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Electrorheological Responses of Particulate Suspensions and Emulsions in a Small-Strain Dynamic Shear Flow: Viscoelasticity and Yielding Phenomena

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Abstract–The dynamic rheological behavior of multiphase electrorheological (ER) fluids was considered, as continuation of a previous paper [Chin and Park, 2000]. Oil-in-oil emulsions, which differ in electrical conductivity and dielectric constant, were employed for an ER-active emulsion and also for a multiphase ER fluid with enhanced performance. The polyaniline particle suspension in an electric field showed viscoelastic behavior within a very limited range of strain amplitude, indicating the transition from viscoelasticity to viscoplasticity. Within the region of viscoelasticity, the linear region was restricted below the amplitude of 0.1%, whereas the ER-active emulsions showed a rather wide linear regime. Frequency dependence of the storage shear modulus in the linear viscoelastic region revealed the typical features of an elastic solid. When the fraction of emulsion drops (Φ) in multiphase ER fluids increased, the limiting strain for viscoelasticity showed a higher value.

Key words: Electrorheology, Multiphase ER Fluid, Dynamic Viscoelasticity, Yielding, Viscoplastic Behavior

INTRODUCTION

Conventional electrorheological (ER) fluids are typical examples of field-responsive materials and have the form of concentrated suspensions of micrometer-sized solid particles in insulating medium. Upon application of an electric field as strong as several kV/mm, a dramatic change of apparent viscosity and resultant yield stress appears for ER fluids. Obviously, this rheological change which appears on a time scale of millisecond is caused by the formation of microstructures among the dispersed phase in medium, so that the electric field-induced polarization process on the bulk, surface, and solid-liquid interfaces is generally regarded as the fundamental mechanism of ER response. Numerous experimental and theoretical studies have been performed on ER phenomena, in order to realize the great potential for developing an ideal electrical-mechanical interface with excellent controllability. However, limited understanding of the mechanism related to ER effects creates significant difficulties in developing the desired materials and application devices.

Rheological behavior of ER materials under the influence of an electric field has been most commonly characterized in terms of their properties during steady shear flow. Due to significant particle-particle interactions, they usually exhibit a yield stress, meaning a certain minimum stress that must be exceeded for flow to occur. The yield stress is hence a material property denoting the transition between "solid-like" and "liquid-like" behavior. However, simple models of Bingham fluids and Herschel-Buckley equations, although adequate in steady flow situations, overlook the properties of the materials at stresses or strains below yielding. This pre-yield region can be effectively studied under oscillatory shear flow [Garmota and Filisko, 1991; Stanway et al., 1989]. Since the fruitful area of application of ER fluids is in the damping of mechanical deviation--and related application devices such as engine mounts and shock absorbers operate in the dynamic or transient mode--understanding the ER dynamic properties is very important. Furthermore, dependence of the rheological properties on the ER fluid structures can be well understood by using small-amplitude dynamic oscillatory experiments.

Previous dynamic studies performed with sufficiently small shear strain amplitudes suggest that ER materials behave as linear viscoelastic bodies [Garmota and Filisko, 1991; Shulman et al., 1989; Vinogradov et al., 1986]. These studies report that the ER material's linear viscoelastic properties, storage and loss shear moduli, are functions of the electric field strength. However, the limit of the linear viscoelastic range of ER materials is usually less than 1%, although many polymer gels and solutions show linear response up to strains of 10% or higher [Oh et al., 1994; Kwon et al., 1999]. Garmota et al. [1993] analyzed the nonlinear viscoelastic properties of ER fluids by Fourier transform analysis of the stress response. They reported that the appearance of the higher harmonics of the Fourier spectrum suggests the onset of a transition in the mode of deformation in ER materials. This transition to nonlinearity was shown to originate from slight structural rearrangement or rupture of particulate columns, leading to unstable configuration even sheared with low strain amplitude and frequency [Parthasarathy and Klingenberg, 1995]. Although understanding nonlinear behavior is important for the interpretation of actual application devices, there is little information available even on linear response of ER fluids, due to the complexity of the rheology in ER materials itself composed of elastic, viscous, and yielding behavior. Recently, Pan and Mckinley [1997a] compared the dynamic behavior of ER fluids with that of physical gel, suggesting the anisotropic network model that can explain the limiting shear strain amplitude leading to structure failure.

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Table 1. Relevant properties of the constituents of emulsion: silicone oil/chlorinated paraffin oil used in this study

Chlorinated paraffin oil				Silicone oil			
Viscosity (Pa s)	Density (g/cm^3)	Dielectric constant	Conductivity (S/m)	Viscosity (Pa s)	Density (g/cm^3)	Dielectric constant	Conductivity (S/m)
4.5	1.16	7.1	8.3×10^{-10}	0.1	0.96	2.6	2.5×10^{-12}
0.3	1.16	7.8	7.1×10^{-10}	0.1	0.96	2.6	2.5×10^{-12}
0.3	1.16	7.8	7.1×10^{-10}	1.0	0.96	2.6	2.5×10^{-12}

In the structures of concentrated ER materials, there exist primary chains of particles spanning the electrodes along the field direction as well as secondary chains tilted with respect to the field direction and interconnecting primary chains. Such a many-body effect of polarized particles causes an inherent limitation in the material strength and narrow range of linear viscoelasticity.

The yield stress behavior and microstructural changes were investigated for ER materials composed of particles and emulsion drops dispersed in a continuous phase in the previous paper of the same authors [Chin and Park, 2000]. The results show not only that enhanced ER activity can be achieved by this route, but also may offer insight into the nature of structure-property relationship in ER phenomena. Dynamic rheological experiments in a small-amplitude shear flow, therefore, can be an effective method for the interpretation of related mechanism and microstructures of such a multiphase ER material.

In this paper, dynamic viscoelastic behavior of an electrorheological oil-in-oil emulsion system is first considered as an ideal case where the dispersed component of emulsion has the higher dielectric constant and conductivity than that of the continuous phase. Of course, these emulsions cannot provide the sufficient material properties required for conventional ER systems. Moreover, the rheological behavior of the emulsion under an electric field is different from that of ordinary particulate suspension in that the dispersed phase experiences a change in shape. Generally, drop deformation, coalescence, electric field-induced re-emulsification, and resultant structural rearrangement of dispersed phase occurs with the influence of shear and electric field [Pan and Mckinley, 1997b]. Nevertheless, the particulate ER suspension can be approximately regarded as an emulsion with infinite value of viscosity of dispersed phase. Therefore, an emulsion system can be studied as a model in ER phenomena in order to investigate the effects of various factors systematically, simply by altering the composition and viscosity of individual components.

Conventional particulate ER suspensions show, as discussed earlier, a linear viscoelasticity in a very limited range of strain amplitude. With increasing strength of electric field, elastic properties become dominant. However, the viscous and plastic nature of the shear stress response (may be included in out-of-phase component) facilitates the complexity in their dynamic rheological response. Addition of emulsion drops with different conductivity may also affect the pre-yield rheological behavior, seeing that such emulsion drops strongly influence the yield stress behavior [Chin and Park, 2000]. Experimental data for the dependence of dynamic viscoelastic functions on the strain amplitude and frequency was presented for the linear and nonlinear regime, within the small strain amplitude that can be approximated as the viscoelastic response. Onset of plastic yielding behavior was also characterized by examining the deviation of stress response from sinusoidal waveform. Emulsion samples of different viscosity ratio were prepared. Dynamic viscoelasticity of ER particle suspensions containing the emulsion drops was investigated in terms of the microscopic change of multiphase ER suspension in an electric field.

MATERIALS AND METHODS

1. Preparation of ER Emulsions

Silicone oils (Shin Etsu Chemical, viscosity η=0.1 and 1.0 Pa·s) and chlorinated paraffin oils (*CPO*; η=4.5 Pa·s, supplied by *Yukong Ltd.* and *Celeclor s45* η=0.3 Pa·s, *ICI Chemical Co.*) were employed. An emulsion of silicone oils and chlorinated paraffin oils was prepared by the simple mixing of a specified amount of individual oils with the magnetic stirrer for 10 min, giving a volume ratio of 90/10 (silicone oil/chlorinated paraffin oil) for a different ratio of viscosity. Individual oils were Newtonian fluids and showed constant viscosity over the wide range of shear rate (0.05-100 s^{-1}). The details of emulsion constituents and their physical properties are summarized in Table 1. The conductivity of chlorinated paraffin oils is about 10^2 times greater than that of silicone oils. Differences in the dielectric constant of these liquids are not so great, as noted in Table 1. Throughout this paper, Φ is used to denote the volume fraction of chlorinated paraffin oil (higher conductive) in the entire liquid phase, and λ denotes the viscosity ratio between the chlorinated paraffin oil and silicone oil.

[$\lambda=\eta$(chlorinated paraffin oil)/η(silicone oil)]

2. Preparation of ER Suspensions Containing Emulsion Drops

As a particulate substrate in ER material, polyaniline used in the previous work [Chin and Park, 2000] was also employed. The conductivity of completely dried polyaniline particle obtained was 5.5×10^{-9} S/m, which is in the range of optimum semi-conductivity. The volume fraction of polyaniline (ϕ_p) was equally set to be 0.130, based on the entire volume of the suspensions. Chlorinated paraffin oil (*Celeclor s45*, η=0.3 Pa·s) with a volume fraction (Φ) of 0.10 and 0.30 were added to the polyaniline in silicone oil suspensions. In these samples, a sharp increase of the yield stress can be observed with increasing the volume fraction of *Celeclor s45* drops [Chin and Park, 2000].

3. Investigation of the Dynamic Rheological Properties

For emulsion systems, forced oscillatory experiments under electric field were performed by using a strain-controlled Advance Rheometrics Expansion System (*ARES*, Rheometric Scientific Co.) equipped with the 100FRTN1 low-range transducer and measuring parallel plate geometry of 50 mm diameter. An electric field (E=

2 kV/mm) perpendicular to the flow direction was applied by high voltage power supply (*Glassman, Model EL5P8L*) to the rotating lower plate by using a low-friction spring electrode. The upper plate was grounded and the gap size was set to 0.5 mm. Since the prepared oil-in-oil emulsions with considerable density difference are unstable and the rheological properties are greatly influenced by both the shear history and the microstructure previously formed by the applied electric field, a constant initial condition was given for each dynamic experiment. The vigorously stirred emulsions (~300 rpm for 1 min) were placed in the test fixture and the individual samples were changed with new ones for each test.

Dynamic rheological properties of the polyaniline in silicone oil suspensions with and without *Celeclor s45* drops were investigated by using an ARES rheometer equipped with 200FRTN1 middle-range transducer and measuring parallel plate geometry of 25 mm diameter. An electric field (E=1, 2, and 3 kV/mm) perpendicular to the flow direction was also applied to the rotating lower plate. The gap spacing was set to be 0.5 mm, too. With the digital oscilloscope (HP 54600B), waveforms of the strain inputs and torque output were recorded and stored in the PC. Storage and loss moduli based on the linear viscoelastic theory were also measured in a limited range of strain amplitude (below 5%) and frequency domain of 0.1-100 rad/s. All the experiments were performed at 25 °C.

RESULTS AND DISCUSSION

1. Viscoelastic Behavior of Electrorheological Emulsions

Characteristics of electrorheological response for an oil-in-oil emulsion system were reported for the first time by Pan and Mckinley [1997b]. In the electric field and dynamic shear flow, the emulsion with more conductive dispersed phase than continuous phase behaves as a viscoelastic material due to the formation of liquid columns that span the electrodes. For the particulate ER suspensions, the limit of the linear range is generally less than 1% strain. Generally, the shorter the range of interparticle force, the smaller is the linear range. However, in the case of emulsion with ER activity, the region of strain for linear viscoelasticity was relatively wide compared with the conventional ER suspension. Fig. 1 gives the result of strain sweep (ω=5 rad/s) for emulsion with Φ=0.1 and λ=45 (see Table 1) under E=0 and E=2 kV/mm. Without an electric field, viscous behavior dominated and linear loss modulus was measured, although stable data could not be obtained for the storage modulus. Under E=2 kV/mm, a clear ER effect can be observed, showing the increase of both G' and G". The linear viscoelastic region can be estimated within the strain range up to 10%, which is a higher limit than that of particulate ER suspensions. When a chain-like column in conventional ER suspension experiences deformation, the process of yielding followed by re-arrangement of chain induces nonlinear behavior even for low strain amplitude. However, the deformed droplets in the electrorheololgical emulsion emerge into the slender worm-like structures, which are readily recovered through complicated processes such as elongation and coalescence, although the dynamic moduli in the emulsion are quite small compared with those of the ER suspension.

In Fig. 2, results of dynamic frequency sweep under linear vis-

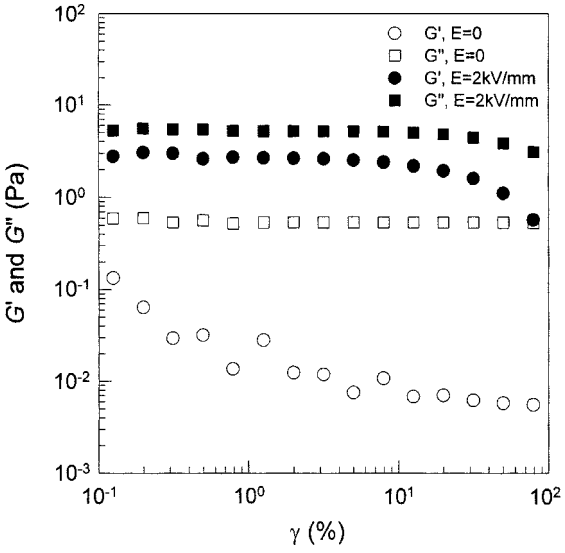

Fig. 1. Storage and loss moduli as a function of strain amplitude with and without electric field; the sample is silicone oil/chlorinated paraffin oil 90/10 (Φ=0.10) emulsion with viscosity ratio λ=45.

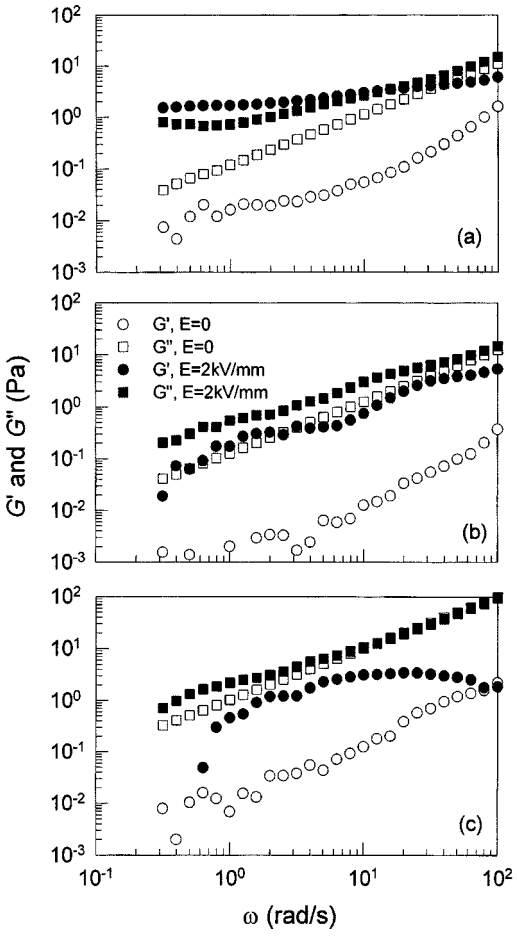

Fig. 2. Dependence of G' and G" on the strain frequency with and without electric field for silicone oil/chlorinated paraffin oil 90/10 emulsion. Viscosity ratio (λ) between chlorinated paraffin oil and silicone oil: (a) λ=45 (b) λ=3 (c) λ=0.3.

coelastic low-strain deformation (5%) are shown for each emulsion in Table 1 with Φ=0.1. Dependence of G' without electric field shows some plateau for emulsion with (λ=45), indicating that dispersed droplets remained spherical due to the large viscosity ratio (λ). For other cases, deformation of droplets can be estimated by the gradual increase of G' and G' with ω. Upon application of an electric field, both G' and G" increased significantly, and as expected, more profound tendency for increasing appears for the emulsion with higher viscosity ratio (λ). This indicates that liquid column structures are more stable for droplets with higher viscosity. At higher viscosity ratio (λ), response of G' became a frequency-plateau, which was similar to that of particulate ER suspension. Especially for the emulsion with viscosity ratio λ=0.3 (less than unity), a negative slope of G' with ω appeared at high frequency. Such a response is due to the largely deformed structure of liquid columns at high frequency, which can be substantially regarded as a nonlinear viscoelasticity. Generally, the increase of the viscous component of stress by the electric field was less than that of the elastic response. In terms of G", the limiting frequency that an electric field effect diminishes was decreased with decreasing the viscosity ratio (λ). Even though we could expect a sinusoidal response of waveform for those emulsion samples in a linear viscoelastic condition and deviating waveform at large frequency, we were unable to achieve clear patterns due to the sensitivity limit of the instruments.

2. Viscoelasticity of Particulate ER Suspensions and Effects of Emulsion Drops

The response of ordinary ER suspensions can be classified in terms of three regions: pre-yield viscoelastic, viscoelastic and plastic at the yield, and post-yield plastic. Such deformation modes are dependent upon the strain amplitude, frequency, and electric field strength [Garmota et al., 1993]. The response of torque waveforms for polyaniline ER suspensions subjected to a sinusoidal strain is shown in Fig. 3, for the polyaniline ϕ_p=0.130 suspension in silicone oil. This set of results examines the variation of torque waveform as a function of strain amplitude while the strain frequency

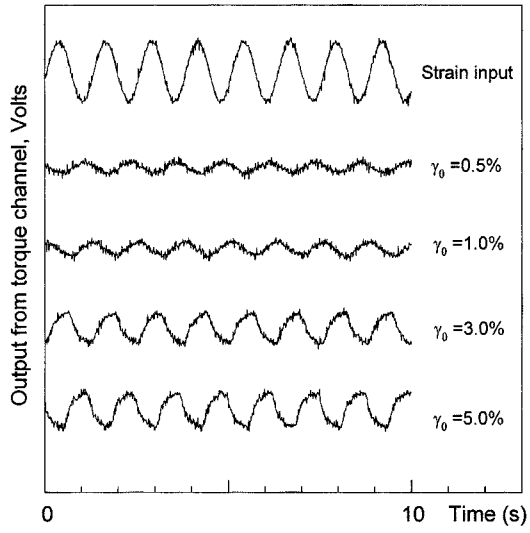

Fig. 3. Response of the torque waveform (viscoelastic region) subjected to sinusoidal strain input for the polyaniline in silicone oil suspension (ϕ_p=0.130) under E=2 kV/mm and low strain; effects of the amplitude of strain (γ_0).

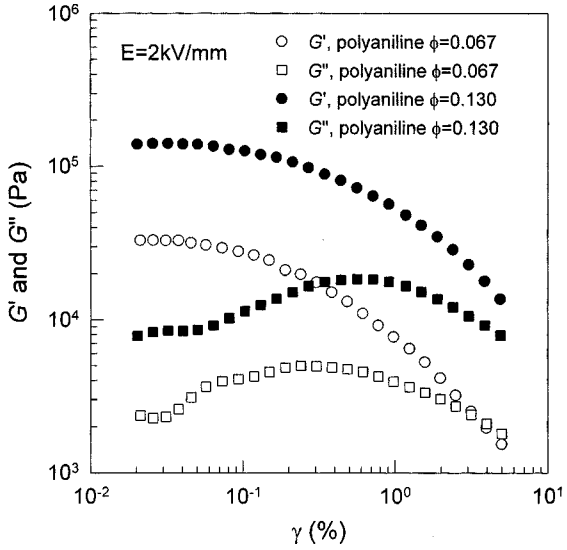

Fig. 4. Dependence of G' and G" on the strain amplitude for polyaniline in silicone oil suspension; volume fraction of polyaniline ϕ_p=0.067 and 0.130.

and electric field strength are held constant (ω=5 rad/s and E=2 kV/mm), although the degree of phase shift is not considered in this figure. At strain as low as 0.5-1.0%, the torque remains nearly sinusoidal. However, as the strain amplitude exceeds 1.0%, the torque clearly begins to deviate from being a sinusoidal response. When such a deviation is pronounced at larger strain, yielding of electric field-induced structures may appear, showing the viscoplastic nature. In that case, the ER material no longer behaves as a viscoelastic body. Therefore, analysis of the ER suspension in terms of the viscoelastic properties should be performed in the region of small amplitude range.

Fig. 4 illustrates the dependence of storage and loss shear moduli (G' and G") on the amplitude of strain until 5%, which can be approximated as the viscoelastic region before yielding. For the polyaniline suspension of ϕ_p=0.130 at 2 kV/mm, the linear viscoelastic region, showing the constant G' and G", was nearly below 0.1%. The limiting strain was even smaller in the case of a more dilute suspension (ϕ_p=0.067). Onset of nonlinearity is characterized as the monotonic decrease of G' and the initial increase and consequent decrease of G", as pointed out by Goodwin [1993].

Effect of the addition of emulsion drops on the dynamic behavior of ER particle suspension is shown in Fig. 5. The magnitude of the storage shear moduli was enhanced with increasing emulsion fraction Φ, indicating the dominating elastic property. In comparison, the magnitude of loss moduli was not sensitive to Φ. Instead, the maximum point of G" showed the shift to higher strain amplitude. When the both G' and G" curves began to decrease with strain amplitude, the breaking of the chain linkages occurred beyond their elastic limit and it may be considered as a yielding of structures, as indicated by Jordan et al. [1992]. Of course, macroscopic breakup of particle structures is not necessarily permanent and ruptured chains often have an ability to reform or join another chain to make a thicker column. The above status of "yielding point" can be considered as the combined region of viscoelasticity and viscoplasticity. Therefore, it may be said that the dynamic behavior of the ER

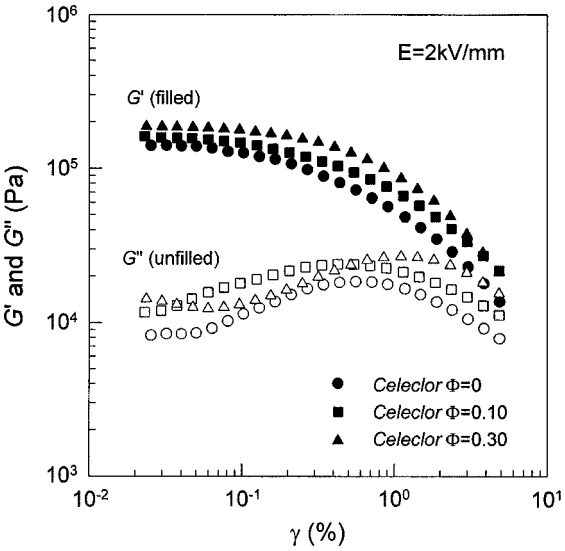

Fig. 5. Dependence of G' and G'' on the strain amplitude for polyaniline in silicone oil suspension (E=2 kV/mm); volume fraction of polyaniline ϕ_p=0.130 and different fraction of *Celeclor s45* emulsion drop Φ=0, 0.10, and 0.30.

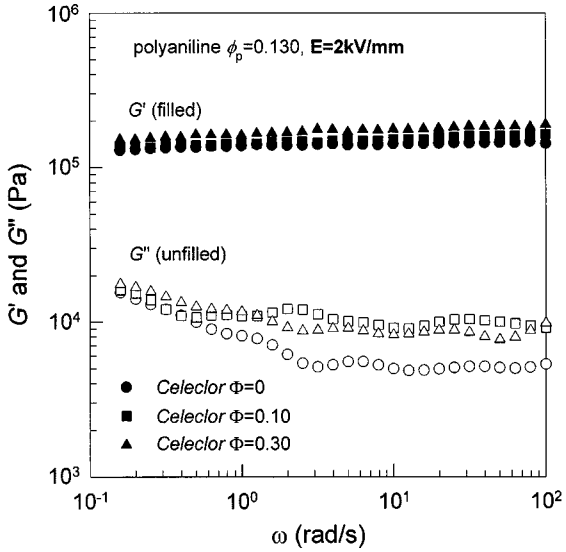

Fig. 7. Dependence of G' and G'' on the strain frequency for polyaniline in silicone oil suspension (E=2 kV/mm); volume fraction of polyaniline ϕ_p=0.130 and different fraction of *Celeclor s45* emulsion drop Φ=0, 0.10, and 0.30.

suspension is composed of narrow, linear viscoelastic region, relatively wide nonlinear region, and yielding region of viscoelasticity-plasticity. Additional liquid columnar structures by the inclusion of emulsion drops (*Celeclor s45*) were found to not only enhance the macroscopic yield stresses of particulate ER suspensions [Chin and Park, 2000] but also to widen the viscoelastic regime and delay the onset of yielding, as clearly seen in Fig. 5.

For the polyaniline/silicone oil suspension with ϕ_p=0.130 and *Celeclor* Φ=0.10, effects of the electric field strength are also shown in the strain amplitude - G', G'' curve of Fig. 6. Nearly similar features were obtained compared with the case of change in emulsion fraction Φ. Irrespective of the volume fraction of particle (ϕ_p) and emulsion drop (Φ), elasticity became dominant and the yielding of structures was delayed, especially at higher electric field strength.

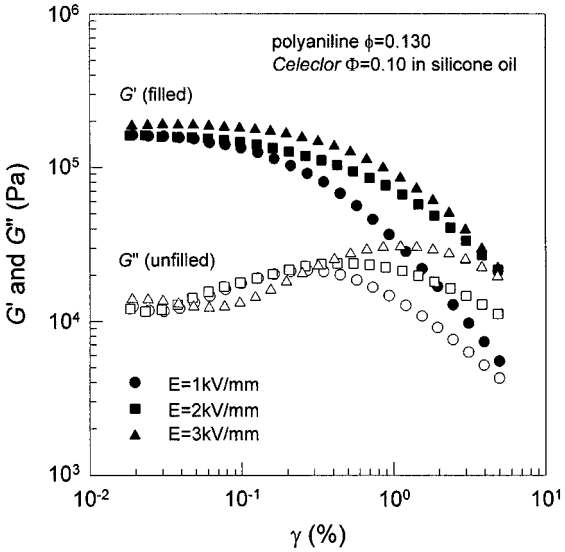

Fig. 6. Dependence of G' and G'' on the strain amplitude at different electric field strength for polyaniline in silicone oil suspension; volume fraction of polyaniline ϕ_p=0.130 and *Celeclor s45* emulsion drop Φ=0.10.

Fig. 7 shows the storage shear and loss modulus as a function of strain frequency for the polyaniline/silicone oil suspension (ϕ_p= 0.130) containing *Celeclor* drops. The electric field strength was set to be 2 kV/mm. The strain amplitude is 0.05%, which is sufficiently low to satisfy the linear viscoelstic condition, as seen in Fig. 4. Obviously, the viscoelastic response dominated in the storage shear modulus, representing a frequency-plateau throughout the range measured. The elastic solid behavior was shown in this small strain condition. Like the dynamic yield stress in post-yield state, the storage modulus was increased with the fraction of emulsion Φ, although the increment was not so profound, probably suggesting the existence of the limit in material strength [Pan and Mckinley, 1997a]. The loss modulus firstly increased with Φ=1.0, but at higher emulsion fraction, the magnitude was decreased again. Complicated behavior of G'' data versus frequency is probably due to the less dominant viscous property at the small strain condition.

3. Yielding and Viscoplasticity of ER Materials

The yielding region of ER fluid is commonly characterized as the material begins to flow or permanently deform, in which the shape of the response of the stress function deviates from that of strain. As the amplitude of strain further increases, the torque waveform takes a truncated shape and the maximum torque in the waves continuously increases with the strain amplitude and electric field strength. Fig. 8 illustrates the waveforms of torque at the yielding region for the polyaniline-based ER suspensions in the various electric field and emulsion volume fractions. The amplitude of torque waveform was gradually increased with the electric field strength, and at the same electric field, it was also increased with the fraction of emulsion drop Φ. Analysis of the data in the yielding region was difficult due to the complex rheological nature of ER materials. One possible explanation can be obtained from the torque-strain

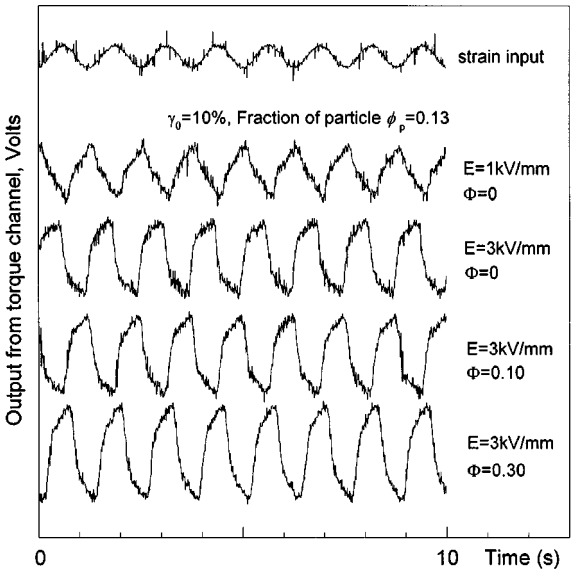

Fig. 8. Response of the torque waveform (yielding region) subjected to sinusoidal strain input for the polyaniline in silicone oil suspension (ϕ_p=0.130) at large strain amplitude for the different electric field strength and emulsion volume fraction Φ.

curves or hysteresis loop. When the hysteresis loops are recorded for the ER material during sinusoidal strain, it takes an elliptical form in the viscoelastic region [Garmota and Filisko, 1991] as seen in the curves in Fig. 9 (a) and (b) with strain amplitude of 1.0%. At higher electric field, the angle between the major axis of the loop and abscissa was increased [compare the curves (a) and (b)]. This indicates the increased elastic component associated with the deformation of ER materials. However, at large strain, the elliptical

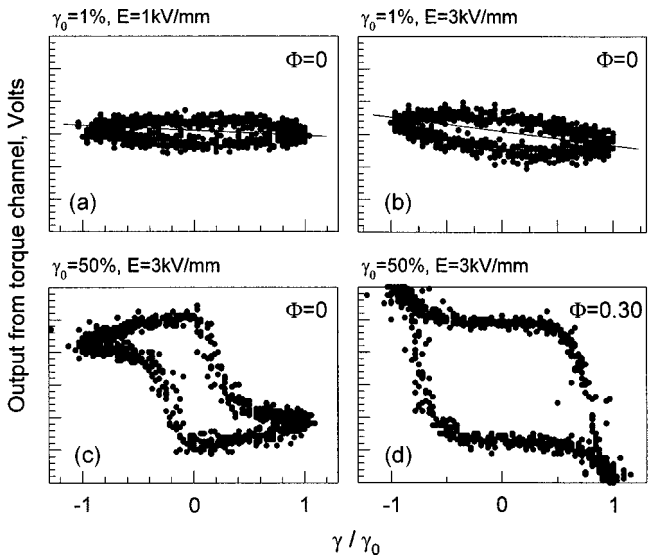

Fig. 9. Hysteresis loops of the torque-strain curves for polyaniline in silicone oil suspensions (ϕ_p=0.130); (a) strain amplitude γ_0=1% and E=1 kV/mm. (b) strain amplitude γ_0=1% and E=3 kV/mm. (c) strain amplitude γ_0=50% and E=3 kV/mm. (d) strain amplitude γ_0=50% and E=3 kV/mm containing *Celeclor s45* emulsion (Φ=0.30).

shape was destroyed and a transition from viscoelasticity to viscoplasticity occurred. The area within the hysteresis loop indicates viscous energy dissipation. Even in the viscoplastic region of large strain (5-50%), a continued increase in the electric field strength and the fraction of emulsion drops (Φ) resulted in an increase of this area, as seen in Fig. 9 (c) and (d). Such a significant deviation from elliptical shape suggests the introduction of some other damping mechanism, especially coulomb damping [Garmota and Filisko, 1991]. However, existing dynamic models cannot explain the complex rheological properties of ER materials in the yielding region. For further investigation of the dynamic behavior of ER fluids, development of an appropriate model that can explain the combined viscoelastic-plastic nature is required.

CONCLUSIONS

Dynamic rheological behavior of ER fluids containing emulsion drops, dispersed particles, and particle-drop bi-dispersed phases was investigated in oscillatory shear flow. Oil-in-oil emulsions with more conductive dispersed phase were considered as the model ER emulsions. Their electrorheological response dominated in the relatively significant increase of G' than G", and the more viscous dispersed phase facilitated the G' enhancement. Moreover, the response of G' was similar to that of particulate ER suspension for the emulsion of large viscosity ratio. The limit of strain amplitude for the linear viscoelasticity was as high as 10%.

For the particulate ER suspension containing semi-conductive polyaniline, torque waveforms subjected to sinusoidal strain input showed deviation even in the small strain of 1.0-5.0%, indicating the transition from viscoelastic to viscoplastic behavior. Within the region of viscoelasticity, the linear region was restricted below the amplitude of 0.1%. However, an increase of the electric field strength and the volume fraction of emulsion drops (Φ) resulted in a higher viscoelastic-limit strain and delayed the onset of viscoplastic yielding. The storage shear modulus in the linear viscoelastic region showed a frequency-plateau, which is the typical feature of an elastic solid.

Subjected to a strain with large amplitude, an ER material cannot be treated as a viscoelastic body. Dominant viscoplasticity and energy damping was characterized by the torque-strain hysteresis curves, which was shown to be a strong function of electric field strength and the volume fraction of ER-active emulsion drops.

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