Nanoscale heat transport studied by high-resolution time-resolved x-ray diffraction

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Abstract. We report on synchrotron-based high-repetition rate ultrafast x-ray diffraction (UXRD) experiments monitoring the transport of heat from an epitaxial La$_{0.7}$Sr$_{0.3}$MnO$_3$/SrTiO$_3$ superlattice (SL) into the substrate on timescales from 100 ps to 4 µs. Transient thermal lattice expansion was determined with an accuracy of 10$^{-7}$, corresponding to a sensitivity to temperature changes down to 0.01 K. We follow the heat flow within the SL and into the substrate after the impulsive laser heating leads to a small temperature rise of ΔT = 6 K. The transient lattice temperature can be simulated very well using the bulk heat conductivities. This contradicts the interpretation of previous UXRD measurements, which predicted a long-lasting expansion of SrRuO$_3$ for more than 200 ps. The disagreement could be resolved by assuming that the heat conductivity changes in the first hundred picoseconds.

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1. Introduction

The development of tailored materials on the nanometer scale demands a detailed understanding of thermal transport properties, especially for multi-gigahertz nanoelectronics and superlattices (SLs) for thermoelectrics [1–4]. Ultrafast x-ray diffraction (UXRD) at synchrotrons offers a direct material-specific probe of local heat expansion on the timescale down to 100 ps, relevant for heat transport on this small length scale [5]. The material is excited (pumped) periodically by short laser pulses which are electronically synchronized with the periodic synchrotron x-ray pulses. This technique has been successfully applied to the detailed analysis of lattice expansion due to coherent acoustic phonons (sound waves) in bulk semiconductor materials [6], to the measurement of ultrafast dynamics in semiconductor SLs [7] and to the unfolding of these SL phonons when they propagate into the substrate [8]. A recent report extends these investigations to the expansion of oxide SLs [9], which have been explored with a time resolution of 150 fs using femto-slicing at a synchrotron [10]. The driving mechanism of coherent acoustic phonons is classified as thermal expansion, in which both electrons and phonons may contribute to the expansion [11]. The coherent acoustic phonons propagating at the speed of sound leave the SL within 80 ps after excitation.

In this paper, we study thermal lattice expansion in a pseudomorphic La$_{0.7}$Sr$_{0.3}$MnO$_3$ (LSMO)/SrTiO$_3$ (STO) SL on timescales from 100 ps to 4 $\mu$s after laser heating of the metallic LSMO layers. X-rays penetrate into the materials, which can be identified in UXRD by their distinct Bragg angles. The individual lattice expansions are deduced from the transient shift of the x-ray diffraction (XRD) curves with an unprecedented accuracy of $10^{-7}$, owing to the high repetition rate (208 kHz) of the laser system. The experimental observations are in excellent agreement with heat diffusion simulations on timescales from 4 ns to 4 $\mu$s, yielding typical bulk values for heat conductivity in the SL. The SL thickness is 338 nm and consists of 15 periods of LSMO/STO double layers epitaxially grown on STO (001) substrate. The LSMO layers are opaque to the 800 nm laser radiation and absorb energy, while the STO layers are transparent.

It has been reported that for a similar SrRuO$_3$ (SRO)/STO SL, the absorbing SRO layers remain considerably expanded even after 200 ps, while at the same time the insulating STO layers are expanded only slightly [12, 13]. On this timescale no modulation due to coherent acoustic phonons remains in the SL and only the hot phonons or electrons contribute to the ‘thermal’ expansion.

We will show that in order to agree with the above observations, the heat diffusion equation would require extremely low values for the heat conductivity of the SL, compared with that of the bulk. Although there are many reports on additional heat resistance in SLs due to nanometer layer thickness and the presence of interfaces, we estimate that the accepted formulae do not
predict considerable deviation from bulk [1, 14]. This may indicate that transient processes reduce the heat conductivity on timescales of $\sim 100$ ps after excitation.

2. Instrumental setup

The pump–probe setup is assembled at the storage ring BESSY II (Berlin, Germany). The EDR bending magnet beam line provides x-ray radiation in the broad energy range of 4–15 keV, which is monochromatized by a single Si(111) crystal. The total monochromatic photon flux was $2 \times 10^5$ photons (s mA mm$^{-2}$)$^{-1}$ at the energy of 8.9 keV.

The pump–probe repetition rate is a compromise between the pulse energy required to induce large structural changes and the laser power. In typical UXRD setups, the repetition rate is of the order of a few kilohertz, allowing for high pump fluences [5, 10, 15]. Such excitation conditions often result in nonlinear sample response. In some cases, a low pulse intensity is preferable where the effects induced in the sample are linear. In this case, the laser repetition rate can be increased, resulting in an advantageous increase of the useable x-ray photon flux. The repetition rate of the x-ray probe photons of 1.25 MHz is determined by the round-trip time of the electron bunches in the storage ring. This high repetition rate facilitates shot-noise-limited single x-ray photon counting and therefore allows detection of weak changes in the sample.

Figure 1 shows a schematic diagram of the experimental setup for the UXRD experiments. A Ti:sapphire laser system (Coherent RegA) with the pulse duration set to 350 fs was synchronized with the master clock of the storage ring in order to operate at one-sixth of the 1.25 MHz ‘bunch marker’ signal ($\sim 208$ kHz). The laser has a synchronization loop in which the actual laser output is detected and compared with the master clock. To keep the output pulses
Figure 2. Typical synchrotron bunch pattern including the optical laser pulse as a function of the single-photon arrival time $\tau$. The full time interval corresponds to the laser repetition rate of $\sim 208$ kHz and is equal to the duration of six electron revolutions in the ring.

synchronized with the ring clock, the length of the laser oscillator cavity is actively controlled and continuously adjusted. The ultrashort laser pulses are used to impulsively heat the SL with an absorbed fluence of $0.7 \text{ mJ cm}^{-2}$. The change in temperature is followed by UXRD.

For the detection of the x-rays, we used a plastic scintillator ($<0.5$ ns rise time). The scintillator converts the x-ray radiation into optical photons with a typical yield of $10,000$ photons MeV$^{-1}$. For a single $8.9$ keV photon about $100$ optical photons are generated, detected by a photomultiplier (Hamamatsu) and sent via a current amplifier (FEMTO) to a single-photon counting module (PicoQuant ‘PicoHarp’) for counting the current pulses. The x-ray photon flux per bunch was small enough to prevent counting multiple x-ray photons as a single event. As the plastic scintillator is transparent for the laser light, the same detector is used to detect attenuated laser pulses. This allows us to coarsely adjust the temporal overlap between the laser pump and x-ray probe pulses.

During the diffraction experiment with full laser power, the detector is shielded by an Al-coated Be window that fully blocks the optical laser reflected from the sample. Figure 2 shows the histogram of the x-ray pulse sequence as a function of the single photon arrival time $\tau$ together with the pump–laser pulse. The time structure of the electrons in the ring becomes visible after summing up the x-ray photon events repeatedly using the $\sim 208$ kHz trigger of the synchronized laser. The histogram shows six periods of the ring bunch pattern, a sequence of $350$ bunches with a period of $2$ ns is followed by a $100$ ns gap filled by a single x-ray pulse (hybrid mode of BESSY II). The strong single spike at $2694$ ns in the histogram corresponds to the optical laser pulses with $\sim 208$ kHz repetition rate. Thus, for every sixth isolated x-ray pulse the sample is excited by the pump pulse. The accuracy in determining the preliminary time overlap using this method is limited by the scintillator response time jitter, which is of the order of half a nanosecond. For more accurate x-ray and laser timing (within $100$ ps) we use ultrafast processes, which occur in the sample after laser excitation described in the next
Figure 3. (a) Experimental diffraction curve (black dots) of the LSMO/STO SL near the (0 0 2) substrate peak and simulation (blue line) according to dynamical diffraction. (b) Barely visible transient shift of the substrate peak (red line: at 4 µs; green line: at 100 ns). (c) Diffraction curves of the zero-order peak (ZOP) of the SL (dash dotted black line: no laser on the sample; dashed red line: at 4 µs; solid green line: at 100 ps). Note the substantial permanent heating (26 K) induced by the laser pulse. The time dependence of the transient heating (positions of green curves in (b) and (c)) is plotted in figure 4.

section. The pump–probe delay can be set by varying the optical path length of the laser with sub-picosecond precision; however, the timing-jitter of the electronic synchronization is of the order of 1 ps and the actual time resolution is given by the x-ray pulse duration of ∼100 ps.

3. Experimental results

We typically scan θ–2θ diffraction curves, keeping the electronically and optically controlled time delays fixed. This allows for the observation of all shape modifications of the rocking curve, including peak shifts and intensity changes. By varying the optical delay for each scan we are able to monitor the XRD changes on sub-nanosecond timescales.

An extended θ–2θ diffraction curve of the LSMO/STO multilayer is shown in figure 3. It consists of the (0 0 2) Bragg reflection of the STO substrate and a number of SL Bragg reflections. The spacing of the SL reflections is defined by the SL period, while the width of the SL reflections is inversely proportional to the total SL thickness.

The position \( q^{(0)} = \frac{2\pi}{c_{av}} \) of the so-called zero-order SL peak (ZOP) corresponds to the average lattice constant of a single double layer (DL) which is given by

\[
c_{av} = \frac{n_M c_M + n_I c_I}{n_M + n_I},
\]

where \( n_M \) and \( n_I \) correspond to the number of unit cells in the metallic LSMO and insulating STO layers with lattice constants \( c_M \) and \( c_I \), respectively. The ZOP is the brightest SL reflection since it profits from the diffraction efficiencies of both bulk constituents, which are significant.
(large envelope) only around their bulk Laue conditions in a $q$-range given by the inverse thickness ($n_M c_M$ and $n_I c_I$) of a single metallic or insulating layer.

The energy deposited in the SL induces its thermal expansion. The time it takes a sound wave—a wavepacket of acoustic phonons—to propagate through the SL is 40 ps [16]. Therefore, at delay times of about 100 ps after excitation, the strain in the SL is governed only by the transient temperature profile.

Figure 3(c) shows the ZOP at 100 ps after laser excitation (solid green), 4 $\mu$s later (red dashed) and without laser illumination of the sample (black dotted). The shift of the diffraction peak at 4 $\mu$s delay describes the static, average temperature rise of the SL, which depends on the average power of the laser. The large shift with respect to the black dotted curve shows that the high average power of the laser leads to a rather large average temperature rise. Clearly the solid green line recorded at a delay of 100 ps shows an additional shift toward lower angles. This demonstrates that laser excitation leads to an ultrafast expansion of the SL, which relaxes toward the static, average thermal expansion on the nano- to microsecond timescale.

Figure 3(b) shows the tiny shift of the substrate reflection after 100 ns (solid green) compared with 4 $\mu$s (red dashed) after excitation.

We note that the diffraction curve at 4 $\mu$s is obtained by counting the corresponding x-ray photons (see figure 2) without the need for scanning the delay. Therefore, for each individual optical delay position the reference peak for 4 $\mu$s delay is measured simultaneously, allowing for a very precise determination of transient peak shifts (for a delay of 4 $\mu$s the actual picosecond delay can be neglected). In addition, the single-photon counting eliminates photomultiplier noise or gain fluctuations, leading to an unprecedented level of accuracy.

Figure 4(a) shows transient peak shifts of the ZOP (blue bullets) and the substrate (0 0 2) reflection (black squares) on the picosecond timescale. The transient shift of the ZOP is fitted by an error function, the derivative of which is a Gaussian with full-width at half-maximum (FWHM) of 100 ps. This is approximately the width of the x-ray pulse. The delay time zero, where x-ray and laser pulses coincide, is therefore given by the time when the peak is half-way shifted. We see no shift of the substrate peak on this timescale, since there is not enough time for the heat to propagate from the SL deep into the substrate. Notably, the SL peak is already shifted for negative time delays by approximately one-tenth of the maximum shift. This is probably due to pre-pulses of the laser system which pump the sample before the main pulse.

The shift of the Bragg peaks can be used to determine the temperature change of the SL and substrate from the relation

$$\cot \theta \Delta \theta = -\alpha \Delta T,$$

where $\alpha$ is the linear thermal expansion coefficient of the corresponding material, $\Delta \theta$ is the change of the Bragg angle from its stationary value $\theta$ and $T$ is the temperature. We take the thermal expansion coefficient measured in thin films, since epitaxial strain may modify the expansion in an anisotropic way. Figure 4(c) shows the transient peak shifts and temperature rise of both the substrate and the SL on an extended timescale. The substrate peak is sensitive to the averaged temperature of the first few microns of the substrate; therefore the shift appears much later than that for the SL. As soon as the heat has diffused deeper than the x-rays penetrate, the shift starts to vanish again. It is worth noting that statistical error bars for the peak positions are significantly smaller for longer times, since more x-ray pulses separated by only 2 ns can be used for counting at fixed resolving power $\Delta t / t$ (cf figure 2), resulting in an increased count rate. The standard deviation for the relative change of the substrate peak position
Figure 4. (a) Transient peak shift of the substrate peak (small black dots) and ZOP of the SL (large blue dots). The error-function fit (blue line) corresponds to the Gaussian FWHM of the synchrotron pulse with a duration of $\sim 100$ ps. (b) The same peak shifts on longer timescales, together with fitting curves according to the diffusion equation with the heat conductivity of $6.9 \text{ W (m K)}^{-1}$ (solid lines). The dashed lines indicate the crude misfit of a simulation with ten times smaller heat conductivity than predicted by previous experiments \[12\] (see text). (c) The same as (b), but with a clearer visibility of the very small error bars down to $10^{-7}$.

is less than $10^{-7}$ degrees, which corresponds to an accuracy in the temperature measurement of 0.01 K.

4. Discussion and simulation of the heat flow in La$_{0.7}$Sr$_{0.3}$MnO$_3$/SrTiO$_3$ superlattices

The experimental measurement of the transient shift of the SL and substrate Bragg peaks (figure 4) can be readily translated into a transient temperature change indicated on the right abscissa using equation (2) with the thermal expansion coefficient $\alpha_{\text{SL}}$ of the SL. Thus, the experiments quantify the heat flow from the SL into the substrate that occurs on the 10–100 ns timescale. The temperature rise in the substrate is smaller because the detection volume is given by the extinction depth of the x-rays ($\approx 1.5 \mu\text{m}$), which is five times larger than the SL thickness. In addition, the temperature rise profile of the excited SL decays as the heat flows into the substrate.
Table 1. Properties of the LSMO/STO SL used in the calculations. The results obtained from simultaneously fitting the SL and substrate data in figure 4 (blue and black solid lines) are in boldface.

| Physical property        | Units          | Material  |
|-------------------------|----------------|-----------|
| Thickness               | nm             | LSMO 8.85 | STO 13.7 | SL 338 |
| Density                 | kg m$^{-3}$    | LSMO 5600 | STO 5120 | SL 5308 |
| Thermal conductivity $\kappa$ | W (m K)$^{-1}$ | LSMO 2.8 [18]–4 [19] | STO 11.1 [20] | SL 6.9 |
| Heat capacity $C$       | J (kg K)$^{-1}$ | LSMO 545.4 [21] | STO 530 [22] | SL 536 |
| Thermal expansion coefficient $\alpha$ | K$^{-1}$ | LSMO 1.16 × 10$^{-5}$ [23] | STO 1.1 × 10$^{-5}$ [23] | SL 1.1 × 10$^{-5}$ |
| Speed of sound          | m s$^{-1}$    | LSMO 6500 [24] | STO 7800 [24] | SL 7232 |

For a quantitative comparison, we simulated the heat transport in a first approximation by treating the SL as a medium with an effective thermal conductivity $\kappa_{\text{SL}}$ and heat capacity $C_{\text{SL}}$. Although $C_{\text{SL}}$ can be calculated as a weighted average of the constituent layers (table 1), the situation for the conductivity is more complex and we choose to determine this value from a fit to the experimental data. For macroscopic systems the heat resistance of a layered material is the direct sum of the heat resistances of its components. Deviations from the simple sum rule were observed on small length scales in SLs, in which the high densities of interfaces result in additional phonon scattering mechanisms and the wave properties of the heat-carrying phonons are modified by zone folding [1, 2]. In the present SL, we can disregard interference since the mean free path of phonons [17] at room temperature is $L = 3\kappa/C \cdot v \approx 1$ nm, where $C$ is the average heat capacity and $v$ the average group velocity of acoustic phonons. In any case, all these additional phonon scattering mechanisms result in a permanent change of heat transport properties, which should be visible on all timescales.

In the following, we show that the static change of heat transport in our SL is negligible, but there must be mechanisms that result in transient effects on heat transport. We use a one-dimensional (1D) model, since the lateral size of the excited region is much larger than the probed depth of the sample. In this model, heat is initially distributed within the 15 LSMO layers of the SL according to the exponentially decaying absorption profile for the 800 nm light pulses. STO is transparent at this wavelength. The heat then equilibrates among the LSMO and STO layers and finally diffuses into the substrate. The thermal properties of the materials are taken from the literature (see table 1). The heat conduction coefficient of the SL and of the substrate, as well as the total absorbed energy density, were free parameters that were determined by solving the diffusion equation, computing the expected Bragg peak position and varying the parameters until the deviations of both experimental transients (SL and substrate) and simulations were minimized globally for all delays.

The results of the heat conduction simulations are shown in figure 5. To check the numerical computation accuracy, the total heat in the sample was calculated for each time. It remains constant within 0.8% throughout the simulated time axis. A specific variation of the...
Figure 5. Contour plot of the calculated temperature rise of the sample as a function of depth and time according to the diffusion equation with the heat conductivity of $6.9 \text{ W (m K)}^{-1}$, which leads to the best fit in figure 4. Note that this simulation predicts complete equilibration of heat between the STO and LSMO layers of the SL within 10–20 ps.

heat conduction coefficients of either the LSMO or the STO layer or the interface does not affect the fit as long as their weighted averages coincide. Figure 4(c) shows that the 1D heat conduction model reproduces the experimental data up to 1000 ns excellently. At later times, the measured cooling rate is higher than predicted in the 1D simulations because the in-plane heat conduction can no longer be neglected and the problem becomes 3D.

The fitted heat conduction coefficient for the SL $\kappa_{\text{SL}} = 6.9 \text{ W (m K)}^{-1}$ agrees well with the value obtained from the sum of heat resistances, with an expected range of $\kappa_{\text{sum,SL}} = 5.2–6.5 \text{ W (m K)}^{-1}$ given by the spread in the literature data for $\kappa_{\text{LSMO}}$ (see table 1). Thus no deviation from the bulk heat conduction due to the nanoscale size of the SL is observed in our experiment.

The effects due to the interference of phonon waves in the SL are negligible according to the small phonon mean free path $L = 1 \text{ nm}$. Assuming fully diffuse and inelastic scattering (in the high temperature limit) and neglecting scattering within the layers, the interface conductivity in the SL can be estimated as [1, 14]

$$\kappa_{\text{SL}} = \frac{C_1 v_1 C_2 v_2}{C_1 v_1 + C_2 v_2} \frac{d_1 + d_2}{2},$$

where $C_i$ is the specific heat, $v_i$ is the sound velocity and $d_i$ is the layer thickness of layer $i$. This formula yields $\kappa_{\text{SL}} = 122 \text{ W (m K)}^{-1} \gg \kappa_{\text{sum,SL}}$, so neglecting the interface scattering is also a valid approximation.

Remarkably, the simulation of heat transport reported in figure 5 predicts that the temperature completely equilibrates within 10–20 ps between the STO and the LSMO layers.
of the SL. This is in stark contrast to previous UXRD measurements on a comparable \textsuperscript{8} SL of the SRO/STO layer, where it has been argued that the heat remains within the metallic SRO layers for at least 200 ps after excitation \cite{12, 13}. In order to increase the LSMO/STO temperature equilibration time in our simulations by one order of magnitude to match \textit{this} equilibration time of at least 200 ps reported in the literature \cite{12, 13}, the effective heat conduction within the SL would have to be reduced by one order of magnitude. For such parameters the heat conduction equation predicts the temperature change shown in figure 4(c) by the dashed lines, which show a strong deviation from the experimental curves in the 1–200 ns time range.

The observation of poor heat conduction shortly after the laser pulse excitation and bulk heat conduction for nanosecond timescales suggests that the thermal conductivity is time dependent. This may be a manifestation of the fact that the concept of thermal equilibrium is not applicable within the first hundred picoseconds. While electron–phonon scattering is much faster (∼1 ps), optical phonons are predominantly excited in ionic crystal lattices. The equilibration of optical and acoustic phonon temperatures may require much longer in oxide SLs. The energy and wavevector of these optical modes cannot be conserved in the dominant optical phonon decay channel into two acoustic phonons of opposite \textit{k} vector known from semiconductors such as GaAs, since the energy of the optical phonons is larger than that for the acoustic phonons with the highest energy. Since optical phonons have a group velocity close to zero, they propagate heat less efficiently. Only after an equilibration time of about 100 ps does the population of the heat-carrying acoustic modes become considerable, supporting efficient heat transport comparable to the bulk heat conduction in thermal equilibrium. This would imply that deposition of heat by optical excitation, used in many heat pulse experiments, could only be described by an effective heat conduction coefficient that is time dependent on the sub-nanosecond timescale. In this scenario, the heat would be confined to the SRO layers after 200 ps, as demanded in \cite{12, 13}, although the heat conduction on the nanosecond timescale is described by the bulk values observed here. Rather challenging future experiments that measure several Bragg reflections simultaneously can in principle clarify whether the rapid heat diffusion within the SL implied by figure 5 really takes place.

In conclusion, we have demonstrated how UXRD at a high repetition rate (208 kHz) can be used to accurately measure the heat diffusion in multilayered samples on the nanoscale and on the timescale from 100 ps to a few \textmu s. The heat conduction simulations using bulk constants describe very well the thermal diffusion from an SL into the substrate on the nanosecond timescale. However, the same simulations predict that heat transport in SLs with layer periods in the nanometer range proceeds in a few picoseconds. This is in clear disagreement with the interpretation of previous UXRD work, which has measured strong differences in the heat expansion of the constituting layers even after 200 ps. The combination of these results suggests that on very short timescales the parameter describing thermal conductivity becomes time dependent.

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\textsuperscript{8} SRO is a ferromagnetic metal with the same crystal structure and a heat conductivity \cite{25} of 5.7 W (m K)\textsuperscript{-1}, which is somewhat larger than the value for LSMO. The layering sequence is very similar as well: \(d_{\text{SRO}} = 7.5 \text{ nm}\) and \(d_{\text{STO}} = 12.9 \text{ nm}\).
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