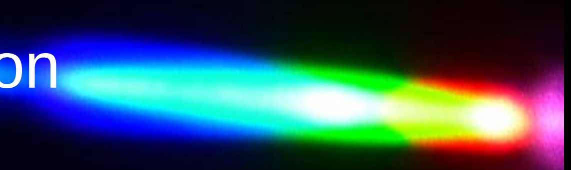
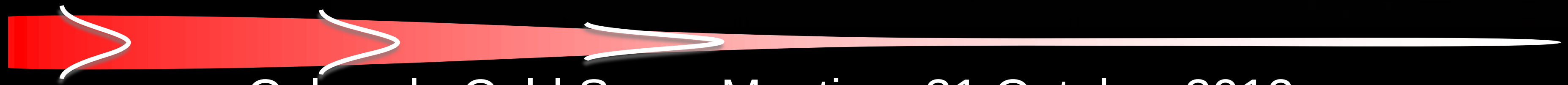
Nanomaterials by Design: The Next Generation of Cold Spray Precursors

Monodispersed Nanomaterials
Femtosecond Laser Energy Deposition
Spatially-Modulated Nanoparticle Nucleation
Filament Reactor
Shaped Pulses for Timed Energy Deposition
Making Snowballs





Colorado Cold Spray Meeting, 31 October 2012

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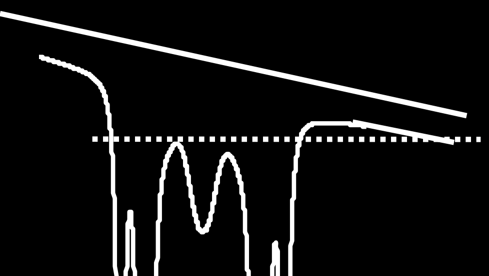


Nanomanufacturing by Design: The Team

Optics Design and Laser Technology

Katharine Moore, Johanan Odhner, Robert Levis

Center for Advanced Photonics Research, Temple University

Reactor Design and Nanomaterials Synthesis

Nicholas Coppa

Nanomaterials Company

Precursor Synthesis for Nanomaterials

Bradford Wayland Group, Michael Zdilla Group

Inorganic Chemistry, Temple University

Nanomaterials Analysis: TEM, SEM, AFM, Dynamic Light Scattering

Daniel Strongin Group, Eric Borguet Group

Physical Chemistry, Temple University

Nanomaterials Analysis: Electrospray Ionization Mass Spectrometry

Paul Flanigan, Santosh Karki, Fengjian Shi, Robert Levis

Analytical Chemistry, Temple University

Temple University Nanomanufacturing by Design Effort for ARL

We offer new enabling technologies developed at Temple University based on nonequilibrium energy deposition from femtosecond laser pulses into precursor materials in the solid, liquid or gas phase. The technologies include:

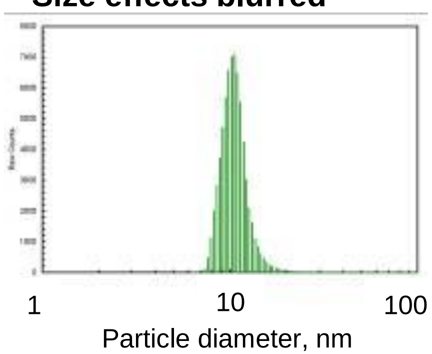
I, Shaped laser pulse reactor capable of preparing millions of different high intensity multifeature laser pulses optimized with genetic algorithms to control nanoparticle size.

II, A laser filamentation reactor that creates ultrahigh intensities over meter path lengths for potential high volume processing.

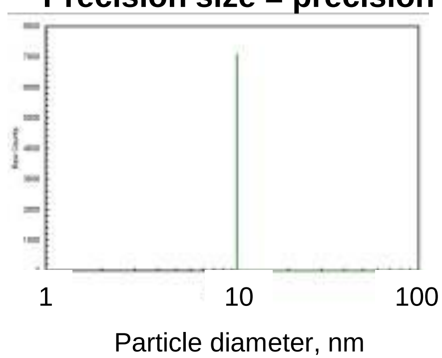
In combination with Nanomaterials Company we seek to determine the genome for nanomaterials manufacturing by performing hundreds of thousands of processing experiments under closed loop control at 1 KHz speeds. The outcomes of the experiments will be used to create the road map of the processing rules allowing the efficient design of new materials with desired characteristics.

In combination with WPI we will engineer new reactor designs to produce nanomaterials in high yield and quantities for subsequent materials processing technologies.

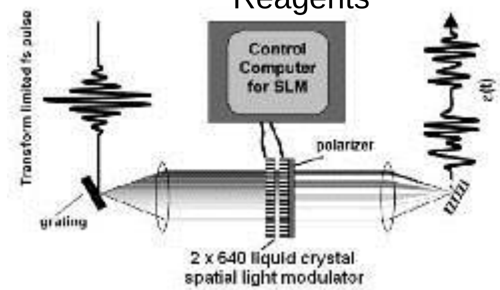
Existing Technology
Thermal process
Statistical size distribution
Size effects blurred



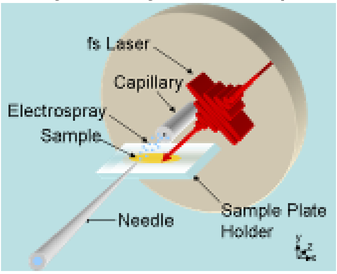
Nonthermal process
Control at the atomic level
Nonstatistical size distribution
Precision size = precision size effects



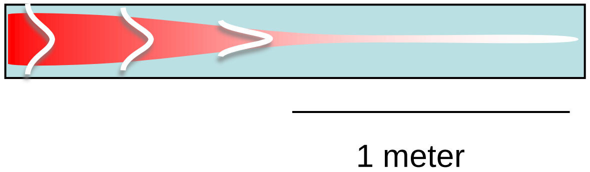
Laser Pulse Shaper Creates Million of Potential Photonic Reagents



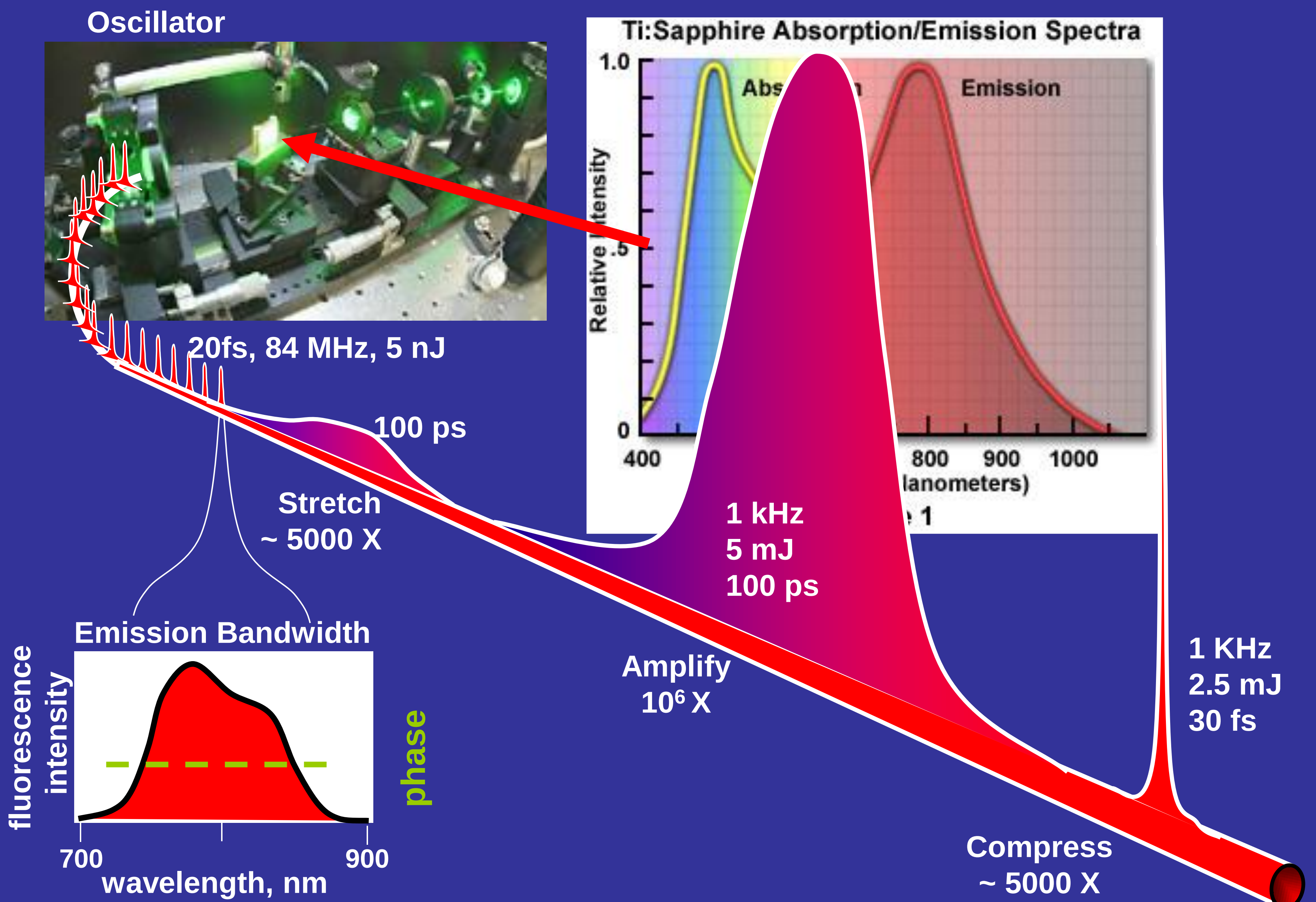
Intense Pulse Shapes Interact with Precursor to Form Nanomaterial as Analyzed by Mass Spec



Laser Pulse Filaments to Form Extended High Intensity Light String for Large Volume Nano Processing

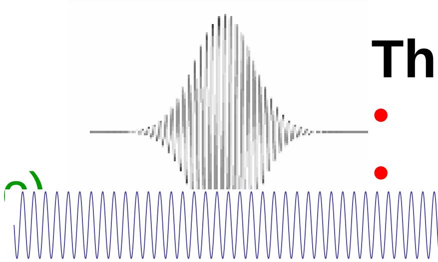


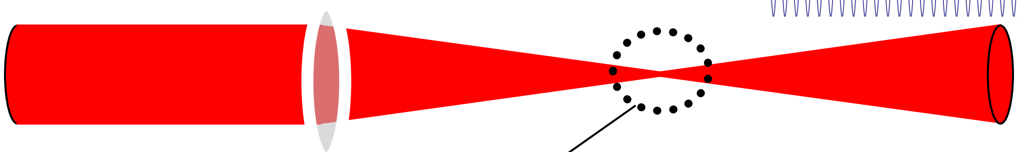
A Primer on Femtosecond Laser Technology



Overview of Ultrafast, Strong Field Molecule Interactions

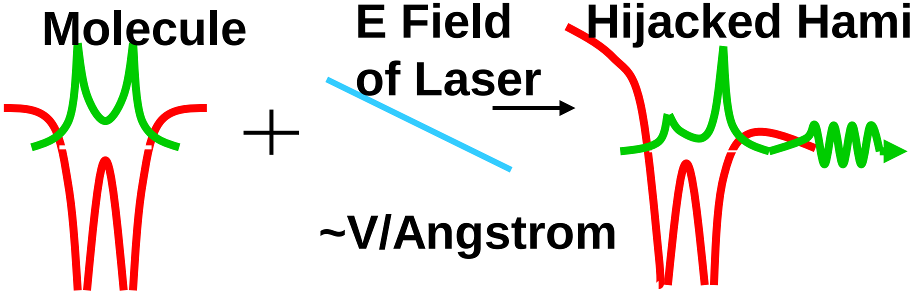
Titanium sapphire regenerative amplifiers:1-10 mJ, 40 fs, 20 oscillations of laser (ns ~ 10^6 osc)





1mJ, 50fs focused to 100 micron spot:
photon density **~10^9 photons/wavelength^3**
electric field strength **~6 Volts per Angstrom**
intensity **~10^{13} W cm^{-2}** **(vs 10^6 W cm^{-2} for ns)**.

The Laser Molecule



The Science:

- Strong Field Chemistry
- Strong Field Physics
- Impulsive Raman Sensing
- Pulse Shaping to Generate N Focal Spots
- Bio-Molecular Microscope
- Time-Dependent Rabi Oscillations
- Remote Detection Technologies
- Hamiltonian Control
- Dimensionality Reduction

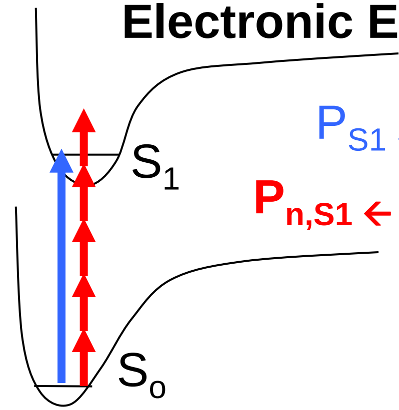
The Products:

O + C + O	**(MPD)**
OC^+ + O	**(Dissociative Ionization)**
$O^+ + C^+ + O^+$	**(Coulomb Explosion)**
$OCO^+ + e^-$	**(Molecular Ionization)**
OCO^*	**(Electronic Excitation)**
$OCO^* + h\nu'$	**(Nonlinear Optics)**

Femtosecond Laser Excitation at 10^{13} W cm^{-2}

- Couples into all molecules via facile multiphoton excitation
- Leads to multiphoton ionization and bond dissociation
- Potential for controlling reactivity

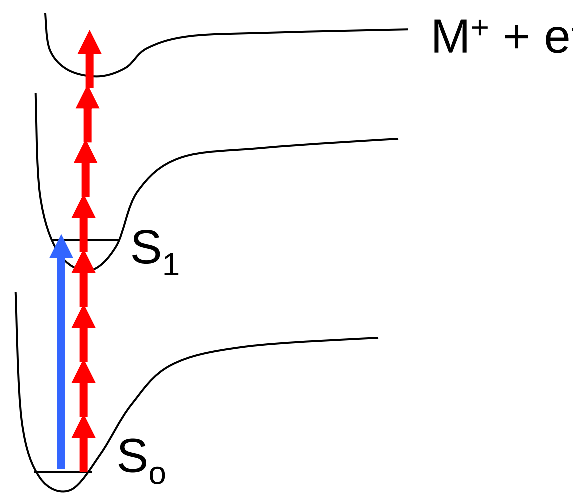
Electronic Excitation

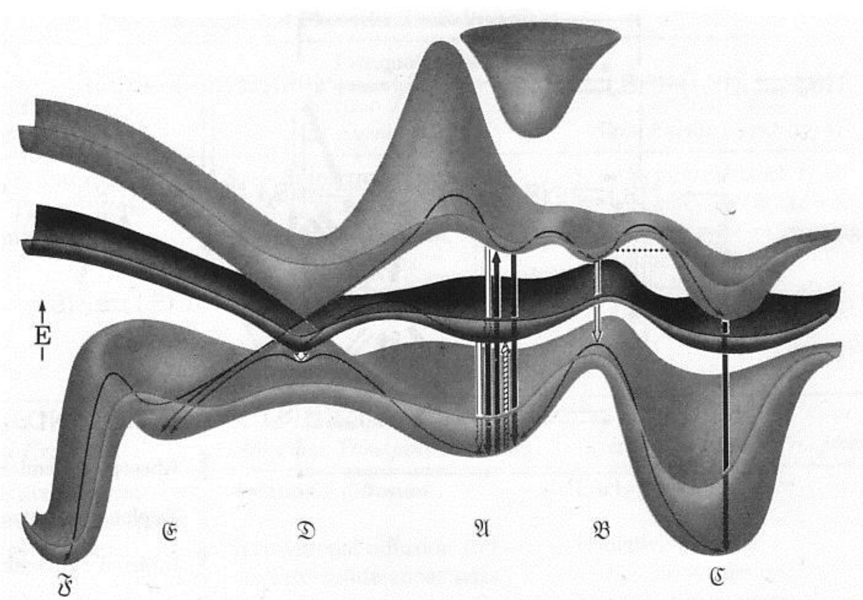


$P_{S1 \leftarrow S0} = I\, \sigma_{S1 \leftarrow S0}$

$P_{n,S1 \leftarrow S0} = I^n\, \sigma_{n,S1 \leftarrow S0}$

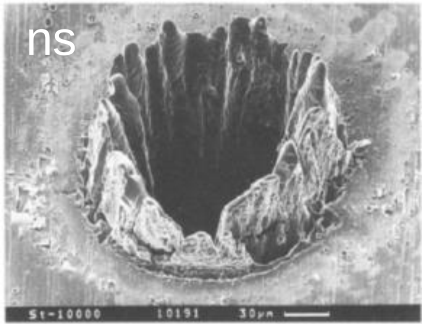
Multiphoton Ionization



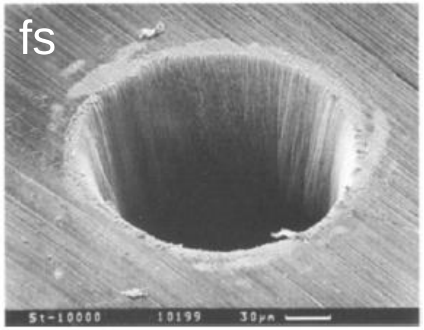


Femtosecond Laser Excitation Leads to Universal Vaporization through Nonresonant Excitation

- Ultrashort pulse durations cause less thermal damage to sample and less fragmentation.
- Femtosecond laser couples into any system through resonant and non-resonant mechanisms.

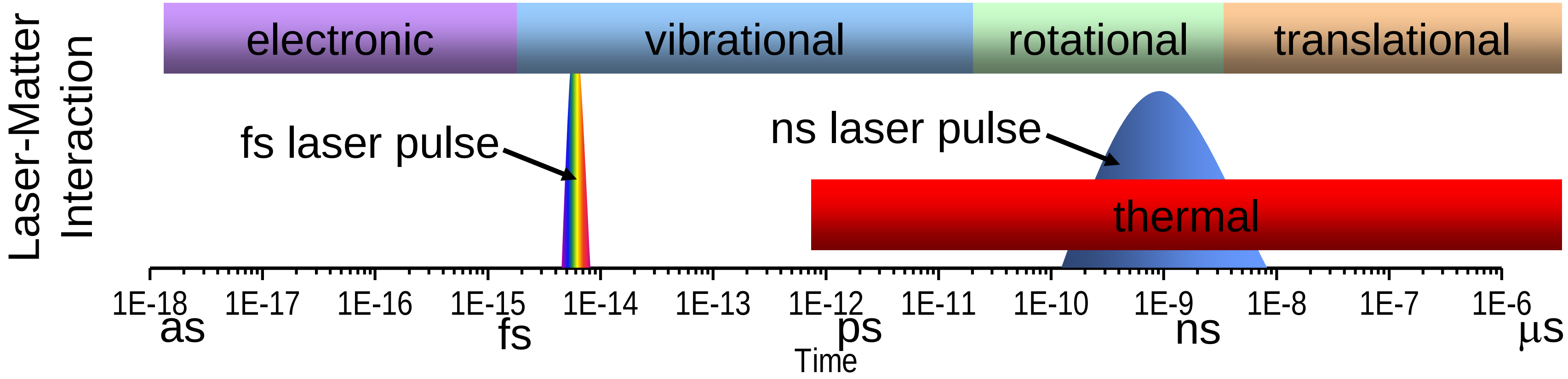


SEM of 100 μm thick steel foil after exposure to 3.3 ns, 1 mJ, F = 4.2 J/cm^2 laser pulses at 780 nm



SEM of 100 μm thick steel foil after exposure to 200 fs, 120 μJ, F = 0.5 J/cm^2 laser pulses at 780 nm

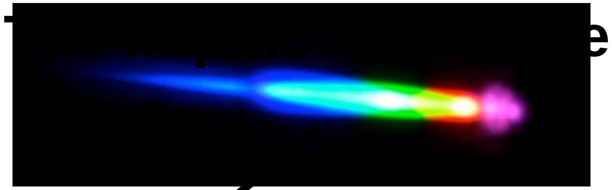
B.N. Chichkov, et al., *Appl. Phys. A.* (1996) 63, 109-115



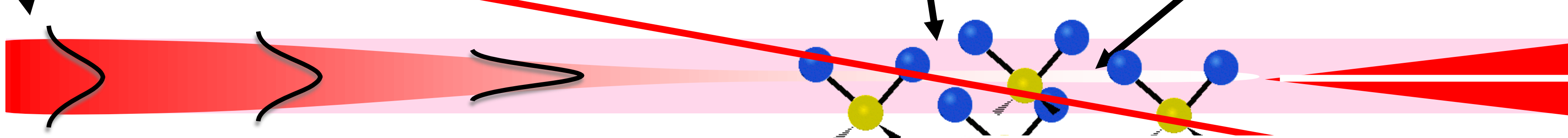
Laser Filaments Form Near Single Cycle Pulses

Pre-Filament
- Kerr lensing → high intensity
- High intensity → self phase modulation

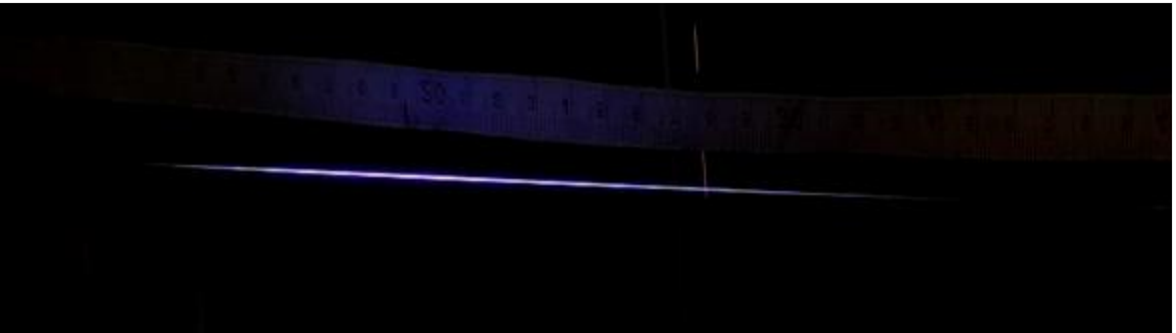
Filament
- High intensity → ionization (10^{16} e^{-} cm^{-3})
- Ionization → intensity clamping 10^{13} W cm^{-2}
- Spatial temporal focusing → self shortening
- Self shortening → < 10 fs pulses

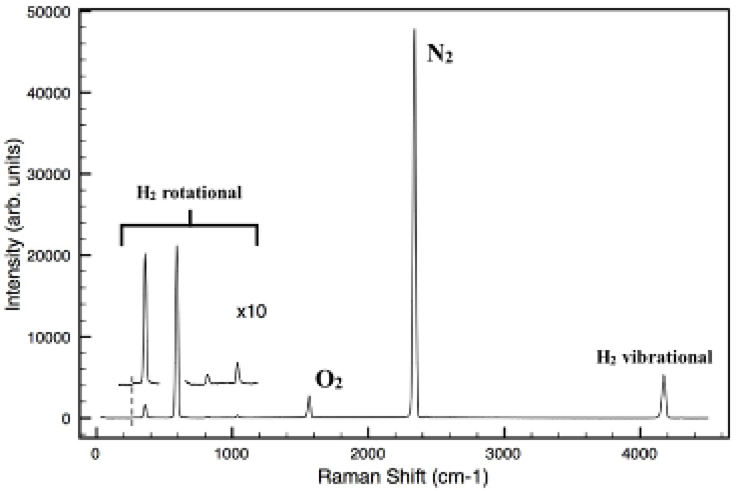


2 mJ 45 fs laser pulse



Short intense pulse provides impulsive rotational and vibrational excitation

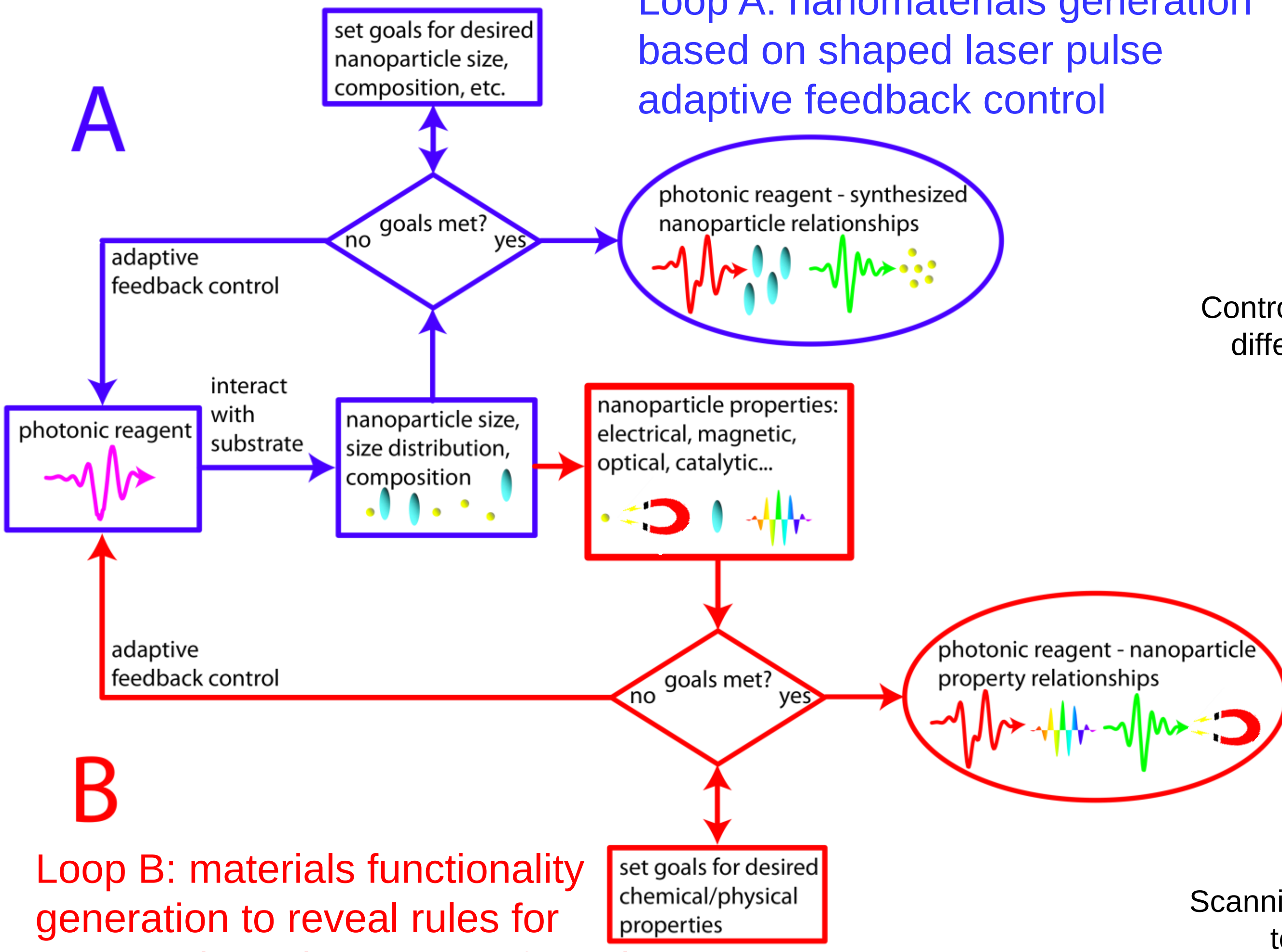




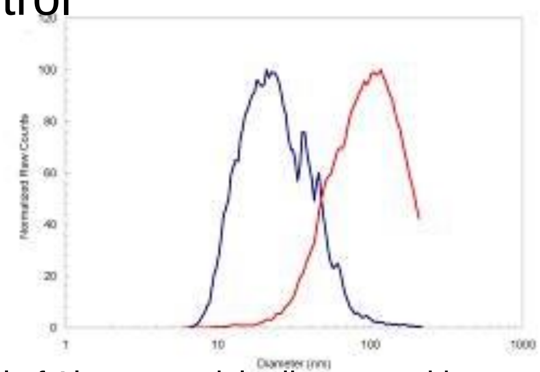
Nanomanufacturing by Design with Shaped Femtosecond Laser Pulses

Shaped laser pulse nanomaterials reactor for determining the rules for femtosecond nanoprocessing using adaptive feedback control

Loop A: nanomaterials generation based on shaped laser pulse adaptive feedback control



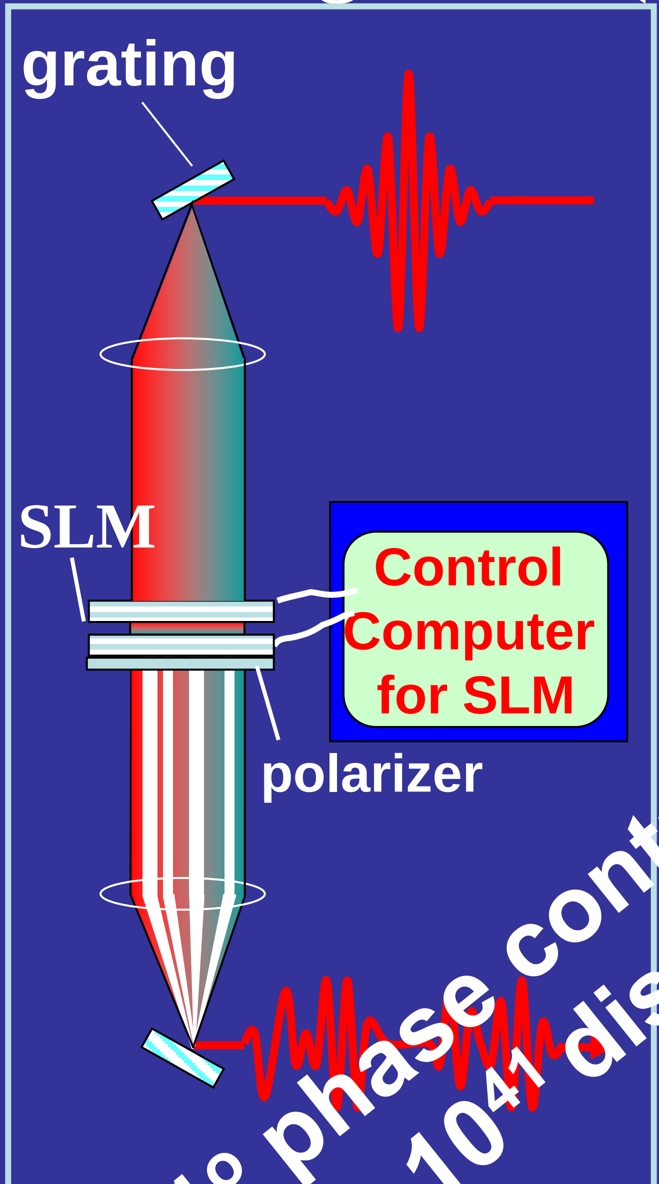
Loop B: materials functionality generation to reveal rules for property-based nanomanufacturing

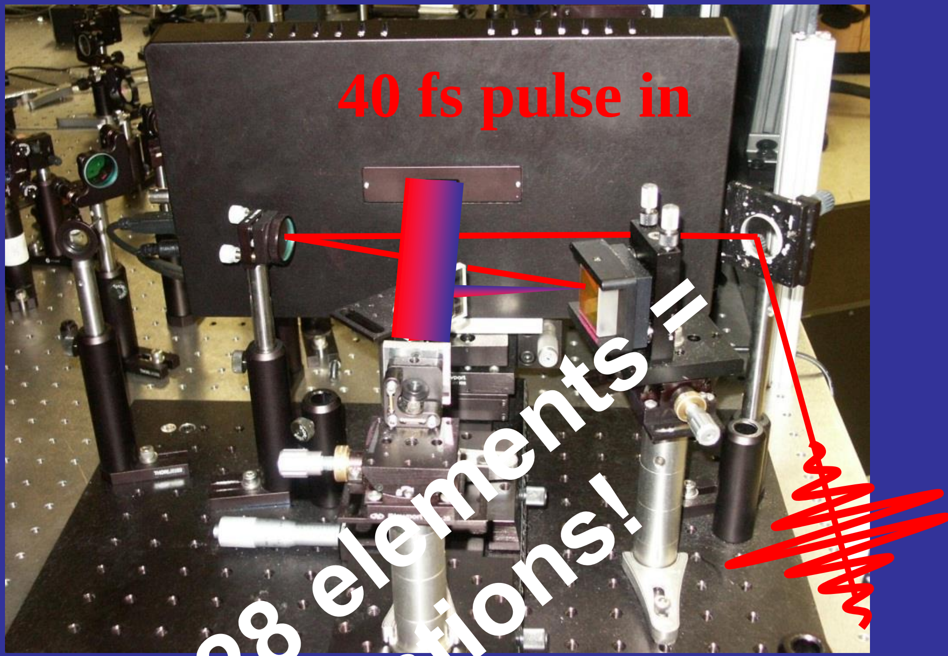


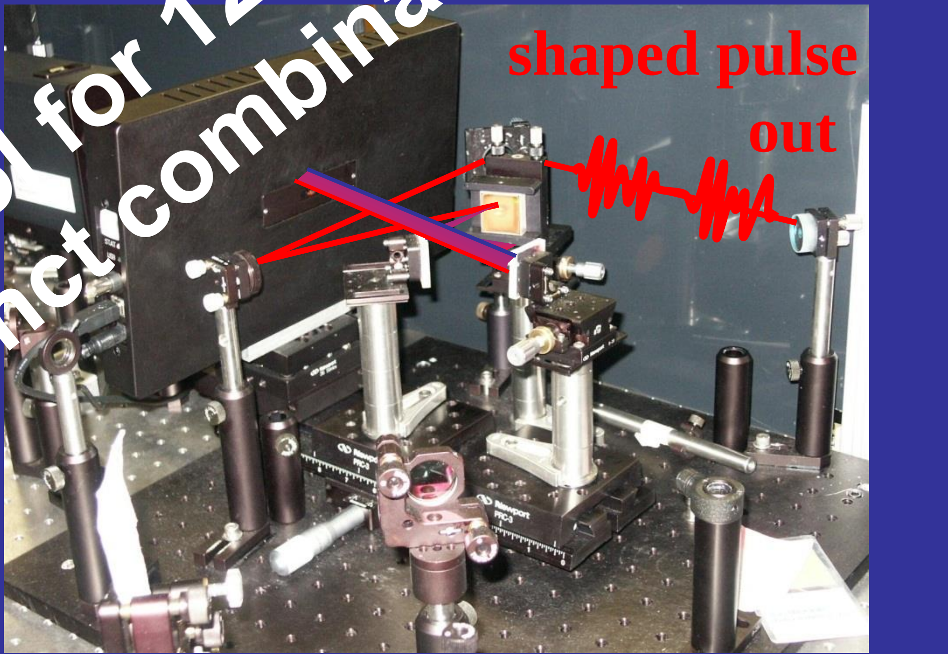
Control of Al nanoparticle diameter with two different femtosecond laser pulse shapes

Scanning mobility nanoparticle size analyzer to generate feedback signal for control

Photonic Reagents, ε(t)

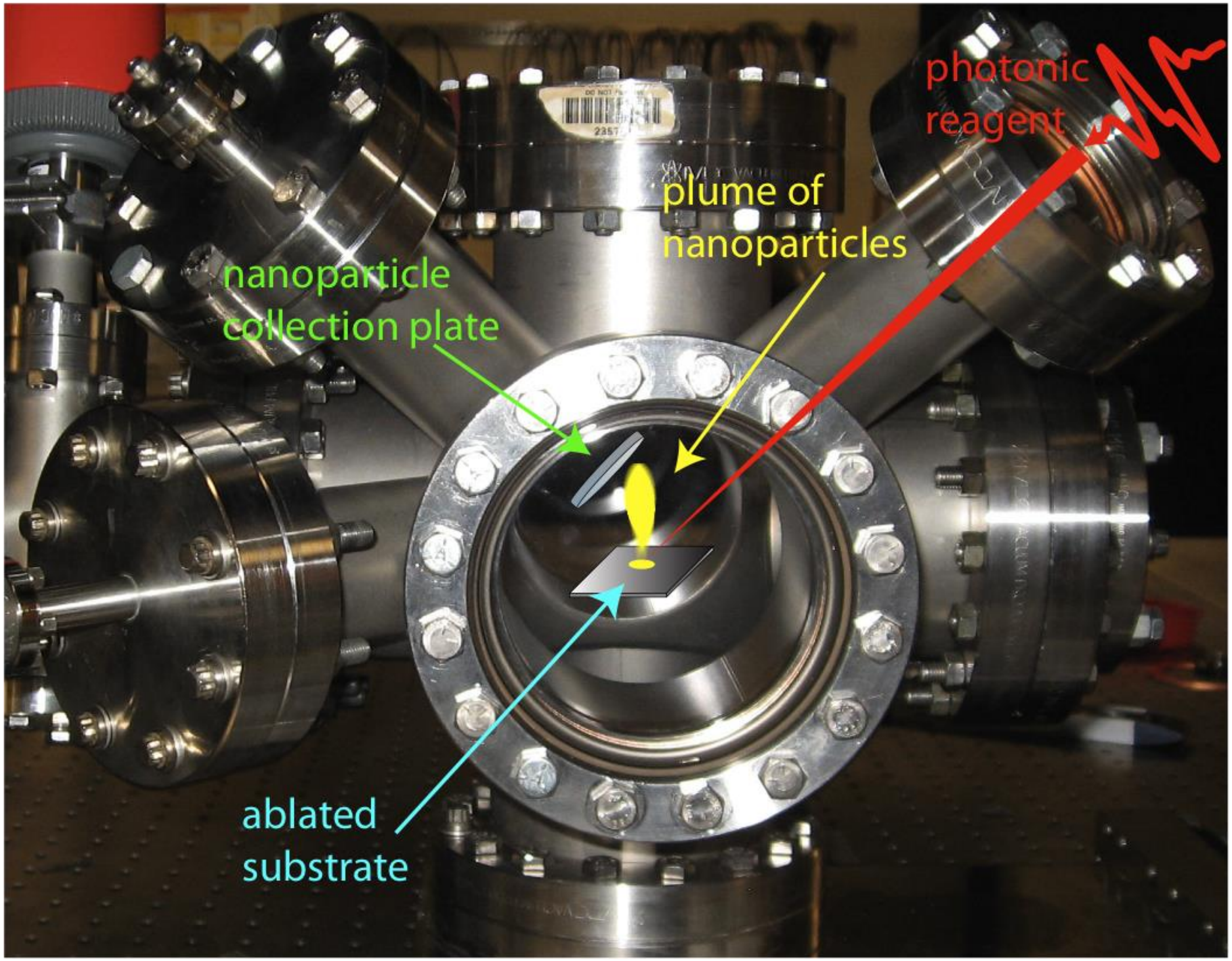




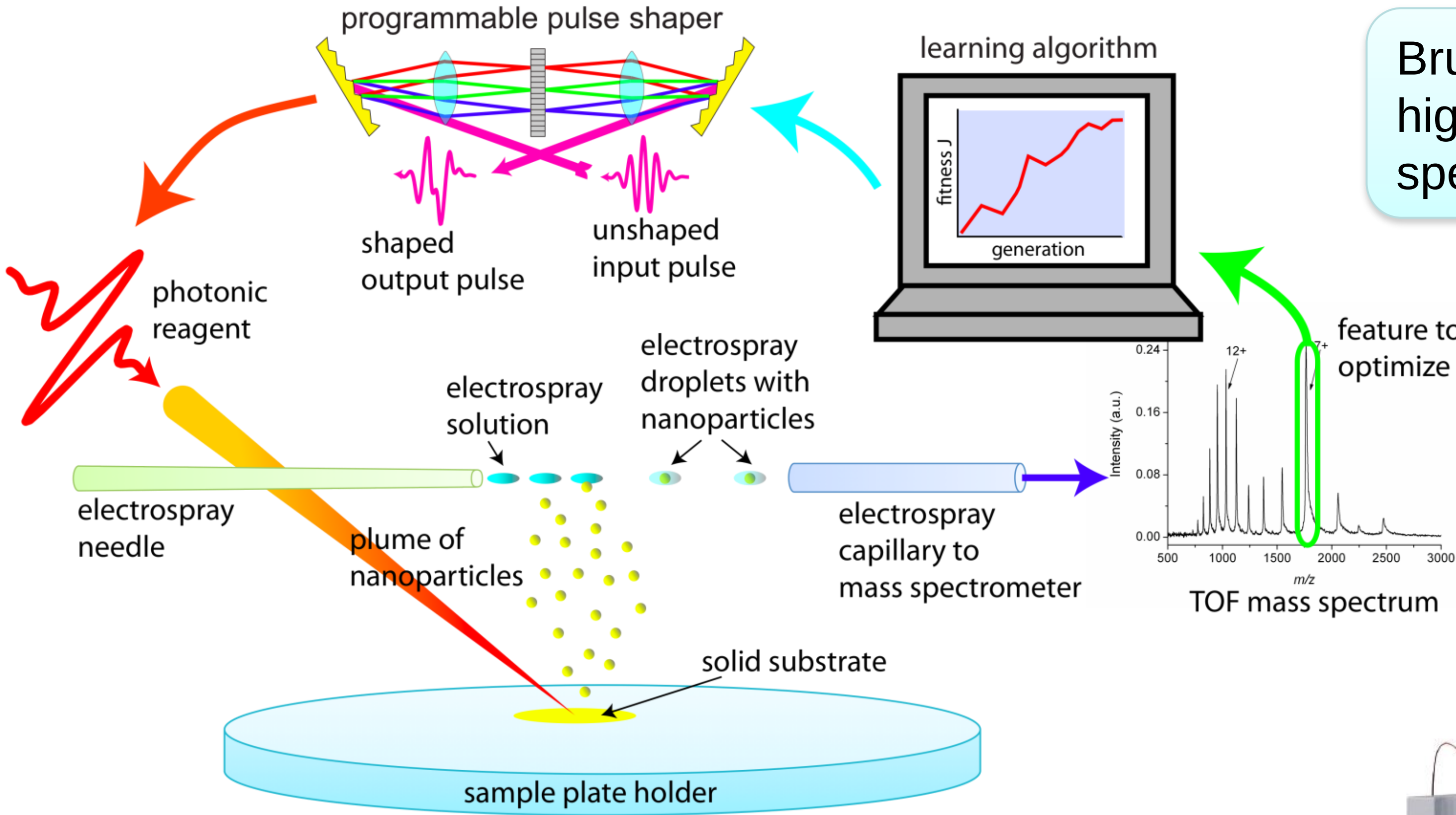


1° phase control for 128 elements =
10^{41} distinct combinations!

Nanomaterials by design reaction chamber



Laser Electrospray Mass Spectrometry for Feedback Control of Nanoparticle Synthesis

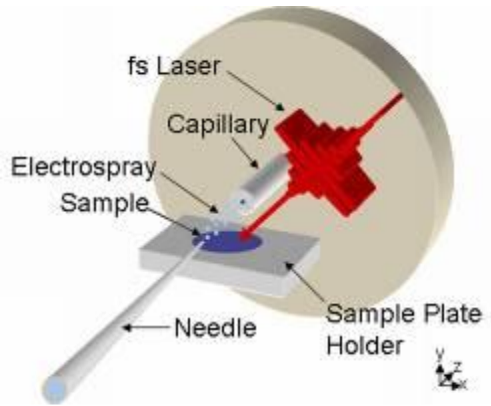


Bruker **micrOTOF** for high-resolution mass spectral analysis

micrOTOF specs

- mass resolution >16,000amu
- mass accuracy <2ppm

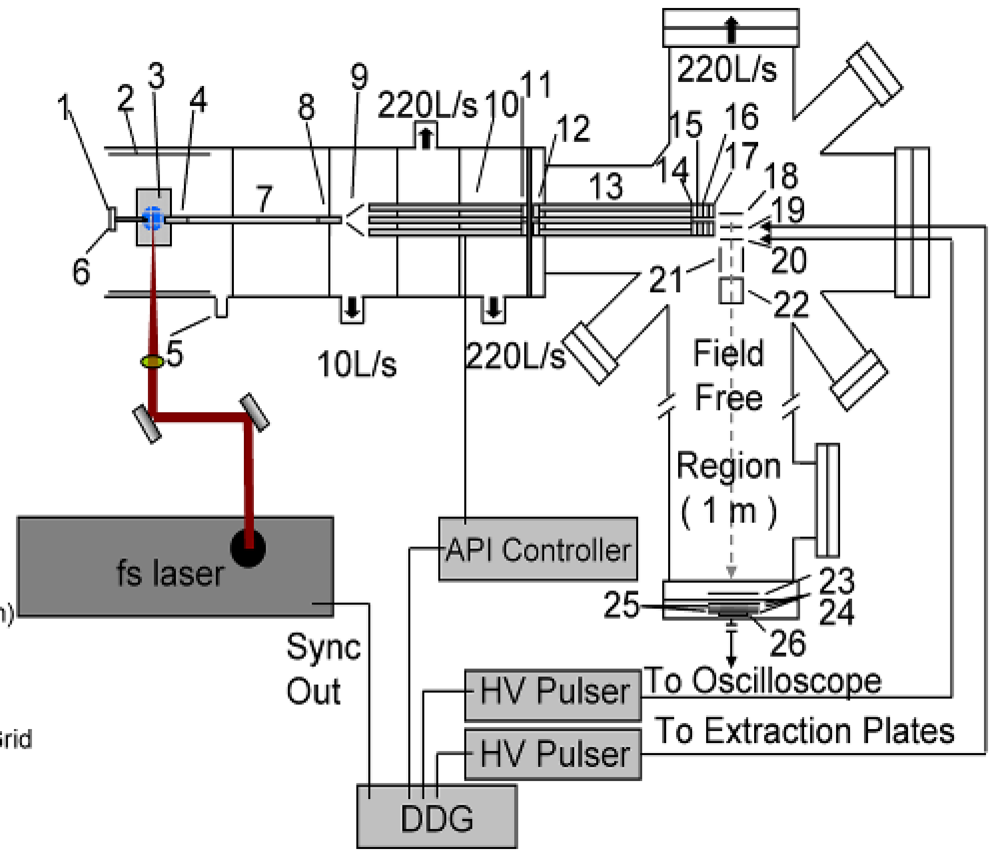
Laser Electrospray Mass Spectrometry System



focus 1 mJ, 50fs pulse to 150 micron diameter spot
10^{13} W cm^{-2}

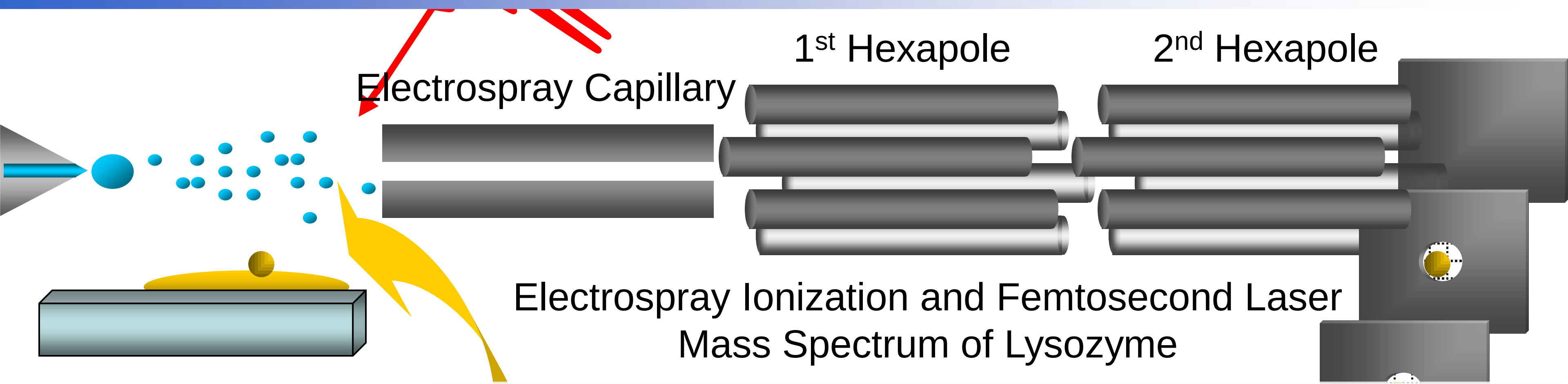
Laser-ESI Source Pulsed Extraction TOF-MS

1. API Needle
2. Source Chamber Electrode
3. Sample Plate
4. Capillary Electrode
5. Dry Nitrogen
6. Nebulizing Nitrogen
7. Glass Capillary
8. Capillary Electrode
9. Skimmer
10. Hexapole Ion Guide
11. DC Lens
12. DC Lens
13. RF Hexapole Ion Guide
14. DC Lens
15. DC Lens
16. X Steering Plates
17. Ground Plate
18. Extraction Plate (mesh)
19. Acceleration Plate (mesh)
20. Ground Plate (mesh)
21. X Steering Plates
22. Y Steering Plates
23. MCP Entrance Screen Grid
24. MCP Bias Plates
25. Microchannel Plates
26. Anode

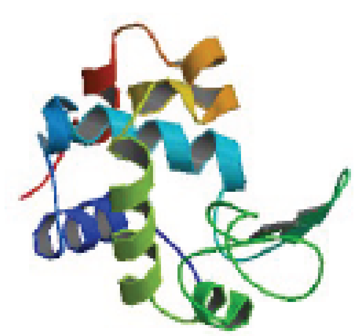


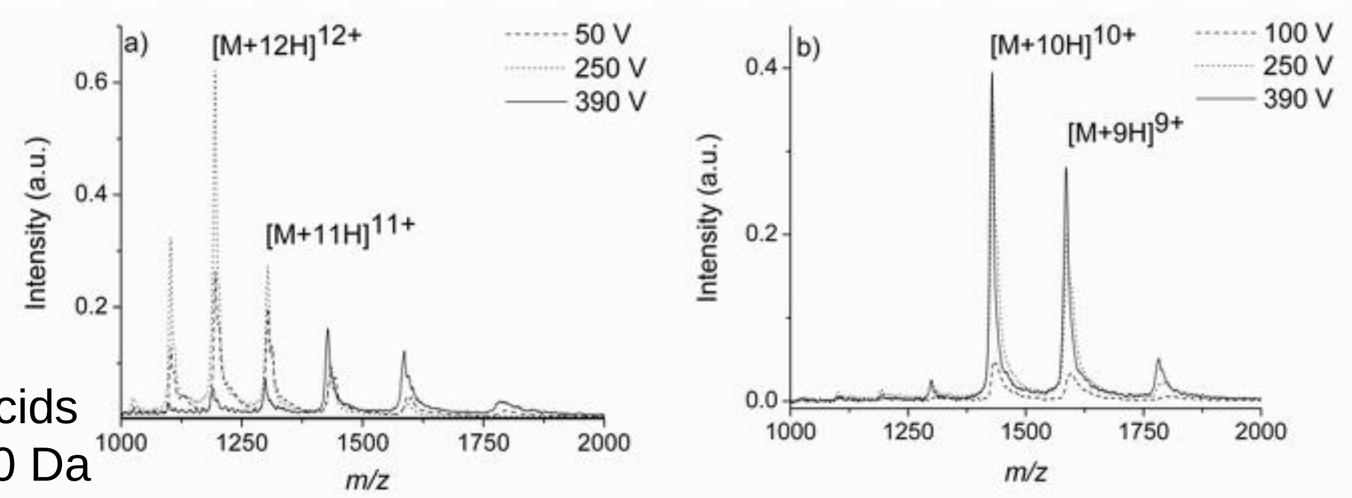
Brady, J.J., Judge, E.J., Levis, R.J., Rapid Commun. Mass Spectrom., **23**, 3151-3157 2009

Femtosecond Non-Resonant Laser Vaporization of Protein Films Provides Universal Analysis



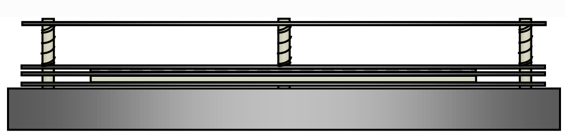
Electrospray Ionization and Femtosecond Laser Mass Spectrum of Lysozyme





Lysozyme 129 Amino Acids
molecular weight 14,300 Da
20 basic AA, 20 acidic AA

No Matrix, Nonresonant Desorption



Detector

Brady JJ, Judge EJ, Levis RJ, 2011; PNAS 108, 12217