Residual stresses and shear-induced overaging in boehmite gels

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Colloidal gels respond like solid solids at rest, whereas they flow like liquids under external shear. Starting from a fluidized state under an applied shear rate \( \dot{\gamma}_p \), abrupt flow cessation triggers a liquid-to-solid transition during which the stress relaxes towards a so-called residual stress \( \sigma_{\text{res}} \) that tallies a macroscopic signature of previous shear history. Here, we report on the liquid-to-solid transition in gels of boehmite, an aluminum oxide, that shows a remarkable non-monotonic stress relaxation towards a residual stress \( \sigma_{\text{res}}(\dot{\gamma}_p) \) characterized by a dual behavior relative to a critical value \( \dot{\gamma}_c \) of the shear rate \( \dot{\gamma}_p \). Following shear at \( \dot{\gamma}_p > \dot{\gamma}_c \), the gel obtained upon flow cessation is insensitive to shear history, and the residual stress is negligible. However, for \( \dot{\gamma}_p < \dot{\gamma}_c \), the gel encodes some memory of the shear history, and \( \sigma_{\text{res}} \) increases for decreasing shear rate, directly contributing to reinforcing the gel viscoelastic properties. Moreover, we show that both \( \sigma_{\text{res}} \) and the gel viscoelastic properties increase logarithmically with the strain accumulated during the shear period preceding flow cessation. Such a shear-induced “overaging” phenomenon bears great potential for tuning the rheological properties of colloidal gels.

Introduction.- Soft Glassy Materials (SGMs) are viscoelastic soft solids ubiquitous in science and engineering [1][3]. These materials display a shear-induced solid-to-liquid transition under large enough stresses, i.e., they flow like liquids above their yield stress \( \sigma_y \), whereas SGMs recover their solidlike properties upon flow cessation [4]. Such a liquid-to-amorphous-solid transition results in the partial relaxation of internal stresses, which usually follows a monotonic decay towards a so-called residual stress \( \sigma_{\text{res}} < \sigma_y \) [5][6]. Residual stresses are critical to the mechanical properties of a broad range of materials across hard and soft condensed matter, for they control both the linear and non-linear material responses [7][8]. In the case of SGMs, the residual stress encodes the sample memory of the plastic deformation accumulated prior to and during flow cessation [9][10]. As such, \( \sigma_{\text{res}} \) depends on the shear rate \( \dot{\gamma}_p \) applied prior to flow cessation, and \( \sigma_{\text{res}}(\dot{\gamma}_p) \) is usually reported to be a decreasing function, typically a weak power-law [11][14], or a logarithm of \( \dot{\gamma}_p \) [15] such that residual stresses become negligible following strong enough shear.

From a microscopic point of view, the origin of the residual stress depends on the details of the SGMs microstructure. On the one hand, residual stresses in dense systems have been linked to the elastic contact forces in soft jammed particles [15][17], and to the so-called “supra-caging” effects in hard-sphere colloidal glasses [9]. On the other hand, residual stresses in dilute systems such as colloidal gels are associated with structural anisotropy frozen into the gel microstructure. Such anisotropy only emerges following low enough shear rates, i.e., low enough Peclet numbers, such that upon flow cessation, particles only move over short distances compared to the range of the inter-particle attraction, hence freezing their latest configuration [14][18]. Yet, the lack of information on residual stresses in colloidal gels compared to denser systems calls for more experiments. Moreover, while residual stresses and, more generally, memory effects in gels are often reported to be driven by the shear rate [19][21], little is known about the impact of the total strain accumulated during the shear period prior to flow cessation, and disentangling the contribution of the shear rate from that of the strain to residual stresses and to final viscoelastic properties remains challenging.

In this Letter, we study the residual stress in acid-induced gels of boehmite, an aluminum oxide. We show that sudden flow cessation from a given shear rate \( \dot{\gamma}_p \) yields an anomalous non-monotonic stress relaxation towards a residual stress that strongly depends on the shear rate \( \dot{\gamma}_p \) applied before quenching the flow. We identify a critical shear rate \( \dot{\gamma}_c \), above which boehmite gels show negligible residual stresses, whereas, for \( \dot{\gamma}_p < \dot{\gamma}_c \), they display significant residual stresses that increase when decreasing \( \dot{\gamma}_p \). Such a residual stress directly impacts the gel viscoelastic properties by reinforcing the elastic and viscous moduli, \( G' \) and \( G'' \), respectively. In particular, for \( \dot{\gamma}_p < \dot{\gamma}_c \), both \( G' \) and \( G'' \) are directly proportional to the residual stress. Moreover, we show that aside from the shear rate \( \dot{\gamma}_p \), the total strain \( \gamma_p \) accumulated before flow cessation plays a crucial role: the residual stress \( \sigma_{\text{res}} \) and the viscoelastic moduli grow logarithmically with \( \gamma_p \), with a prefactor that depends on \( \dot{\gamma}_p \). Our results are prototypical of shear-induced “overaging”, and show that boehmite gels can be tuned by both shear- and strain-controlled memory effects in the limit of low enough shear rates.

Materials and methods.- Steady shear and flow cessation experiments are performed on 4% vol. boehmite gels prepared by dispersing a boehmite powder (Plural SB3, Sasol) in an aqueous solution of nitric acid at 14 g/L [22].
After 35 min of mixing, the suspension contains unbreakable aggregates of diameter $2R_{ag} \simeq 100$ nm, which further assemble into large clusters of typical diameter $2R_{cl} \simeq 300$–1200 nm, leading to a sol-gel transition after a couple of hours [25]. The sample is left at rest for at least seven days prior to any rheological test, until the pH stabilizes to $pH = 3.5$ [24][26]. About 10 mL of the gel sample is then transferred into a smooth cylindrical Couette cell of gap 1 mm connected to a stress-controlled rheometer (AR-G2, TA Instruments).

**Results.**- The gel is first rejuvenated by a constant shear rate $\dot{\gamma} = 1000$ s$^{-1}$ during 600 s into a liquid suspension that displays no residual stress. Then the flow is quenched to the shear rate of interest $\dot{\gamma}_p$ for a duration $t_p = 600$ s, which is sufficient for the shear stress to reach a steady state whatever the value of $\dot{\gamma}_p$ in the range of 1 s$^{-1}$ to 1000 s$^{-1}$. Finally, the flow cessation, which sets the origin of time, is induced by imposing $\dot{\gamma} = 0$ s$^{-1}$, while we monitor the stress relaxation $\sigma(t)$, as reported in Fig. 1 for shear rates $\dot{\gamma}_p$ ranging from $\dot{\gamma}_p = 3$ s$^{-1}$ to 500 s$^{-1}$. For all $\dot{\gamma}_p$ in the experimental range, the stress displays a remarkable non-monotonic evolution, which becomes more pronounced for decreasing values of $\dot{\gamma}_p$. More precisely, the stress response shows a dual behavior relative to a critical shear rate $\dot{\gamma}_c \simeq 40$ s$^{-1}$. For $\dot{\gamma}_p > \dot{\gamma}_c$, the stress response first shows a linear decay until $t = t_{\text{min}} \simeq 10$ s, followed by a stress overshoot, before decreasing again towards a negligible stress value, below the rheometer resolution. In stark contrast, upon flow cessation from a shear rate $\dot{\gamma}_p < \dot{\gamma}_c$, the stress response decreases down to a minimum value reached at time $t_{\text{min}} \lesssim 10$ s, and then increases towards a final and steady residual stress $\sigma_{\text{res}} > 0$ [see Fig. S1 in Supplemental Material for the temporal evolution of the shear rate $\dot{\gamma}$ effectively applied, and Fig. S2 for longer stress relaxations and additional data recorded in a cone-and-plate geometry]. In that case, both $t_{\text{min}}$ and $\sigma_{\text{res}}$ depend on the shear rate $\dot{\gamma}_p$ applied before flow cessation.

Figure 2(a) presents the residual stress $\sigma_{\text{res}}$ measured 2000 s after flow cessation as a function of $\dot{\gamma}_p$ over three decades [see also Fig. S3(a) in Supplemental Material for $t_{\text{min}}(\dot{\gamma}_p)$] and quantitatively confirms the dual behavior of boehmite gels: the residual stress is negligible for $\dot{\gamma}_p > \dot{\gamma}_c$, whereas it increases logarithmically with decreasing $\dot{\gamma}_p$, up to $\sigma_{\text{res}} \simeq 9$ Pa for $\dot{\gamma}_p = 1$ s$^{-1}$. These results show that boehmite gels display little if no memory of shear history for $\dot{\gamma}_p > \dot{\gamma}_c$, whereas they build up some frustration while quenching the gel from $\dot{\gamma}_p < \dot{\gamma}_c$, hence freezing residual stresses. We further characterize the properties of these soft solids by measuring their steady-state linear viscoelastic properties at $t = 3000$ s after flow cessation and at a single frequency of 1 Hz. Such a choice of frequency does not impact the gener-
ality of our results since the viscoelastic moduli of the present boehmite gels depend on the frequency as very weak power laws. Consistently with $\sigma_{\text{res}}(\dot{\gamma}_p)$, the elastic and viscous moduli, $G'$ and $G''$, both show a similar dual behavior [see also Fig. S3(b) in Supplemental Material for $\tan \delta = G''/G'$]. Indeed, for $\dot{\gamma}_p > \dot{\gamma}_c$, the viscoelastic moduli are insensitive to shear history, with $G'_0 = 444 \pm 37$ Pa and $G''_0 = 10 \pm 2$ Pa. In contrast, for $\dot{\gamma}_p < \dot{\gamma}_c$, $G'$ and $G''$ increase logarithmically for decreasing $\dot{\gamma}_p$, which suggests that $\dot{\gamma}_p$ affects both the skeletal backbone of the gel network, and its fractal microstructure, most likely through more anisotropy for smaller $\dot{\gamma}_p$.

In summary, the shear rate $\dot{\gamma}_p$ applied before flow cessation appears to control the terminal viscoelastic properties of boehmite gels. However, one should keep in mind that so far, the shearing duration was kept constant for all values of $\dot{\gamma}_p$ so that the samples experienced different levels of strain prior to flow cessation.

In order to disentangle the impact of the shear rate $\dot{\gamma}_p$ from that of the accumulated strain $\gamma_p$ prior to flow cessation, we vary the shearing duration $t_p$ for given values of $\dot{\gamma}_p$, so as to generate gels with different levels of accumulated strain $\gamma_p = \dot{\gamma}_p t_p$ [see Fig. S4 in Supplemental Material for corresponding stress relaxation data, which all display a non-monotonic evolution]. Note that for $t_p < 600$ s, the stress does not reach a steady state for all levels of strain during the shearing step at $\dot{\gamma}_p$. Figure 3 reveals the impact of both $\gamma_p$ and $\dot{\gamma}_p$ on $\sigma_{\text{res}}, G'$ and $G''$. For a fixed shear rate $\dot{\gamma}_p < \dot{\gamma}_c$, all three quantities increase logarithmically for increasing strain $\gamma_p$ [see colored lines in Fig. 3]. This result holds over three decades of strain for various shear rates. Moreover, the prefactors of this logarithmic dependence increase weakly for decreasing $\dot{\gamma}_p$ [Fig. 3(d)-(f)], i.e., the lower the applied shear rate, the larger $\sigma_{\text{res}}, G'$ and $G''$ for a fixed accumulated strain $\gamma_p$. In conclusion, the impact of both $\dot{\gamma}_p$ and $\gamma_p$ on the various observables of interest, $X = \sigma_{\text{res}}, G'$, or $G''$, is captured by:

$$\sigma_{\text{res}}, G', \text{ and } G'' = \alpha_X(\dot{\gamma}_p) \log \gamma_p + \beta_X(\dot{\gamma}_p),$$

(1)

where both $\alpha_X(\dot{\gamma}_p)$ and $\beta_X(\dot{\gamma}_p)$ are decreasing functions of $\dot{\gamma}_p$. While the slope $\alpha_X(\dot{\gamma}_p)$ depends only weakly on $\dot{\gamma}_p$, the intercept $\beta_X(\dot{\gamma}_p)$ accounts for the strong sensitivity on $\dot{\gamma}_p$ reported in Fig. 2. Note that for $\dot{\gamma}_p > \dot{\gamma}_c$, neither the strain nor the shear rate have any impact on the residual stress or on the viscoelastic properties.

Finally, the robustness of Eq. 1 prompts us to eliminate the strain and to directly link the gel viscoelastic properties to the residual stress. As pictured in Fig. 4, this allows us to collapse all data obtained for various shear histories, i.e., various shear rates $\dot{\gamma}_p$ applied over different durations $t_p$, hence yielding various strains $\gamma_p$, onto a master curve. Moreover, as expected from Eq. 1, the elastic and viscous moduli both increase proportionally to the residual stress, starting from the two reference values $G'_0$ and $G''_0$ measured for $\dot{\gamma}_p > \dot{\gamma}_c$ [see also Fig. S5 in the Supplemental Material for the corresponding master curve for $\tan \delta$]. Note that such a collapse does not depend on frequency (here 1 Hz), for viscoelastic properties of boehmite gels show a very weak power-law frequency dependence. Our results thus show that the viscoelastic properties of boehmite gels are reinforced by the residual stress, which grows due to an extended period of shear at low shear rates.
cate driving force that relocates the system into deeper
framework, moderate shear was shown to act as a deli-
effect of thermally activated dynamics [36, 37]. In this
quence of the system exploring its “energy landscape”
coelastic moduli [25]. Such physical aging is a conse-
acterized by a logarithmic time dependence of the vis-
gels also develop internal stresses, which bear some mem-
anisotropy. Here, we show that for \( \dot{\gamma}_p \) the gel, which builds up some shear-induced structural
follows: low enough shear rates do not fully rejuvenate
\( \dot{\gamma}_p \) Ref. [25] to account for observations with \( \dot{\gamma}_p \)
contact [25, 29–32]. The hypothetical scenario proposed in
shear to the bond force between two particles at con-
as a critical Mason number comparing the strength of
thus confirm the robustness of \( \dot{\gamma}_p \) in Ref. [25]). Our results
before imposing the shear rate of interest \( \dot{\gamma}_p \) for the present boehmite gels as hypothesized in Ref. [25], and with the
emergence of some anisotropy in the microstructure of
based on shear-induced alignment is consistent with the
alignment of the microstructure under flow, and the
formation of bonds upon flow cessation due to interpar-
ticle attractive forces that locally deform the bulk mate-
rial, hence inducing internal stresses [52]. This picture
based on shear-induced alignment is consistent with the
emergence of some anisotropy in the microstructure of
boehmite gels as hypothesized in Ref. [25], and with the
growth of residual stresses only observed for \( \dot{\gamma}_p < \dot{\gamma}_c \) in
the present study. In that framework, boehmite gels
develop internal stresses, which bear some mem-
ory of the flow history.

Moreover, as reported in a broad variety of SGMs [33–
35], boehmite gels show spontaneous aging at rest char-
acterized by a logarithmic time dependence of the vis-
coelastic moduli [24]. Such physical aging is a conse-
quence of the system exploring its “energy landscape”
by going from one metastable state to another under the
effect of thermally activated dynamics [36, 37]. In this
framework, moderate shear was shown to act as a deli-
cate driving force that relocates the system into deeper
energy minima, thus inducing “overaging” in SGMs [38–
40]. Here, the logarithmic increase of \( G' \) and \( G'' \) with
the strain accumulated under low enough shear rate is strik-
ingly reminiscent of aging and constitutes a clear instance
of “overaging” in a colloidal gel. Finally, we note that for
vanishingly low shear rates, our results may be also linked
to the so-called “directed aging” recently achieved under a
static load or strain in amorphous solids [41, 42]. Yet,
the microscopic process underpinning shear-
induced “overaging” remains to be identified in the
present boehmite gels. It most likely originates either
from local compaction, i.e., the slow increase in local
volume fraction turning the local mechanical response
from that of gel to that of a glass [43, 44], or from the
strain-induced alignment of the microstructure [15, 47].
Interestingly such a strain-controlled reinforcement pro-
cess seems to reach a limit for large enough strains.
Indeed, when imposing \( \dot{\gamma}_p = 2 \) s\(^{-1}\) prior to flow cessation,
the residual stress reaches a maximum at \( \gamma_p^* \approx 2.10^3 \) be-
fore decreasing [see yellow symbols in Fig. 3(a)]. The
same effect is visible beyond \( \gamma_p^* \approx 2.10^4 \) for \( \dot{\gamma}_p = 7 \) s\(^{-1}\)
[see red symbols in Fig. 3(a)], suggesting that strain be-
comes detrimental beyond a certain point, breaking the
strong microstructure that builds up at \( \gamma_p < \gamma_p^* \). Such a scenario
remains to be investigated with numerical tools,
including molecular dynamics (MD) simulations, which
would allow one to assess whether plasticity is responsi-
ble for the drop in residual stress beyond \( \gamma_p^* \), and whether
it results from rearrangements within gel strands [48], or
occurs at larger length scales, between clusters [31, 49].

Another signature of shear-induced “overaging” on
the present boehmite gels for \( \dot{\gamma}_p < \dot{\gamma}_c \) is their non-
monotonic stress relaxation following flow cessation.
Such an anomalous relaxation, common to disordered
solids [50, 51], has been recently reported in viscoelas-
tic liquids and attributed to two key ingredients, namely
the alignment of the microstructure under flow, and the
formation of bonds upon flow cessation due to interpar-
ticle attractive forces that locally deform the bulk mate-
rial, hence inducing internal stresses [52]. This picture

Discussion and conclusion.- Let us now summarize and
discuss the most prominent results of this Letter. First,
we have identified a critical shear rate \( \dot{\gamma}_c \) above which
boehmite gels are insensitive to shear history. Such a crit-
ical shear rate is similar to the one reported in Ref. [24],
although determined with a different rheological proto-
col. In particular, as a key difference with Ref. [25],
we have identified a critical shear rate \( \dot{\gamma}_c \) in Ref. [25]. Our results
around the same val-
ues of \( \dot{\gamma}_c \) \( (\dot{\gamma}_c = 30 \pm 10 \) s\(^{-1}\) in Ref. [24] \). Our results
thus confirm the robustness of \( \dot{\gamma}_c \) which was interpreted
as a critical Mason number comparing the strength of
shear to the bond force between two particles at con-
tact [25, 29, 32]. The hypothetical scenario proposed in
Ref. [24] to account for observations with \( \dot{\gamma}_p < \dot{\gamma}_c \) is as
follows: low enough shear rates do not fully rejuvenate
the gel, which builds up some shear-induced structural
anisotropy. Here, we show that for \( \dot{\gamma}_p < \dot{\gamma}_c \) boehmite
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by going from one metastable state to another under the
effect of thermally activated dynamics [36, 37]. In this
framework, moderate shear was shown to act as a deli-
cate driving force that relocates the system into deeper

FIG. 4. Elastic modulus \( G' \) vs. residual stress \( \sigma_{res} \) for a boehmite gel submitted to various shear histories, i.e., various
shear rates \( \dot{\gamma}_p \) over different durations \( t_p \). Data replotted from
Fig. 3(a)–(c). Inset: Viscous modulus \( G'' \) vs. \( \sigma_{res} \). Black
lines are the best linear fits for \( \sigma_{res} < 8.0 \) Pa, respectively
\( G' = G'_0(1 + \lambda' \sigma_{res}) \) and \( G'' = G''_0(1 + \lambda'' \sigma_{res}) \), with \( G'_0 =
444 \pm 37 \) Pa, \( \lambda' = 0.35 \pm 0.04 \), \( G''_0 = 10 \pm 2 \) Pa, and \( \lambda'' =
0.69 \pm 0.06 \).
Our data set thus constitutes a unique opportunity for future numerical work to identify the minimal ingredients necessary to reproduce such an anomalous relaxation. We anticipate that the anisotropy of boehmite particles and their noncentral interaction play a key role in the stress dynamics.

Finally, in cases where the stress relaxes to vanishingly small values, the late stages of the dynamics are consistently characterized by erratic fluctuations of $\sigma(t)$ [see Fig. 1 and Fig. S4 in the Supplemental Material]. Such fluctuations could be the signature of large-scale, heterogeneous dynamics occurring in the bulk or in the vicinity of the walls. Future experiments offering local insights into the sample microdynamics upon flow cessation should be undertaken to answer this open question.

**Conclusion.** Our study unravels a deep connection between residual stresses and viscoelastic properties in colloidal gels: the elastic and viscous modulus of boehmite gels are reinforced proportionally to the residual stress trapped upon flow cessation. Interestingly, such a linear relationship was previously reported in gels of fractal-like particles experiencing van der Waals attractive interactions [11]. However, in contrast with the low shear rates explored here, the reinforcement in these fractal gels was observed in a shear-thickened state obtained only after a period of intense shearing, and following a monotonic stress relaxation. Such a discrepancy with our observations suggests that the linear dependence of the viscoelastic moduli with $\sigma_{res}$ is somewhat universal in colloidal gels, regardless of microscopic details. As such, our experimental results constitute a benchmark for future modeling of residual stresses at the particle scale [12], not only to rationalize their relation to elasticity and reinforcement, but also to understand their dependence with the accumulated strain, here reported to be logarithmic, as well as their temporal evolution after flow cessation.

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Figure S1(a) displays the temporal evolution of the shear rate $\dot{\gamma}$ effectively applied (as measured by the rheometer) during the flow cessation step and normalized by $\dot{\gamma}_p$. In all cases, it takes about 0.5 s for the shear rate to reach $\dot{\gamma}/\dot{\gamma}_p = 10^{-3}$. Therefore, we may assume the shear rate to be effectively zero for $t > 1$ s, which is the reason why we report the stress response starting at $t = 1$ s in Fig. 1 in the main text. For the sake of completeness, we also report in Fig. S1(b) the stress responses for $t > 0.5$ ms.

FIG. S1. (a) Temporal evolution of the shear rate $\dot{\gamma}$ effectively applied and normalized by the shear rate $\dot{\gamma}_p$ imposed prior to the flow cessation step. The black horizontal dashed line highlights $\dot{\gamma}/\dot{\gamma}_p = 10^{-3}$. (b) Stress response $\sigma(t)$ of a boehmite gel following flow cessation induced by imposing a decreasing step of shear rate from $\dot{\gamma} = \dot{\gamma}_p$ to $\dot{\gamma} = 0$ at $t = 0$ s. The colors code for $\dot{\gamma}_p = 3$ s$^{-1}$ (■), 5 s$^{-1}$ (■), 10 s$^{-1}$ (■), 15 s$^{-1}$ (■), 40 s$^{-1}$ (■), 50 s$^{-1}$ (■), 200 s$^{-1}$ (■), and 500 s$^{-1}$ (■).
Figure S2 reports additional stress responses following flow cessation as described in the main text. Using the same Couette geometry and the same rheometer as in the main text, we checked that the stress has reached a stationary value by monitoring $\sigma(t)$ over $10^4$ s instead of 2000 s (see square symbols). Moreover, measurements performed over 2000 s using a sandblasted cone-and-plate geometry connected to a different stress-controlled rheometer (MCR 302, Anton Paar) display a very similar non-monotonic evolution and quantitatively point to the same residual stresses (see dotted lines for $\dot{\gamma} = 2$ and $5 \, \text{s}^{-1}$). These measurements emphasize the robustness of the anomalous stress relaxation towards significant residual stresses for $\dot{\gamma}_p < \dot{\gamma}_c$. Note that the stress relaxations do not strictly coincide in the two different geometries, which is not surprising as the details of the global stress dynamics following flow cessation must depend on the local deformation of the microstructure, which is itself likely to depend on the (geometry-dependent) local shear rate and shear stress fields.

**FIG. S2.** Stress response $\sigma(t)$ of a boehmite gel following flow cessation induced by imposing a decreasing step of shear rate from $\dot{\gamma} = \dot{\gamma}_p$ to $\dot{\gamma} = 0$ at $t = 0$ s. Square symbols correspond to experiments performed in the same Couette geometry as in the main text but over a longer duration of $10^4$ s, while dotted lines refer to experiments performed over 2000 s in a sandblasted cone-and-plate geometry (angle of $2^\circ$ and diameter of 40 mm) connected to an MCR 302 (Anton Paar) stress-controlled rheometer. The colors code for $\dot{\gamma}_p = 2 \, \text{s}^{-1}$ (■), $5 \, \text{s}^{-1}$ (■), and $10 \, \text{s}^{-1}$ (■). The duration of the shear step at $\dot{\gamma}_p$ is $t_p = 600$ s in all cases.
COMPLEMENTARY ANALYSIS OF THE STRESS RESPONSES

In Fig. S3, we show complementary observables extracted from Fig. 1 in the main text, as a function of the shear rate $\dot{\gamma}_p$, namely the time $t_{\text{min}}$ at which the stress reaches a minimum [Fig. S3(a)], and the loss factor $\tan \delta$ [Fig. S3(b)]. Both observables display a dual behavior depending on the position of $\dot{\gamma}_p$ relative to $\dot{\gamma}_c$. For $\dot{\gamma}_p < \dot{\gamma}_c$, the data are well fitted by a logarithmic dependence in $\dot{\gamma}_p$, while they become independent of $\dot{\gamma}_p$ above $\dot{\gamma}_c$. Such a behavior is fully consistent with the trends observed in Fig. 2 in the main text for the residual stress and the viscoelastic moduli. Note that the stress minimum becomes hardly measurable after strong shearing, as the stress response flattens with $\dot{\gamma}_p$, hence the large error bars on $t_{\text{min}}$ for the highest values of $\dot{\gamma}_p$. Moreover, the fact that $\tan \delta$ increases with decreasing $\dot{\gamma}_p$ indicates that the dissipative component increases more steeply than the elastic component as $\dot{\gamma}_p$ decreases.

![Graph](image)

**FIG. S3.** (a) Time $t_{\text{min}}$ at which the stress reaches a minimum and (b) loss factor $\tan \delta$ vs. the shear rate $\dot{\gamma}_p$ applied prior to flow cessation. The vertical dashed line highlights the critical shear rate $\dot{\gamma}_c = 40 \text{ s}^{-1}$ below which the sample grows a residual stress. Blue lines highlight $t_{\text{min}} = 11 \pm 1 \text{ s}$ and $\tan \delta = 0.0228 \pm 0.0005$, and red lines are the best logarithmic fits of the data for $\dot{\gamma}_p < \dot{\gamma}_c$: $X = a_X \log \dot{\gamma}_p + b_X$ with $a_t = 4.45 \text{ s}$, $a_\delta = -0.01$, $b_t = 2.96 \text{ s}$, and $b_\delta = 0.04$. 
INFLUENCE OF THE SHEARING DURATION ON THE STRESS RELAXATION

Figure S4 presents the stress responses measured after flow cessation following shearing at $\dot{\gamma}_p$ over different durations $t_p$. It reveals that the accumulated strain $\gamma_p = \dot{\gamma}_pt_p$ has a crucial influence on the stress relaxation, and thus on the subsequent residual stress, as further analyzed in Fig. 3 in the main text.

FIG. S4. Stress response $\sigma(t)$ of a boehmite gel following a flow cessation induced by imposing a shear step from $\dot{\gamma} = \dot{\gamma}_p$ to $\dot{\gamma} = 0$ at $t = 0$ s. The colors code for (a) $\dot{\gamma}_p = 3$ s$^{-1}$, (b) $\dot{\gamma}_p = 5$ s$^{-1}$, (c) $\dot{\gamma}_p = 10$ s$^{-1}$, and (d) $\dot{\gamma}_p = 15$ s$^{-1}$, and for different durations $t_p$ as indicated in the graphs.
MASTER CURVE FOR THE LOSS FACTOR VS. THE RESIDUAL STRESS

From the master curves for $G'$ and $G''$ vs. $\sigma_{\text{res}}$ and the linear fits shown in Fig. 4 in the main text, it is straightforward that the loss tangent $\tan \delta = G''/G'$ should also follow a master curve as a function of the residual stress, independent of the shear rate $\dot{\gamma}_p$ and duration $t_p$. This is checked in Fig. S5, where the ratio of the fits for $G''$ and $G'$ (see black line) is shown to account well for the evolution of the loss tangent with $\sigma_{\text{res}}$.

FIG. S5. Loss factor $\tan \delta$ vs. residual stress $\sigma_{\text{res}}$ for a boehmite gel submitted to various shear histories. Colors code for $\dot{\gamma}_p = 2 \text{ s}^{-1}$ (■), 3 s$^{-1}$ (■), 5 s$^{-1}$ (■), 7 s$^{-1}$ (■), 10 s$^{-1}$ (■), 15 s$^{-1}$ (■), and 50 s$^{-1}$ (■) imposed over various durations $t_p$ in independent experiments. The black line corresponds to the ratio of the linear fits of $G''$ and $G'$ vs. $\sigma_{\text{res}}$ shown in Fig. 4 in the main text, namely $G''_0(1 + \lambda''\sigma_{\text{res}})/[G'_0(1 + \lambda'\sigma_{\text{res}})]$, with the fit parameters given in the main text.