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ABSTRACT

We propose, test, and analyze a new step-wise excitation approach to quickly obtain, or determine with high accuracy, the resonance frequency of a microparticle confined in the space charge region of a low pressure plasma operated above a horizontal surface. The local value of this resonance frequency can directly be related to the charge of such a plasma-immersed microparticle and is, therefore, essential to be obtained with respect to the interpretation of many dusty and complex plasma laboratory experiments. The presented approach applies a step-wise change in plasma power, after which the damped harmonic oscillatory motion of the plasma-confined microparticle toward its “new” equilibrium confinement position is monitored temporally resolved and fitted with the theory describing this damped harmonic oscillator. Proof-of-principle experiments demonstrate that the results from this method match the results of the traditional frequency sweep resonance method within 5%. Comparing to existing—for instance, phase-resolved or single particle injection—methods to determine the resonance frequency, this approach offers the experimenter a unique possibility to choose the trade-off between reduced measurement time—essential when working with particles that are vulnerable to plasma etching—and measurement accuracy.

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I. INTRODUCTION

The physics of complex and dusty plasmas—i.e., the study of small (nanometer- to micrometer-sized) solid and charged particles confined in low pressure plasmas—has been an active research topic for several decades already.^{1–3} The related ecosystems studied can roughly be divided in two subsets.

The first subset includes systems in which particles are grown inside reactive gas-plasmas, such as silane,⁴ methane,⁵ acetylene,^{6,7} and hexamethyldisiloxane.⁸ Once grown sufficiently large, these particles may be confined in the positive plasma potential due to the permanent negative charge they obtain from the plasma bulk. In these so-called dusty plasmas, particle sizes range from the size of large molecules up to several micrometers while particle number densities as high as 10^{15} m^{-3} are found depending on the plasma parameters and the phase in the particle formation process.⁹ The reactive plasmas, in which these particles can be grown, have been recognized to have numerous applications ranging from the synthesis of nanostructures¹⁰ to thin film deposition.¹¹

The second subset of ecosystems—often referred to as complex plasmas—includes plasmas, which contain a countable number of

particles, often externally injected. The used particles in such systems are often well-defined in terms of morphology, size, and material composition. The advantage of such systems is that the cloud of confined microparticles in this regime may lead to strongly coupled systems,¹² allowing them to be used as macroscopic systems to study fundamental phenomena, such as crystallization,¹³ phase transitions,¹⁴ and (dust) density waves under both laboratory conditions on Earth,¹⁵ and under microgravity conditions in the International Space Station.¹⁶

The fundamental importance of micrometer-sized particles inserted in plasmas is based on the fact that they become electrically charged by the collection of plasma species and may levitate in the space charge region—often called the plasma sheath—which is self-induced by the plasma upon contact with a surface. Surfaces immersed in a plasma are usually charged negatively due to the higher mobility of electrons compared to that of the ions. This effect creates a region where electrons are repelled in the vicinity of the surface. Hence, a thin layer, with a thickness of typically 1 to 10 mm for low temperature and low pressure plasmas, is created in which the combination of depleted electrons and (streaming) ions leads to the buildup of a space charge,

which, in turn, leads to high, local, and time-averaged electric fields and a positive plasma potential compared to the plasma-facing surface potential.

Despite the importance of the presence of the plasma sheath for many industrial applications, such as those in the solar cell industry,¹⁷ lithography,¹⁸ material processing,¹⁹ glass fiber production,²⁰ contamination control,²¹ the exploration of this region has dramatically appeared complex. Traditional probe techniques have appeared too invasive²² and optical—Stark-based—techniques^{23,24} suffer from insufficient signals due to too low electric fields present in the sheath region.

To experimentally explore the sheath region of such low pressure plasmas, the aforementioned microparticles have been utilized as (almost) noninvasive probes in several studies.^{25–30} From a basic point of view, a microparticle will—after being injected into and charged by the plasma—levitate at the exact position in the sheath region where the sum of all (plasma-induced) forces working on it is zero. With information about the microparticle charge, one could learn more about the relevant fundamental plasma–particle interactions and the different (plasma-induced) forces working on the microparticle.

As becomes clear from the above, for both the analysis and interpretation of complex plasmas (and connected research efforts in general), and the interpretation of the plasma sheath probing experiments using microparticles, the particle charge is a crucial parameter to determine. However, since the particle charge is mutually intertwined with most of the fundamental plasma–particle interactions, determining that this charge is a complex task. Traditional theoretical frameworks to determine this charge—for instance, based on the Orbital Motion Limited (OML) theory²²—become less applicable in the sheath region because in this, quasi-neutrality does not hold and high electric fields, inherently present there, induce an ion flow. All these effects complicate the modeling of the particle charge and demand for experimental data to reveal new insights and to obtain additional information.

Up to now, several experimental approaches have successfully been developed and applied to this challenge. For instance, tracking the temporal evolution of the interaction between two Coulomb-colliding particles can give insight about the charge that these particles carry with them.³¹ Optical tweezers, i.e., complex setups where two counter propagating laser beams trap a single particle by laser pressure in the plasma sheath or bulk, or even without plasma present, have been developed by Schneider and Kersten.³² Tracking the damped oscillatory trajectories of microparticles toward their equilibrium confinement position upon external injection in the plasma was studied by Tomme *et al.*³³ From such trajectories, those authors obtained information regarding the local value of the microparticle's charge. Another trajectory based technique used an ion source to alter the vertical trajectories of injected hollow microspheres to investigate the influence of fast neutrals in the ion beam on the microparticles.³⁴ Maybe the mostly used and most elegant way of determining the charge of plasma-confined microparticles is the resonance method, which was originally introduced by Melzer *et al.*³⁵ and later modified by Homann *et al.* to investigate only one single microparticle in a crystal lattice.³⁶ In the resonance method, a single plasma-confined particle is excited—for instance, by amplitude-modulating the driving radio frequency (RF) voltage—after which the response of the microparticle's motion to a frequency sweep (typically between 1 and 100 Hz) reveals the “eigen-frequency” or undamped resonance frequency. This resonance frequency can then be related to the particle's charge once

local plasma parameters are known, for instance, from modeling efforts. Zafiu *et al.* demonstrated that non-linear effects become significant for larger particle excitation amplitudes.³⁷ Furthermore, a phase resolved resonance method for accurate fitting of both the real and imaginary parts of the resonance curve was introduced by Carstensen *et al.*³⁸

Despite being useful and elegant, these resonance-based methods require a full frequency sweep with a sufficient spectral resolution in the spectral domain around the (expected) resonance frequency. From an experimental point of view, this takes considerable time to conduct, of course, depending on the desired resolution and measurement accuracy. Taking into account that most experiments in the literature use Melamine-Formaldehyde (MF) microparticles, which, in general, are considerably affected by plasma-etching,^{29,39,40} the total measurement time should be as short as possible to prevent the decreasing accuracy due to a drifting change in mass, particle size and, therefore, a drift in the exact position where the particle probes the plasma sheath.

Although Tomme *et al.*³³ experimentally derived the resonance frequency for a single response in the time-domain, repeating the experiments with their particle drop technique to achieve higher accuracy would introduce additional uncertainties with respect to the measurement location, size (i.e., charge), and mass of the used microparticles. These uncertainties are introduced because each additional measurement requires a new microparticle to be injected for which the size and mass may differ with respect to the same properties of the particle used for the first experiment. Due to the spread in mass, not only a measurement error is introduced when it comes to the resonance frequency but also the exact location at which such microparticles fulfill their probing “duty.”

In this work, we introduce and propose step-wise excitation of the probing microparticle after which the particle's response is monitored in the time-domain (rather than phase resolved). Analyzing this response provides information about the resonance frequency and damping, which, in turn, could be used to obtain information about the microparticle's charge and for the modeling and interpretation of various dusty and complex plasma experiments. This method of particle excitation can be applied to an already confined microparticle and is initiated by instantaneously changing the plasma power. In contrast to phase-resolved methods, only one single step excitation experiment could provide the needed information about the resonance frequency and damping. Hence, the measurement time would significantly be shortened while the experiment can be repeated for statistical accuracy depending on the desired measurement accuracy.

By applying the approach discussed in this work, the experimenter is given the freedom to choose the optimal trade-off between the total duration of the experiment and the increased accuracy achieved by repeating the experiment with exactly the same particle over and over again.

This work is organized as follows. Section II introduces some theoretical considerations regarding the plasma particle confinement and the response of a plasma-confined microparticle against a step excitation. The experimental approach—including the experimental hardware, the measurement procedure, and the data processing—is subsequently discussed in Sec. III. In Sec. IV, the primary results are shown after which a comparison is made with results obtained with the traditional resonance method (Sec. V). Section VI concludes this work.

II. THEORY

As explained in Sec. I, the proposed method is based on the response of a single microparticle confined in the sheath region of a low pressure plasma after being excited by a step-wise change in the plasma power.

In this section, we first briefly summarize the mechanism behind microparticle charging in low pressure plasmas (Sec. II A) and the dominantly involved (plasma-induced) forces working on a microparticle leading to a stable equilibrium position in the plasma sheath (Sec. II B). A qualitative summary of the relevant forces is briefly given in Sec. II C. How the (local) particle resonance frequency is determined from the particle's response to the applied step-wise excitation is discussed from a theoretical perspective in Sec. II D.

A. Charging of plasma-immersed microparticles

Upon injection into a low pressure radio frequency (RF) driven discharge, a microparticle usually acquires a permanent negative surface charge Q_p by collecting incoming plasma fluxes. Because the plasma is generated by an oscillating radio frequency (RF) electric field of 13.56 MHz, the electrons usually have a temperature of a few electron volts¹⁹ (i.e., a multiple of 11.6×10^3 K) while the ions remain at room temperature. Initially, the highly mobile electrons collide with the injected particle more often than the ions do. As electrons stick to the surface of the microparticle, this process negatively charges the particle surface. The developing negative surface potential of the microparticle enhances the ion flux and suppresses additional electron fluxes toward the particle until a surface equilibrium potential (and related surface equilibrium charge) is reached at which the electron and ion fluxes equalize. This theoretical framework for determining the charge of a plasma-embedded microparticle—based on equalizing the electron and ion fluxes toward its surface—is called the Orbital Motion Limited (OML) theory²² and is applied throughout the field. In the quasi-neutral plasma bulk, plasma-embedded microparticles are predicted to obtain negative charges up to 10^4 to 10^5 elementary charges depending on parameters, such as the particle size and the temperature of the electrons.⁴¹ In most dusty and complex plasma experiments on Earth, gravity pulls the microparticle toward the lower boundaries of the plasma, i.e., in the case of this work toward the plasma sheath region just above the lower (horizontally aligned) electrode (see Fig. 1). In this region, the microparticle is embedded in a plasma region with a (time-averaged) depleted electron population. Encumbered by their inertia, the relatively heavy ions cannot follow the applied frequency of the electric field and, thus, are only subjected to the time-averaged sheath electric field induced by the plasma itself. This time-averaged electric field accelerates the ions toward the plasma-facing electrode. Looking back from the microparticle's point of view, these two phenomena—the depletion of the electron population and the sheath-electric-field-induced streaming of the ions—lead to the values of the microparticle equilibrium charge, which are up to two orders of magnitude less negative, compared to particle charges obtained in the quasi-neutral plasma bulk region.⁴² Additionally, even in weakly ionized plasmas as used here, ion-neutral collisions with the particle surface can alter the final value of the particle charge, making it less negative.⁴³

FIG. 1. Overview of force vectors and the driving phenomena, working on the particle probe confined in the plasma sheath.

B. Dominant forces acting on plasma-immersed microparticles

In this section, we briefly discuss the dominant forces acting on a single microparticle that is confined in the sheath region of a low pressure plasma. The ecosystem of a charged microparticle confined above a horizontally oriented (with respect to gravity) electrode in the plasma sheath region (as is the case in this work), and the forces working on the particle are schematically shown in Fig. 1.

1. Electrostatic force

As discussed, high and time-averaged electric fields $\vec{E}$ (up to 10^4 to 10^5 V m⁻¹)⁴² are present in the plasma sheath region, which points from the plasma in the direction of the plasma-facing surface. This electric field acts on the negative particle charge Q_p , which results in an electrostatic force

$$\vec{F}_E = Q_p \cdot \vec{E}, \quad (1)$$

which is directed away from the surface (see Fig. 1). Since both Q_p and $\vec{E}$ are functions of the particle confinement position, the electrostatic force $\vec{F}_E$ is also position dependent.

2. Ion drag force

The sheath electric field $\vec{E}$ leads to a streaming flux of positive ions from the plasma toward the plasma-facing surface, which can exert momentum—and, thus, a force which is often called the ion drag force—to the microparticle either by direct impact or through Coulomb interactions.

The direct impact, or the collisional part, of the ion drag force, can be expressed as

$$\vec{F}_i^{\text{Coll}} = n_i m_i u_i \vec{u}_i \pi r_p^2 \left[1 - \frac{2e\phi(r_p)}{m_i u_i^2} \right], \quad (2)$$

where n_i is the local ion density, m_i is the ion mass, $\vec{u}_i$ is the local ion velocity, u_i is its magnitude, e is the elementary charge, r_p is the microparticle's radius, and ϕ is its equilibrium potential at the confinement position.⁴¹

The Coulomb scatter part of the ion drag force is due to the streaming ions, which closely pass by the charged microparticle and Coulomb-interact with it, but do not hit the microparticle's surface. As theoretically each passing ion is interacting with the microparticle, the cylindrically symmetric ion flow will exert a net ion drag force on the microparticle, which is generally pointing from the plasma toward the electrode surface. This part of the ion drag force is commonly expressed as⁴⁴

$$\vec{F}_i^{\text{Coul}} = 4m_i v_{i,\text{tot}} n_i \vec{u}_i \pi b_{\pi/2}^2 \ln \left(\frac{\lambda_s + b_{\pi/2}^2}{r_p + b_{\pi/2}^2} \right), \quad (3)$$

where $v_{i,\text{tot}}$ is the total ion speed, which is the vector sum of the ion speed plus the thermal speed,⁴⁵ and λ_s is the linearized Debye length given by $\lambda_s = \frac{\lambda_{D,e} \lambda_{D,i}}{\sqrt{\lambda_{D,e}^2 + \lambda_{D,i}^2}}$, in which $b_{\pi/2}$ is the asymptotic impact parameter of which the asymptotic orbit angle is $\pi/2$ ⁴⁶ and $\lambda_{D,e/i} = \sqrt{\frac{\epsilon_0 T_{e,i}}{en_{e,i}}}$ is the electron/ion Debye length, respectively, where $T_{e,i}$ is the electron and ion temperature, n_e is the electron density, and ϵ_0 is the permittivity of vacuum. The resulting vector sum of both sub-forces is referred to as the total ion drag force

$$\vec{F}_i = \vec{F}_i^{\text{Coll}} + \vec{F}_i^{\text{Coul}}, \quad (4)$$

which depends—as well—on the confinement position of the microparticle.

3. Gravitational force

The gravitational force only depends on the microparticle's mass and the Earth's gravitational field $\vec{g}$ and can be expressed by

$$\vec{F}_g = 4/3 \pi r_p^3 \rho \vec{g}, \quad (5)$$

where ρ is the microparticle's mass density. In the case of the currently presented experiments with a microparticle levitated above a horizontally aligned electrode (see Fig. 1), the gravitational force vector is pointing toward the electrode, away from the plasma.

4. Other non-dominant forces

Next to the above-mentioned dominant forces, there are several other forces known that may work on plasma-confined particles. These are—in the current experiment—non-dominant and include the thermophoretic force induced by thermal gradients in the system, the neutral drag force caused by momentum transfer from a neutral gas flow, and the radiation pressure force caused by the irradiating laser. Since no significant thermal gradients are present, gas flows are minimal near the particle's position and the used laser power is also low, these forces can be considered negligible with respect to the above-mentioned electric force, ion drag force, and gravitational force.

C. Resulting microparticle confinement

A qualitative overview of all previously mentioned forces acting on the probing microparticle is schematically shown in Fig. 1. This schematic view is made of the focus plane of the used high-speed camera as will be discussed in Sec. II D.

Under certain conditions, an injected microparticle may find a stable vertical confinement position at which the sum of all previously mentioned forces—for the case of this work usually working in the vertical direction—is exactly zero. It is well known from the literature that in plasma sheath regions, microparticles can be confined for hours and even longer.²⁹

Although horizontal components of forces—such as the horizontal force exerted by the irradiation laser and small horizontal components of the gravitational force caused by a slight misalignment of the electrode with respect to the horizontal plane—are negligible with respect to the earlier mentioned dominant forces, they may result in loss of particles from the camera focus area in the horizontal direction. To prevent this, horizontal confinement is achieved by a confinement well, i.e., a 1.0 mm deep indent milled in the electrode's flat surface (elaborated on in Sec. III A). Since the space charge region slightly follows this indent, the particle is forced to be horizontally confined above the center of this indent.

D. Particle's response after step excitation

From an experimental point of view, in our proposed method, a single microparticle is first (e.g., for $t < 0$) stably confined at its equilibrium position under steady-state plasma conditions. This means that under these initial plasma conditions, and at an equilibrium position of the microparticle, the sum of all plasma-induced forces working on the microparticle is zero.

At a certain moment in time (e.g., at $t = 0$), the RF plasma driving voltage V_{rf} is instantaneously changed by a fraction ΔV_{rf} and, with that, also the plasma power is suddenly changed. Driven by this step-wise change in the plasma power, the plasma sheath structure suddenly changes as well and, as a result, the position at which the microparticle would experience a zero net force—i.e., the microparticle's equilibrium position—is instantaneously redefined at another position.

Caused by the relatively large inertia of the microparticle compared to the electrons and ions in the plasma, the overall response of the plasma to this sudden change—which occurs at time scales on the order of 100 μs (Ref. 47)—is much faster than the response of the microparticle itself to the changing environment it is embedded in. Hence, this yields decoupling of the plasma and microparticle dynamics time scales. In contrast, in the aforementioned traditional phase-resolved particle resonance methods, the microparticle is excited by amplitude modulating the plasma with a frequency comparable to the eigen-frequency of the microparticle. Consequently, this may lead to coupling of the plasma and microparticle dynamics time scales.

Now, for $t \gg 100 \mu\text{s}$, the microparticle starts to move toward its new equilibrium position in a plasma environment, which has been changed with respect to $t < 0$, but can be considered steady-state. The microparticle will display a damped harmonic oscillating motion toward this new equilibrium position. By carefully analyzing the microparticle's trajectory and by comparing and fitting it with the theory, both the local resonance frequency and the damping ratio can be retrieved as explained below.

Assuming a linear restoring force, after excitation by the step in RF driving voltage and RF power, the following equation of motion applies to the confined microparticle:

$$m_p \cdot \ddot{z} + 2\gamma m_p \cdot \dot{z} + m_p \omega_0^2 \cdot z = \begin{cases} \pm \alpha & t \leq 0 \\ 0 & t > 0 \end{cases}, \quad (\text{N}) \quad (6)$$

where $z(t)$ is being the time-dependent microparticle position above the electrode, $\dot{z}$ and $\ddot{z}$ are the first and second order derivatives of z with respect to time t , respectively, m_p is the microparticle's mass, $\gamma = \zeta \cdot \omega_0$, which is the product between the damping ratio ζ and the natural/undamped angular resonance frequency ω_0 of the probing microparticle. α is the perturbation of force (due to the difference in RF driving power) to have the microparticle confined at the pre-step equilibrium position. The solution of the equation of motion of the microparticle after the step-excitation toward its equilibrium position (which is to be probed) is given by⁴⁸

$$z(t) = D \cdot e^{-t/\tau} \cdot \cos(\omega_d \cdot t + \phi_s) + z_0 \text{ (10}^{-3} \text{ m)}, \quad (7)$$

in which D is the arbitrary initial amplitude of the response, ω_d is the damped angular resonance frequency, τ is the decay time constant of the oscillation amplitude, z_0 is the new or post-step probing microparticle's equilibrium position, and ϕ_s is the starting phase at $t = 0$, which is defined as

$$\phi_s = \begin{cases} \pi/2 & \text{"falling"} \quad (\alpha > 0) \quad (\text{rad}) \\ 3\pi/2 & \text{"rising"} \quad (\alpha < 0) \quad (\text{rad}). \end{cases} \quad (8)$$

The damped angular resonance frequency is related to the natural angular frequency by³³

$$\omega_d = \omega_0 \sqrt{1 - \zeta^2} \text{ (rad s}^{-1}\text{)}. \quad (9)$$

The decay time constant τ in the exponent of Eq. (7) is related to the damping ratio by

$$\tau = \frac{1}{2\pi\zeta \cdot f_0} \text{ (s)}, \quad (10)$$

where f_0 is the natural frequency of the microparticle. Rewriting the above relation for ζ , substitution of $\omega_{d,0} = 2\pi \cdot f_{d,0}$ and some algebra, Eq. (9) can be solved for the natural resonance frequency f_0 as

$$f_0 = \sqrt{f_d^2 + \left(\frac{1}{2\pi\tau}\right)^2} \text{ (s}^{-1}\text{)}. \quad (11)$$

From fitting Eq. (7) to the experimentally obtained data (i.e., the time-resolved particle trajectory after step excitation), the time decay constant τ and the damped resonance frequency f_d can be found, which, in turn, delivers the natural frequency f_0 by using Eq. (11). From this framework, no difference in the solution given by Eq. (7) is expected between situations for which α is positive and situations for which α is negative.

III. EXPERIMENTAL

All experiments presented in this work have been performed on one and the same single microparticle levitated in the sheath region of a parallel plate, capacitively coupled low pressure argon plasma.

In this section, the overall experimental setup will be described in Sec. III A, followed by a discussion of the procedure that was followed to obtain the proof-of-principle measurements in Sec. III B. Finally,

the post-processing of the data from the high speed camera system is briefly discussed in Sec. III C.

A. Experimental setup

A schematic overview of the overall setup used for the experiments—including a vacuum vessel, particle injection, illumination and tracking equipment, and gas handling—is depicted in Fig. 2. Centered inside this vacuum vessel is the electrode configuration, which is depicted in greater detail in Fig. 3.

The used vacuum vessel shown in Fig. 2 had a volume of ~ 4 L and contained three ISO K-100 and two ISO K-160 ports. In the vacuum vessel, the background pressure was $< 1 \times 10^{-4}$ mbar, which was checked before the measurements using a Hornet IGM400. During the experiments, the operating argon gas pressure in which the plasma was created was 16.2 ± 1.6 Pa as measured using a CVG101 Worker Bee [Fig. 2(b)] pressure gauge.

The inlet of gas was controlled by a Brooks GF40 mass flow controller with full scale flow capacity of 10 SCCM. This gas left the vessel through a stationary HiPace 80 turbo pump [Fig. 2(d)], which was directly connected to the vacuum vessel. Down the vacuum line, two vacuum valves in succession choked the exhaust gas flow toward an operating HiPace 80. This turbo molecular pump was backed by a Leybold Scrollvac 10.

The electrode assembly [Fig. 2(g)] was mounted on the bottom flange of the 18 cm cube vacuum vessel of black anodized aluminum as shown in Fig. 2.

The plasma discharge was generated between two, a 5.0 mm thick, 70×70 mm² 316L stainless steel electrodes with rounded edges, which were aligned in the horizontal plane, and they were separated by a 40 mm distance from one another (see Fig. 3). The top electrode [Fig. 3(a)] was grounded via the wire frame [Fig. 3(f)], which held both electrodes in place. The 15 mm hole in the middle of this top electrode [Fig. 3(b)] granted from the top injected microparticles access to the discharge region, to have them finally reaching the sheath region of interest just above the bottom electrode. The bottom electrode

FIG. 2. Complete vacuum vessel, with the (for visual purposes) cut surfaces shown in red: (a) high speed camera and optics, (b) pressure gauge, (c) in-house developed particle injector, (d) stationary turbo molecular pump, (e) laser assembly, (f) laser beam, and (g) electrode assembly.

FIG. 3. Close-up of the RF electrodes, with: (a) grounded electrode, (b) the 15 mm hole, (c) powered electrode, (d) exchangeable inset, (e) particle probe, (f) wire frame, and (g) PEEK insulators.

[Fig. 3(c)] was powered with a radio frequency (RF) voltage signal and was electrically insulated from the wire frame by customized PEEK insulating parts [Fig. 3(g)]. A 1 mm deep, $5 \times 10 \text{ mm}^2$ slot-shaped indent was milled in an exchangeable insert [Fig. 3(d)], which was fixed in the powered bottom electrode to create a potential well to ensure that the probing microparticle remained just above the center of the bottom electrode [Fig. 3(e)], i.e., in the limited depth of field of the optical system.

The plasma power was generated by using two function generators in succession: the first function generator's output of -5 to 5 V was fed to the modulation input of the second function generator. The second function generator provided a constant 13.56 MHz carrier signal, which was amplitude-modulated by a set percentage by the output of the first function generator. This modulated RF signal was sent to a Mini Circuits LZY-22+ amplifier, which, via a MFJ Versa Tuner II (model: MFJ-969), applied the RF voltage on the bottom electrode of Fig. 3. The plasma power was determined by an Octiv Poly VI probe, which was installed in the RF line prior to the measurements.

The used microparticle was made from SiO_2 (which are pristine argon plasma resistant^{39,49}) and had a diameter of $4.96 \pm 0.20 \text{ }\mu\text{m}$, with a manufacturer (Microparticles GmbH) reported mass density of 1850 kg m^{-3} . The microparticle was injected in the same way as was done by Beckers *et al.*^{29,42,50,51} before. This was done by a “salt” shaker like device [Fig. 2(c)], which had two $\approx 40 \text{ }\mu\text{m}$ holes in its bottom plate. To activate the shaker, a brief pulse of 24 V was put on its atmospheric coil, which suddenly attracted the particle container inside the vacuum, to be returned to its initial position by a spring after the pulse.

The laser beam [Fig. 2(f)] used to illuminate the microparticle was generated by a 450 nm laser diode (Thorlabs L450P1600 MM), which was compressed in the direction of the perpendicular axis ($\theta_\perp$) by a cylindrical lens with $f = 24.88 \pm 0.25 \text{ mm}$, with the laser diode situated at the focal line of the lens [Fig. 2(e)]. The CW laser beam

power at the position of the particle was roughly 5 kW m^{-2} . The laser beam was finally dumped on the anodized blind side of the vacuum vessel.

The camera used for imaging was a monochrome, 12-bit Phantom Miro C110 [Fig. 2(a)]. This camera was fitted with a band-pass filter (center wavelength of 450 nm and a FWHM of 10 nm) and “machine vision” optics, Thorlabs MVL50TM23, with a fixed focal length of $f = 50 \text{ mm}$. The resulting spatial resolution at the position of the probing microparticle was $16.5 \text{ }\mu\text{m/pixel}$.

B. Measurement procedure

Before the start of an experiment, the vacuum vessel as shown in Fig. 2 was pumped down to the base pressure and an argon gas flow of $\approx 0.6 \text{ SCCM}$ was introduced in the vessel while the pressure in the vessel was allowed to stabilize at an operating pressure of $16.2 \pm 1.6 \text{ Pa}$. Then, the plasma was switched on using an RF power of $3.2 \pm 0.3 \text{ W}$ after which the system was allowed to thermally stabilize for roughly 3 h . After these 3 h of plasma-on time, several microparticles were injected into the plasma vessel by using the particle injector. By tilting the relatively small vacuum vessel, more and more microparticles were allowed to escape from the confining horizontal potential well above the powered electrode until a single particle remained trapped. This remaining single microparticle was the particle with which all experiments presented in this manuscript have been conducted.

The absolute value of the applied step in RF power was chosen such that the microparticle would not move too quickly for the camera to resolve it (raw data shown in Fig. 4), which was smaller than 15 mm s^{-1} in our most extreme case. However, the step in power was chosen sufficiently large to be able to resolve several periodic oscillations of the microparticle toward its new equilibrium position after excitation. This is more extensively discussed in Sec. V B.

The post-step plasma power used to achieve a suitable microparticle equilibrium position z_0 was $3.2 \pm 0.3 \text{ W}$. For the measurements in which the plasma power was suddenly *increased* to this value—in this work these experiments are indicated as *rising step excitation*—the pre-step plasma power was set to $1.5 \pm 0.2 \text{ W}$. The corresponding pre-step equilibrium position of the microparticle is denoted by z_1 . Similarly, for the measurements in which the plasma power was suddenly *decreased* to the value of $3.2 \pm 0.3 \text{ W}$ —in this work these experiments are indicated as *falling step excitation*—the pre-step plasma power was set to $6.6 \pm 0.7 \text{ W}$. The corresponding pre-step equilibrium position of the microparticle is denoted by z_h .

Statistical information has been obtained by using a modulation signal, which was a 2 Hz square wave with a duty cycle of 50% . The repetition rate was sufficiently low to ensure fully restored and stable plasma confinement of the involved microparticle. The high speed camera was triggered on the rising/falling edge of the modulation voltage, until the memory section ($\approx 183 \text{ s}$) of the camera was saturated. The set high speed camera settings were as follows: $180 \text{ Frames Per Second (FPS)}$, with an exposure of 5.55 ms per frame at the maximum exposure index of $25\,000$.

C. Data processing

The amount of data is drastically reduced by taking two pictures of the stably levitating microparticle probe, with different camera frame sizes. In the first image, the microparticle and the electrode were

FIG. 4. The SiO_2 microparticle's response when excited from an instantaneous lower (left) and higher (right) plasma power, together with fits of Eq. (7) and the equilibrium positions before (z_1 and z_h for rising and falling step excitation) and after (z_0) the step excitation. The applied RF power is depicted in red.

photographed in one frame. The second image was taken with a smaller frame size, which only included the microparticle. Because the microparticle probe was verified to be at a stationary location, the two pictures were related to one another and, from that, the height of the exact position of the microparticle could be determined without having to record the electrode in each frame. By this referencing between the overall and small photographs, almost a 30-fold data reduction per frame was achieved. The additional error in the particle position made by this data-saving approach is assumed to be less than 1 pixel or $16.5 \mu\text{m}$. The nature of this error is a systematic one and is, therefore, not shown in the results in this work.

The referenced images were processed by the MATLAB software developed by van Minderhout *et al.*,²¹ after being adapted to the tracking of a single particle. For each consecutive video frame, this software enabled to resolve, with sub-pixel resolution, the particle position despite the resolution of the optical system used. The resulting particle locations were fitted with Eq. (7) in the time-domain by using the “cfit” functionality of the MATLAB software. Fitting of the particle locations was done by omitting the first few data points of the damped oscillation, as Eq. (7) only holds for the oscillatory part of the motion. Hence, the acceleration part of the movement is omitted, which does not affect the found resonance frequency and damping time constant. The parameters that were fitted by the MATLAB software on Eq. (7) were as follows: the amplitude of the oscillatory motion D , the resonance frequency f_d , the damping time constant τ , the offset z_0 , the initial phase ϕ_s , and an offset in time t . Statistical information of the total data set has been obtained by using the “mean” and the “std” basic functions provided in MATLAB.

IV. PROOF-OF-PRINCIPLE EXPERIMENTS

The feasibility of the proposed method to determine the resonance frequency and damping ratio of a vertically excited

microparticle confined in the sheath of a low pressure plasma operated above a horizontal surface is demonstrated in Sec. IV A. The accuracy of this method as a function of the number of experimental repetitions (with exactly the same particle) is discussed in Sec. IV B. Finally, the results are compared with the established resonance method technique in Sec. IV C.

A. Proof-of-concept

Exemplary experiments of one single probing microparticle subjected to a *rising step excitation* (i.e., an instantaneous increase in plasma power from 1.5 ± 0.2 to 3.2 ± 0.3 W) and a *falling step excitation* (i.e., an instantaneous decrease in plasma power from 6.6 ± 0.7 W to 3.2 ± 0.3 W) were conducted. The recorded vertical particle position data as a function of time after these excitations are shown in Fig. 4.

As can be seen from this figure, while having different initial ($t < 0$) equilibrium positions, z_1 is on average 3.519 ± 0.003 mm and z_h is on average 3.654 ± 0.018 mm for the respective plasma powers of 1.5 ± 0.2 W and 6.6 ± 0.7 W. Nevertheless, the particle is found to oscillate eventually toward the same (on average) equilibrium position $z_0 = 3.555$ mm (the lack of error estimation is explained in Sec. V C) for both the rising and the falling step excitation experiments. Also included in Fig. 4 are fits of Eq. (7) through the experimental data points. These fits clearly demonstrate that—already from a single step excitation—the resonance frequency and the damping ratio of the system can immediately be obtained.

Zooming in on the raw data plot of the falling step excitation experiment shows that there is a missing data point around $t \approx 0.015$. This is caused by the lack of optical signal from the particle due to a too high velocity of the probing microparticle in the captured frames (discussed further in Sec. V B). Hence, such data points are not taken into account when fitting the raw data with Eq. (7).

B. Determination of the microparticle's resonance frequency and damping ratio

To investigate the validity and the accuracy of the proposed experimental method and the influence of experiment repetition on it, both the *rising step excitation* and the *falling step excitation* experiments presented in Fig. 4 have been repeated 365 times. The raw data of each individual step response has been fitted with Eq. (7), and from the fitting parameters, the decay time constant τ and the damped resonance frequency f_d have been obtained. Statistics have been applied to the obtained parameters, from which the standard deviations were estimated. Figures 5 and 6 show, for the rising and for the falling step excitation experiments, the mean values of these parameters together with the standard error on these “means” as a function of the amount of experiment repetitions. Although structural drifts in process parameters (e.g., thermal drifts, see Sec. V C) were insignificant, the experiments making up the subsets used to construct Figs. 5 and 6 have been randomly chosen from the overall set of experiments, of course, while distinguishing between rising and falling step excitation.

The two graphs immediately show the flexibility that this method provides for the experimenter. If measurements need to be done quickly—for instance, in cases where plasma etching of the used microparticles is significant—a few measurements will give a rough estimate of the resonance frequency and damping time constant already. If the experimental situation allows to perform more successive measurements, the accuracy of the overall experiment increases.

From the full sets of experiments (i.e., 365 repetitions for each type of experiment), the values of the means and standard deviations converge to values that are shown in Table I.

From the resulting parameters in this table, it can be concluded that—although relatively close—both the damped resonance frequency f_d and the corresponding standard deviation σ_d for the rising step excitation experiment are slightly higher than those for the falling step excitation experiment. The same trend was found for the values of the decay time τ and its standard deviation σ_τ , where it should be noted that the spread in the obtained time constant for the rising step

FIG. 5. Standard error on the mean of the fitted decay time constant as a function of the number of repetitions.

FIG. 6. Standard error on the mean of the fitted resonance frequency as a function of the number of repetitions.

excitation experiment is significantly larger than the spread in the obtained time constant for the falling step excitation experiment. This larger spread is most likely caused by the relatively smaller initial amplitude of the damped oscillation for rising step excitation experiment.

The values of the obtained resonance frequency and the damping ratio together with their standard deviations are used to calculate the natural frequency f_0 by substitution in Eq. (11) (see Table I). The damping ratio ζ displayed in the same table is calculated from rewriting Eq. (10) and the substitution of the natural frequency and the decay time.

It can be seen from the obtained mean ζ that the damping ratio of the falling step excitation experiment is 5% larger compared to the rising step excitation experiment. However, this difference is relatively small when comparing the found damping ratios with the calculated uncertainties.

C. Comparison with the linear sweep method

For benchmarking and comparison, a frequency sweep experiment, as done in the work of Melzer *et al.*,³⁵ has been conducted with exactly the same microparticle and under identical plasma conditions during one and the same experimental run. To this end, the first function generator (elaborated on in Sec. III A) was programmed to generate a 0.01 to 60.01 Hz linear increasing frequency sweep at a rate of 0.25 Hz s⁻¹ during a total experiment duration of 240 s. (Note that one single step excitation experiment can be conducted on sub-second time scales.) The second function generator modulated—with the

TABLE I. Fitted and calculated parameters for the step-wise excitation experiments.

	f_d (Hz)	σ_d (Hz)	τ (ms)	σ_τ (ms)	f_0 (Hz)	ζ
Rising	25.6	0.51	45.7	7.32	25.9 ± 0.6	0.140 ± 0.024
Falling	24.9	0.31	43.1	2.44	25.2 ± 0.3	0.147 ± 0.010

FIG. 7. Raw data of the linear frequency sweep, with two fits through the particle's maxima and minima positions. A discrepancy of 0.52 Hz is found between the fitted resonance frequencies.

momentary frequency provided by the first function generator—the RF power by $\pm 12\%$. The microparticle position evolution is plotted in Fig. 7, which was tracked with the high temporal-resolution of 120 FPS during this frequency sweep. The resonant behavior of the microparticle is already clearly visible in the raw data. Table II shows the obtained resonance frequencies and damping ratio from the fits through the particle maxima and minima, i.e., f_{res} and ζ_{res} . The calculated natural frequencies of the probing microparticle are also shown.

First, it can be concluded that the mean of the vertical microparticle equilibrium position, i.e., $z_{0,\text{sweep}} = 3.56$ mm, perfectly agrees with the average of $z_0 = 3.555$ mm for both step excitation experiments within experimental error bounds (see Fig. 8 for details). This means that the proposed step-wise excitation approach is able to probe the plasma at exactly the same vertical microparticle equilibrium position as is done by traditional resonance techniques.

Second, when comparing the resonance frequency obtained from the frequency sweep measurements with those obtained by the step-wise excitation approach, the following is observed. For the frequency sweep case, we simply determine the resonance frequency f_{res} by using resonance curve fits in the time domain through the maxima and minima in the microparticle positions. As can be seen from Fig. 7, the fit through the maxima delivers a $f_{\text{res}} = 25.1$ Hz, which is somewhat higher than the $f_{\text{res}} = 24.5$ Hz obtained from the fit through the minima. Notably, this is in the same direction as the discrepancy observed between the damped resonance frequencies for the rising step excitation ($f_d = 25.6$ Hz) and the falling step excitation ($f_d = 24.9$ Hz) and

TABLE II. Fitted parameters of the top and bottom particle locations of the linear frequency sweep.

Frequency sweep (Hz/s)	f_{res} (Hz)	ζ_{res}	f_0 (Hz)
Maxima	25.05	0.122	25.43
Minima	24.53	0.115	24.85

FIG. 8. Fitted equilibrium position z_0 of both the rising and falling step excitation experiments, together with the particle size on the same scale.

may possibly originate from a slightly non-linear sheath structure. Overall, regardless of the direction of step excitation (rising or falling), it can be concluded that the obtained damped resonance frequencies are within 5% with respect to those values obtained from the traditional resonance method.

Since the damping ratios for both methods, i.e., step excitation and frequency sweep, are close together as well, it is clear that also the obtained natural resonance frequencies f_0 from both methods match within 5% accuracy.

V. ADDITIONAL DISCUSSION

In addition to the discussion of the proof-of-principle experiments and the comparison of those with the linear frequency sweep method, this section elaborates on three important topics as follows: comparing the experimentally found damping rates with the classical damping rate predicted by Epstein, optimally resolving the particle trajectories, and the (thermal) drift of the microparticle's probing location during the total measurement time.

A. Comparison of the damping rates with the theory

In this work, the obtained damping ratios can be compared to the classical damping rate defined by Epstein *et al.* The Epstein damping rate is defined as⁵² $\beta_{\text{eps}} = \delta \frac{4}{\pi} \frac{p}{\rho v_{\text{th}} r_p}$, where p is the gas pressure, v_{th} is the thermal velocity of the neutrals, and δ is a dimensionless factor that depends on whether the neutrals collide with the particle's surface by diffusive or by specular reflections (1.44 or 1, respectively) of.⁵³ When calculating the Epstein damping rate for the parameters used in this work, one obtains $\beta_{\text{eps}} = 16.2 \pm 1.7 \text{ s}^{-1}$ assuming diffuse reflections. Converting the experimentally found ζ to the Epstein damping rate via $\zeta * \omega_0 = \beta$, for the rising and falling step excitation experiments, the values of 22.8 ± 3.9 and $23.3 \pm 1.6 \text{ s}^{-1}$ are found, respectively. Both values are larger than the damping rate predicted by Epstein *et al.* Calculating the same damping rates for the results of the linear frequency sweep, the maxima and minima of the results in

Table II give damping rates of 19.49 and 17.96 s⁻¹, respectively. Hence, it is clear that the experimentally found values for the damping rate deviate more in the case of the step excitation method (compared to the frequency sweep method). This might be caused by the relatively large steps in plasma power, used to excite the probing particle to a sufficient extent. Pustynnik *et al.* have reported that the classic Epstein damping rate should be corrected by the so-called Delayed Charging Effect,⁵² which is dependent on the plasma parameters and is defined as $\beta_{DCE} = \frac{\tau_{ch}}{2} \frac{eE}{m_p} \frac{\partial Q_p}{\partial z} \big|_{z=z_0}$, where τ_{ch} is the charging time. According to Pustynnik *et al.*, in such cases the damping rates can be summed to a total damping rate as $\beta = \beta_{eps} + \beta_{DCE}$. In conclusion, this means that the step excitation method introduced here is more influenced by the Delayed Charging Effect than the linear frequency sweep method is.

B. Resolving the particle trajectories

For optimally resolving the microparticle's damped oscillatory trajectory, there is always a trade-off between, on one side, a sufficiently large oscillation amplitude in order to achieve sufficient spatial resolution and, on the other side, a sufficiently low velocity of the microparticle to have sufficient optical signal to detect the particle at all times.

In the view of achieving sufficiently large oscillation amplitudes, the applied step excitation method needs—in general—more power modulation ($\approx 50\%$ and $\approx 100\%$ for the rising and falling probe experiments, respectively) than the linear frequency sweep method requires (in the case of this work $\approx 12\%$). However, as discussed before, due to the much shorter plasma dynamics time scales compared to the microparticle dynamics time scales, these time scales can be considered fully decoupled in the case of step excitation, while this may not be the case for the linear frequency sweep method.

Furthermore, it is not surprising that the standard deviations on the mean values of f_d and τ for the rising step excitation experiments (having a relatively small initial oscillation amplitude) are significantly higher than those for the falling step excitation experiments (having a relatively large initial oscillation amplitude). At the same time, just after the step excitation in the falling probe case, the microparticle's velocity is too high such that the optical system and post-processing fail to detect the first few data points, as can be seen in Fig. 4.

Overall, as the optical irradiation and detection systems seem to be the limiting factor in resolving the probing microparticle, a more powerful/focused laser beam enables faster and less noisy camera images. Here, the experimenter must be aware of the risk of additional and unwanted effects, such as higher radiation pressure, increased particle heating, light scattering by the vacuum vessel etcetera. Additionally, a more sensitive/intensified camera can be used to enhance the measurements' signal-to-noise ratio.

C. (Thermal) drift of the microparticle's probing location

With the step excitation with a 50% duty cycle, the time averaged plasma power for the rising step excitation (from 1.5 ± 0.2 W to 3.2 ± 0.3 W) is 2.4 W. This is much lower than the plasma power used in the falling step excitation experiments (from 6.6 ± 0.7 to 3.2 ± 0.3 W), being 4.9 W on average. Assuming that a significant fraction of this power is deposited on the bottom (RF powered) electrode of this asymmetric discharge, it can be assumed that the thermal

load on the bottom electrode during the falling step excitation experiments is higher than that during the rising step excitation experiments. To investigate the possible disturbing effect of an additional upward thermophoretic force on the overall equilibrium position of the particle, the microparticle's equilibrium position (z_0) as a function of consecutive step excitation experiments is plotted in Fig. 8 for both the rising and falling step excitation experiments.

As can be seen from this figure, a drift in particle equilibrium position of the microparticle is indeed observed. This drift is—as described above—upwards for consecutive falling step excitation experiments and downwards for consecutive rising step excitation experiments. However, this drift is astonishingly small with a maximum drift in either direction of only 14 μm for a measurement duration of 183 s. For comparison, the size of the used microparticle is plotted in the figure as well, highlighting not only the high spatial resolution of the used optical system and post-processing, but more importantly also the stability of the microparticle's probing position.

This effect can be fully mitigated by allowing the setup to reach thermal equilibrium with the same average plasma power used during the step excitation waveform. For example, if the experimenter wants to perform the rising step excitation experiment with a duty cycle of the excitation wave of 50%^o and power steps from 1.5 to 3.2 W, the constant (no modulation) plasma setting for reaching the thermal equilibrium should be set at 2.35 W. Of course, the same approach works for reaching the required thermal equilibrium with respect to the falling step excitation experiment.

VI. CONCLUSION

We have introduced a new step-wise excitation approach to quickly obtain, or with high accuracy, the resonance frequency of a microparticle confined in the space charge region of a low pressure plasma operated above a horizontal surface. This step-wise excitation was achieved by suddenly changing the plasma power, which instantaneously altered the equilibrium position of the confined microparticle as well. By resolving the trajectory of the microparticle toward its new equilibrium position and comparing this trajectory with the theory describing the damped harmonic oscillator, the local value of the resonance frequency can be retrieved.

Such a resonance frequency, together with the obtained damping ratio, can be used to calculate the natural frequency of the particle probe. This, in turn, is essential to obtain for the interpretation of many dusty and complex plasma laboratory experiments, because it can directly be related to the particle's charge.

The step-wise excitation approach was compared to the traditional frequency sweep particle resonance method and the results of both methods match one another within 5%.

The found damping rates are compared to the classical Epstein damping rate, where the results from the step excitation experiments show a larger discrepancy from the classic damping rate, than the results from the linear frequency sweep do. This is most likely caused by the Delayed Charging Effect.

With the proof-of-principle experiments presented in this work, we have demonstrated the flexibility that this method provides for the experimenter. If measurements need to be done quickly—for instance, in cases where plasma etching of the used microparticles is significant—a few measurements (with sub-second measurement time per experiment repetition) will give a rough estimate of the resonance

frequency and the damping time constant. If the experimental situation allows time to perform more successive measurements, the accuracy of the parameter determination increases.

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DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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