Probing Ultrafast Carrier Dynamics in Silicon Nanowires

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(Invited Paper)

***Abstract*—We present the first ultrafast optical pump–probe spectroscopic measurements, to the best of our knowledge, on silicon nanowires (SiNWs). In this study, we performed femtosecond pump–probe measurements on vapor–liquid–solid-grown SiNWs to investigate the influence of the NW diameter, pump and probe polarizations, and pump fluence on the observed dynamics while tuning the probe wavelength below and above the indirect bandgap in Si. For smaller NW diameters, carriers were found to relax more rapidly into both extended and localized states, indicating that a surface-mediated mechanism governs the observed dynamics. The magnitude of the photoinduced transmission change exhibited strong polarization dependence, showing that optical transitions in these quasi-1D systems are highly polarized along the NW axis. Finally, density-dependent experiments revealed that the relaxation time decreases with increasing photoexcited carrier density for an above bandgap probe; however, no significant density-dependent changes in the relaxation dynamics were observed when probed below the bandgap. In short, our experiments reveal the influence of diameter, polarization, and carrier density on carrier dynamics in SiNWs, shedding light on the phenomena that govern carrier relaxation in these important nanosystems and giving insight on their future use in nanophotonic applications.**

***Index Terms*—Nanotechnology, optical spectroscopy, semiconductor materials, silicon, ultrafast optics.**

I. Introduction

SEMICONDUCTOR nanowires (NW) have been the subject of great interest in recent years, as their quasi-1D nature gives rise to many unique properties while making them particularly suitable for many applications. One of the most promising of these applications is in nanophotonics, where they can potentially serve multiple roles (e.g., transistors, light emitters, and photodetectors) within photonic and electronic devices that are integrated on a single chip. Silicon NWs (SiNWs) are particularly well suited for this purpose, as modern electronic devices are typically based on silicon, and therefore, it would be advantageous to explore a NW material that could easily be integrated with these devices from a compatibility and manufacturing point of view. There has already been some initial success in understanding growth, controlling dopant incorporation and developing a process for large-scale direct assembly of SiNWs to prepatterned electrical contacts [1]. These advances open up the possibility of incorporating SiNWs into mainstream semiconductor technology. In fact, SiNWs have already been used as active components in a wide variety of proof-of-principle photonic and electronic devices, such as transistors [2] and photodetectors [3]. In addition, efficient photoluminescence (PL) has been observed in Si nanostructures, such as porous Si and Si nanocrystals [4], [5], raising hopes that SiNWs can overcome the inefficient light emission from bulk Si that limits its use in nanophotonics. This is further supported by the observation that the energy gap in SiNWs was found to increase with decreasing NW diameter (d) from $E_g = 1.1\,\mathrm{eV}$ for $d = 7$ nm to $E_g = 3.5\,\mathrm{eV}$ for $d = 1.3$ nm [6]. This interesting experimental demonstration could lead to applications such as SiNW-based light-emitting diodes and lasers, since the wide bandgap is predicted to be direct [6]. Hence, the recent progress in large-scale production of uniform, thin, and clean SiNWs [7] enables an in-depth investigation of their optical properties and will aid in determining potential applications.

As in most NW systems, the most heavily studied optical property of SiNWs is their PL, due to the relative ease of performing PL experiments. However, several studies on SiNWs have found that defect states strongly contribute to the observed PL spectra, leading to emission from visible to IR frequencies [8]–[10]. These defects have been variously attributed to oxygen impurities [11], [12], Au contamination from the catalyst nanoparticles [13], and defects at the interface between the Si core and the postgrowth native oxide layer [14]. The net result of these studies is to demonstrate that SiNWs contain defect states both above the conduction band edge as well as throughout the bandgap; the latter can trap carriers from the band edge before they can radiatively recombine, which would reduce the efficiencies and raise the thresholds of potential SiNW-based LEDs and lasers.

Although considerable progress has been made in investigating the source of luminescence and identifying defect states in SiNWs, further progress in the area of SiNW-based optical

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device design will depend on an understanding of nonequilibrium carrier dynamics in these nanostructures on a femtosecond (fs) timescale. This will lead to new insights into the influence of the defect states on carrier dynamics in SiNWs, in particular the competition between rapid carrier trapping and radiative recombination. Although carrier relaxation in SiNWs has been recently investigated on the nanosecond timescale using time-resolved PL (TRPL) spectroscopy [15], no in-depth study has been done to date on how carrier relaxation occurs in SiNWs at shorter timescales, which could yield valuable information on how defect states influence carrier relaxation immediately after injection. In addition, one limitation of TRPL is that it can only directly probe radiative transitions, which are proportional to the *product* of electron and hole distributions (i.e., both an electron and a hole must have the same momentum for radiative recombination to occur with high probability). In contrast, ultrafast pump–probe spectroscopy is sensitive to the *sum* of electron and hole distributions at a given photon energy (i.e., only one species of carrier is enough to modify the absorption of subsequent photons) and therefore has the potential to probe both radiative and nonradiative transitions with fs time resolution, giving deeper insight into energy transfer processes that occur on a subpicosecond timescale [16].

Here, we present the first fs optical spectroscopic measurements on SiNWs to the best of our knowledge. In particular, we examined the influence of changes in the NW diameter on ultrafast carrier dynamics by measuring the time-resolved photoinduced change in transmission ($\Delta T/T$) of a probe laser pulse tuned above and below the indirect bandgap in Si. As in our previous observations on germanium NWs (GeNWs) [17], carriers were found to relax faster in thinner NWs, indicative of a surface mediated mechanism. However, for the SiNWs studied here, we observed a reversal of this trend for NWs with $d > 80$ nm when probing above the bandgap. In addition, the $\Delta T/T$ signal measured below the bandgap for $d = 10$ nm NWs strikingly differed from the signals measured on larger NWs, possibly due to quantum confinement effects. We also found that the magnitude of the measured $\Delta T/T$ signals exhibit strong polarization dependence, confirming that optical transitions and carrier generation are highly polarized along the NW axis. Finally, we explored the dependence of relaxation rates on the photoexcited carrier density, studies that are particularly relevant to applications in NW lasing. These measurements show a strong density dependence when probed above the bandgap, with faster relaxation at higher pump fluences that may be due to Auger processes. However, there was no significant change in the relaxation dynamics when probing mid-gap defect states as a function of pump fluence. Overall, these first ultrafast optical experiments on SiNWs give insight on carrier relaxation processes after fs photoexcitation, revealing dynamics similar to that observed in other NW systems [16]–[19], [21] along with physics that may be unique to SiNWs.

II. Experimental Details

The vapor–liquid–solid (VLS) technique was used to grow SiNWs through chemical vapor deposition [1], [20] on Si(111) substrates, using different diameter Au colloids (10, 30, 50, 80, and 100 nm) with a SiH_4 (50% in H_2) gas precursor at a temperature of 530 °C and a silane partial pressure of 1.5 Torr. Fig. 1(a) shows an SEM image of a typical dense array of randomly positioned SiNWs. TEM analysis [see Fig. 1(b)] showed that the NWs were single crystalline, as indicated by the clean lattice fringes with a $\langle 111 \rangle$ growth orientation. We note that for the SiNW diameters investigated here with $d > 10$ nm, bulk bandgap values are assumed, with $E_{\Gamma 1} = 3.4\,\mathrm{eV}$ (365 nm), $E_g = 1.12\,\mathrm{eV}$ (1107 nm, the indirect band gap), $E_X = 1.2\,\mathrm{eV}$ (1033 nm), and $E_L = 2\,\mathrm{eV}$ (620 nm) [22]. These structures were then dry transferred onto a sapphire substrate for optical measurements with an areal density of $5.5 \times 10^6\ \mathrm{NWs/cm^2}$. It is important to note that we acquired time-resolved data at three to five locations on each sample and averaged the results to minimize effects originating from the nonuniformity of the NW ensemble across the sample surface, which caused the amplitude of the measured signals (but not the dynamics) to vary substantially from spot to spot [21].

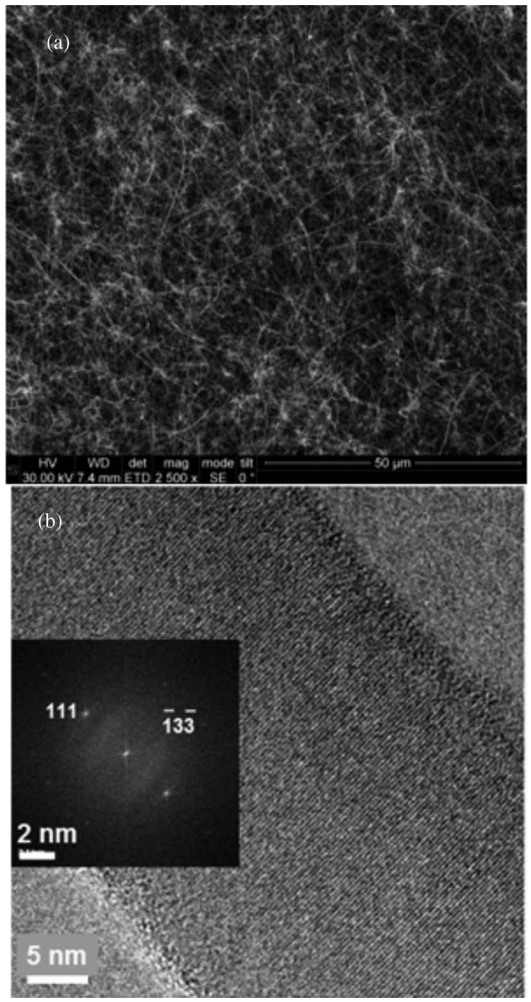

Fig. 1. (a) SEM images of VLS-grown 30-nm SiNWs. (b) TEM image of a 32-nm-diameter SiNW showing (111) lattice fringes. Inset: a fast Fourier transform of the TEM image, indicating the $\langle 111 \rangle$ growth orientation.

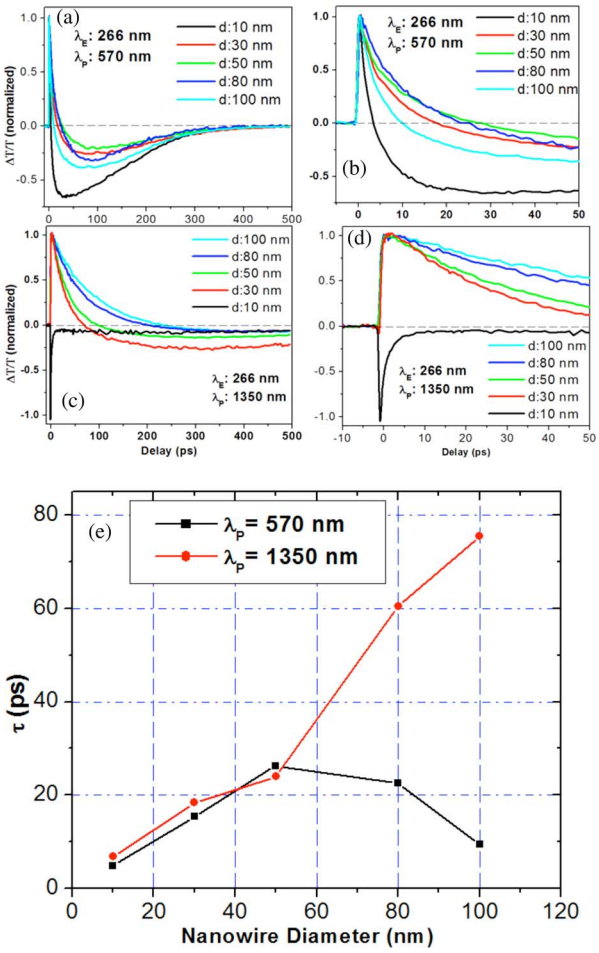

Fig. 2. (a) UV-pump (266 nm), visible-probe (570 nm) measurements on SiNWs of various diameters; (b) data from (a) during the first 50 ps. (c) UV-pump (266 nm), IR probe (1350 nm) measurements on SiNWs of various diameters; (b) data from (c) during the first 50 ps. (e) Graph showing the variation of τ with NW diameter at 570 and 1350 nm.

A 100-kHz regeneratively amplified laser producing 50 fs, 10 μJ pulses at 800 nm (1.55 eV) was used to pump a visible optical parametric amplifier (OPA), as well as to generate 266 nm (4.66 eV) pulses in a nonlinear optical crystal through third harmonic generation. The signal wavelength from the OPA was tuned to generate the probe pulses, with wavelengths of 570 nm (2.17 eV), 1045 nm (1.18 eV), and 1350 nm (0.92 nm). The 570 and 1045 nm probes are absorbed through indirect (phonon-assisted) transitions, while the 1350 nm probe and high-energy pump photons are directly absorbed. All experiments were performed at room temperature. Further details of the pump–probe experiments have been given elsewhere [21].

III. Discussion

A 266-nm ultraviolet (UV) pump pulse was used to excite the SiNWs well above the bandgap, creating nonequilibrium carriers in the valence and conduction bands with excess kinetic energy. In Fig. 2, we present a diameter-dependent study of the SiNWs at two probe wavelengths, 570 and 1350 nm, probing above the *L* valley and below the *X* valley, respectively, to obtain a relationship between carrier lifetimes and NW diameter. The pump fluence in these experiments was $400\,\mu\mathrm{J/cm^2}$, corresponding to an approximate initial photoexcited carrier density of $N \sim 3.2 \times 10^{19}\,\mathrm{cm^{-3}}$. With the 570 nm probe, the general trend in the signals seen in Fig. 2(a) for all diameters is an initial decrease in the probe absorption through state filling, resulting in a positive normalized pump-induced change in probe transmission ($\Delta T/T$), with typical amplitudes of $\sim 5 \times 10^{-3}$ at $t = 0$. This is followed by a decrease in the probe transmission, which crosses zero around ∼5–25 ps and reaches a minimum around ∼25–75 ps, until the carriers return to equilibrium around ∼350–400 ps. Physically, after photoexcitation, the photogenerated carriers rapidly relax to lower energy states in the conduction band *L* and *X* valleys. In particular, the initial rise in $\Delta T/T$(570 nm) is due to the state filling-induced change in the indirect absorption of the 570-nm probe photons into the *L* valley. The initial decay in $\Delta T/T$(570 nm) is then due to relaxation of these carriers from the *L* valley to the *X* valley and into mid-gap defect states. Carriers trapped in these defect states can then reabsorb probe photons, leading to the negative signal observed between ∼5–350 ps; this signal disappears as the photoexcited carriers leave these defect states and return to equilibrium. It is worth mentioning that negative $\Delta T/T$ signals in semiconductors and their nanostructures are often attributed to free carrier absorption (FCA)[16]. However, in our samples, FCA is unlikely to play a significant role in the measured dynamics, as negative signals at 570 nm are only observed for $t >$ 5 ps. At this point, the majority of carriers have been trapped into defect states, as supported by the positive signals observed with the 1350 nm probe [see Fig. 2(c) and (d)].

It is clear from Fig. 2(b) that the initial relaxation dynamics at 570 nm are strongly dependent on the NW diameter *d*; this is shown more explicitly by plotting the time constant τ for this process, obtained from exponential curve fits to the measured data, as shown in Fig. 2(e) as a function of *d*. For $d = 10$–50 nm, τ increases with *d*, indicating that surface-meditated trapping and recombination processes strongly influence the observed dynamics, as was observed in GeNWs with similar diameters [17]. However, for $d = 80$–100 nm, τ actually decreases with increasing *d*, which is inconsistent with previously observed trends in semiconductor NWs [16], [17], [23]. TEM images of $d = 80$–100 nm NWs revealed patches on the surface along the length of the wires, which may induce localized surface defects. Carrier trapping at these defect states could thus explain the decrease in τ for these NW diameters. Another possibility is the increased density of twinning defects observed in some of our NW samples with larger diameters. However, further studies are necessary to fully understand the decrease in τ for $d = 80$ and 100 nm.

Fig. 2(c) and (d) depicts $\Delta T/T$(1350 nm), which examines carrier relaxation into and out of mid-gap defect states; it is worth noting here that the observation of a signal using a mid-gap probe in itself confirms the presence of defect states within the forbidden bandgap of Si. We first focus on the $\Delta T/T$ signals for $d = 30$–100 nm; the signal for $d = 10$ nm will be discussed later in this section. The rise time of these signals is nearly identical to that at 570 nm (∼700 fs), indicating that we are probing

a transition from the defect states to the conduction band; i.e., when photoexcited carriers relax into the L and X valleys, they block absorption of the 1350 nm probe photons from carriers trapped in defect states, causing the positive $\Delta T/T$ signal. The subsequent relaxation corresponds to carriers returning to equilibrium, supported by the observation that both $\Delta T/T$(570 nm) and $\Delta T/T$(1350 nm) return to zero around 350–400 ps; it is worth noting that these carriers may relax into additional lower energy defect states along the way, which can result in additional induced absorption processes as seen for the $d = 30$ nm NWs in Fig. 2(c). Here, τ (1350 nm) consistently increases with diameter for $d = 30$–100 nm [see Fig. 2(e)]. The most probable explanation for this behavior is that the density of surface defects increases with decreasing NW diameter, as has been observed recently for wurtzite NWs [24]. Therefore, it is likely that carriers in the mid-gap defect states examined by the 1350 nm probe can return to equilibrium through additional surface states at lower energies, whose density increases as the NW diameter decreases. However, we cannot rule out other possibilities, e.g., changes in the nature of the surface-related defects (e.g., their energetic position) with the NW diameter could also lead to the observed dependence of τ (1350 nm) on d. We note that our experiments do not enable us to determine the origin of the defect states that govern carrier relaxation in SiNWs. Nevertheless, it is likely that the defects that govern the initial relaxation of the 570-nm probe signal are different than those probed at 1350 nm, as the 570-nm probe photons can be absorbed from defects that exist at energies both above and below those that are probed at 1350 nm. In addition, the nature of these defects is likely to be different in SiNWs than in bulk Si. This, along with the strong wavelength dependence of the dynamics, makes comparisons of our data on SiNWs to previous ultrafast optical experiments on bulk Si [25]–[27] difficult, as all of these experiments were performed at different pump and probe wavelengths than those used here.

The $\Delta T/T$(1350 nm) signal drastically changes when the NW diameter is reduced to 10 nm [see Fig. 2(c) and (d)]; here, we observe a negative photoinduced transmission change at $t = 0$, indicating induced absorption. Quantum confinement effects could account for the observed signal, since a recent report revealed a blueshift in the absorption edge of SiNWs as compared to the bulk [9]; if the mid-gap defect states probed for $d = 30$–100 nm shifted to higher energies for $d = 10$ nm, then induced absorption of the 1350-nm probe photons could dominate over state filling. In a similar manner, bandgap renormalization [16] could also influence the observed dynamics by causing the absorption of the probe beam to increase after photoexcitation. Finally, changes in the nature of the defect states or contamination from the catalyst Au nanoparticles could also cause the anomalous $\Delta T/T$(1350 nm) signal for $d = 10$ nm. In general, a more detailed study of NWs with diameters close to the exciton Bohr radius must be carried out to obtain a more complete understanding of the unique physics observed here.

In order to understand the influence of the laser polarization on carrier dynamics in SiNWs, we also performed time-resolved polarization-dependent experiments on the $d = 50$ nm SiNW sample with a 1045 nm probe. The inset of Fig. 3 on the lower right shows that most of the SiNWs are horizontally aligned when observed under an optical microscope, making this study possible. Fig. 3 shows the pump-induced change in the probe transmission for four different sets of measurements where both the pump and probe polarization were varied. It is clear that $\Delta T/T$ ($t = 0$) is maximum (minimum) when the polarizations of both pump and probe beams are parallel (perpendicular) to the NW axis. This is consistent with previous linear absorption measurements [28], [29], which demonstrated that both absorption and PL are maximum when the electric field of the incident light is polarized parallel to the NW axis, due to the large dielectric contrast between the NW and the air surrounding it. Therefore, the highest carrier density is generated when the pump is polarized parallel to the NW axis. Similarly, the probe is maximally absorbed when it is polarized along the NW axis. The resulting polarization dependence of $\Delta T/T$ ($t = 0$) can then be easily understood by noting that the photoinduced change will be the largest when the photoinduced carrier density ($\propto \Delta T$) and probe absorption ($\propto (1-T)$) are concurrently maximized. In addition, the inset of Fig. 3 on the upper left reveals that the dynamics do not significantly change with pump and probe polarization. Similar studies will be carried out on single NWs to further confirm and understand this behavior.

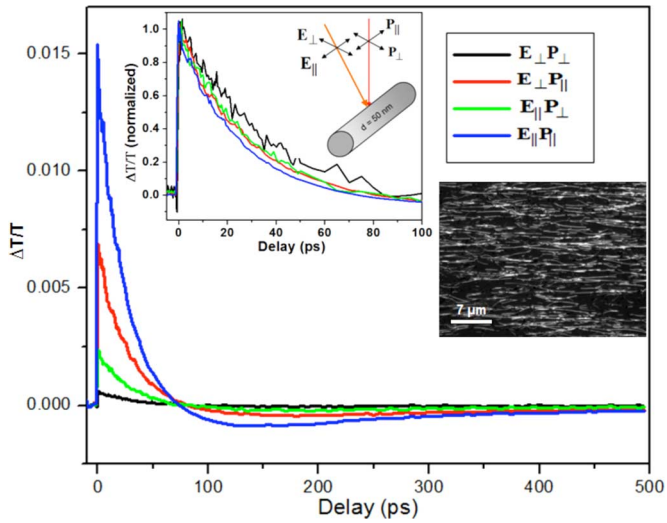

Fig. 3. Polarization-dependent transmission in $d = 50$ nm SiNWs at a pump fluence of $400\ \mu\mathrm{J/cm^2}$ and a probe wavelength of 1045 nm. Here, "E" indicates the pump polarization and "P" indicates the probe polarization. The upper left inset shows the corresponding normalized data, and the lower right inset shows an optical microscope image of the NWs after dry transfer onto the sapphire substrate.

Finally, we also examined the dependence of ultrafast dynamics in SiNWs on pump fluence. Fig. 4 depicts the photoinduced change in transmission for $d = 50$ nm SiNWs at probe wavelengths of 570 and 1350 nm for several pump fluences. It is clear that the carrier lifetime decreases significantly with increasing fluence for the 570 nm probe, but varies substantially less with fluence at 1350 nm.

Fig. 5 more quantitatively confirms this by depicting the variation of the initial relaxation time constant $\tau$with pump fluence for both probe wavelengths. Several possible mechanisms could

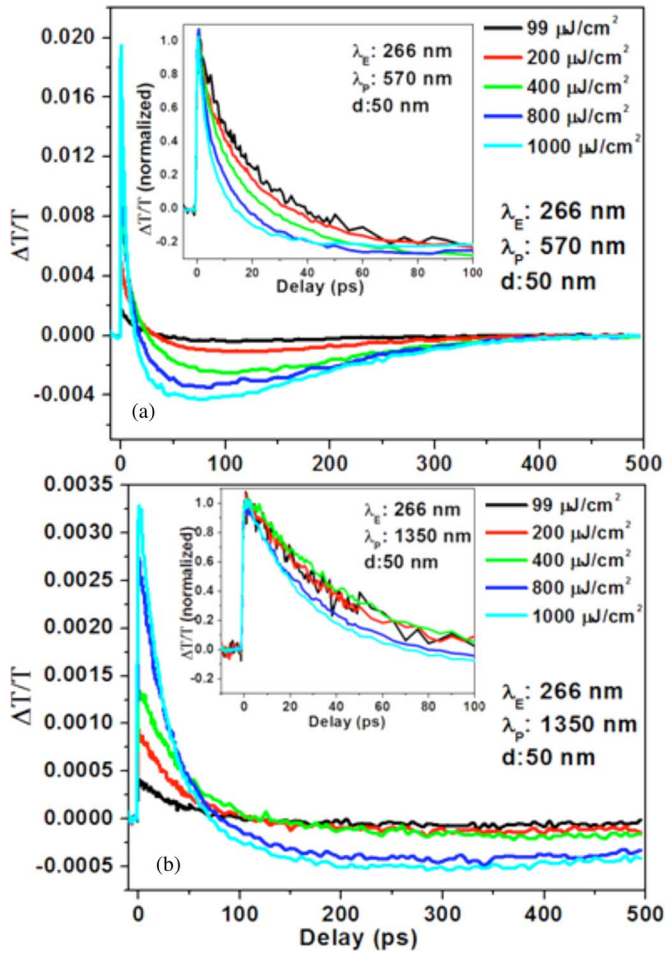

Fig. 4. Pump-fluence-dependent carrier relaxation dynamics in $d = 50$ nm SiNWs probed at (a) 570 nm and (b) 1350 nm. Insets show the data on a 100 ps timescale with peak amplitudes normalized.

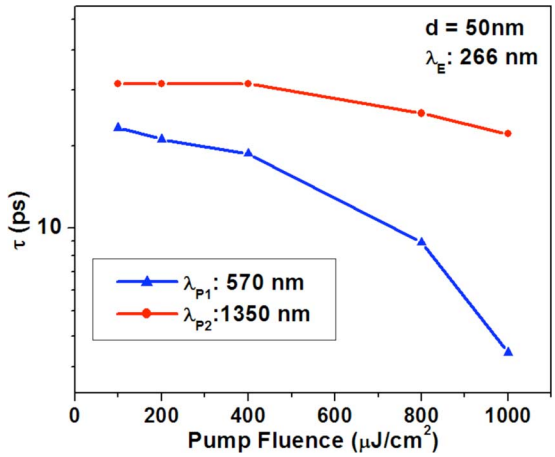

Fig. 5. Plot of τ versus pump fluence for $d = 50$ nm SiNWs probed at 570 and 1350 nm.

explain the dependence of τ(570 nm) on pump fluence. For example, Auger recombination (AR) is a dominant process in low-dimensional systems, due to the fact that spatial confinement increases carrier–carrier interactions as compared to bulk semiconductors [16] and has been shown to influence relaxation dynamics in SiNWs [30] as well as in other NW systems [31]. However, the wide size distribution in our NW samples made it difficult to unambiguously fit conventional expressions for either bimolecular (exciton–exciton) or three-carrier AR [31] to our data. Bandgap renormalization could also influence the observed fluence-dependent dynamics, since the photoexcited carrier density in our experiments is $>10^{19}/\mathrm{cm}^3$, which is larger than the Mott carrier density for silicon [32]. In this scenario, the bandgap would transiently shrink with increasing carrier density, which could cause the probe photons to examine higher energy states in the conduction band relative to the renormalized band minimum from which carriers would relax more rapidly. A final possibility is that, given the complex nature of defect states in the SiNWs, a laser-induced transformation of one defect state into another could also explain the observed density-dependent carrier relaxation at 570 nm [21].

Fig. 5 also reveals that τ varies significantly less with fluence at 1350 nm than at 570 nm, and in fact it is essentially constant for fluences $\leq 400\,\mu\mathrm{J/cm}^2$. The changes in τ (1350 nm) at higher fluences could also be due to bandgap renormalization, which could make additional transitions between lower energy defect states and the conduction band possible. Future controlled growth of SiNWs with fewer defects and concurrent investigation into the nature of defects in SiNWs will provide us with further insight into these observations.

IV. Conclusion

In conclusion, we have performed the first fs pump–probe experiments on SiNWs, to the best of our knowledge. These experiments were performed as a function of the NW diameter, pump and probe polarizations, and pump fluence to examine the influence of these parameters on the observed dynamics. We find that surface traps and recombination centers govern carrier relaxation in SiNWs, particularly those with $d \leq 50$ nm. The magnitude of the measured photoinduced transmission changes also strongly depends on the pump and probe polarizations. Finally, we also observe that the carrier lifetime significantly decreases with increasing pump fluence, likely due to many-body interactions between photoexcited carriers. These first ultrafast optical experiments on SiNWs demonstrate that carrier relaxation processes occur on a subnanosecond timescale and indicate that minimizing the influence of defect states on carrier dynamics (e.g., through surface passivation) will be essential in future nanophotonic applications of these quasi-1D systems.

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