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FAST TRACK COMMUNICATION

Linear and nonlinear rheology of dense emulsions across the glass and the jamming regimes

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Abstract
We discuss the linear and nonlinear rheology of concentrated microscale emulsions, amorphous disordered solids composed of repulsive and deformable soft colloidal spheres. Based on recent results from simulation and theory, we derive quantitative predictions for the dependences of the elastic shear modulus and the yield stress on the droplet volume fraction. The remarkable agreement with experiments we observe supports the scenario that the repulsive glass and the jammed state can be clearly identified in the rheology of soft spheres at finite temperature while crossing continuously from a liquid to a highly compressed yet disordered solid.

S Online supplementary data available from stacks.iop.org/JPhysCM/25/502101/mmedia

(Some figures may appear in colour only in the online journal)

An amorphous solid is a state of matter that has both liquid- and solid-like attributes: it has a liquid-like structure, yet mechanically responds to an applied stress as a solid. The conceptually simplest amorphous solid is composed of monodisperse spheres that are non-interacting at a distance yet are repulsive when they make contact [1–5]. The repulsive interaction potential at contact can be *soft* or *hard*. Upon rapidly quenching a disordered system above a sphere volume fraction of $\phi \sim 0.6$, a supercooled liquid of spheres enters an amorphous phase, since crystallization is suppressed. Despite decades of intense research on the nature of this transition, it is still not fully understood [6]. In the zero-temperature limit, there are no entropic contributions to the free energy and solidity emerges simply from what is known as *random close packing* or the *jamming* transition [2, 3, 7]. The location of this transition is well established at $\phi_J \simeq 0.64$ and it plays a key role in the properties of granular spherical packings from the micro- to the macro-scale. At finite temperatures, however, an additional phase boundary at densities several percentile below jamming, the ergodic to nonergodic glass transition ϕ_g, has been conjectured by mode coupling theory (MCT) and as an explanation of light scattering experiments and for hard-sphere systems [4, 8–10]. In this scenario a nonergodic *glass* is a weak amorphous solid having a density $\phi_g < \phi < \phi_J$ [4, 8–10, 5]. An ideal MCT glass is a solid that does not

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relax in time. In reality a glass has a finite viscosity (and α-relaxation in light scattering) that evolves in time, or ages, but the modulus and yield stress will not, for the repulsive (soft and hard) colloidal spheres considered here over relevant time scales.

While many detailed experimental and theoretical studies can be found as ϕ approaches ϕ_g from the liquid side [4, 8–13], quantitative studies in which ϕ crosses both ϕ_g and ϕ_J are scarce [14]. The very existence of a solid glassy phase has been questioned, and it has even been suggested for hard spheres that the glass and jamming transition are the same $\phi_g \equiv \phi_J$ [15]. Results obtained from experiments on only one type of experimental system have not yet been able to unravel the general question, mainly because it is very difficult to determine exact volume fractions of submicron sized particles [16], as a result of non-zero nanoscopic length scales associated with stabilization. Moreover, the very concept of hard-sphere-like systems has been questioned recently [17] due the difficulty in designing colloids having a sufficiently sharp repulsive interface.

In this letter, we study the elastic shear modulus and the yield stress of a colloidal model system of (sub)microscale soft repulsive spheres. We discuss the experimental evidence for a solid glass and a jammed state of dense uniform emulsions. Moreover we demonstrate that applying simple theoretical concepts to these technically relevant systems provides accurate quantitative predictions about their linear and nonlinear mechanical properties.

An emulsion is a dispersion of droplets of one liquid in a different immiscible liquid [18]. The nano- to micrometer sized droplets of the dispersed phase can be kept almost indefinitely in a metastable state by decorating their interfaces with amphiphilic surfactant molecules. In emulsions, because of the existence of a thin film of continuous phase between droplets, lubrication is present, so solid–solid friction cannot play a role. In contrast to compressible star polymers or microgels [14, 20, 13, 19], which do not have volume fractions that are precisely defined, in an emulsion, the droplet volume fraction is well defined because the liquid within the droplets is incompressible, even if the droplet interfaces can deform under extreme compression. To prevent droplet coalescence, a short-range, screened electrostatic repulsive interaction, provided by an ionic surfactant, is typically present. For the anionic surfactant, sodium dodecyl sulfate (SDS), at 10 mM concentration in water, the Debye screening length is $\lambda_D \simeq 3.5$ nm [21]. Herein, we interpret measurements of the linear and nonlinear rheology of silicone oil in water emulsions stabilized by 10 mM SDS for droplet radii $R =$ 250, 370 and 530 nm. Using depletion-induced droplet size segregation [22] the distribution of radii R of the droplets can be made quite uniform, $\Delta R/R \sim 10$–12%, while keeping the polydispersity sufficiently high in order to suppress crystallization [8–12]. The experimental data that we consider for the elastic modulus G_p and the yield stress σ_y have been published previously, and technical details can be found elsewhere [23, 18].

To provide perspective, we summarize briefly very recent theoretical predictions for the elastic shear modulus and the yield stress in the glass and in the jamming regimes. Theoretical and numerical work by Ikeda *et al* [25, 26] addresses the glassy dynamics and jamming of soft spheres. The authors lay out a theoretical scenario about how to distinguish between the mechanical response of a *glassy* system and that of a *jammed* system. Extensive simulations on idealized spheres that have harmonic repulsions beyond contact were carried out in order to map out the transition. What sets this study apart is their explicit focus on describing the physics of soft colloids crossing all relevant regimes. Most previous attempts for model colloidal systems have either concentrated on the approach to the glass transition $\phi \leq \phi_g$ [4, 8–13] or on the jamming physics $\phi > \phi_J$ [2, 3, 27, 29–31]. By considering both, Ikeda *et al* find that glassy and jammed colloids display qualitatively different characteristics with respect to their mechanical responses. The details of the transition, however, depend sensitively on the nature of particle elasticity (i.e. repulsive interaction) when particles touch. For very soft spheres, the transition from the glass to a jammed solid is smeared out and phenomenologically disappears [28].

The mechanical properties of the glass are entropically driven and scale with temperature and size as $\sim k_B T/R^3$. Ikeda *et al* suggest the following scaling relation for the yield stress covering the entire glass phase for hard spheres [25]:

$$\frac{\sigma_y}{k_B T/(2R)^3} \simeq c_1 \left[1 + c_2 \frac{(\phi - \phi_g)^{0.7}}{(\phi_J - \phi)^{0.8}} \right] \tag{1}$$

with constants c_1 and c_2 of order unity. MCT calculations for both the yield stress and the modulus are consistent with this scaling, the parameters c_1 and c_2 however strongly depend on the specific approximations made [32–34]. The second term in equation (1) takes account of the expected divergence of σ_y close to the jamming of hard spheres [35, 25]. Since the divergence is avoided for soft spheres, the transition must be somewhat smeared, as it is also apparent in simulation results [25].

We now turn our attention to the packing fraction dependence of the shear modulus above the jamming threshold $\phi > \phi_J$. In the jamming scenario the modulus is given by a product of the bond strength k and the excess number of contacts [2, 3] which has been found to scale as $\Delta Z \propto \sqrt{\phi - \phi_J}$ [36, 37] and thus

$$G_p = a_1 \frac{k}{\pi R} \sqrt{\phi - \phi_J} \tag{2}$$

where a_1 is a constant of order unity [2, 3, 19, 38, 39]. Note that $\pi R G_p$ is the spring constant of a particle harmonically bound in a matrix having G_p [40–42]. When applying a sufficiently large stress σ_y the sample will yield and subsequently flow. Recent simulation work suggests $\sigma_y \sim k(\phi - \phi_J)^{1.2}$ [43, 44][4] which in turn provides expressions for

[4] In the supplemental material, figure S3 (available at stacks.iop.org/JPhysCM/25/502101/mmedia), we compare the model predictions for slightly different scaling exponents, 1 and 1.5, also reported in the literature for harmonic spheres [45, 46].

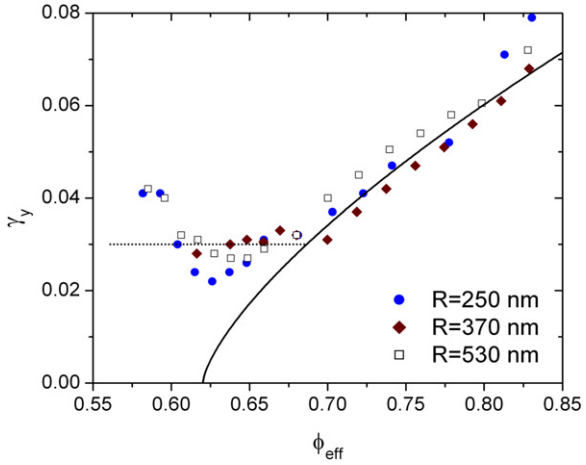

Figure 1. Yield strain γ_y as a function of the effective volume fraction (data from [23]). Dotted line: constant value of 0.03. The solid line shows the prediction from equation (4) with $a_2/a_1 = 0.2$, and $\phi_J^{eff} = 0.62$.

the yield stress and the yield strain, where a_2 is a constant

$$\sigma_y = a_2 \frac{k}{\pi R}(\phi - \phi_J)^{1.2} = (a_2/a_1)G_p \times (\phi - \phi_J)^{0.7} \quad (3)$$

$$\gamma_y \sim \sigma_y/G_p = a_2/a_1(\phi - \phi_J)^{0.7}. \quad (4)$$

We first compare the theoretical predictions with experimental data for the yield strain, figure 1. The yield strain should be scale invariant and thus the data for different sizes should collapse on a master curve. As shown previously, this is indeed the case for microscale droplets provided the bare droplet volume fraction is converted to an effective volume fraction $\phi_{eff} = \phi(1 + h/2R)^3$, where a ϕ-dependent effective thickness $h \sim 10$ nm of the interfacial layer is used to account for screened electrostatic interactions between droplet interfaces [23, 18]. Moreover the yield strain should be independent of the bond strength according to equation (4). Indeed, as shown in figure 1, well above ϕ_J the yield strain rises and the data is consistent with the scaling predicted by equation (4) with $a_2/a_1 \sim 0.2$.

We note that while the concept of an effective volume fraction captures rather well the influence of the interfacial layer for microscale droplets, this approximation is certainly not perfect [24]. This explains for example why the experimental effective jamming density $\phi_J^{eff} \simeq 0.62$ [23] is found to be slightly lower than the expected value 0.64 (see also figure 3). The effect is even more pronounced for nanoscale droplets, $R \leq 100$ nm, where the size begins to approach the Debye screening length; in this limit disordered nanoemulsions can become elastic solids at bare volume fractions, as low as about $\phi \simeq 0.2$ [24].

Over $0.60 < \phi_{eff} < 0.68$ the yield strain data are approximately constant, as figure 1. At the very lowest measured $0.58 \leq \phi_{eff} \leq 0.60$, well below ϕ_J^{eff} and very close to measurements of ϕ_g, γ_y is reported to be slightly larger, which would indicate a different scaling of G_p and σ_y when approaching the jamming transition (i.e. a different c_1 and c_2 parameter in equation (1), such that the modulus would rise faster than the yield stress approaching ϕ_J^{eff}). However this observation could also be due to an increasing uncertainty in the line intersection method on the log–log plot of stress versus strain, which defines the yield point, as the emulsion's low-strain response begins to transition from dominantly elastic to dominantly viscous [23]. Given the uncertainty in the experiments and analysis, it is reasonable to say that the yield strain is approximately constant for ϕ_{eff} between ϕ_g and ϕ_J^{eff}, at about 0.030 ± 0.005. The same value for σ_y/G_p has been reported by Ballauff, Fuchs and co-workers in a careful study of microgel core–shell particles approaching the glass transition from below [47].

We now return to the discussion of the modulus and the yield stress of an emulsion. The scaling relations equations (2) and (3) have been suggested for harmonic spheres having pair-wise interactions described by $V(r) = \epsilon(1 - r/2R)^2$, where the spring constant is $k = \epsilon/2R^2$ [2, 3, 38, 39, 25]. We make the assumption that these relations can also be applied to the case of emulsions but we allow the bond strength k to depend explicitly on the center-to-center distance r. The bond strength can be calculated by taking the second derivative of the effective interaction potential $V(r)$ between two emulsion droplets in the jammed state [36, 37],

$$V(r) = 3^{-\alpha}\epsilon[(2R/r)^3 - 1]^\alpha. \quad (5)$$

In the limit of small droplet compression ($1 - r/2R \ll 1$) the potential reduces to the more common form $V(r) = \epsilon(1 - r/2R)^\alpha$, $\epsilon = 2C\gamma R^2 3^\alpha$. Here γ is the surface tension. For our case $\gamma = 9.8 \times 10^{-3}$ N m^{-1} and the characteristic constants are $C \simeq 0.36$, $\alpha \simeq 2.32$ [36, 37]. For example for $R = 250$ nm, $\epsilon \simeq 1.4 \times 10^6 k_B T$. We find for $k(r) = \partial^2 V(r)/\partial r^2$,

$$\frac{2R^2 k(r)}{\epsilon} = \frac{6\alpha}{3^\alpha}\left(\frac{2R}{r}\right)^5\left[\left(\frac{2R}{r}\right)^3 - 1\right]^{\alpha-1} + \frac{9\alpha(\alpha-1)}{2\cdot 3^\alpha}\left(\frac{2R}{r}\right)^8\left[\left(\frac{2R}{r}\right)^3 - 1\right]^{\alpha-2}. \quad (6)$$

In a bulk emulsion the interdroplet distance r is set by the droplet number density which in turn is related to the volume fraction occupied by the oil droplets and thus $\phi \propto r^{-3}$. At the jamming transition ($\phi_J \simeq 0.64$) the droplets are in direct contact $r = 2R$ thus $\phi(r) = \phi_J(2R/r)^3$ and we can write $r/2R \simeq (\phi_J/\phi)^{1/3}$ [27]. Combining equations (6) and (2) provides a simple expression for the shear modulus,

$$\frac{G_p}{\gamma/R} \simeq 6a_1[\phi^{8/3}(\phi - \phi_J)^{0.82} + \phi^{5/3}(\phi - \phi_J)^{1.82}]. \quad (7)$$

For an estimate of the prefactor a_1 in equation (7) we compare our model to simulation results for G_p reported by Lacasse *et al* [36]. For the comparison we have slightly rescaled the ϕ-values of the simulation data by a numerical factor of 0.978 to have them extrapolate to the same critical value $\phi_J = 0.64$. We find excellent agreement for $a_1 = 0.25 \pm 0.01$ as shown in figure 2. This also sets an

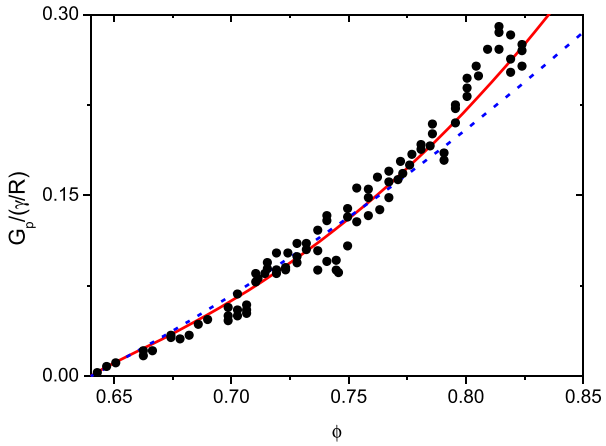

Figure 2. Comparison of equation (7) (solid line, $a_1 = 0.25$) with numerical simulations of the shear modulus G_p (symbols) [36]. Dotted line: scaling $G_p \simeq 1.6\phi(\phi - \phi_J)$ as reported in [49].

estimate for the yield stress parameter $a_2 = 0.2a_1 \simeq 0.05$.[5] Remarkably, equations (3) and (7) now provide quantitative analytic predictions for the shear modulus and yield stress of emulsions that can be tested directly against experimental data. In figure 3 we compare the predictions in the jammed state, equations (3) and (7), with the experimental data for the shear modulus and the yield stress. The agreement is remarkable given the difficulty of such a quantitative comparison.

While other formulas exist for predicting the modulus and yield stress of a disordered uniform emulsion as a function of volume fraction [23], they are empirically based and have not, up to now, been derived analytically in a microscopic model that properly incorporates the potential of interaction between droplets and the scaling of the coordination number. Moreover, the scaled $G_p(\phi)$ and $\sigma_y(\phi)$ are self-consistent in this model (i.e. use the same microscopic parameters) and provide good agreement with both linear and nonlinear measurements. Earlier experiments on polydisperse emulsion suggested $G_p \sim \phi^{1/3}(\phi - \phi_J)\gamma/R$ [48]. Subsequently it has been shown that the behavior of uniform emulsion is better described by the semi-empirical relation $G_p \sim \phi(\phi - \phi_J)\gamma/R$ [49]. Our predictions, based on the correct scaling of the excess number of contacts ΔZ, are very close to these predictions (i.e. within about $\pm 10\%$) up to densities of about $\phi \sim 0.85$. For $\phi \to 1$ our model extrapolates to $G_p \times (R/\gamma) \simeq 0.88$, a value slightly larger than reported in [49], yet this value satisfyingly matches a calculation for dry foams and emulsions [50][6].

[5] Simulations for harmonic spheres in the zero-temperature limit [25] are well described by equation (3) with a yield stress parameter $a_2 \simeq 0.03$, in fairly good agreement with the estimate reported here. The remaining difference might be due to the different interaction potentials, the different particle size distributions, and due to difficulties in assessing absolute values of the yield stress experimentally with high precision.

[6] We note that for simplicity we have neglected an additional weak concentration dependence of $r \sim \phi^{0.09}$, suggested in [27], and of α and C in the interaction potential $V(r)$ as discussed in [36]. Both effects are due to the rise in the number of contacts Z upon compression and could easily be incorporated in the model.

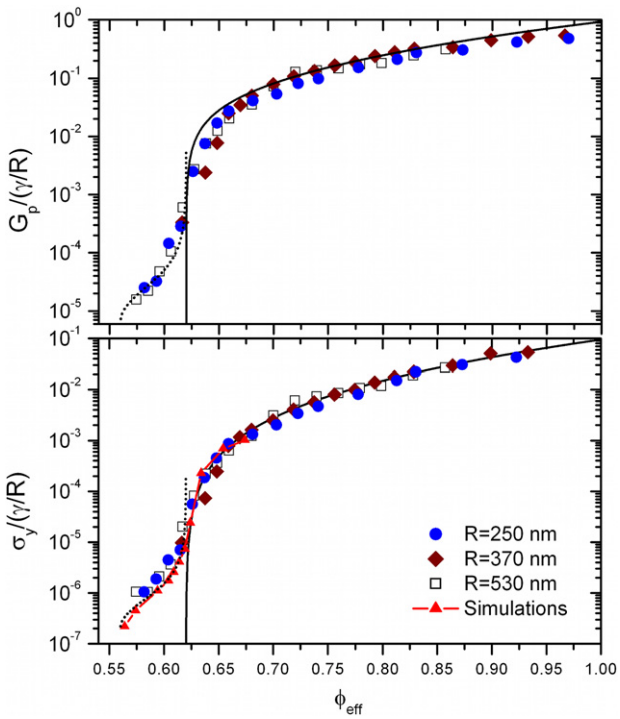

Figure 3. Upper panel: shear modulus of emulsions with radius $R = 250$, 370 and 500 nm (data from [18, 23]). Lower panel: yield stress for the same samples. Dotted lines: scaling predictions for yield stress in the glass phase, equation (1), with $c_1 = 0.25$, $c_2 = 2.5$ and for the modulus $G_p(\gamma/R) = \sigma_y/0.03$. Solid lines: theoretical predictions in the jammed state, equations (3) and (7), with $a_1 = 0.25$, $a_2 = 0.05$ and $\phi_J^{eff} = 0.62$, $\phi_g = \phi_J^{eff} - 0.06 = 0.56$. Solid triangles: simulation results for harmonic spheres with $k_BT/\epsilon = 10^{-6}$ (data reproduced from figure 2(b) [25]).

Next we address the properties in the glass state and the transition from the glass to the jamming physics, which is governed by the particle softness. Ikeda's measure of softness is given by $T_e = (k_BT)/\epsilon$ [25]. Computer simulations for harmonic spheres show that for values larger than 10^{-4} (very soft spheres) the glassy phase narrows and the transition is smeared out to an extent that in an experiment or simulation glassy physics and jamming cannot be distinguished any more. For our droplets $T_e \sim 10^{-7}$–10^{-6} which should allow us to distinguish both regimes. The glass physics and the onset of jamming should not depend on the details of the interaction potential. We can thus attempt to compare the simulation results for $T_e = 10^{-6}$ reported in [25] with the experimental data for the yield stress σ_y. Note that the numerical data in [25] is given in units of $\epsilon/(2R)^3$ while our data is normalized by γ/R. For our emulsion droplets however $(\epsilon/(2R)^3)/(\gamma/R) \simeq 1$ and thus the comparison can be done directly. To match the jamming transition of the emulsions we slightly shift the numerical data such that $\phi_J^{eff} = 0.62$ in both cases. As shown in figure 3 the agreement is excellent all the way from the glass to the jamming regime. Moreover, comparing the numerical data with equation (1), we can estimate $c_1 \simeq 0.25$ and $c_2 \simeq 2.5$ using again $G_p \simeq \sigma_y/0.03$ in the glass (dotted lines in figure 3).

Despite the excellent agreement reported here it is worth mentioning that over the range of sizes covered

the experimental data do not scale with the droplet size as suggested by entropic origin of the glass elasticity, equation (1). We are unable to discern whether this is due to limitations of the theory, or due to the experimental difficulties in determining such small values with sufficient accuracy, or both. To resolve this question additional experiments covering a larger range of droplet sizes will be required in the future.

Notwithstanding the above, the present work suggests that the glass and jammed state are well distinguishable for experiments on uniform emulsions. Both the modulus and the yield stress are characterized by a sharp rise close to the jamming transition. This indicates that the smearing of the transition is limited and our emulsion droplets indeed behave as soft spheres with a sufficient stiffness ϵ/k_BT or bond strength. In reality, emulsion droplets stabilized by an ionic surfactant are slightly more complicated. The presence of the Debye layer will soften the droplet interactions. However, as droplets come into contact, the Debye layer is rapidly compressed and the stiffness increases until it approaches the value ϵ/k_BT of the elastically coupled droplet core [24]. Our results suggest that this process takes place over a very limited range of concentrations for the droplet sizes $R \geq 250$ nm considered here. The situation might be different when considering smaller droplets [24] or particles having a different interaction potential.

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