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Granular fragility under thermal cycles

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Abstract We study the effect of thermal cycling of granular materials on both the granular packing fraction and the position of an intruder object within a granular sample. We demonstrate that the packing fraction consistently increases with thermal cycling, regardless of the relative coefficients of thermal expansion for the grains and their containers. The exact magnitude of the packing fraction increase appears to be influenced by factors other than the thermal expansion coefficients, with the intergrain friction and the friction between the grains and their container likely to be important. We also examined the motion of spherical intruders with thermal cycling as a function of the size and density of the intruders, and find that such intruders sink within the grains when the pressure from the intruder exceeds a threshold. These data demonstrate the fragility of dense granular ensembles to changes in the ambient temperature.

Keywords Granular · Thermal cycling · Fragility

1 Introduction

The nature of granular packing, i.e., the random arrangement of a dense collection of granular particles, continues to receive considerable attention [1–3]. A typical granular pack can support a load of static stress with only minimal deformation, behaving much like a solid [4]. The same granular pack, however, can be rearranged by the application of relatively small vibrations. The packing fraction, defined as the fraction of sample volume filled by grains rather than by empty space, can also change significantly under such vibrations [3], and the position of intruder particles can be systematically changed by weak vibrations, e.g., the Brazil nut effect [5,6]. This sensitivity to perturbations is a reflection of the "fragile" nature of a dense granular system [7], which arises in part because the majority of the stress in a granular pile is supported by an inhomogeneous network of force-bearing grains [8]. Small changes at grain-grain contacts can cascade along the network, and cause substantial changes to the bulk properties of a granular sample [9].

An interesting manifestation of fragility can result from changing the ambient temperature of the grain environment. Temperature changes induce microscopic expansions or contractions of the individual grains that can impact the bulk physical granular properties. For example, the "apparent" weight of a granular pile depends sensitively on temperature [10,11]; pressure accumulation has been reported in industrial scale storage silos subjected to diurnal temperature changes [12]; and a small change of the temperature of a single grain in a granular pile can affect the path of sound transmission in granular media [13]. Recent experiments have also demonstrated significant changes in the packing fraction of granular materials under thermal cycles [14–16]. The fractional change in size of the grains or their containers induced by temperature changes is typically on the order of 10^{-6} to 10^{-4}, orders of magnitude less than the resultant bulk effect. In this paper, we report further studies of the effects of thermal cycling, focusing on thermally induced packing fraction relaxation of a range of granular materials with different coefficients of thermal expansion, which are cycled in containers that also have different coefficients of thermal expansion. We also investigate the motion of an intruder

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particle within a granular sample which is subjected to thermal cycling.

2 Experimental details

We conducted all measurements on samples prepared by filling graduated cylindrical containers through a funnel that was raised slowly at a constant rate by a stepper motor. The resultant system had a consistent packing fraction of $58.9 \pm 0.2\%$ which was vertically uniform to within the uncertainty of our measurements. Each experimental measurement was performed on a freshly prepared sample with the exception of runs which explicitly probed the effects of repeated thermal cycles. We also compared the results using fresh grains and those with grains that had been repeatedly heated and cooled, and we observed no noticeable difference in the data.

We used spherical granular materials with different coefficients of thermal expansion (CTE), including soda lime glass (Jaygo Inc., $d = 0.2, 0.5, 0.9$, and 3 mm), polystyrene (MaxiBlast Inc., $d = 0.98$ mm), and high density polyethylene or HDPE (US Plastic, $d = 3$ mm). The cylindrical containers were made of either polymethylpentene (PMP) plastic or borosilicate glass with diameters ranging from 14 to 102 mm. The CTE of all of these materials are listed in Table 1. In all experiments, we conducted thermal cycles by placing the samples into an oven and heating them until thermal equilibrium was reached at a pre-set temperature, and then allowing them to cool slowly to ambient temperature (22 ± 2°C) before measurement. We denote the highest temperature reached in each such thermal cycle as the "cycle temperature". Thermal cycling required more than 10 h to reach the cycle temperature and then about the same amount of time to return to ambient temperature, limiting the total number of cycles which could be performed. Test runs in which temperature probes were placed at the center of the grain pack were performed to determine the proper heating and cooling time so that thermal equilibrium was reached. Great care was taken to avoid any effects from mechanical agitation of the sample. Test runs with no thermal cycling were performed to ensure that vibration from environment or during data acquisition had no measurable effects.

Table 1 Linear coefficient of thermal expansion (CTE) of materials used in the packing experiment

Granular media	Linear coefficient of thermal expansion
Soda lime glass grains	9×10^{-6}/K
Polystyrene grains	70×10^{-6}/K
HDPE plastic grains	110×10^{-6}/K
Container material	
PMP plastic	117×10^{-6}/K
Borosilica glass	3×10^{-6}/K

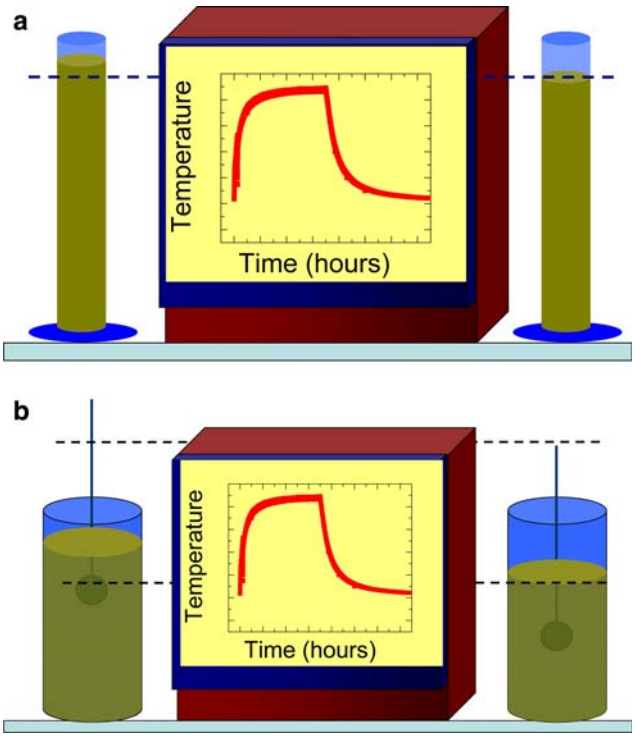

Fig. 1 Experimental setup. **a** Experimental setup of packing experiment. Graduated cylinders are filled to a well-controlled initial packing fraction and placed into the oven for thermal cycling. The packing fractions of the samples are then measured after the thermal cycle. The dashed horizontal line shows the change of pile height after thermal cycles. **b** Experimental setup of intruder experiment. Intruders are buried in the granular pack, with vertical position indicated by a very thin stiff stainless steel post attached to the intruder, which extends above the granular surface. Positions of both the pile surface and the intruder are measured before and after the thermal cycle. The dashed horizontal lines show the change of pile height and intruder position after thermal cycles (Color online)

In the packing studies, packing fractions were measured after thermal cycles and compared with the initial packing. The setup of the packing measurement is illustrated schematically in Fig. 1a, and details have been published previously [14]. The packing fraction is determined using the equation $P = \frac{m}{\rho V}$, where P is the packing fraction, m is the mass of grains in the container, ρ is the density of the grain material used in the experiment, and V is the volume of the granular pile read from the container graduation.

We show the experimental setup of the intruder studies in Fig. 1b. In these measurements, we only studied the polystyrene grains in borosilicate glass containers (10 cm in diameter, 16 cm in depth) (Measurements of intruders in a pile of glass grains with a PMP container yielded no measurable relative displacement when the intruder was placed at the center of the container), with the intruders positioned along the symmetry axis of the container. Spherical intruders of

different sizes (0.64–3.81 cm) and materials (aluminum and brass) were tested. The thermal expansion coefficients of aluminum (23×10^{-6}/K) and brass (19×10^{-6}/K) are nearly equal and both much less than that of the grains. The expansion of the intruder diameter is much less than the grain diameter during thermal cycling. Denser intruders, that would sink into the granular pile under their own weight, were slowly lowered from the top surface after the pile was filled, until they came to rest within the pile. To minimize the disturbance to the packing of the grains which might be caused by pushing the intruder into the grain pack, for less dense intruders ($<4\,\mathrm{g/cm^3}$), we filled the pile to a preset depth, set the intruder in place on the top of the pile, and then filled grains to bury the intruder within the pile. Sample preparation was completed by flattening the pile surface with a soft brush, and the initial burying depth of the intruder was 2–3 mm (2–3 layers of grains). The position of the buried intruder was indicated by a thin vertical stainless steel post (0.4 mm diameter and ~15 cm in length) which was attached to the top of the intruder and stuck out through the top surface of the grains. Thin posts are chosen to minimize the possible friction between the post and the grains. These posts also serve to limit rotation of the intruder within the grains. The buried surface area of the post is a small fraction of that of the intruders (at most 3%), and we therefore judge friction between the vertical post and the grains to have a negligible effect. The position of the intruder and the pile surface were determined before and after thermal cycling by photographing against a ruler with a minimum division of 1 mm. The relative displacement after cycling is the change of distance between the pile surface and the top of the intruder, and we define a downward relative displacement of the intruder as negative. No time dependence of displacement was observed after thermal equilibrium was reached.

3 Experimental results: packing fraction

As our group has previously reported, thermal cycling leads to clear changes in the packing of glass grains in PMP cylinders, a process in which the grains expanded upon heating much less than the containing cylinder [14]. This effect was shown to be independent of sample depth and width over a fairly broad range, and it is also strongly dependent on the cycle temperature [14]. An open question, however, is how much the results depend on the relative thermal expansion of the grains and the container. One might even expect the existence of a simple scaling law for the effects of thermal cycling, with the thermal expansion coefficients as the scaling parameters [15].

In the present study, we examined grain and cylinder materials with rather different CTE, including pairings where the cylinder material expanded less than the grain material. We find that, even for systems with very small thermal expansions, small but non-trivial changes of packing fraction are observable. The change of the packing fraction as a function of cycle temperature for the different material combinations is plotted in Fig. 2a. The value of each point and the error bar are respectively the average value and standard deviation of at least 12 measurements. Figure 2b shows the packing fraction relaxation of polystyrene grains under multiple successive cycles. The packing fraction continues to rise with decreasing increments. The results can be fit to a double relaxation function ($y_0 + A_1 e^{-x/t_1} + A_2 e^{-x/t_2}$) similar to the results in

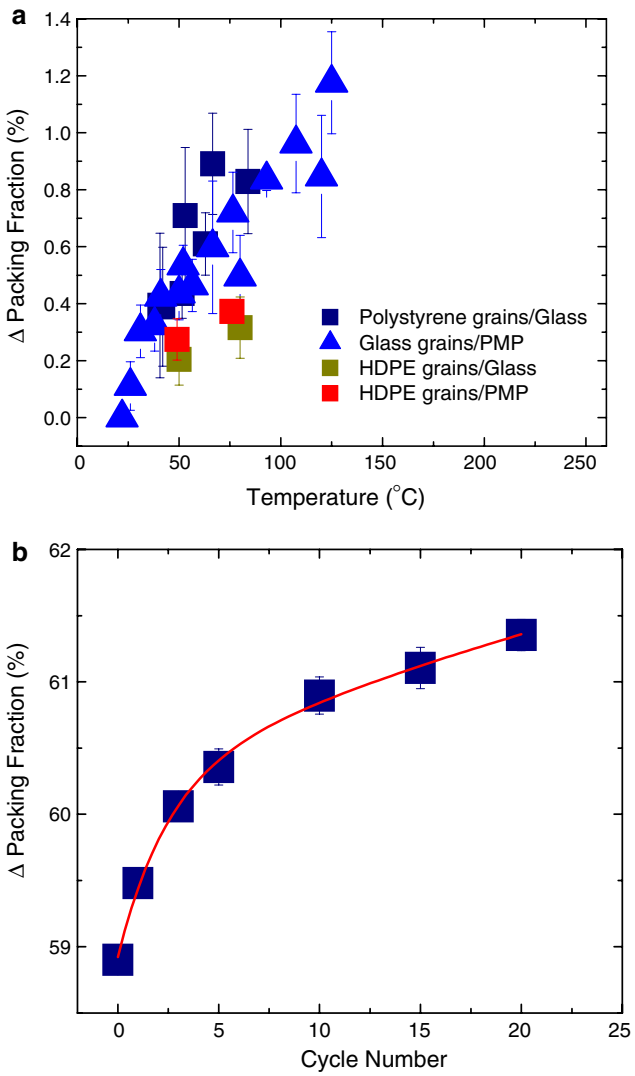

Fig. 2 Change of granular packing fractions with one thermal cycle. **a** The change of packing fractions as a function of cycle temperature. **b** The change of packing fraction as a function of number of cycles (polystyrene grains in glass container). The results can be fit to a double relaxation function (*red solid line*) $y_0 + A_1 e^{-x/t_1} + A_2 e^{-x/t_2}$ with fitting parameters $y_0 = 64$, $A_1 = -0.90 \pm 0.07$, $t_1 = 2.72 \pm 0.48$, $A_2 = 4.12 \pm 0.06$ and $t_2 = 132 \pm 10$. All the samples have an initial packing fraction of 58.9 ± 0.2 (Color online)

Ref [14]. Consistent with our earlier studies, no significant filling depth or container size dependence was observed.

Note that the different thermal expansion coefficients of the materials imply that the size changes of the grains and the container create perturbations at either the boundary (when the container expands much more than the grains inside) or the bulk of the pile (when the container expands much less than the grains) or both (when both the container and the pile material have similar thermal expansion coefficients). The packing fraction relaxation observed in glass grains in glass containers is much less than that from glass grains in a plastic container [17]. This is an example of thermally-induced packing relaxation originating at the container boundary. The data for plastic grains in glass containers, in which the grains expand much more than the container, provide new evidence that the packing fraction relaxation under thermal cycling can also occur in the bulk of the pile, rather than strictly near the boundaries. Curiously, however, the data for plastic grains in glass containers and glass grains in plastic containers seem to lie almost on top of each other in Fig. 2a, even though the physical process dictated by the relative CTE (i.e., the thermal expansion coefficients of glass or plastic used for the containers or the grains) must be quite different.

From a pure geometric point of view and ignoring intergrain friction, the change of packing faction might be expected to only depend on the magnitude of the size changes induced by temperature. The data, however, do not show any scaling to the fractional changes of the grains and container under thermal cycles. This suggests that there are other factors besides the thermal expansion coefficient that affect the change of packing fraction under thermal cycles, such as the friction between the grains or between the grains and the container wall [18–20]. In fact, such a scaling law may simply be experimentally inaccessible due to the importance of the other factors, such as friction, which are quite difficult to control when changing the material being studied.

4 Experimental results: intruder motion

Our intruder study is a thermal cycling analog of experiments which study segregation induced by vibrating a granular bed. While vibration can cause the intruder to rise or fall within the granular medium [5,6], when placed under thermal cycles the intruders in our experiments either do not move or they sink. As shown in Fig. 3, the relative displacement of brass intruders increases with the cycle temperature, and the 2.54 cm diameter brass intruder sinks more than the 1.91 cm diameter brass intruder at the same cycle temperature. The effect of intruder size, seen clearly in Fig. 3, was studied by using brass intruders of different diameters. Figure 4a shows the size dependence of the relative displacement of these intruders after single thermal cycles to 76°C. No relative

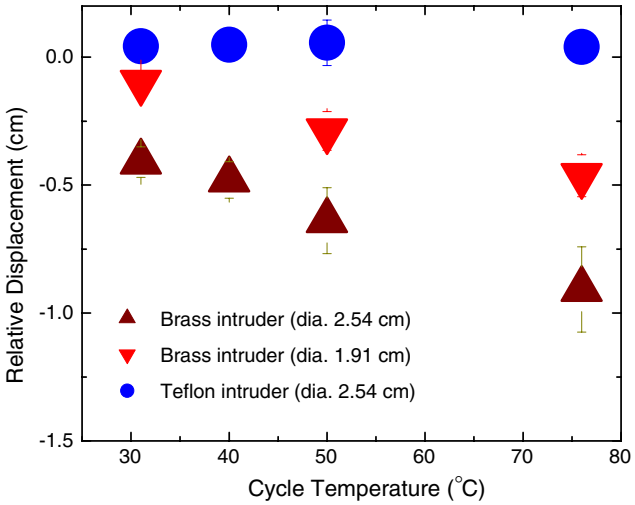

Fig. 3 Relative displacement between the intruder and the pile surface as a function of cycle temperature for a single thermal cycle. Teflon (2.54 cm diameter, *blue circle*) and brass (1.91 cm diameter, *red triangle*, and 2.54 cm diameter brown triangle) intruder were thermally cycled polystyrene grain pack (Color online)

displacement was observed for intruders of the smallest size, but we observe an increase in the magnitude of the displacement as the diameter is increased. As we show in Fig. 4b, the relative displacement also depends on the intruder density, which was varied by loading 3.81 cm diameter hollow aluminum intruders with metal shot. Intruders with the lowest density have zero relative displacement, but the displacement starts to increase with intruder density for densities above a threshold (approximately 2 g/cm^3 for the intruder diameter studied). This agrees with the observed zero relative displacement from a 2.54 cm diameter Teflon intruder with a density of 2.2 g/cm^3. When placed under multiple successive thermal cycles, intruders continue to sink as shown in Fig. 5, while the increment of displacement decreases with cycle number. The displacement of the pile can be well fitted to a double exponential decay function ($y_0 + A_1 e^{-x/t_1} + A_2 e^{-x/t_2}$) similar to the packing relaxation of granular materials under multiple thermal cycles, where y_0 is the saturation depth the intruder can reach under thermal cycles [14]. This suggests that there are at least two different relaxation processes at work (as in the case of packing fraction relaxation [14]), the relaxation of single grains and the relaxation of blocks of grains [2]. Single grain relaxation occurs when the deformation of the grains creates enough room to allow a single grain to relocate, and the block relaxation describes the collective settling of a block of grains without relative rearrangement within the block.

Our observed displacement of heavy intruders in a granular pile appears essentially to be packing fraction relaxation under an overload. Furthermore, the dependence of relative displacement on both the intruder size and density

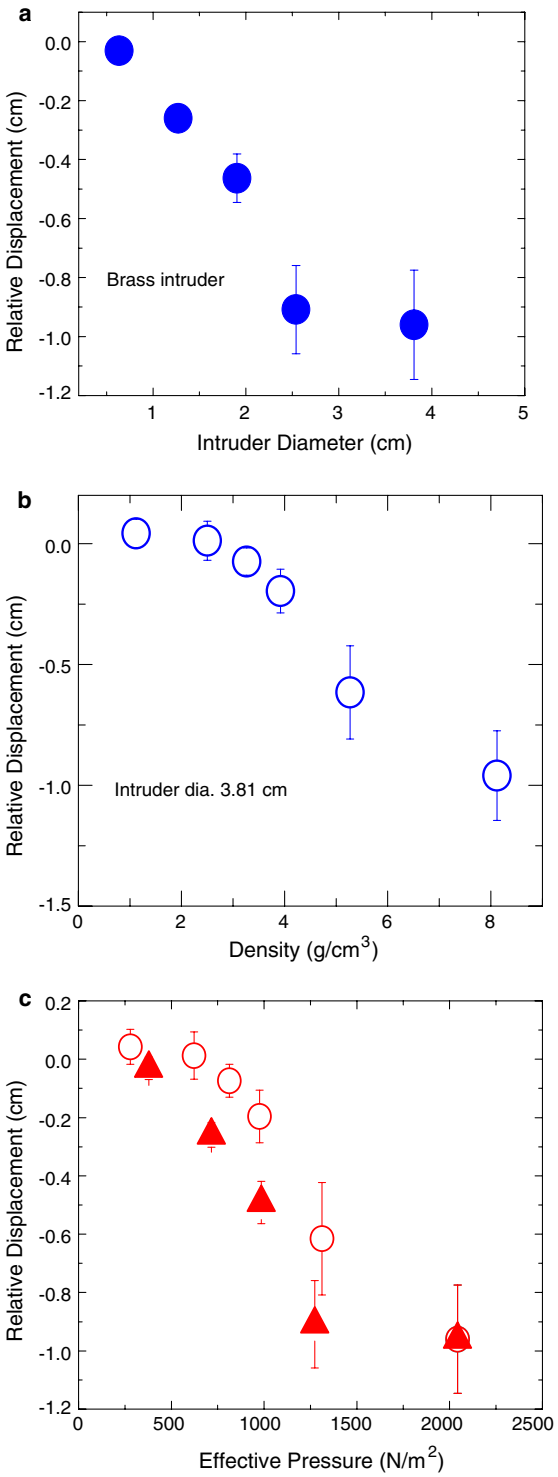

Fig. 4 Relative displacement as a function of intruder size and density **a**. Relative displacement between the intruder and the pile surface as a function of intruder size for cycle temperature of 76°C. **b**. Relative displacement between the intruder and the pile surface as a function of intruder density. **c**. Relative displacement between the intruder and the pile surface as a function of effective pressure. Filled triangles are data from (**a**) and open circles are data from (**b**). The pressure is calculated as the ratio between the intruder weight and the cross-section area (Color online)

suggests that the effective pressure, i.e., the ratio between the weight of the intruder and the cross-sectional area of the sphere, may be the essential parameter of intruder displacement under thermal cycles as shown in Fig. 4c. Each grain in the granular pack is in a static equilibrium state as a result of forces from contacting grains. This creates a contact network that supports the weight of the grains and any external load placed on the pack. When the pressure from the intruder is low, the granular pile is able to support the intruder without having to "rearrange" the contact network supporting the intruder. This elastic [21] response occurs despite the overall increase in packing of the grains with thermal cycling, which is independent of the intruder. When the pressure from the intruder increases, extra stress is introduced to the grains below it. Under thermal cycles the motion of these stress-bearing grains leads to greater relaxations than those of grains that are not supporting the weight of the intruder, resulting in a displacement of the intruder relative to the granular pile. For a given cycle temperature, the magnitude of the relative displacement is determined by the volume of grains affected by the extra stress introduced by the intruder. The data show that the displacement only occurs when the density exceeds a certain threshold, and that it is subsequently proportional to the effective pressure of the intruder with increasing intruder density. This proportionality makes physical sense because the jamming nature of granular packing limits the depth the extra stress can penetrate, and heavier intruders affect grains further from the intruder surface [22]. The zero relative displacement in glass piles and plastic containers seems to indicate that the granular packing fraction relaxation within a moving boundary is not homogenous with minimal relaxation at the center of the pile, where it is furthest from the source of perturbation [18]. The frictional force between the intruder and the grains does not seem to affect the intruder displacement significantly so long as the intruder size is much greater than the grain diameter. The intruder is supported by a network of contacts below it, and the motion involves the rearrangement of all those grains intead of only those in immediate contact with the intruder. Since the packing fraction increases with successive thermal cycles [14], the rearrangement of grains under the intruder is also inhibited with more cycles as shown in Fig. 5. This strongly suggests that the intruder motion in thermally cycled granular piles stems from the packing fraction relaxation of the grains under the intruder.

5 Conclusions

Our observed packing fraction relaxation and the displacement of an intruder in thermally cycled granular media presumably result from small perturbations that propagate in

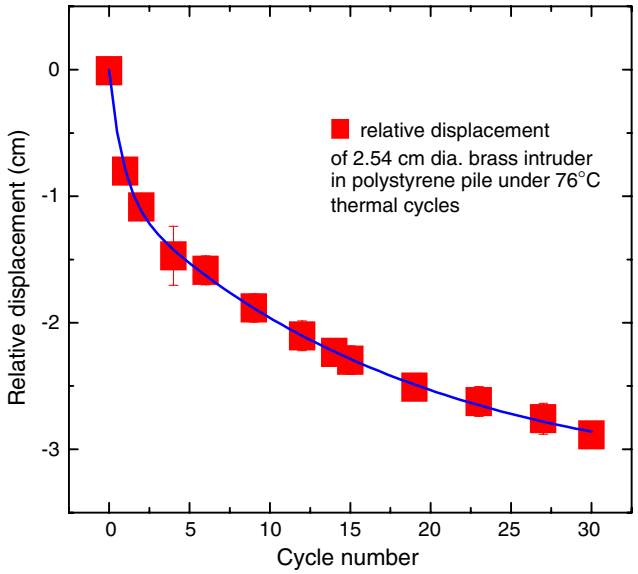

Fig. 5 Intruder displacement under multiple thermal cycles to 76°C (this temperature is chosen to be as large as possible without risking plastic deformation of the grains). Relative displacement of 2.54 cm diameter. brass intruder under multiple cycles; the displacement of the intruder can be fit to a double exponential decay function $y_0+A_1e^{-x/t_1}+A_2e^{-x/t_2}$ with fitting parameters $y_0 = -3.3$, $A_1 = 0.97$, $t_1 = 0.87$, $A_2 = 2.34$, and $t_2 = 18.03$) (*blue solid line*) (Color online)

the system, creating and destroying contacts in the network of intergrain contacts [11]. The data show that thermal processes provide a tool through which granular response to very small perturbations can be probed. The detailed mechanisms through which microscopic thermal changes in grain size can trigger motions several orders of magnitude greater in the bulk material deserves further theoretical, numerical [15], and experimental investigation. Experimentally, real-time photo-elastic imaging or confocal microscopy study of granular contact networks under thermal cycles or thermal shocks may shed light on the evolution process of thermally cycled granular media [23].

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