Time-resolved scattering

Paul G. Evans

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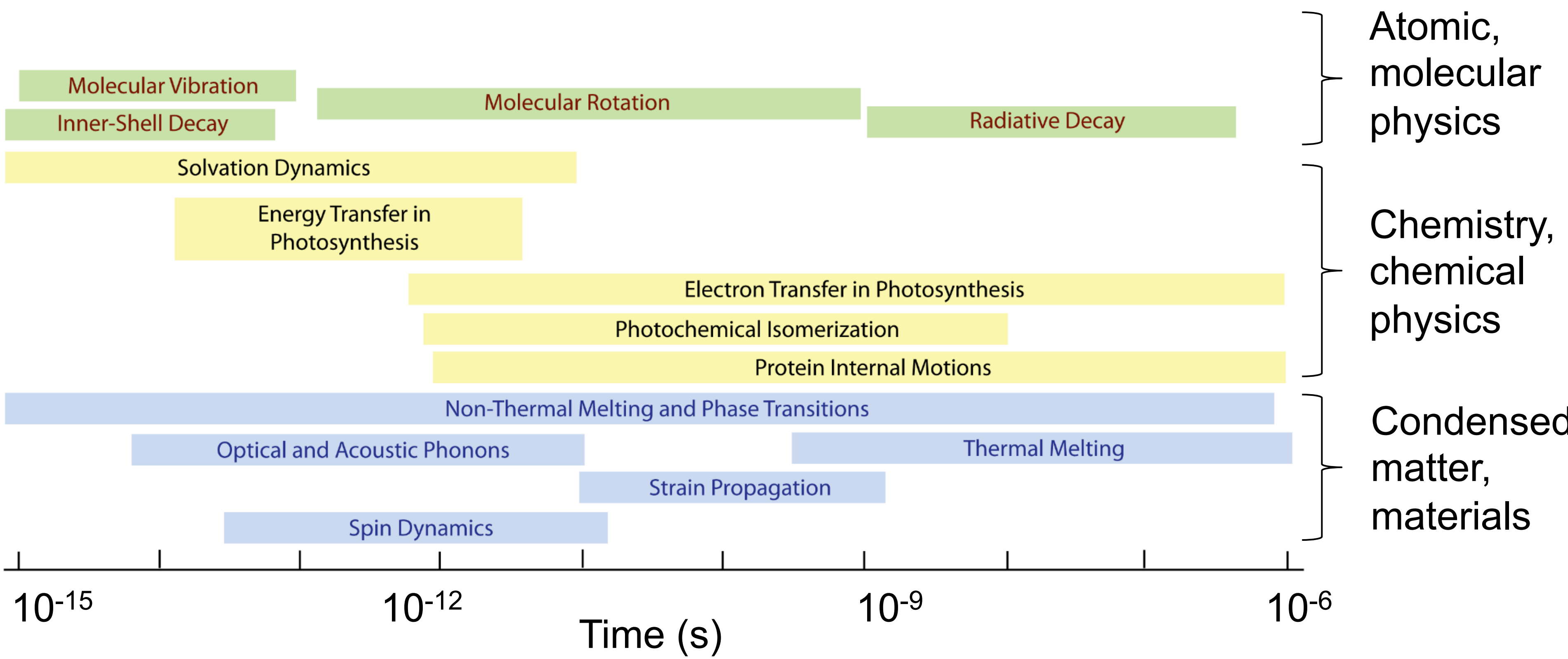
June 20, 2014

Motivations: Timescales of Dynamical Processes

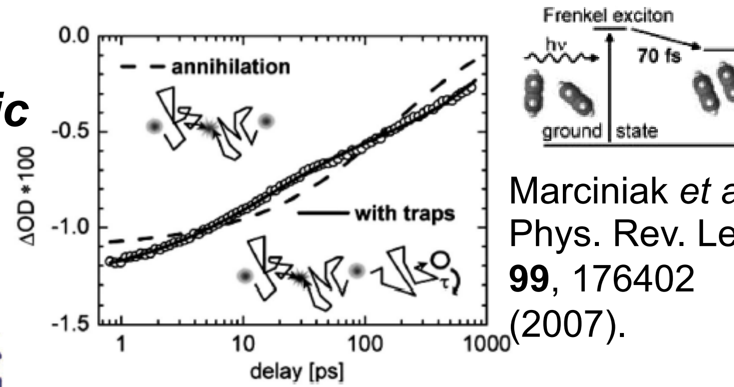


Examples of Dynamical Phenomena in Condensed Matter 1

Fundamental Excitations

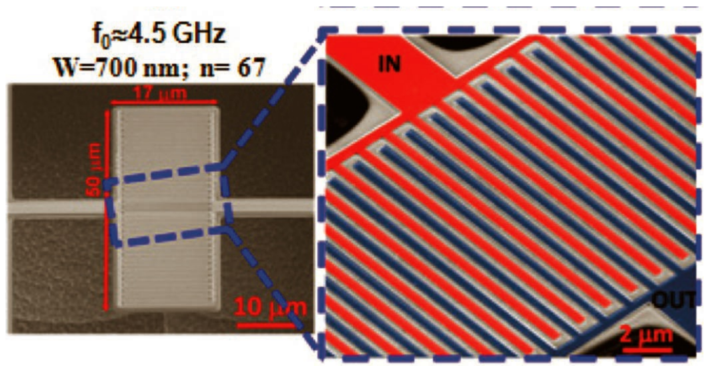
- Structure and Dynamics of Excitons in Organic Semiconductors

- Phonon Generation and Dynamics



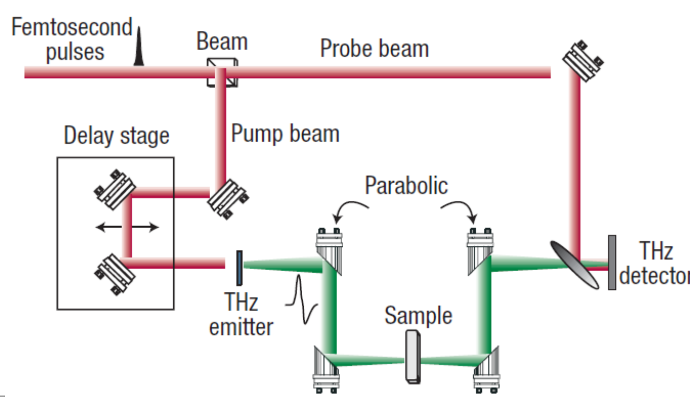
Marciniak *et al.*, Phys. Rev. Lett. **99**, 176402 (2007).

Rinaldi *et al.*, IEEE Trans. on Ultrasonics, Ferroelectrics, and Freq. Control **57**, 38 (2010).



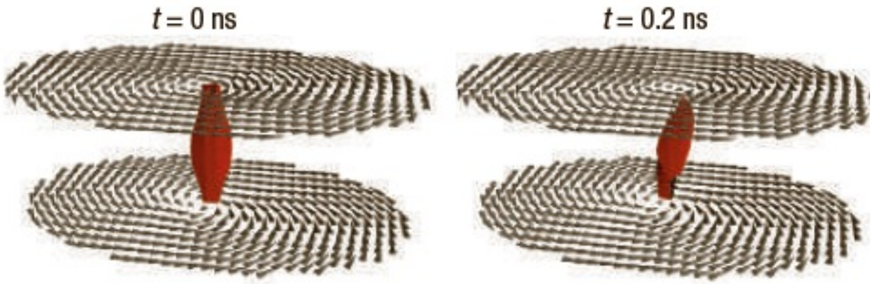
GHz Mechanical Materials and Devices

THz Electronics and THz Properties of Materials



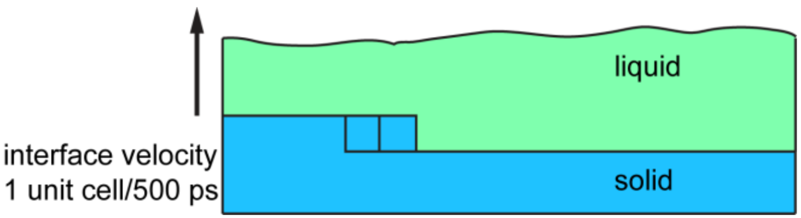
B. Ferguson and X.-C. Zhang, Nature Mater. **1**, 26 (2002).

Examples of Dynamical Phenomena in Condensed Matter 2



Pribiag, *et al*., Nature Phys. **3**, 498 (2007).

Magnetic Dynamics: Spin-transfer Torque



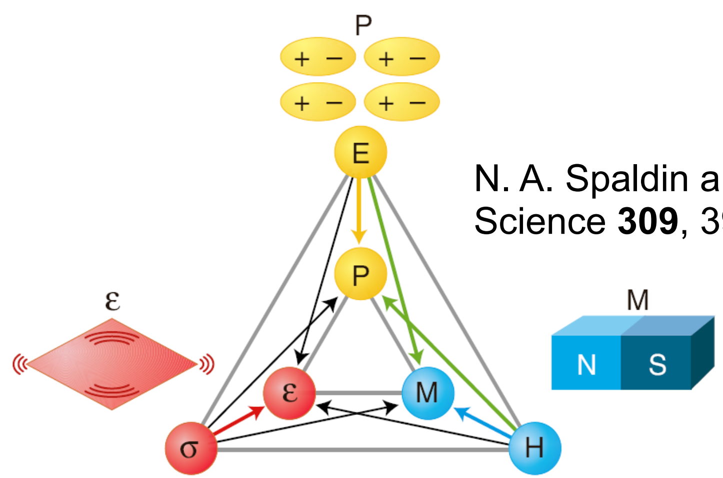
M. A. Scarpula, *et al*., Appl. Phys. Lett. **82**, 1251 (2003).

Atomic and Thermal Transport

- Pulsed Laser Materials Processing

- Nanomaterials Thermal Transport

Dynamics in Complex Oxides



N. A. Spaldin and M. Fiebig, Science **309**, 391 (2005).

Small and Fast Go Together

- Small and fast
  - If you want to look at dynamical processes in small areas you need to be fast
  - If you want to look at small areas the dynamics can be fast.
  - 1 km/sec = 1 nm/ps (~sound)
  - (10^8 m/sec=100 nm/fs)
- Timescales/wavelengths of probes must be chosen to match the problem.
- X-rays ($\lambda \approx 1$ Å) match the size-scale of atomic-to-nanoscale processes.
- **How can we use x-rays to study dynamics?**

What about Bandwidth?

- **Question: Can we just find a way to chop up x-rays and do the same experiments we'd do with longer pulses? At synchrotrons: yes.**
- The energy bandwidth and duration of optical pulses are related by an uncertainty relation called the time-bandwidth product: $\Delta E \Delta t > h$
- A 2 fs visible pulse with λ=600 nm has a bandwidth of nearly 300 nm, spanning nearly the whole visible spectrum.
- X-rays have far higher frequency, and the same frequency width is not a concern, even for fs pulses.

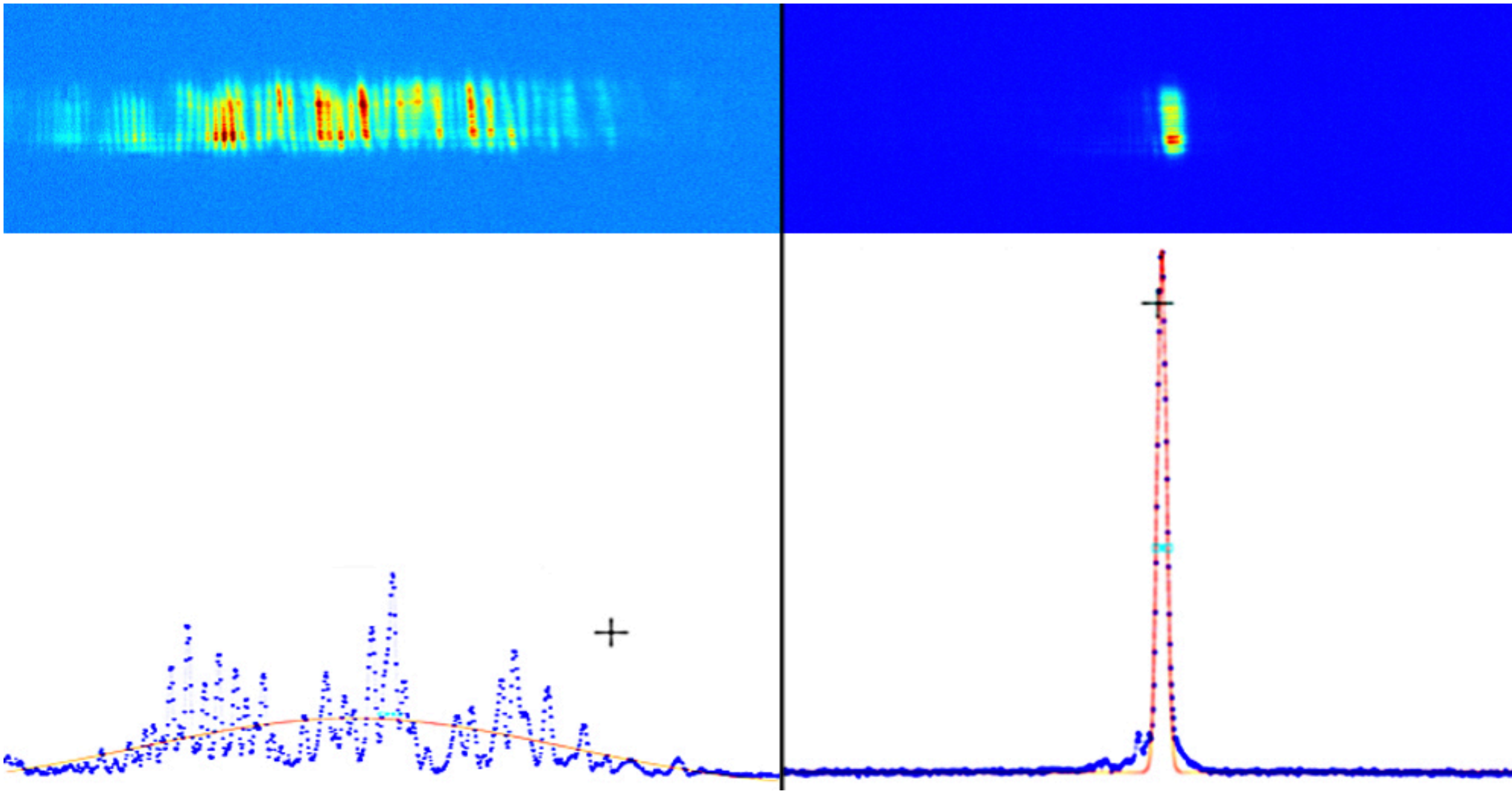
X-ray Sources are Not Yet Transform Limited

- APS: 100 ps duration pulse, 100 eV bandwidth from undulator, typical 1 eV bandwidth selected for diffraction.

  $\Delta E \Delta t$=30000 h (!!!)

- LCLS and other FELs based on spontaneous emission are closer, but not there yet.
- So far, just use the pulsed x-ray sources as short duration lamps.

How Close are FELs? Really Close!



LCLS normal operation: SASI, several modes

LCLS self seeding test 0.5 eV bandwidth at 8 keV

J. Amman *et al*., Nature Photonics **6**, 693 (2012).

What if we were close to the transform limit?

Theory of scattering with transform-limited pulses from electron orbital wavepackets.

PNAS PNAS PNAS

Imaging electronic quantum motion with light

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Edited by Margaret M. Murnane, University of Colorado at Boulder, Boulder, CO, and approved May 1, 2012 (received for review February 7, 2012)

Imaging the quantum motion of electrons not only in real-time, but also in real-space is essential to understand for example bond breaking and formation in molecules, and charge migration in peptides and biological systems. Time-resolved imaging interrogates the unfolding electronic motion in such systems. We find that scattering patterns, obtained by X-ray time-resolved imaging from an electronic wavepacket, encode spatial and temporal correlations that deviate substantially from the common notion of the instantaneous electronic density as the key quantity being probed. Surprisingly, the patterns provide an unusually visual manifestation of the quantum nature of light. This quantum nature becomes central only for non-stationary electronic states and has profound consequences for time-resolved imaging.

X-ray imaging | attosecond science | quantum electrodynamics

The scattering of light from matter is a fundamental phenomenon that is widely applied to gain insight about the structure of materials, biomolecules and nanostructures. The wavelength of X-rays is of the order of atomic distances in liquids and solids, which makes X-rays a very convenient probe for obtaining real-

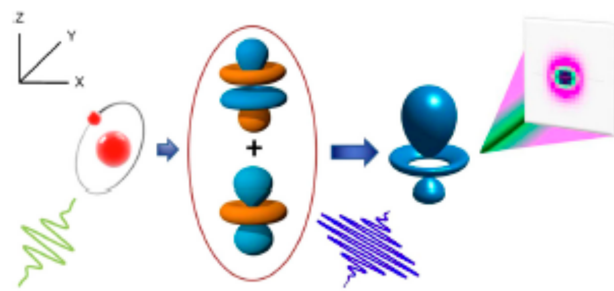


Fig. 1. Schematic of the time resolved X-ray imaging scenario used as an example throughout this work. An electronic wavepacket is prepared by a laser pump pulse (indicated in green) as a coherent superposition with equal population of the 3*d* and 4*f* eigenstates of atomic hydrogen with projection of orbital angular momentum equal to zero. The polarization direction of the generated wavepacket is aligned with the laboratory *z*-axis. The electronic dynamics of the wavepacket is probed by an ultrashort X-ray pulse (indicated in blue) propagating along the *y* direction. A series of scattering patterns obtained by varying the pump-probe time-delay serve to image the electronic motion with high spatial and temporal resolution.

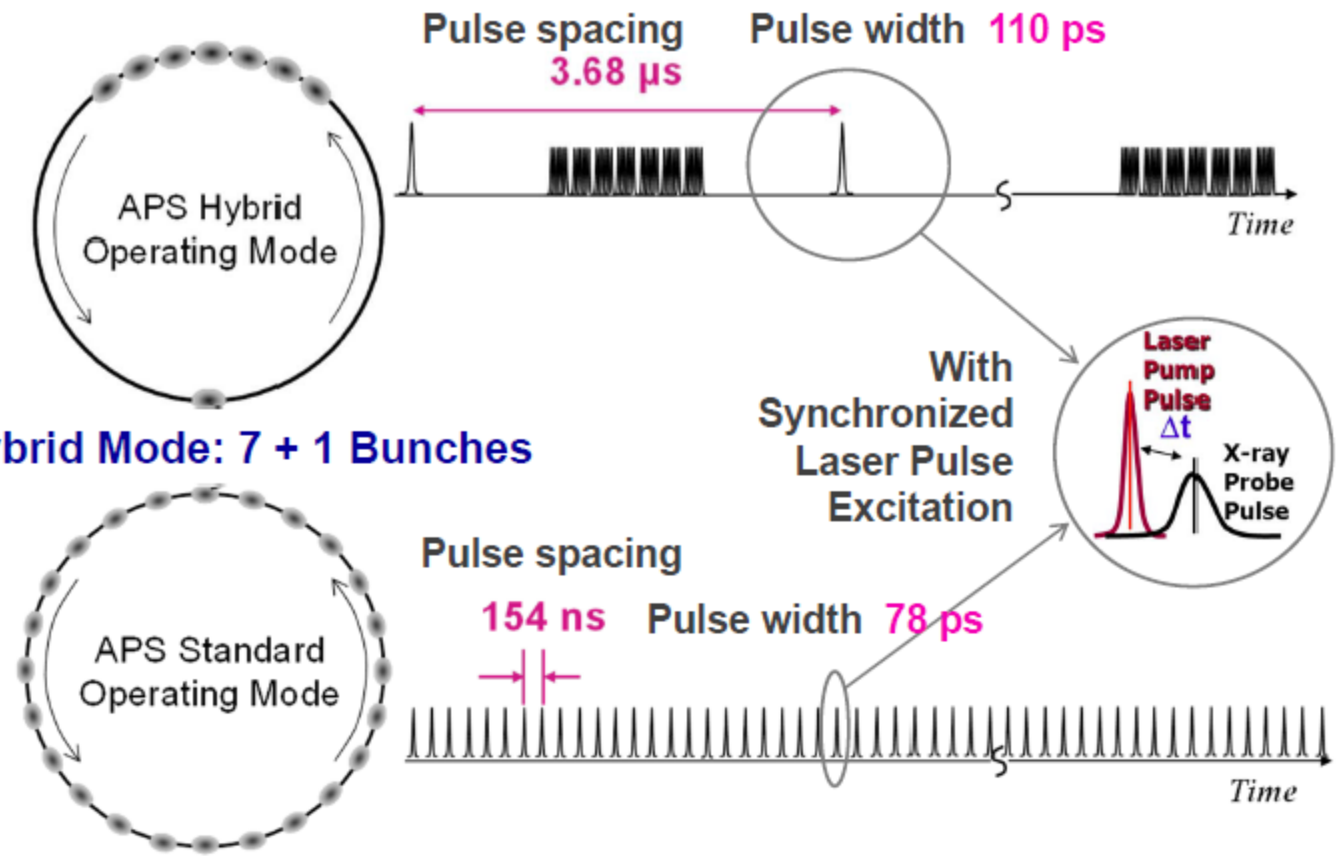
Dixit *et al.*, Proc. Nat. Acad. Sci. **109**, 11636 (2012)

We don't have to worry about this in experiments yet!

Key Parameters and Sources of Short-Duration X-ray Pulses

- Experimental design parameters: pulse duration, x-ray photon energy, repetition rate, photons per pulse
- **Synchrotrons**
  - 40-100 ps duration, 100 eV-50 keV, MHz-GHz repetition rates, 10^3-10^7 photons/pulse
  - (*) Also laser slicing sources, etc. for niche applications with short durations but very low flux.
- **Free electron lasers**
  - LCLS ~100 Hz repetition rate, 800 eV-8 keV, 2 fs, >10^{10} photons/pulse
  - European XFEL: similar, but with ~30 kHz repetition rate

APS Operating Modes: 3 Available



X-ray Pulse Frequency

Hybrid Mode: 7 + 1 Bunches — 272 kHz

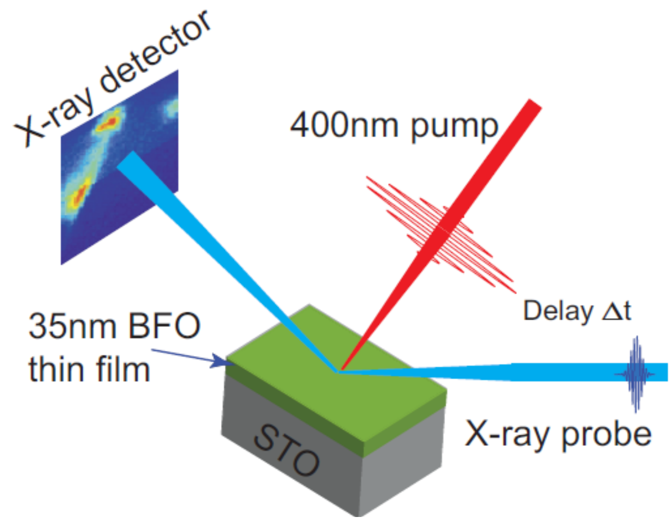
24-Bunch Mode ("Standard") — 6.5 MHz

324-Bunch Mode — Pulse spacing 11.4 ns, Pulse width 50 ps — 88.1 MHz

D. Tiede (ANL)

Experimental Strategies 1: Diffraction from Thin Films

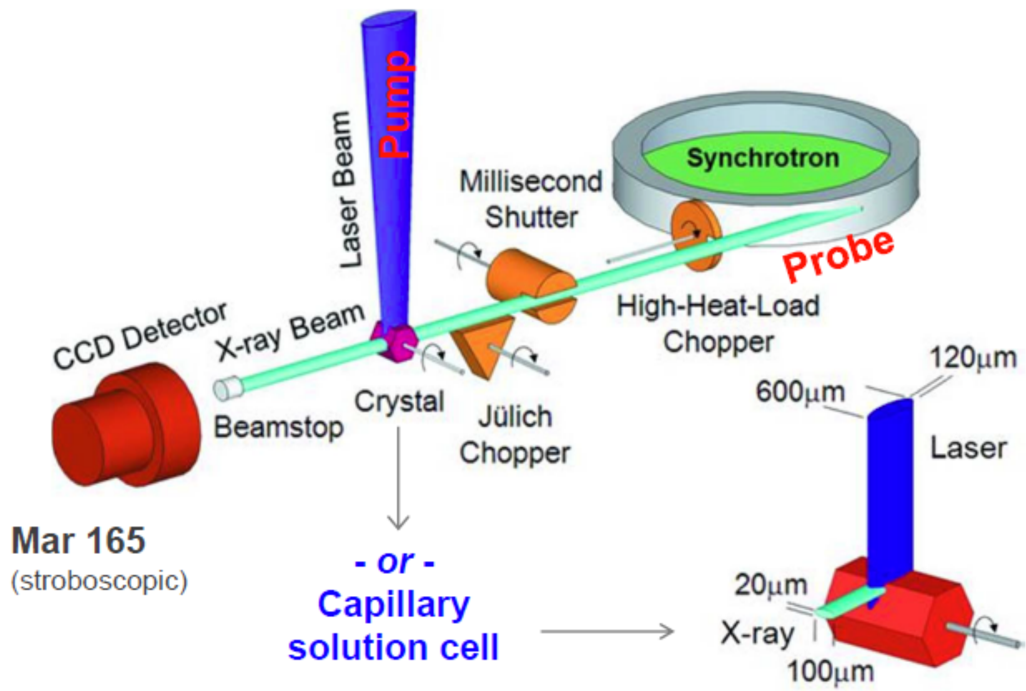
- Excite sample with a short transient, e.g. a laser pulse
- Repeat diffraction experiment at a series of times.



H. Wen *et al.*, Laser-driven strain in $BiFeO_3$ thin flims, *submitted*
APS ID-7

Experimental Strategies 2: Solution Scattering

Beamline Diagram for BioCARS APS ID-14



Source: Graber et. al. J. (2011) J. Synchrotron Rad. 18: *online*

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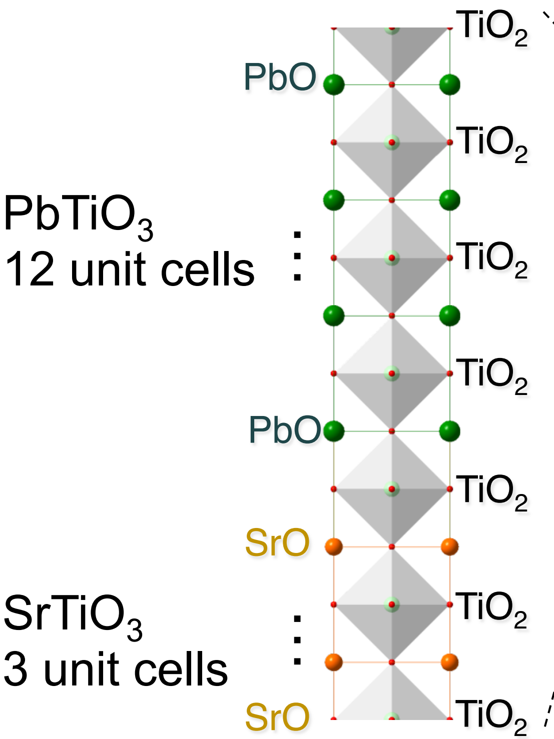
Examples of Experiments

1. **Condensed Matter and Materials Science**

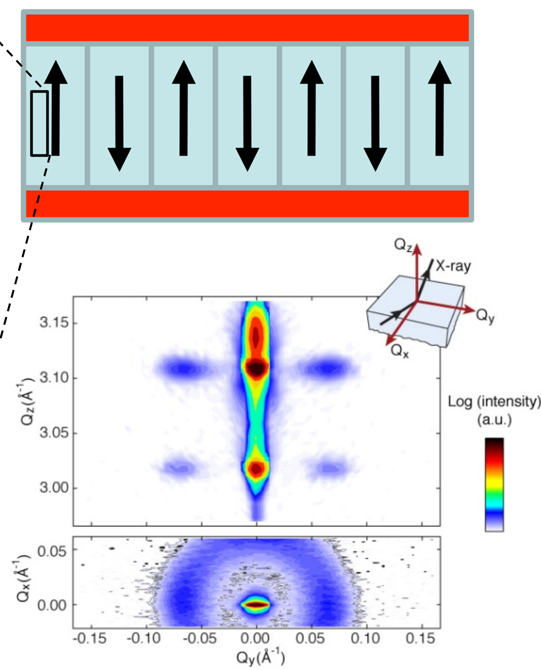
2. Chemical Transformations

3. Biological Molecules

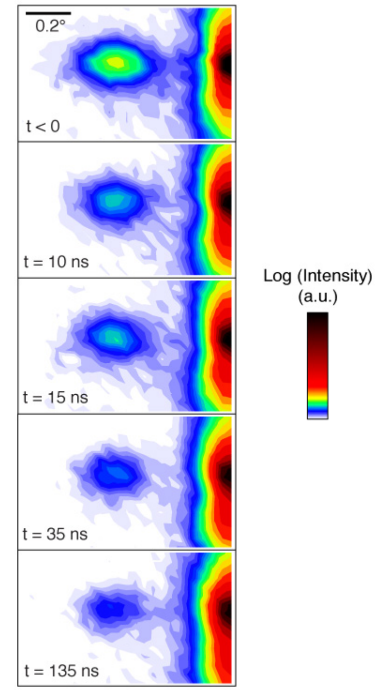
Example 1: Dynamics in artificial ferroelectrics: ferroelectric/dielectric superlattices



Self-organized nm-scale domains



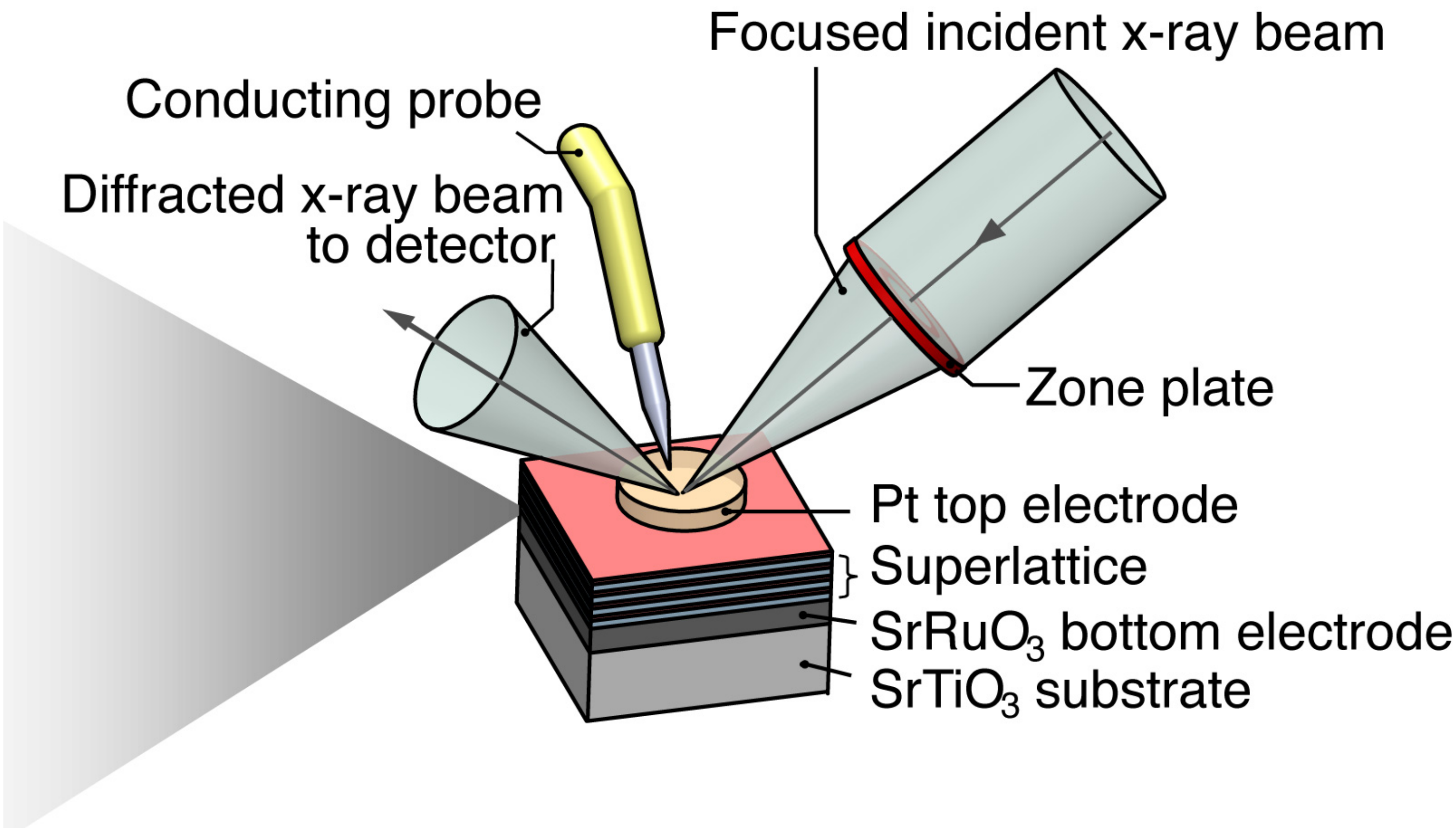
Evans group, in preparation (2011).

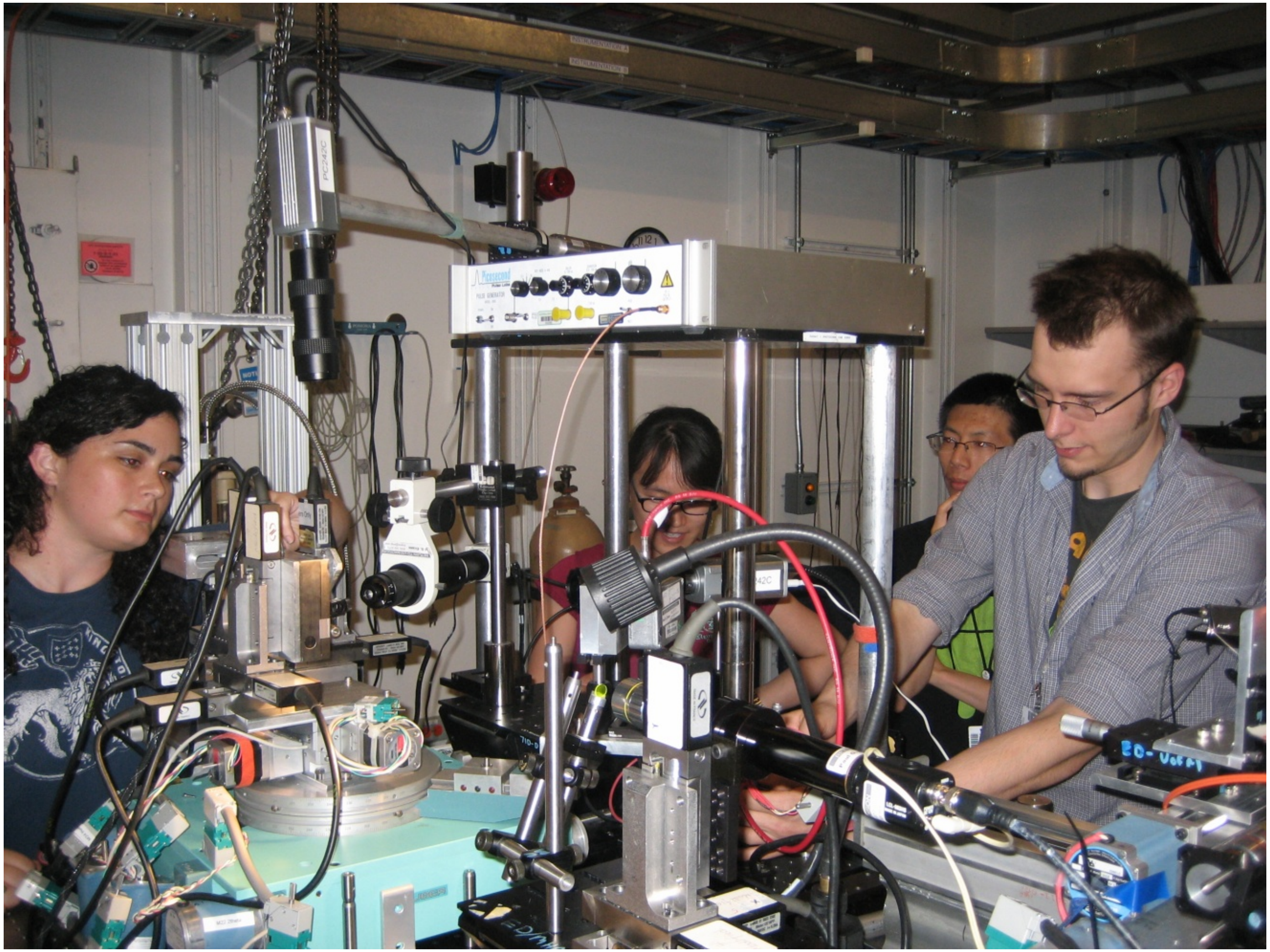


Electronic properties arise from the E-field driven reconfiguration of domains.

Probing ultrafast *structural* dynamics is crucial. Future sources will provide insight into the mechanism of switching and the field-distorted structure of the superlattice.

Time-Resolved Diffraction: $SrTiO_3/PbTiO_3$





Nanodiffraction at APS sector 7: 100 nm spot size, ~100 ps time resolution

Domains

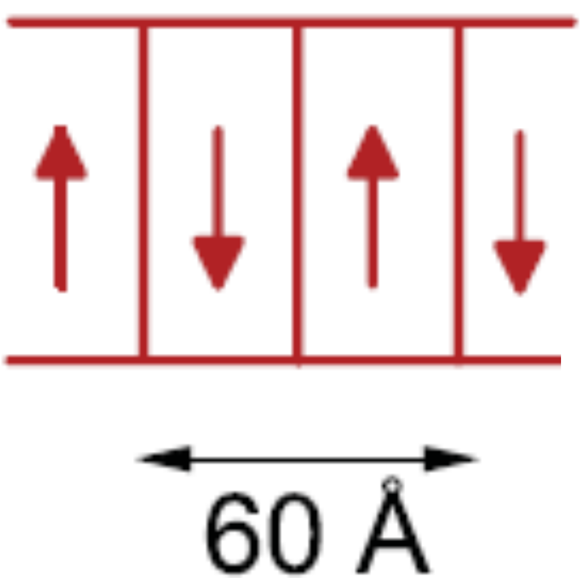
PRL **104**, 187601 (2010) PHYSICAL REVIEW LETTERS week ending 7 MAY 2010

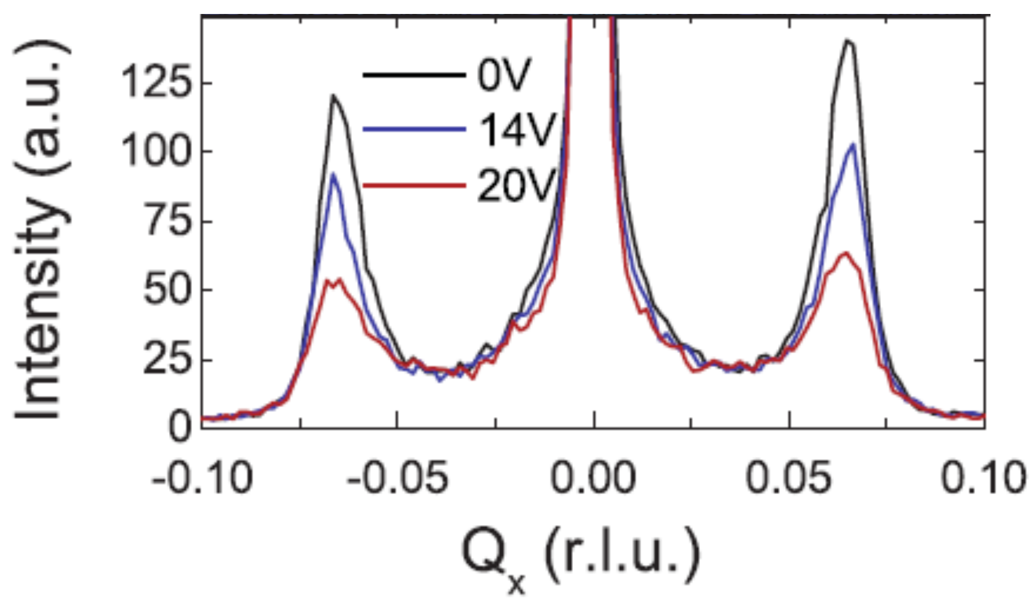
X-Ray Diffraction Studies of 180° Ferroelectric Domains in $PbTiO_3/SrTiO_3$ Superlattices under an Applied Electric Field

P. Zubko,* N. Stucki, C. Lichtensteiger, and J.-M. Triscone
Department of Condensed Matter Physics, University of Geneva, 24 Quai Ernest-Ansermet, 1211 Geneva 4, Switzerland
(Received 24 November 2009; published 7 May 2010)

The dielectric response of $PbTiO_3/SrTiO_3$ superlattices is studied using electrical and structural measurements. While the dielectric response of paraelectric superlattices is well accounted for by the lattice contribution, superlattices with ferroelectric compositions exhibit an enhanced permittivity. X-ray diffraction allowed the presence of ordered nanodomains in ferroelectric superlattices to be established and their displacement under an applied bias to be directly probed, demonstrating that the enhanced permittivity in these artificial materials is due to domain wall motion.

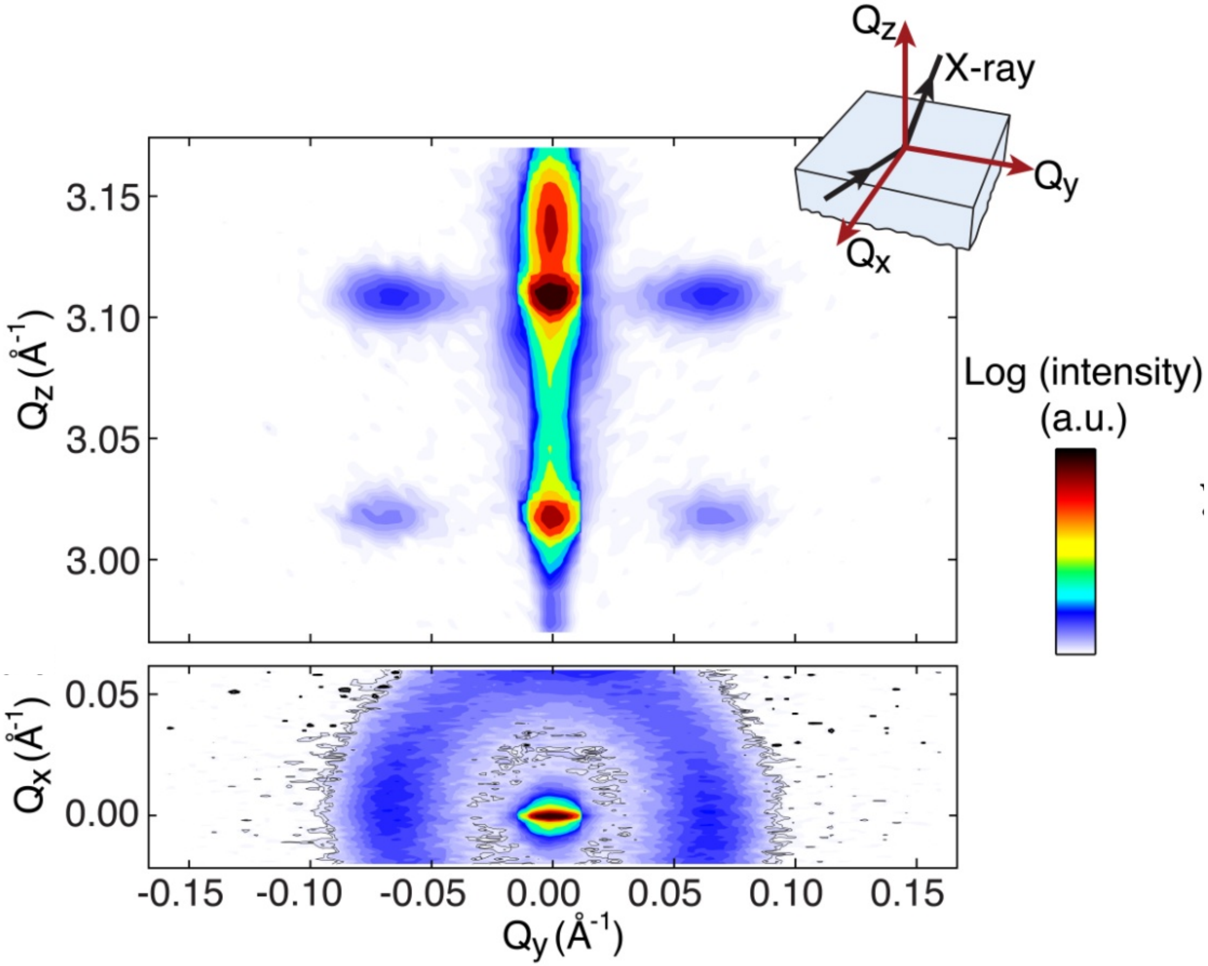
DOI: 10.1103/PhysRevLett.104.187601 PACS numbers: 77.80.Dj, 61.05.C−, 77.84.Cg, 77.84.Ek





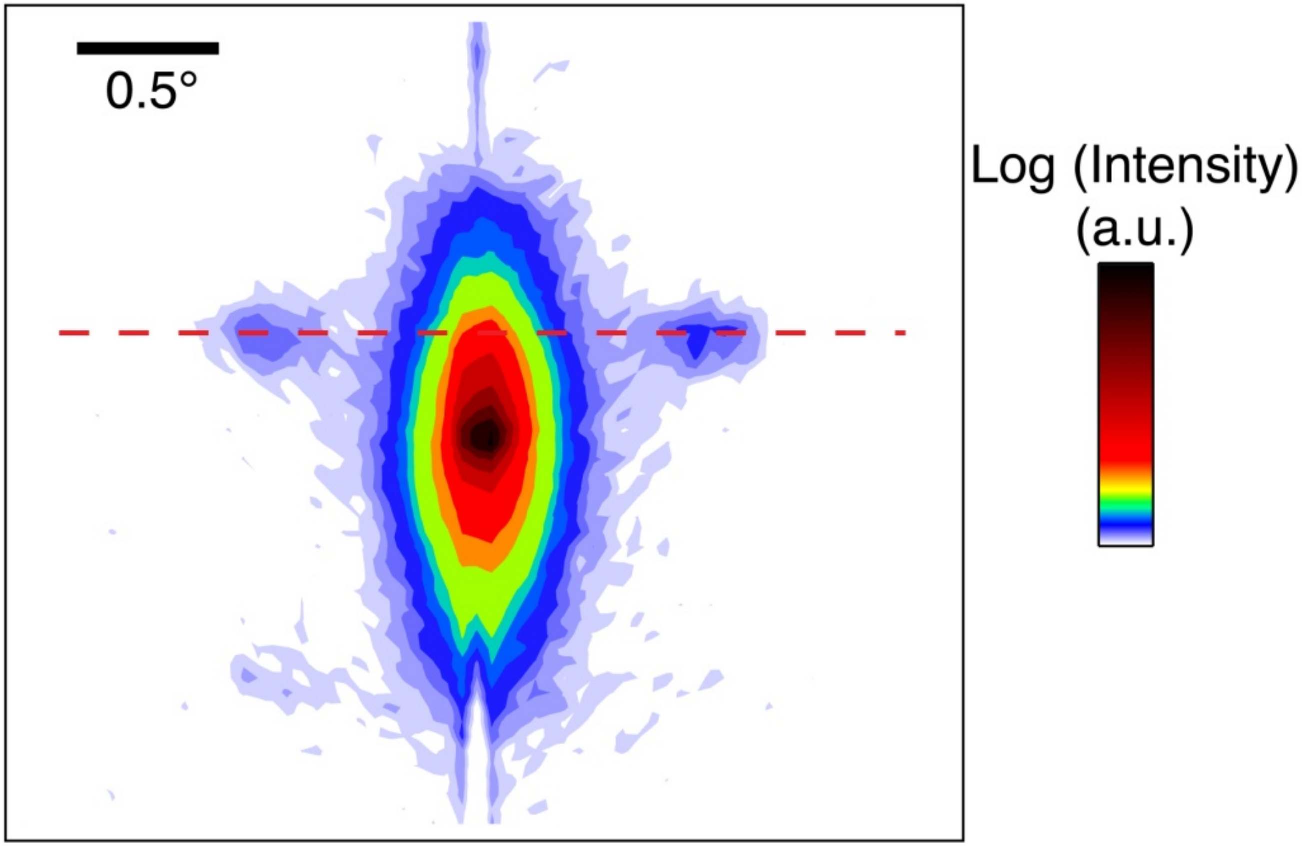
Zubko, *et al*., Phys. Rev. Lett. **104**, 187601 (2010)

Domain Diffuse Scattering in 12($PbTiO_3$)/ 3($SrTiO_3$)



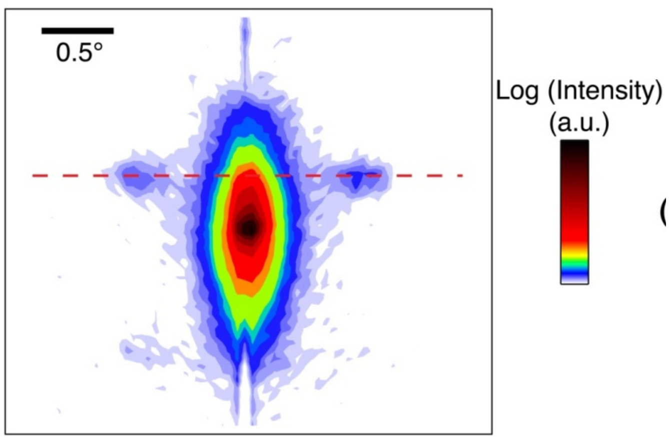
Domain period 9.5 nm
Coherence length only 20 nm

Piezoelectric Expansion in the Superlattice Structural Reflection

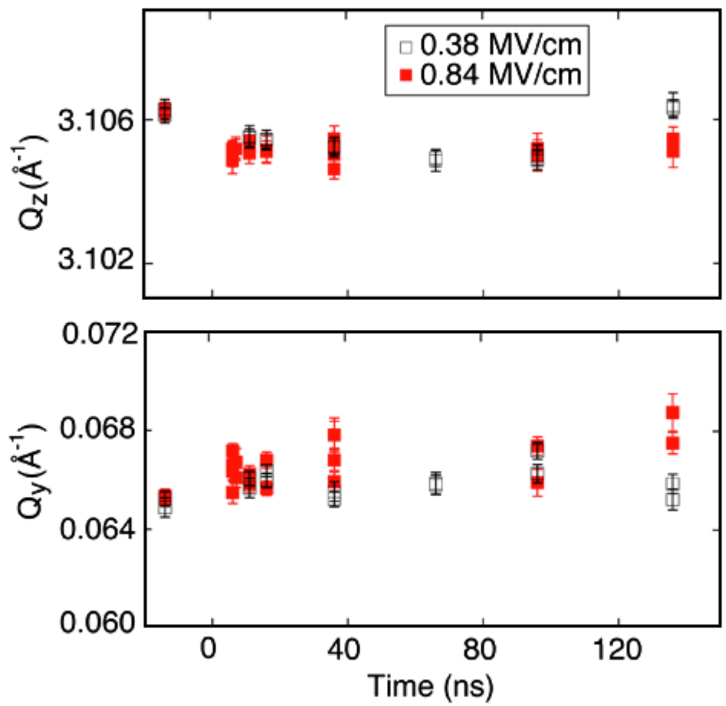


Piezoelectric coefficient: 36 pm/V

No Piezoelectric Expansion within Domains



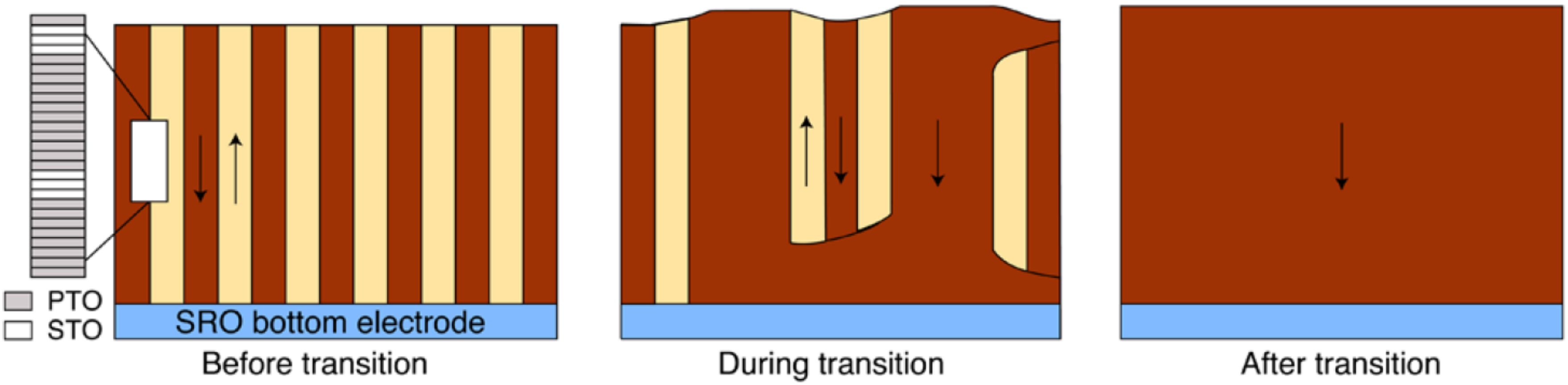
Domain wavevectors



Out-of-plane

In-plane

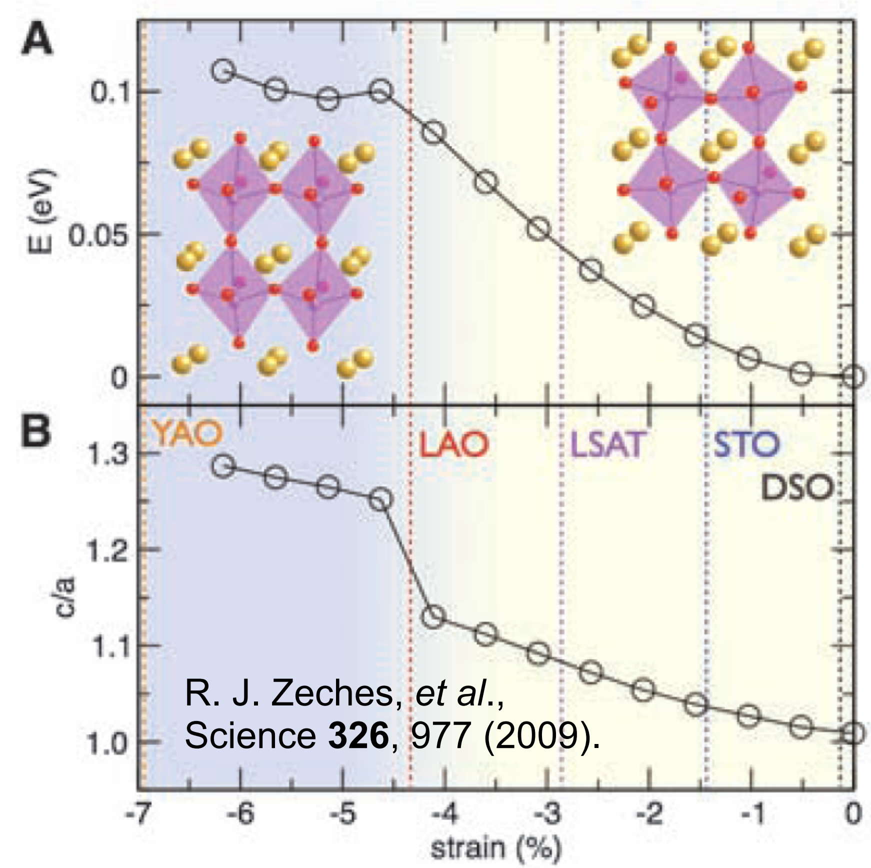
Proposed Switching Mechanism



Challenges: 2D (or 3D) imaging of domains? During switching?

Timescales for Structural Phase Transitions

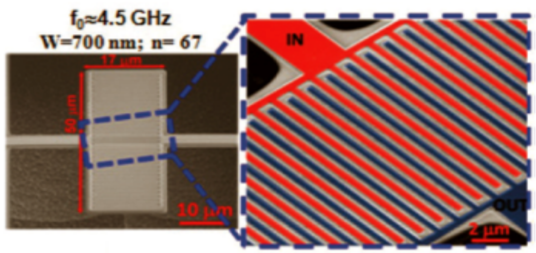
- Strain drives a transition between rhombohedral-like and tetragonal-like phases – and causes huge distortion.
- What are the dynamics of these structural transitions? Expect that it proceeds at the sound velocity, ~1 nm/ps.
- Pump with THz radiation, probe with X-ray pulses.



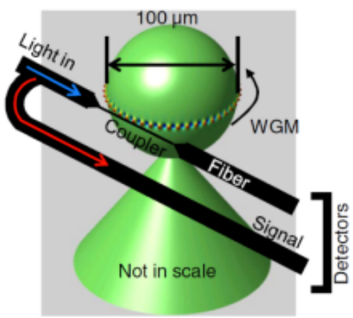
R. J. Zeches, *et al.*, Science **326**, 977 (2009).

"Morphotropic" phase boundary in $BiFeO_3$

Example 2: Ultrafast Mechanics



4.5 GHz NEMS resonator
Rinaldi *et al*., IEEE Trans. on Ultrasonics, Ferroelectrics, and Freq. Control **57**, 38 (2010).

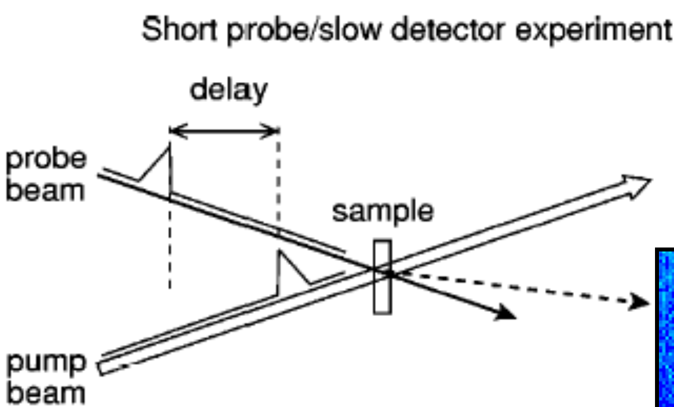


11 GHz whispering gallery mode resonator
M. Tomes and T. Carmon, Phys. Rev. Lett. **102**, 113601 (2009).

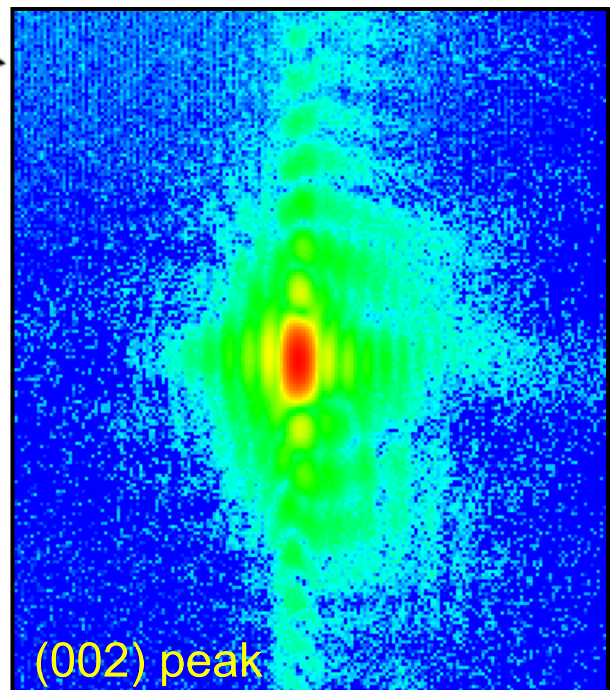
- **High *Q* and high frequency are desirable but highly challenging in materials and device design.**
- **What are the mechanical modes?**
- **Where does nonlinearity come from?**

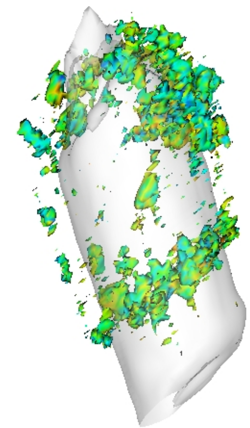
100 ps is not fast enough to capture the relevant effects.

Ultrafast Coherent Diffraction Imaging of Strained Nanostructures

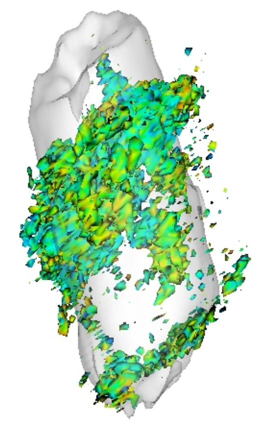


Ni-capped ZnO nanorod coherent diffraction





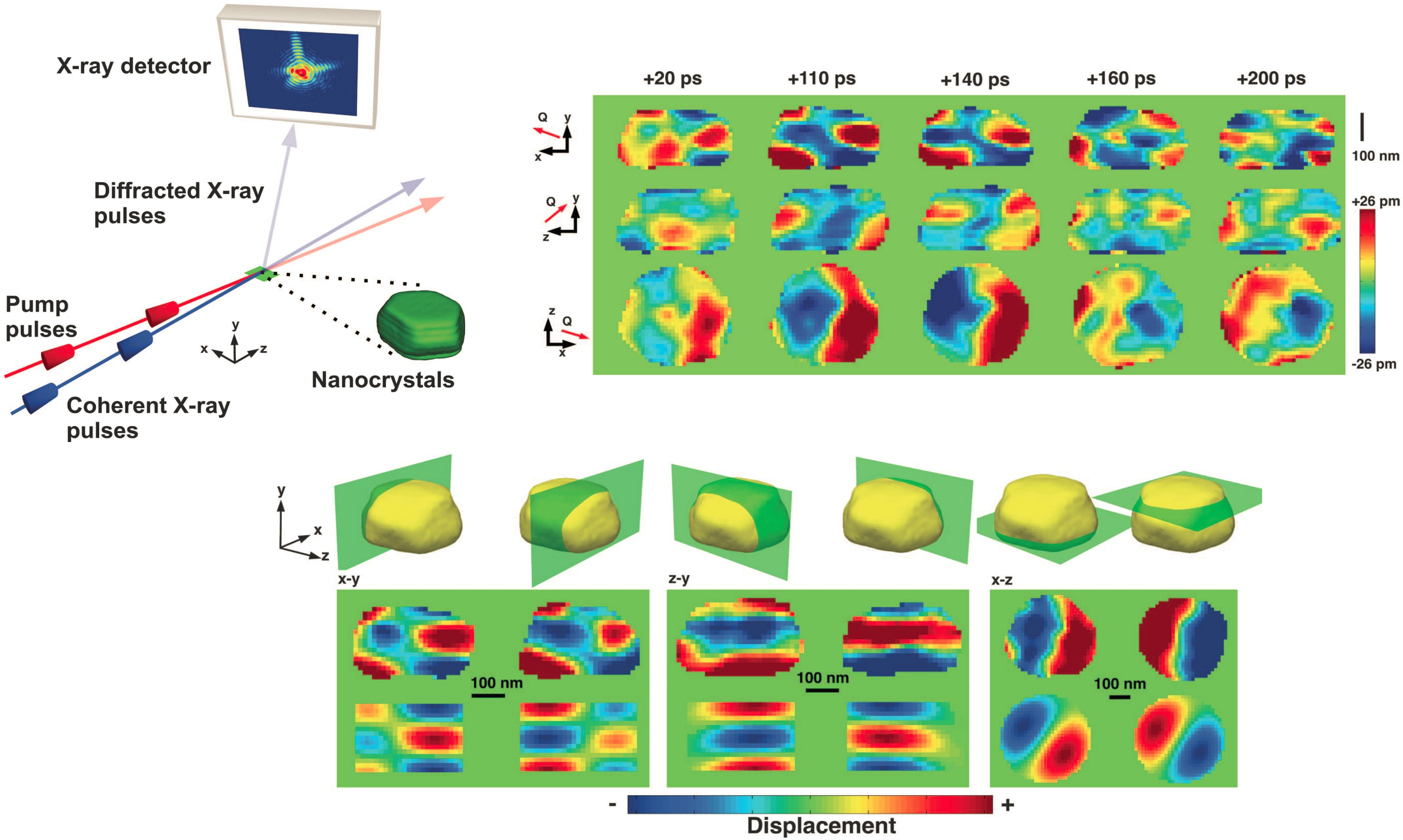
Time zero



Time +50ps

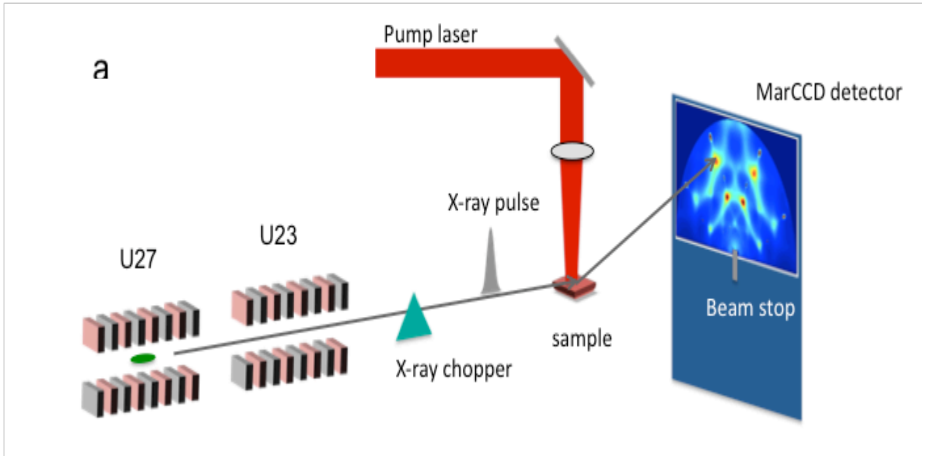
R. Harder (ANL)

Acoustic Modes of Nanocrystals



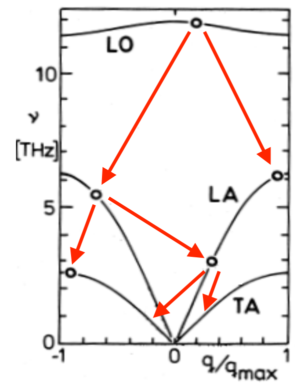
J. N. Clark *et al*., Science **341**, 56 (2013).

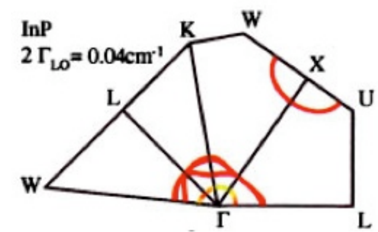
Example 3: Tracking phonons in time and momentum



M. Trigo, *et al*., Phys. Rev. B, **82** 235205 (2010).

Ultrafast thermal diffuse scattering





A. Debernardi, *et al.,* Phys. Rev. B., **57,** 12847 (1998).

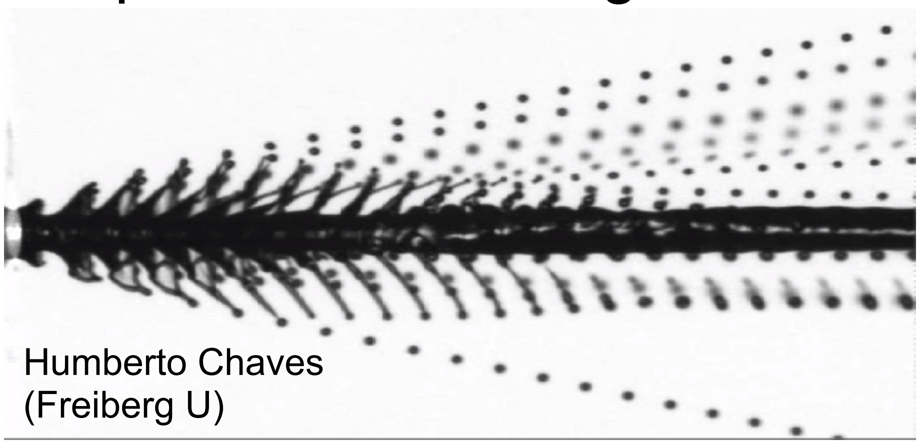
Nonthermal distributions, anharmonic decay

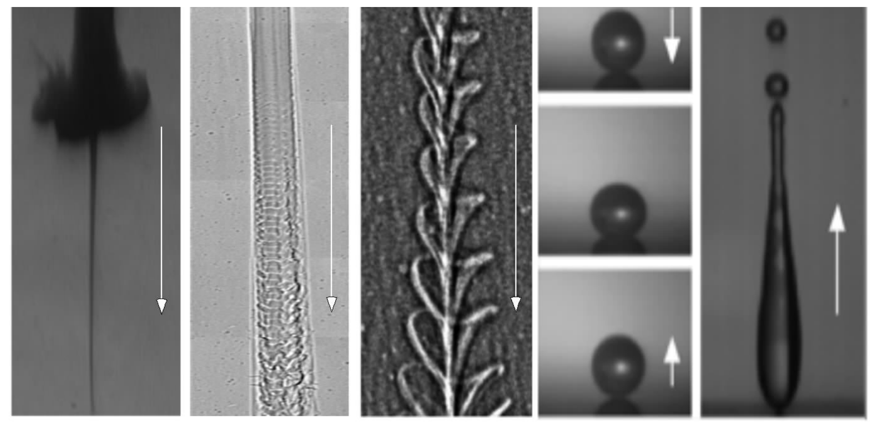
- Phonon-phonon, electron-phonon interactions
- **Time-domain** often necessary to access these non-equilibrium states
- Future sources will match x-ray scattering to the natural timescale of these interactions.

PULSE STANFORD

Example 4: Imaging Microscopic Multiphase Liquid Flows

- Can high-speed flows (often turbulent) be a well-controlled process?
- Engine sprays, high-pressure industrial sprays. Immediate implication for next-generation fuel and combustion.





High-pressure injector | *High-pressure diesel spray* | *Jet with in-nozzle vorticity* | *Liquid drop bouncing on a solid surface*

Challenges:
•Optically dense due to many interfaces
•Highly dynamic – high temporal resolution
•Even more difficult on micrometer length scale-high spatial resolution

•X-rays provide easily interpreted image of mass density.

J. Wang (ANL)

Examples of Experiments

1. Condensed Matter and Materials Science

2. **Chemical Transformations**

3. Biological Molecules

Example 1: Photo-decomposition of Iodomethane

Identifying excited state reaction pathways.

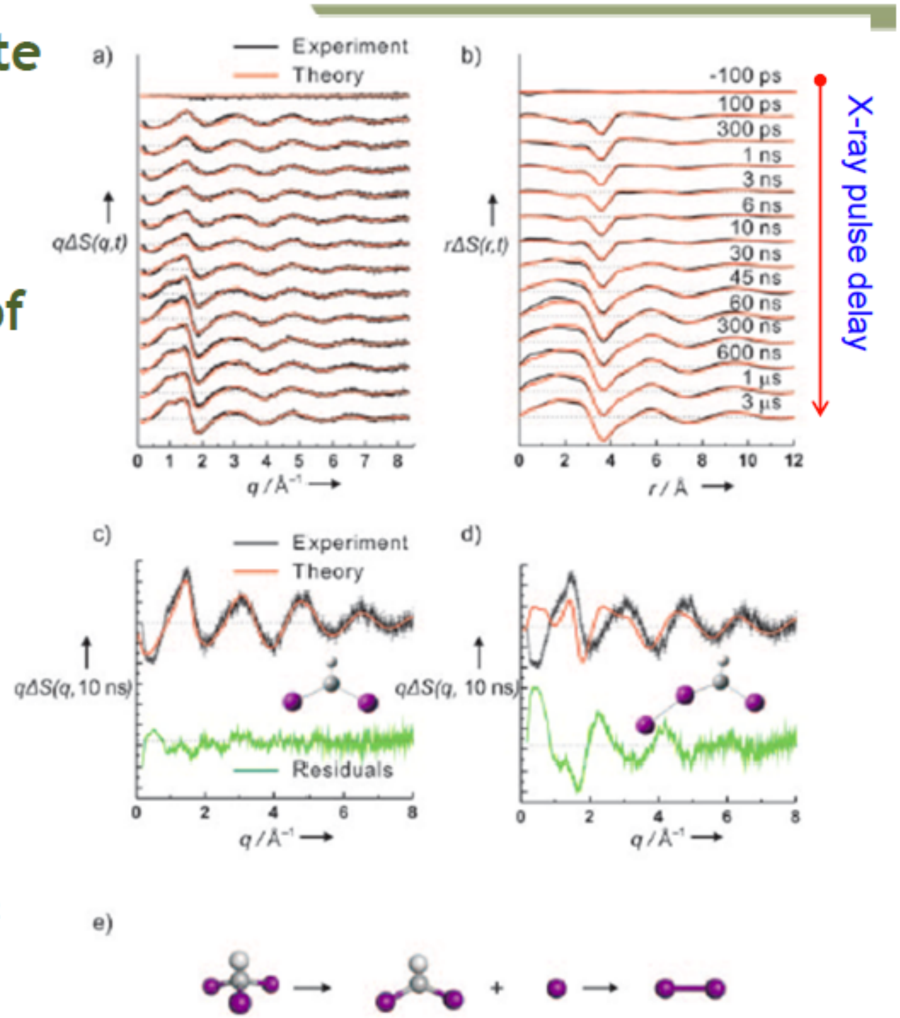
Example: photo-decomposition of iodoform, CHI_3

(many other examples too!)

$$CHI_3 \xrightarrow{hv} [\ CHI_3^{*}\ ]$$

$[\ CHI_3^{*}\ ] \rightarrow$ or $\rightarrow [\ CHI_2\text{-}I\ ]$?

$$2CHI_2 + 2I^{\bullet} \longrightarrow 2CHI_2 + I_2$$



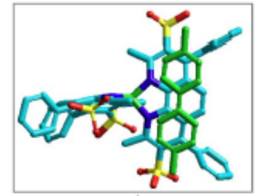
Kong, Lee, Plech, Wulff, Ihee, Koch, Angew. Chem. (2008) 120: 5632–5635; Angew. Chem. Int. Ed. (2008) 47: 5550–5553.

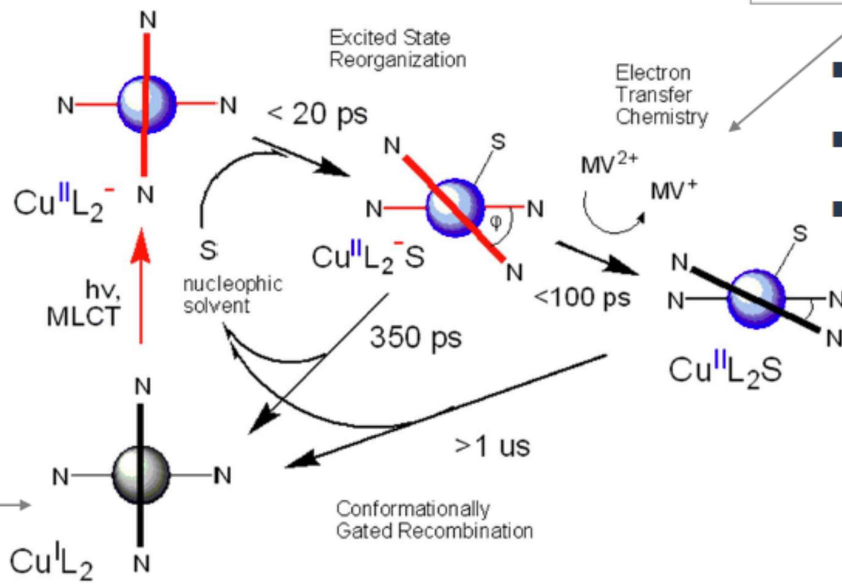
D. Tiede (ANL)

Example 2: Molecular Excited States

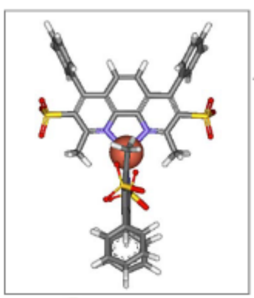
Elaboration on Cu(I) diimide excited-state scheme for electron transfer:
Need for multiple time scales

ET complex





- Molecular recognition
- ET complex formation
- Cage escape



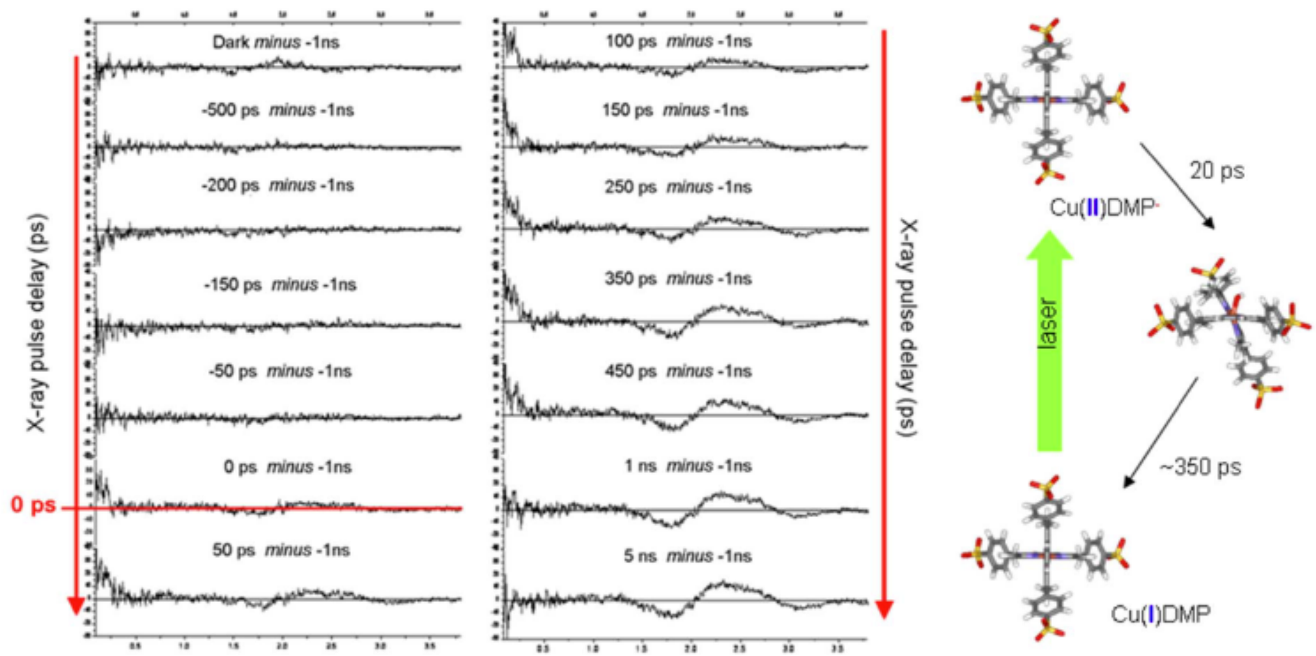
- Inner and outer shell structural events control efficiency of electron transfer
- Dynamic processes cover ultrafast photophysics to multi-scale chemistry
- Model for novel and biomimetic solar conversion

D. Tiede, L. Chen (ANL)

Example 2 (continued): Molecular Excited States

First Pump-Probe Scattering on 11-ID-D using Monochromatic X-rays :

Cu(I) diimide excited-state reaction dynamics



- Demonstration feasibility to do pump-probe TR-scattering experiment using **monochromatic X-rays** at synchrotron light-source
  - **Dilute (6 mM) 1st row transition metal complex**

D. Tiede, L. Chen (ANL)

Examples of Experiments

1. Condensed Matter and Materials Science

2. Chemical Transformations

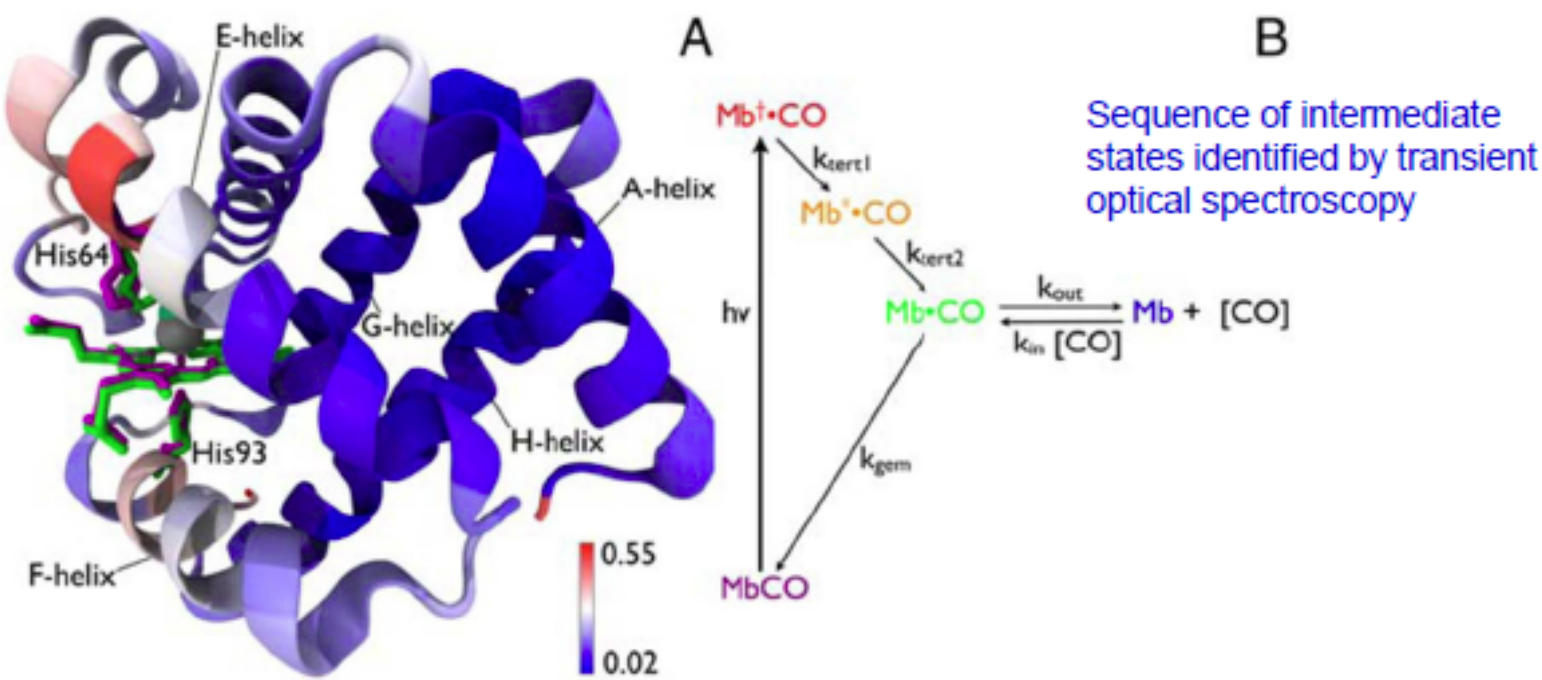
3. **Biological Molecules**

Example: Photo-deligation in Myoglobin

Time-resolved applications in macromolecular photochemistry:

Start in carbon monoxy form (MbCO), optically induced transition to deoxy form (Mb).

Example: Photo-deligation in CO-Mb



Cho *et al*., Proc. Nat. Acad. Sci. **107**, 7281 (2010).

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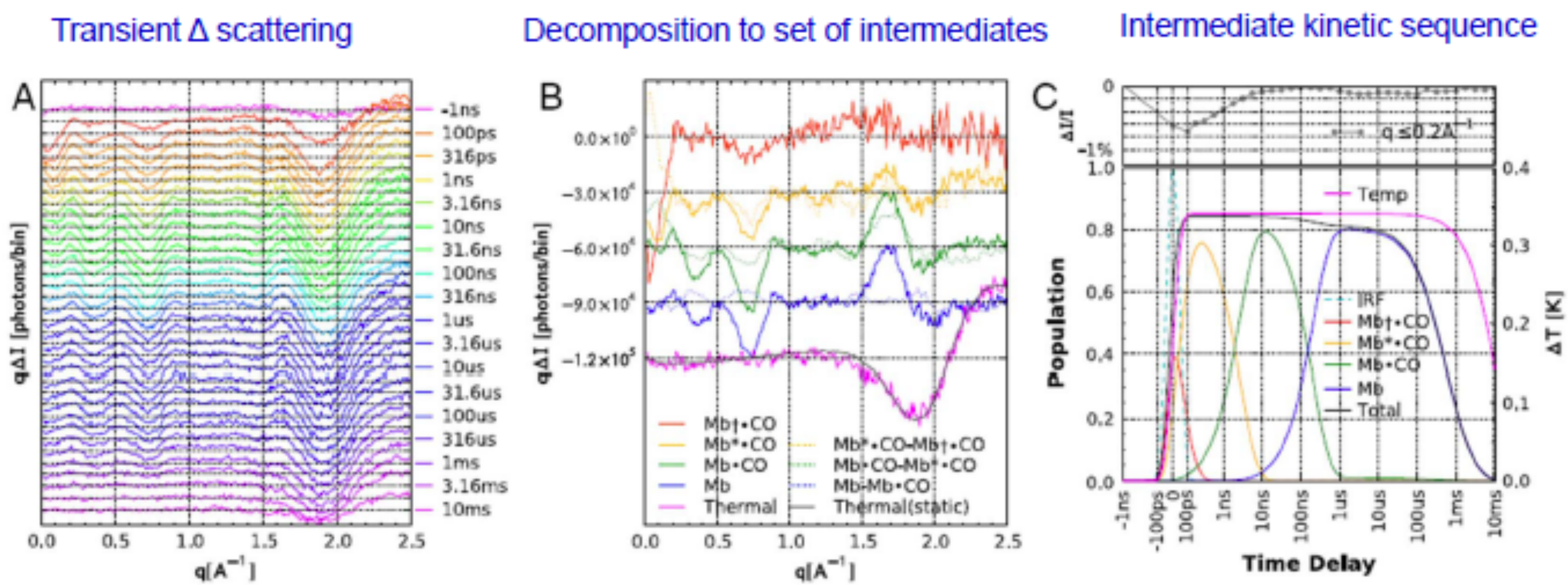
Example: Photo-deligation (continued)

Time-resolved approach has
applications in macromolecular photochemistry:

Example: Photo-deligation in CO-Mb (APS-BioCARS)



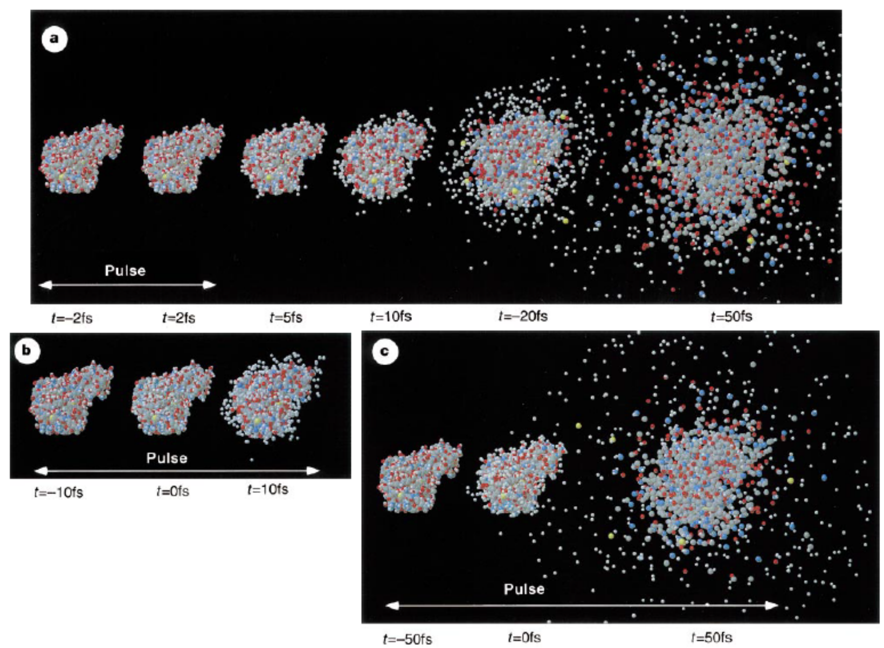
Cho *et al*., Proc. Nat. Acad. Sci. **107**, 7281 (2010).

D. Tiede (ANL)

New Strategies: Single-Shot Experiments at FELS

- FELs have sufficient intensity that the sample can be damaged or destroyed by the first pulse! (Each LCLS pulse has the photons of 10^3-10^5 synchrotron bunches, but in 2 fs.)
- Can you get the information you need in one pulse?
- Can the pulses be short enough to get the information before the structure explodes?

Prediction (2000): Neutze *et al.* Nature **406**, 752 (2000).



Femtosecond Nanocrystallography

LETTER

doi:10.1038/nature09750

Femtosecond X-ray protein nanocrystallography

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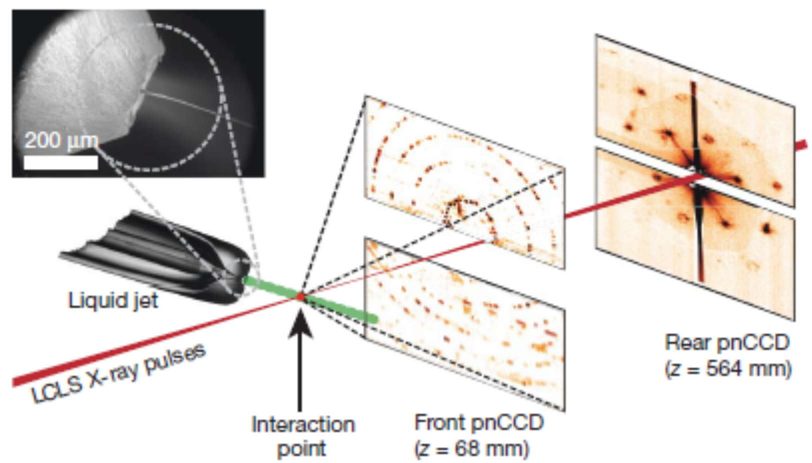
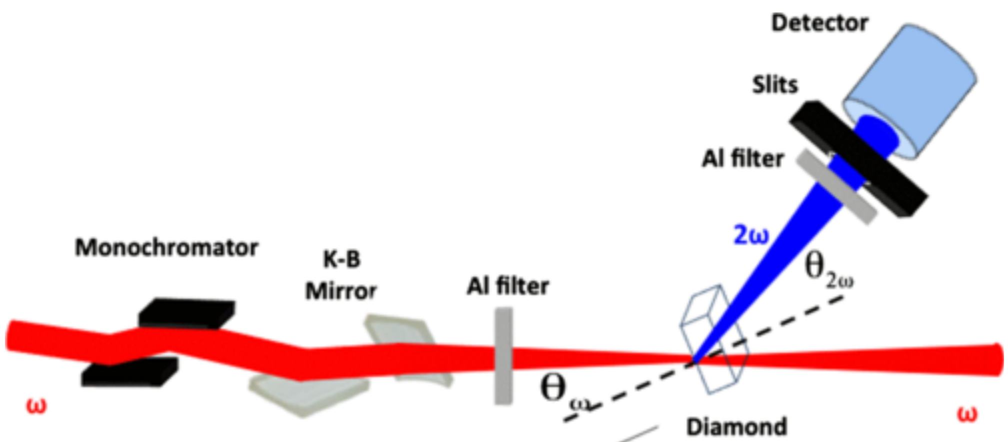
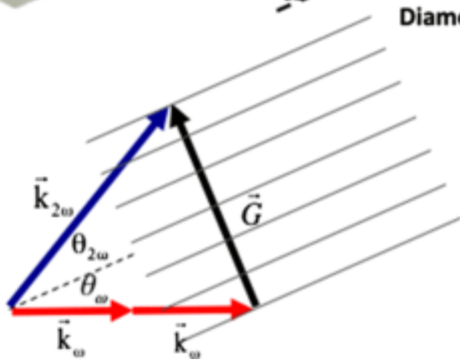
3 FEBRUARY 2011 | VOL 470 | NATURE | 73

Experiment (2011)



X-ray Nonlinear Optics





PRL 112, 163901 (2014) PHYSICAL REVIEW LETTERS week ending 25 APRIL 2014

X-Ray Second Harmonic Generation

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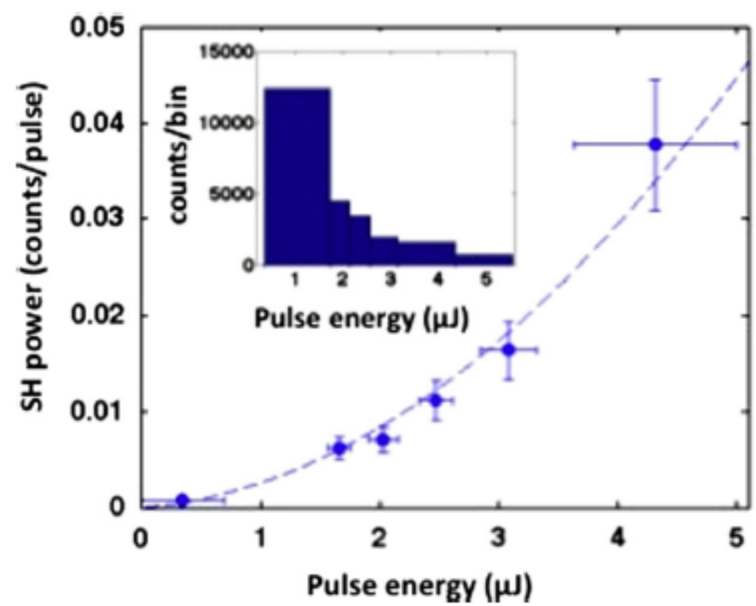
(Received 1 November 2013; published 22 April 2014)

We report clear experimental evidence for second harmonic generation at hard x-ray wavelengths. Using a 1.7 Å pumping beam generated by a free electron laser, we observe second harmonic generation in diamond. The generated second harmonic is of order 10 times the background radiation, scales quadratically with pump pulse energy, and is generated over a narrow phase-matching condition. Of importance for future experiments, our results indicate that it is possible to observe nonlinear x-ray processes in crystals at pump intensities exceeding 10^{16} W/cm^2.

DOI: 10.1103/PhysRevLett.112.163901 PACS numbers: 42.65.Ky, 78.70.Ck



New Sources are Coming Soon

- Improvements in synchrotron sources
- New FELs
  - **Building/Built:** SACLA (Japan), European XFEL, Pohang (Korea), Swiss FEL

Current and Future FELs

	BES FEL Light Sources								
FEL Facility	**LCLS/LUSI**	**LCLS-II**	**NGLS**	**FLASH**	**FLASH-II**	**XFEL**	**SACLA**	**SWISS FEL**	**PAL FEL**
Laboratory, Country	SLAC, USA	SLAC, USA	LBNL, USA	DESY, FRG	DESY, FRG	DESY, FRG	SPring8, JPN	PSI, CH	PAL, ROK
First Operation	Sep 2009	~2018	~2022	Jun 2006	~2014	~2015	Jun 2011	~2016	~2015
Construction Cost [M$]	474.3	405	CD0 Range 900-1500			~1525		294	400
FY12 Annual Ops Cost [M$]	123.9	NA	NA		NA	~140			~60-80
Status	Operating	Construction	Design	Operating	Construction	Construction	Operating	Construction	Construction
E-Beam Energy [GeV]	2.2-15	7-13.5	2.4	0.5-1.25	0.5-1.25	17.5	8.5	2.1-5.8	3, 10
Peak Brightness	1.0E+33	1.9E+33	3.0E+32	2.0E+30	1.0E+31	8.7E+33		2.0E+32	5.0E+31
Average Brightness	3.00E+21	5.80E+21	8.00E+24			3E+24		2.00E+21	
Wavelength Range [Å]	1.3-50	0.7-50	10-120	41-450	40-800	0.5-10	0.63-2	7-70, 1-7	10-100, 0.6-7
Photon Energy Emphasis	Hard X-Ray	Hard X-Ray	Soft X-Ray	Soft X-Ray	Soft X-Ray	Hard X-Ray	Hard X-Ray	Hard X-Ray	Hard X-Ray
Peak Power [GW]	90	190	0.3-3	1-3	1-5	37	40	3	
Pulse Energy [mJ]	0.1-6	0.1-12	0.002-1	0.4	0.5	3.7	0.5	0.005-0.2	
Pulse Duration FWHM [fs]	1-500	1-500	1-300	50-200	10-200		<20	5-50	
Linac Type, Freq [GHz]	NC, 2.856	NC, 2.856	SC, 1.3	SC, 1.3	SC, 1.3	SC, 1.3	NC, 5.7	NC, 5.7	NC, 2.856
Rep Rate [Hz]	120	120	≥1E6	10	10	10	10-60	100	60
Bunches per Cycle	1 now, 2 later	1 now, 2 later	CW	500	4000	2700		2	1 or 2
Number of Undulators	1	2	3 now, 10 later	1	1	3	1 now, 5 later	2	2
Number of Instruments per Undulator	6	3	≥2	5	5	2		3	1-2, 2-4

Storage Ring Sources

	BES Storage Ring Light Sources													
Storage Ring Facility	ALS	NSLS XRAY	NSLS-II w/ Damping Wiggler	SSRL	APS	APS-U	SLS	MAX IV	SIRIUS	ESRF	Spring-8	PETRA-III	ESRF Phase I&II Upgrade	Spring-6 Upgrade
Laboratory, Country	LBNL, USA	BNL, USA	BNL, USA	SLAC, USA	ANL, USA	ANL, USA	PSI, CH	MAX, SWE	LNLS, FRB	ESRF, FRA	SPring8, JPN	DESY, FRG	ESRF, FRA	SPring8, JPN
First Operation	1993	1984	2014	1974(2004)	1996	~2018	2001	2015	~2016	1992	1997	2010	~2019	~2019
Construction Cost [M$]	146	24	912		812	391	171		320	~500	1240	~260	413	~450
FY 12Annual Ops Cost [M$]	60	36	NA	34.9	123.9	NA	38.7	NA	NA	~140	~95		NA	NA
Status	Operating	Operating	Construction	Operating	Operating	Construction	Operating	Construction	Construction	Operating	Operating	Operating	Design	Design
E-Beam Energy [GeV]	1.9	2.8	3	3	7	7	2.4	3	3	6	8	6	6	6
Emittance [nm]	2.2	59	0.5	9.6	3.1	3.1	5.5	0.33	0.28	4	3.4	1	0.13	0.0675
Average Brightness	4E19@1 keV	2E+17@3keV	3E+21@3keV	1.1E19@12keV	1.4E20@8keV	4E20@8keV	4E19@1keV	4E21@10keV	2E21@10keV	3E20@8keV	8E20@10keV	1E21@10keV	1E22@8keV	4E22@10keV
Circumference [m]	197	170	792	234	1104	1104	288	528	518.2	844	1436	2304	844	1436
Photon Energy Emphasis	Soft X-Ray	Hard X-Ray	Hard X-Ray	Hard X-Ray	Very Hard X-ray	Very Hard X-ray	Soft X-Ray	Hard X-Ray	Hard X-Ray	Very Hard X-ray	Very Hard X-ray	Very Hard X-ray	Very Hard X-ray	Very Hard X-ray
Beam Current [ma]	500	300	500	450	100	150	400	500	500	200	100	100	200	100
RMS Pulse Duration [ps]	30	145	15	3-21	33	0.9	0.1-35							
Number of Straights	12	8	30	18	40	40	12	20	20	32	48		32	48
FY12 Annual Users	1995	2453	NA	1597	4360	~5000	1793	NA	NA				NA	NA

Conclusions

- Dynamics in condensed matter, materials science, chemistry, and macromolecules have wide relevance to important questions.
- X-ray techniques can address these questions using dynamical versions of techniques we're familiar with.
- Near Future: Many new sources, new techniques.

- Further Future: Transform-limited x-ray pulses.