Highly efficient hot electron harvesting from graphene before electron-hole thermalization

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Although the unique hot carrier characteristics in graphene suggest a new paradigm for hot carrier–based energy harvesting, the reported efficiencies with conventional photothermoelectric and photothermionic emission pathways are quite low because of inevitable hot carrier thermalization and cooling loss. Here, we proposed and demonstrated the possibility of efficiently extracting hot electrons from graphene after carrier intraband scattering but before electron-hole interband thermalization, a new regime that has never been reached before. Using various layered semiconductors as model electron-accepting components, we generally observe ultrafast injection of energetic hot electrons from graphene over a very broad photon energy range (visible to mid-infrared). The injection quantum yield reaches as high as ~50%, depending on excitation energy but remarkably, not on fluence, in notable contrast with conventional pathways with nonlinear behavior. Hot electron harvesting in this regime prevails over energy and carrier loss and closely resembles the concept of hot carrier solar cell.

INTRODUCTION

The bandgap ($E_g$) character of conventional semiconductor materials enables their applications in light-harvesting and optoelectronic devices, but at the same time, sets the fundamental limit of the spectral response and power conversion efficiency. For instance, the energy below $E_g$ is not captured and any excess energy exceeding $E_g$ is lost irreversibly to lattice as heat, leading to, e.g., Shockley-Queisser limit for photovoltaic cells (1). One way to overcome this limit and achieve far higher efficiency is extracting all energies from photoexcited carriers while they are still hot (2). This can be realized in semiconductors where scattering among photoexcited electrons is faster than electron cooling and recombination, coupled with equally fast carrier extraction by an energy selective contact.

Among potential candidate materials including semiconductor (3) and metallic (4) nanostructures and thin films, graphene with atomic layer thickness has been proposed as a promising system to explore hot carrier–assisted energy harvesting (5). Because of its unique linear band structure, graphene exhibits uniform (~2.3%) spectral response spanning infrared (IR) to visible regions (Fig. 1, i) (6, 7) and more importantly, very rapid electron-electron scattering assisted by strong Coulomb interactions (3, 8–10). Although very early stage carrier dynamics in graphene have not been explicitly resolved and analyzed, more experimental studies including ultrafast spectroscopic dynamics (8, 9, 11–13), broadband photoluminescence (PL) (14, 15), and stimulated emission in graphene (16, 17) have suggested that in the first ~10 fs, the initial photoexcited electron and hole assemblies quickly evolve into two separate hot Fermi-Dirac (FD) distributions (“quasi-thermalization”) with distinct chemical potentials ($\mu^{+}(0)$) and temperatures ($T^{+}(0)$) in conduction and valance bands, respectively, through intraband electron-electron scattering (Fig. 1, ii). Following that, there is consensus in literature that electron-hole coalescence into a single hot FD distribution (“full thermalization”) with one temperature ($T_e$) and chemical potential ($\mu$, $\mu = 0$ for charge neutral graphene) in 100 to 150 fs through interband electron scattering (including Auger and inverse Auger processes; Fig. 1, iii) and strongly coupled optical phonon emission (Fig. 1, iv) (5, 8, 10–13, 18). The thermalized hot carrier cools down slowly by further dissipating energy to phonons in picoseconds (directly or assisted by supercollision). Because of small electron heat capacity and vanishing density of states near the Dirac point, hot electrons in graphene can reach a high temperature (a few thousand Kelvin) and stay hot over a long time (up to tens of picoseconds).

Many efforts have exploited hot carrier–assisted energy harvesting in graphene, with most based on the photothermoelectric effect with in-plane junction devices (19–24). Unfortunately, these devices typically show limited photoactive areas, low quantum efficiency (~1%), and sublinear photoresponse on illumination power, limited by the operation principle and a small Seebeck coefficient of graphene. On the other hand, graphene-semiconductor
vertical heterostructures where the entire graphene is exposed as a photoactive region and vertical charge transfer (CT) is enabled has attracted increasing interest (25–31). An initial study on a graphene-WSe$_2$-graphene heterostructure with chemical vapor deposition graphene shows sub-bandgap photocurrent generation with superlinear dependence on excitation power (26). Hot thermalized electron in graphene has a long tail toward higher energy, and the portion above a potential barrier can transfer into a semiconductor through thermionic emission, denoted as conventional one-μ photothermal emission (1μPTE) pathway. However, the quantum efficiency was rather low (~2%) as a substantial amount of energy and carrier loss has occurred during thermalization (5, 10, 18), as well as subsequent electron injection, which is in competition with hot electron cooling (26). A very recent study on WS$_2$-graphene heterostructure exhibits photocarrier generation in WS$_2$ after sub-bandgap excitation (27). The authors referred to direct interlayer CT transition to explain observed photocarrier generation (27). However, the interlayer CT transition is usually too weak (compared to intralayer transition) to serve as an efficient pathway. Given that energies and carriers are mostly stored and conserved in the initial intraband scattering process (5, 9, 10, 18), an ideal energy-harvesting strategy would be efficiently extracting quasi-thermalized hot carriers before electron-hole coalescence sets in, denoted as two-μ photothermal emission (2μPTE) pathway.

We proposed and experimentally demonstrated the 2μPTE pathway for highly efficient hot electron harvesting from graphene to various transition metal dichalcogenides (TMDs, including MoS$_2$, MoSe$_2$, WS$_2$, and WSe$_2$) as model electron-accepting components. Using ultrafast spectroscopy, we observed highly efficient upconverted hot carrier–assisted photocarrier generation in TMD layers over a broad excitation wavelength (visible to mid-IR) (Fig. 2A). Our measurement shows ultrafast (~25 fs) electron injection from graphene to TMDs under sub-bandgap excitation with quantum yield (QY) as high as ~50%. QY shows strong dependence on photon energy but remains nearly constant with varying photon density, in good agreement with the 2μPTE but not conventional 1μPTE pathway. The temperature- and gating-dependent studies further support this new pathway.

RESULTS

We fabricated graphene/TMD (MoS$_2$, WS$_2$, MoSe$_2$, and WSe$_2$) heterostructure samples on SiO$_2$ by mechanical exfoliation and van der Waals stacking (see experimental details). The heterointerface was kept away from any polymer contamination during fabrication, and the samples were annealed under high vacuum. We kept the graphene layer on top for all samples to eliminate potential substrate effects on graphene. Figure 2B shows an optical image of a representative monolayer graphene/WS$_2$(Gr/WS$_2$) heterostructure. The atomic force microscopy height profile (Fig. 2C) shows a small height difference of approximately 0.4 nm between graphene and WS$_2$, suggesting a well-coupled interface (27, 32). We draw the energy level diagram (Fig. 2D) based on the 1.4-eV offset between the graphene Dirac point and the WS$_2$ valance band maximum from angle-resolved photoemission spectroscopy measurement (33) and a quasi-particle bandgap (~2.3 eV) of WS$_2$ (34, 35) without considering excitonic effect or interfacial effect. The estimated energy barrier ($\phi_B$) between WS$_2$ conduction band minimum (CBM) and the graphene Dirac point is thus ~0.9 eV and will be ~0.2 eV smaller if we consider interlayer exciton binding energy (35–37).

We characterized the sample using reflectance contrast (ΔR) (Fig. 2E) and PL (fig. S9) spectroscopy. Reflectance contrast is calculated as $\delta R = (I_R^2 - E_r^2) / I_R^2$, where $I_R$ and $E_r$ are the reflected light intensity from sample and substrate (SiO$_2$), respectively. For atomically thin samples on a transparent SiO$_2$ substrate, $\delta R$ directly reflects the absorption property (6). Monolayer WS$_2$ exhibits a sharp A exciton peak at 2 eV, which is significantly broadened and red-shifted in the Gr/WS$_2$ heterostructure. This, together with orders of magnitude quenched WS$_2$ PL, suggests strong electronic coupling and ultrafast interfacial CT in the heterostructure (32, 38). It is worth noting that the small trion feature on WS$_2$ PL disappeared in heterostructures, suggesting a slight static electron flow from WS$_2$ to graphene (38). We determined graphene in the heterostructure to be weakly p-doped with a doped carrier concentration of ~1 x 10$^{12}$ cm$^{-2}$ and a chemical potential of ~0.13 eV below the Dirac point from Raman spectroscopy (section S1). We compared a summed $\delta R$ spectra (gray dashed line) of isolated graphene and WS$_2$ monolayers to that of the Gr/WS$_2$ heterostructure (Fig. 2E). As was shown previously, they overlap well below WS$_2$ absorption (lower than 1.8 eV) (38), indicating most graphene direct interband transition with little (<5%) CT transition contribution in the Gr/WS$_2$ heterostructure.

We investigated the photocarrier generation in graphene/TMD heterostructures using ultrafast transient reflectance (TR) spectroscopy (see experimental details in section S1). We pumped the heterostructures with photon energies below the TMD bandgap to excite graphene only and probed the electron population in TMD through a white light continuum. For the Gr/WS$_2$ heterostructure, we chose excitation energies of 1.6 eV (775 nm), 1.37 eV (900 nm), 1.2 eV (1.03 μm), 0.95 eV (1.3 μm), 0.83 eV (1.5 μm), 0.73 eV (1.7 μm), 0.62 eV (2.0 μm), 0.52 eV (2.4 μm), and 0.36 eV (3.5 μm). Under sub-bandgap excitation, hot electron distribution has a long tail toward higher energy from electron-electron scattering and only upconverted energetic electrons above the barrier can transfer to WS$_2$. The color plot of TR spectra of Gr/WS$_2$ at 1.6-eV excitation (pulse duration of ~30 fs) is shown in Fig. 3A. Upon photoexcitation, TR spectra show a dominant bleach peak at ~1.98 eV, corresponding to A exciton peak of WS$_2$. We observed no TR signal on an isolated WS$_2$ monolayer under the same condition. The WS$_2$ A exciton bleach under sub-bandgap excitation can only be attributed to carrier-induced broadening and band-filling effect, indicating carrier injection from photoexcited graphene to WS$_2$ (39). Selective carrier extraction from the Gr/WS$_2$ heterostructure (section S2) confirms electron (not hole) injection to WS$_2$, which is consistent with the energy level alignment and previous photocurrent measurements (26). In a small signal regime (< 0.01), the TR signal is linearly proportional to carrier population; thus, TR kinetics directly yields the electron transfer kinetics (section S4).

The TR kinetics of WS$_2$ A exciton bleach is shown in Fig. 3B. Fitting the kinetics yields a rising time of 27 ± 4 fs and a biexponential decay with a half-life time of ~1.2 ps, which corresponds to hot electron transfer from graphene to WS$_2$ and subsequent back electron transfer to graphene, respectively. Previous studies on graphene-TMD van der Waals heterostructures also reported an ultrafast interfacial process, mostly limited by experimental time resolution (27, 36, 40). This interfacial electron transfer time is orders of magnitude faster than the value (~40 ps) inferred from photocurrent measurement (26). This time scale is comparable with graphene intraband electron scattering time, which is a prerequisite for efficient hot carrier harvesting.

Key insights into the hot electron transfer process comes from examining how the TR signal peak amplitude (TR$_{\text{max}}$) changes with photon energy (hν) and absorbed photon density (N$_{\text{photon}}$). We
varied \( h\nu \) over a wide range while always maintaining the TR signal in a small signal regime, and we observed photoelectron generation in WS\(_2\) for all excitation conditions (fig. S10). We first analyzed the dependence of the TR max and injected electron population on \( N_{\text{photon}} \), as shown in Fig. 3C. \( N_{\text{photon}} \) was calculated with precise pump/probe beam profiles and absorption properties (section S5). We assumed constant absorption (2.3\%) from graphene in the investigated range (6, 7). Unexpectedly, TR max increases linearly with \( N_{\text{photon}} \) for all \( h\nu \) applied, except the onset of sublinear deviation with low-energy photons at high \( N_{\text{photon}} \). This linear power dependence precludes multiphoton processes and indicates constant hot electron injection efficiency from photoexcited graphene, in notable contrast with conventional photothermionic (26, 28) and photothermoelectric graphene devices (19, 22–24), which show superlinear and sublinear power dependence, respectively. Similar linear behavior has been observed and ascribed to direct CT transition (27). CT transition cannot occur below \(-0.9\) eV as the band alignment sets a cutoff. CT could contribute to photocarrier generation for photon energy above 0.9 eV, but we expect this contribution to be small (<5\%) on the basis of the absorption spectra. As we will show later, this linear power dependence can be well described by the proposed 2\( \mu \)PTE model but not the conventional photothermionic model.

Increasing \( h\nu \) leads to a larger TR signal, which can be clearly seen from the slope of linear power dependence. We determined the QY of hot electron injection from graphene, which is the figure of merit for most applications (see section S4 for details), and the results are shown in Fig. 3D for the Gr/WS\(_2\) heterostructure. We note that the error bars are calculated from completely different sets of measurements on the same sample. The way we determined QY sets the upper bound and has a relative uncertainty of 10\% (section S3). QY exhibits a strong dependence on \( h\nu \), increasing monotonically from ~1\% for the lowest \( h\nu \) (0.36 eV) to as high as ~55\% for 1.6-eV excitation (~0.4 eV below the WS\(_2\) bandgap), highlighting the importance of \( h\nu \) on hot electron extraction yield. The hot electron extraction yield here is significantly higher than the internal QY in previously reported graphene hot carrier–based devices (22, 26, 28).

We performed the same measurements on another Gr/WS\(_2\) sample and obtained similar results (fig. S11).

Under sub-bandgap photon excitation, only electrons with enough energy gained from an electron scattering process (“energetic electrons”) can overcome the interfacial barrier \( \varphi_B \) and inject into a semiconductor conduction band (“photothermionic emission”). In the conventional photothermionic emission (1\( \mu \)PTE) pathway (26), photoexcitation heats up carriers on graphene and creates a thermalized hot carrier distribution for the whole electronic system

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f_\epsilon(\xi) = \frac{1}{\exp\left(\frac{\epsilon - \mu}{k_B T_e}\right) + 1}
\]
with one well-defined $T_e$ and unaltered $\mu$. The hot electron distribution extends to high energy, and the fraction of electrons above $q_B$ contribute to photocurrent via thermionic emission (26, 28). We calculated hot electron distribution and injection QY at different $h\nu$ (0.35 to 1.6 eV) and $N_{\text{photon}}$ ($10^{11}$ to $10^{13}$ cm$^{-2}$) values using the 1µPTE model for charge-neutral graphene (see section S5 for details). As substantial energy ($\sim$30%) (26, 41) loss has occurred during the initial thermalization process, for simplicity, we assume that 70% of photon energy go to the electronic system in the 1µPTE model. This percentage does not affect the predicted trend.

The calculated $T_e$ as a function of $h\nu$ and $N_{\text{photon}}$ is shown in fig. S6. The representative FD distributions ($f(\epsilon)$) at three different $N_{\text{photon}}$ values are shown in Fig. 4A, and the corresponding electron distributions [calculated as $f(\epsilon)g(\epsilon)$, where $g(\epsilon)$ is graphene density of states] in Fig. 4B. The calculated QY with $q_B$ of 0.7 eV is shown in Fig. 4C, assuming that all hot electrons above $q_B$ emit into the semiconductor conduction band with unity yield. As expected, hot electron temperature and injection QY show a strong dependence on both $h\nu$ and $N_{\text{photon}}$. A hotter electron distribution, thus higher QY, can be achieved by increasing either $h\nu$ or $N_{\text{photon}}$ i.e., they are interchangeable. QY reaches $\sim$30% for $h\nu$ of 1.6 eV and $N_{\text{photon}}$ of $10^{13}$ cm$^{-2}$. The injected electron density at a fixed $h\nu$ shows a superlinear dependence on $N_{\text{photon}}$ or excitation power (fig. S7), consistent with previous photocurrent measurements (26, 28). Simply speaking, as $T_e$ in graphene depends on excitation power ($P$) as $T_e \sim P^{1/3}$ and thermionic current increases exponentially with $T_e$, photothermionic current exhibits an overall superlinear dependence (26, 28). Apparently, the high QY value ($\sim$50% at 1.6 eV) and especially the linear power dependence (or constant QY versus $N_{\text{photon}}$) observed experimentally cannot be explained by the conventional 1µPTE model.

Instead, the ultrafast electron transfer from photoexcited graphene to the TMD semiconductor suggests that hot electron injection likely occurs before electron-hole coalescence through the 2µPTE pathway. In this pathway, on the order of $\sim$10 fs, photoexcited electrons and holes quickly establish separate quasi-thermal equilibrium

$$f_{e(\nu)}(\epsilon) = \frac{1}{\exp\left(\frac{\epsilon - \mu_{e(\nu)}^*}{kBT_{e(\nu)}}\right) + 1}$$

with distinct temperatures ($T_{e(\nu)}$) and separated chemical potentials ($\mu_{e(\nu)}^*$) for electrons and holes, respectively. Given the ultrafast time scale and phase space constraint of a graphene Dirac spectrum, both photon energy (half into each band) and photoexcited electrons and holes ($= N_{\text{photon}}$) can be approximated to be stored and conserved in each band (16, 18).

We calculated hot electron distribution and injection QY using the proposed 2µPTE model. The calculated $T_e$ and $\mu_e^*$ as a function of $h\nu$ and $N_{\text{photon}}$ is shown in fig. S6. The representative FD distributions and corresponding electron distributions above the Dirac point are shown in Fig. 4 (D and E). Obviously, the 2µPTE model yields a significantly higher electron temperature and more energetic electrons above $q_B$ than the 1µPTE model, especially at low $N_{\text{photon}}$. For low and moderate $N_{\text{photon}}$ (e.g., less than $10^{13}$ cm$^{-2}$), $T_e^*$ increases linearly with $h\nu$ and $\mu_e^*$ is mostly negative (below the Dirac point).
corresponding to a hot and dilute Maxwellian electron gas (Fig. 4D). Therefore, the electron distribution increases almost uniformly (Fig. 4E) with \( N_{\text{photon}} \), and the fraction of energetic electrons above \( \phi_B \) (i.e., injection QY) remain nearly constant at fixed \( \hbar \nu \) and \( \nu_B \) is fixed at 1.6 eV. Electron distribution above the Dirac point in graphene with (B) 1µ and (E) 2µ models at different absorbed photon densities. Hot electrons above the barrier height \( \phi_B \) can inject into the semiconductor conduction band. A much higher fraction of energetic electrons are observed in 2µ model. Color plot of predicted electron injection QY as a function of photon energy and density with (C) 1µPTE and (F) 2µPTE models. \( \phi_B \) was chosen to be 0.7 eV, and all electrons above \( \phi_B \) were assumed to inject into the semiconductor conduction band. QY shows a strong dependence on both photon energy and density in the 1µPTE pathway but remains nearly constant with varying photon density in the 2µPTE pathway.

In Fig. 3D, we show calculated QY versus \( \nu_B \) with \( \phi_B \) of 0.6 to 0.8 eV and experimental QY values mostly fall into the calculated range. The \( \phi_B \) 0.2 to 0.3 eV smaller than values from quasi-particle band alignment can be attributed to interlayer exciton binding energy, which is 0.2 to 0.3 eV in two-dimensional layered heterostructures (36, 37, 43). Considering no free parameters, this simple 2µPTE model reproduced the experimental results reasonably well and captured the key essence: the highly efficient harnessing of energetic electron from intraband scattering, prevailing over energy and carrier loss due to interband scattering and phonon emission.

To further support the proposed 2µPTE pathway, we measured the hot electron injection yield as a function of sample temperature.
thorough study on a Gr/WSe\textsubscript{2} heterostructure with $\hbar\omega$ of this broadband energy-harvesting strategy (figs. S8 and S12). A

We extended this hot electron-assisted photocarrier generation under sub-bandgap excitation to other TMD semiconductors

DISCUSSION

Compared to conventional pathways, $2\mu$PTE proposed here yields significantly higher hot electron extraction QY especially at a technically relevant low-fluence regime, e.g., solar radiation. The highly efficient hot carrier generation and extraction from graphene over a broad spectral range through the $2\mu$PTE pathway closely resembles the concept of “hot carrier solar cell” introduced in the beginning. This, together with a new route for extremely efficient and flexible energy-harvesting and optoelectronic devices beyond the standard limit. Furthermore, the efficient generation and harnessing of energetic electrons above ($\sim$1 eV) the Dirac point with low-energy photons also suggest graphene as potential broadband upconverting light-harvesting component to drive chemical reactions (e.g., photocatalysis). The absorption can be increased using optical waveguides and/or plasmon enhancement. The fast back electron transfer to graphene (i.e., charge recombination loss) can be retarded by constructing judiciously designed multicomponent vertical heterostructures (e.g., Gr/WS\textsubscript{2}/MoS\textsubscript{2}; see section S2).

MATERIALS AND METHODS

Sample preparation

Isolated graphene and TMD layers were mechanically exfoliated onto transparent SiO\textsubscript{2} or SiO\textsubscript{2} (100 nm)/Si substrates from bulk crystals (HQ graphene). The number of layers was confirmed by atomic force microscopy, PL, and Raman spectroscopy. After the TMD and graphene monolayers were exfoliated onto the substrates, we picked up the graphene monolayer with polycarbonate (PC) and transferred on top of a TMD flake under an optical microscope to form a Gr/TMD heterostructure. The PC was melted at 180°C and dissolved with chloroform. The samples were annealed for 1 hour at 300°C in high vacuum.

Optical characterization

Steady-state micro-area PL, Raman, transmittance, and reflectance contrast measurements were taken on a home-built microscope setup. A supercontinuum laser (NKT, super compact) was used as broadband light source for transmittance and reflectance contrast measurement and a 532-nm continuous-wave laser (full width at half maximum, <0.001 nm) for PL and Raman measurements. Spectra were recorded with a liquid N\textsubscript{2} cooled detector (Princeton Instruments). For femtosecond TR spectroscopy (mFemto-TR100, Time-Tech Spectra LLC) (39), the fundamental output (1030 nm, $\sim$170-fs pulse duration) from a Yb:KGW laser (PHAROS, Light Conversion Ltd.) was separated into multiple light beams. One was introduced to a noncollinear optical parametric amplifier (OPA) to generate visible and near-IR wavelengths for pump beam (775, 900, 1300, 1500, 1700, and 2000 nm, with a pulse duration of 30 to 100 fs depending on the wavelength), and one was introduced to a collinear OPA to generate 2.4 and 3.5 $\mu$m with a pulse duration of $\sim$70 fs. The third was focused onto an yttrium–aluminum–garnet crystal to generate continuum white light as a probe beam. Both pump and probe beams were sent to a microscope colinearly and focused using a reflective microscope on the sample. The exact beam profile for both the pump and the probe can be seen in fig. S5. Samples were in a liquid nitrogen–cooled cryostat for low-temperature TR measurements. Back gate voltage was applied with prepatterned electrodes and KEITHLEY 2614B.

SUPPLEMENTARY MATERIALS

Supplementary material for this article is available at http://advances.sciencemag.org/cgi/content/full/5/11/eaax9958/DC1

Section S1. Determining doping level and chemical potential in graphene

Section S2. Selective carrier extraction from Gr/WS\textsubscript{2} heterostructures and charge recombination retardation

Section S3. QY calibration using WSe\textsubscript{2}/WS\textsubscript{2} heterostructure and direct high-energy excitation

Section S4. Absorbed photon density calculation

Section S5. Calculation with $1\mu$PTE model and $2\mu$PTE model

Section S6. Hot electron transfer from photoexcited graphene to WSe\textsubscript{2}

Section S7. Additional figures

Fig. S1. Determining doping level and chemical potential in graphene.

Fig. S2. Selective carrier extraction and charge recombination retardation.

Fig. S3. CT in WSe\textsubscript{2}/WS\textsubscript{2} heterostructure.

Fig. S4. TR measurements of Gr/WS\textsubscript{2} under above-gap excitation.

Fig. S5. Beam profile and absorption properties of heterostructures.

Fig. S6. Calculated carrier temperature and chemical potential with different models.

Fig. S7. Calculated injection electron density.

Fig. S8. Hot electron transfer from photoexcited graphene to WSe\textsubscript{2}.

Fig. S9. PL spectra of WS\textsubscript{2} monolayer and Gr/WS\textsubscript{2} heterostructure.

Fig. S10. All TR kinetics of Gr/WS\textsubscript{2} heterostructure at WS\textsubscript{2}, A exciton bleach, at different excitation photon energies and densities.

Fig. S11. TR results from another Gr/WS\textsubscript{2} sample.

Fig. S12. TR spectra of Gr/MoS\textsubscript{2} and Gr/MoSe\textsubscript{2} heterostructures.

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