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Resonant third harmonic generation of KrF laser in Ar gas

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Investigations of emission of harmonics from argon gas jet irradiated by 700 fs, 5 mJ pulses from a KrF laser are presented. Harmonics conversion was optimized by varying the experimental geometry and the nozzle size. For the collection of the harmonic radiation silicon and solar-blind diamond semiconductor detectors equipped with charge preamplifiers were applied. The possibility of using a single-crystal CVD diamond detector for separate measurement of the 3rd harmonic in the presence of a strong pumping radiation was explored. Our experiments show that the earlier suggested 0.7% conversion efficiency can really be obtained, but only in the case when phase matching is optimized with an elongated gas target length corresponding to the length of coherence. © *2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4902346]

I. INTRODUCTION

Harmonic generation in a gas phase target at relatively low laser intensities is driven by multiphoton excitation and described by perturbation theory.[1–6] The generation of a specific harmonic can be greatly enhanced if there is a real bound state to which the multiphoton resonance can occur.[1] That is the case in argon with a three-photon near-resonance of KrF laser at 248.5 nm.[7] The ponderomotive energy of the ionized electron is smaller for UV radiation as compared to IR or visible radiation with the same field amplitude. This makes a Keldysh parameter greater than one, thus photoionization occurs with multiphoton processes. As a result the UV light of excimer lasers results in a dominance of the multiphoton process in harmonic generation. Harmonic intensity and the phase matching ruled by a perturbative formalism in the UV laser range worsens drastically with the harmonic order.[1]

For the short-wavelength (UV) laser pulses of a KrF laser even low order harmonics have high photon energy, thus efficient generation of intense coherent extreme ultraviolet (EUV) radiation is possible. A high intensity source of this wavelength is needed to study nonlinear phenomena in the EUV, and optical damage of solid and biological samples can also be investigated. Harmonic generation due to its coherent nature conserves the short duration of the pulse while spectrally upconverting it into harmonics. Experiments of Dölle *et al.*[7,8] showed the possibility of resonant frequency tripling of KrF laser radiation in Ar gas. In their experiment a conversion efficiency of ∼0.7% was claimed for a 10 mJ laser pulse, thus obtaining intense coherent radiation at the 82.8 nm wavelength. That conversion measurement was carried out by using vacuum photodiodes where the separation of the harmonics from a stronger laser signal was complicated. Dispersion gratings were used for the separation, for which tabulated efficiency data had to be taken into account causing a lot of uncertainty. Clearly, applying solar blind detectors, which are not sensitive to 248-nm laser radiation, is advantageous. In the experiment we attempted to use a diamond detector (DD), which has a high discrimination ratio between the fundamental KrF laser radiation and its harmonics, thus allowing a direct measurement of the produced harmonic content.

Dölle *et al.*[7,8] measured the high conversion efficiency using a small f-number focusing, thus obtaining a relatively long Rayleigh length, much longer than the size of the used gas targets. However, the obtainable good phase matching[7] permits for the use of longer gas targets and thus obtaining even higher conversion. In the present experiments we show that using several mm long gas-jet targets matches the coherence length of phase-matching, and thus allows the amplification up to several mm. Different types of density-calibrated gas-jet targets with a 0.65, 3, and 9 mm length were utilized, thus extending the amplification up to several mm and obtaining the possibility to demonstrate phase matching up to theoretically estimated geometrical coherence length of 4.4 mm.

II. EXPERIMENTAL ARRANGEMENT

The experimental arrangement for measuring harmonic radiation is shown in Fig. 1. The laser used in the experiments was a hybrid dye-excimer system with a KrF amplifier.[9,10] The excimer laser technology made it

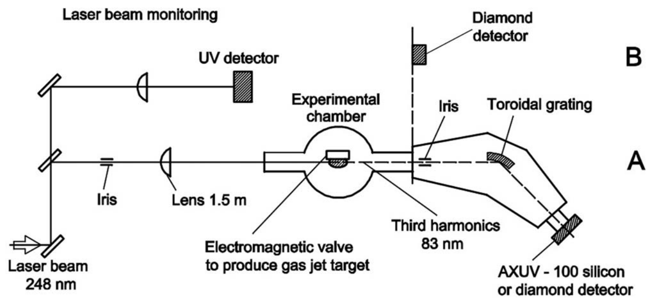

FIG. 1. Experimental arrangement (not to scale) to measure harmonics with different gas-jet valves. In configuration A, radiation is detected using a monochromator; in configuration B, the diamond detector directly recorded the radiation.

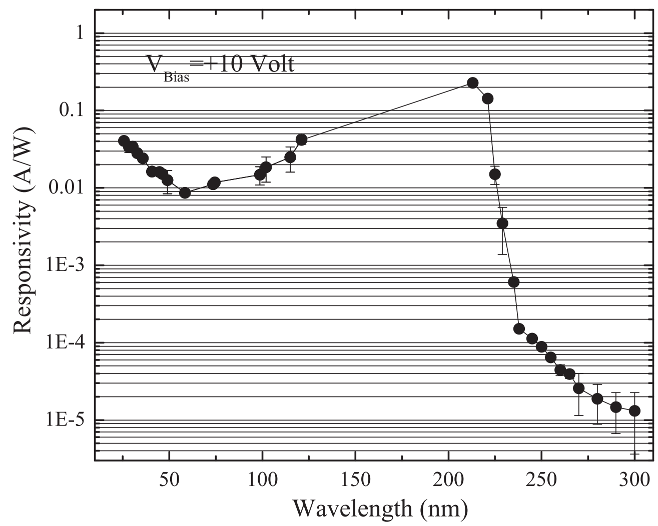

FIG. 2. Spectral sensitivity of the DD and its drop at 225 nm.

possible to achieve multiterawatt laser pulses.[11,12] Moreover, the generated laser pulses are to some extent a wavelength and duration tunable.[10,13] Such a laser is capable to amplify short pulses with a pulse duration down to a few tens of femtosecond.[9,14,15] For the present experiment a 15 GW system was used. Laser pulses of 700 fs and energy up to 9 mJ were focused with a 1.5-m lens and also in some of the measurements with a 0.75-m lens (at correspondingly less laser energy). The energy of laser pulses was monitored with the use of DET 210, THORLABS UV energy meter. In most of the experiments, a single stream nozzle of 0.65-mm diameter (Parker Instrumentation) was used which was characterized by x-ray backlighting.[16] Longitudinal nozzles of 3 and 9 mm length developed in the Institute of Optoelectronics (Warsaw) were also used. Gas density was varied by the delay between the opening time of the valve and the time of the laser pulse coming. The generated harmonic radiation was investigated and optimized with the use of the EUV monochromator (Jobin-Yvon prod.) equipped with a 550 l/mm toroidal grating (see Fig. 1, A configuration). To collect the radiation behind the monochromator a silicon semiconductor detector AXUV-100 (IRD prod.) 10×10 mm^2 in size or a single-crystal CVD DD 4×4 mm^2 in size (SCD301 type, Tor Vergata University) were used. In some measurements, harmonic radiation was recorded directly from the gas-jet target (without using of the monochromator). In that case only the DD was employed, which is blind to the fundamental radiation of the laser (Fig. 1, B configuration).

The AXUV100 is a commercial detector for which calibration data are available. It is not solar blind (i.e., not selective for 82.8 nm—3rd harmonic of the KrF laser). It is sensitive in a broad range down to 10 nm due to a special semi-transparent structure of the surface electrode layer.

The DD detector was fully fabricated at Tor Vergata University including the fabrication of the starting material. The diamond is characterised by a broad band-gap of 5.5 eV resulting in a cut-off of sensitivity of a DD at around 225 nm, which makes it very suitable for production of solar blind EUV detectors.[17,18] The DD used in the experiment was manufactured in a multilayered structure obtained by a three-step deposition process. A conductive p-type boron-doped diamond layer was homoepitaxially grown on a commercial low cost diamond substrate (and later used as a back electrode). Next, an intrinsic diamond layer of a 20-μm thickness was grown on the doped one and made an active (sensitive) layer. It is a single-crystal structure. Finally, a semi-transparent front Pt electrode of ~5 nm thickness was thermally deposited on the intrinsic diamond surface. Such a structure, operating in transverse configuration, acts as a Schottky barrier diode.[19] In our experiment a bias up to 28 V was applied to the detector in order to increase the dynamic range of the response. The detector was preliminarily tested at the Tor Vergata University over a wide spectral range spanning from the EUV up to visible radiation. The He-Ne laser and EUV toroidal grating monochromator were used to study the detection system in the range of 20–120 nm. An optical tuneable laser (210–300 nm) was used to investigate the visible-band properties of the detector. In the spectral range between 20 and 120 nm, the DD has the responsivity minimum at about 60 nm corresponding to the minimum in the Pt-contact transmission curve (see Fig. 2). At around 215-nm there is the maximum responsivity after which it drops down dramatically resulting in three orders of magnitude difference between 225- to 248.5-nm wavelength, and more than 4 orders of magnitude around 300-nm. This demonstrates a high capability of the DD in discriminating visible light.

Signals from the detectors were collected with the use of two charge preamplifiers; one home-built, based on a TL071 operational amplifier, which is of a low-sensitivity (about 1 mV/MeV, Si equiv.). The TL071 was useful for direct measurement of harmonics from gas-jet target. The second preamplifier was a commercial CSTA2 (TU Darmstadt, Germany) which features a high sensitivity (100 mV/MeV, Si equiv.) and is utilized in nuclear spectroscopy. In our study it was used for a measurement of weak higher harmonics at the output of the monochromator.

III. SOURCE OPTIMIZATION AND CHARACTERIZATION

The generation of harmonics was optimized by adjusting the position of the beam focus relatively to the position of the nozzle, the backing gas pressure, opening time, and delay

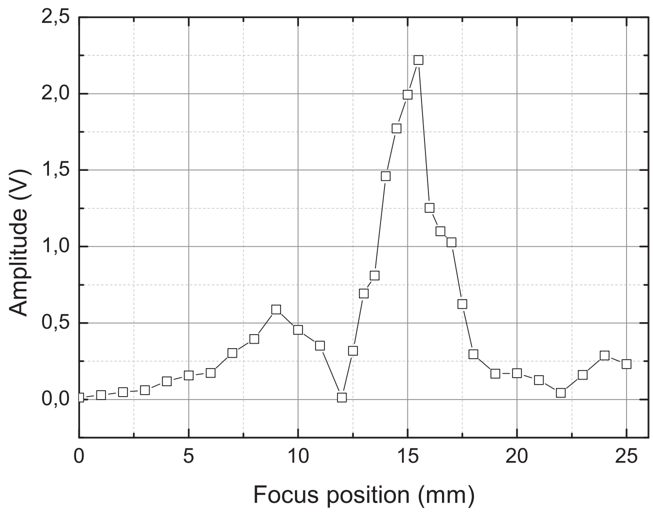

FIG. 3. Dependence of 3ω harmonic emission on focus position measured with the circular nozzle and DD (bias: 28 V), laser energy $\approx$3 mJ, opening time of 1.8 ms, gas backing pressure of 3 bars.

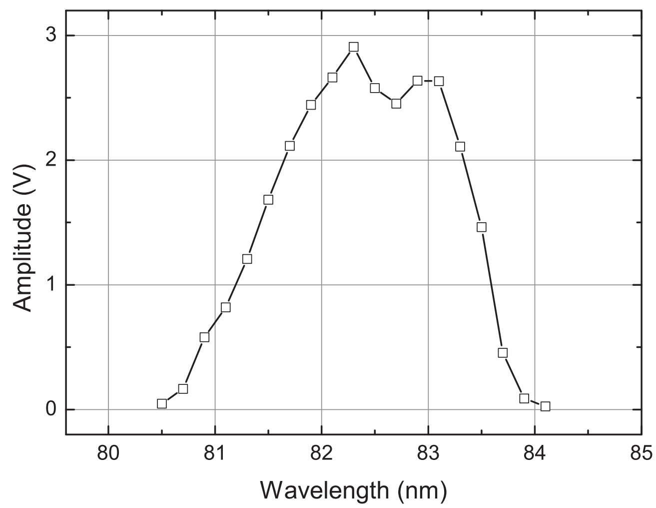

FIG. 5. Spectrum of the 3rd harmonic measured with the DD, opening time of the circular nozzle of 1.8 ms, focus position of 15.5 mm, gas pressure of 3 bars, detector bias of 28 V.

of the laser pulse in regard to the starting time of the gas jet. All above measurements were realized in A configuration (Fig. 1). In this configuration pulses coming to a detector were of relatively low intensity and the detector operated in the linear range (see Sec. III B). Due to the high sensitivity of the detection system based on the charge preamplifier, it was possible to capture high harmonics up to 7th. As the Parker valves were characterized earlier we could optimize the generation of 3ω radiation by varying the backup pressure and the valve opening time. The results of the optimization using the Parker valve nozzle are shown in Figs. 3 and 4. The optimal opening time was of 1.7 ms and the optimal gas backup pressure of about 3 bars.

The typical 3rd harmonic spectrum is shown in Fig. 5. It shows a dip structure, which probably reflects the same dip structure of the fundamental radiation observed in other experiments[20,21] and which are seen also in the harmonic spectra recorded by Dölle *et al.*[7] For this reason the spectral width (FWHM) of the harmonics is of about 2 nm.

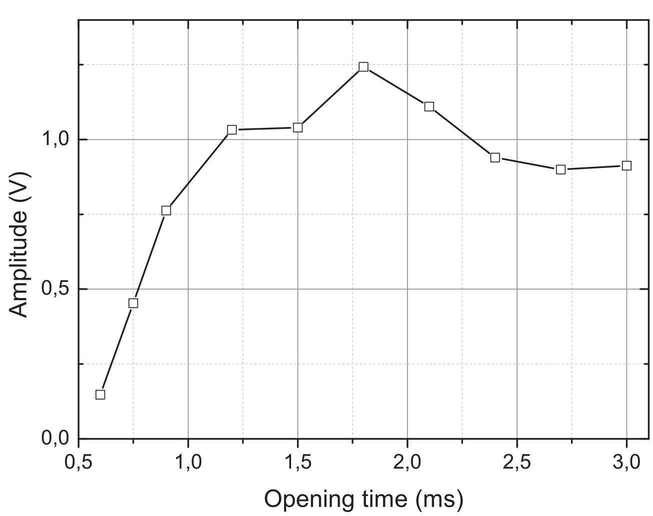

FIG. 4. Dependence of 3ω harmonic emission on opening time of the nozzle measured with the DD (bias: 28 V).

A. Results and discussion

The dependence of harmonic intensity on the laser energy shows that saturation of harmonic generation (due to a strong ionization of the gas) occurs for laser energies above 4 mJ, which corresponds to a pump intensity of around 2×10^{14} W/cm^2. To avoid saturation by ionization, the pump intensity was reduced by the laser beam defocusing.

The qualitative results obtained with the circular Parker valve[16] were comparable to those reported by Dölle *et al.*[7,8] Similarly, we observed that depending on energy, harmonic generation was stronger when placing the gas jet on either side of the focal spot rather than in the focus. At a lower laser energy the maxima for three different positions were observed, as it is shown in Fig. 3. While using the circular valve with an orifice of 0.65 in diameter, the expected coherence length was longer than the interaction length. As it was discussed in detail by Dölle *et al.* the 3rd-harmonic power, at a low pump power P_ω,—when neglecting its absorption—has a cubic dependence on the pumping power ($P_{3\omega} \sim F \cdot P_\omega^3$) with the phase matching function F given by

$$F = N^2 L^2 \frac{\sin^2(\Delta k L/2)}{(\Delta k L/2)^2}, \tag{1}$$

with N being the density of neutral particles, L the interaction length, and Δk the wave vector (phase) mismatch. The total mismatch Δk consists of three parts: a dispersive neutral gas (atomic) contribution $\Delta k_{\text{disp}} = N\Delta k_{\text{disp,A}}$ (proportional to the vector mismatch per atom $\Delta k_{\text{disp,A}}$), a contribution from the free electrons with $\Delta k_{electr} = 8(\lambda/3) r_e N_e$ (where λ is the wavelength of the laser, N_e is the electron density, and r_e is the classical electron radius) in the case of some ionization a plasma dispersive term is present, and a geometrical contribution (which can be approximated by $\Delta k_{\text{geom}} = 4/b_\omega$ for a weakly focused pump beam—with the confocal parameter b_ω).

The total phase mismatch is thus

$$\Delta k(N, \lambda) = N \Delta k_{disp,A}(\lambda) + \Delta k_{geom} + \Delta k_{electr}. \qquad (2)$$

According to Dölle *et al.*,[7] the second and third terms always give a positive contribution to the phase mismatch of $\Delta k = k_{3\omega} - 3k_{\omega}$ and the neutral atomic term can be either positive, zero, or negative, depending on the wavelength. This behaviour makes a perfect phase matching possible in the case of negligible ionization allowing a relatively long range of harmonic amplification. In Ref. 7, a gas-jet target diameter was chosen to be shorter than the available phase matching length. In the present experiments we reproduced their results using a similar gas target system, then employing longer gas-jet targets we could increase the conversion efficiency using the full phase-matching interaction length.

Interestingly, further 3rd harmonic intensity enhancement was observed for laser parameters causing a visible plasma column creation in the gas jet which can be attributed to nonlinear propagation effects (also found in high-order harmonic generation (HHG) with Ti:Sa lasers).[22] This led to extending of favorable condition for efficient HHG beyond the coherence and absorption lengths. To our knowledge the effect relays on spatio-temporal reshaping of the fundamental laser pulse propagating under filament-like conditions resulting in a spatial flattening of Gaussian pulse distribution (thus a greater part of the beam profile matches to optimum HHG), changing of the geometrical phase-matching contribution to Eq. (2), and also the free electron contribution due to the redistribution of the ionization rate along the gas jet.[23]

An interesting consequence of the strong sensitivity of the dispersive contribution to wavelength and atomic number was the observed oscillating property of harmonic conversion on the particle density for 247.7-nm wavelength. As illustrated in Fig. 6, we obtained similarly to Dölle *et al.*[8] an oscillating behaviour of the 3rd harmonic intensity vs. the particle density which is caused by the periodicity of the phase mismatch factor, i.e., $\Delta k_m = (2m + 1)\pi/L$, in which the periodicity (m = 0, 1, 2...) is caused by the atomic factor proportional to the density.

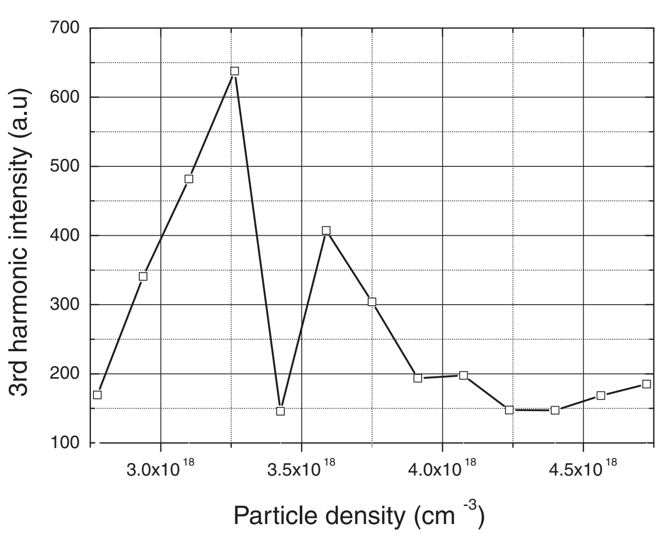

FIG. 6. Dependence of 3ω conversion on the atomic density.

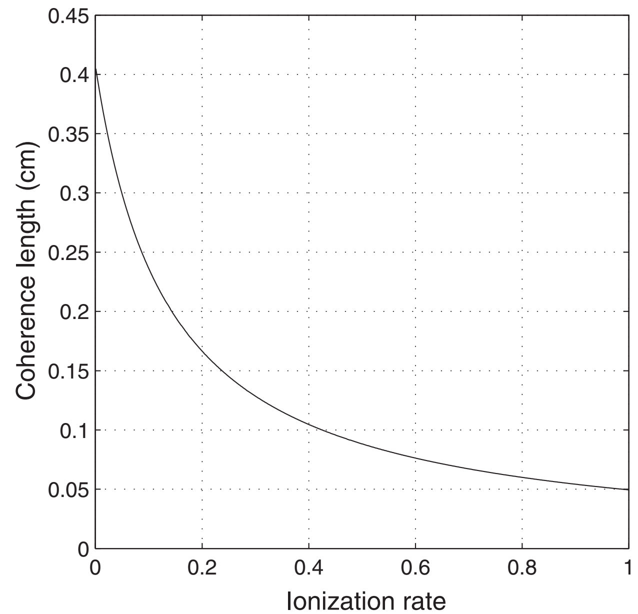

FIG. 7. The calculated coherence length of 3rd harmonic as a function of the ionization degree for the atomic density of 3×10^{18} cm^{-3}, beam diameter of 2.5 cm and focal length of 1.5 m.

In the following we assume the special case when the atomic contribution of phase mismatch is set to zero and the phase mismatch is the sum of the geometrical and the free electron term. The calculated corresponding coherence length ($L_{coh} = \pi/\Delta k$) is shown in Fig. 7 as a function of ionization rate for typical experimental parameters. It can be seen that a coherence length as high as 4.4 mm can be obtained in the case when the gas remains neutral. With increasing ionization the obtainable coherence length drops to ~1 mm—the size of the gas jet used in Ref. 7—when the ionization rate exceeds 40%. In order to take an advantage of the actual coherence length, experiments were carried out with 3 and 9 mm long rectangular-shaped elongated nozzles. The increase of the 3rd harmonic conversion efficiency by 80% was observed for the 3-mm nozzle length and 3rd harmonic intensity was 2.5 times more for the 9-mm nozzle than with the Parker valve. The length of the longest nozzle was greater than the expected length of coherence. Thus, the nonlinear propagation effects, pump depletion and harmonic absorption (which may have been present in this case) did not cause significant loss. Thus, the propagation in a longer coherence length was probably the reason of HHG generation improvement.

B. Application of the diamond detector to direct measurement of harmonic radiation

When exposed directly to radiation from the gas-jet target, a DD is irradiated simultaneously by UV fundamental radiation at 248.5 nm of the KrF laser propagating in the same direction, which almost freely penetrates through the gas jet, and EUV harmonic radiation (with the most intense 3rd harmonic at 82.8 nm). In the case of a DD, the

248.5-nm radiation is out of the range of sensitivity, but still it interacts very weakly in the whole volume of the active layer by carrier recombination (via defect states acting as recombination centres—monomolecular process). The monomolecular process leads to a linear dependence of response on the input signal. Instead, the harmonic radiation, which falls into the short-wave range of high sensitivity of the detector, interacts strongly and thus penetrates very shallowly (in nanometer scale). The interaction undergoes two coexisting processes, band-to-band recombination (bimolecular process) and the monomolecular process mentioned above. In this case, the dependence of detector response to harmonic radiation on the intensity of the laser pulse is expressed by a power function with the exponent ranging from 0.5 to 1 depending on which process dominates (determined by the density of impurities and intensity of radiation).[24,25] The exponent 0.5 is expected at full switching to bimolecular process that occurs at high intensity.

The response of our detector was investigated by varying the laser energy up to 16 μJ with the use of an ArF laser operating at 193 nm which is falling in the range of the DD's high sensitivity (Fig. 8). As the detector had an area of 16 mm^2, the energy corresponds to power density of 60 μJ/cm^2. The dependence of the photoresponse amplitude on the energy of laser pulses is characterised by the exponent 0.38 which is close to 0.5. However, as it is out of the range 0.5–1, it may indicate a participation of an additional process enhancing nonlinearity, which could be carrier-carrier scattering. Also, the loss of pulse energy in the Pt front electrode can affect the dependence curve. The 0.5 exponent and saturation at higher pulse energy were reported for the polycrystalline CVD DDs using pulses from an ArF laser at energy density above 6 μJ/cm^2.[26,27]

In our direct measurement of radiation from the gas jet, the DD was placed at a distance of 77 cm from the gas jet on the laser beam axis. To exclude the likely detector overloading, a filter made of a kapton film covered with a thin Cu layer with a number of holes of 50 μm in diameter was applied. Attenuation of the filter measured with the use of α-particles

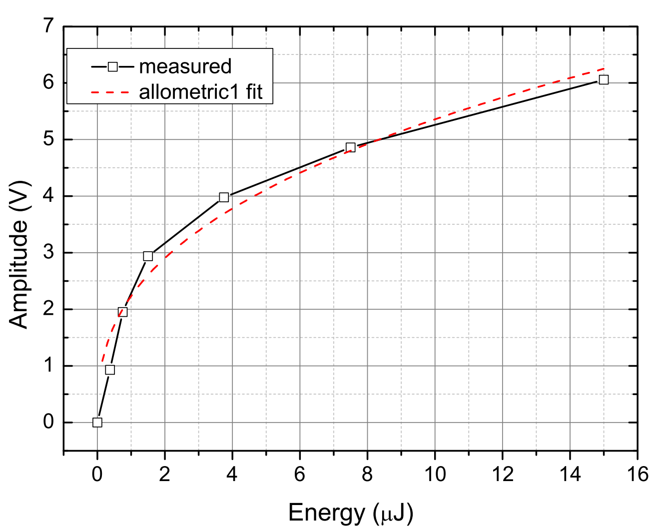

FIG. 8. The response of the DD (SCD301) biased at 10 V to 20 ns pulses of ArF laser. The dotted line is the fit with the scaling exponent of 0.38.

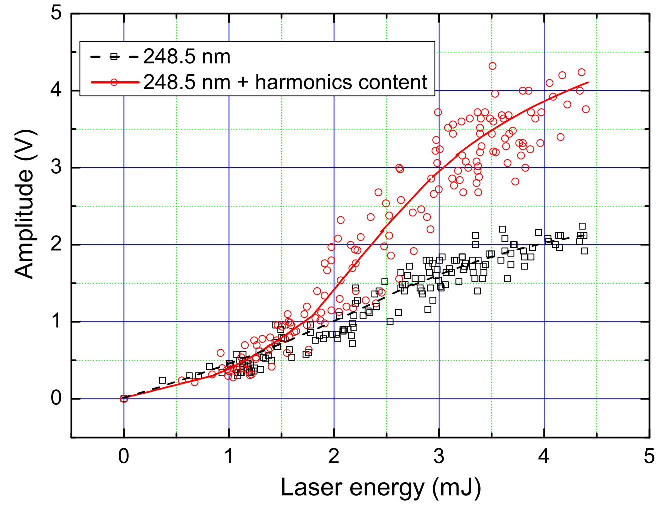

FIG. 9. The DD signal dependence on laser energy with the gas jet (upper curve) and without it (lower curve).

was 7.5 $\pm$ 0.4. Figure 9 shows the response of the detector as a function of the incoming laser energy in two cases: one when gas jet is switched on and the combined harmonic and fundamental radiation reach the detector, and when it is switched off and only fundamental radiation strikes the detector. The test (carried out with the circular Parker-valve) shows that the background signal from the fundamental radiation occurred to be quite large and it is comparable with the signal from harmonics. Also, the dependence of the background signal on energy is not exactly linear. Harmonics start to be generated for the laser energy above 1 mJ. The pure signal corresponding to the harmonics (dominated by the 3rd harmonic) is obtained by subtracting the signals of the two measurements: with and without a gas jet. It is assumed that the loss of fundamental radiation propagating through a gas jet is negligible.

To obtain absolute value of harmonic emission, both absolute calibrations of the detector for the harmonic and for fundamental radiation in the full relevant range of laser pulse energy are needed. The calibrations should be made in the condition of pulse stimulation, which is different than that of continuous stimulation. The calibration for fundamental radiation is straightforward, while for 82-nm 3rd harmonic it is more complicated due to lack of a suitable intense pulsed source at this wavelength or nearby. A synchrotron source does not deliver pulses intense enough, e.g., the Bessy II in Berlin produces pulses of only up to 50 nJ in a single bunch mode. We plan to use a capillary laser, which operates at 46.9 nm.[28] It is encouraging that the responsivity of our detector (in the case of a steady stimulation) practically does not differ at 46.9 and 82 nm, as it is seen in Fig. 2.

At present we could make a rough estimation of the conversion efficiency to the 3ω radiation. In the case of incoming 4 mJ laser energy the harmonic signal adds linearly in the detector response to that of the laser. It is seen in the experiment that these signals are approximately of the same strength (Fig. 9). For our present approximation we use the earlier calibration done at Tor Vergata University, Rome, in which a low-intensity cw source was used. In that case the detector behaved linearly even for the 82.8 nm radiation. In Fig. 2, a responsivity ratio of 1000 times for the two wavelengths (fundamental ω and 3ω) is shown. Assuming that this proportion is also valid in the case of pulse stimulation, the observed energy of the 3ω radiation is approximated to be 4 μJ. Additionally, our calibration with a pulsed source (as seen in Fig. 8) refers to a nonlinear behaviour and for the observed range of amplitudes (between 2 V and 4 V), the detector was effectively about 3 times less sensitive for the harmonic pulse. This gives an estimate of 12 μJ for the harmonic energy. However, taking into account the higher conversion efficiency attributed to the elongated target, it can be concluded that at the optimum phase matching length the harmonic energy of about 30 μJ can be obtained. The conversion efficiency corresponding to this energy is however still lower by factor of 3 than the ~0.7% obtained by Dölle *et al.*[7]

IV. CONCLUSIONS

Measurements of harmonics produced by a femtosecond KrF laser in argon gas jets were carried out using different types of nozzles, jet timing, laser parameters, and different types of detectors. A very sensitive method of harmonics detection was introduced by applying charge preamplifiers. The highest harmonic conversion was obtained by using a long coherence and a corresponding long-jet nozzle. When employing longer gas-jet targets a conversion efficiency increase was observed up to a full phase-matching length. The length of the longest nozzle (9 mm) was approximately twice longer than the expected length of coherence. The obtained improved conversion can indicate that besides the appearing nonlinear propagation effects, harmonic absorption and possible pump depletion do not cause significant loss. The saturation of harmonic generation (due to ionization) was observed above 4 mJ (~2×10^{14} W/cm^2) of laser energy. Oscillating property of harmonic conversion in dependence on the particle density was observed for the 3rd harmonics.

For the first time, a single-crystal EUV CVD DD was used for direct measurement of the harmonic radiation (dominated by 82.8-nm 3rd harmonic) produced by interaction of 248 nm pumping radiation from a KrF laser with the gas-jet argon plasma. The harmonic radiation interacts very shallow in the detector material, which leads to very high surface density of carriers causing nonlinear response described by a power function. The response to intense 248 nm radiation occurred to be large in spite of the high out-of-band attenuating factor and was comparable to the response to harmonics. Nevertheless, the detector proved to be useful in detection of the EUV harmonics and pumping VUV radiation. It can be used for absolute measurements when properly calibrated in a pulsed mode in the relevant range of radiation intensity.

The present estimation of conversion efficiency gives lower conversion efficiency than that of Dölle *et al.*[7] On the other hand, it is shown that by optimizing the coherence length the conversion efficiency suggested therein can be potentially obtained. These studies show that it is possible to obtain harmonic efficiency at 82.8 nm against the laser energy of the order of 1%.

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