Ultrafast melting of colloidal crystals observed in pump-probe experiments at LCLS

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Photonic crystals

Photonic crystals in nature

Artificial photonic crystals

Photonic band gap materials

https://www.theochem.kth.se/research/phot_crys/t/Photonic_Crystals.html
Tuning of properties by external fields

Pumping energy


Y. Y. Hui et al., ACS Nano 7 (8), 7126 (2013)

Incremental heating

M. Daryl et al., PRL 108, 033902 (2012)

http://www.st-andrews.ac.uk/micro photonics/Gallery.php
Colloidal crystals grown by self-organization
Growth of the colloidal crystal film

Vertical deposition method

Polystyrene colloidal crystal

Diameter = 160nm

Polystyrene (PS) spheres

Prototype of bio-molecule
Structural evolution of colloidal crystal films in the process of melting

E. Sulyanova et al., Langmuir (2015) (accepted)

See also the Poster on the Users Meeting
X-ray diffraction patterns measured *in situ* during incremental heating

**Experiment A**

15 keV, 50 x 50 μm unfocused beam

<table>
<thead>
<tr>
<th>T = 293 K</th>
<th>T = 364 K</th>
<th>T = 376 K</th>
<th>T = 378 K</th>
<th>T = 382 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Experiment B**

8 keV, 3.5 x 2.8 μm focused beam

<table>
<thead>
<tr>
<th>T = 300 K</th>
<th>T = 364 K</th>
<th>T = 376 K</th>
<th>T = 378 K</th>
<th>T = 382 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>(b)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
X-ray diffraction pattern of the experiment A measured at room temperature.

Same pattern with SAXS contribution subtracted.

Enlarged area of (b) showing Bragg peak indexing.
Temperature evolution

Mesoscopic scale

- Lattice distortion parameter $g_q$
- Domain misorientation parameter $g_\varphi$

Nano-scale

- Polystyrene particle diameter $D$
- Average lattice parameter $<a_{[110]}>$
The model of colloidal crystal melting process

Nano-scale

Mesoscopic scale
Study of dynamics in colloidal crystals
Observation and tuning of hypersonic bandgaps in colloidal crystals

- Polystyrene spheres in air, glycerol, PDMS and silicon oil
- $D = 256\, \text{nm, 307\,nm}$
- Brillouin spectroscopy
- No sintering

Brillouin light scattering spectra of dry and wet opals and phononic gap

Hypersonic modulation of light in three-dimensional photonic and phononic band-gap materials

- Silica spheres, 359 nm diameter, 10-12 layers
- IR energy converted into vibrations with an 100 nm thick Al foil (“hypersonic transducer”)
- Sintered crystal, coupling parameter $\chi = D/2a - 1 = 0.015 \pm 0.005$
- Reflectivity measurements

Set up for pump-probe experiment

Reflectivity measurements

Pump-probe experiment on colloidal crystals at FLASH
Study of colloidal crystal in the temporal domain

- Elastic vibration of the spheres (Lamb modes)
- Collective vibrations (phonons)
- Order-disorder transitions

Pump-probe experiment:

- Pump: 800 nm IR laser
- Probe: 8 nm FEL radiation
- Time delay from -100 ps up to 1000 ps, with 50 ps steps

Single-shot diffraction patterns at different time delay

The momentum transfer vector $\mathbf{Q}$ and the horizontal $W_x$ and vertical $W_y$ size of the peaks were analyzed

Theoretical calculations of vibrations of a 400 nm isotropic elastic sphere based on the Lamb theory reveal a 5.07 GHz eigenfrequency of the ground (breathing) mode.
Pump-probe experiment on colloidal crystals at LCLS

See also the Poster on the Users Meeting
Experimental setup

Pump-Probe experiment on colloidal crystals
Experimental setup@XPP

Bandwidth
0.4 eV at 8keV

Diamond (111)

CRL

Sample

Fly tube
Filed with He

Length ~10 m

Detector CSPAD

1 pixel ~110 µm

Another experimental hatch
Experimental setup@XPP
Experimental setup@XPP
CSPAD detector
Samples
Top to middle of sample. At the top the sample is quite damaged, but the middle region with a lot of layers and larger area’s between the cracks has higher quality.

Distance between cracks around 50 μm

In the film parts lines typical for stacking defects can be seen. These indicate that even at this thick region the film is still ordered.
1-st experimental geometry

IR Laser

X-ray Laser

IR Laser

Al foil 50 nm

Colloidal crystal

Detector
2-nd experimental geometry

IR Laser

X-ray Laser

$\theta_B = 35^\circ$

Detector
Parameters of X-ray and IR laser beams

1. X-ray beam
   - $E=8$ keV
   - Pulse duration: $\leq 50$ fs
   - $\text{Flux}_{\text{sample}} \sim 10^9$ ph/pulse
   - Focus $\sim 50$ $\mu$m
   - Energy bandwidth $\sim 10^{-4}$

2. Laser beam
   - $\lambda_{\text{las}} = 800$ nm
   - Pulse duration: $\leq 50$ fs
   - $E \sim 2$ mJ
   - Power: $P \sim 4\times 10^{10}$ W
   - Focus $\sim 100$ $\mu$m
Energy of infrared laser

Fitted curve

- Experimental data

<table>
<thead>
<tr>
<th>WP</th>
<th>Energy (μJ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>104</td>
</tr>
<tr>
<td>13</td>
<td>173</td>
</tr>
<tr>
<td>15</td>
<td>229</td>
</tr>
</tbody>
</table>

Laser pulse energy (μJ)

Waveplate angle (degree)
Pump-Probe experiment on colloidal crystals
Damage produced by laser power $7^\circ$ wave plate angle (~50 µJ)
Study of ultrafast melting of colloidal crystals
Run 187 first 100 frames at one position X-rays only

Detector frame in linear scale

Intensity distribution along angular direction (ANG)

Intensity distribution along radial direction (RAD)
Pump-probe experiment

Position 1  Position 2

Laser trigger

Time delays from \( \tau = -10 \text{ ps to } +1000 \text{ ps} \)
Decay of integrated intensity
Decay of integrated intensity of the peaks

\[ \frac{\Delta I(\tau)}{\langle I \rangle} = \frac{I_{1}^{on}(\tau) - \langle I^{off} \rangle}{\langle I^{off} \rangle} \]

- \( \tau \) is time delay between laser and X-ray pulses
Decay of the interplaner distance
Decay of the interplanar distance

\[ \Delta Q(\tau) = \frac{Q_{\text{on}}(\tau) - \langle Q^{\text{off}} \rangle}{\langle Q \rangle} = \langle Q^{\text{on}} \rangle - \langle Q^{\text{off}} \rangle \]

- \( \tau \) is time delay between laser and X-ray pulses
Bragg peak’s broadening in q-direction
Bragg peak’s broadening in $q$-direction

\[ \frac{\Delta w_q(\tau)}{\langle w_q \rangle} = \frac{w_{q_1}^{on}(\tau) - \langle w_q^{off} \rangle}{\langle w_q^{off} \rangle} \]

- $\tau$ is time delay between laser and X-ray pulses

$w_q$ - FWHM in $q$-direction
Bragg peak’s broadening in $\varphi$-direction
Bragg peak’s broadening in $\phi$-direction

$$\frac{\Delta w_\phi (\tau)}{\left<w_\phi \right>} = \frac{w_{\phi,1}^{on}(\tau) - \left<w_{\phi}^{off}\right>}{\left<w_{\phi}^{off}\right>}$$

- $\tau$ is time delay between laser and X-ray pulses

$w_\phi$ - FWHM in $\phi$-direction
Model of ultrafast melting of colloidal crystal

1. Energy transfer from IR laser to colloidal crystal

2. Response of colloidal crystal lattice
Energy transfer from IR laser to colloidal crystal

- IR wavelength: 800 nm
- Energy: 1.5 eV
- Energy of chemical bonds: (C-C, C-H) \( \sim 3-4 \) eV
- Absorption coefficient of 800 nm radiation in polystyrene: \( 10^{-4} \)
- Temperature raise: one-two degrees
Members of the team

On site

1. I. Vartaniants (DESY, Germany) (spokesmen)
2. M. Chollet (LCLS, USA) (LCLS beamline responsible)
3. J.M. Meijer (University of Utrecht, Nederlands)
4. R. Kurta (DESY, Germany)
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Experiment is over!!!
Thank you for your attention