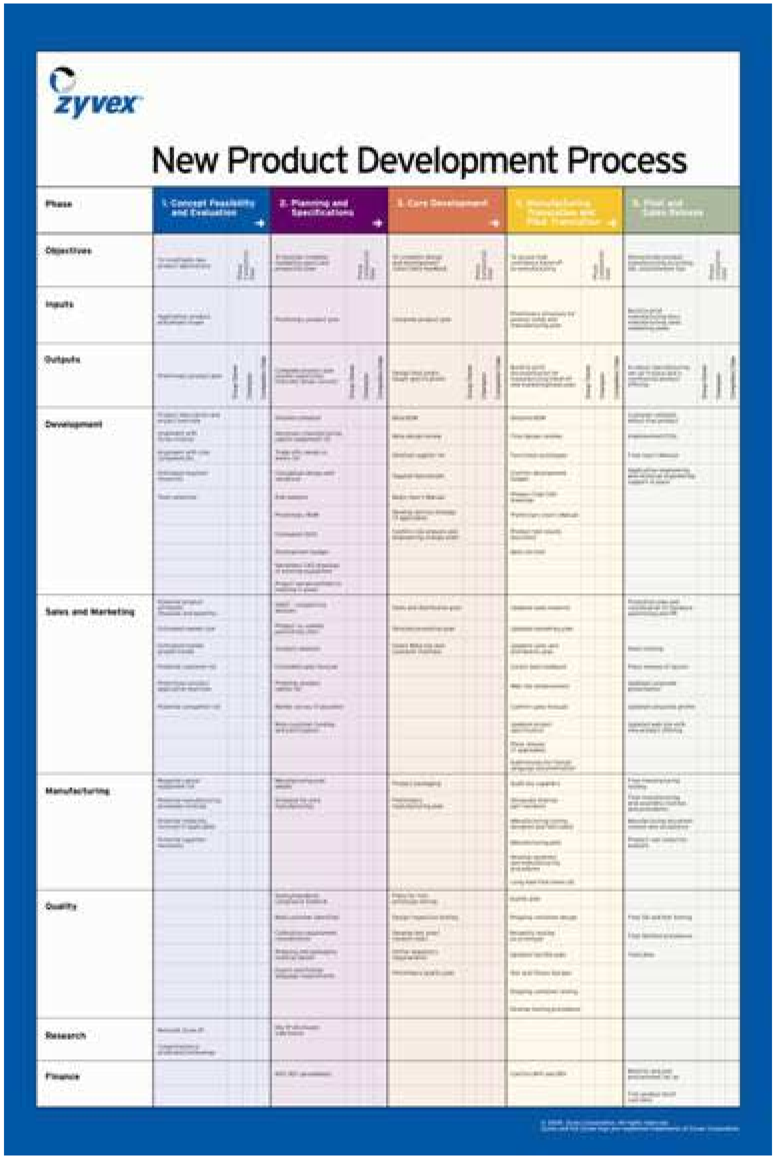
BUDGET AND SCHEDULE



Budget ($207k), 12 month program

DARPA DSO Seedling Program

5 Stage New Product Development Process



1. Concept Feasibility and Evaluation
2. Planning and Specifications
3. Core Development
4. Manufacturing and Pilot Translation
5. Pilot and Sales Release

Time to Market

Late 2001	Conception and First Lab demonstration
April 2002	Presentation to Patent Review Board
May 2002	Filed patents on CNT functionalization
July 2002	JACS Paper on CNT functionalization
October 2002	Started New Product Development Process
December 2002	First Industrial Research Contract
February 2003	Phase I SBIR with NASA JSC
September 2003	First sample sold to Boson Scientific
February 2004	Second Industrial Research Contract
January 2004	Phase II SBIR with NASA JSC
June 2004	Supply Chain Certification Program
July 2004	DARPA Funding for CNT Fibers

Time to Market

- August 2004 — Third Industrial Research Contract
- September 2004 — Easton Announces CNT reinforced Bike parts
- October 2004 — Forth Industrial Research Contract
- December 2004 — Fifth Industrial Research Contract
- January 2005 — Phase I SBIR with NASA Marshall
- January 2005 — Sixth Industrial Research Contract
- February 2005 — Seventh Industrial Research Contract
- May 2005 — Easton releases line of CNT Baseball Bats
- May 2005 — Eighth Industrial Research Contract
- June 2005 — Notification of R&D 100 Award for NanoSolve
- June 2005 — Eight Industrial Research Contract
- June 2005 — Aldila releasing golf club shafts to Pros

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Easton
The Ballpark
"Range-Baseball :25/:05"
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TRT: 30 Seconds
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ka-chew!

Commercialization Successes

- Easton and Aldila are the first customers in production our epoxy CNT products
- We are working seriously with several other companies:
  - Honeywell
  - Intel
  - Boeing
  - Lockheed Martin
  - Raytheon
  - Others (Confidential)

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