THz Electric Field-Induced Second Harmonic Generation in Inorganic Ferroelectric

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Second Harmonic Generation induced by the electric field of a strong nearly single-cycle terahertz pulse with the peak amplitude of 300 kV/cm is studied in a classical inorganic ferroelectric thin film of (Ba0.8Sr0.2)TiO3. The dependences of the SHG intensity on the polarization of the incoming light is revealed and interpreted in terms of electric polarization induced in the plane of the film. As the THz pulse pumps the medium in the range of phononic excitations, the induced polarization is explained as a dynamical change of the ferroelectric order parameter. It is estimated that under action of the THz pulse the ferroelectric order parameter acquires an in-plane component up to 6% of the net polarization.

Photoinduced nonequilibrium phase transitions triggered by femtosecond or picosecond laser pulses is a subject of intense and multidisciplinary research1–2. Fundamentally, these are counterintuitive phenomena in which subtle excitations of atoms or spins are able to lead to dramatic changes in crystallographic, electric, or magnetic properties of media. In ferroics, ultrafast control of the order parameter (electric polarization or magnetization) is a particularly important problem, understanding of which might be essential for future progress in information processing technology. In the last decades a substantial progress has been achieved in ultrafast optical control of spins in magnetically ordered materials and all-optical magnetic recording. It has been shown that exciting magnets on a timescale much faster than characteristic times of atomic, orbital and spin motion can steer magnetization dynamics along yet unexplored non-thermodynamic routes1. Ultrafast coherent control of the magnetic phase transitions via active optical pumping of the soft mode4,5 and magnetization reversal via a strongly non-equilibrium state6 have been demonstrated. Thermodynamically, critical behaviors of the electric polarization and the magnetization are very similar7. Nevertheless, the possibility of switching of the electric polarization by ultrashort laser pulses has not been reported until now. Naturally it raises interest to the problem of ultrafast optical control of electric polarization in ferroelectric materials.

Despite the enormous amount of experiments reporting on optical control of spins, there are very few studies of ultrafast dynamics of the electric polarization8–16. Obviously, measurement techniques based on electric contacts are not able to provide the required temporal resolution. An elegant solution for a detection of the order parameter in ferroelectrics can be based on time-resolved X-ray diffraction. However, this technique is very challenging and, as a matter of fact, it is available in very few places in the world17–19. Nonlinear optical technique of the Second Harmonic Generation (SHG) is known to be very sensitive to the order parameter in ferroelectrics16,20–22 and it is by far less challenging than the studies in the X-ray spectral range23–25. However, despite this fact practically nothing is known about ultrafast nonlinear optical response of ferroelectrics26. Consequently, this lack of knowledge considerably hampers optimization and interpretation of experiments in which ultrafast dynamics in ferroics is probed with the help of second harmonic generation and visible light.

Here we report about experimental study of ultrafast nonlinear optical response of ferroelectric (Ba0.8Sr0.2)TiO3 (BST) to the electric field of nearly single-cycle THz pulses picosecond pulses. For this we employ a pump-probe method. Freely propagating nearly single-cycle THz pulses with the amplitude up to 300 kV/cm excites ferroelectric ((Ba0.8Sr0.2)TiO3) film. Femtosecond pulses in the near-infrared spectral range probe the response of the system by SHG. Analysis of the time-resolved non-linear optical response reveals that the

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Results

We have used single-cycle THz pulses to excite the ferroelectric. The result of the excitation was probed by a femtosecond laser with the central wavelength of 1240 nm. Upon non-linear interaction of this pulse with the ferroelectric medium, second harmonic light was generated with the central wavelength of 620 nm. The time delay $\tau_d$ between the THz-pump and optical-probe pulses was changed with the help of a delay line. The pump beam was at normal incidence. The angle of incidence of the probe beam was 23 degrees. Schematics of the experimental setup is shown in Fig. 1a. In the following orientation of the electric fields of the pump and probe pulses will be described in laboratory frame with $X_L$, $Y_L$, and $Z_L$ axes, as shown in the figure. The sample was oriented with its [100] axis parallel to the $X_L$ axis. The electric field vector of the THz pump pulse was oriented in the $X_L$-$Y_L$ plane at angle $\psi$ with respect to the $X_L$ axis. The electric field (i.e. polarization) of the near-infrared probe pulse could be rotated with respect to the $X_L$ axis by angle $\phi$ or set in the P-state ($\phi = 0$). The polarization of the SHG signal was set either in the $P_{out}$ or $S_{out}$-state. Dots correspond to experimental data and lines are fits (calculations see in Supplementary Information). Values for S-state multiplied by 3, 2 and 50 for (b–d) respectively.

Figure 1. Experimental geometry and polarization diagrams of the SHG intensity for various experimental geometries. (a) Experimental geometry. The axes of the chosen laboratory frame $X_L$, $Y_L$, $Z_L$ correspond to [100], [010] and [001] crystallographic directions, respectively. $\varphi$ - the angle between the electric field of near-infrared probe and the $X_L$-axis, $\psi$ - the angle between the electric field of the THz pump pulse and the $X_L$-axis; (b) dependence of the SHG signal on $\varphi$ without any THz pump; (c) dependence of the SHG signal on $\varphi$ when the THz field is applied parallel with respect to the $X_L$-axis. The polarization of the SHG signal was set either in the $P_{out}$ or $S_{out}$-state; (d) dependence of the SHG signal on $\psi$ when the probe polarization was set to the P-state ($\varphi = 0$). The polarization of the SHG signal was set either in the $P_{out}$ or $S_{out}$-state. Dots correspond to experimental data and lines are fits (calculations see in Supplementary Information). Values for S-state multiplied by 3, 2 and 50 for (b–d) respectively.

experimental results can be explained assuming that under action of the THz pulse the ferroelectric order parameter acquires an in-plane component up to 6% of the net polarization.
Hence the data show that the THz electric field clearly affects the process of the second harmonic generation. To reveal ultrafast dynamics of these electric field induced changes, we performed pump-probe measurements of the SHG signal. In particular, the signal was measured as a function of the delay $\tau_d$ between the THz-pump and near-infrared probe pulses. Figure 2a shows time-domain trace of the electric field of the THz pulse obtained with the help of electro-optical sampling. The measurements of the SHG signal from the BST film (Fig. 2b) reveal a similar dynamics during the overlap of the probe and THz-pump pulses. It points out that the non-linear response is proportional to the THz electric field.

For comparison, we also measured temporal evolution of the SHG signals from centrosymmetric crystals excited by the intense THz pulse. The SHG transients for SrTiO$_3$ (STO) and Si are shown in Fig. 2c (red and blue line, respectively). First of all, for the both cases of the centrosymmetric crystals the SHG signal of the unper-turbed media are zero. Secondly, in the maximum of THz modulated signal the absolute values of the intensity of the SHG from BST is two orders of magnitude higher than those for the centrosymmetric crystals. Thirdly, the shape of the SHG response differs from the shape of the THz pulse. Qualitatively, the frequency of the time transients of the SHG response for centrosymmetric STO and Si is doubled with respect to the frequency of the temporal variations of the electric field of the THz pulse.

The spectra obtained as Fourier transforms of the time-traces for BST and STO as well as for THz pulse are plotted in Fig. 2d and e. For BST, the frequency-domain signal follows the input pulse, while for STO the frequency is doubled. Power dependencies of the SHG intensity on the THz electric field are plotted in Fig. 2f in logarithmic scale and reveal linear and quadratic for BST and STO, respectively.

Finally, from the measured SHG traces we deduced how the induced electric polarization changes upon a change of the electric field of the THz pulse. These dependencies were compared with the hysteresis loops measured with the help of the Soyer-Tower technique (Fig. 3a, bottom solid line). The first, i.e. static hysteresis loop, is quite typical for BST films with 20% of Strontium $^{37}$. We also compared the data with loops obtained by measuring the SHG signal in an electric field which varies with the frequency of 100 Hz. To deduce the loops we employed the procedure of reconstruction of the electric polarization as described previously $^{28}$. It is based on the dependence of SHG intensity on ferroelectric polarization:

$$\Gamma^{\omega} = 1^{bg\omega} + \alpha(P_0 + P(E_\omega))^2, \quad (1)$$

where $1^{bg\omega}$ is the incoherent component of the unswitchable part of the second harmonic signal; $P(E_\omega)$ is the ferroelectric (switchable) polarization, which depends on the electric field of the THz pulse; $P_0$ is remanent polarization i.e. unswitchable polarization; $\alpha$ is the proportionality coefficient, which is determined by the Fresnel factors and the nonlinear optical susceptibility.

In order to deduce the field dependence of the electric polarization $P(E_\omega)$, one has to subtract from the SHG intensity the background signal $1^{bg\omega}$ which is independent on the electric field. Then one has to take the square root

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**Figure 2.** THz-induced dynamics of nonlinear-optical response of the crystals. (a) Time trace of the electric field of the THz pump pulse; intensities of the SHG-signal: (b) for BST, (c) for STO (red line) and Si (black line); (d–f) Fourier transform spectra of the time traces shown in panels (a–c), respectively ((f) plotted in doubled frequency scale); (g) dependence of the SHG intensity on the THz field (logarithmic scale) for BST (top line) and STO (bottom line).
from the residual signal. It gives the value, which is proportional to the electric polarization \(P_0 + P(E \Omega)\). The result of such a data processing is shown in Fig. 3a (top solid line). Additionally, we can mirror the loop with respect to the x-axis at negative electric fields and thus obtain a loop that resembles largely the dielectric hysteresis loop obtained in statics (Fig. 3a, dashed line). Since in the THz measurements both electric field and SHG intensity are measured as a function of time, hysteresis loop is set parametrically. After excluding time from these dependences and performing the same procedure for the SHG intensity (except taking the square root because of linear dependence of the SHG intensity), the THz SHG loop is obtained. In Fig. 3a points show hysteresis loops for two values of the THz electric field (E-field). Although these loops are quite noisy, a similarity with the low-frequency loop (with hysteresis and coercively) is obvious.

**Discussion**

In centrosymmetric crystals (SrTiO\(_3\), Si), in the electric-dipole approximation, it is possible to generate second harmonic signal by applying an external electric field, which breaks the inversion symmetry. This is so-called electric field induced second harmonic (EFISH). For electric field oscillating at THz frequency we will call it TEFISH. Taking into account that \(\Omega \ll \omega\) and \(\Omega + \omega + \omega \cong 2\omega\), TEFISH polarization can be described as

\[
\vec{P}(2\omega) = \chi^{(2)}E_\Omega \vec{E}_\omega + \chi^{(3)}E_y \vec{E}_\omega.
\]

Analogously to the linear optical Kerr effect, which is described by a tensor of the same rank, it has electronic and ionic contributions\(^{29}\).

In noncentrosymmetric crystals with nonzero electric dipole contribution \(\vec{P}_{\text{cryst}}(2\omega)\), formally the same electric field induced mechanism is also valid:

\[
\vec{P}(2\omega) = \vec{P}_{\text{cryst}}(2\omega) + \vec{P}_{\text{TEFISH}} = \chi^{(2)}E_\Omega \vec{E}_\omega + \chi^{(3)}E_y \vec{E}_\omega.
\]

For ferroelectric material electric field dependent nonlinear optical polarization has several contributions (analogously to linear optics\(^{29-31}\)): electronic, ionic and piezoelectric. As the employed THz pulses pump the medium in the range of phononic excitations, it is natural to assume that the THz electric field induces ferroelectric polarization due to the ionic contribution.

Without any electric field, the sample is split into two types of domains with the polarization aligned along the [001] axis. For nonzero angle of incidence, for the SHG intensity one finds

\[
I_{\text{SHG}} \sim (\vec{P}_{\text{cryst}}(2\omega))^2.
\]

When the in-plane electric field is applied at angle \(\psi\) with respect to \(X_x\) axis, a part of the domains line up along the field. In analogy with ref. 32, the net response will be defined by volume fractions of domains \(V^+_y\) and \(V^-_y\) (\(i = x, y\)) (\(i = x, y\)), where \(x, y\) denote the crystallographic axis along which the polarization is aligned, but “+” and “-” show the direction of the alignment. The differences of the fractions of the positively and the negatively oriented domains determines the electric field dependent contribution to the nonlinear optical polarization as

\[
P_i = P_{x0} + \Delta V P_y, \quad \Delta V_y = V^+_y - V^-_y.
\]

Thus, the volume contributions to the corresponding domain directions for any angle \(\psi\) of the applied THz E-field results in the following dependences:

\[
\Delta V_x = \gamma \cos \psi,
\]

\[
\Delta V_y = \gamma \sin \psi,
\]
where \( \gamma \) is the ratio of the fraction of in-plane switched domains to the fraction of [001]-oriented unswitched domains.

Generally, SHG intensity for the THz E-field oriented along arbitrary direction in the plane of the sample can be written as

\[
I_{2\omega} \propto \left( P_{2\omega}^{(0)} \Delta V_x + P_{2\omega}^{(10)} (E_{1\omega}) \Delta V_x + P_{2\omega}^{(10)} (E_{1\omega}) \Delta V_y \right)^2.
\]  

(6)

When the in-plane electric field is applied along the [100] axis \((V_x \neq 0, V_y = 0)\), a polarization is induced along the same axis and the intensity acquires an additional contribution:

\[
I_{2\omega} \propto \left( P_{2\omega}^{(0)} \Delta V_x + 2P_{2\omega}^{(10)} (E_{1\omega}) \Delta V_y \Delta V_x + (P_{2\omega}^{(10)} (E_{1\omega}) \Delta V_y \right)^2.
\]  

(7)

Figure 4 shows examples of polarization dependences obtained for different \( \gamma \): (a,d) \( \gamma = 0.05 \); (b,e) \( \gamma = 0.5 \); (c,f) \( \gamma = 1 \). Blue and red lines on (a and d) represent the linear plots of fitting curve from Fig. 1c and d.
Conclusions
In our experiments with ferroelectric thin film, we observed a very strong modulation of the SHG signal by the electric field of a THz nearly single cycle pulse. Polarization dependences of the SHG intensity were explained in terms of THz field-induced second harmonics generation. As the THz pulse pumps the medium in the range of phononic excitations, the modulation of the nonlinear signal can be explained in terms of a change of the ferroelectric order parameter. In particular, under action of the THz pulse the latter acquires an in-plane component up to 6% of the net polarization.

The net SHG signal responds to the THz excitation as if the THz pulse induces a 90-degrees switching of the polarization in parts of the sample. The possibility of such a switching on the time scale of the period of the soft mode was recently suggested by ref. 34.

Methods

Samples. Heteroepitaxial ($Ba_0.8Sr_0.2$)TiO$_3$ (BST) thin film (thickness 500 nm) was deposited on MgO (001) substrate by RF-sputtering of stoichiometric polycrystalline target. Transparent and mirror-smooth film was realized by means of layer-by-layer growth (Frank-van der Merwe mechanism). Details of the growth conditions have been previously reported in ref. 35. The vertical and azimuthal film misorientations were found to be less than 0.4° by high resolution XRD analysis. ($Ba_0.8Sr_0.2$)TiO$_3$ solid solutions belong to ferroelectric perovskites. In the paraelectric phase, they have a tetragonal unit cell (space group Pm3m). In the ferroelectric phase below the Curie temperature ($T_c$ = 353 K) they have a tetragonal unit cell (space group P4mm, point group 4mm). At room temperature, the soft mode is overdamped with the following parameters: $\Omega = 46$ cm$^{-1}$ (2.40 THz), $\Delta \Omega = 50$ cm$^{-1}$ (1.57 THz)$^{36,37}$. The as-grown film consists of 180-degrees domains which are not compensated due to an interfacial strain and show built-in polarization in the [001] crystallographic direction$^{38,39}$. The size of domains is about 500 nm.

Experimental setup. The THz pulses were generated by optical rectification of the femtosecond laser pulses, generated by high-energy 10 Hz Cr:Forsterite chirped-pulse amplification laser at the central wavelength of 1240 nm with 100 fs pulse duration and 20 mJ output energy, in the nonlinear organic crystal OH1$^{39}$. The polarization of the terahertz pulse coincided with the polarization of the femtosecond pump laser pulse. The rotation of the terahertz electric field was carried out by simultaneous rotation of polarization of incident beam and nonlinear organic crystal OH1 on the same angle. The energy of the THz pulses was up to 2 $\mu$J at pump laser energy of 700 $\mu$J. Two parabolic mirrors were used to guide and to focus the THz pulses on the sample surface. The first mirror collected and collimated THz pulses emitted from the nonlinear crystal. The second one focussed the THz beam into a spot with the diameter of 900 nm. Such parameters provided a high amplitude of the THz electric field up to 300 kV/cm. The amplitude of the THz electric field was estimated based on the measurements of the pulse duration and pulse fluence. We have used about 3% of the output energy of the Cr:Forsterite laser beam reflected by a thin optical glass plate to probe the electric polarization in the studied medium. These optical probe pulses passed through a delay line and were focused into a spot of 200 $\mu$m in diameter. After the sample, the fundamental optical radiation at the wavelength of 1240 nm was blocked and the SHG signal at the wavelength of 620 nm was transmitted by a bandpass filter ($\Delta \lambda = 10$ nm). The SHG signal was detected by a Photo-Multiplier Tube in the current regime.

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Author Contributions

A.V.K. together with M.B.A. conceived the project. K.A.G., N.A.I., A.V.O. and O.V.C. designed experimental set-up and carried out experiments. K.A.G. automated experimental set-up and prepared the figures. V.M.M. prepared the samples and performed electro-physical measurements. E.D.M. and N.E.S. performed data analysis and developed phenomenological description. E.D.M and N.E.S wrote the paper with contributions from A.V.K and M.B.A. All authors discussed the results and commented on the manuscript.

Additional Information

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