Studying Stimulated Raman Activity in Surface-Enhanced Femtosecond Stimulated Raman Spectroscopy by Varying the Excitation Wavelength

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Supporting Information

ABSTRACT: We present the first multiwavelength surface-enhanced femtosecond stimulated Raman spectroscopy (SE-FSRS) study, as well as the first observation of anti-Stokes vibrational features in SE-FSRS spectra. We compare stimulated Raman loss (SRL) and stimulated Raman gain (SRG) signals at three pump wavelengths chosen to sample different portions of nanoparticle aggregate localized surface plasmon resonances. The SE-FSRS signals exhibit similar signal magnitudes in the SRL or SRG regions of the spectra regardless of Raman pump or probe wavelength. The spectral lineshapes, however, differ dramatically with excitation wavelengths. The observed trends in spectral line shape show a strong dependence on the relative position of the excitation fields with respect to the plasmon resonance but do not match predictions from any existing SE-FSRS theory. These results suggest the need for further theoretical efforts with complementary experimental studies of individual aggregates to remove the effects of inherent ensemble averaging.

Studying the fundamental structural dynamics of bond breakage and formation requires techniques that can characterize deformities of molecular equilibrium structures on time scales corresponding to collective nuclear motion. To investigate the dynamics of bond-breakage and formation, vibrational spectroscopy is ideal because it can directly detect changes in molecular structure based on changes in the vibrational spectrum. Coherent Raman scattering (CRS) techniques have been utilized in stimulated Raman gain (SRG), stimulated Raman loss (SRL), with molecular resonance (FSRRS), and surface-enhancement (SE-FSRS). When performed off resonance with the analyte molecule, FSRS has the benefit of lineshapes that are directly comparable to spontaneous Raman scattering, a considerable advantage compared with coherent anti-Stokes Raman scattering (CARS) where interference between nonresonant and resonant vibrational terms in the optical susceptibilities lead to dispersive lineshapes and broad backgrounds. When performed on resonance, FSRS exhibits dispersive lineshapes that vary as the pump wavelength is scanned through the molecular electronic absorption band. The dispersive signals are attributed to the interference of hot luminescence (HL) and resonance Raman scattering (RRS) pathways upon generating an excited-state population. However, even when SE-FSRS is performed far from molecular resonance, dispersive lineshapes are observed. SE-FSRS lineshapes are strongly dependent on the location of the excitation pulses with respect to the plasmon resonance, as observed in a previous two-point plasmon resonance comparison. In that study, the dispersive lineshapes were attributed to Fano-like coupling between narrowband vibrational coherences and the broadband plasmon resonance. The behavior seen in Frontiera et al. was independently reproduced theoretically by two research groups using either a purely quantum-mechanical time domain approach, which supports the hypothesis of purely Fano-like coupling as described above, and semiclassical mixed frequency-time domain approach, which proposes that the lineshapes result from the combination of two Fano-like contributions arising from the interference between both the real and imaginary components of the Raman susceptibility and the plasmon resonance. Our current study examines three points of interest: (1) observe for the first time SRL in SE-FSRS experiments, (2)
understand differences in SE-FSRS probe enhancement by comparing signal magnitudes of SRG to SRL, and (3) investigate the wavelength dependence of SE-FSRS lineshapes by Raman pump-tuned SRG and SRL experiments. By exploring these three points, we demonstrate the versatility of plasmonic enhancement in SE-FSRS as applied to a variety of experimental conditions. Moreover, this study contributes to further understanding of the plasmonic enhancement in SE-FSRS, as we can compare experimental results to recent SE-FSRS theoretical predictions.

Two different molecule-plasmonic nanoparticle assemblies are used in this study: 90 nm Au cores aggregated with trans-1,2-bis(4-pyridyl)ethylen (BPE) and 60 nm Au cores aggregated with 4,4’-bipyridine (BPY). The aggregated cores are overcoated with silica (Figure S1). The 90 nm BPE nanoparticle assemblies (BPE90) have been used in several previous SE-FSRS studies and have been characterized to consist of both monomers and aggregates including dimers, trimers, and larger clusters. The 60 nm BPY nanoparticle assemblies (BPY60) are analogous to the 60 nm BPY assemblies used in Frontiera et al., except for the difference in reporter molecule; they consist of a similar range of aggregate sizes but with a relatively higher proportion of monomers, although the exact composition was not quantified. Extinction spectra of BPE90 exhibit an aggregate localized surface plasmon resonance (LSPR) near 1000 nm, while the aggregate LSPR of BPY60 occurs near 850 nm. Both samples also exhibit a monomer LSPR at shorter wavelengths, near 575 nm for BPE90 and 555 nm for BPY60. BPE90 and BPY60 are investigated by SE-FSRS with three distinct Raman pump wavelengths (Pu) 795 nm for BPE90 and 555 nm for BPY60. BPE90 and BPY60 are investigated by SE-FSRS with three distinct Raman pump wavelengths (Pu), 750, 795, and 840 nm, and the corresponding SRG and SRL signals are obtained using Raman probe fields (Pr) with spectral width sufficient to stimulate vibrational modes from 700 to 1900 and −700 to 1900 relative cm⁻¹, respectively. Multiple pump wavelengths were chosen to sample different portions of the aggregate LSPR with SE-FSRS, such that different plasmonic responses to either the pump or probe fields could be interrogated. These conditions include positioning both excitation fields on the rising edge of the aggregate LSPR, both on the falling edge, one on either side, and even both fields far off resonance of the aggregate LSPR. The specific conditions probed are summarized in the Table 1 and are illustrated schematically above the corresponding spectra.

SE-FSRS spectra are collected using a 1 MHz repetition rate system that has been previously described. In brief, the system consists of a noncollinear optical parametric amplifier (Clark-MXR iNOPA) pumped by a Yb-doped fiber oscillator/amplifier (Clark-MXR Impulse). The NOPA output is sent through a spectral grating filter to narrow the spectral bandwidth and generate the picosecond Raman pump. Residual white light not used for NOPA seeding is filtered using a pair of edge-pass filters to generate the broadband Raman probe. For this study, the wavelength of the pump beam was adjusted by tuning the NOPA and spectral grating filter accordingly, while the probe wavelength range was adjusted by exchanging edge-pass filters. All SE-FSRS experiments were performed with 320 μW of average pump power (320 pJ/pulse) and enough probe to reach ~80% dynamic range filling on the charge-coupled device (CCD) camera. The probe power was not rigidly controlled because Raman gain is invariant with probe intensity and maintaining equal dynamic range filling of the camera prevents the signal-to-noise variations between data sets. The pump beam is mechanically chopped to collect alternating pump-on and pump-off spectra. As discussed by McAnally et al., the first published SE-FSRS spectra were incorrectly phased due to an issue with the chopper; this issue has been corrected and the SE-FSRS spectra presented here are plotted properly as pump-on divided by pump-off. Further experimental details are given in the Supporting Information.

We start by comparing the Stokes and anti-Stokes signals of non-plasmonically enhanced FSRS. Other studies have demonstrated that the anti-Stokes side of the FSRS spectrum is dominated by negative SRL features that are equal in both magnitude and line width to the positive SRG features that appear on the Stokes side of the FSRS spectrum. This serves as a stark contrast with spontaneous Raman experiments, in which the ratio of anti-Stokes to Stokes signals is governed by the Boltzmann distribution for population of excited vibrational states. A theoretical description of FSRS attributes the SRL features to a different set of four-wave mixing processes than those that generate SRG features, a result that explains their equal magnitudes but opposite signs. Our 1 MHz laser system produces lower energy pulses than the 1 kHz systems typically used for FSRS; while this is beneficial for SE-FSRS studies as it mitigates sample degradation, it limits the molecules that we can study without plasmonic enhancement. Thus we chose to use neat cyclohexane in our comparison of SRG and SRL for non-plasmonically enhanced FSRS as it is a much stronger Raman scatterer than saturated aqueous solutions of either BPE or BPY, the analytes used in our SE-FSRS studies. Figure 1 shows the SRG (red) and SRL (blue) responses of neat cyclohexane pumped at 795 nm. The spectra were collected using 1.1 mW (1.1 μJ/pulse) of Raman pump and are plotted as a function of the absolute value of the Raman shift to display them simultaneously and facilitate comparison between spectral features. The SRG and SRL responses agree with results from the literature with regards to signal magnitude and sign. This comparison was used at every Raman pump wavelength to ensure that SE-FSRS spectra could be compared accurately such that changes in SE-FSRS magnitudes and signs can be assigned to plasmonic influences.

Next, we examine the Stokes and anti-Stokes SE-FSRS signals of BPE90 (Figure 2) pumped at three different wavelengths. When the Raman pump is tuned to 750 nm, it is far off resonance with the aggregate LSPR of BPE90 (Figure 2A, top). As with unenhanced FSRS, the signal magnitudes (Figure 2A, bottom) are nearly identical between SRG (red) and SRL (blue). Precise determination of the signal magnitudes is hampered by the varying dispersivities of the lineshapes.

<table>
<thead>
<tr>
<th>sample</th>
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<th>λ_Pu = 750 nm</th>
<th>λ_Pu = 795 nm</th>
<th>λ_Pu = 840 nm</th>
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<td>λ_Pu &lt; λ_LSPR</td>
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<td>SRG</td>
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<td></td>
<td>SRL</td>
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<td>λ_Pu &lt; λ_LSPR</td>
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Table 1. Summary of Experimental Conditions for SE-FSRS Experiments

The Journal of Physical Chemistry Letters
Letter
DOI: 10.1021/acs.jpclett.7b01342

3329
While a previous SE-FSRS study of BPE90 fit the spectral features to Fano lineshapes, the fits are entirely empirical; the line width and line asymmetry parameters cannot be decoupled and the interplay between them additionally affects the fit intensities, leading to multiple solutions when fitting complex spectra with overlapping lineshapes. The lack of a unique fit precludes a more rigorous quantitative comparison of intensity and dispersiveness between spectra in which the lineshapes differ considerably, such as those shown here. The SRG shows highly dispersive lineshapes across all modes, while the SRL has less dispersive lineshapes. It is important to note that, unlike the FSR spectra of cyclohexane in which “loss” features are strictly negative and “gain” features are strictly positive, the dispersivity of the SE-FSRS lineshapes can obscure the interpretation of spectral features as originating from SRL versus SRG without prior knowledge of the excitation conditions. For example, within the Fano line shape equation, the asymmetry parameter, \( q \), describes the coupling between the narrow vibrational mode and the broad plasmon resonance; the phase of the measured signals depend on both the sign of the molecular signal and the sign of the coupling parameter. If a positive amplitude “gain” feature and a negative amplitude “loss” feature experience couplings of equal magnitude but opposite sign, then the phases of the resulting spectral features will be the same.

Figure 2B shows the SRG and SRL responses (bottom) of BPE90 to a 795 nm pump, which is closer in wavelength to the aggregate maximum LSPR but still off-resonant (top). Again, the SRG and SRL signals are nearly identical in intensity but display differing dispersivities. The SRL and SRG signals are both highly dispersive, but the SRG lineshapes exhibit a strong frequency-dependent dispersive character between modes. The pair of modes around 1600 cm\(^{-1}\) are significantly less dispersive than the modes between 1000 and 1400 cm\(^{-1}\). Finally, Figure 2C shows the SRG and SRL responses of BPE90 to an 840 nm pump. While still off-resonant with the aggregate LSPR of BPE90, the pump is closer to the resonance than in either of the two prior pump wavelengths. As in the previous data sets, the signal magnitudes remain approximately equal between SRG and SRL, while the dispersivities of the lineshapes differ.

Figure 1. FSRS spectra of cyclohexane. The SRG (red) and SRL (blue) spectra, plotted as a function of absolute Raman shift, show signals with equal magnitudes but opposite signs.

Figure 2. SE-FSRS spectra of BPE90 pumped at three wavelengths. Extinction spectra with schematic pump and probe pulses (top) and SE-FSRS spectra (bottom) for BPE90 pumped at 750 (A), 800 (B), and 840 nm (C). The spectra are plotted as a function of absolute Raman shift, with SRL shown in blue and SRG shown in red, and are vertically offset for clarity.
To confirm that the trends in signal magnitude and line shape are not unique to BPE90, we obtained SE-FSRS spectra of BPY60 for the same set of excitation conditions (Figure 3). For each pump wavelength, the SRL and SRG signal magnitudes are nearly identical. The lack of any noticeable difference in intensity is particularly interesting in the case of pumping at 750 nm (Figure 3A), as the probe is resonant with the aggregate LSPR in the SRG experiment but far off resonance in the SRL experiment. Additionally, the SRG exhibits almost no dispersive character while the SRL lineshapes are dispersive. In contrast, when pumped at 800 nm (Figure 3B) the spectral features are more dispersive for SRG than for SRL. Both the SRG and SRL responses appear to become less dispersive when the pump wavelength is further increased to 840 nm (Figure 3C).

Comparing the SE-FSRS spectra collected at all three pump wavelengths, we observe that for BPE90, which has an aggregate LSPR near 1000 nm, both SRG and SRL lineshapes become less dispersive as the wavelength of the Raman pump increases from 750 to 840 nm (Figure 2). This is a surprising result as McAnally et al. predicts more dispersive lineshapes when the excitation field is closer to the plasmon resonance.\(^\text{13}\) Likewise, we see that for SE-FSRS of BPE60, which has an aggregate LSPR near 850 nm, the lineshapes are less dispersive when pumped at 840 nm (Figure 3C) compared with pumping at 795 nm (Figure 3B). As the Stokes probe in the 840 nm pump experiment is 910–1025 nm, this challenges the prediction by Mandal et al. that lineshapes should become more dispersive as excitation fields become redder than the plasmon resonance.\(^\text{12}\) Instead, we observe a new trend in the lineshapes that has not been suggested by theory. SE-FSRS lineshapes generally appear less dispersive when the probe field interacts with a slowly varying region of the LSPR. For this trend to hold, we must also take into account the presence of a monomer LSPR at shorter wavelengths. Thus the least dispersive lineshapes are observed when the probe field falls within either the maximum of the aggregate LSPR or the dip between the monomer LSPR, while more dispersive lineshapes are observed when the probe field falls on the rising or falling edge of either the monomer or aggregate LSPR. The dependence of the SE-FSRS line shape on the LSPR slope can even be observed within a single spectrum, such as in the SRG response of BPE90 when pumped at 795 nm (Figure 2B). As mentioned previously, the pair of modes at 1600 cm\(^{-1}\) is noticeably less dispersive than the lower energy modes. When we look at how the probe spectrum overlaps the LSPR, we can see that the redder probe wavelengths are nearly on-resonance with the aggregate LSPR; these are the wavelengths that correspond to the less dispersive 1600 cm\(^{-1}\) modes.

In summary, we have observed SRL in SE-FSRS experiments for the first time, a step that will allow us to explore the effects of molecular and plasmonic resonances on SE-FSRS signals and determine experimental limitations. By probing molecular resonances at different positions along the aggregate LSPR, these studies can help us understand how the mechanism of plasmonic enhancement in CRS differs from that in spontaneous Raman scattering. While surface-enhanced Raman scattering (SERS) experiments see a simple local field enhancement (LFE) of the incoming excitation and incoherently scattered fields, coherent Raman signals exhibit an additional strong dependence on the coherently driven third-order molecular polarization that interacts with the plasmon resonance.\(^\text{12,13}\)

Figure 3. SE-FSRS spectra of BPY60 pumped at three wavelengths. Extinction spectra with schematic pump and probe pulses (top) and SE-FSRS spectra (bottom) for BPY60 pumped at 750 (A), 800 (B), and 840 nm (C). The spectra are plotted as a function of absolute Raman shift, with SRL shown in blue and SRG shown in red, and are vertically offset for clarity.
By examining the SRL and SRG responses for the BPE90 and BPY60 substrates at different pump wavelengths, we can conclude that the frequency dependence of SE-FSRS signal magnitudes on excitation wavelength is complex and slowly varying. We see nearly identical signal intensities between SRL and SRG for a given pump wavelength and only small differences (<10%) in signal intensity between spectra obtained at different pump wavelengths. The weak dependence of plasmonic enhancement on excitation wavelength has also been observed in SERS experiments. While the SE-FSRS lineshapes have a strong dependence on the relative position of the excitation wavelengths with respect to the LSPR, this dependence did not match predictions based on any current SE-FSRS theory. This suggests that current theories are incomplete, as demonstrated by the incorrectly predicted amounts of dispersive character either near an LSPR or at wavelengths redder than an LSPR. Thus further theoretical efforts are needed and should be aided by the collection of additional experimental data over wider ranges of excitation wavelengths. Both theoretical models were applied to single aggregates; the presence of dispersive peaks in the both sets of calculated SE-FSRS spectra indicates that the complex lineshapes observed experimentally are not simply an artifact of the ensemble measurement. However, to better correlate with theoretical results, experimental studies of individual aggregates should be made to remove inherent ensemble averaging. Such measurements would also eliminate any possibility that the broadband probe pulse is being chirped by propagation through the colloidal sample, potentially creating a phase shift in the stimulating field that could add an additional dispersive element to the measured lineshapes and convolute their wavelength dependence.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.jpcl.7b01342.

Additional data and experimental details. (PDF)

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All authors have given approval to the final version of the manuscript.

Funding
National Science Foundation Division of Chemistry grants CHE-1414466 and CHE-1506683. National Science Foundation Division of Graduate Education grant DGE-0824162.

Notes
The authors declare no competing financial interest.

ACKNOWLEDGMENTS

L.E.B., M.O.M., N.L.G., G.C.S., and R.P.V.D. were supported by the National Science Foundation (NSF) Center for Chemical Innovation (CCI) dedicated to Chemistry at the Space-Time Limit (CaSTL) Grant CHE-1414466. M.O.M. and N.L.G acknowledge support from the NSF Graduate Research Fellowship Program (DGE-0824162). All instrumentation used in these experiments was purchased by NSF CCI - CaSTL Grant CHE-1414466. We thank STA Technologies for providing the nanoparticle assemblies.

ABBREVIATIONS

FSRS, femtosecond stimulated Raman spectroscopy; SRG, stimulated Raman gain; SRL, stimulated Raman loss; SE-FSRS, surface-enhanced femtosecond stimulated Raman spectroscopy; BPE, trans-1,2-bis(4-pyridyl)-ethylene; CARS, coherent anti-Stokes Raman spectroscopy

REFERENCES


Supporting Information

Studying Stimulated Raman Activity in Surface-Enhanced Femtosecond Stimulated Raman Spectroscopy by Varying the Excitation Wavelength

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SUPPLEMENTAL MATERIALS AND METHODS

SE-FSRS samples. All SE-FSRS experiments are performed on commercially-available nanoparticle assemblies provided by STA Technologies consisting of one or more gold cores topped with a sub-monolayer coverage of a reporter molecule and overcoated with a silica shell. One sample (BPE90) consists of gold cores with a diameter of approximately 90 nm aggregated with trans-1,2-bis(4-pyridyl)ethylene (BPE), while the other sample (BPY60) comprises approximately 60 nm gold cores aggregated with 4,4'-bipyridine (BPY) (Fig S1). SE-FSRS samples are prepared by suspending the nanoparticle assemblies in water and placing the colloidal mixture in a cuvette with a path length of 2 mm. The sample is stirred constantly during data acquisition using a magnetic stir bar.

1 MHz laser system. The laser system is summarized in Figure S2. The laser system is based on a one-box diode-pumped Yb-doped fiber oscillator/amplifier system (Clark-MXR Impulse). The fundamental output (1 MHz, 1.034 µm, 250 fs, 11 µJ/pulse) is directed into a noncollinear optical parametric amplifier (Clark-MXR iNOPA) where it is split into two beams. Approximately 12% of the fundamental beam is focused into an yttrium aluminum garnet (YAG) plate to generate a broadband white light continuum (WLC) while the other 88% is focused into a type I β-barium borate (BBO) crystal to generate 2.5 W of 532 nm via second harmonic generation (SHG). Half of the WLC is mixed noncollinearly with the SHG in a type I BBO to generate femtosecond bandwidth light at 750, 795, or 840 nm. The beam is sent through a prism compressor, then directed into a home-built 4-f spectral grating filter, which is tuned to each wavelength in order to narrow the spectral bandwidth and create the Raman pump (1 ps; 3.0 nJ/pulse of 750 nm, 3.5 nJ/pulse of 795 nm, 1.4 nJ/pulse of 840 nm). The other half of the WLC, after being filtered through pairs of edge-pass filters (Table S1), is sent through a prism
compressor to generate the stimulating probe field (~30 fs). The pump and probe beams are focused collinearly onto the samples with a 10 cm focal length lens. The beam diameters at the sample are adjusted to be ~55 µm. The probe beam is recollimated and the Raman pump is removed using pairs of optical filters, as summarized in Table S1.

**Data collection.** On each system, the probe and phase-matched (SE-)FSRS signal are sent through a spectrograph (Princeton Instruments Acton SP2300) and focused onto a CCD camera (Princeton Instruments PIXIS 400BR) for self-heterodyned detection. Pump-on and pump-off spectra are collected by chopping the Raman pump at 250 Hz. Subsequent pump-on and pump-off spectra are divided to provide the background-free stimulated Raman gain spectrum.
Supplemental Figure S1. Schematic depiction of nanoparticle assemblies. The nanoparticle assemblies consist of gold nanoparticles with an average diameter of either 90 nm (left) or 60 nm (right) and a broad (roughly 12%) distribution of individual particles sizes. The 90 nm particles are aggregated with trans-1,2-bis(4-pyridyl)-ethylene (BPE) while the 60 nm particles are aggregated with 4,4'-bipyridine (BPY) overcoated with a silica shell. These samples have been characterized previously to contain a range of aggregate sizes ranging for single cores to dimer, trimers, and larger aggregates.¹
Supplemental Figure S2. Schematic of the 1 MHz laser system. Abbreviations: second harmonic generation (SHG), β-barium borate crystal (BBO), lens (L), white light continuum (WLC), yttrium aluminum garnet (YAG), long pass filter (LPF), short pass filter (SPF), prism (P), grating (G), cylindrical lens (CL), detection filters (DF).
Supplemental Table S1. Summary of filters used to collect SRL/SRG signals at various pump wavelengths.

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<th>Detection Filters</th>
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<td>Loss</td>
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<td>Gain</td>
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<td>Loss</td>
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<td>Gain</td>
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<td>840 nm</td>
<td>Loss</td>
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<td>Gain</td>
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LP = long pass; SP = short pass

SUPPLEMENTAL REFERENCES