

The shear-induced solid–liquid transition in yield stress materials with chemically different structures

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Abstract

The shear-induced solid–liquid transition of viscoplastic materials has been studied extensively through various steady-shearing experiments including steady stress sweep, stress ramp, creep and strain recovery. The results are consistent in showing a clear change in behaviour of the materials from solid-like to liquid-like. A transition stress plateau separates the regions of solid behaviour and liquid behaviour. The slope of the plateau reflects the uniformity of the structure, and hence the distribution of bonding strength within this structure. Depending on the structure of the material, the yielding process of viscoplastic materials can occur over a wide or a narrow range of stress, which represents “ductile-type” or “brittle-type” failure. Altering the concentration and extent of particle flocculation (for suspensions) or polymer chain entanglements (for polymer gels) can vary the bonding strength and strength distribution, and therefore change the slope of the stress plateau. The continuous solid structure exhibits creep at stresses well below the yield stress and fails at a critical strain. The yielding of the material seems to be characterised by a critical strain rather than by a critical stress or a critical shear rate. The deformation of viscoplastic materials can be recovered fully or partially in the solid-like region once the stress is removed. This is a significant difference from the behaviour of purely viscoelastic materials. Strain recovery tests result in two characteristic strain values that can be used to define the two commonly used “yield stress” values, the higher one of which is in good agreement with the traditional value of yield stress as measured by vane torsion, for example. © 2004 Elsevier B.V. All rights reserved.

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1. Introduction

Very soft (weak) solids such as concentrated mineral suspensions, drilling muds, solutions of thickeners, many food stuffs and cosmetics are widely processed on a large scale. During processing, or use, conditions alternate between sheared bulk flow in “liquid” form, and gelation or solidification under unsheared, stagnant conditions. Solid behaviour is often a specified requirement for these materials, as in the “slope disposal” of mineral tailings; in oil drilling-fluids which must be able to entrap bore-solids within a structured matrix; long-distance pipeline of suspensions which must be able to carry large particles without settling; in formulating

foods, cosmetics and paints, which must be spreadable but must not flow once shear is removed.

The strength of such weak solid-like materials has been characterised traditionally by a yield stress. Although the inadequacy of the rheological definition of this property has generated worldwide debate on the existence of a true yield stress [1–9], the concept remains useful, and the yield stress remains an important parameter in rheological characterisation of complex fluids and in understanding their processing characteristics in industrial applications. Yield stress is generally regarded as the transition stress between elastic solid-like behaviour and viscous liquid-like behaviour, and is related to the presence of an internal network structure. It was found that at small deformation up to a limit, there is a linear zone of stress versus strain and a yield stress as the transition point from linear to non-linear zones can be determined at

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such limit [10]. However, many researchers have suggested that transition typically occurs not at a single point, but instead over a range of stress starting at a lower limit, corresponding to progressive transition between elastic and plastic deformation and ultimately ending at a higher limit, corresponding to the transition between plastic deformation and bulk viscous flow [11–13]. This leads to the definition of two types of yield stress, i.e. a static and a dynamic yield stress [14–17]. The presence of a stress plateau has been taken as indicating the existence of a yield stress, and a redefinition in terms of this plateau has been suggested [8]. Uhlherr et al. [13,18] have found that, for traditional yield stress materials, the stress plateau coincides with an unambiguous transition from solid-like behaviour to liquid-like behaviour. However, the presence of a stress plateau is not uniquely associated with a yield stress but occurs for many shear-thinning polymer solutions as well [8]. Yield stress is an important and much used rheological concept, but its definition is in urgent need of clarification. In this paper, the transition behaviour of traditional yield stress materials is investigated extensively by various steady-shearing tests. The results provide information about the yielding behaviour of such materials and its dependence on material structure.

Yield stress materials normally are multiphase systems. For example, in a suspension, the solid particles are dispersed in a liquid medium and interact with each other to create a continuous network with a flocculated structure. The suspension flows only when the stress is large enough to break down the network structure. Hence, the yielding process of the material must be related to the strength of the coherent network structure. The yield stress has been analysed in a number of studies [19–26] in terms of surface chemistry conditions and physical parameters of the materials, such as zeta potential, solids concentration, particle diameter and particle size distribution. However, an understanding of the role of the material structure in the transition process of yield stress materials is still lacking. As already stated, the transition of rheological behaviour from solid-like to liquid-like is characterised by a stress plateau. It is hypothesized here that the steepness of the stress plateau reflects the uniformity of the solid structure, and hence the distribution of “bond” strength. Depending on the width of this distribution, the yielding process could occur over a wide or narrow range of stress, representing a “ductile-type” or a “brittle-type” of failure. To test the above hypothesis, systematic studies have been carried out on the transition behavior of chemically different structured materials using a range of techniques. The information gained from this study adds to our understanding of the yielding process of viscoplastic materials.

2. Materials and experimental work

Materials with chemically different structures have been formulated including kaolin, TiO_2 and $\text{SiO}_2/\text{TiO}_2$ suspensions, as well as Carbopol 934 gels. The different structures

of these materials are expected to influence the observed shear-induced transition behaviour. Electrostatic coagulation is normally considered to be responsible for the bonding of particles in kaolin and TiO_2 suspensions, with bond strength of the network thus dependent on surface chemistry conditions. The morphology of kaolin and of TiO_2 pigment is very different: TiO_2 pigment contains sphere-like particles, while kaolin is present as plate-like particles. The surface chemistry and rheology of kaolin suspensions are more complicated than those of TiO_2 suspensions because of the presence of heterogeneously charged edges and faces on each particle. A suspension of SiO_2 mixed with small amount of TiO_2 represents a different flocculated state again. The incorporation of small quantities of metal oxides into aqueous silica slurry can develop strong colloidal interaction, which has a marked effect on the flow properties leading to a significant increase in yield stress [27]. The electrostatic attraction between the small TiO_2 particles ($0.3\ \mu\text{m}$) and the larger SiO_2 particles ($9\ \mu\text{m}$), which are negatively charged at neutral pH, leads to the formation of large flocs that hold a quantity of water. Carbopol gel is commonly used as model material in studies of viscoplastic behaviour because of its stable and transparent nature. The strength of Carbopol gel may come from the entanglements and confinement between microgel particles.

A grade A-HR TiO_2 pigment from Tioxide Australia Pty Ltd. was used to prepare the TiO_2 suspensions with different concentrations and pH values. The pigment is made in anatase form, with no inorganic coating, but treated with organics. The average crystal size is $0.15\ \mu\text{m}$ (supplier's data). A grade 400G silica flour, which was obtained from Unimin Australia Ltd., was used for suspensions with small amount of R-HD2 grade TiO_2 pigment, which was also from Tioxide Australia Pty Ltd. Kaolin suspensions were prepared using a product from Commercial Mineral Pty. Ltd.

The particle size distributions for TiO_2 pigments and silica flour were measured using a Malvern MasterSizer/E. To break up loosely agglomerated particles, samples were dispersed by intensive stirring and ultra-sonication. The volume average particle size is found to be $0.3\ \mu\text{m}$ for R-HD2 TiO_2 , $0.5\ \mu\text{m}$ for A-HR TiO_2 and $9\ \mu\text{m}$ for 400G silica flour, respectively. Zeta potential of the pigments was measured as a function of pH by electrophoresis using a Malvern Zetasizer 4. The isoelectric point (IEP) is found at around pH 2.4 for A-HR TiO_2 , pH 6.5 for R-HD2 TiO_2 and pH 1.5 for silica flour.

Mixed suspension was formulated with a $\text{TiO}_2/\text{SiO}_2$ ratio of 0.12. At natural pH, the A-HR TiO_2 pigment is well dispersed in water without significant flocculation, and no yield stress is detected for the suspension. To prepare a stock suspension, HNO_3 in deionised water was added to create a suspending medium with low pH, and then TiO_2 pigment was dispersed in this acidic water. The pH of the resulting suspension was further adjusted by adding more 2 M HNO_3 . The suspension was then gently agitated for at least 24 h to ensure an equilibrium state. The stock suspension has a concentration of 50 wt.% solid and a pH value of 4.8. Suspensions with different pH values and concentrations were obtained by

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