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Use of creep recovery protocol to measure static yield stress and structural rebuilding of fresh cement pastes

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ABSTRACT

In this study, a creep recovery shear rheological protocol was applied to fresh cement pastes. A viscosity bifurcation behavior was observed through applying a range of creep stresses. When applied stress is sufficiently low viscosity increases and the material yields, exhibiting viscoelastic solid-like behavior. Beyond a critical stress viscosity decreases and the material flows, exhibiting viscoelastic liquid-like behavior. Through examining this bifurcation behavior we found that the transition of viscosity occurs at very low strains. The strains at which this transition occurred were compared with critical strains measured through low amplitude oscillatory shear. Results provided support that the solid-liquid transition occurs beyond the critical stress measured through creep, thereby tying it to static yield stress. The protocol was implemented to probe pastes modified with attapulgite clays, a highly thixotropic system, and was found to be effective in characterizing static yield stress and thixotropic rebuilding.

1. Introduction

Yield stress has been extensively studied in non-Newtonian fluids, including colloidal gel, bentonite, polymer suspension, food, paints, waxy crude oil, pastes, foams, etc. [1–5]. Specifically in cementitious materials, yield stress is important in quantifying flowability [6] and is correlated to conventional field-friendly measurement methods such as slump flow and flow table test, both experimentally and in simulation [7]. The large application and research on self-consolidating concrete (SCC) has aroused great interest and study on yield stress because it is closely related to formwork pressure [8–14], stability of coarse particles in self-consolidating concrete (SCC) [15] and multi-layer casting [16].

Since the publication of the Bingham model [17], many models have been proposed to fit the equilibrium flow curve. The most common models for cementitious materials are Bingham, $\tau = \tau_y + \eta_{pl}\dot{\gamma}$, where τ is the shear stress, $\dot{\gamma}$ is the shear rate, τ_y is the Bingham yield stress and η_{pl} is the plastic viscosity; and Herschel-Bulkley, $\tau = \tau_y + k\dot{\gamma}^n$, where k and n are constants based on material properties. Mortar and SCC are usually regarded as Bingham materials, showing a linear relationship between shear stress and shear rate [8,18,19]. While, because of shear thinning of cement paste [20] and shear thickening of normal concrete [21], cement paste and normal concrete are commonly fitted with the Herschel-Bulkley model, in which $n < 1$ for shear thinning and $n > 1$ for shear thickening. With these models, the equilibrium flow curve quantifying the relationship between equilibrium shear stress and shear rate is fitted and the dynamic yield stress is obtained.

In contrast to the dynamic yield stress obtained from the steady-state flow curve, thixotropic materials also exhibit a static yield stress [22,23]. The dynamic yield stress is the stress necessary to terminate flow, measured after the material has been sheared and the microstructure has been broken down. In contrast, the static yield stress is the stress necessary to initiate flow, corresponding to a well-connected microstructure. A common protocol to characterize static yield stress is to apply an intermediate constant shear rate and to record the resultant shear stress [23,24]. The typical response of cement-based materials is an initial increase to a peak and subsequent decrease to an equilibrium value over time. The increase in stress is related to the gradual deformation of the material and the peak stress marks the static yield stress of the suspension that must be overcome to initiate continuous flow. However, this method is shear rate dependent [25]. This presents an experimental challenge when attempting to characterize systems that exhibit very high rates of flocculation. For instance, systems modified with highly purified attapulgite clays were found to significantly accelerate thixotropic rebuilding kinetics [26]. As a result, the stress growth protocol is not a suitable approach to characterize such systems. Another method is to apply creep recovery, which was implemented by Struble and Schultz on cement paste systems [20]. This is a stress-controlled test, where the material will not flow until the applied creep stress is

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higher than the critical creep stress. They observed the transition from solid-like behavior to liquid-like behavior with a slight increase in applied stress. They compared the measured stress at which this transition occurs to the dynamic yield stress measured through fitting the flow curve with a steady-state flow model. Here, we adopt their approach and tie it to static yield stress, then extend it to characterize thixotropic rebuilding over time.

In this study, strain and viscosity development of cement paste under a creep recovery test will be presented and discussed. Through incrementally increasing applied stress, a viscosity bifurcation is found to occur beyond a critical stress. Through low amplitude oscillatory shear, the critical strain at which the solid-liquid transition occurs is measured and is compared to the shear strains measured in the creep recovery test. The strains at which the material transitions to either flow or no flow were found to exceed the critical strain only once the critical stress had been exceeded. This supports that the critical stress measured through the creep recovery test is static yield stress. The test is extended to characterize thixotropic rebuilding of cement pastes modified with attapulgite clays by measuring the development of static yield stress over time, up to 60 min.

2. Theoretical background of approach

2.1. Creep recovery test

Creep recovery test has long been applied to probe the viscoelastic properties of materials. The protocol and various viscoelastic responses are shown in Fig. 1. A shear stress is applied instantaneously to the material, held constant for a period of time, and then removed to (Fig. 1a) [20,27]. For an elastic solid (Fig. 1b) the load-deformation relationship is proportional, where the strain changes instantaneously with stress and stays constant; when the stress is removed the strain instantaneously drops to 0 and stays constant. For a viscous liquid (Fig. 1c) the load-deformation rate relationship is proportional, where the strain linearly increases with time with no initial strain; when the stress is removed there is no recovery and the strain remains constant. For viscoelastic

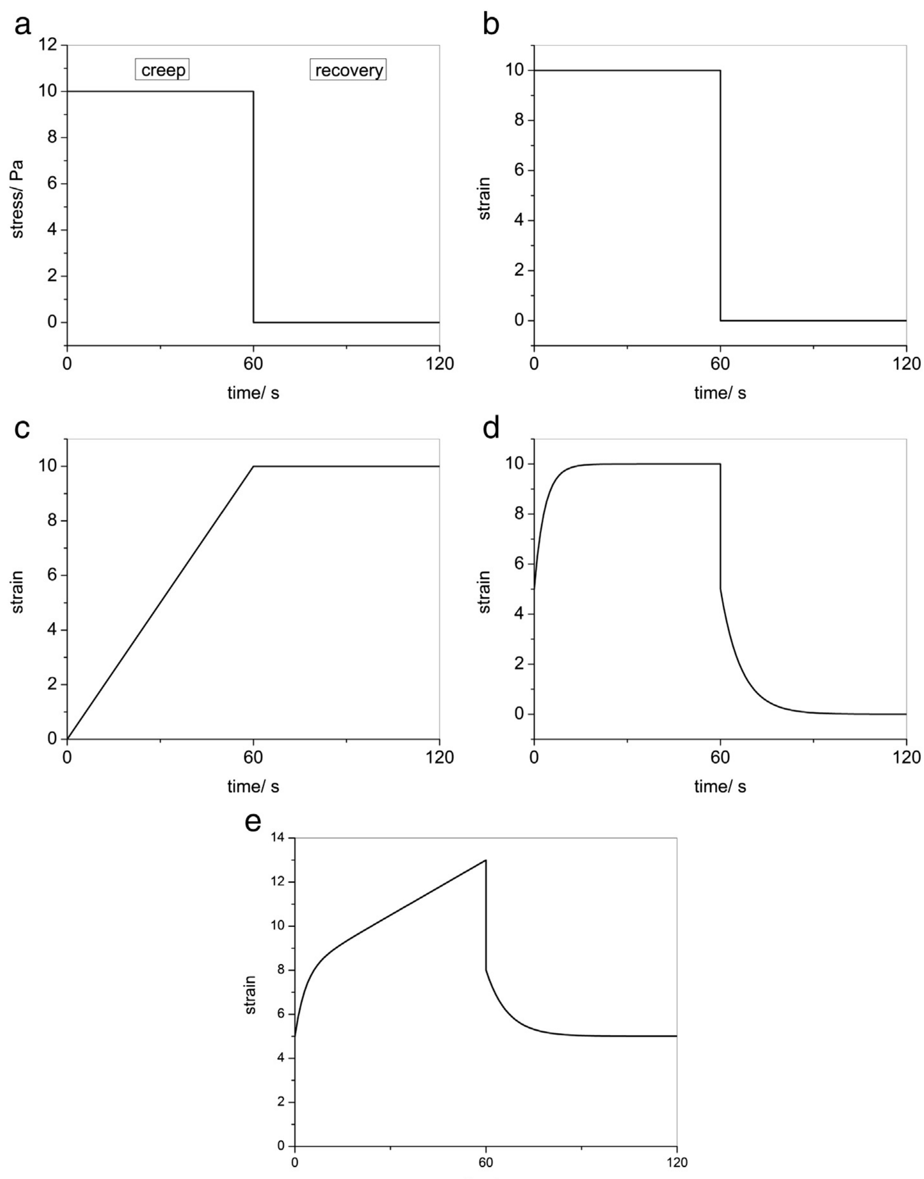

Fig. 1. (a) Applied creep recovery protocol, and strain development of (b) elastic solid, (c) viscous liquid, (d) viscoelastic solid and (e) viscoelastic liquid.

materials, there is an instantaneous strain, which is then followed by a time-dependent strain. For viscoelastic solids the strain reaches an equilibrium value (Fig. 1d) while for viscoelastic liquid the strain continues to increase (Fig. 1e). Upon removal of stress, viscoelastic solids can recovery substantially while viscoelastic fluids exhibit little to no recovery.

Many researchers have found that under different stresses, fresh cement-based materials show different strain development. Struble and Schultz [20] found that under low shear stresses cement pastes show a creep recovery response similar to viscoelastic solids, with strain reaching equilibrium under creep and partial recovery of strain upon removal of stress. Under higher shear stresses cement pastes show a similar response to viscoelastic liquids, where strain continues to increase under creep and exhibits no recovery. Struble and Schultz reasoned that the solid-liquid transition occurs within a narrow range of shear stresses and can be linked to the yield stress of the material.

Fresh cementitious materials are thixotropic, exhibiting flocculation due to diffusion/coagulation processes and formation of CSH bridges due to early hydration under rest (structural rebuilding) and deflocculation and breakage of CSH bridges under shear (shear rejuvenation) [28]. With respect to macroscale flow, this translates to increase in viscosity under rest and decrease under shear. Using the inclined plane test, Coussot et al. [29,30] observed avalanche behavior in yield stress fluids. There exists a critical slope, which corresponds to a critical stress applied on the fluid. Within the critical slope the fluid exhibits little to no flow, while with only a slight increase in slope the fluid flows much longer and faster. It is believed to be the effect of thixotropy and the competition between structural rebuilding and shear rejuvenation. When stress is not sufficiently high structural rebuilding is dominant over shear rejuvenation, thus the flow stops eventually; while with slightly higher stress shear rejuvenation becomes dominant and the material exhibits continuous flow that accelerates until steady-state is reached. In this study, it is proposed that the creep recovery protocol can be implemented to observe similar behavior through viscosity bifurcation and subsequently used to characterize static yield stress.

2.2. Oscillatory test

The oscillatory shear approach has been largely applied to study the viscoelastic properties of fresh cement pastes. Low amplitude oscillatory shear (LAOS) can be applied to obtain the evolution of storage and loss modulus [31]. And amplitude sweeps can be applied to obtain measures of critical strain [32]. The theory is briefly explained here.

An oscillatory strain is applied as a sine function:

$$\gamma = \gamma_0 \sin(\omega t) = \gamma_0 \sin(2\pi f t) \tag{1}$$

where γ_0 is maximum strain amplitude [rad], t is time [s], and ω is angular frequency [rad/s] and f is frequency [Hz]. If the strain is sufficiently low so that the particles in the suspension remain close to each other, the microstructure is not disturbed and the material can recover elastically. In this case (linear regime), the measured response in terms of stress is as follows:

$$\tau = \gamma_0 \left[G' \sin(\omega t) + G'' \cos(\omega t) \right] \tag{2}$$

where G' is the storage modulus, and G'' is the loss modulus. The storage modulus represents the elastic or in-phase component and the loss modulus represents the viscous or out-of-phase component. For an ideal elastic solid, G'' equals 0; and for an ideal fluid, G' equals 0.

The first step in performing oscillatory shear rheometery is to find the linear viscoelastic region (LVR), where G' (and also G'') is independent of applied frequency and strain. For a flocculated suspension, particles are able to recover elastically as strain is oscillated until some critical strain amplitude is reached. Within this critical strain, which is the LVR, strains are sufficiently small so that the structural integrity of the flocculated network is maintained. Above the critical strain, beyond the LVR, strains are sufficiently high so that particles are not able to recover elastically, modulus decreases, and the material behaves as a viscoelastic liquid.

To find the LVR a strain sweep is performed on cement pastes, during which the sample is subjected to an oscillatory strain of constant frequency and increasing amplitude, which may range in the order of 5 or 6. The corresponding stiffness G' curve will exhibit two main branches, as shown in Fig. 2: a plateau region corresponding to the LVR and a decreasing branch indicating that the sample is experiencing a shear-induced breakage. The strain at which there is an apparent drop of stiffness marks the end of the LVR and is considered to be the critical strain. Frequency must also be considered to find the LVR. If the frequency is too high, the structure cannot recover and the measured LVR will be inaccurate.

Here, we will implement the amplitude strain sweep to measure critical strain, at which the transition from viscoelastic solid to viscoelastic liquid occurs. This will serve to supplement the results of the creep test to verify that the stress measured can be tied to static yield stress. Critical strain can also provide some insight into the fresh-state microstructure. Therefore the effect of attapulgite clay addition on critical strain will be measured.

3. Material and procedures

3.1. Materials and experimental setup

Type I Portland cement is used in all mixes. According to ASTM C150 [33], its compressive strength at 28 days is 44.8 MPa, the Blaine fineness is 420 m^2/kg and the chemical constituents are summarized in Table 1. Mixes are prepared with tap water. Highly purified attapulgite clay is also added to select mixes. The nanoclay is a highly purified magnesium alumino-silicate. It is a commercially available clay that is chemically exfoliated from bulk attapulgite to remove all impurities. When dispersed, it is needle-like with an average length of 1.75 μm and diameter of 3 nm, so it will be referred to as nanoclay herein. In this study, no chemical admixtures are used.

Mixes have a water-to-cement ratio (W/C) of 0.36 by mass. As a parameter, the amount of nanoclay varies from 0, 0.1, 0.3 and 0.5% by mass of cement.

Nanoclay powder is blended with the mixing water in a Waring blender for 2 min to produce a clay suspension, which remains stable after 6 h at rest. Immediately after preparing the suspension, it is poured into a beaker. Cement powder is slowly added to the suspension and mixed by hand for 1 min, then mixed with a hand mixer at a speed of 1100 rpm for 3 min. The fresh cement paste is loaded into the cup of the rheometer with a syringe to ensure each sample is the same volume.

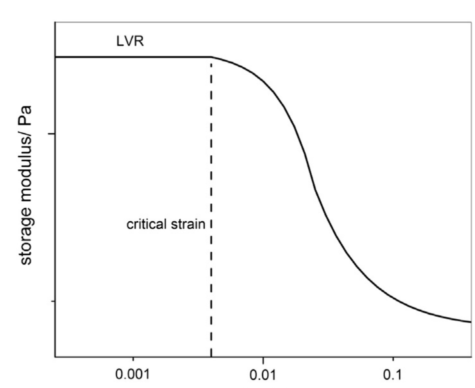

Fig. 2. Critical strain measured through low amplitude oscillatory strain sweep.

Table 1
Cement chemical constituents.

Constituents	% by mass
SiO_2	19.22
Al_2O_3	4.98
Fe_2O_3	3.42
CaO	62.42
MgO	3.87
SO_3	2.72

We perform all tests on a HAAKE MARS III rotational rheometer. We use a coaxial cylinder geometer – the radius of the bob is 12.54 mm and the gap between the bob and cup is 1.06 mm.

3.2. Shear rheological protocols

As cementitious materials are thixotropic, the flow behavior is greatly dependent on the flocculation state. It must be consistent before each test to guarantee repeatability. For all protocols in this study, each sample is presheared at 600 s^{-1} for 4 min until the shear stress reaches equilibrium, after which the material is allowed to rest for a period of time for stress relaxation and to reach a stable state. Then, either the creep recovery test or oscillatory test is applied. In the creep recovery protocol, we apply the creep step for 60 s at each applied stress level and monitor its recovery for up to 60 s. In the strain amplitude sweep test, we apply strains from 10^{-5} to 10^{-1} at a fixed frequency of 1 Hz, as conducted in many studies on cement paste [31,34,35].

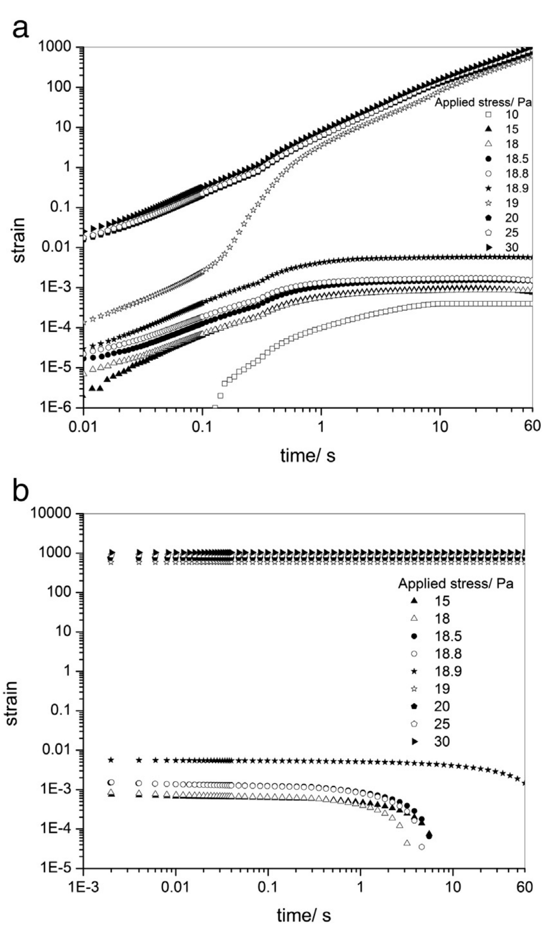

Fig. 3. Strain development of cement paste under (a) creep and (b) recovery.

4. Results and discussion

4.1. Creep-recovery - viscosity bifurcation

We implement the creep-recovery protocol on neat cement pastes. Fig. 3 and Fig. 4 show the strain and viscosity development under various creep stresses, respectively. As shown in Fig. 3a, when creep stress is below 18.9 Pa the strain reaches a plateau and flow ceases. When creep stress is higher than 19 Pa the strain increases and the material continues to flow until creep stress is retrieved. Supplementing this with the recovery, Fig. 3b, it is observed that within 18.9 Pa the material exhibits some recovery (viscoelastic solid) and beyond 19 Pa there is no recovery (viscoelastic fluid).

As could be seen in Fig. 4, under creep the viscosity exhibits an initial plateau and then either increases or decreases. When creep stress is below 18.9 Pa the viscosity increases to infinite values; when higher than 19 Pa the viscosity decreases to diminishingly small values. This resembles the avalanche behavior of thixotropic materials, as discussed prior. Thus this bifurcation point can be tied to the transition between viscoelastic solid to liquid, and the corresponding critical stress may be considered to be the static yield stress. It can be seen that the transition occurs at the very early ages of creep. Under stresses higher than the static yield stress, the material transitions from a solid-like structure to a liquid-like structure. It is also assumed that this transition is related to the microstructure of material. This is explored through LAOS and discussed in the next section.

4.2. Solid-liquid transition

4.2.1. Amplitude sweep - critical strain

We implement amplitude and frequency sweeps on fresh cement pastes. Through monitoring G', we measure the LVR and corresponding critical strain. A frequency sweep experiment is performed in which frequencies from 0.1 Hz to 20 Hz are applied at a fixed strain of 10^{-4}. Results of frequency sweeps indicated that 1 Hz is within the LVR, which is also commonly applied in other studies [31,35,36]. Therefore this fixed frequency is used for all amplitude sweeps. The results of an amplitude sweep on neat paste are shown in Fig. 5, where strains ranging from 10^{-5} to 0.1 are applied. Beyond a critical strain, storage modulus G' decreases dramatically, showing that the structure is broken down. In addition, beyond this point G'' becomes greater than G', indicating the transition from solid-like to liquid-like behavior. The average value of the critical strain measured through low amplitude oscillatory sweep of neat paste is 7.69×10^{-4}, which corresponds to the same order of magnitude as those reported in other works [31,37].

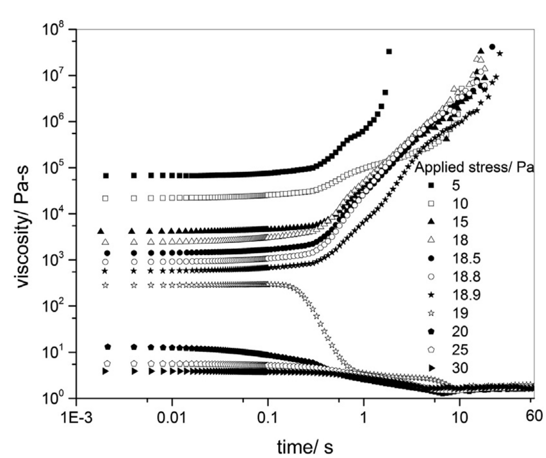

Fig. 4. Viscosity bifurcation of cement paste.

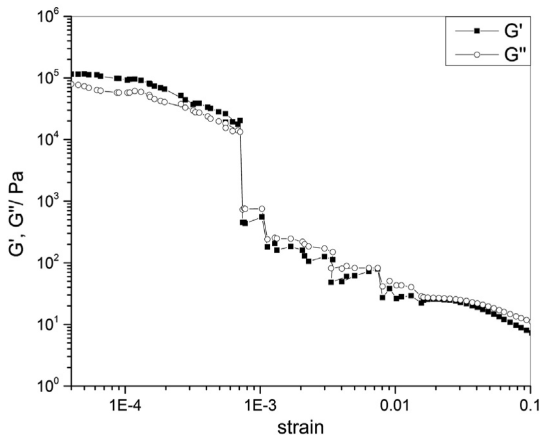

Fig. 5. Strain sweep of cement paste with w/c at 0.36.

4.2.2. Solid-liquid transition

The transition strain is recorded from viscosity evolution plots (Fig. 4), considered to be when the viscosity becomes 20% higher or lower than the initial plateau value, at each creep stress. Transition train is plotted against applied stress and presented in Fig. 6. In addition, these values are compared against the critical strain as measured by LAOS and critical stress as measured by creep protocol, indicated by the dotted lines. With increasing creep stress, the transition strain increases, as shown in Fig. 6a. It could be seen that under creep stresses lower than the critical stress the transition strain remains within the critical strain, implying that the microstructure remains intact, as shown in Fig. 6b. Further, under creep stresses higher than the critical stress the transition strain exceeds the critical strain, indicating the microstructure is no longer able to recovery elastically and the solid-liquid transition occurs. Only when applied creep stress exceeds the static yield stress could the material show solid-liquid transition. This will be discussed further in the following subsection.

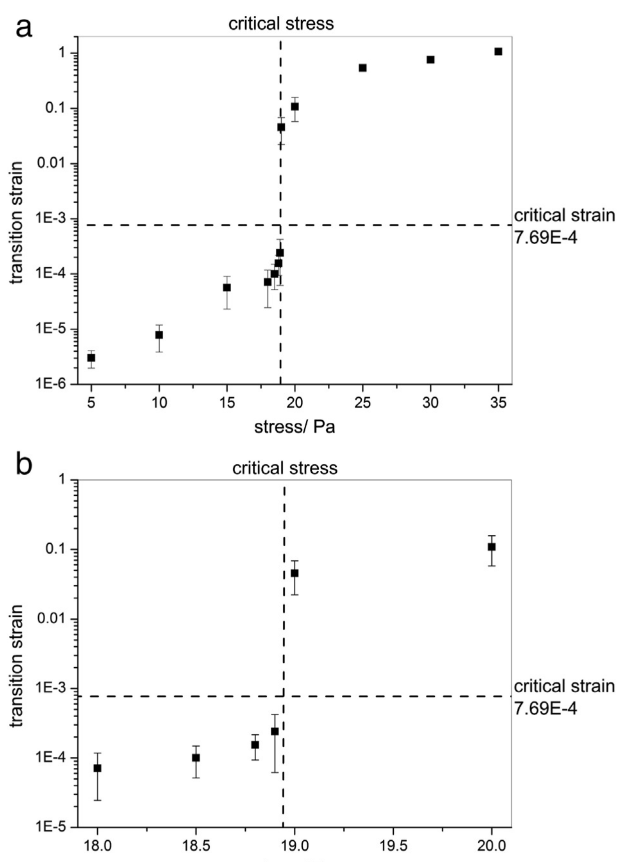

Fig. 6. Transition strain versus applied creep stress, in comparison with critical stress and critical strain – (a) full and (b) partial plot.

4.2.3. Schematic diagram of creep behavior

Baudez and Coussot [38] found that under creep in a couette geometry, typical yield stress materials (i.e. Carbopol gel and foam) initially deformed homogeneously. Then beyond a critical deformation the inner rotor region deformed much more quickly than the region near the wall, indicating solid-liquid transition occurred near the rotor region. Although Carbopol gel and foams have much higher critical strains than cement pastes, they still show very similar yield stress and viscoelastic properties. The authors believe that before the transition between flow and no flow, the viscosity at the plateau in Fig. 4 is the instantaneous response of the viscoelastic solid material. At this point, the transition has not yet occurred, so the material is homogeneously deformed. And the higher the creep stress, the higher the transition strain.

After the transition, the bifurcation behavior is determined by whether the strain exceeds the critical strain. As illustrated in Fig. 7, with increasing stress the transition strain increases. For creep stresses within the static yield stress, the transition strain is smaller than the critical strain. The structure is not broken so viscosity approaches infinity and flow ceases. In contrast, for creep stresses exceeding the static yield stress the transition strain is higher than the critical strain. The structure is broken, the flow continues and the viscosity decreases due to shear rejuvenation. The agreement between the oscillatory amplitude sweep and creep-recovery tests provides support that the critical stress measured through the creep protocol is static yield stress.

4.3. Effect of nanoclay

4.3.1. Static yield stress

As mentioned prior, due to the rate dependence of the stress growth protocol in measuring static yield stress it is not a suitable approach for characterizing highly thixotropic mixes. Therefore here we implement the creep-recovery protocol to measure the static yield stress of cement pastes modified with highly purified attapulgite clays, which exhibit high rate of thixotropic rebuilding at short time scales [26]. The results of static yield stress as measured by the creep-recovery protocol on cement pastes with nanoclay addition are shown in Fig. 8. It is shown that there is a linear increase of static yield stress with nanoclay addition. With 0.5% of nanoclay addition over cement, the static yield stress increases 3 times. This is in good agreement with the results found that

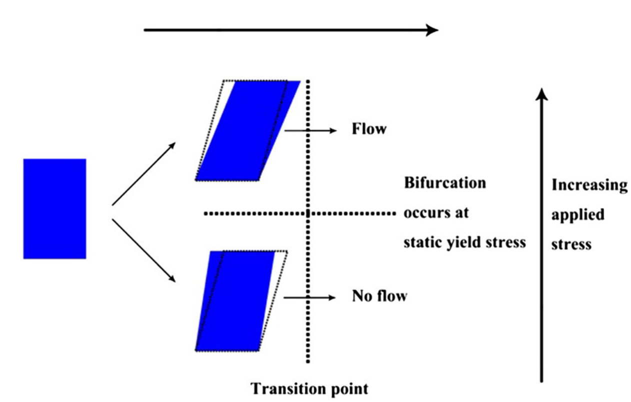

Fig. 7. Schematic depicting the comparison between transition strain (creep) and critical strain (LAOS) – different applied stress leads to different flow behaviors.

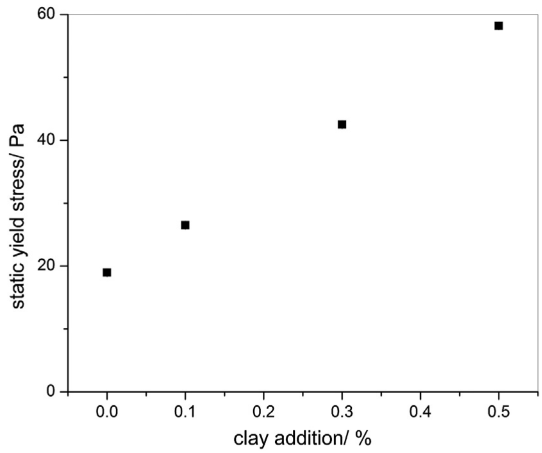

Fig. 8. Effect of nanoclay addition on static yield stress.

measured the effect of these nanoclays on cohesive strength as measured by the tack test [37].

It is considered that [16] of the two components of thixotropy, i.e. de-flocculation under shear and flocculation at rest, the latter is more important because most of the problems encountered in SCC application (i.e. formwork pressure, multi-layer casting, and stability) are all related to resting. The accurate quantification of static yield stress is the key to solving these problems. A_{thix} quantified as the increment of yield stress over resting time, shown in Eq. (3), has been proposed as a useful parameter to describe the thixotropy of materials [16].

$$\tau_0(t) = \tau_0 + A_{thix}t \tag{3}$$

Nanoclay has been found to effectively decrease formwork pressure in lab-scale studies [39]. And this has been attributed to its effect on thixotropic rebuilding over time. Therefore we measured the effect of nanoclay on static yield stress over time. The results are shown in Fig. 9. It is apparent that with increasing resting time, the static yield stress of both cement paste with and without nanoclay increase. From 1 to 30 min, the thixotropic rate A_{thix} of cement paste with 0 and 0.5% nanoclay are 2.35 Pa/min and 6.61 Pa/min, respectively. From 30 to 60 min, A_{thix} of cement paste with 0 and 0.5% nanoclay are 5.43 Pa/min and 11.25 Pa/min, respectively. With addition of nanoclay, the cement paste exhibits higher rate of thixotropic rebuilding. As modeled by Ovarlez and Roussel [11], the higher the A_{thix}, the lower the formwork pressure. Thus, this effect on static yield stress with resting time by the nanoclays can help partially explain the substantial decrease in formwork pressure over time measured in previous studies [39].

A_{thix} also increases with longer resting time. From the first 30 min to the second 30 min of resting, the A_{thix} of cement paste without nanoclay increases 2.31 times, while it increases 1.70 time with 0.5% of nanoclay. A potential explanation may be tied to cement hydration. Studies by Kawashima [26] showed that after 30 min of resting, storage modulus of cement pastes without nanoclay becomes higher than those with 0.5% nanoclay, implying that the effect of nanoclays on thixotropic rebuilding may be hindered by the progression of hydration. This is currently under investigation.

4.3.2. Critical strain

The yield stress of cement paste originates from the microstructure of an attractive particle-particle network, connected through colloidal interaction or direct contact. The microstructure sustains a certain amount of stress before it is broken and starts to flow, which defines the yield stress. Other studies have measured the clay's effect on floc size and strength. However, characterization was limited to steady-state shear conditions [40,41]. To gain insight into how the attapulgite clays affect the undisturbed fresh-state microstructure at rest, we measure the critical strain.

Results of LAOS on nanoclay-modified cement pastes are shown in Fig. 10. They indicate that critical shear strain decreases with nanoclay addition. Due to their fine size compared to cement particles – nanoclays are 1.75 μm in length, 3 nm in diameter while Ordinary Portland Cement has an average particle size of 15 μm – we may attribute this to the clays filling the voids between cement particles and connecting the bonds between cement particles; or due to surface and edge charge effects nanoclay itself also forms card-house type structures, contributing to the connectivity of the cement paste microstructure. In section 4.2.2 it was discussed how the critical strain measured by LAOS agreed well with the solid-liquid transition measured by creep. The authors would like to point out that similar behavior was found for all pastes modified with nanoclay addition, i.e. at 0.1, 0.3 and 0.5%, tested in this study. So the theory described by Fig. 7 also applies to pastes systems with nanoclay.

5. Conclusions

The creep recovery test is applied to probe the viscoelastic behavior of cement paste and a viscosity bifurcation is observed. Within a critical stress the strain approaches equilibrium, indicating no flow. Beyond a critical stress the strain increases dramatically and no equilibrium value is reached, indicating flow is initiated and continues. The critical stress corresponding to this viscosity bifurcation is considered to mark the solid-liquid transition of the material and so is considered to be a

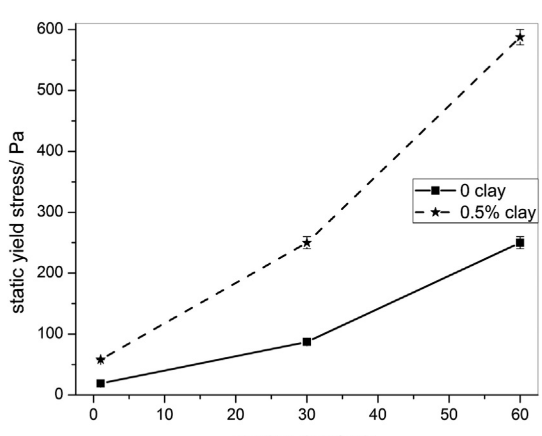

Fig. 9. Effect of resting time on static yield stress of cement pastes with 0 and 0.5% of nanoclay.

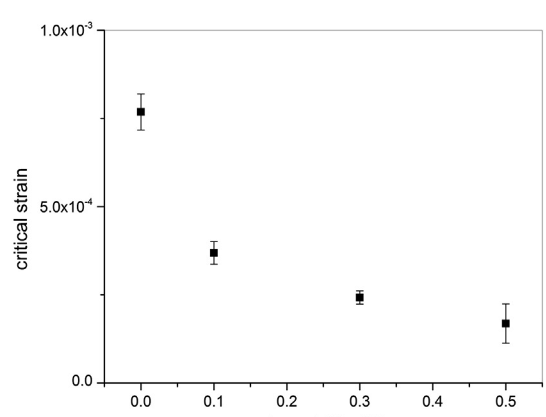

Fig. 10. Effect of nanoclay addition on the critical strain of cement pastes, measured through low amplitude oscillatory sweep.

measure of static yield stress. Measures of critical strain by LAOS are found to support this. The methods are applied to characterize the thixotropic rebuilding of cement pastes modified with nanoclays up to 0.5% by mass of cement. The creep protocol is found to effectively capture the static yield stress of highly thixotropic systems. Further, the increase in static yield stress over resting time is found to be higher for cement paste with 0.5% nanoclay over neat paste. This helps to elucidate how nanoclays can reduce formwork pressure over time. Nanoclays are also found to decrease critical strain measured by LAOS, which provides evidence that they are increasing the interconnectivity of the fresh-state microstructure.

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