NIST
National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

High Throughput Methods for Nanocomposite Materials Research

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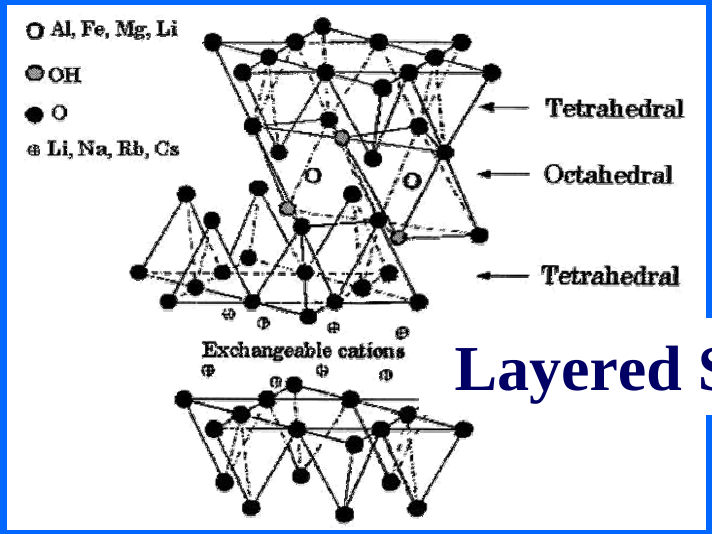
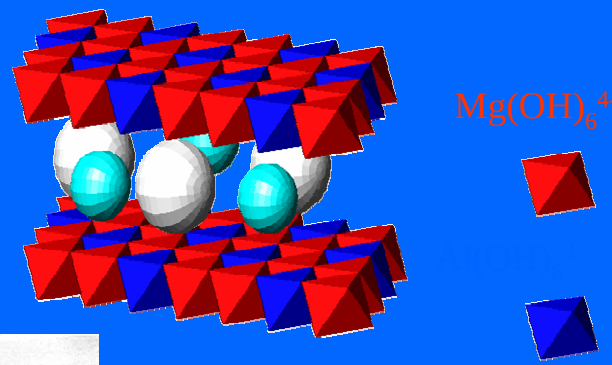
Nano-additives



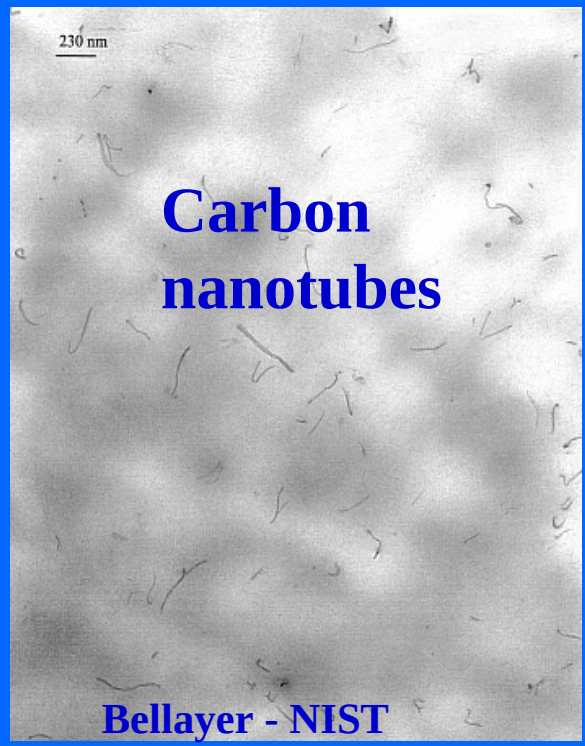
Layered Silicates

Layered Double Hydroxide



Zammerano-Trieste

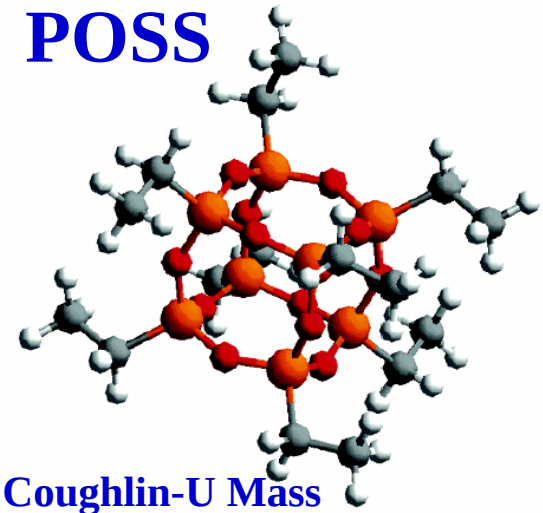
Nano silica



Carbon nanotubes

Bellayer - NIST

POSS

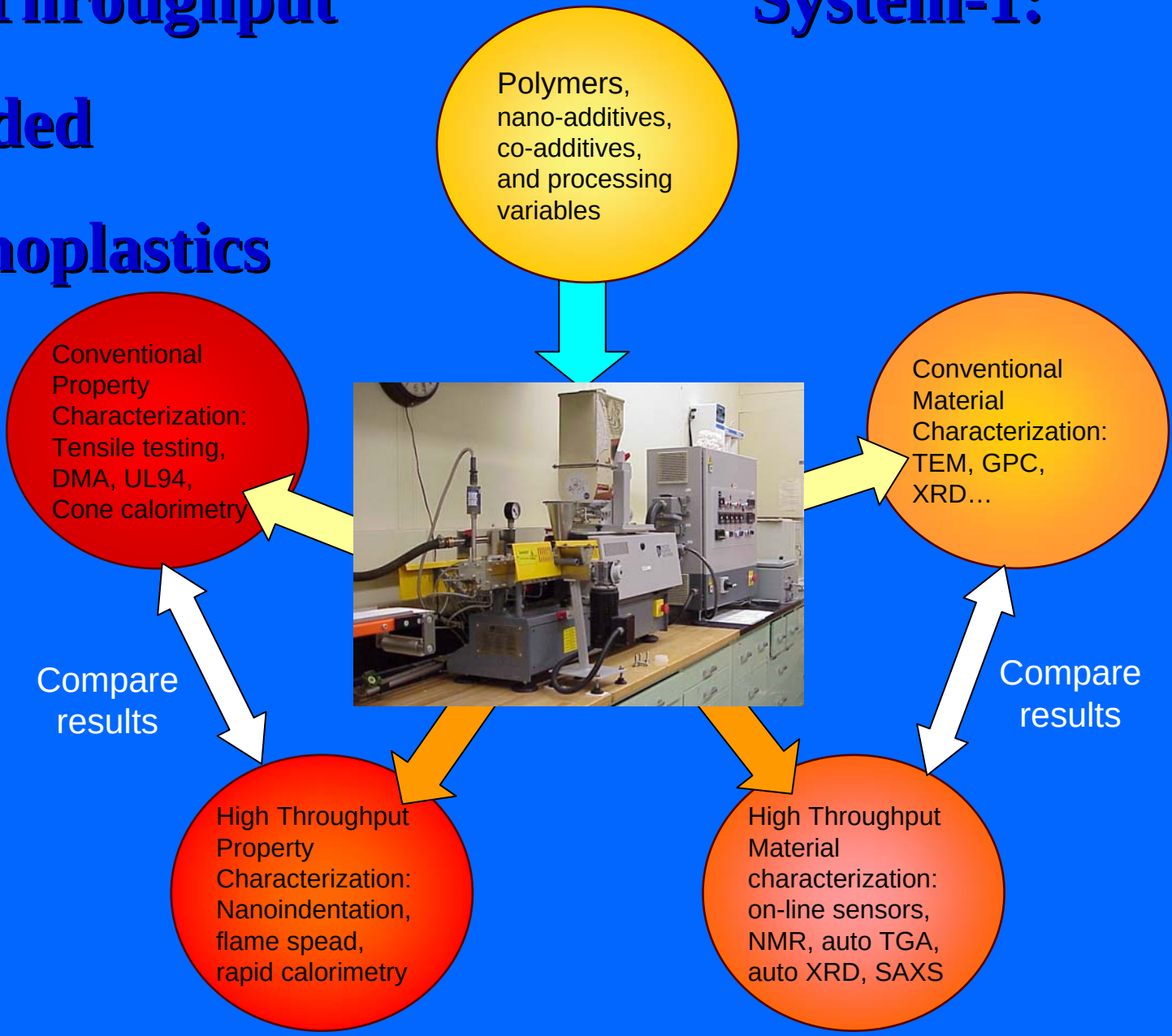


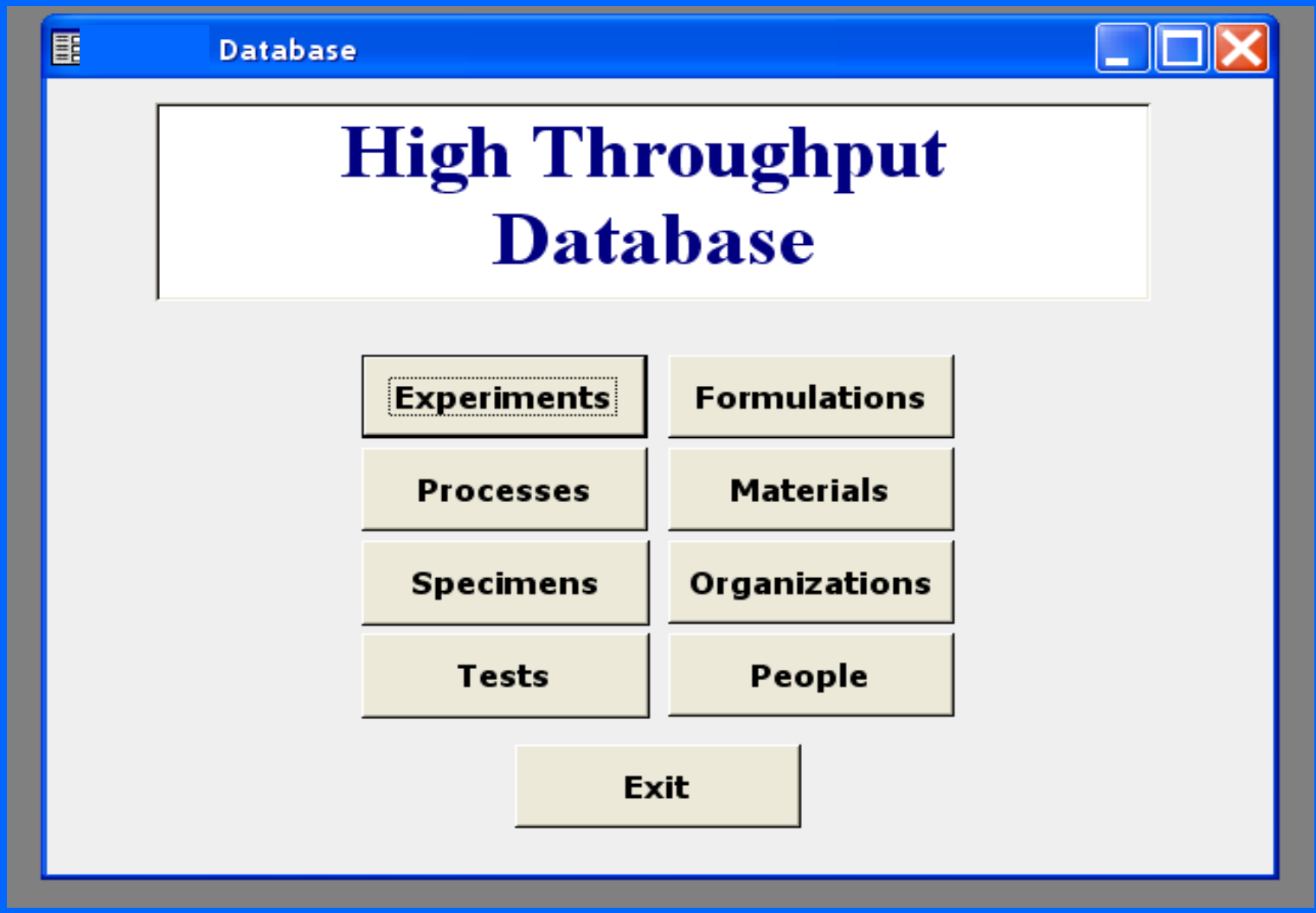
Coughlin-U Mass

Parameter Space (~ 10^6 Experiments) for Polymer Nanocomposites

Polymer	Nano-additive	Organic Treatment	Processing Conditions	Other additives	Flame Retardant
PE	clay	Alkylammonium	Temperature	Stabilizers	Phosphate
PP	POSS	Imidazolium	Shear	Processing	Halogenated
PS	Carbon	Chelates	Residence time	UV	Silicon Based
PA6	Silica	Silated	.	Antioxidant	.
PU	.	Alkyl	.	Fillers	.
PVC	.	Carboxylate	.	Pigments	.
PC	.	.	.	.	.
PEO	.	.	.	.	.
PMMA	.	.	.	.	.
EVA	.	.	.	.	.
Epoxy	.	.	.	.	.
.	.	.	.	.	.
.		.	.	.	.

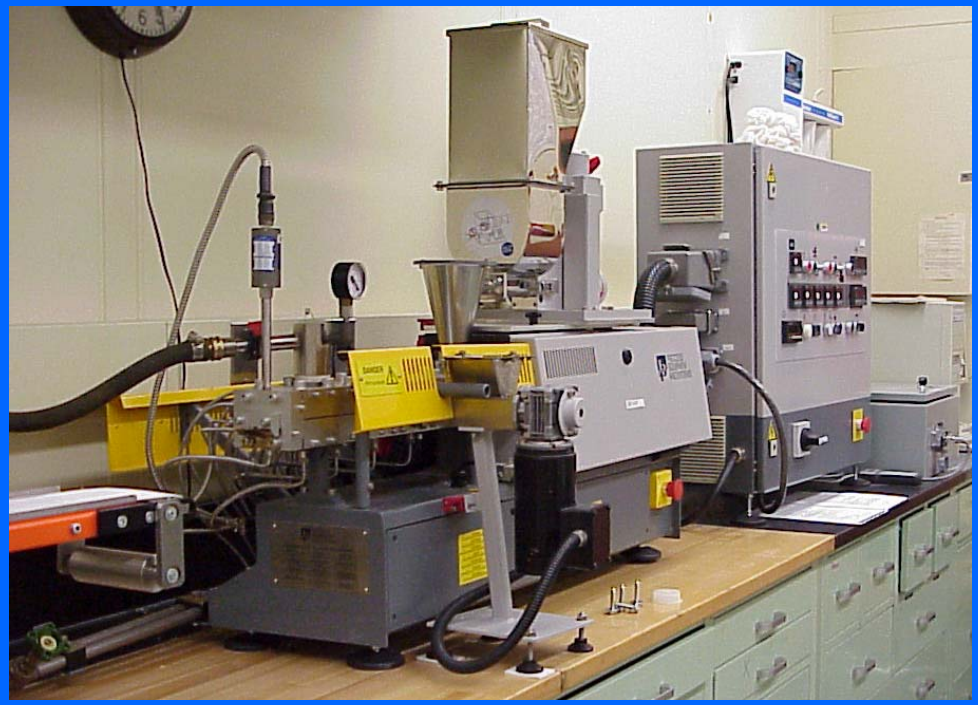
High Throughput Extruded Thermoplastics System-1:





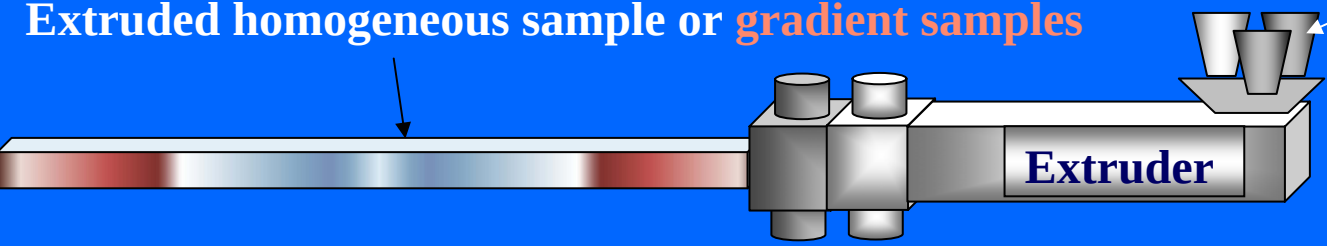
Standardized: inventory control, experimental design, data input, analysis and visualization

Extrusion of Samples



Gravimetric feeders

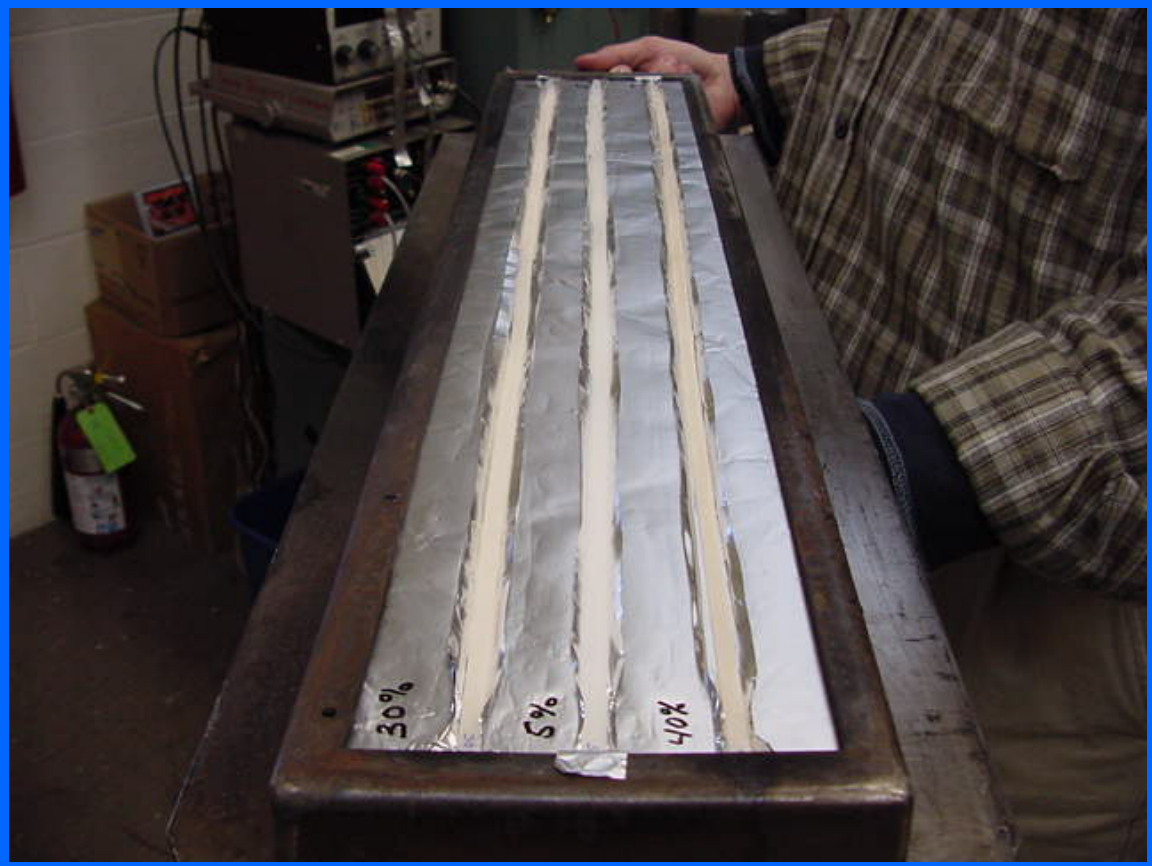
Extruded homogeneous sample or gradient samples



Off line High Throughput:
TGA, X-Ray scattering, NMR,
nanoindentation,
Flame spread

~ In-line sensors: UV-Vis, FTIR, optical,
dielectric

Extruded samples



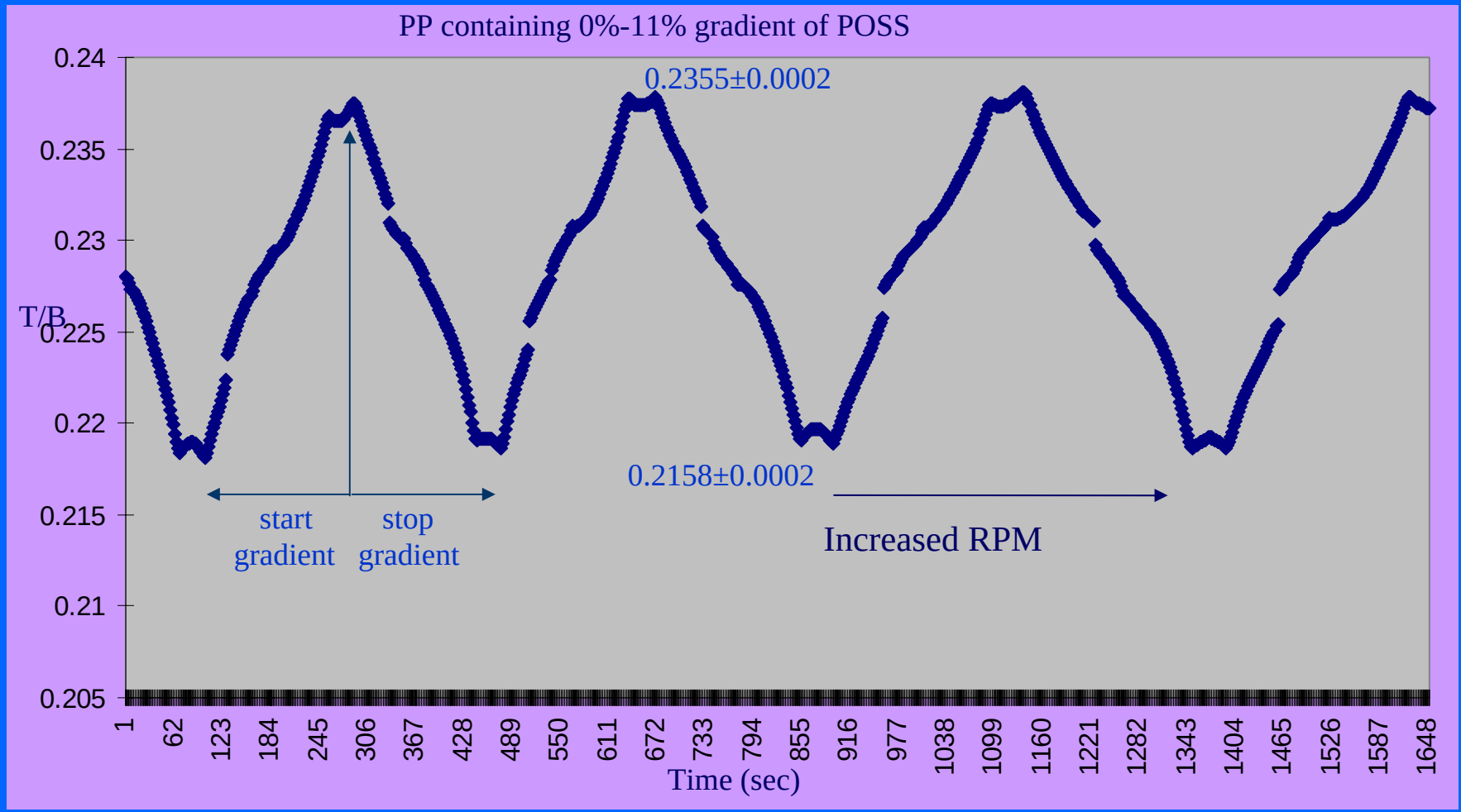
No pelletizing or injection molding

Additive Filled Polymers: Gradient and Single Composition

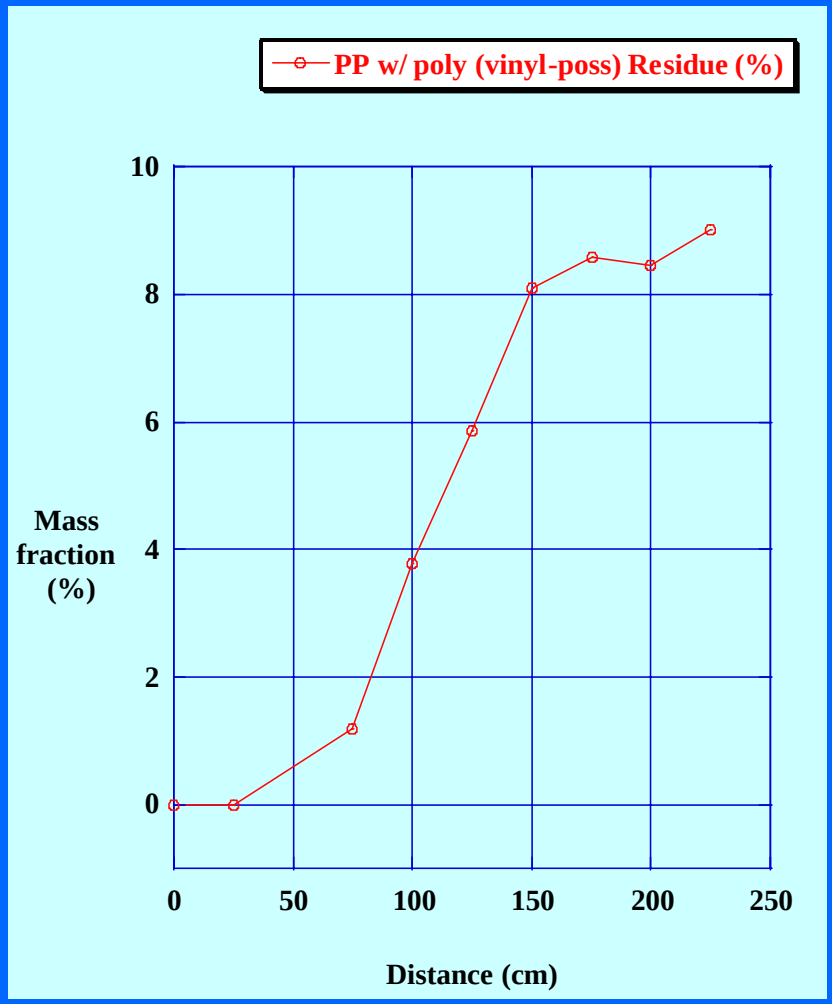
1 Day

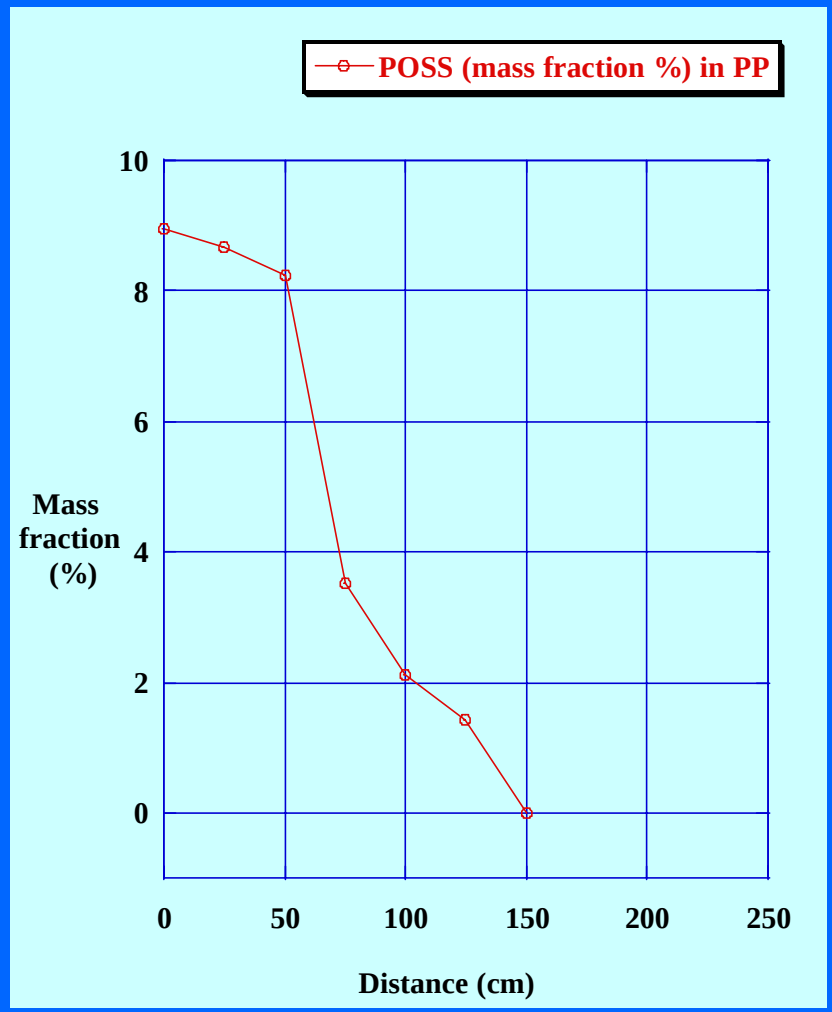
- PP: Pure and FR
  - Poly(phenyl-POSS) : (0-11 mass-%)
  - Poly(vinyl-POSS) : (0-11 mass-%)
- ABS
  - Poly(vinyl-POSS) : (0-11 mass-%)
- 9 different single-composition strands
- 3 replicates each
- up to 50-60 different compositions

Fiber-Optic Sensor



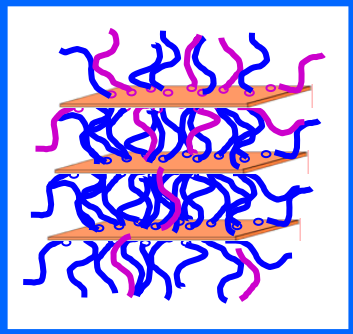
POSS/PE Gradients

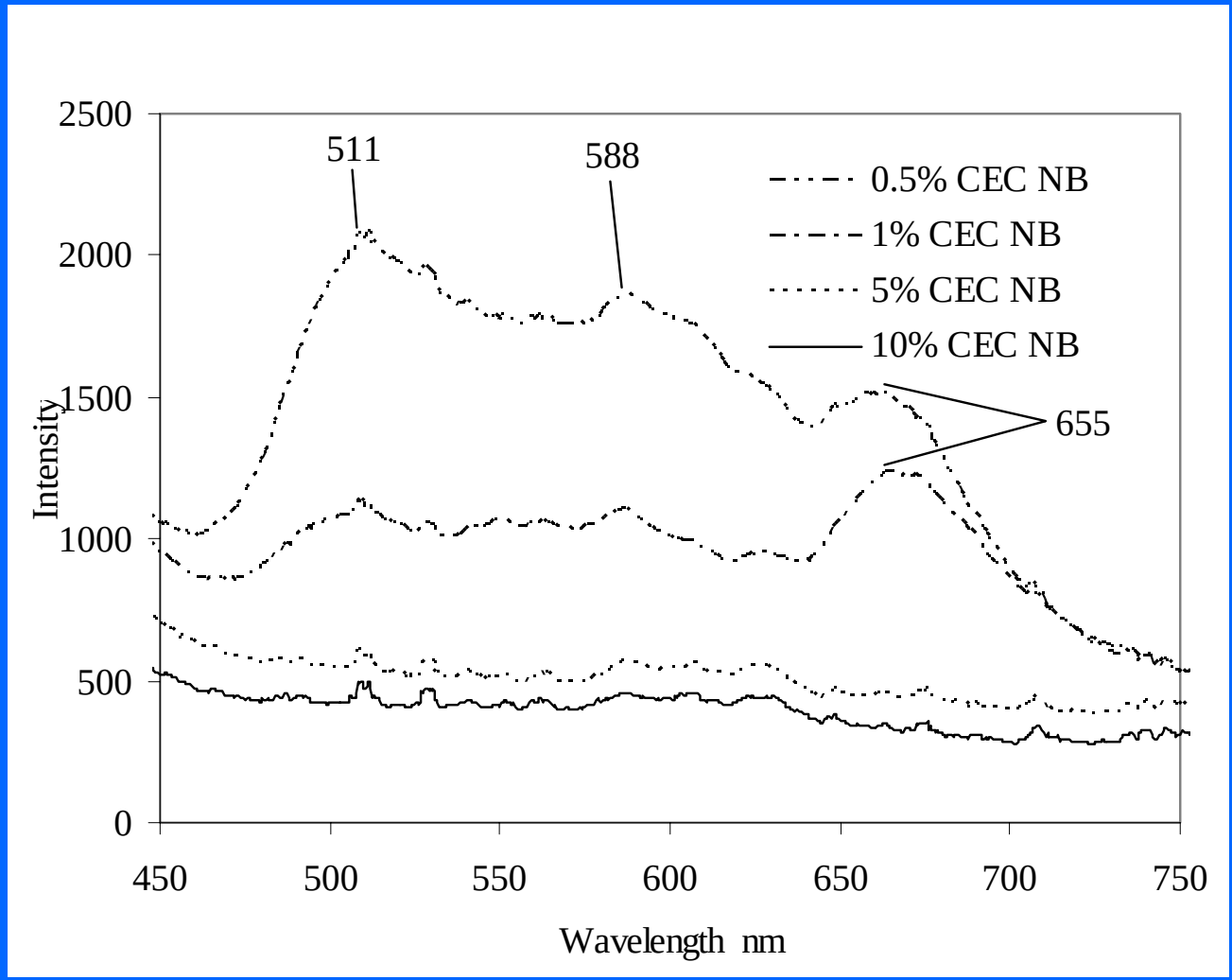




Fluorescence of NB-MMT Clay

Nile-Blue-clay





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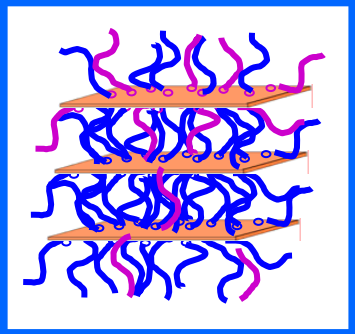
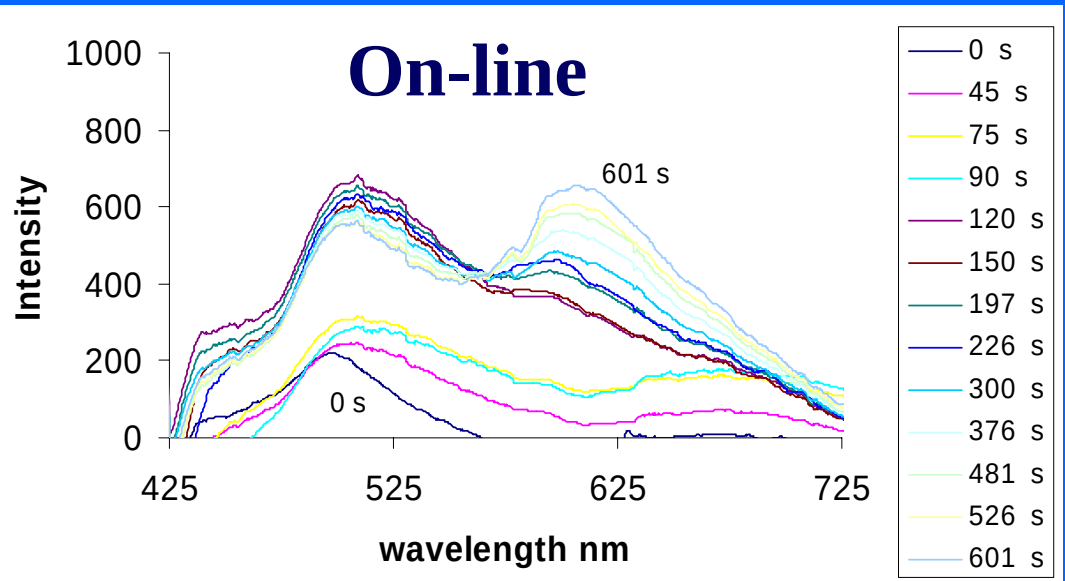
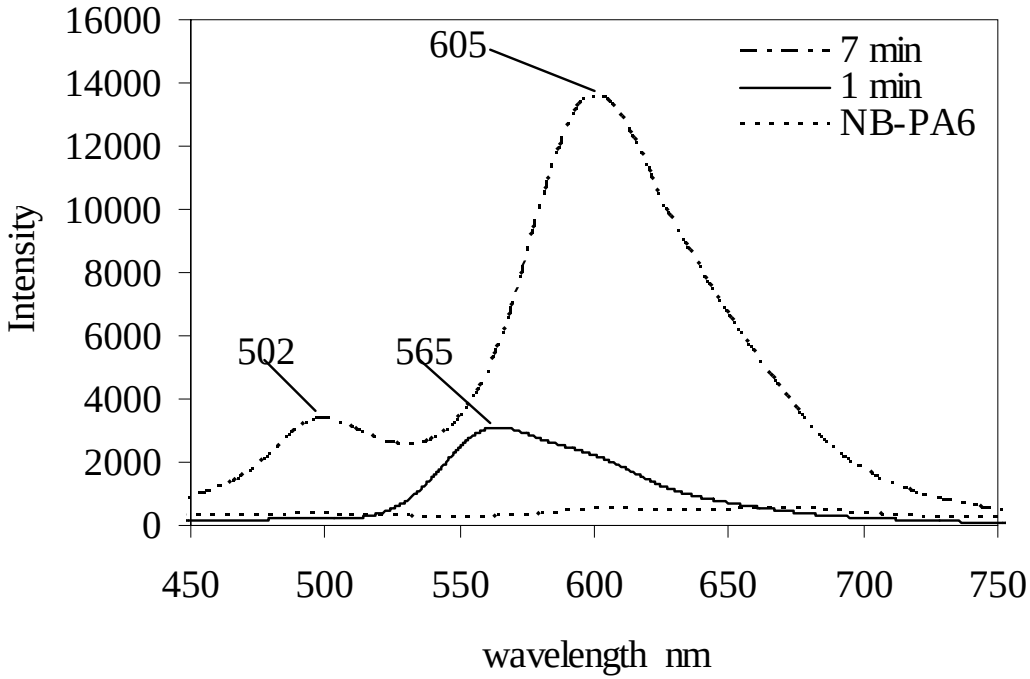
Fluorescence Monitoring of Exfoliation

Nile-Blue-clay







Post processing

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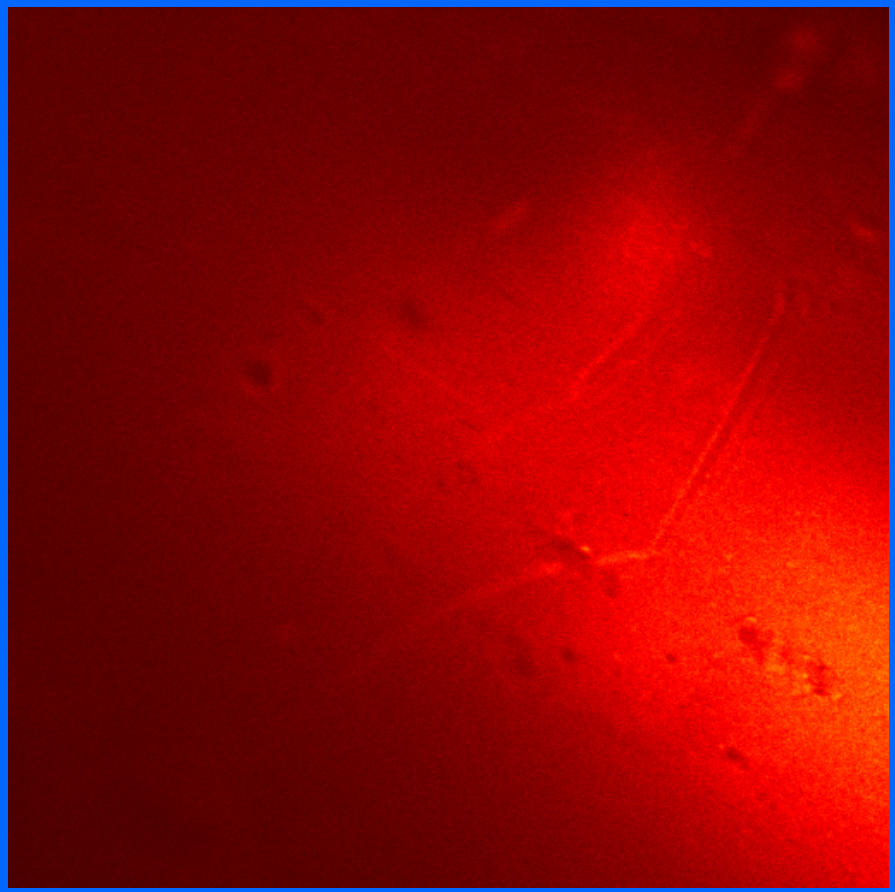
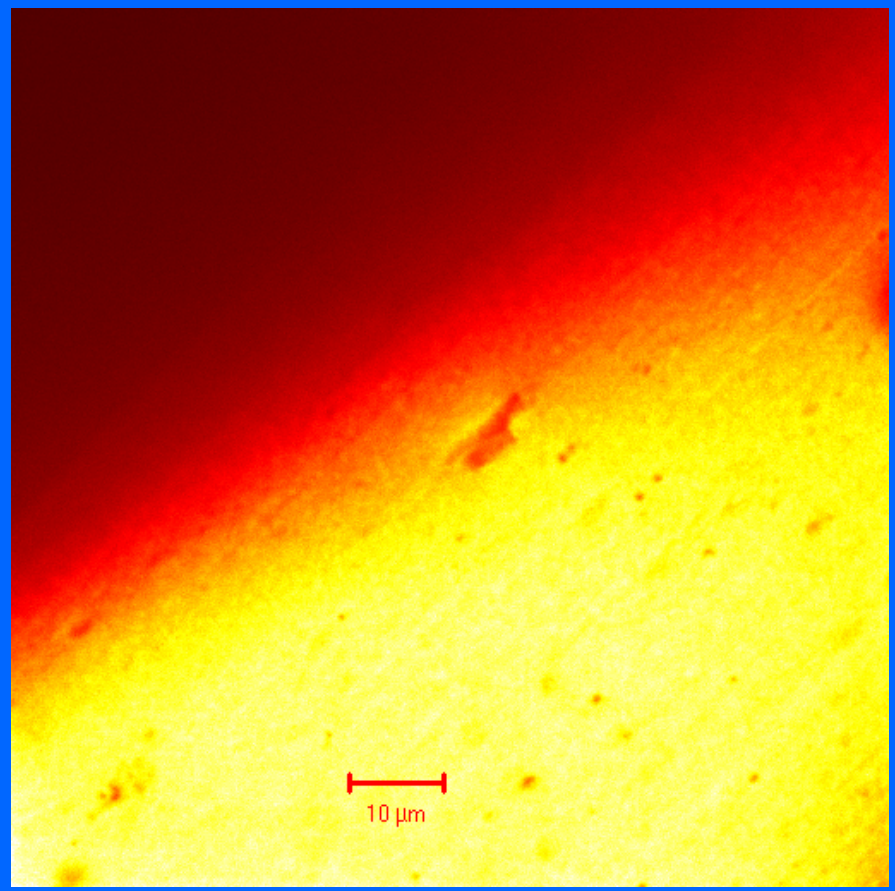
Fluorescence Monitoring of Exfoliation

PA-6 2% IM/NB MMT 1 min



PA-6 2% IM/NB MMT 7 min

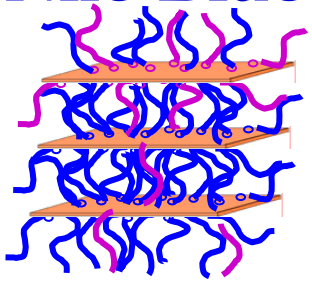


Laser Scanning Confocal Microscope; 80 images, 370 nm slice

Charles Wu, Lipiin Sung

Optical Monitoring of Exfoliation

Nile-Blue-clay



Micro/intercalated

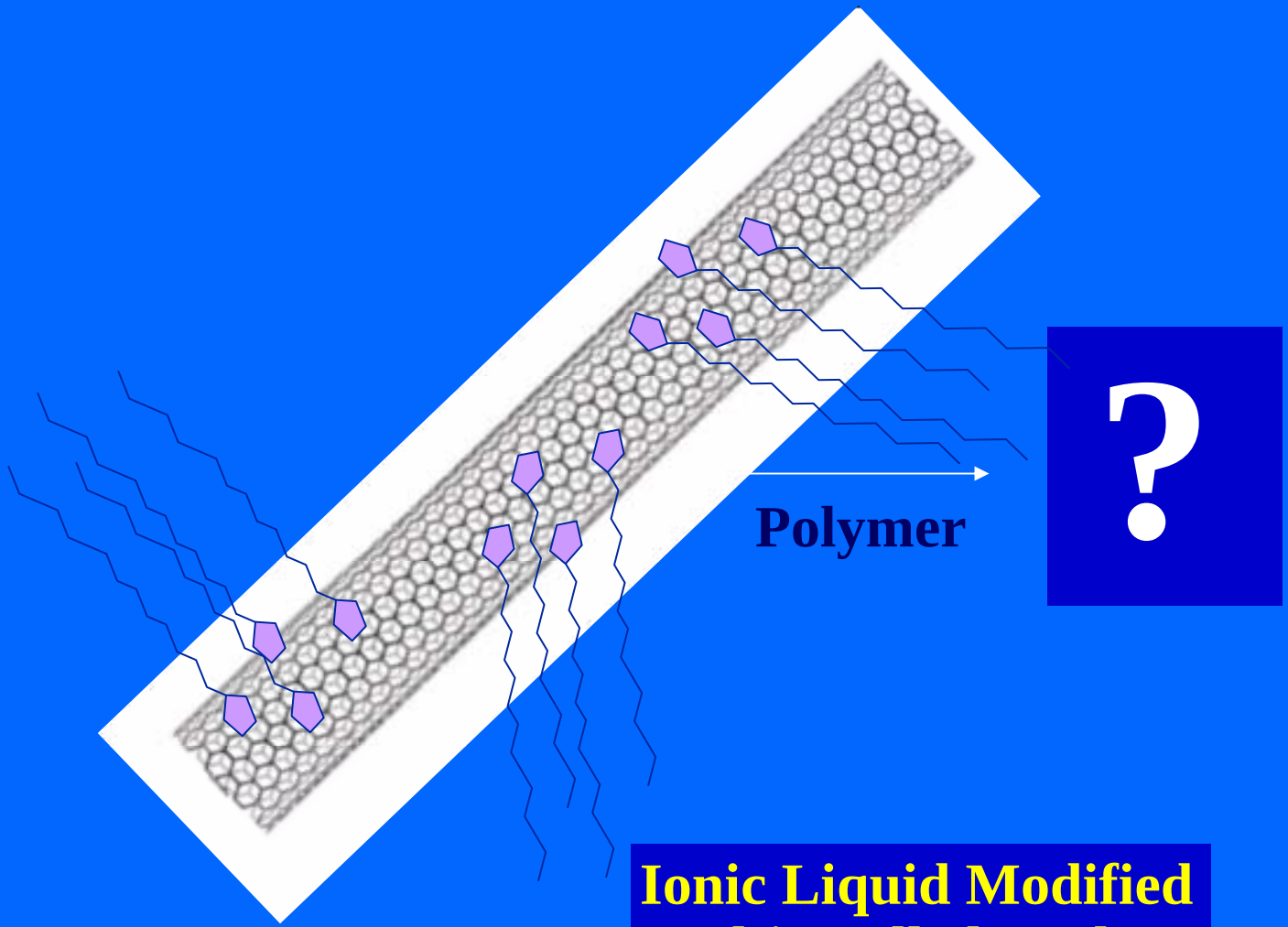
PA-6 2% IM/NB MMT extruded-1 min

Disordered exfoliated

PA-6 2% IM/NB MMT extruded-7 min

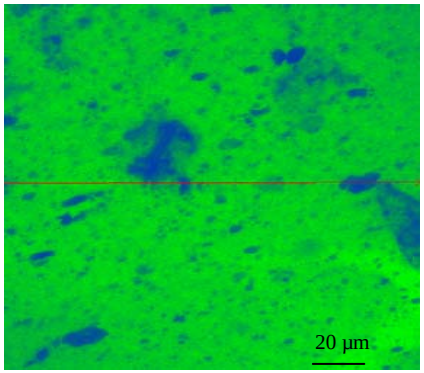
Maupin, et al, Macromol. Rapid Comm. April, 2004
Gilman et al, PMSE, 2004

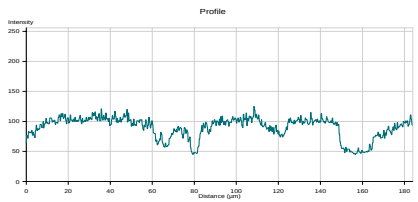
Multi-Walled Carbon Nanotube Polymer Composites



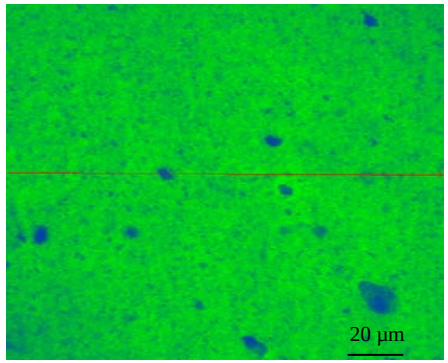
Ionic Liquid Modified Multi-Walled Carbon Nanotube

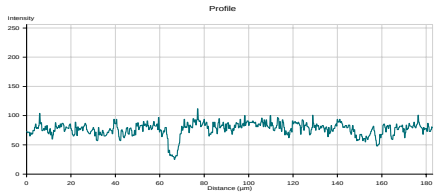
Confocal Microscopy of PS-MWNT



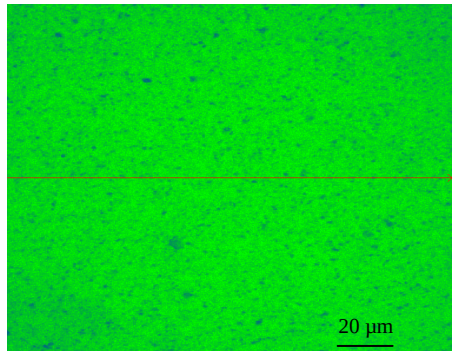


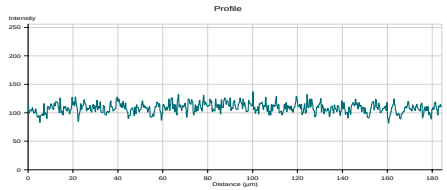
PS + 0.5% MWNT (Nile Blue) 1 min 200 RPM 195°C



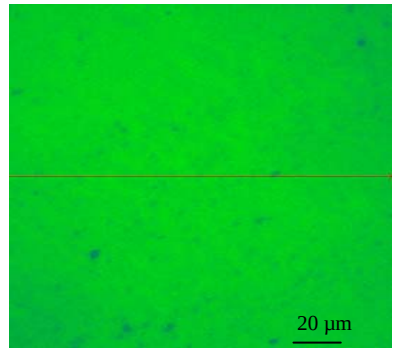


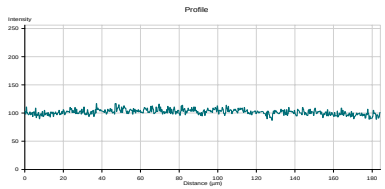
PS + 0.5% MWNT (Nile Blue) 10 min 200 RPM 195°C





PS + 0.5% MWNT+ 0.5% DMHDIm-TFB (Nile Blue) 10 min 200 RPM 195°C





PS + DMHDIm-TBF (Nile Blue) 10 min 200 RPM 195°C

Quantitative Image Analysis of PS/MWNT Nanocomposite

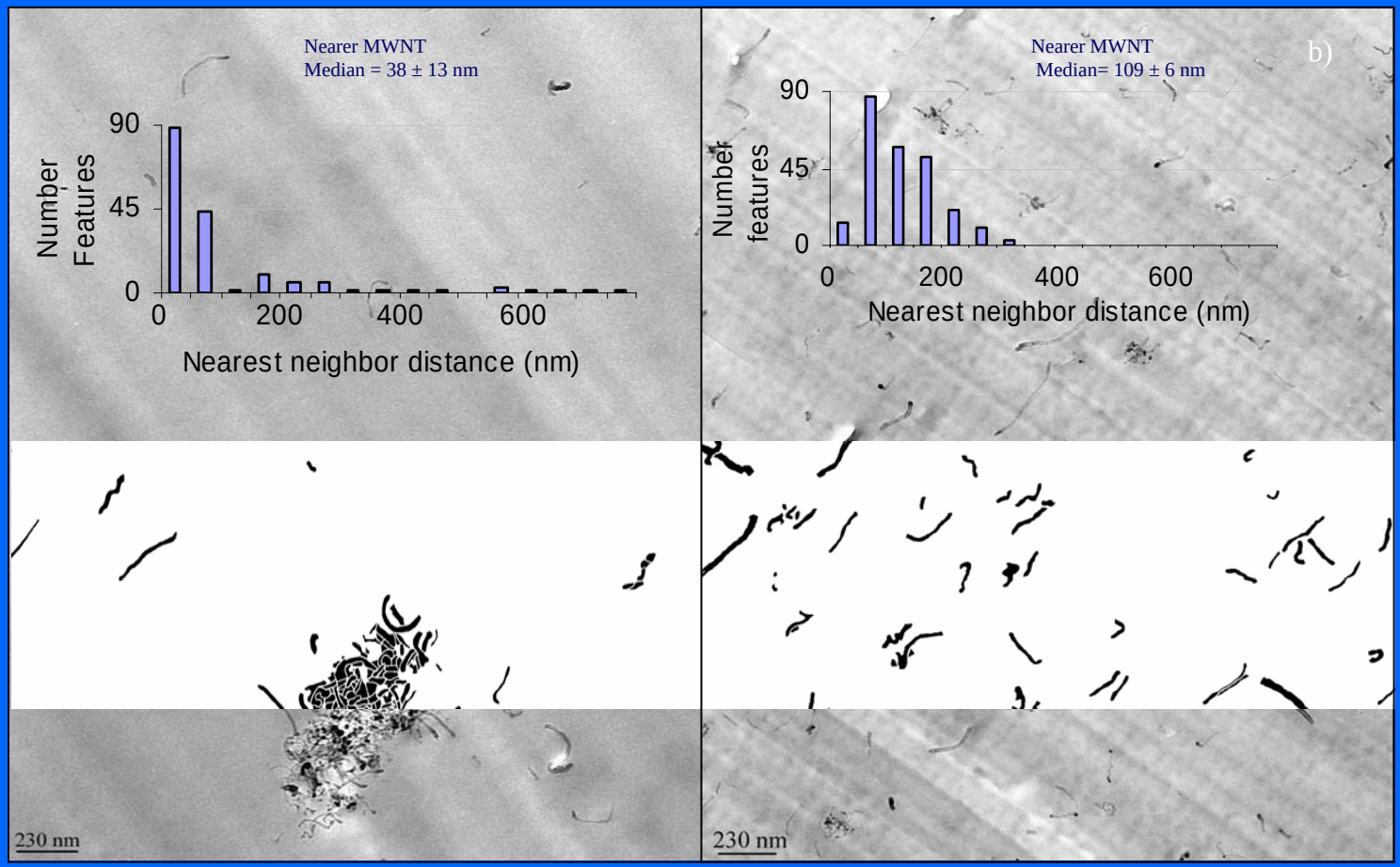
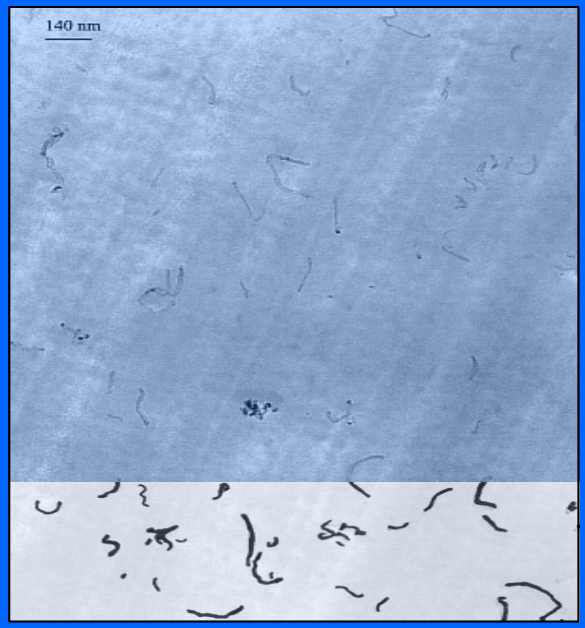
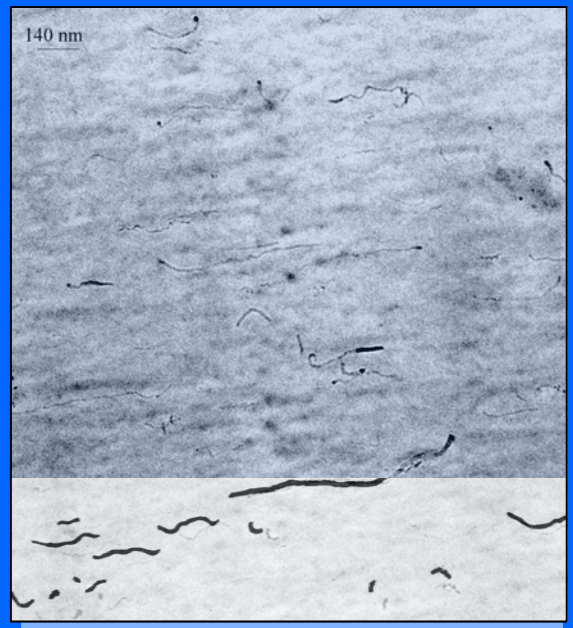


Figure : TEM pictures of a) PS/MWNT untreated sample and b) PS/MWNT/(1:1)DMHDIm-TFB sample, both partly threshold into binary images.

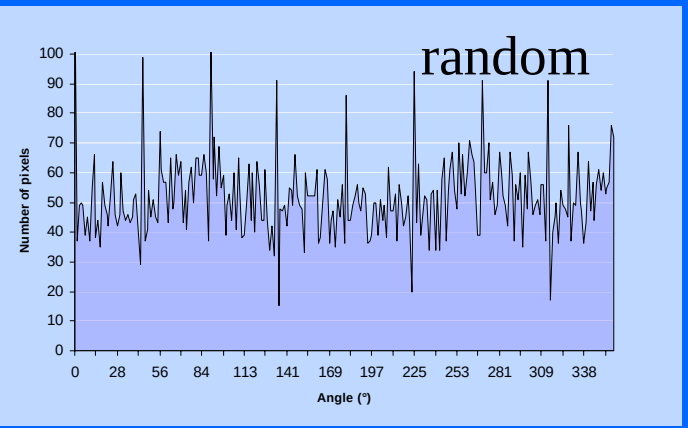
Image analyses to quantify nanotube orientation

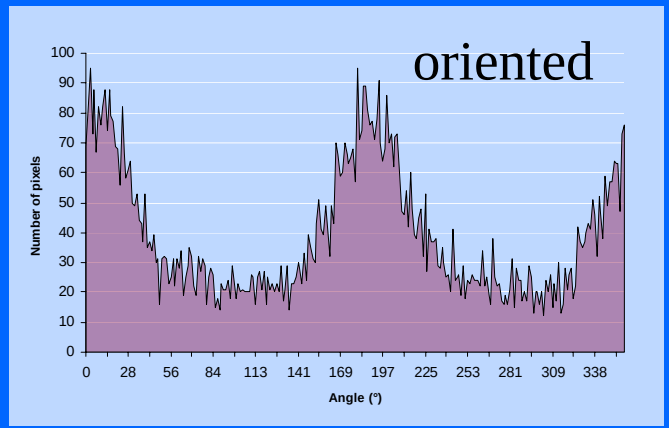


TEM images of PS/MWNT (Hyperion Catalysis) mixed by extrusion (195°C, 200 RPM, 5 min). Image analysis performed after removing the background

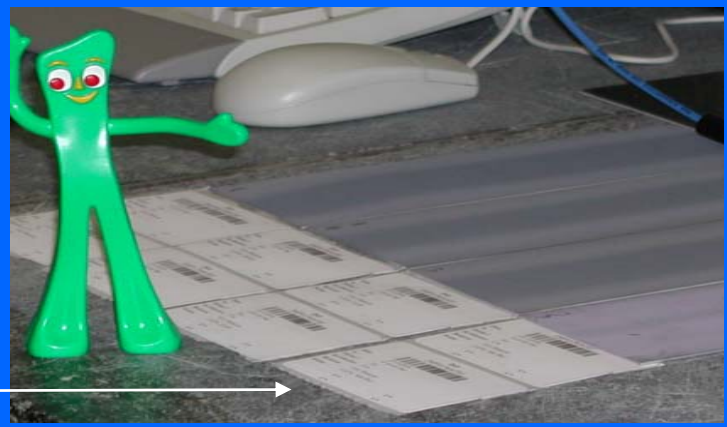


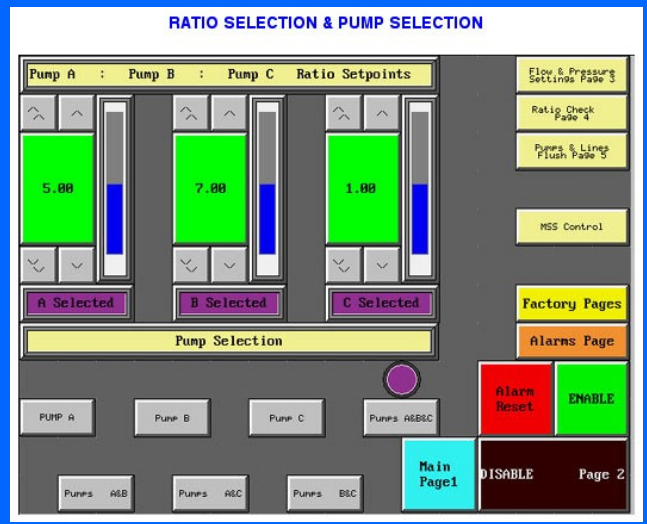
*TEM images of PS/MWNT (Hyperion Catalysis) mixed by extrusion (195°C, 200 RPM, 5 min) and **drawn** into fiber. Image analysis performed after removing the background*





High Throughput System-2
Gradient Coatings

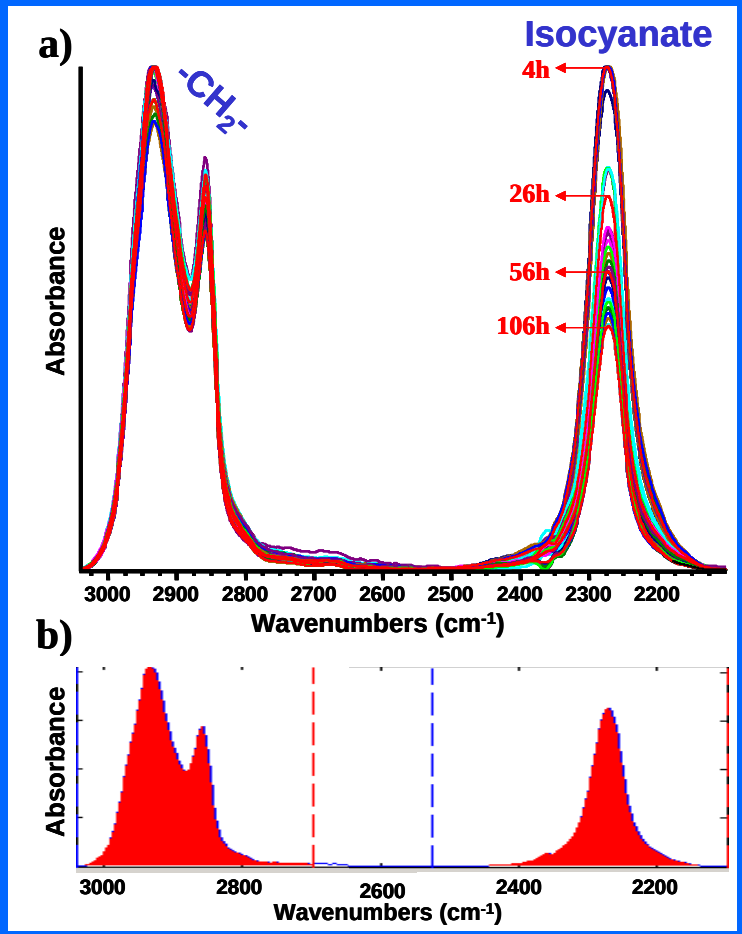




High Throughput Screening of Military Aircraft Topcoat Accelerators Project

Micro FTIR cure monitoring

Naomi Eidelman

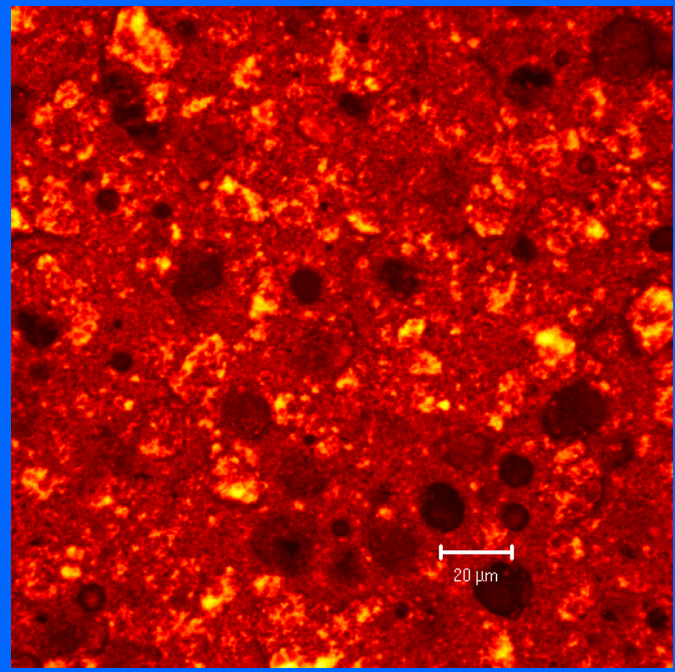


a) Micro-IR spectra taken from six spots between 4 h to 106 hours after the coatings were sprayed. All spectra were normalized to the –CH2- peaks.
b) The isocyanate and the -CH2- peaks in the respective spectral regions used for the peak ratios.

Laser Scanning Confocal Microscope;

100s of images, 100-1000 nm slices

Lori Brassell, Severine Balayer, Lipiin Sung



High Throughput Screening of Military Aircraft Topcoat Accelerators Project

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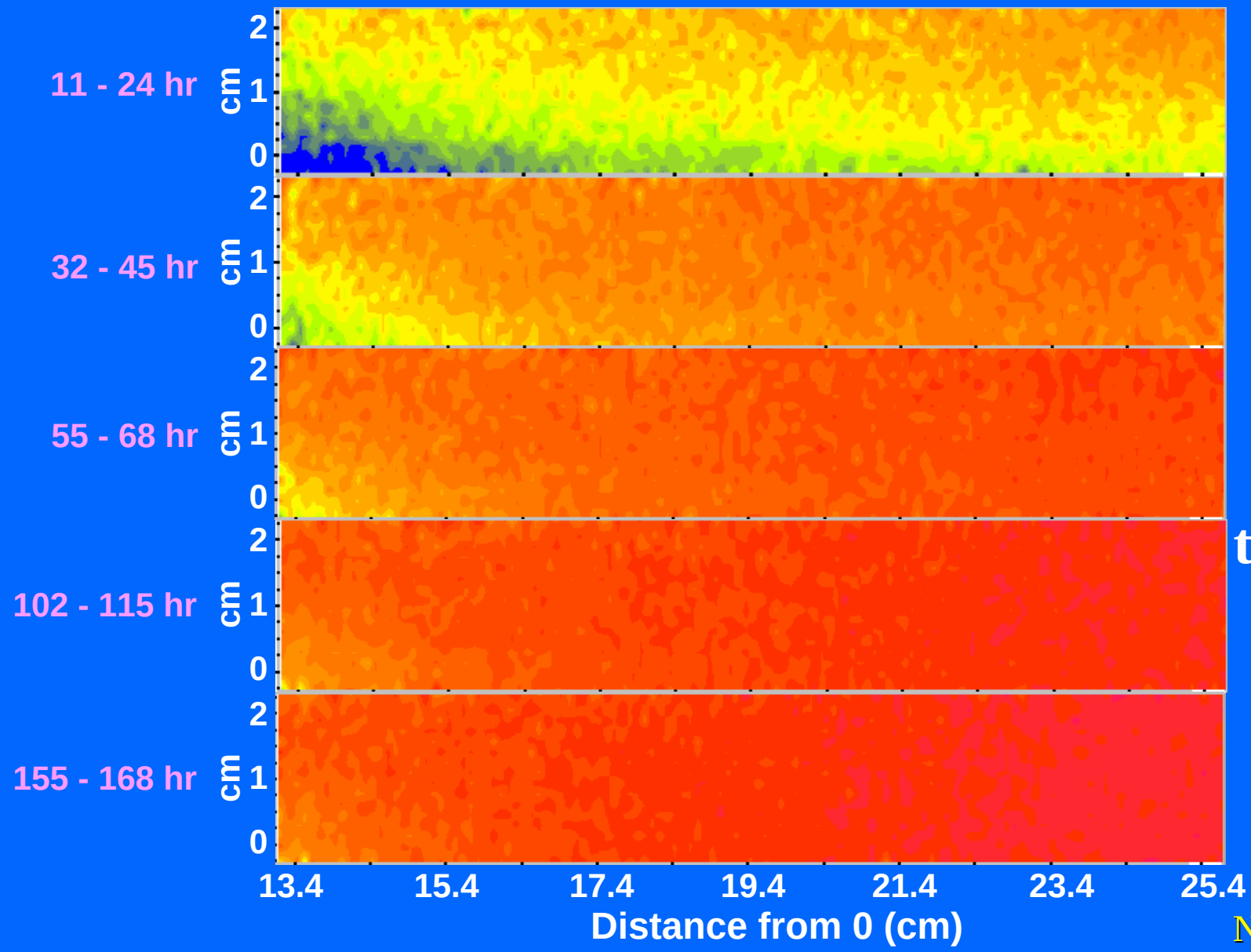
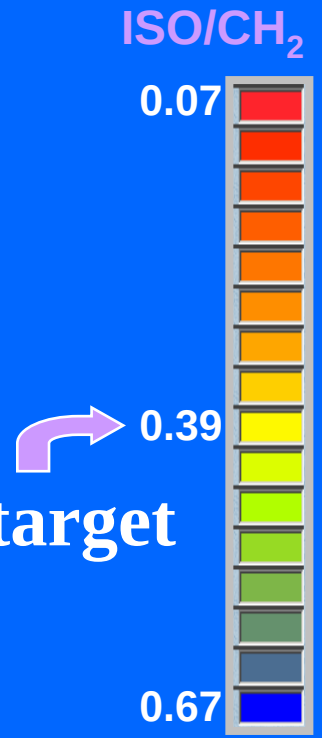
Technology Administration, U.S. Department of Commerce

Gradient #3, 1000, 1600, 32s, (2100- 2551)/(2700-3040)cm^{-1}





Naomi Eidelman

Conclusions

- Extrusion offers rapid sample preparation and with in-line sensors the potential of HT characterization
- Gradient Spray Coating Facility provides rapid sample preparation
- FTIR-Microscopy, Optical probes and Confocal are inherently High Throughput vs. alternatives

Research Team

Rick Davis, Takashi Kashiwagi, Marc Nyden, Richard Harris, Greg Linteris, John Shields, Walid Awad, Lori Brassell, Michael Smith - BFRL/NIST;

David VanderHart, Atsuchi Asano, Anthony Bur - MSEL/NIST

Rick Beyer, Mark Vanlandingham –ARL

Joe Lichtenhan –Hybrid Plastics

Paul Maupin –DOE

Paul C. Trulove and Hugh DeLong -Air Force Office of Scientific Research

Doug Fox – NRL, Naval Academy

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AFRL – High Throughput Screening of Military Aircraft Accelerators - Joel Johnson

High Throughput Methods for Materials Flammability Consortium (AFRL, FAA, Rhodia, Dow)

FAA, Richard Lyon at William J. Hughes Technical Center (IAA- DTFA03-99-X-90009)