Transient filamentous network structure of a colloidal suspension excited by stepwise electric fields

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Jamming and force networks observed in electrorheological (ER) fluids bear many similarities to those observed in various granular and colloidal systems. We have measured the time evolution (transient stresses) of filamentous networks of colloidal particles in suspensions subjected to continuous tensile strain concomitant with the switching on and off of electric fields. The density of particle chains was found to increase exponentially with the applied tensile strain via a rapid formation of single chains followed by a slower coarsening (aggregation) of the chains. The two processes can be ascribed to the field-induced short-range and long-range interparticle forces, respectively, along with the tensile viscous force.

I. INTRODUCTION

The intriguing nature of jammed structures and networks in granular materials has recently attracted much interest [1–5]. When a granular material undergoes a jamming transition and/or exhibits a finite yield stress, significant changes also occur in the viscosity and nature of the flow. Although jamming is considered to be a bulk property, it is accompanied by local changes in the granular networks and interparticle forces. The force networks observed in jammed systems have been studied experimentally and theoretically [2,3] and jamming phase diagrams for attracting particles have been given [4,5]. Both the adhesion and friction forces between particles appear to be involved in determining the networks, and have been studied using various theoretical models for the interparticle interactions [6]. Although the jamming conditions and the equilibrium structures in granular materials and colloidal suspensions have been widely studied, the dynamics, time evolution, and transient behavior of (jammed) networks has not. Jamming processes are usually rapid and nonlinear, and the dynamics of these processes, e.g., of the changing three-dimensional network, are difficult to observe directly. Also, precisely repeatable measurements are often hard to conduct on granular systems adding to the difficulty of studying these systems.

Fortunately, there is a class of colloid suspensions in which the interparticle interactions can be well controlled by applying an external electric field. Such particles usually form a network of filaments oriented along the field direction, which are similar to the networks observed in jammed granular systems. The formation of structures composed of chains leads to an increase in the bulk viscosity, and to a more "solidlike" behavior of the suspension. Such suspensions are known as electrorheological (ER) fluids, which are noted for rapid (10 ms) and reversible changes of their bulk viscosity, often by several orders of magnitude, upon the application of a high external field [7]. The dynamic restructuring processes of the ER fluid studied here, which are simi-lar to the transient jamming processes occurring in granular materials, occur over relatively short time scales, on the order of seconds or minutes. Further, and much slower, structural transitions, on the order of tens of minutes or hours, have recently been reported in such systems [8]. The mechanical performance of ER fluids is widely recognized to be related to the structures of both single chains and bundles or columns of these chains [7], some of which can be crystalline [9]. The single chains are formed by the short-range forces between the particles, while the bundles or columns are formed by the long-range forces [7,10]. The strength of chain bundles has been found to be higher than the sum of the individual (isolated) chains [11]. Upon the sudden application of an electric field, ER fluids undergo transient stress changes, especially when sheared [12–17] or compressed [18]. The behavior of ER fluids subjected to static tensile forces has also been studied [19,20].

There are many similarities in the conformations of particles, chains, or fractal networks of jammed granular materials, colloidal suspensions, and ER fluids [2,7]. In this investigation, we have measured the transient tensile stresses and time evolution of filamentous networks upon the sudden application or switching off of external electric fields, and have compared the ER fluid behavior with the jamming in granular and colloidal systems. The fundamental molecular, interparticle, or "colloidal" forces (e.g., van der Waals, double layer) in all these systems are the same. However, both granular materials and ER fluids are subject to strong additional external forces: gravitational (buoyancy) forces in the former case, and electric field forces in the latter (other systems can have magnetic forces). These external forces give rise to ordering and jamming effects, and while the external forces are different in each case, their effects are often very similar. Our aims are not to model granular systems, but to better understand alignment and jamming effects in ER fluids, and how these may be similar to those observed in other systems.

In the experiments described here, an external electric field was suddenly applied to a previously stressed ER suspension (in tension) to abruptly increase the interparticle forces and drive the suspension into the jammed state. The

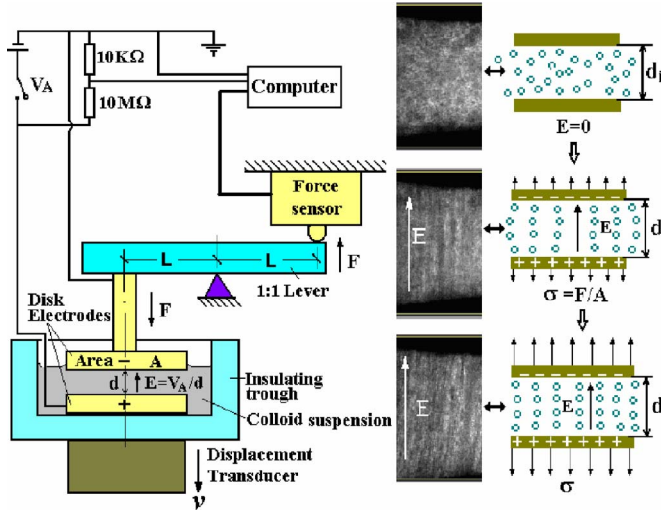*Email address: Jacob@engineering.ucsb.edu

FIG. 1. (Color online) Schematic of the experimental system. The colloidal suspension was confined between two parallel disk electrodes of diameter 2.0 cm (area $A=3.14$ cm^2) and mean surface roughness <1 μm. The lower electrode surface could be moved up or down at a velocity v using a computer-controlled motor, and an electric field E could be applied across the gap d.

transient rheological changes as the system entered the jammed state were measured, and the time evolution of the chain density during the jamming processes determined. Two modes were found for the rate of chain density increase; the first corresponded to the formation of individual chains, the second to the aggregation of individual chains into larger chain bundles (coarsening). These two processes are attributed to the action of the short-range and long-range interparticle forces excited by the applied external electric field, respectively, along with the viscous tensile force. This is also the first report on the transient tensile behavior of ER suspensions.

II. EXPERIMENT

The ER fluid was a suspension of micron-sized (diameter $1-8$ μm) spherical surface-modified zeolite particles with high conductivity ($C_p=1\times10^{-4}$ S m^{-1}) and high dielectric constant ($\varepsilon_p=30\,000$ at 100 Hz), with a volume fraction of $\phi=27\%$, suspended in a silicone oil with a viscosity of $\eta_0=50$ mPa s (20 °C) [12]. The suspension was stable over the experimental time scale—sedimentation occurring only over the course of weeks or months. The suspension was confined between two parallel steel electrode disks in a bathing chamber (Fig. 1) and could be put under tension by moving the chamber (and the lower electrode disk) downwards. The tensile force was recorded by a rigid force sensor (Kistler, Model PCB209B02) with a measuring range of $0-10$ N ($0-1$ kg), a sensitivity of 5 mN, and a response time of 5 μs. The upper disk was fixed at one end of a 1:1 lever to convert the tensile force between the surfaces to a compressive force acting on the force sensor.

All experiments were carried out at room temperature, 23 °C. In the experiments, the initial gap distance between

the disks was set at $d_0=1$ mm. First, a dc voltage, V_A, between 1200 and 1800 V, was applied across the suspension for >20 sec, during which time the colloidal particles were seen to rearrange (Fig. 1) but neither the stress nor the gap width d_0 changed. Then the lower surface was moved downwards at a constant speed of $v=0.2$ mm s^{-1} for 5 sec. The instantaneous gap distance was therefore $d=d_0+vt$. The motion was stopped when d reached $d=2$ mm. During each 5 sec run, the voltage V_A was repeatedly switched on and off (starting at V_A at $t=0$) at various intervals while the instantaneous voltage and tensile force F were simultaneously recorded. The instantaneous tensile stress is given by $\sigma=F/A$, where A is the area of the electrode disks. The instantaneous field is $E=V_A/d$. Taking $i=0$ corresponds to the preapplied voltage V_A, $i=1,2,3,\dots$ corresponds to each subsequent sudden application of the same voltage, ε_i is the instantaneous tensile strain of the ith tension, $\dot{\varepsilon}_i$ is the tensile strain rate, d_i is the gap thickness at the time the ith voltage V_A was applied. The instantaneous tensile strain is

$$\varepsilon_i = (d - d_i)/d_i = vt/d_i, \tag{1}$$

and the tensile strain rate is

$$\dot{\varepsilon}_i = d\varepsilon/dt = v/d_i. \tag{2}$$

III. RESULTS AND DISCUSSIONS

Figure 2(a) shows typical curves of the applied voltage V_A, stepped between 1400 V and zero, and resulting changes in the tensile stress σ, plotted against time t. The inset shows that within 2 ms of switching on the voltage the tensile stress increases roughly exponentially from near zero to a value of $\sigma_{\text{i-s}}$, the stable tensile stress of the tension corresponding to the ith voltage step. after application of the ith V_A, the tension before $\sigma_{\text{i-s}}$ is reached is referred to as the "transient tension." when the applied voltage is turned off, σ decreases rapidly (within 2 ms) to a very low value that is due to the viscous drag force of fluid flowing into the gap from the reservoir as the surfaces are separated at a velocity v. this regime will not be analyzed in this paper; here we concentrate on the near exponential rise of σ in the transient regimes. for the ith transient, σ can be expressed as

$$\sigma_i(t) = \sigma_{\text{i-s}}\left[1 - \exp\left(\frac{t - t_i}{\tau_i}\right)\right], \tag{3}$$

where t is the time, t_i is the time V_A was switched on, and τ_i is the characteristic rise time of the ith tension ($i=1,2,3,\dots$) [cf. inset in Fig. 2(a) where $\tau_i=0.035$ s and $\sigma_{\text{i-s}}=1.31$ kPa for $i=2$]. Figure 2(b) shows the stresses as a function of the gap thickness d for four different voltages V_A ranging from 1200 to 1800 V. The inset shows the characteristic times τ_i as a function of the strain rates, $\dot{\varepsilon}_i$, defined by Eq. (2), at the different voltages tested. The values of τ_i are seen to be independent of V_A and to decrease slightly with increasing strain rate $\dot{\varepsilon}_i$.

During the separation of the electrode surfaces at a velocity v, the purely viscous force per colloid particle is $3\pi\eta_0av'$, where a is the particle diameter, v' the particle
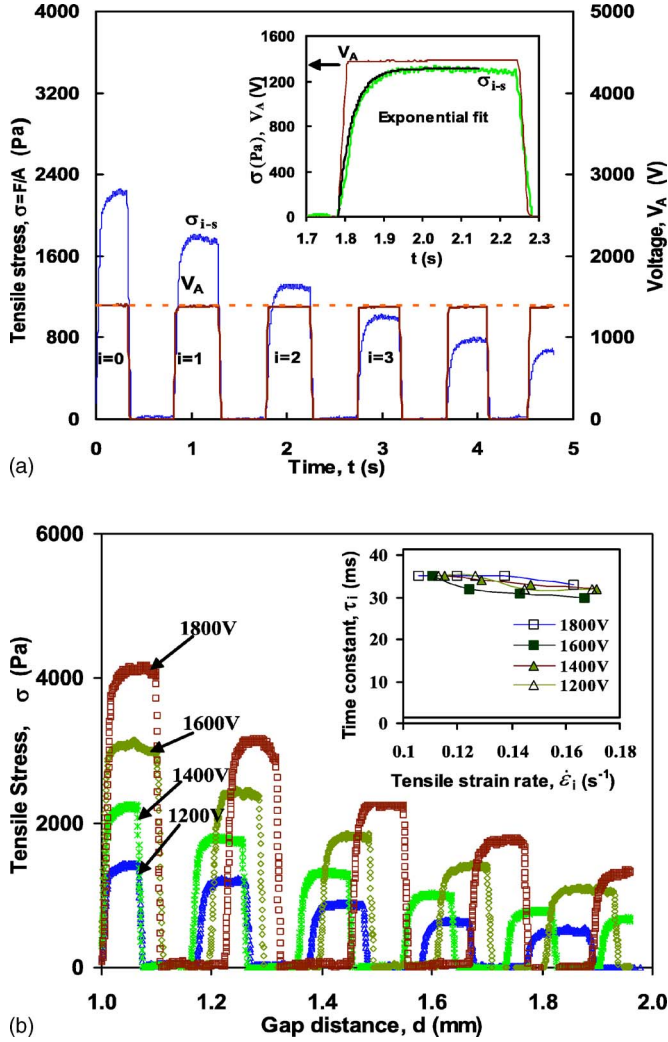
(a)

(b)

FIG. 2. (Color online) (a) Typical curves of tensile stress versus time for a 5 sec run where a voltage of $V_A = 1400$ V was switched on and off six times. Inset: Exponential fit using Eq. (3) to the second transient tension ($i = 2$), giving a characteristic time of $\tau_2 = 0.035$ s. (b) Original four test runs with different applied voltages V_A and the fitted characteristic times τ_i for the different transients (insets). Note that as the gap distance varied from $d = 1.0$ to 2.0 mm in 5 s, the tensile strain rate varied from $\dot{\varepsilon} = 0.2$ to 0.1 s^{-1}, as given by Eq. (2).

velocity (related to v, approximated by $v' \simeq va/d$), and η_0 is the viscosity of the host liquid. This contribution to the transient stress is negligible when the colloidal particles associate into filaments oriented along the applied electric field E, as shown in the middle right panels of Fig. 1.

The transient tensile stresses are determined by the strength of each filament and the number density of filaments in the suspension. The tensile strength of a filament is determined by the attractive force of two neighboring particles along the electric field direction, which is given by [7]

$$F_0 = \frac{3\pi}{16}\varepsilon_0\varepsilon_f a^2 \beta^2 E^2 \left(\frac{a}{a'}\right)^4, \qquad (4)$$

where ε_0 is the dielectric permittivity of free space (vacuum), ε_f is the relative dielectric constant of the suspending fluid, β

is the polarizability of the particles given by $\beta = (C_p - C_f)/(C_p + 2C_f)$ (approximately one in this study because the conductivity of the silicone oil is $C_f \simeq 1 \times 10^{-12}$ S m^{-1}), a is the particle diameter, and a' is the distance between particle centers (which is related to the applied electric field strength, the particle volume fraction, and the particle structure formed in the suspension [7,9]). Equation (4) assumes that the induced dipole moment per particle in the field is βE, and that the interactions between the particles are typical dipole-dipole interactions [7]. The attractive stress between two particles can be estimated as

$$\sigma_c = \frac{F_0}{4a'^2} = \frac{3\pi}{64}\varepsilon_0\varepsilon_f \beta^2 E^2 \left(\frac{a}{a'}\right)^6. \qquad (5)$$

If the chain number density in the surface perpendicular to the tension direction in the suspension is Γ (number per unit area), the transient tensile stress σ is therefore

$$\sigma = \Gamma\sigma_c \quad \text{or} \quad \Gamma = \sigma/\sigma_c. \qquad (6)$$

It should be noted that Eqs. (4) and (5) are valid for $a \ll a'$, and can underestimate the experimental value by over one order of magnitude for the ER strength at high volume fractions. Up to now, no one has succeeded in accurately predicting the particle interaction strength when the yield stress of an ER fluid is above kPa, though many have tried both theoretically and experimentally [7,11,12]. This makes the real chain number density in an ER fluid not obtainable directly from a mechanical measurement. However, Eqs. (4) and (5) do show the basic effects of dipole-dipole interactions in ER fluids. Previous researchers have been mainly concerned with the validity of the "ideal" relationship $\sigma_c \propto E^2$, which is usually not verified by the experiments [7–21]. Instead, Eq. (6) can be modified to

$$\sigma = \Gamma\sigma_c = \Gamma k E^m \quad \text{or} \quad \Gamma k = \sigma/E^m, \qquad (7)$$

where k is a parameter determined by the factors used in Eqs. (4) and (5), except the electric field, and is usually thought to be a constant for a given ER fluid; m is the exponent for the particle interaction strength and the electric field (ideally, $m = 2$, but most researchers have obtained lower values [7–21]). Also, it has been found that ER bundles or columns have a higher strength than the sum of the individual (isolated) chains [11], a result of "many-body" interactions [7,10,21]. Thus the chain number density in Eq. (7) comes from two parts, one is from the single chains which can be determined directly from observations of single chain structures, the other from the long-range many-body interactions of neighboring particles and chains, which changes with time as the structure evolves (e.g., coarsens, or the chains slowly crystallize into ordered columns).

If E and a' are constant, σ_c is constant, then $\Gamma \propto \sigma$. However, the instantaneous field $E = V_A/d$ decreases with time during the tests. Thus σ_c is not constant, so that if we want to determine Γ from measurements of σ during the transient tensions using Eq. (7), the effect of E on σ_c [apart from the E^2 term in Eq. (5)] has to be established. To do this, we plot in Fig. 3(a) the normalized stresses σ/E^2 vs ε for two different values of V_A: 1200 and 1800 V. All the transient tensions
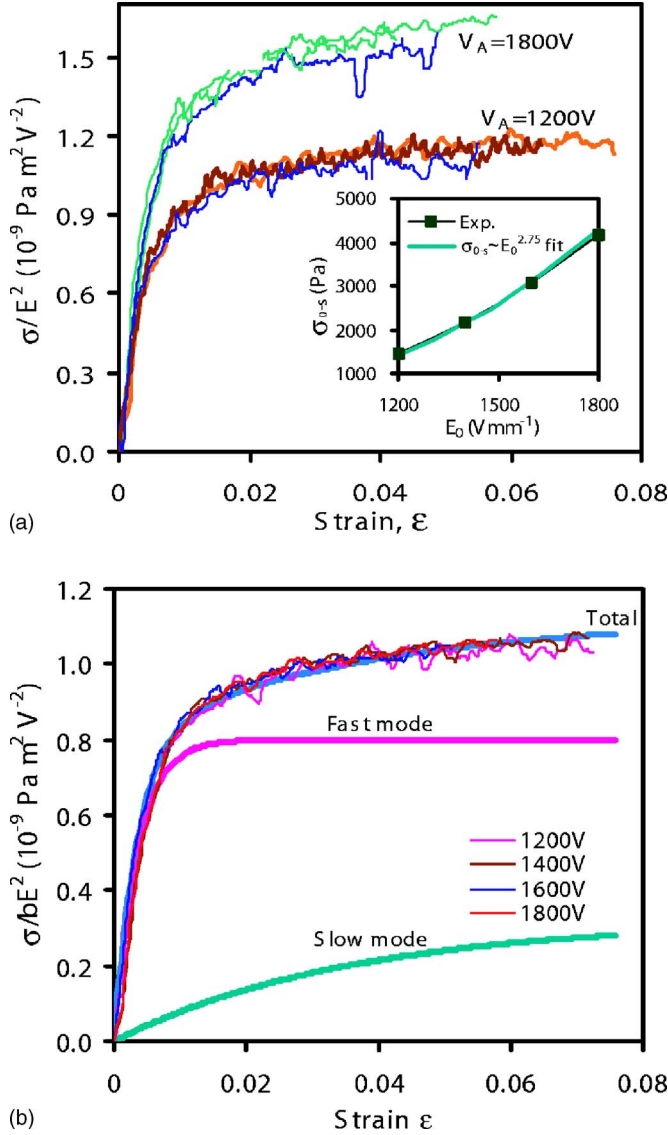
(a)

(b)

FIG. 3. (Color online) (a) Curves of the normalized transient tensile stresses σ/E^2 versus ε for two voltages, $V_A = 1800$ and 1200 V. Inset: $\sigma_{0\text{-s}}$ versus $E_0 = V_A/d_0$ for all the four test runs shown in Fig. 2(b), and the fitted curve of $\sigma_{0\text{-s}} = 4.8 \times 10^{-6} E_0^{2.75}$ ($\sigma_{0\text{-s}}$ in Pa, E_0 in V mm^{-1}) corresponds to a dimensionless factor $b = E_0^{0.75}$ (E_0 in kV mm^{-1}) used in the further normalization. (b) Curves of $\sigma/bE^2 = \Gamma k$ versus ε taken from Fig. 2(b) at four V_A, and three fitting curves corresponding to the single filament forming process (Fast mode), the coarsening process (Slow mode), and their sum (Total), as calculated using Eq. (8) with parameters $\Gamma_1 k = 0.8$ (10^{-9} Pa m^2 V^{-2}), $\Gamma_2 k = 0.32$ (10^{-9} Pa m^2 V^{-2}), $\varepsilon_{c1} = 0.0035$ s, and $\varepsilon_{c2} = 0.036$ s.

for the same V_A agreed reasonably well with each other, and similar results were obtained for $V_A = 1400$ and 1600 V (not shown). These plots show that σ/E^2 does depend on V_A, with $\sigma_{\text{i-s}}/E^2$ increasing as V_A (or E) increases. The increase in stable stresses at higher field strengths could be due to a number of reasons: (i) a memory or hysteresis effect; (ii) the trapping of more particles between the electrodes; (iii) a decrease in a' resulting in an increase in a/a' in Eq. (5). If the stable tensions have an additional, *universal*, dependence on

the initial preapplied electric field of $E_0 = V_A/d_0$, then a plot of $\sigma_{0\text{-s}}/E_0^n E_0^2 = \sigma_{0\text{-s}}/bE_0^2$ against ε for different values of V_A should yield a master curve for *all* the data for some universal value of the exponent n. It should be noted that this n is different from m in Eq. (7). Figure 3(b) shows that for $n = 0.75$ [$b \propto E_0^{0.75}$ or $b \propto V_A^{0.75}$, fitted as shown in the inset of Fig. 3(a)] the data do fall on the same master curve. This indicates the same change in the number density of filaments Γ with tensile strain ε for all the transient tensions in Fig. 2. These findings are similar to other studies of transient processes of ER suspensions which show a weak dependence of characteristic time on the electric field but a strong dependence on the applied strain [15–21].

As shown in Fig. 3(b), each curve can be divided into a rapidly rising part followed by a slower increase. Visual observation of the aggregation of the colloidal particles following the sudden switching on of a field shows a rapid association into single filaments followed by a slower aggregation of the filaments into bundles or columns. The former process is undoubtedly caused by the strong short-range dipole-dipole pair interactions; the second by the long-range forces between the filaments themselves and the increased viscous drag between them [10]. This interpretation agrees with a recent experimental study on the cluster morphology of attracting colloidal particles that formed tenuous and branched clusters for short-range forces, and more compact clusters for longer-ranged interactions [5]. A similar phenomenon occurred in the transient tensions in this study, as shown in the middle column of Fig. 1. The coarsening of single filaments into columns apparently increases the overall strength of the suspension [11,22,23], i.e., the interactions are cooperative, and can be considered as being equivalent to increasing the density of single filaments, Γ. We proposed the following equation to simultaneously describe these two processes

$$\Gamma(\varepsilon)k = \Gamma_1 k(1 - e^{-\varepsilon/\varepsilon_{c1}}) + \Gamma_2 k(1 - e^{-\varepsilon/\varepsilon_{c2}}), \qquad (8)$$

where Γ_1 is the final single chain density for the fast mode with a characteristic tensile strain ε_{c1} that corresponds to interactions between neighboring particles in the same chain, and Γ_2 is the final equivalent chain density for the slow mode with a characteristic tensile strain ε_{c2} that accounts for the long range interactions from particles of neighboring chains. Figure 3(b) shows how well the data of the "master" curve is fitted by Eq. (7) using the parameters of $\Gamma_1 k = 0.8$ (10^{-9} Pa m^2 V^{-2}), $\Gamma_2 k = 0.32$ (10^{-9} Pa m^2 V^{-2}), $\varepsilon_{c1} = 0.0035$ s, and $\varepsilon_{c2} = 0.036$ s. As mentioned above, we do not have an accurate estimate for the constant k of an ER fluid, so the real values of Γ_1 and Γ_2 cannot be obtained through the above scaling. However, since $\Gamma_1 k/\Gamma_2 k = \Gamma_2/\Gamma_1$ is independent of k, this ratio provides information about the *relative changes* of single chains and chain bundles and their short-range and long-range interactions, thus providing the first quantification of the transient tension of ER fluids excited by a stepwise electric field.

The aggregation time depends mainly on the applied electric field, the viscosity of the solvent, and the particle volume fraction of the suspension [10]. Under no external field, the diffusion coefficient at room temperature, $D = k_B T/3\pi\eta_0 a$, is about 8.6×10^{-15} m^2 s^{-1}, corresponding to a displacement x

of about 0.01 μm per particle in one second (using $\langle x \rangle^2 = 2Dt$). Compared to the particle diameter of ~ 1 μm and the volume fraction of ~ 0.3, the particles cannot diffuse to their equilibrium positions in one second at room temperature. Under an electric field, the much stronger attractive dipole-induced forces between the particles quickly drive them together. A simple estimate of this aggregation time can be made as follows: $t_s = 16\eta_0 / \varepsilon_0 \varepsilon_f \beta^2 E^2$ [7], where the particles' inertia and Brownian motion are ignored. Taking $\eta_0 = 50$ mPa s, $\varepsilon_f = 2.5$, $\beta = 1$, and $E = 1.2 - 1.8$ kV mm^{-1}, yields values of $t_s = 10 - 25$ ms. Higher fields, lower solvent viscosity, and higher particle volume fractions should lead to faster coalescence, as found in the quiescent state [10,14].

Examinations of the fast and slow modes have found that the fast mode takes milliseconds, while the slow mode takes hundreds of milliseconds to several seconds [7]. For instance, a direct measurement of the quiescent diffuse transmitted light intensity and the current passing through an ER suspension with a particle volume fraction of 0.33 in a silicone oil with a viscosity of 0.02 Pa s, subjected to a stepwise electric field found characteristic time constants of $4 - 9$ ms for the fast mode and $90 - 150$ ms for the slow mode [17]. Compared to our case with a smaller volume fraction of 0.27 and silicone oil with a higher viscosity of 0.05 Pa s, according to the relationship of $t_s \propto \eta_0 / \phi^{1.5}$ [7], the fast mode in our case at the quiescent state should be about $14 - 30$ ms, and the slow mode $300 - 500$ ms. This estimate of the fast mode is close to the fitted characteristic time constant of $\varepsilon_{c1} = 3.5$ ms (with a total aggregation time of about $4\varepsilon_{c1} = 14$ ms). However, the above estimate of the slow mode is much larger than the fitted characteristic time constant of $\varepsilon_{c2} = 36$ ms (with a total aggregation time of about $4\varepsilon_{c2} = 144$ ms). This discrepancy is due to the effect of the tension flows on the aggregation processes. A mechanical field such as shearing, compression or tension acting on the suspension will act to modify the forces needed to drive the particles to aggregate [13–16]. There are no equations that describe how such mechanical fields affect the aggregation yet; but qualitatively, we expect and show that the tension flow enhances the bundling process while slowing down the single chain forming process.

A ratio of the characteristic times of the fast mode to the slow mode has been reported to be about 0.05, obtained from optical observations [17] under static (no flow) conditions, i.e., in the absence of shear flow induced viscous forces. Our results yield a ratio of $\varepsilon_{c1} / \varepsilon_{c2} \simeq 0.1$ for the tensioned suspension, implying that single filament formation is about ten times faster than the coarsening of the filaments. The increase of the characteristic time ratio shows the effect of the tensile flow on the slow and the fast modes. Also, the ratio of $\Gamma_2 / \Gamma_1 = 0.4$ means that the chain coarsening of the slow mode increases the mechanical strength of the suspension by 40%. This increase is usually ascribed to many-body effect or structural effects [7,21] and is quantified in this investigation as an equivalent to forming 40% more single particle chains in the suspension, which cannot be obtained from direct observations.

Some of our results can be compared with similar studies of the jamming of granular materials or colloidal suspen-sions. For instance, the overlapping of the normalized stress curves in Fig. 3 agree with recent molecular dynamics simulations by Ostojic *et al.* [2] who found a scale invariance and universality of force networks in static granular materials. For various values of the confining pressure and other parameters the networks were found to have identical scaling exponents and scaling functions. The simulations thus showed that the structural changes are independent of the details of the interparticle potentials. However, if other forces are comparable to the interparticle potentials, this invariance might be changed, i.e., if the tensile flow is fast, the evolution of the structure will be significantly modified from that obtained in this investigation.

Continuing our comparison between ER fluids and granular materials, the particle interaction potential in ER fluids can be easily modulated by many orders of magnitude by changing the applied electric field strength, which can change the mechanical properties of the whole suspension from a liquid to a solid. The shear yield stress of this kind of solid can be as high as 200 kPa [24]. The interparticle potential can be further manipulated by external mechanical fields arising from shearing, compression, and tension. The interparticle force in ER fluids along the electric field direction is usually much higher than that in other directions. In contrast, in the jamming of granular materials or colloidal suspensions, the interactions are usually not so strongly anisotropic. It is possible to apply more than one kind of external field to a suspension of particles (ER, colloidal, or granular) simultaneously, such as electric field and magnetic field, so as to further control both the strength and directionality of the interparticle interactions [25].

IV. CONCLUSIONS

The transient tensile stress of an ER fluid has been measured and normalized to obtain the time evolution of filamentous networks of colloidal particles in the suspension subjected to stepwise electric fields. The density of particle chains was found to increase exponentially with the applied tensile strain via a fast formation of single chains and a slow chain coarsening process. Those structure changes are ascribed to the electric field-induced short-range and long-range particle interaction potentials, respectively. We compare our results to recently reported studies on granular or colloidal systems where similar chain networks and jamming were observed. ER fluids are shown to be good model systems for granular materials and colloidal suspensions, with the added advantage of the high flexibility of controlling interparticle potentials over a wide range.

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