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Universal Relationship between Linear Viscoelasticity and Nonlinear Yielding in Soft Materials

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The nonlinear response of yield stress fluids remains difficult to predict and control. Here, we show that the height of the overshoot in the loss modulus G'', a key characteristic of yielding, depends only on linear viscoelastic properties. Furthermore, the position of this overshoot depends on linear viscoelastic and flow properties, demonstrating the important and enduring role of elasticity in yielding. The physics governing linear viscoelasticity is therefore not only preserved during yielding but also controls two commonly reported yielding metrics.

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Yielding is a nonlinear physical phenomenon in which soft materials transition from deforming recoverably to unrecoverably under an applied load. Yield stress fluids (YSFs) are typically thought of as behaving like viscoelastic solids below the yield stress and liquids when the yield stress is exceeded. Although yielding has been observed for over a century [1], the precise mechanisms by which materials undergo this transition remain poorly understood. This absence of understanding is caused at least in part by the significant ambiguity over how to identify, and define, yielding [2,3].

Multiple rheological protocols have been used to interrogate material failure and flow, including creep tests in which the viscosity bifurcates about the yield stress [4], shear start-up [5], serial creep divergence [6], fatigue tests [7,8], and flow sweeps with controlled shear history [9]. The protocol perhaps most commonly employed to identify yielding is large amplitude oscillatory shear [10–15], in which sinusoidal stresses or strains are applied at varying amplitudes. The periodic material response can then be used to determine the dynamic moduli, referred to as the storage (G') and loss (G'') moduli, which are proportional to the average energy stored and dissipated per cycle [16]. Although not observed for all classes of YSFs, one common feature of yielding rheology in oscillatory shearing is an overshoot in G'', which has formed the basis of measures of both the yield stress and the yield strain [3].

At large amplitudes, the rheological response can no longer be fully described by a linear differential equation with constant coefficients, and the response is said to be nonlinear. Such nonlinear responses from oscillatory shearing have been mathematically analyzed through procedures such as Fourier transform rheology [17,18] and stress decomposition [19], but physical insight from these methods has been difficult to obtain. Recently, an experimental procedure called recovery rheology has been used to gain insight into nonlinear rheological responses according to the relative acquisition of recoverable and unrecoverable strains. This technique has been used to show that the commonly observed overshoot in the loss modulus G'' [20] arises from the acquisition of unrecoverable strain [21]. The decomposition into recoverable and unrecoverable strain components forms the basis of the recent KDR model [22], which accurately models many of the features of the yield transition.

In this Letter, we show that the size of the G'' overshoot apparent in the nonlinear response of YSFs—a feature that is now known to arise due to acquisition of unrecoverable strain—is determined by the loss tangent in the linear regime, which is defined only in terms of recoverable behaviors. We further show that the amplitude at which the overshoot occurs is determined by a combination of linear viscoelastic and steady shear metrics. These relationships

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are shown to hold across a library of model YSFs and prior results reported in the literature. Specifically, we analyze polymer-linked emulsions (PLEs) with tunable yield transitions and compare their nonlinear responses to those reported in the literature for other classes of materials. For all materials analyzed, we find that the height of the G'' overshoot, quantified by the magnitude of the maximum relative to the small amplitude value, and the corresponding strain amplitude at which it occurs $\gamma_{0,\mathrm{peak}}$ can be collapsed onto master curves that are functions solely of linear recoverable metrics and combinations of linear recoverable and unrecoverable steady shear metrics, respectively. Furthermore, we show through both direct numerical solutions and theoretical approximations that these universal curves are accounted for by the KDR model. From this agreement between theory and experiment, we demonstrate that the physics responsible for the linear recoverable response of materials persists across the yield transition to control the nonlinear response where unrecoverable processes arise and ultimately dominate. These findings unify our physical interpretation of the nonlinear yield transition with linear viscoelasticity and identify nondimensional parameters that control yielding. These metrics can therefore serve as simplified targets for the design and optimization of soft materials.

We examine the linear and nonlinear rheological responses of a wide range of soft materials prepared in our previous works, including PLEs [23–25], colloidal gels [6,26], and fibrillar networks [26]. Their rheology is characterized on a TA Instruments HR-20 rheometer with parallel plate or cone-and-plate geometries. Linear rheology is collected through oscillatory frequency sweeps as a function of angular frequency ω with a constant strain amplitude γ_0 within the linear viscoelastic regime (LVR). Nonlinear rheology is measured by varying γ_0 at a constant frequency ω, and steady shear data are collected by measuring the steady state stress σ as a function of shear rate $\dot{\gamma}$. Additionally, we analyze rheological data available in literature for YSFs displaying an overshoot in G'', including high-internal-phase emulsions [27,28], colloidal glasses and gels [29–32], hydrogels [33–38], linked emulsions [39], polymer melts [40], and rubber composites [41–43]. Materials from literature are further detailed in Supplemental Material [44].

The typical rheological response of soft materials is exemplified by PLEs, as shown in Fig. 1(a). The PLEs shown here are prepared from 50 vol % decanol-in-water emulsions that are linked into a cohesive elastic network by the addition of a telechelic, triblock copolymer (POJO) with hydrophobic end blocks and a hydrophilic, bottlebrush midblock [25]. We tune the linear rheology by varying the polymer concentration c. At low c, these PLEs are elastic at high frequency but relax viscoelastically at lower frequencies as a result of the weakly percolated network. These viscous relaxations are suppressed on experimentally

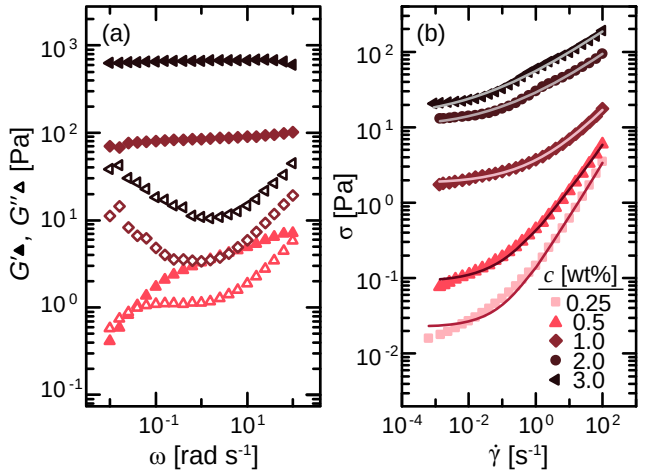

FIG. 1. (a) Storage modulus G' (closed symbols) and loss modulus G'' (open symbols) as a function of oscillation frequency ω. (b) Stress σ as a function of shear rate $\dot{\gamma}$ for samples containing the specified concentration of POJO-250. Curves are fits to the Herschel-Bulkley model.

accessible timescales at higher c as the network density increases, resulting in G' becoming nearly independent of frequency. Moreover, these stiff PLEs exhibit a pronounced minimum in G'' at $\omega \approx 1$ rad s^{-1}, which we attribute to an interplay between glassy colloidal dynamics and segmental fluctuations of polymer midblocks [25]. The steady shear flow curves, in which the stress σ is measured as a function of shear rate $\dot{\gamma}$, are shown in Fig. 1(b). Similar to the linear frequency sweeps, we observe an increase in σ with c as the network becomes more robust. The flow sweeps are well described by the Herschel-Bulkley model $\sigma(\dot{\gamma}) = \sigma_y + k\dot{\gamma}^n$, where σ_y is the yield stress, k is the consistency index, and n is the flow index [45]. From both small amplitude oscillatory shear and steady shear experiments, we find that the rheological behavior of PLEs can be tuned solely by the concentration of bridging polymers. This tunable rheology allows us to investigate the relationship between linear viscoelasticity and yielding independent from structural or compositional changes that may occur in other systems.

To characterize the yielding behavior, we show the results of amplitude sweeps at various angular frequencies and concentrations in Fig. 2. For each test, we observe the expected behavior [20], including a linear viscoelastic region at small strain amplitudes ($\gamma_0 \lesssim 10^{-2}$) where the dynamic moduli are independent of the strain amplitude, followed by a decrease in G' and an overshoot in G'' at larger amplitudes. This nonlinear response depends strongly on both polymer concentration and frequency. With increasing c, shown in Fig. 2(a), and thereby with increasing network elasticity, the decay in G' becomes steeper as a function of γ_0, and the relative maximum of the G'' overshoot becomes larger. Physically, these differences in yielding behavior indicate that a greater number of

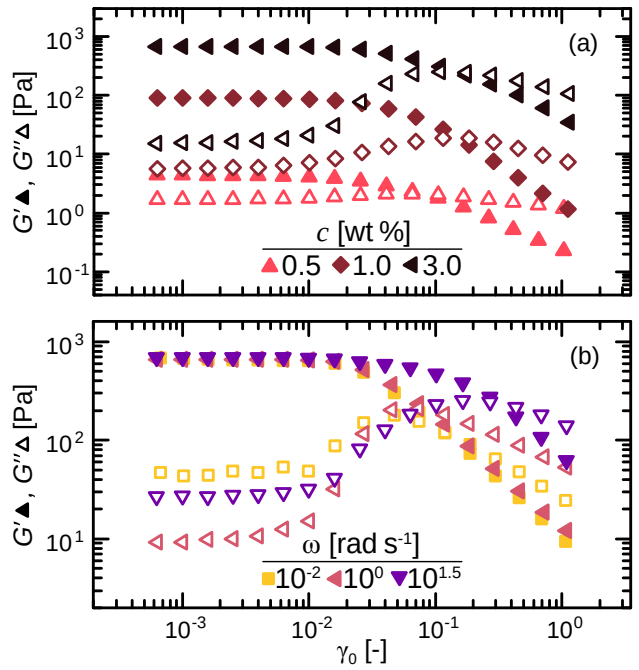

FIG. 2. Storage modulus G' (closed symbols) and loss modulus G'' (open symbols) as a function of strain amplitude γ_0 (a) for samples containing different polymer concentrations c at the same frequency $\omega = 10$ rad s^{-1} and (b) for samples containing $c = 3$ wt% POJO-250 at different frequencies ω.

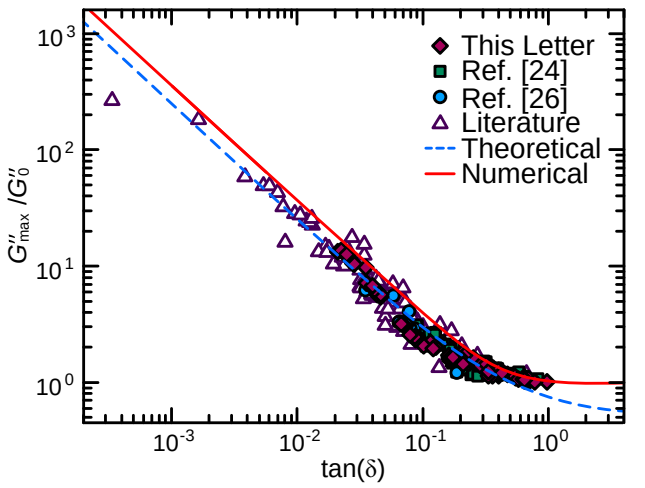

FIG. 3. Normalized magnitude of the G'' overshoot ($G''_{\max}/G''_0$) as a function of the LVR value of $\tan(\delta)$. Literature sources of yielding metrics are from Refs. [27–43] and are described in Supplemental Material [44]. Curves indicate numerical (red) and theoretical (blue) predictions based on the KDR model.

elastic elements transition to dissipating energy via unrecoverable processes upon yielding in PLEs formed at higher polymer concentrations, consistent with the increase in density of elastic bridges quantified in earlier work [25].

The dependence on frequency is more complex, as shown in Fig. 2(b). For a 3 wt% PLE, the yielding behavior varies significantly as a function of ω. First, we observe that the G'' overshoot is significantly larger at intermediate frequencies ($\omega = 10^0$) than at low frequencies ($\omega = 10^{-2}$) where G'' is larger, but the decrease in G' occurs at approximately the same strain amplitude. Second, the magnitude of the G'' overshoot is similar at low ($\omega = 10^{-2}$) and high ($\omega = 10^{1.5}$) frequencies with comparable values of G'', but the decrease in G' occurs at significantly larger amplitudes when tested at higher frequencies. Qualitatively similar behavior is observed for PLEs prepared at different c [44].

For linear deformations, it is well known that the rate of deformation relative to the rate of relaxation dictates the relative contributions from elastic deformation and viscous flow. Because such intrinsic dynamics are also present during nonlinear deformations, we expect that they should manifest in the frequency dependence of the yield transition. We explore this hypothesis by comparing linear and nonlinear rheological metrics. Specifically, we note that the relative contributions of viscous and elastic elements, quantified by the loss tangent $\tan(\delta) = G''/G'$, and the magnitude of the overshoot in G'' both depend strongly on ω. Thus, we plot the value of G'' at the overshoot normalized by the loss modulus G''_0 in the linear viscoelastic regime ($G''_{\max}/G''_0$) as a function of $\tan(\delta)$ and find a universal collapse, as shown in Fig. 3. The collapse of the overshoot height as a function of $\tan(\delta)$ holds across orders of magnitude in PLE moduli and frequency.

The dependence of the size of the overshoot in G'', a nonlinear behavior, on $\tan(\delta)$ defined in the linear regime is unexpected and surprising. It is now known that the overshoot in G'' arises due to the acquisition of unrecoverable strain [21], and yet $\tan(\delta)$ in the linear regime represents recoverable behavior. The dependence also indicates that the physics governing the material response to small deformations are preserved across the yield transition. Furthermore, given that the collapse is independent of material class, the physics controlling how soft materials yield must be universal and independent of material structure, chemistry, and the nature of interparticle interactions. To explain this phenomenon, we turn to the recently developed KDR model [22], which separates the strain into recoverable and unrecoverable components $\gamma = \gamma_{\text{rec}} + \gamma_{\text{unrec}}$ and describes yielding according to nonlocal physics where the rapid acquisition of recoverable strain enhances plastic unrecoverable deformation. With this framework, the relationship between stress and strain is described by the differential equation,

$$\sigma + \frac{\eta_f(\dot{\gamma}) + \eta_s}{G}\dot{\sigma} = \eta_f(\dot{\gamma})\left(\dot{\gamma} + \frac{\eta_s}{G}\ddot{\gamma}\right), \tag{1}$$

where $\eta_f(\dot{\gamma})$ is the Herschel-Bulkley viscosity under steady flow, $\eta_s = G''_0/\omega$ is a structural viscosity defined in the linear regime, and G is the linear elastic modulus.

We numerically solve this differential equation for oscillatory deformations $\gamma(t) = \gamma_0 \sin(\omega t)$ over orders of magnitude in γ_0, ω, and $\eta_f(\dot{\gamma})$. Specific ranges are given in Supplemental Material [44]. The moduli are calculated according to $G' = \omega/(\pi\gamma_0^2) \oint \sigma(t)\gamma(t)dt$ and $G'' = 1/(\pi\gamma_0^2) \oint \sigma(t)\dot{\gamma}(t)dt$, respectively [16,21]. These values correspond to the most common outputs from commercial rheometers and remain well defined in the nonlinear regime even when the geometric interpretation of in-phase and out-of-phase contributions no longer holds.

We find that for all conditions investigated, the overshoot height, $G''_{\max}/G''_0$, collapses as a function of $\tan(\delta)$, as shown in Fig. 3. This agreement exists without any fitting parameters, indicating that the same physics dictates both the linear recoverable properties and the nonlinear flow behavior captured by the overshoot height. Although the numerical solution slightly overestimates the magnitude of the G'' overshoot, it accurately captures the inverse relationship at low $\tan(\delta)$ and the turnover to a plateau as $\tan(\delta) \to 1$. The single numerical solution across such a wide range of amplitudes, frequencies, and sample properties demonstrates that the height of the G'' overshoot is solely determined by the relative contributions of elastic and viscous elements in the linear viscoelastic regime, quantified by $\tan(\delta)$. From this relationship, we can accurately predict one aspect of the nonlinear response of YSFs from purely linear metrics.

We gain additional insight into the physical mechanisms controlling this relationship by returning to the physics described by the KDR differential equation given in Eq. (1). This model is analytically solvable for sufficiently small γ_0, as is expected for linear responses, but cannot be directly evaluated once $\gamma_0 \gtrsim (\sigma_y)^{1/n}/k\omega$ or $\gamma_0 \gtrsim \sigma_y/\omega\eta_s$. Instead, following the foundations of the KDR model, we decompose the loss modulus into solidlike and fluidlike elements such that $G''/G''_0 = (G''_{\text{fluid}} + G''_{\text{solid}})/G''_0$, which can be rewritten as $G''/G''_0 = \gamma^2_{0,\text{rec}}/\gamma^2_0 + [\eta_f(\dot{\gamma})/\eta_s](\gamma^2_{0,\text{unrec}}/\gamma^2_0)$ by substituting their corresponding definitions. To predict the G'' overshoot height, we therefore require a relationship between flow $\eta_f(\dot{\gamma})$ and structural η_s viscosities. We build this relationship by recognizing that the stress in the recoverable and unrecoverable elements must be equal and that the stress of the recoverable element is described by $\sigma = G\gamma_{\text{rec}} + \eta_s\dot{\gamma}_{\text{rec}}$. Substituting this expression into Eq. (1) yields a differential equation describing purely unrecoverable elements according to

$$\sigma + \frac{\eta_s}{G}\dot{\sigma} = \eta_f(\dot{\gamma}_{\text{unrec}})\left(\dot{\gamma}_{\text{unrec}} + \frac{\eta_s}{G}\ddot{\gamma}_{\text{unrec}}\right). \tag{2}$$

We now make two simplifying assumptions. First, we approximate the instantaneous response of the system by their averaged parameters such that $\dot{\gamma} \approx \gamma_0\omega$ and $\ddot{\gamma} \approx \gamma_0\omega^2$, from which it follows that

$$\eta_f(\dot{\gamma}) = \frac{G\gamma_{0,\text{rec}}(1 + \eta_s\omega/G)}{\omega\gamma_{0,\text{unrec}}}. \tag{3}$$

Second, we assume that the recoverable and unrecoverable strain amplitudes are equal at the overshoot such that $\gamma_{0,\text{rec}} \approx \gamma_{0,\text{unrec}}$, which is reasonable based on experimental observations but likely overestimates the magnitude of unrecoverable strain. With this assumption, we can simplify the expression to

$$\frac{G''_{\max}}{G''_0} \approx \frac{1}{2} + \frac{1}{4}\frac{G}{\eta_s\omega} = \frac{1}{2} + \frac{1}{4}\tan(\delta)^{-1}. \tag{4}$$

Equation (4) is independent of any flow behavior and shows that the size of the overshoot in the loss modulus, which is a nonlinear property that arises according to the acquisition of unrecoverable strain, is dependent only on the linear viscoelastic value of $\tan(\delta)$, which corresponds to the acquisition of recoverable strain. The full details of this derivation are provided in Supplemental Material [44].

This theoretical approximation is in good agreement with both experimental data and the numerical solution shown in Fig. 3. It captures the appropriate scaling at small $\tan(\delta)$ and agrees quantitatively with experimental findings across materials and frequencies. At large $\tan(\delta)$, however, our simplified expression misses the appropriate plateau and instead predicts a value of $G''_{\max}$ smaller than G''_0. This discrepancy presumably occurs because the assumption that the maximum in G'' occurs when $\gamma_{0,\text{rec}} \approx \gamma_{0,\text{unrec}}$ overestimates the magnitude of $\gamma_{0,\text{unrec}}$, consistent with our observations that $G''_{\max}$ occurs at strain amplitudes close to, but always slightly smaller than, the strain amplitude at the crossover [22]. Even within the limitations of our approximations, this simple theoretical relationship is surprisingly robust and confirms that the nonlinear yielding response of soft materials can, at least in part, be predicted by linear recoverable measurements. Our results also confirm that elastic behaviors play an important role during yielding, and that the G'' overshoot exhibited by soft materials can be fully understood as a transition from energy storage via recoverable processes to energy dissipation via unrecoverable processes.

Lastly, we find that the strain amplitude at the G'' overshoot $\gamma_{0,\text{peak}}$ exhibits a universal collapse as a function of linear and steady shear properties, shown in Fig. 4. We observe that $\gamma_{0,\text{peak}}$ scales linearly with the ratio of the flow stress $\sigma_f = \eta_f(\dot{\gamma})\omega\gamma_{0,\text{peak}}$ to the linear elastic modulus G modulated by $\tan(\delta)$. This is a universal collapse across all frequencies, polymer concentrations, and material classes. Furthermore, we confirm the linearity of this relationship from the numerical solution to the KDR model, which predicts a prefactor of $\approx$1.5 that is in excellent agreement with experimental data. To understand the mechanisms controlling this transition, we return to the KDR model, subject to the same assumptions as above. Starting with

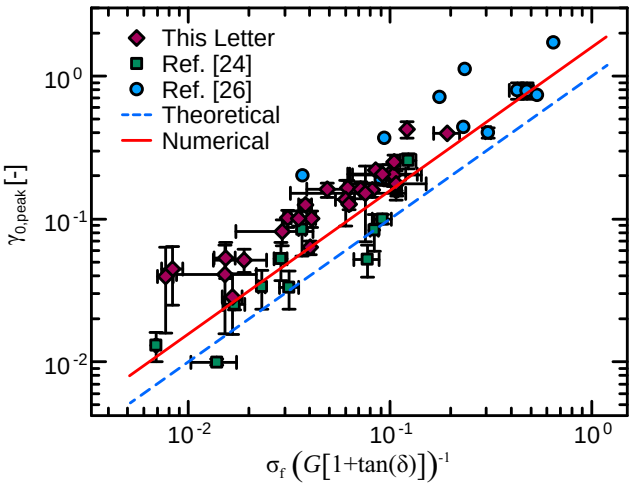

FIG. 4. Strain amplitude at the G'' overshoot $\gamma_{0,\mathrm{peak}}$ as a function of the nondimensional parameter of flow stress σ_f, linear storage modulus G, and $\tan(\delta)$. Lines show numerical (red) and theoretical (blue) predictions.

Eq. (2) and substituting in the expressions for the stress associated with recoverable strain and average values in place of instantaneous derivatives gives

$$G\gamma_{0,\mathrm{rec}} + 2\eta_s\gamma_{0,\mathrm{rec}}\omega + \frac{\eta_s^2\gamma_{0,\mathrm{rec}}\omega^2}{G} = \eta_f(\dot{\gamma})\gamma_{0,\mathrm{unrec}}\omega + \frac{\eta_f(\dot{\gamma})\eta_s\gamma_{0,\mathrm{unrec}}\omega^2}{G}. \quad (5)$$

Further assuming that $\gamma_{0,\mathrm{rec}} \approx \gamma_{0,\mathrm{unrec}}$ at the G'' overshoot results in

$$\gamma_{0,\mathrm{peak}} \approx \frac{\eta_f(\dot{\gamma})\omega\gamma_{0,\mathrm{peak}}}{G[1+\tan(\delta)]} = \frac{\sigma_f}{G[1+\tan(\delta)]}, \quad (6)$$

where we approximate $\eta_f(\dot{\gamma}) \approx \eta_f(\omega\gamma_{0,\mathrm{peak}})$ according to the empirical Rutgers-Delaware relationship [10], which is an extension of the Cox-Merz relationship specifically applied to YSFs relating the viscosity measured under steady and oscillatory shearing [46,47]. The KDR model assumes that the magnitude of the flow viscosity depends on the total shear rate so that the Rutgers-Delaware relationship always holds [47]. Our theoretical approximation underestimates the experimental data but achieves the correct scaling and is within the appropriate order of magnitude. As with the size of the overshoot, this deviation is expected given our assumption that the recoverable and unrecoverable strain components are equal at the overshoot. The explicit dependence of $\gamma_{0,\mathrm{peak}}$ on $\tan(\delta)$ is shown in Supplemental Material [44]. The near quantitative agreement between experiments, numerical solutions, and theoretical approximations demonstrate that the strain amplitude corresponding to the overshoot in G'' follows from linear and steady shear rheological metrics.

Our results help to explain why there has been significant ambiguity over how to identify yielding from oscillatory shearing. Although the yield transition is marked by a transition from recoverable to unrecoverable acquisition of strain, most yielding metrics are defined in terms of the total strain only. Additionally, while the overshoot in G'' is known to result from the acquisition of unrecoverable strain [21], our results demonstrate that its magnitude is determined by recoverable properties only and that the corresponding total strain amplitude is determined by the relative contributions of recoverable elasticity within the linear regime and unrecoverable deformation under flow. Even when brittility is taken into account [48], our derivation remains unchanged, confirming that both brittle and ductile yielding modes result in similarly sized overshoots. The size and position of the G'' overshoot, a major feature of the oscillatory shear rheology of YSFs, therefore correspond to intrinsic material properties and do not serve as accurate metrics by which to determine the extrinsic yield transition.

In this Letter, we have demonstrated that the nonlinear yielding rheology of soft materials exhibits universal characteristics that can be quantitatively predicted from linear and steady shear flow properties. Specifically, we have shown that the magnitude of the overshoot in G'', which is known to be caused by the acquisition of unrecoverable strain, depends exclusively on $\tan(\delta)$ in the linear viscoelastic regime, which is defined entirely in terms of recoverable properties. Further, we have shown that the amplitude at which the overshoot occurs, $\gamma_{0,\mathrm{peak}}$, depends on $\tan(\delta)$ and the viscous stress developed under steady shear flow. These relationships persist across all classes of YSFs that exhibit a G'' overshoot, across all deformation frequencies, and across orders of magnitude in $\tan(\delta)$. YSFs that do not exhibit this overshoot [49] or those that undergo more complex transitions such as strain stiffening [50,51], may form distinct classes or possess sufficiently small ratios of unrecoverable dissipation that the overshoot is experimentally undetectable [52]. Although the precise mechanism by which materials yield may be specific to a material class [53], the universality of these relationships indicates that yielding is governed by elements of the same nonlocal physics represented by the KDR model, in which elastic recoverable properties enhance plastic unrecoverable behaviors. This demonstrated relationship between linear and nonlinear properties in YSFs will accelerate the development of novel complex fluids and soft materials by shifting design targets and performance metrics from experimentally challenging characterizations of the yield transition to the relatively straightforward optimization of their linear viscoelasticity.

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Data availability—The data that support the findings of this Letter are openly available [54].

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