Observation of resonance-enhanced high-order harmonics from direct excitation of metal nanoparticles with femtosecond pulses

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We report on the observation of resonance enhanced high-order harmonic generation (HHG) from a dense medium composed of indium or tin nanoparticles. Unlike previous studies of resonance enhanced high harmonics from laser produced plasma plume from a prepulse, we generate HHG directly, thus eliminating the undesired contributions from various components in the plasma. We discuss the mechanisms for our observed resonance effect in the absence of preionized particles and the implications of the experimental results on the HHG mechanism in metal nanoparticles.

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I. INTRODUCTION

Over the last three decades, extensive research on high-order harmonic generation (HHG) [1–3] have been performed which covered various types of generation medium including gases [4–13], clusters [14–18], liquids [19–21], solids [22–32], low-density plasma plumes [33–37] and dense plasma surfaces [38,39], etc. In the regime of moderate, non-relativistic driving laser intensities ($<10^{16}$ W/cm^2), atoms and small molecules have been the most used targets for HHG. For species that are in gas phase in their normal form (such as noble gases and small molecules), targets are prepared by using gas jets or gas cells. For atomic species that are in solid phase in their normal form, the majority of the studies employed the laser ablated plasma scheme [33–37] in which a nanosecond or picosecond pulse [the heating pulse (HP)] is first focused on the solid target surface to produce a plasma plume and then a femtosecond laser pulse [the driving pulse (DP)] is focused at the plume to generate harmonics from it.

One of the attractive special features in the harmonics spectra from plasma plumes of several species of metals that are absent in gaseous atoms is the resonant enhancement of a single order harmonic [40–42], that is, the yield of a particular order is higher than the neighboring harmonics by an order of magnitude or more. While it is believed that the formation of the resonant harmonics (RHs) are tied to the fact that the photon energy of these harmonics coincide with the energy of some particular strong electronic transitions in the metal ions, two competing interpretations on how the ionic transition play its role in the enhancement process have been proposed. One is based on single-atom response [43–45] and the other is based on macroscopic phase matching effect [46–48].

Interestingly, RHs have also been observed in the plasma plume of the aforementioned metals but in the form of nanoparticles (NPs) [49]. This is, in fact, somewhat surprising as one may anticipate that the characteristics of the electronic resonances of a tightly bound cluster should be different from an isolated atom. On the other hand, however, the constituents of the plasma plumes produced by ablating metal NPs is not entirely clear. Although there is evidence showing that the plumes contain NPs, which remain intact after interacting with the laser pulses [50,51], it is uncertain that the plumes are free of isolated atoms and ions. In other words, it is still unclear whether the observed RHs are mainly contributed by the NPs or by the isolated atoms.

In this work, we report on the observation of RHs from ablating a thin medium consists of metal NPs with femtosecond near-infrared laser pulses. In contrast to previous work that utilized a HP to produce a preformed plasma plume, we produce RHs directly from a femtosecond DP. Therefore, the measured harmonics can be unambiguously attributed to the interaction of the driving pulses with NPs alone instead of a preformed multi-component mixture containing not only NPs but also isolated atoms and ions. We observed RHs from NPs of indium (In) and tin (Sn), species which have already shown RHs in previous studies with preformed plasma. While the general mechanism of HHG from such a medium is not entirely clear, the fact that RHs survive clearly reveals that HHG from NPs indeed retains some properties of atomic HHG. Our findings help answer the question of whether the enhancement is a microscopic or macroscopic effect.

II. EXPERIMENT

Our experiments use a 0.8-μm Ti:sapphire amplifier system (Spectra Physics: Spitfire Ace). It delivers a 35 fs, 5.5-mJ

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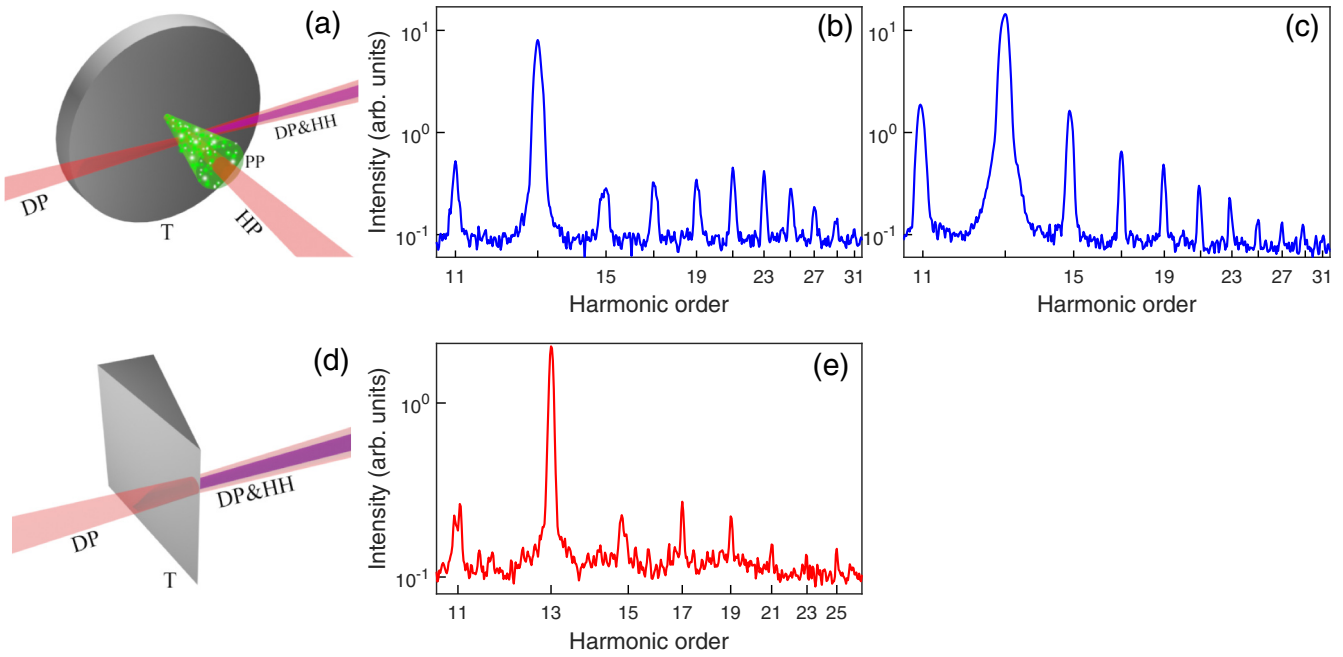

FIG. 1. Schematic setup diagrams and corresponding experimental results with indium targets. T: target; PP: plasma plume; HH: high harmonics. The panels in the upper row are for the case with HP: (a) is a schematic diagram, (b) and (c) are the HH spectra generated from NPs and a bulk target, respectively. The panels in the lower row are for the case without HP: (d) is a schematic diagram, and (e) is a HH spectrum from NPs.

beam and a 200 ps, 1.5-mJ beam (which is a split-off from the amplified beam before compression). The 35-fs pulse is directed to a long optical path in order to create a ∼80-ns delay between it and the picosecond pulse. Due to propagation through transmissive optical components and optical path in air, the 35-fs pulse is broadened to about 52 fs when it arrives at the experimental setup. Unless specified otherwise, the measurements are performed with a laser pulse repetition rate of 200 Hz. The target sample is prepared by first using a hydraulic press (with a pressure of about 8 MPa) to compress indium NPs (average diameter = 50 nm) to form a cylindrical solid of diameter 10 mm and thickness 3 mm. Then it is abraded to form a wedge with a sharp edge of thickness about 100 μm. The sample is placed in a vacuum target chamber and the femtosecond beam is focused on its surface close to the sharp edge, as schematically shown in Fig. 1(d). The full width at half maximum (FWHM) diameter of the focal spot is about 50 μm. When irradiated by the focused laser, the material in the focal region is removed progressively due to laser ablation. The harmonics signal is observed when the sample is drilled through by the laser beam, and the signal vanishes as the ablated hole eventually becomes too big that the laser can no longer interact with the material effectively. After the completion of each harmonic measurement, the sample is translated vertically so that the beam interacts with a fresh surface region in the subsequent measurement. For comparison, we also measure harmonic spectra under the conventional setup of plasma HHG [as shown in Fig. 1(a)], in which a sample with a flat surface of a compressed NPs sample or a piece of bulk metal is first irradiated by a focused picosecond HP (200 ps, intensity $\sim 10^{10}$ W/cm^2) at normal incidence to form a plasma plume. Then HHG from the plasma is driven by a femtosecond DP, which arrives at the plasma plume region at about 80 ns after the HP. The harmonics radiation enters an extreme ultraviolet spectrometer located behind the target chamber. After passing through the entrance slit of the spectrometer, the harmonics radiation is first reflected by a cylindrical mirror onto a 1200 grooves/mm flat field grating, then the dispersed harmonics are detected by a microchannel plate with a phosphor screen and imaged by a CCD camera. A diagram of the target chamber and spectrometer has been presented in Ref. [52].

III. RESULTS AND DISCUSSION

We first describe the results obtained from the conventional plasma HHG setup. The intensity of the DP is kept at 6×10^{14} W/cm^2. The harmonic spectra generated from the plasma of a compressed solid of indium NPs and the plasma of a bulk piece of indium are shown in Figs. 1(b) and 1(c), respectively. In both cases, the yield of the 13th harmonic (the RH) is more than an order of magnitude higher than the neighboring harmonics, similar to previous work in the literature [40,49]. Although there are some differences between the two spectra in terms of the relative intensity of each harmonics order, the cutoff energies of both spectra are essentially the same and the difference in the overall harmonics yield is typically not more than a factor of 2. While the fact that the two spectra are not identical probably implies that the plasma constituents in the two cases have some differences, we do not have sufficient evidence to conclude that the plasma plume for Fig. 1(b) is dominated by NPs and the plasma plume for Fig. 1(c) is dominated by atoms.

As for the measurements of harmonics without preformed plasma, the intensity of the DP is kept the same and the HP is not used. A spectrum is shown in Fig. 1(e). The presence of

the strong 13th order harmonic is still observed. No harmonics signal is detected when the polarization of the DP is changed from linear to circular using a quarter wave plate, which is an expected result as it is a characteristic feature of HHG originated from the strong-field three-step process [6].

An absolute comparison on the harmonics yields between the two setups is out of the scope of this study, but the following key difference should be noted. In the case with preformed plasma, the harmonics signals, which although diminish gradually as the sample surface is progressively destructed by laser ablation, typically survive for at least hundreds of laser shots before disappearing. On the other hand, the harmonics signals in the case of without HPs can only sustain for very few numbers of laser shots. It is because the size of the ablated hole increases rather rapidly as a function of number of laser shots. And as the ablated hole becomes significantly larger than the FWHM diameter of the DP focus spot, then the central intense part of laser beam can no longer interact with the NPs to generate harmonics. To illustrate this phenomenon, we switch the laser to a single-shot mode and perform the following experiments. First, we fire laser pulses (one pulse at a time) on a fresh sample and monitor whether the laser pulse has penetrated through the sample with a camera behind the sample. We kept firing pulses until the camera detects a clear laser spot from the back side of the sample (which implies that a small hole has just been created). The camera is then removed. Subsequently, we fire laser pulses again at the same hole (one pulse at a time) and detect the harmonic signal generated from each pulse. We stop firing pulses as the camera could no longer detect any harmonic signal.

The red circles in Fig. 2(a) represent the detected 13th harmonic intensity as a function of pulse number. It can be seen that the intensity is the strongest at the first few pulses and it decreases gradually. The background noise is indicated by the grey dots. The harmonic signal persisted for about 10 pulses. In order to compare the hole size before and after the harmonic generation process, we used the laser to make another small hole on another fresh area nearby the previous one with the same procedure. After all the experiments, we acquired images of the two holes using a scanning electron microscope (SEM). As shown in Fig. 2(c), the size of the hole, which did not undergo harmonic generation process, is comparable to the focal spot size of the laser. On the other hand, as shown in Fig. 2(b), the hole which underwent harmonic generation process is at least twice as big.

The main finding of this study, that is that the presence of the resonant harmonic in the case without preformed plasma, might appear to be somewhat unexpected at first sight since no HP is present to produce singly ionized particles for the DP in this case. In previous studies of HHG from the plasma of metal NPs [49,51,53], resonant harmonics do not exist when the HP intensity is low and it was speculated that the reason is because there is a lack of singly charged particles in the plasma plume since the HP is weak. However, regardless of the condition of the HP, the ionization caused by the DP itself should not be overlooked. If the DP is sufficiently intense, it should be possible that even the rising edge of the pulse can ionize a significant portion of the particles (which are all neutral initially, if the HP is ignored) and subsequently for the central portion of the pulse to interact with these ionized

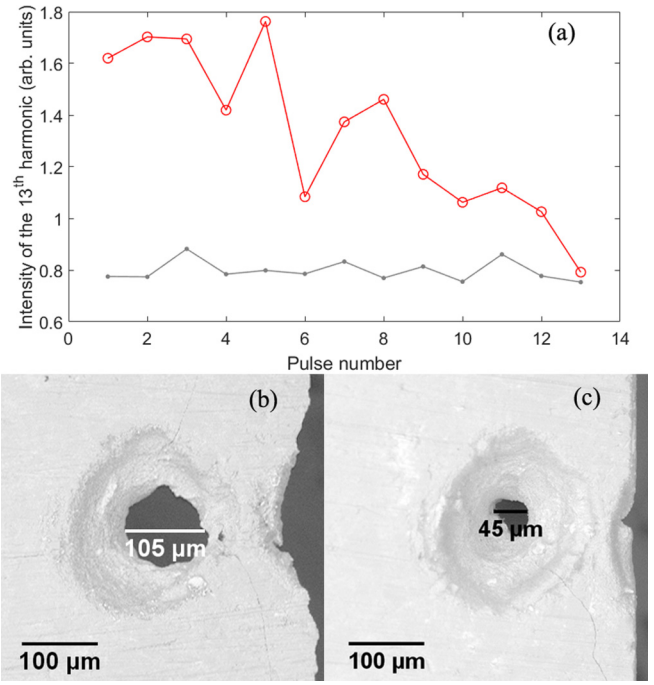

FIG. 2. (a) Intensity of the 13th harmonic as a function of pulse number (see text). (b) SEM image of the front view of a sample surface after the harmonic generation experiment. (c) SEM image of the front view of a sample surface with a preformed small hole but was not used for harmonic generation experiment.

particles to generate resonant harmonics. To investigate such a possibility, we use the Perelomov-Popov-Terent'ev (PPT) formula of ionization rate [54], which has been shown to be applicable for ionization of various atoms in both multiphoton and tunneling regime [55–57], to calculate the ionization yields of indium atom and ions irradiated by a femtosecond DP. In the calculation, the peak intensity of the DP is 3×10^{14} W/cm^2, which is the lower bound value for the intensity distribution of the region within the FWHM diameter of the focal spot in our experiment. The pulse envelope is assumed to have a sine-squared shape. The FWHM pulse width and central wavelength are set to match the experimental values. The initial population of neutral atom is unity and the populations of singly, doubly, and triply charged ions are all zero. The populations of the charge states as a function of time are obtained by numerical solving a set of coupled rate equations [58] where the ionization rates are computed using the PPT formula. Note that only sequential ionization mechanism is considered. The results are shown in Fig. 3. It can be seen that as the laser pulse starts ramping up, the population of In^+ rapidly saturates. Subsequently, as the central part of the DP approaches, most of the In^+ population is further ionized. This calculation indicates that at the intensity level of our experiment, even without using the HP, a significant amount of In^+ are produced by the rising edge of the DP and are available to contribute to the harmonics generation process. Note that our ionization calculations did not include the collisional ionization mechanism for simplicity, which means that the overall ionization probabilities could have been even higher than we estimated. Regardless, this would not go against our

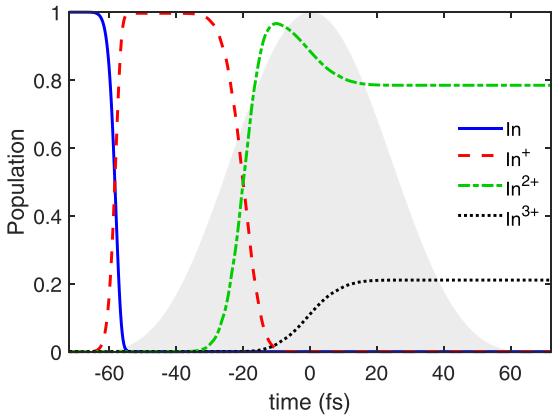

FIG. 3. Calculated population of different charge states of an indium atom irradiated by a DP (see text). The temporal profile of the DP intensity is represented by the shaded region.

conclusion that the rising edge of the driving pulse is capable of producing a significant amount of ions.

Given the significant amount of ionization events, one may question whether the NPs remain intact during the interaction with the DP. To estimate the expansion of an indium NP caused by the DP, we perform calculations using the expanding fluid model [59], which has been extensively used in strong-field studies of rare gas and metal clusters (see Ref. [60] and references therein). With the same intensity and pulse profile as the calculation in Fig. 3, the calculation shows that the NP diameter is expanded by less than 10%. Here the percentage of expansion refers to the difference between the initial diameter (50 nm) and the diameter at the instant when the DP reaches its peak. If the laser intensity in the calculation is increased to 6×10^{14} W/cm^2, which is the estimated experimental intensity at the center point of the focused laser spot, the percentage of expansion is less than 20%. Therefore, our calculations suggested it is likely that the NP has not yet been completely disintegrated during the HHG process, although its structure may have been evolving significantly. Note that these calculations only model the expansion of an isolated NP but not the interaction between neighboring NPs.

Tin is another species that is known to show RHs from previous HHG studies with preformed plasma. As shown in Fig. 4(a), the enhanced 17th harmonic is clearly observed in a spectrum generated from a HP produced plasma from a bulk piece of tin. To test whether RH can be generated from tin NPs without performed plasma, we perform the same measurement as illustrated in Fig. 1(d) but with a sample composed of tin NPs. The result is presented in Fig. 4(b), it can be seen that the enhancement of the 17th harmonic is still clear. The other peaks in the spectrum are due to plasma emissions from the singly and doubly charged ions of tin and oxygen.

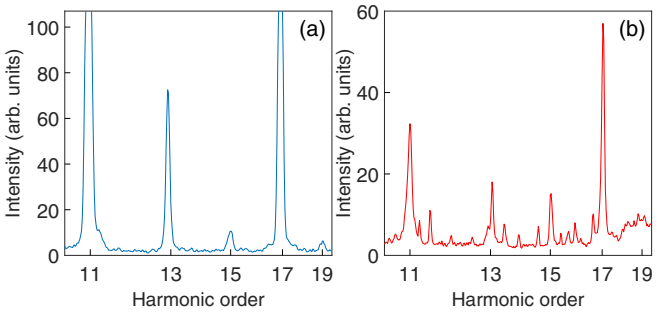

FIG. 4. (a) Harmonics spectrum generated from the plasma plume of a bulk piece of tin. (b) Harmonics spectrum generated from tin NPs without preformed plasma.

As mentioned, there have been two main proposed mechanisms for the RHs from metal ions in the literature. In the microscopic mechanism proposed by Strelkov [44], the key process is that the returning electron recombines to an autoionizing state and then relaxes to the ground state, instead of directly recombining to the ground state. For the case of In plasma, it has been suggested that the responsible autoionizing state is $4d^9 5s^2 5p(^2D)^1P_1$ of In^+ [40]. The transition energy between that state and the ground state $4d^{10}5s^2\ ^1S_0$ is 19.92 eV, which is very close to the photon energy of the 13th harmonic of 0.8-μm laser. As for Sn plasma, the energy of transition between $4d^9 5s^2 5p^2(^1D)^2D_{5/2}$ and $4d^{10}5s^2 5pP_{3/2}$ of Sn^+ is very close to the photon energy of the 17th harmonic of 0.8-μm laser [42].

The macroscopic mechanism, on the other hand, is based on the speculation that the resonance of the ions significantly contributed to the dispersion properties of the plasma plume in a way such that the refractive index of the plasma at the wavelength of a particular harmonics becomes close to the refractive index at the wavelength of the driving laser, thus leading to a favorable phase matching condition for the harmonics whose frequency is close to the resonance frequency. However, to the best of our knowledge there has been no experimental data of the dispersion properties of laser-produced indium plasma in the spectral region of interest and therefore it has been very difficult to directly justify the explanation. However, despite the lack of exact knowledge about the plasma dispersion properties, in our case it is still very reasonable to anticipate that the refractive index of the plasma produced from the sample is significantly different from the refractive index of the compressed sample itself. It is because the density of free electrons and ions are very different from each other in the two cases. Therefore, the fact that the enhanced 13th order harmonics are observed in both cases seems to suggest that the macroscopic properties of the generation medium is not the decisive factor for creating the enhancement. Note that the results of several recent experimental investigations on discriminating the two mechanisms [61–63] are also consistent with the microscopic mechanism and no clear evidence of the phase matching effect have been observed so far.

Until now, research on the HHG mechanism from metal NPs is still scarce [64]. In particular, the mechanism of electron recollisions for NPs and how it is different from the case of single atom is not entirely clear. It is noteworthy that in the HHG studies of rare gas clusters (which is essentially another type of NP), different mechanisms have been proposed and are still debatable. Some studies suggested that the electron wave function responsible for HHG is partially delocalized, which spreads over the cluster [16,17]; other studies suggested the harmonics are just emitted from individual atoms in the cluster [15,18]. If one accepts that the RH is indeed originated from

the microsopic mechanism due to electronic resonance of a single ion [44], our observation of RH from NPs seems to indicate that the latter mechanism plays a significant role in HHG of NPs. It is because the delocalized electron wave-function model does not seem to be compatible with the mechanism in Ref. [44], which is based on the wave function of a single ion.

IV. CONCLUSIONS

In conclusion, we have demonstrated HHG and observed the RHs from NPs of indium and tin without utilizing a preformed plasma plume produced by a HP. Although the appearance of the RH in the absence of preionized particles might appear, at first glance, inconsistent with the proposed mechanisms of the RH, strong-field ionization calculation indicated that the rising edge of the intense DP alone can produce the singly charged ions required for the resonance process. Also, our results seem to suggest that the electron recombination at individual atoms play a significant role in HHG from NPs. In addition, the experimental scheme can be applied to other species of NPs and potentially can act as an alternative method to study HHG from NPs.

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