

Observing the Glass and Jamming Transitions of Dense Granular Material in Microgravity

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The present Letter investigates a weakly pulsed granular system of polystyrene spheres under longtime microgravity conditions on the International Space Station. The spheres are measured using diffusing wave spectroscopy (DWS) and are described by mean square displacements (MSDs). Our aim is to use this technique to provide the first experimental evidence for glassy dynamics in dense granular media in microgravity and subsequently compare these results with ground-based measurements to see how the nature of these dynamics changes without the influence of gravity. Our results show that as we densify the sample in microgravity, glassy dynamics appear at a volume fraction 1.6% lower than on ground. We also show how the influence of gravity can affect how dense a granular system one can prepare by comparing the final jamming point of our sample on the ISS compared to our ground setup. We show that jamming occurs at a volume fraction 0.5% lower in space compared to on ground, showing that we can create denser states when a granular system is in the presence of a stronger gravitational field.

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Granular material is one of the most abundant and widely utilized types of materials in our society [1,2]. Grains feature in every aspect of life, from the coffee one drinks to the paths one walks on [1,3]. They also feature heavily in industries such as pharmaceuticals, agriculture, and energy production [1]. Understanding the way grains move and interact then becomes a field of key interest, especially when they are produced, stored, and used in a dense state [4,5]. One of the key characteristics of granular material is gravitational settling [1,6]. Gravity is an attractive force that influences the dynamics of the system [7]. It is therefore a natural question to ask, what if the force of gravity were removed from such a system? How would the dynamics change? Such a system has been simulated in [8], and granular material has been investigated in microgravity experiments prior to this Letter, but up to this point, not in such a dense state. The previous works have tended to revolve around less dense systems such as granular gases [9–11] due to the natural difficulty of capturing the complete dynamics of a dense, opaque system, combined with the challenge of obtaining enough experimental time in microgravity to resolve slow relaxation dynamics of dense systems. This provides the motivation for our

investigation, where we used an established light scattering technique such as diffusive wave spectroscopy (DWS) to measure dynamical responses of dense granular systems on the International Space Station and compare our findings to those seen on the ground [12–15].

Dynamics is a broad term used to describe how the system moves. The goal of our experiment was to measure the slowing down of dynamics as we densified our granular system to investigate the glass and jamming transition. When considering the state diagram of granular media [16], one can see that the dynamics are controlled by the density and the driving force. It has long been hypothesized that changing the density of a dynamical granular system, while keeping the driving force constant, should yield similar characteristics to those of a glass [17,18]. We then looked to measure and quantify the slowing down of the dynamics of our system as we increased our density, in order to identify the point at which a similar dynamical response to that of a glass [17] is seen. Measurements were conducted in both microgravity and on the ground to enable a direct comparison of the effect gravity has on these transitions.

For our experiment, as shown in Fig. 1, samples are illuminated with a 532 nm laser, intensity traces are collected in both the transmission and backscattering geometries, via single-mode optical fibers, referred to as “bulk” and “wall” dynamics, respectively. Also in the backscattering geometry is a line camera which records a time lapse of 500 speckles, via multimode optical fibres. DWS is then performed by correlating individual speckles and then averaging over all speckles. The 130 μm mean diameter polystyrene particles with the distribution of particle sizes can be found in [19] and

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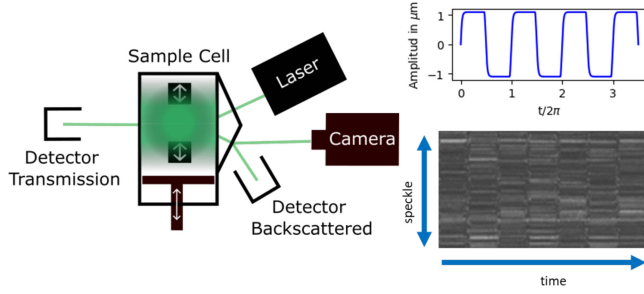


FIG. 1. Schematic of the setup with piezo agitation source in the sample volume with agitation profile, piston for volume control, laser source, detectors for collecting intensity fluctuations in transmission and backscattering geometry, and a camera for time-resolved speckle detection.

are contained in an $11 \times 10 \times 5$ mm sample cell. The height of the sample cell is adjustable, from 11 to 3.6 mm, which enables our change in volume fraction (VF) [20], where the $VF = V_{\text{Sample}}/V_{\text{SampleCell}}$ is expressed as a percentage. Each measurement produces an intensity correlation function g_2 , from which the field correlation, g_1 , can be calculated via the Siegert-Relation [21],

$$g_2(t) = 1 + \Lambda |g_1(t)|^2, \quad (1)$$

where Λ , referred to as the coherence area factor, is assumed to be 1. The later analysis assumes that we can connect $g_1(t)$ with the mean square displacement (MSD) $\langle \Delta r^2 \rangle$ by using the DWS approximations [13,14]

$$g_1(t) = \exp\left(-\frac{1}{3} \left(\frac{kL}{l^*}\right)^2 \langle r^2 \rangle\right), \quad (2)$$

where $k = 2\pi/\lambda$ is the wave vector and L is the length of the sample cell. The effective mean free path length $l^* = l/(1 - \langle \cos(\theta) \rangle)$, where l is the mean free path length determined by the scattering cross section $\sigma(\theta)$; for more details see Ref. [15]. A full description of how the oscillation from the piezo influences the correlation function g_1 can be seen in the Appendix.

We conducted similar experiments both on the ISS and on the ground with the same material, agitation frequency, and agitation time. The sample cells are filled on the ground packing fraction at ambient air pressure at a maximum packing fraction. The cell internal maximum design pressure has been calculated to be 1.25 bar. In Figs. 2(a) and 2(c) we show the intensity correlation functions for ground and in microgravity as we compress the sample. The results show correlation functions acquired over 10-hour measurement runs in both the transmission and backscattering geometry. The volume fractions compared are different as on ground the minimal VF one can construct due to gravitational settling is 55%, whereas in space, our minimum based on our sample cell geometry was 44%. The minimum is calculated

when taking into account the total volume of grains and the maximum volume of the sample cell when the piston is fully extended. In both cases, the bulk dynamics appear faster than the wall dynamics, e.g., a faster decay. In the bulk dynamics on the ground, a systematic slowing down is seen as the sample is compressed. This is less distinguishable for the microgravity sample, but a qualitative difference can be seen in the faster initial part of the decay. On the ground, the wall results show a much slower decay than its counterpart on the space station. When viewing the ground results in the MSD representation, Fig. 2(b), one can distinguish different regimes. Firstly a ballistic-like motion, followed by a plateau, and finally subdiffusive behavior shown by a power law exponent of 0.5. This is similar to results in microgravity [Fig. 2(c)], with ballistic behavior, this time without a recognizable plateau and a final subdiffusive behavior that follows a power law exponent of 0.2. Further discussion of the implications of this can be found in the Appendix. From the MSD representation on the ground, we also see that the measurements at the wall and in the bulk results tend to collapse to show the same curve. This is in contrast to the microgravity experiments, where the two dynamics show different behavior in the MSD representation. We note that while packing is expected to be different near the walls compared to inside the bulk, as per [22], the effect on the calculated l^* and the subsequent MSD conversion is negligible.

With a understanding of the key differences of results in space and on the ground, we can now extend our view over a larger range of VFs. Figure 3 presents a case for identifying both the glass transition and the resulting jamming for a set of experiment runs where the sample is systematically compressed and then measured for 1 hour while agitating. When analyzing Figs. 3(a) and 3(b) qualitatively, one can observe the slowing down of the dynamics as the sample gets denser for both ground and microgravity, shown by the decreasing MSD slopes. Figure 3(c) shows the MSDs at 10 s, plotted as a function of the VF. The time of 10 s is chosen because this is the time after caging. At some VF, a significant decrease in MSD is observed. On ground, this is at $60.6\% \pm 0.1$, and on the ISS at $59.0\% \pm 0.1$. The highest VF plotted for both sets of measurements represents the maximum densification point that was possible for that sample. With the influence of gravity, this point is $61.1\% \pm 0.1$, and in microgravity, this is $60.3\% \pm 0.1$. Where the uncertainty is calculated from the uncertainty in the mass of the sample and the uncertainty in the encoder position.

The results in Fig. 3 show only correlation functions formed from the fiber. As mentioned, included in the experimental setup on the ISS is a line camera, which shows a time evolution of change in speckle intensity with a time resolution of approximately 1 ms. The intensities of these individual speckles are then correlated to form our correlation functions. One of the key advantages of the line

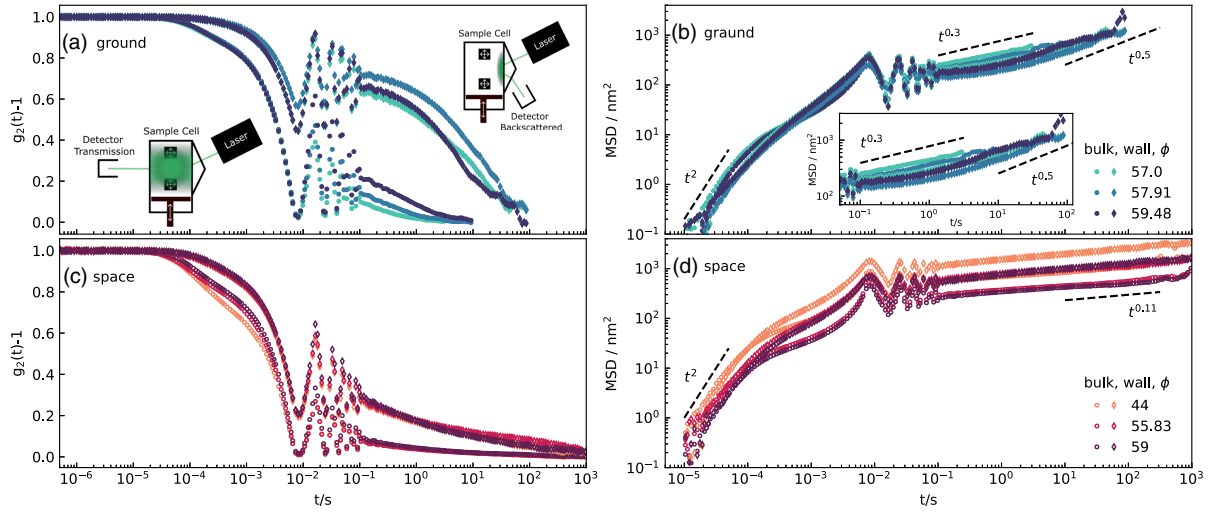


FIG. 2. Intensity correlation functions and mean squared displacements respectively for backscattering (wall) and transmission (bulk) measurements for ground in (a),(b), and for microgravity in (c),(d).

camera is the ability to easily remove the oscillations imprinted into the correlation functions, thereby showing the underlying particle dynamics. Figure 4 shows a full spectrum of VFs measured starting from 45% to a maximum of 61.3%, from both the fiber and line camera. This figure shows a complete picture of our experimental campaign conducted on the ISS. We again see the slowing of the longtime relaxation as the VF increases and the system becomes denser. Figures 4(a) and 4(b) include all correlation functions and corresponding MSDs for all 1, 5, and 10 h experiment duration runs in microgravity. When considering our fiber measurements, we see the dominating influence of the oscillation. But by fitting the data using the method outlined in the appendix, we can remove the oscillation, which just leaves the underlying dynamics of the particles. The camera reproduces results from the fiber, lending credence to the reliability of our setup and the reproducibility of our results. Figure 4(c) shows a calculated root mean square displacement as a fraction of the diameter of the particles, extracted from the line camera MSDs. We can describe this as a localization length. These values are then plotted as a function of VF for all expansion and densification runs. What is seen is what appears to be a state change in a range of VFs between 59% and 59.5%, which agrees with the results seen in Fig. 3, where visually one can see that at that VF range the localization length starts to decrease significantly. This presents a clear change of state over a large set of experimental measurements in a microgravity environment.

With the complete picture of the experimental results now presented, we can discuss the interpretation of our results. First, let us address the difference in apparent wall and bulk dynamics. It can be assumed that the light detected in backscattering geometry has been scattered a smaller number of times compared to in transmission [23]. The

total number of scattering events make up the scattering path. Particle movement results in the decay of the correlation function. The shorter the length of the scattering path, as assumed when observing the wall dynamics, the smaller number the scattering events there are. Therefore, for a smaller scattering path length, with a similar amount of particle movement, the decay of the correlation function would then appear slower [13]. But the MSD representation takes into account our scattering path length differences. Our results in Fig. 2(b) on the ground show that even with different-looking correlation functions for the different scattering geometries, a single measurement for the wall and bulk dynamics collapse into the same result in the MSD representation. A difference remains in Fig. 2(d) when looking at the MSDs for the wall and bulk dynamics in microgravity. From this one could infer that the system on the ground is more homogeneous than in microgravity, as the dynamics shown by the MSD representation are similar at the wall and in the bulk. This could be due to it being easier to establish contacts between particles on the ground and therefore transfer the energy produced by the piezos throughout the system more efficiently. Therefore particles at the boundaries of our sample cell experience a similar agitation to those in the majority of the bulk, which results in a more homogeneous dynamical response throughout the whole sample [1].

Now to determining the glass and jamming transition. Within the field of granular matter, the processes of glass and jamming transitions vary in terms of their exact definitions [24–27]. We define a glass transition as the sudden change in the generalized dynamics of the granular system from a fluid, liquid-like state to that of an amorphous solid [28]. We define a jamming transition as an extreme extension into the realm of glassy dynamics, in which a mechanically stable state has been reached [25,29],

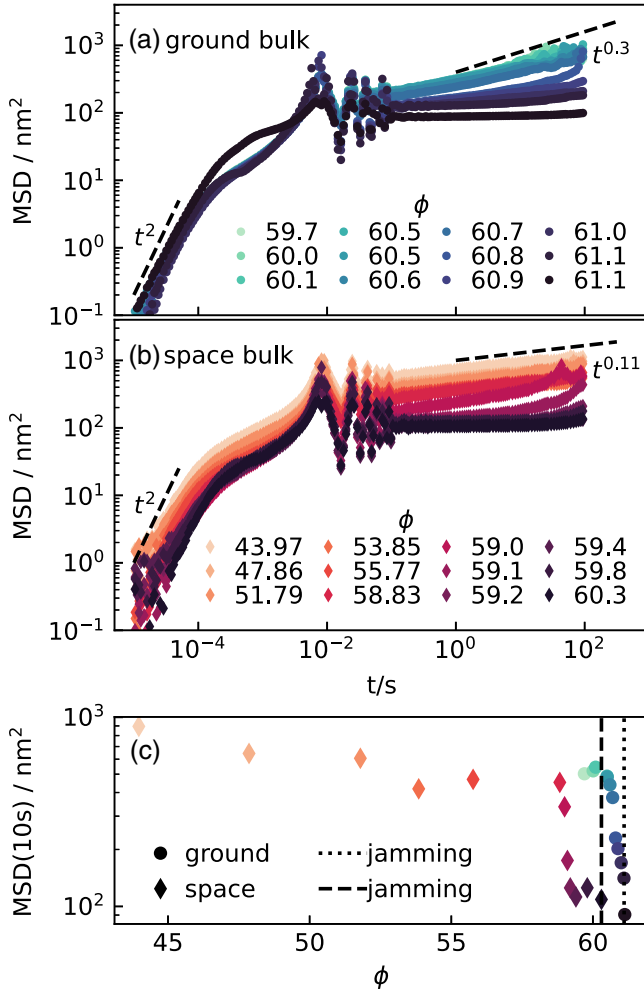


FIG. 3. Volume fraction dependent mean squared displacement (a) on the ground, (b) in space, and (c) mean squared displacement at 10 s depending on Volume fraction for ground and space illustrating glass transitions at $59.0\% \pm 0.1$ and $60.3\% \pm 0.1$ as well as jamming transition at $60.6\% \pm 0.1$ and $61.1\% \pm 0.1$.

in our case above a timescale of 10 s. Within the context of this experiment, we define a glass transition as when the dynamics start to dramatically decrease with increased density, and jamming is when the piston gets stuck and can no longer densify the system. For our system, the piston can exert a maximum force of 70.5 N. For both of these transition points in microgravity, our results in Fig. 4 show that the exact position is history dependent [30]. When similar experimental procedures are implemented both on ground and in microgravity, as shown in Fig. 3, two things become apparent. Firstly, the glass transition occurs at a higher density on ground, specifically at a VF that is 1.6% higher. Secondly, the jamming transition occurs at a VF that is 0.5% higher on ground compared to in space. This second point demonstrates that we can produce a more dense system on ground, as shown in previous experiments [31]. Additionally, both the glass and jamming transition positions do fall in the range of VFs predicted by [32].

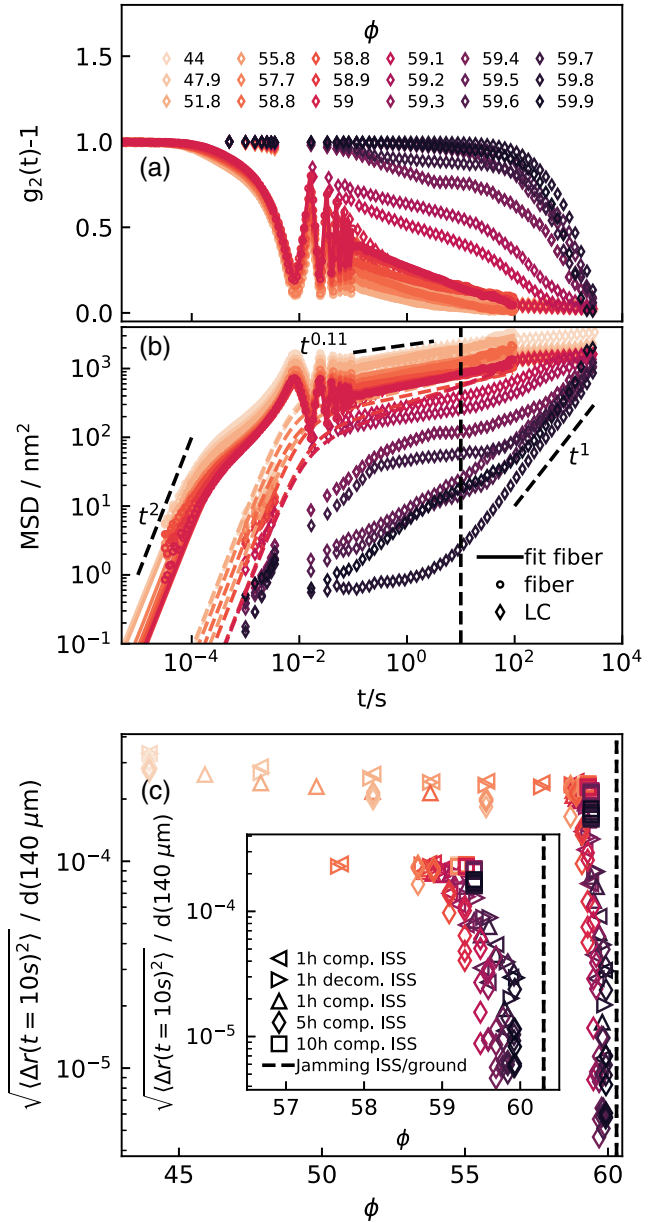


FIG. 4. Camera and fiber measurements in backscattering geometry presented as (a) intensity correlation functions and (b) mean squared displacements; the dashed lines at 10 s indicate the time where the plateau value is extracted for (c) the volume fraction dependent comparison to root MSD normalized to the particle diameter.

So why does this occur? In microgravity the cohesive interparticle forces such as friction, Van der Waals, and the electrostatic force [33], are the dominant forces acting on the system. These interactions limit the ability of the granular system to rearrange into more dense configurations compared to a system present in a gravitational force field, where gravity can apparently aid motion to overcome barriers with additional gravitational acceleration. This results in the earlier onset of glassy dynamics, due to the higher stability of each configuration in microgravity.

With regards to jamming, the reduction in the rearrangement ability better maintains the long force chains that permeate throughout the system to the boundaries and make the system mechanically stable [34], limiting the ability for the piston to compress the sample. Both inter-particle and inter-surface friction will also play a major role with regard to the dynamics of our system. Both frictional effects should decrease in microgravity, due to the absence of a strong gravitational force, which one would expect to facilitate easier compression and particle movement [30,35,36]. However, our results suggest the opposite. This implies that the increased ability to reconfigure provided by the presence of a gravitational field more than compensates for the resulting increase in friction. We hope to try and quantify the exact role of friction by repeating the experiment with friction-reduced Aerosil-coated particles in the future [37,38]. Finally, while the effect of gravity on granular dynamics has been modeled using discrete element method simulations [39], there has been little work with regards to dense systems such as ours. We hope that our experimental results can provide the motivation for such an investigation.

In summary, we investigated a weak pulse-driven granular system under microgravity conditions on the ISS. We utilized DWS to describe the system by MSDs of the investigated polystyrene spheres. The sample cell creates volume fractions as low as 45% on the ISS. While the piston is densifying the sample, glassy dynamics are observed at volume fractions of $\phi_g^{\text{iss}} > 59.0\% \pm 0.1$ compared to $\phi_g^{\text{gr}} > 60.3\% \pm 0.1$, which we believe is the first evidence of glassy dynamics experimentally shown for a dense granular system in microgravity. Under the influence of gravity, the jamming transition appears at a higher volume fraction, $\phi_{\text{jam}}^{\text{gr}} = 61.1\% \pm 0.1$, compared to in space, $\phi_{\text{jam}}^{\text{iss}} = 60.6\% \pm 0.1$. This shows that we can produce states in microgravity that are less dense but are as mechanically stable as denser states prepared on ground.

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Data availability—The data that support the findings of this article are not publicly available. The data are available from the authors upon reasonable request.

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End Matter

Appendix: Additional information—

Experimental setup: The intensity signal is collected from the sample split and fed into two avalanche photodiodes, in both transmission and backs-scattering

geometries. These are then cross-correlated using an ALV USB correlator series-7004. The volume of the sample is calculated using the stated density of 1057 kg/m^3 of the PS grains and the mass [40]. The

height of the sample cell is given by an encoder position provided by a magnetic encoder scale. Inside the sample cell are 4 piezo cubes, of size $2 \times 2 \times 2$ mm, located about 1.5 mm away from the walls at each corner. These provide an agitation amplitude of $2.2 \mu\text{m}$ and an acceleration of approximately 1 g at 60 Hz. The volume of the piezo elements is then deducted from the volume of the container when calculating the VF [20]. The agitation frequency can be tuned to values between 5 Hz and 2 kHz. An agitation frequency of 60 Hz was chosen after preliminary tests, where 60 Hz showed the best-resolved dynamics after 1 hour-long measurements.

MSD: Further analysis of the MSD representation shows that both ground and space samples show sub-diffusive behavior at longer time scales with maximum MSD power law exponents of 0.5 and 0.2 respectively. This sub-diffusive behavior is similar to that seen in liquids for the high-frequency contributions [41,42]. Consequently, we can infer from our measurements that ground samples present behavior closer to a normal liquid-like behavior resulting finally in diffusion not seen because of accessible timescales and length scales. To reach the point where diffusion appears we would either need a stronger agitation method as seen in [15], or to analyze longer length scales by probing with a laser with a longer wavelength, e.g., in the THz regime [43,44]. However given our current setup, we could infer that ground measurements are closer to reaching a state similar to a liquid on our measured time scale ($\beta = 0.5$) compared to in space where it appears to be a deeply supercooled glass due to its similarity with the high-frequency power laws seen in molecular systems ($\beta = 0.2$). Intuitive one would expect granular material to be more similar to a liquid without gravity because gravity is irrelevant for molecular liquids. Leaving an open question to be tackled.

Another open question concerns the length scale of particle dynamics that is probed in the experiment and the exact nature of these dynamics. We have shown that we have seemed to resolve the nanometer movement of 140-micrometer particles [45,46]. We believe that the length scales calculated could describe two processes. First, we could measure the deformation of the particles on contact, as there is precedent in literature called clapping contacts. The deformations are on the order of 10 nm, depending on excitation energy, and stay in contact for about 1×10^{-6} s [7,47–49]. This is not in our resolution limit but the calculation only considers normal contact forces, so this could still be responsible for the ballisticlike motion as shown in [15]. Alternatively, this could be a true motion which is a combination of translation and rotation [7]. In which most of the sample exhibits minimal to no movement in comparison to the particles contacted directly by the piezos thereby reducing the average movement of the whole sample to nanometers [49]. Verification of these length scales comes in two-fold; first DWS theory tells us that our

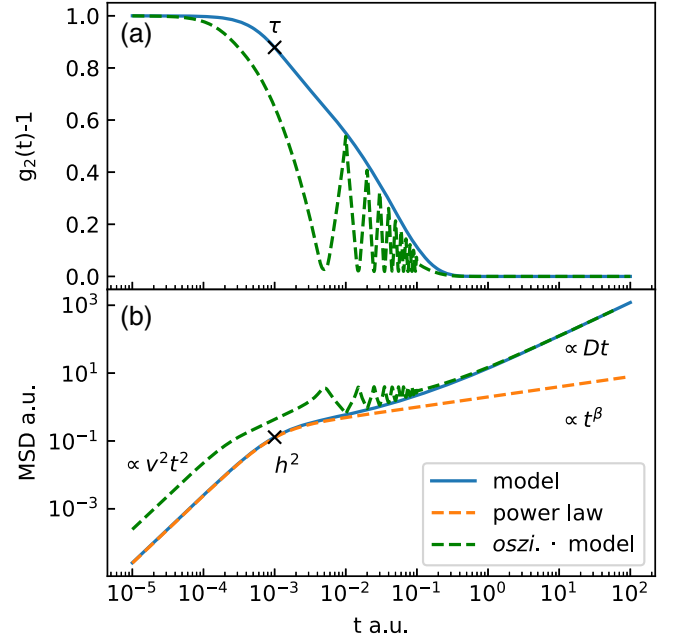


FIG. 5. Schematic illustration of (a) correlations function and (b) mean squared displacement of the model function with (green) and without (blue) agitating oscillation as described in the text.

resolving length scale is defined by the wavelength, and specifically the wavelength split across a lot of scattering events [13,50–52]. Secondly, other experiments that have looked at more dilute systems of similar-size particles also achieved length scales in this range [23,53,54]. One of the key unknown factors in calculating our MSD is determining our l^* or randomization length. This has been a challenge to verify experimentally with regard to light scattering on granular material [16,17,53–55]. There is also a disagreement when comparing length scales with those predicted from mode coupling theory (MCT) [56,57]. However, these calculations assume frictionless hard spheres, which presents a significant difference compared to our experiment.

Modeling the agitation: We assume that the measured intensity correlation function $g_{2,m}$ is a convolution, weighted with C , between the oscillation $O(t)$ with the intensity correlation function $g_2(t)$, and the pure $g_2(t)$ correlation function

$$g_{2,m} = g_2(t)[CO(t) + (1 - C)]. \quad (\text{A1})$$

The oscillation $O(t)$ of the agitation is given by (derivation see Ref. [58])

$$O(t) = \frac{1}{T} \int_0^T \exp(-\kappa^2 A^2 [P(\omega(t+t')) - P(\omega t')]^2) dt' \quad (\text{A2})$$

with amplitude A , the variance of scattering vectors κ , time of period T and the correlation of the periodic rectangular agitation function $P(t)$ is modeled by the profile shown

in Fig. 5 which is obtained by measuring the applied voltage profile. We measure $g_2(t)$ and assume that the field correlation function $g_1(t)$.

To construct $g_1(t)$ we use a model function for the MSD with power laws for ballistic, subdiffusion, and diffusion behavior

$$\langle \Delta r^2 \rangle = \frac{h^2}{\left(\frac{t}{\tau}\right)^2 + \left(\frac{t}{\tau}\right)^\beta} + 6Dt + 6D\tau(e^{-\frac{t}{\tau}} - 1) \quad (\text{A3})$$

with ballistic time constant τ , diffusion coefficient D ,

localization length h , and power law exponent β (illustrated in Fig. 5) see also Ref. [15].

Multispeckle intensity correlation function: The intensity correlation function for the line camera images is defined as

$$g_2(t, t + \Delta t) = \frac{\langle I(t)I(t + \Delta t) \rangle_{\text{px}}}{\langle I(t) \rangle_{\text{px}} \langle I(t + \Delta t) \rangle_{\text{px}}} \quad (\text{A4})$$

with delay time Δt between image lines of same time and the pixel average $\langle \dots \rangle_{\text{px}}$ over one line.