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Highlights

- We present the first non-destructive 4π particle scatterometer of its kind
- Controlled acoustic levitation allows measuring the sample from any angle
- Polarimetric angular maps of light scattering from a mm-sized sample are demonstrated
- The non-contact sample manipulation allows handling high-value fragile samples

Journal Pre-proof

4 π Scatterometer: A new technique for understanding the general and complete scattering properties of particulate media

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Abstract: Knowing the optical properties of a sample is important in many scientific fields, such as space science, climate studies, and medicine. In many of these applications, the samples are fragile, unique or available in limited quantities, and have to be subsequently studied by **additional** techniques, implying that sample preservation is important. Established light scattering single particle measurements involve attaching the sample to a holder or measuring a laminar flow of particles, neither of which allows a controlled, unperturbed non-destructive measurement.

Acoustic levitation is a state-of-the-art and non-contacting approach capable of assisting light scattering measurements. However, a full 4 π measurement (i.e., a measurement from any direction on the full 4 π solid angle) has hitherto been impossible due to levitation instabilities. Here we present and describe the instrument capable of performing a full 4 π light scattering measurement. We measure light scattering properties of millimeter-sized samples at any direction. This is enabled by introducing a novel non-contacting sample holder based on acoustic levitation, which allows a disturbance-free measurement of an orientation-controlled sample. The instrument is scalable and currently employs polarized visible light (400–700 nm). It also measures beam and sample stability as well as temperature and humidity, to ensure consistency of measurements.

We demonstrate the 4 π capabilities of the instrument by measuring an angular map of light scattering from a polystyrene foam sample, as well as a multi-angular measurement (two semicircular measurements 90 degrees apart) of a sample consisting of agglomerated 500 nm silica spheres. The upper left 2 \times 2 submatrix of the Mueller matrix is measured from the sample along the (polar) scattering angle in semi-circular sweeps, changing the azimuthal scattering angle for each sweep. Our results allow **for verification of** theoretical models by mimicking the conditions of the simulations and by making the measurements directly comparable to model predictions.

1. Introduction

Light scattering theory plays a major role in **terrestrial** atmospheric science, where aerosol particles directly affect our climate by interacting with incoming sunlight. Mineral aerosols are present especially in desert areas, and they are released in large quantities during volcanic eruptions. Understanding the single scattering behavior of these particles is essential for modeling their effect on light scattering in the atmosphere [1, 18].

Light scattering is also an important tool when observing cosmic dust, cometary coma, and the regolith of airless solar system bodies, such as asteroids and moons [2]. Since directly acquiring samples is often hard in these cases, the scattering and polarization of light is an invaluable tool for determining the material properties of these mineral particles.

Light scattering models for atmospheric and interplanetary dust particles are often based on Mie scattering theory, which assumes the individual scattering elements to be spherical. This is a reasonable approximation for liquid droplets, but mineral aerosols are highly irregular in shape [3]. More accurate scattering models could potentially be constructed by utilizing robust experimental data obtained for real dust particles. Computational models based on simulations exist that provide the Mueller matrix as a function of the polar and azimuthal scattering angles (Fig. 1a). Such modeling gives the relationship between scattered light and incoming light [4,5]. Unfortunately, their accuracy can only be determined by direct comparison to empirical measurements of equivalent samples.

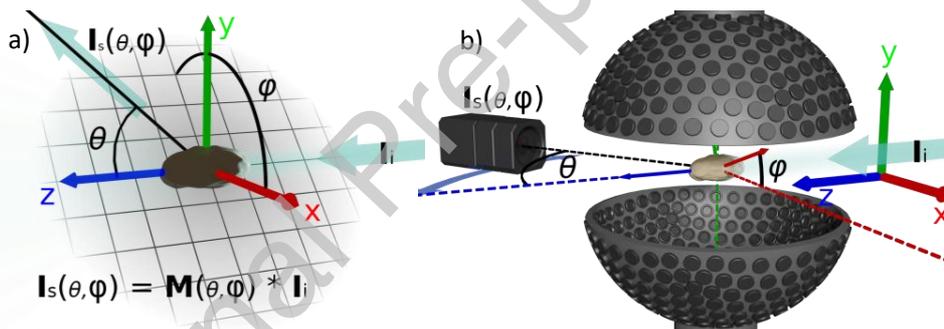


Fig. 1: a) Scattered and incident light, described by the Stokes vectors. The coordinate system is chosen such that the incident beam is aligned with the z-axis. The scattering angle is denoted by θ and the azimuthal scattering angle is denoted by ϕ . b) Angular light measurement in a levitating environment. Here the azimuthal scattering angle (ϕ) is defined by the rotation of the particle's local coordinate system around the z-axis. Incoming and scattered light intensity are denoted I_i and I_s , respectively.

Previous developments in this field, representing the current state of the art, have been made by Muñoz *et al.* [6]. Their instrument measures the averaged scattering properties from a laminar flow of small particles, as well as from static samples placed on a thin pedestal. Their light scattering database is a valuable resource that provides optical properties of various mineral dusts [7]. Alternative goniometric setups are designed for measuring light scattered by surfaces [8-11]. Due to multiple scattering interactions between the individual scatterers within closely packed medium measurements are more difficult to interpret compared to single particle [12, 13].

Here we advance the state of the art by measuring light scattering from individual particles suspended in midair from any angle (Fig. 1b). This dictates the requirements of our system. The sample must be preserved, to allow it to be measured again at different orientations, and to allow it to be characterized by other methods. The sample must remain in midair at a specific orientation. Finally, we need to be able to measure light scattering at any solid angle, since we do not average our measurement results over multiple orientations, and therefore cannot assume that our results are rotationally symmetric. Our solution to achieve these capabilities is orientation-controlled acoustic levitation. This solution provides a full 4π measurement that is also non-destructive and non-contacting. The approach allows us to make repeatable measurements of individual particles and opens the door for measuring precious samples available in limited quantities. One such example is samples gathered during space missions.

In this paper we describe, in detail, the design choices and features of the scatterometer we have developed. We outline its working principles and the measurement procedure. In the results section we demonstrate the 4π capability. Finally, we discuss the compromises that were made during the development as well as possible future improvements to this instrument.

2. System description

The scatterometer is designed to measure light scattered by a mm-sized sample, covering any solid angle, with the exception of narrow two cones around the forward- and backscattering directions. The setup is depicted in Fig. 2. The Mueller calculus that forms the matrix elements from scatterometer measurements is described in [12]. Four polarization configurations are required along the optical path. These are acquired by placing polarizers before and after the sample.

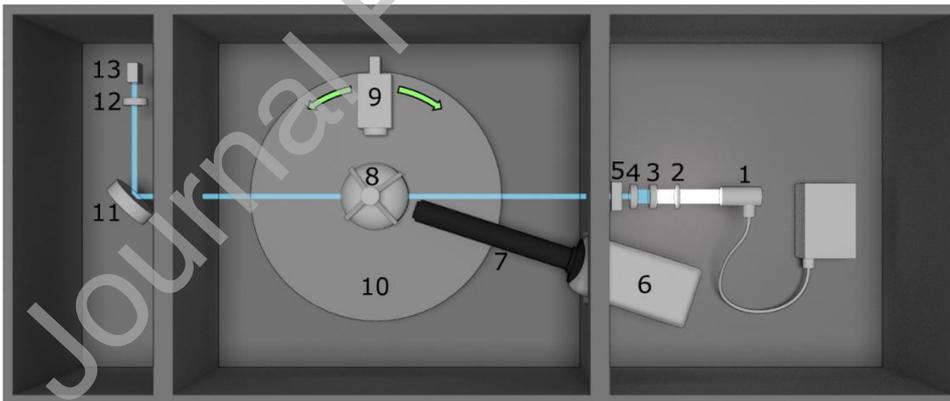


Fig. 2: Simplified top-down schematic of the instrument, featuring thirteen parts: 1. Collimator 2. Focusing lens 3. Color filter 4. Aperture 5. Motorized polarizer 6. High speed camera 7. High magnification objective 8. Acoustic levitator 9. Measurement head 10. Rotation stage 11. Optical flat 12. Neutral density filter 13. Reference PMT. The green arrows show the measurement head movement along the scattering angle.

The sample is trapped inside the levitator using ultrasound. The levitator is mounted in the center of the horizontally aligned rotation stage. The scattering angle θ (in relation to the incident beam) is scanned by moving the detector on a rotational stage, while the azimuthal scattering angle φ is controlled by rotating the sample around the beam axis. The instrument is divided into three enclosed compartments that are covered by a diffuse black velvet-like material, which prevents specular reflections from the environment and minimizes stray light.

The instrument has five major features: The light source, the sample holder, the analyzer, the sample monitoring system, and the beam intensity monitor (Fig. 3).

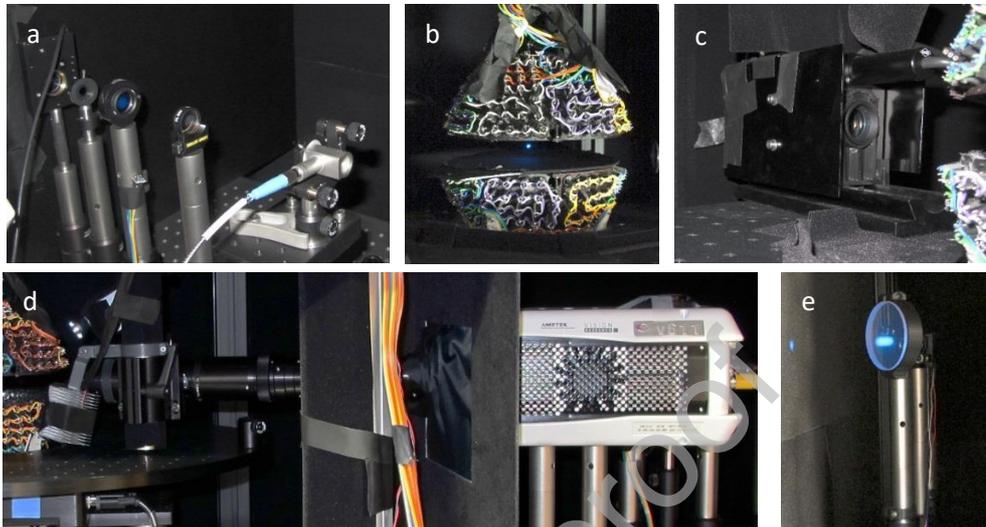


Fig. 3: Main features of the scatterometer: a) Light source (Fig. 2 numbers 1-5) b) Acoustic levitator (8) c) Analyzer (9-10) d) Sample monitoring system (6-7) e) Beam intensity monitor (11-13).

2.1 Light source

Since different samples pose different challenges, our instrument features a modular fiber-coupled beam collimator, which permits use of different light sources without having to realign the beam path. Our primary light source is an Energetiq EQ-99 Laser Driven Light Source (LDLS). It features a smooth, continuous spectrum in the entire visible range. The light is collimated by a Thorlabs RC08FC-P01 mirror collimator and a laser line filter (Thorlabs FL488-10, 488 ± 5 nm) is used to limit the frequency range for measurements. An adjustable aperture limits the beam width to prevent scattering from the edges of optics down the line. The beam is focused onto the sample by a planoconvex spherical lens ($f = 700$ mm) and polarized by a birefringent calcite polarizer (Newport 10GL08) on a motorized mount. The beam is then passed into the second chamber, where the scattering measurement is done.

For cases where high single-frequency light intensity is needed, the LDLS can be exchanged for a tunable Argon-Krypton laser (Melles Griot KAP 41, [12]), which produces a monochromatic vertically polarized beam with high coherence length. The same light source is in use in IAA, Granada [6]. This requires replacing the birefringent polarizers with film polarizers, to avoid interference fringes, as well as addition of quarter wave plates to rotate the polarization of the beam.

2.2 Sample holder

What makes our instrument unique is that the sample is held in place by an acoustic levitator featuring accurate sample orientation control. The main advantage of this system is its non-contacting nature. The sample is suspended in midair with no other structural parts interfering with the light scattering. Additionally, this allows full manipulation of fragile samples without the risk of deformation or breakage. Orientation control is achieved by digitally adjusting the shape of the acoustic field. The levitator comprises two hemispherical transducer arrays

opposing each other and creating a standing wave acoustic field in the center (Fig. 3b). The transducer elements on each hemisphere are grouped into 12 phase-controlled channels, allowing precise computer control of the field shape. The standing waves are adjusted to form an asymmetric potential well, where the largest physical dimension of the sample aligns itself with the axis of lowest gradient. While most acoustic levitators allow the sample to spin freely around one axis, this asymmetric trap design keeps the sample stable and minimizes any movement. By gradually altering the trap shape the sample can be rotated around any axis (pitch, roll, and yaw). Like with the modular light source, the levitator can be swapped out and be replaced with e.g. a conventional pedestal for samples which are too large for the acoustic levitator. A more complete description of the ultrasonic levitation system can be found in [17].

2.3 Analyzer

The analyzer (Fig. 3c) is used to obtain the specified polarization data with the scatterometer instrument. The sensor is a Hamamatsu microPMT H12403-01 photomultiplier tube (PMT) module, connected to a Thorlabs TIA60 amplifier. The PMT provides both high sensitivity and high dynamic range. It is integrated into a measurement head, featuring a motorized film polarizer (Thorlabs LPVISE100-A), a motorized aperture, which also works as a shutter, and an infrared (IR) filter that protects it against the illumination used by the sample monitoring system. Despite its low extinction ratio, a film polarizer was chosen rather than a calcite one, since it allows for a compact design. The measurement head is mounted on a circular breadboard ($\varnothing=600$ mm) which is turned by a motorized rotation stage (Standa 8MRB240-152-59D) with a resolution of $15'$. Both the breadboard and the rotation stage have a central 150 mm aperture for mounting the sample holder.

2.4 Sample monitoring system

The sample monitoring system is closely related to the acoustic levitator, in that its main purpose is to measure the position and rotation stability of the levitating sample. An additional feature arising from this is that a 3d-model of the sample surface can be reconstructed from the footage using the Structure from Motion (SfM) algorithm [14]. The sample is levitated and recorded by a Phantom v611 hi-speed camera (6242 fps at full resolution), using a microscope objective with 120 mm working distance, providing high resolution close-up images of the sample. The sample monitoring system uses its own light source to provide sufficient illumination. It consists of two 850 nm near-IR LEDs. Near-IR illumination was chosen because the hi-speed camera sensor is sensitive in that range, and because it can be filtered out from the analyzer without affecting the measurement, which is conducted in the visible range. Thus, the PMT's are protected from the sample monitoring illumination. While the sample could theoretically be monitored and measured at the same time, this approach is avoided, since the LEDs and uncovered camera lens produce artifacts in the scattered light pattern.

2.5 Beam intensity monitor

While the light source is chosen to have high stability in the intensity and other beam characteristics, we still want to ensure that any minor deviations in beam intensity are recorded and compensated for. We also need to measure the difference in intensity between the vertical and horizontal polarization of the beam, since it is challenging to create and maintain a perfectly unpolarized beam. Some existing instruments approach the problem by modulating the beam intensity [15], whereas we measure the beam directly using a PMT. The beam intensity monitor, located in the third compartment of the scatterometer, measures the beam exiting from the measurement chamber. The light is reflected against an optical flat (Zerodur®, Edmund optics, $\lambda/20$) at 45° incidence angle, to give the Fresnel reflection coefficients. It is then attenuated by a neutral density filter (OD XX), before being measured

by a PMT, equivalent to the one in the analyzer (Hamamatsu microPMT H12403-01). In the data analysis stage, the measured scattering intensity is then divided by the corresponding beam intensity.

2.6 Computer control

The instrument is controlled by a computer interface based on the PXIe platform from National Instruments (NI). The control software is developed in NI LabVIEW and features a unified graphical user interface to control motor movements, to acquire data, and to set up the measurement routine. Data acquisition is facilitated by a multichannel digital oscilloscope (NI PXIe-5171R, 250 MHz, 14 bit) measuring the PMT signals and a general-purpose digital/analog I/O interface (NI USB-6000) that reads temperature and humidity sensors. Once the desired measurement parameters (e.g. start and stop angle, number of measurement points, exposure time) have been set, the measurement and data logging process itself is autonomous, limiting the risk of human errors. All relevant controls are motorized and can be adjusted remotely without opening the light-blocking curtains, which allows the temperature inside the compartments to stay stable.

3. Measurement steps

The developed acoustic levitator is capable of holding a sample in the range of 0.5 to 3 mm in size. Smaller particles are still levitated, but not orientation controlled [17]. This limitation comes from the wavelength of the acoustic waves. For different length scales, different transducers can be used. Better orientation control is achieved for samples with an elongated shape than for round samples. Other than the size limitation, the levitator can hold a wide range of samples. The measurement procedure is outlined in Fig 4. A video presentation showing the measurement steps in detail can be found in [13].

The mechanical calibration of the scatterometer is described in detail in [12] and consists of leveling the analyzer plane (rotation stage), followed by aligning the beam using pinholes and mirrors. This is typically only done when assembling the instrument, or when there is a reason to suspect that something has moved, e.g. during swapping of the light source.

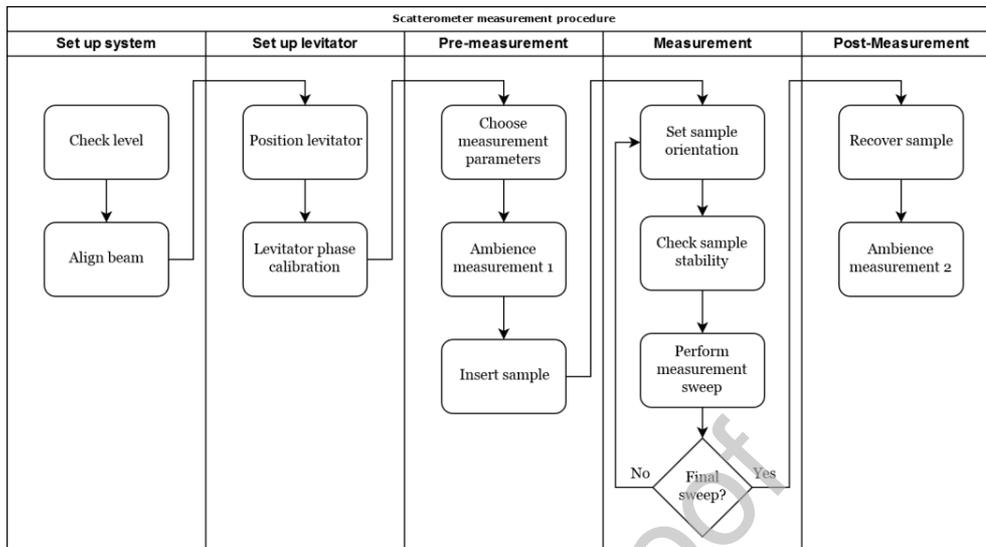


Fig. 4: Flowchart of measurement procedure. The system set-up step is usually omitted unless components have been swapped

The acoustic levitator must be calibrated at the start of every measurement session, since the shape of the acoustic field is sensitive to variations in temperature, humidity, and air pressure. The acoustic field is probed by an air-coupled transducer with a narrow aperture and the phase and amplitude of each channel is adjusted individually.

Before inserting the sample, the light scattering of the empty scatterometer is measured, which gives an estimate of the uncertainty level caused by ambient light and electric noise. The sample is typically introduced with a “spoon” made from acoustically permeable mesh (Fig. 5). Another acoustically permeable mesh spans across the lower hemisphere of the levitator, acting as a safety net for the sample. Once the desired orientation has been chosen for the sample, its stability is checked with the sample monitoring system. The software then performs a set number of sweeps, measuring the four different polarization combinations for each point on each sweep. Once all desired orientations are measured, the sample is recovered, and a second measurement is done of the empty scatterometer, making sure that the ambient level has not drifted.



Fig. 5: Mesh “spoon” for sample insertion

4. Results

Our setup is unique in its capability of measuring light scattering of a levitated sample along both the scattering (θ) and azimuthal scattering (φ) angles, something we refer to as 4π capability. The azimuthal scattering angle can be set by rotating the sample along the beam axis, while the scattering angle is scanned over by the analyzer. This allows us to cover any solid angle, except for two narrow cones (11°) around the back and forward scattering directions, since the analyzer must not be allowed to block the beam. The 4π capabilities of the instruments were tested by measuring light scattering from a piece of polystyrene foam at 20° intervals over both φ - and θ -angles.

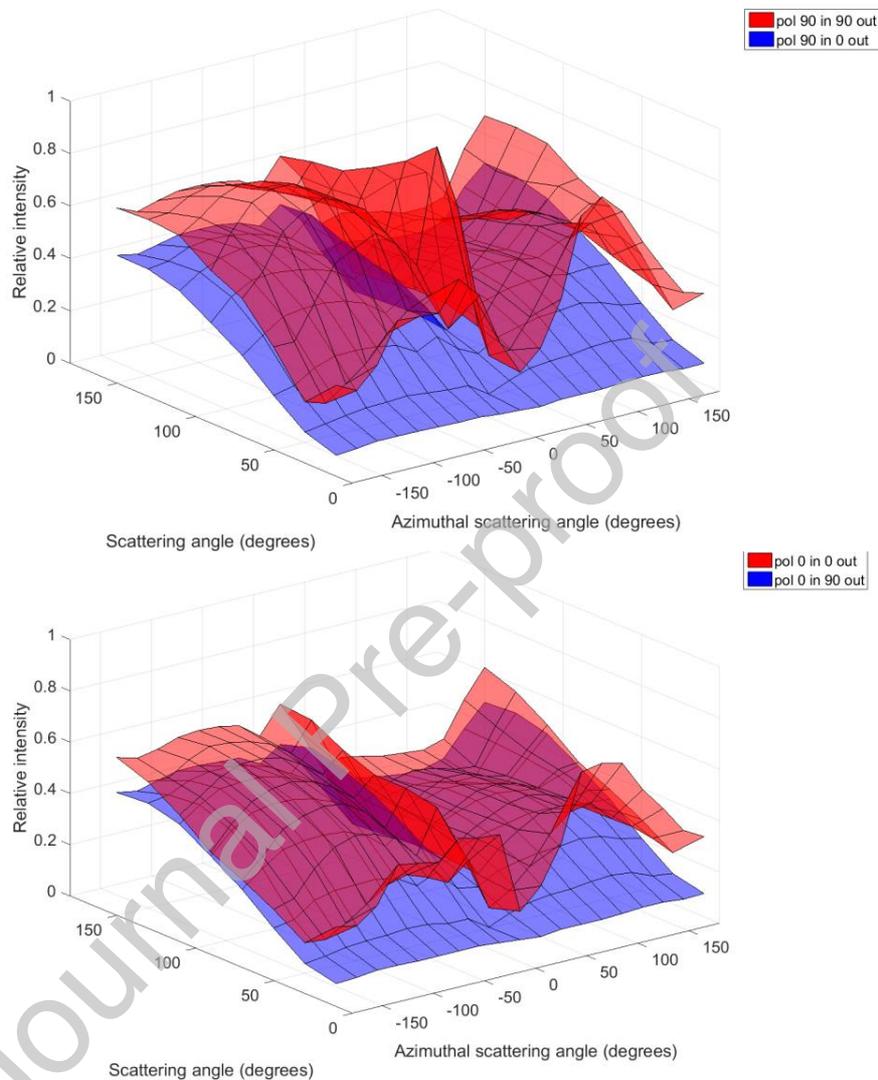


Fig. 6: Light scattering from a polystyrene foam sample. A scattering angle of 0° represents the forward scattering direction.

The sample shape and positioning were reconstructed using camera tracking and the light scattering was measured in θ sweeps going from 170° to 10° in relation to the forward scattering axis. For each azimuthal scattering angle, the θ sweep was repeated three times, to reduce the chance of outliers. Figure 6 shows the light scattering, separated into different polarizations, while fig 7 shows the sample and its 3D reconstruction. The local coordinate system of the particle was defined such that the azimuthal scattering angle is 0° when the y-axis is horizontal, the z-axis is pointing down and the forward scattering direction is along the x-axis.

The irregular shape of the sample means that the scattered intensity varies by up to 70% as a function of the azimuthal scattering angle (Fig. 8, left). In the forward scattering direction, the light tends to preserve its polarization (Fig. 8, right).

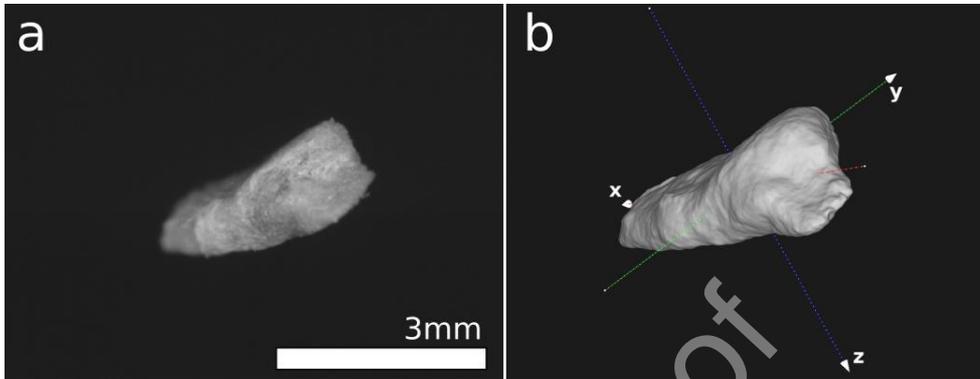


Fig. 7: Photograph (left) and 3D reconstruction (right) of the polystyrene sample, as well as its assigned local coordinate system.

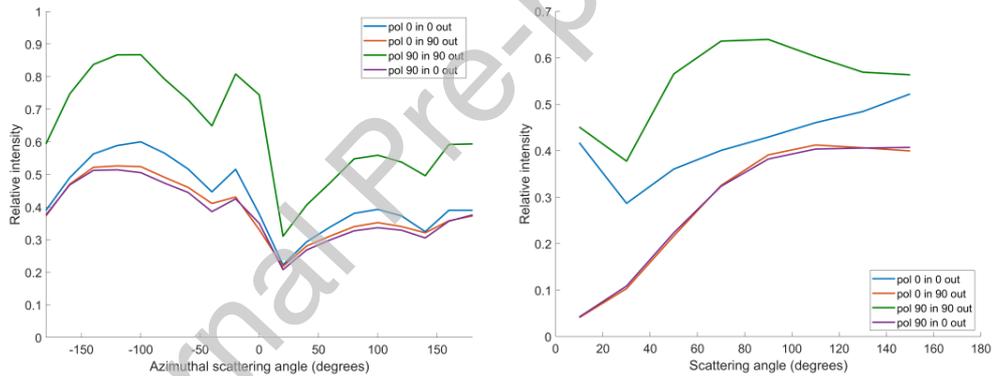


Fig. 8: Azimuthal light scattering in 360°, sampled at a scattering angle of 90° (left), and light scattering averaged over all azimuthal scattering angles (right).

A multi-angular measurement was conducted on a sample consisting of spherical 500 nm SiO_2 particles, naturally aggregated to form an irregular shape (Fig. 9). This material was previously used to investigate clustering of space dust and its resulting optical properties [16]. The spherical shape of the particles makes the sample suitable for comparison to simulations [13]. The light color was filtered to 488 ± 5 nm by a laser line filter (Thorlabs FL488-10).

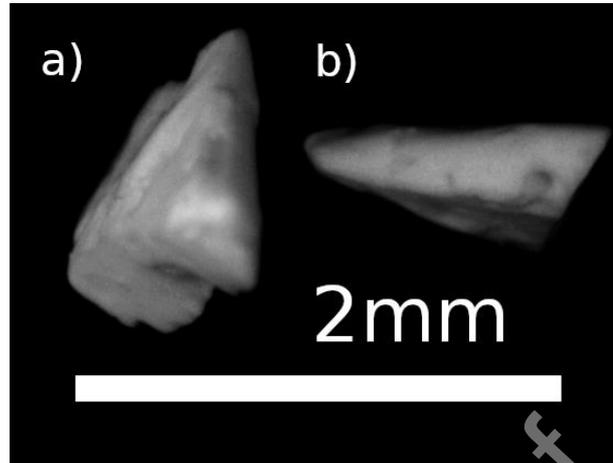


Fig. 9: SiO₂ aggregate sample, imaged by the sample monitoring system, rotated a) 90° and b) 180° around the beam axis

The sample was first measured upright, i.e. $\varphi = 90^\circ$ (Fig. 9a) and then rotated to $\varphi = 180^\circ$ before measuring it again. Each angle was measured three times to ensure repeatability of the measurement. The light scattering intensity was divided by the measured beam intensity and normalized relative to the highest scattered intensity. Figure 10 shows the measured scattering intensities for the different beam and analyzer polarizations. The sample shows characteristics of a typical diffuse reflector, scattering light equally in all directions and depolarizing the incoming light. Some grazing angle reflections are seen in Fig. 10a) close to the forward scattering directions, where the light reflecting from the side of the sample retains its polarization. This is less pronounced when the sample is rotated 180°, since the flat side of the sample is not facing the analyzer.

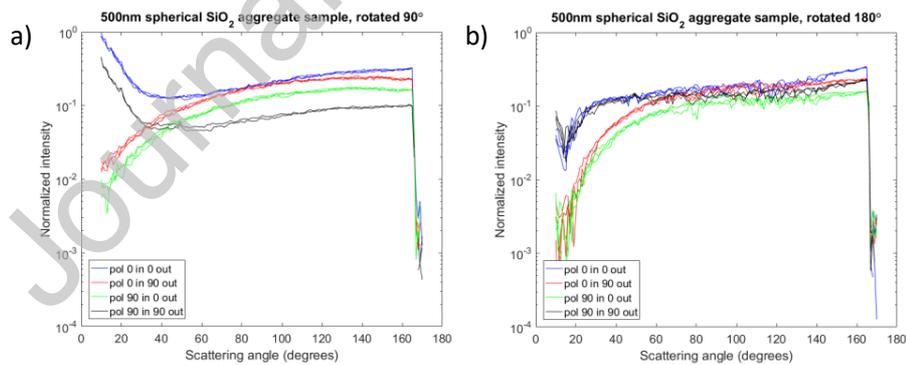


Fig. 10: Light scattered from SiO₂ aggregate sample, rotated a) 90° and b) 180°. Pol 0 denotes a horizontal polarization while 90 is vertical. “In” refers to the polarization of the beam and “out” refers to the analyzer polarization.

5. Discussion

Our instrument is the first full 4π scatterometer for measuring mm-sized samples. It is based on a non-destructive and non-contacting method for manipulating the sample during measurement. This gives it advantages over existing setups based on particle flows or mechanical holders: It allows repeated measurements conducted simultaneously with sample characterization, and it provides ability to measure rare, fragile, and valuable samples. Several novel features had to be developed to have these capabilities, the most remarkable being a levitating sample holder with absolute orientation control. The sample monitoring system was needed as a stability feedback mechanism for the levitator, but it also provided the added benefit of enabling photogrammetric sample 3D reconstruction. This gives the sample an absolute and repeatable reference frame for determining its orientation. The 3D reconstruction provides important input for simulations, when comparing theory and experiment, since it gives the exact position and orientation of the sample when the measurement was acquired.

The acoustic sample holder is the most novel feature of our scatterometer. It is modular and can be swapped out. This allows us to retain comparability to other scatterometers, e.g. the IAA Cosmic dust laboratory. Like in their design, a conical pedestal with a circular tip can be used as a sample holder to support small rock samples as well as engineered test samples, such as glass ball lenses.

The levitator is unique in its orientation control abilities, however, being a prototype, it has limitations. Manipulating dense samples is challenging, since high acoustic power reduces the stability of the orientation control. Some orientations are in this situation more challenging than other ones. In the vertical orientation, the weakest gradient of the acoustic trap is the one that fights gravity, and this orientation is therefore limited to light samples (e.g. polystyrene foam) only.

Several light sources were considered for the scatterometer, before settling for the LDLS source. Laser-based sources provide well-defined frequency, high monochromatic intensity, and ease of collimation and focusing. However, while their coherence can be an advantage in many cases, it is not comparable to the behavior of natural light scattering. Speckle causes unevenness in the scattered light and certain optics, like birefringent polarizers, can cause the laser beam to interfere with itself. The LDLS source solves these issues by providing unpolarized, incoherent white light while retaining the convenience of being fiber-coupled. The light spectrum is chosen by filters. This gives the flexibility of choosing any wavelength or spectrum that is made possible by available filters, but also has the drawback that a narrow laser line filter reflects almost all light, leaving a low intensity beam. Our compromise was to use a filter with 10 nm FWHM, and a convex lens that focuses the light around 100 mm past the sample. With this arrangement, the resulting beam intensity is comparable to that of a fiber-coupled KAP 41 Ar-Kr-laser.

The sample monitoring system was built with the camera in the measurement plane. This compromise allowed the system to be installed without modifying the levitator geometry, but the specular reflection of the objective lens means that it must be covered during measurement. This restriction means that the camera monitoring system can only provide a statistical measure of sample vibrations, rather than real-time deviation data. A next version of the levitator is under development, which provides higher stability for challenging orientations, as well as accommodation for sample imaging outside the imaging plane. Combined with stroboscopic lighting and PMTs with integrated solid-state shutters, true real-time sample monitoring can be achieved.

Other planned improvements include a more compact measurement head on the analyzer. Using off-the-shelf components, the angular width of the measurement head can be reduced to half, narrowing the prohibited sectors around the beam axis from 11° to 5° . Moving the

camera out of the measurement plane would allow up to 14 measurement heads to be placed on the rotation stage, which would allow the sample to be measured from both sides simultaneously. This would speed up the measurement.

6. Conclusions

We demonstrated the capability of making fully non-contacting 4π light scattering measurements of millimeter-sized particles suspended in midair. This capability is unique and, together with shape reconstruction of the sample, allows validation of complex computational models for light scattering from highly irregular particles. The instrument can be used to characterize rare and precious materials, such as mineral dusts collected by sample-return missions in space, in a novel way - with full orientation control in the course of the measurement and sample preservation. With the addition of absolute intensity calibration, our approach would also allow measuring the albedo of the sample in a way that is explicitly tied to the scattering angle, offering an advantage over the traditional integrating sphere approach.

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Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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