Material response to laser energy deposition: non-thermal processes

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Structural Control of Proteins at Electrified Interfaces

Hot Electron Electrochemistry

Lasers in Cultural Heritage

Far Field

Near Field

Laser Medicine
Excitation mechanisms of solids

Metals: Two-temperature model
- Fundamentals
- Thin films
- Metal ablation

Dielectrics: Multiphoton and Avalanche Ionization
- Dielectric ablation
- Coulomb Explosion
- Non-thermal melting, X-ray

Material Patterning
- Laser-induced periodic surface structures
- Electron pressure
- Photopolymerization
Outline

- Excitation mechanisms of solids
  - Metals: Two-temperature model
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  - Laser-induced periodic surface structures
  - Electron pressure
  - Photopolymerization
Overview of Laser Matter Interaction

- Carrier Excitation
  - Absorption of photons
  - Impact ionization
  - Carrier-carrier scattering
  - Carrier-phonon scattering

- Thermalization

- Carrier Removal
  - Radiative recombination
  - Carrier diffusion
  - Auger recombination
  - Ablation & evaporation
  - Thermal diffusion
  - Resolidification

- Thermal and Structural Effects

Accord. To E. Mazur
Mechanisms for exciting carriers in a solid

(a) single photon absorption — direct

(b) single photon absorption — indirect

(c) multi-photon absorption

(d) free-carrier absorption

(e) impact ionisation

Accord. To E. Mazur
Mechanisms for exciting carriers in a solid

Interband Excitation by Photons
Interband *single photon absorption* (SPA)

Multiphoton absorption (MPA)

Intraband Excitation by Photons
*Free carrier absorption*

Impact Ionisation

Accord. To E. Mazur
Mechanisms for exciting carriers in a solid

The three regimes of strong field ionisation:

- multiphoton ionisation
- tunnelling regime
- over-the-barrier regime

Mechanisms for exciting carriers in a solid

Carrier Redistribution, Thermalisation and Cooling

Carrier redistribution and thermalisation:

**carrier distribution** following laser excitation but before scattering

thermalised carrier distribution created by **carrier-carrier**

\[ k_1 + k_2 = k'_1 + k'_2 \]
\[ E(k_1) + E(k_2) = E(k'_1) + E(k'_2) \]

and **carrier-phonon** scattering.

Accord. To E. Mazur
Mechanisms for exciting carriers in a solid

Carrier Redistribution, Thermalisation and Cooling

Specific to crystalline semiconductors: intravalley and intervalley scattering.

\[ \delta E_G = D \left( \delta a / a \right) \]

Intravalley scattering

Intervalley scattering

(b) carrier-phonon scattering

\[
\frac{1}{\tau_{e-ph, \text{deformation}}} \propto \text{constant}
\]

\[
\frac{1}{\tau_{e-ph, \text{polar-optical}}} \propto \frac{1}{q^2}
\]

Accord. To E. Mazur

Department of Physical Chemistry

Wolfgang Kautek
Ultrafast laser excitation of solids

- $10^{-15}$: electron-electron scattering: $T_{e-h} \gg T_L$
- $10^{-14}$: electron-phonon scattering: $T_{e-h} \downarrow, T_L \uparrow$
- $10^{-13}$: non-thermal
- $10^{-12}$: $T_{e-h} \approx T_L$
- $10^{-11}$: phonon-phonon scattering
- $10^{-10}$: thermal
- $10^{-9}$: heat conduction
- $10^{-9}$: (thermal) melting
- $10^{-9}$: ablation

Time [s]

Wolf 2006
Ultrafast laser excitation of solids

Characteristic time scales:

• Electronic excitation: $\tau_L$

• Thermalization: $\tau_R$ \hspace{1cm} $\tau_L << \tau_R << \tau_E < \tau_M$

• Energy transport: $\tau_E$

• Mass transport: $\tau_M$

• Spatial and temporal localization of energy deposition:
  • Non-equilibrium
  • Impulsive heating at constant volume
  • High temperature and high pressure phases of matter:
    „warm dense matter“
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  - Electron pressure
  - Photopolymerization
2 Temperature Model (TTM)

\[ C_e(T_e) \frac{\partial T_e}{\partial t} = \nabla [K_e(T_e, T_l) \nabla T_e] - G(T_e)(T_e - T_l) + S(\vec{r}, t) \]

\[ C_l(T_l) \frac{\partial T_l}{\partial t} = \nabla [K_l(T_l) \nabla T_l] + G(T_e)(T_e - T_l) \]
Relaxation phases following optical excitation of metals

(a) $t = 0$

nonequilibrium electrons
electron-electron collisions

(b) $T_e \gg T_\ell$
electrons in thermal equilibrium
electron-phonon collisions

(c) $T_e = T_\ell$
electrons and lattice in thermal equilibrium

2 Temperature Model:
Time and energy dependence of the electron distribution in gold

E. Carpene, PHYSICAL REVIEW B 74, 024301 (2006)

50-fs
\( h = 1.55 \text{ eV} \)
5 J/cm²
2 Temperature Model: Hot electron balance

\[ C_e(T_e) \frac{\partial T_e}{\partial t} = \nabla [K_e(T_e, T_l) \nabla T_e] - G(T_e)(T_e - T_l) + S(\vec{r}, t) \]

\( C \):  heat capacities

\( K \):  thermal conductivities

\( G(T_e) \):  electron-phonon coupling factor

\( S(\vec{r}, t) \):  source term of local energy deposition by the laser pulse

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
2 Temperature Model: Hot electron balance

Electron heat capacity

High \( T_e \):

\[
C_e(T_e) = \int_{-\infty}^{\infty} \frac{\partial f(\varepsilon, \mu, T_e)}{\partial T_e} g(\varepsilon) \varepsilon d\varepsilon
\]

- \( g(\varepsilon) \): electron DOS at the energy level \( \varepsilon \)
- \( \mu \): chemical potential at \( T_e \)
- \( f(\varepsilon, \mu, T_e) \): Fermi distribution function

\[
f(\varepsilon, \mu, T_e) = \left\{ \exp\left[\frac{(\varepsilon - \mu)}{k_B T_e}\right] + 1 \right\}^{-1}
\]

Low \( T_e \):

\[
C_e(T_e) = \gamma T_e
\]

- \( \gamma \): electron heat capacity constant

\[
\gamma = \frac{\pi^2 k_B^2 g(\varepsilon_F)}{3}
\]

\[
\gamma = \frac{\pi^2 n_e k_B^2}{2 \varepsilon_F}
\]

- \( g(\varepsilon_F) \): electron DOS at the Fermi level

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
2 Temperature Model: Hot electron balance

Electron-phonon coupling factor

\[ \frac{\partial E_e}{\partial t} \bigg|_{ep} = G(T_l - T_e), \quad G = \frac{\pi^2 m_e C_s^2 n_e}{6 \tau(T_e) T_e} \]

- \( m_e \): is the effective electron mass
- \( C_s \): is the speed of sound
- \( n_e \): is the number density of the electrons
- \( \tau(T_e) \): electron relaxation time defined as the electron-phonon scattering time, \( \tau_{e-ph} \)

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
2 Temperature Model: Hot electron balance

$T_e$-dependent electron-phonon coupling factor $G(T_e)$

$$G(T_e) = \frac{\pi \hbar k_B \lambda \langle \omega^2 \rangle}{g(\varepsilon_F)} \int_{-\infty}^{\infty} g^2(\varepsilon) \left( - \frac{\partial f}{\partial \varepsilon} \right) d\varepsilon$$

$g(\varepsilon)$: electron DOS at the energy level $\varepsilon$

$f(\varepsilon, \mu, T_e)$: Fermi distribution function

$\omega^2$: second moment of the phonon spectrum

$\lambda$: electron-phonon mass enhancement parameter

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
Aluminium

Electron DOS &
Fermi distribution function

Electron-phonon coupling factor

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
Gold

Electron DOS & Fermi distribution function

Electron-phonon coupling factor

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
Nickel

Electron DOS & Fermi distribution function

Electron-phonon coupling factor

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
Titanium

Electron DOS & Fermi distribution function

Electron-phonon coupling factor

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
Conclusions from strong electron-phonon nonequilibrium

- **Al:** Free Electron Gas (FEG) provides a good description of the temperature dependence of the electron heat capacity, but fails to predict a relatively moderate 40% increase in the strength of the electron-phonon coupling with increasing electron temperature.

- **Au:** the electron heat capacity and the electron-phonon coupling factor are strongly enhanced by the thermal excitation of d band electrons at electron temperatures exceeding several thousand Kelvins.

- **Ni:** Fermi level at the high density of states edge of the d band results in the opposite trend when the thermal excitation of d band electrons leads to a drastic decrease in the electron-phonon coupling factor and large negative deviations of the electron heat capacity from the linear dependence on the electron temperature.

- **Ti:** Fermi level in the middle of a partially filled d band, in a local dip in the electron DOS, results in complex nonmonotonic dependences of the thermophysical properties on the electron temperature.

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
Conclusions
from strong electron-phonon nonequilibrium

- The strong deviations of the thermophysical properties from the commonly used approximations of a
  (a) constant electron-phonon coupling and a
  (b) linear dependence of the electron heat capacity,
  have important implications for ultrafast processes associated with laser interaction with metals
during the time of electron-phonon equilibration:
- threshold fluences
- laser-induced stress wave
- emission of electrons
- depth of the melting
- heat-affected zone

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
Nonlinear energy absorption noble metals

Ag: Nonlinear absorption is caused by the excitation of d-band electrons below the Fermi surface
not observed in Pt

Z. Lin, L.V. Zhigilei, V. Celli, PHYSICAL REVIEW B 77, 075133 (2008)
Time dependence of electron and lattice temperatures

Bulk

Thin film

100-nm films
single 200-fs
400-nm laser pulse
23 mJ=cm²

Thickness dependence of damage thresholds

Dashed lines: Heat diffusion equation

Solid lines: TTM

248-nm, 14-ns
400-nm, 200-fs

Thickness dependence of damage thresholds

Threshold depends on the film thickness whenever this is smaller than the range of electronic energy transport.

Importance of electron–phonon coupling is reflected by the great difference in electron diffusion depths of noble and transition metals.

Au: electron diffusion is the dominant process. Transient optical properties and ballistic energy transport must be accounted for.

Transition metals: Ballistic transport can be neglected.
Substrate influence on electron–phonon coupling in thin Au films

Rate of energy loss from the electron system beyond e-p coupling

Excess electron energy loss via electron-substrate transport: three temperature model TTR

**DLC thickness dependence of damage thresholds**

- $\alpha_{\text{eff}}$ in a-C$_x$N$_y$ $\sim$ 110 nm in accordance with two-photon absorption
- Ballistic hot electrons and heat diffusion length are negligible.

\[ l_{\text{tot}} = \alpha_{\text{eff}}^{-1} \]

Ablation of metals

\[ d = \frac{2}{\pi e \rho \Omega_{\text{vap}}} \frac{A_h F}{\alpha} \]

High \( F \): heat propagation
\( \rho \Omega_{\text{vap}} \): Enthalpy of evaporation,
\( A_h \): absorptance,

Low \( F \): Beer-Lambert Law

\[ d = \frac{1}{\alpha} \ln \left( \frac{F}{F_{th}} \right) \]

High \( F \): Thermal zone
Low \( F \): Optical zone

Ablation of metals: Thermal melting & Spallation, Phase Explosion

Ablation of metals: Thermal melting & Spallation, Phase Explosion

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- Material Patterning
  - Laser-induced periodic surface structures
  - Electron pressure
  - Photopolymerization
Dielectric ablation threshold fluence vs. pulse duration $\tau$

Below the Electron-Phonon Relaxation Time

Fused silica, $\lambda = 780$ nm, $N = 80$

$\tau = 3$ ps  
(F = 19.9 Jcm$^{-2}$)

$\tau = 220$ fs  
(F = 10.7 Jcm$^{-2}$)

$\tau = 20$ fs  
(F = 11.1 Jcm$^{-2}$)

$\tau = 5$ fs  
(F = 6.9 Jcm$^{-2}$)

Avalanche Ionization

free carrier absorption...

Linear $n_l$

Accord. To E. Mazur
Impact Ionization

...and impact ionization

Accord. To E. Mazur
Free-electron generation in laser-irradiated dielectrics

B. Rethfeld, PHYSICAL REVIEW B 73, 035101 (2006)
Laser field ionization

Accord. To E. Mazur

Nonlinear In

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Dielectrics: Collisional and multiphoton ionization: rate equation approximation

\[
\frac{\partial N(t)}{\partial t} = P(I) + \beta N(t) + \gamma N^2(t) + \zeta N(t)
\]

- \(N(t)\) Electron Density in Conduction Band
- \(\beta\) Avalanche Ionization Coefficient
- \(\gamma\) Coefficient of inelastic Electron-Electron Scattering
- \(\zeta\) Coefficient of Electron-Phonon Scattering

Laser energy couples to the electrons

Energy transfer to the lattice

fs → ps → ns

According to Stoian, 2002
Nonstationary electron energy distribution at the initial stage of ionization

Transition to the asymptotic avalanche regime at longer time scales

B. Rethfeld, PHYSICAL REVIEW B 73, 035101 (2006)
Impact-ionized electrons depend only on the product of intensity and pulse duration

B. Rethfeld, PHYSICAL REVIEW B 73, 035101 (2006)
Ablation of dielectrics


Calculated with MRE
A: surface region depleted of electrons, electric drag force $eE$ dominates electron diffusion $D$ toward the depleted region.

B: electric field is small, a region with negative charging is formed.

C: reduced positive charge

Charging of **dielectric** surfaces causes a **sub-picosecond electrostatic rupture** of the superficial layers, i.e. Coulomb explosion (CE)

- CE strongly **inhibited** for **metals and semiconductors** as a consequence of superior carrier transport properties

Non-thermal melting

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Non-thermal melting

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Non-thermal melting of Si

Molecular dynamics (MD) simulations on the basis of an electronic tight-binding Hamiltonian in real-space: Rapid excitation of electrons within a few 10 fs.

Lattice dynamics on time-dependent potential energy surfaces.

Massive instability in the crystal lattice due to perturbation of the interatomic bonds.

Non-thermal melting of Si

Silicon (64 atoms)
\( \tau = 20 \text{ fs} \)

<table>
<thead>
<tr>
<th>Pulse duration (fs)</th>
<th>Ablation threshold (eV per atom)</th>
<th>Ablation threshold (J/cm²)</th>
<th>Melting threshold (eV per atom)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>3.7 ± 0.3</td>
<td></td>
<td>2.6 ± 0.3</td>
</tr>
<tr>
<td>500</td>
<td>6.2 ± 0.5</td>
<td></td>
<td>5.4 ± 0.4</td>
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</tbody>
</table>

Time-resolved x-ray diffraction (TXRD)

- Laser plasma X-ray source
- Laser pulse to generate a plasma
- X-ray probe pulse
- X-ray mirror
- Optical excitation pulse
- Sample
- Bragg diffracted X-rays
- Variable delay
- Rocking curve
- X-ray CCD camera

Wolf 2006
fs-laser plasma: “femtosecond x-ray tube”


fs-laser: ~100fs, 10 ... 100 mJ

I ≈ 10^{16} ... 10^{18} W/cm²
X-ray diffraction data

CCD camera image

Integration

Dispersion

Rocking Curve

Integrated reflectivity

different lattice constants of Ge and Si

Wolf 2006
X-ray diffraction experiment
AG von der Linde/Sokolowski-Tinten, University Essen

Wolf 2006
X-ray diffraction: Ultrafast melting of InSb

X-ray diffraction: Ultrafast melting of InSb

Changes in the interatomic potential and atomic displacements

A. M. Lindenberg, 308 SCIENCE 208,392 (2005)
Ultrafast non-thermal melting

Laser 0.47 J/cm²

Sample

Silicon

electronic excitation

0.1 ps 0.3 ps

50 ps 0.1 ns

non-thermal melting

ablation

optical spectroscopy

R(λ) of solid Si

300 fs 50 fs -20 fs -120 fs

liquid

Ultrafast non-thermal melting

Intense electronic excitation

> 10 % of all valence electrons!

K. Sokolowski-Tinten et al., PRB 61, 2643 (2001)

P. Stampfli et al., PRB 49, 7299 (1994)

lattice instability
X-ray diffraction: Ultrafast melting of Ge

170 nm Ge on Si; (111)-diffraction spot

K. Sokolowski-Tinten et al., PRL 87, 225701 (2001)
Time-resolved photoemission of Gd(0001) bulk

Gd(0001) surface: p-pol. probe

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Fig. 9a–f. SEM pictures (60°) of damage in silicon generated with Ti:sapphire laser pulses in air. 
a $\phi_0 = 1.0 \, \text{J/cm}^2$, b $1.3 \, \text{J/cm}^2$, c $1.8 \, \text{J/cm}^2$ ($\lambda = 780 \, \text{nm}$, 
$\tau = 100 \, \text{fs}, N = 100$). d $\phi_0 = 2.0 \, \text{J/cm}^2$, e $2.8 \, \text{J/cm}^2$, f $4.1 \, \text{J/cm}^2$ ($\lambda = 800 \, \text{nm}, \tau = 130 \, \text{fs}, N = 100$)

Fig. 6a–d. Laser ablation of TiN on silicon in air \((\tau = 150 \text{ fs}, \lambda = 800 \text{ nm}, \phi_0 = 3.0 \text{ J/cm}^2)\) for circular polarization. Columnar structures were formed in the silicon substrate after drilling through the TiN layer was performed.

\(a \ N = 50; \ b \ N = 80; \ c \ N = 100; \ d \ N = 200 \) laser pulses

Non-linear correlation of periodicity with energy

As ripples grow, **nonlinear effects** become important, ripples become asymmetric and the wavelength increases due to merging events (**bifurcations**).

---

\[ \lambda = 800 \text{ nm}, \ \tau = 130 \text{ fs}, \ N = 100, \ 1 \text{ kHz (interval } = 1 \text{ ms}), \ F = 5.6 \text{ J/cm}^2 \]

Theory of Ripple Formation II

• \( \Lambda \sim \lambda \) type \[^{[1]}\]:

  • Interference of the incident and the reflected/refracted light with the scattered light near the interface (normal incidence).

    \[
    \Lambda = \lambda
    \]
    \[
    \Lambda = \frac{\lambda}{n}
    \]
    \[
    n = \varepsilon^{1/2}
    \]
    \[
    \varepsilon(\omega) = \varepsilon'(\omega) + i \varepsilon''(\omega)
    \]

  • Orientation:

    Metals: \( \perp \) to the laser field vector. \( \perp \) if \( |\varepsilon'| > 1 \)

    Dielectrics: \( \parallel \) if \( |\varepsilon'| < 1 \) & \( |\varepsilon'| > \varepsilon'' \)

Generation of stretched pulses (150 fs – 5 ps) and of double pulse sequences with delay times (300 fs – 11 ps).

Polyimide

- Ripples with $\Lambda \sim \lambda$
- ll to E $\rightarrow$ dielectric
- Transition from ordered $\rightarrow$ disordered structures
  - Pulse Duration $> 500$ fs
  - Double Pulse Delay Time $> 2$ ps

(a) $N=10$, $\tau=150$ fs, $F=1.4$ J/cm$^2$
(b) $N=10$, $\tau=800$ fs, $F=1.5$ J/cm$^2$

Theory of Ripple Formation

- $\Lambda \ll \lambda$ type ("Nanoripples"):
  1. Interference effects along with transient changes in the optical properties during laser irradiation? \[1\]
  2. Second harmonic generation? \[2\]
  3. Excitation of surface excited waves? \[3\]
  4. Self-organisation! \[4\] \[5\]

Non-thermal melting of Si

Vibration dynamics of ellipsoidal silver nanoparticles execute anisotropic shape oscillations triggered by the thermal expansion of the optically heated particles. Expansion is caused by two mechanisms:

1. lattice anharmonicity and the
2. extremely large pressure of the hot conduction electrons

Conventional Photopolymerization

![Diagram of conventional photopolymerization process]

- **UV laser**
- **Liquid resin**

The diagram shows a UV laser focused on liquid resin, creating a curing effect.

Key components:
- **Scanning system**
- **Laser**
- **Sweeping wiper blade**
- **Vat**
- **Part**
- **Platform**
- **Photocurable liquid resin**
- **Elevating platform**
- **Supports**
Monomers, Photoinitiators & Sensitizers

A radical polymerization:

\[ \text{hv} \quad M \xrightarrow{\text{light}} M^* \xrightarrow{\text{PI}} (M)_n \xrightarrow{\text{PI}} P \]

\[ \equiv \xrightarrow{R \cdot} R^* \xrightarrow{\text{R}} COOR_m \]

Initiation: intramolecular charge transfer followed by an intermolecular electron transfer.
2-Photon Absorption

\[ \frac{dn_p}{dt} = \delta NF^2 \]

- \( n_p \): absorbed photons
- \( \delta \): TPA cross section (VERY SMALL!)
- \( N \): absorbing molecules
- \( F \): laser flux

Virtual state (\( \tau \sim \text{fs} \))
Advantages of 2PP

According to J. Mertz, Boston University
Advantages of 2PP

- CCD camera for online monitoring
- Galvo-scanner
- fs-pulses: 80 fs, 80 MHz, < 1 nJ
- High NA objective
- Oil
- Glass Frame
- 150 µm
- 100 µm
- 150 µm
- Resin

Laser Focus World 2004
2-Photon Lithography

J. Stampfl, TU Vienna
2-Photon Lithography

J. Stampfl, TU Vienna
Femtosecond Laser
Apertureless Scanning Nearfield Optical Microscope

Tip-enhanced highly confined photon source:
Nanostructuring
Femtosecond Laser
Apertureless Scanning Nearfield Optical Microscope

C. Huber, P. Grabner, A. Trügler, U. Hohenester, W. Kautek, to be published.
Summary: Non-thermal processes

Fundamental differences in material responses on fs-excitation in metals and dielectrics

<table>
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<th>Semiconductors / Dielectrics</th>
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<td>Electron heating</td>
<td>Free electron generation</td>
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<td>Electron diffusison</td>
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<td>Electron thermalisation</td>
<td>Electron-electron thermalisation</td>
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<td>Free carrier absorption</td>
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<td>Coulomb explosion</td>
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<td>Electron-phonon relaxation</td>
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<td>(scattering interaction)</td>
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<td>Supercritical fluid formation</td>
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<td>Thermal diffusion</td>
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<tr>
<td>Ablation via phase explosion</td>
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<tr>
<td>Resolidification</td>
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</table>
Summary: Non-thermal processes and electronic and structural dynamics in semiconductors and dielectrics on excitation with short laser pulses.