ORIGINAL CONTRIBUTION

Experimental observation on the flow-induced assembly of Carbon nanotube suspensions to form helical bands

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Abstract This paper describes optical observations on the way aggregates of multi-walled carbon nanotubes (CNTs) in an epoxy matrix can form unusual helical bands (HBs) that are aligned perpendicular to the shear flow. By applying specific flow conditions, HBs were formed from isotropic aggregates of CNTs suspended in an essentially Newtonian epoxy matrix. Both optical and matching rheological data are presented together with a schematic model of the way HBs are formed. It was discovered that the steady shear rheology of the CNT suspension did not substantially change during the observed micro-structure change. The HB structure may have relevance in terms of potential applications for CNTs.

Keywords Fibre suspension · Shear-induced structure formation · Rheo-optics

Introduction

Carbon nanotubes (CNTs) are semi-rigid cylinders of rolled graphene sheets belonging to a relatively new class of fibrous material with length scales larger than polymer chains but smaller than classical glass or carbon fibres (Dresselhaus and Avouris 2001; Iijima 1991). Because of their intrinsic high mechanical strength, low density and electronic properties, CNTs are being extensively

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researched and evaluated (Baughman et al. 2002), for example as polymer fillers (Ajayan et al. 1994; Calvert 1999) and molecular-scale electronic devices (Saito 1997; Tans et al. 1998). Obtaining uniform dispersions of CNT within a processing matrix is important but also a difficult task. The way in which CNT can be dispersed has been discussed in a number of recent studies (Sandler et al. 1999; Shaffer et al. 1998; Zheng et al. 2003), and it has been shown that a mixing time of several hours may be necessary to establish a stable rheology for a CNT dispersion. Even then, it is not guaranteed that the dispersion will be uniform at the nano level (Huang et al. 2006). In this paper, we identify a mechanism by which certain CNTs can be processed to give a highly heterogeneous banded meso-structure.

Banded meso- and micro-structure have been reported before for both CNT and other suspensions (see review by Vermant and Solomon 2005). A recent publication (Hobbie and Fry 2006) reported a regime of flow where banding of multi-wall CNT suspensions was observed within poly-isobutylene solutions. They reported a phase diagram for different concentrations and shear rates, dividing flow regimes into isolated aggregates, vorticity banding and "cavitated networks". Other researchers have observed banding in different systems, namely thixotropic clay gel (Pignon et al. 1997), silica in polydimethylsiloxane (Degroot et al. 1994), silica in hexadecane (Varadan and Solomon 2001), attractive emulsions (Montesi et al. 2004) and thermotropic liquid crystalline polymers (Grazian and Mackley 1984a, b; Gervat et al. 1995). It is generally accepted that anisotropic organisation of suspending particles/droplets occurs in flow situations where interactions between particles are comparable to hydrodynamic forces, and that aggregate size is generally determined by balancing these forces (see review by Vermant and Solomon 2005). Various mechanisms have been proposed to explain shear

induced banding (see for example Hoekstra et al. 2003 and Montesi et al. 2004); however, at present, there is no universal consensus to describe all results.

Experiments

Materials and methods

Multi-walled CNTs were produced by the chemical vapour deposition method (Singh et al. 2003) and were supplied by the Macromolecular Materials Laboratory at the Department of Materials Science and Metallurgy, University of Cambridge. According to scanning electron microscope (SEM) images, CNTs used in this study typically have a diameter of less than 100 nm. The length of the nanotubes was not fully characterised, but the optical images suggested that it is about 30 μm. The epoxy matrix (UV 60-7155) was supplied by Epoxies, in the USA. It is essentially Newtonian and with a viscosity of 0.4 Pa.s. Ten milligrams of multi-walled CNTs with no prior chemical surface treatment were weighted into 30 g of epoxy to prepare a 0.03% suspension. The suspension was mixed using a homogeniser (Silverson L4R) for 5 h.

Microstructure characterisation

Optical micrographs of sheared CNT suspensions were obtained using a Cambridge Shear System (Bower et al. 1998; Mackley et al. 1999) manufactured by Linkam Scientific Instruments, and a schematic diagram of the apparatus is shown in Fig. 1. The CNT suspension was
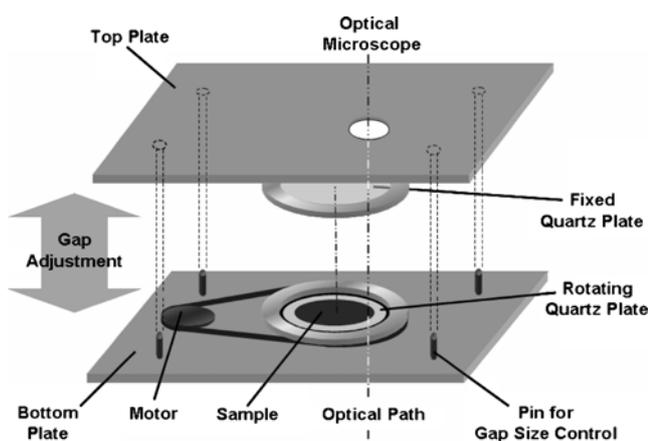

Fig. 1 Experimental configuration: the Cambridge Shear System (Linkam Scientific Instruments) consists of a shear cell and an optical microscope. The sample was confined between two quartz plates and the bottom plate was connected to a motor. The gap size was varied between 55–230 μm. Steady rotational shear was applied to the sample via the bottom plate. Microstructure formation in the sample was viewed using the optical microscope

confined between two parallel quartz discs with a controllable gap size tolerance of ±5 μm and a temperature of 25 °C. A stepper motor was used to control the rotation speed of the bottom disc and thus the shear rate. Optical images were viewed using an optical microscope and were captured by a JVC colour video camera (TK-C1480E) with a shuttle speed of 1/1,000 s. The photographs shown in the following figures were generally obtained immediately after shear to maximise clarity. It was established using video imaging that the optical textures seen during and immediately after shear were essentially the same.

Rheological measurement

Rheological properties of the CNT suspension were measured using an ARES strain-controlled rheometer with 50 mm parallel plates and a gap size of 130 μm. The temperature for all measurements was maintained at 25 °C. To obtain steady values of apparent viscosity, viscosity measurements were made 100 s after a particular shear rate was applied.

A parallel plate configuration for both rheological and optical measurements was used to help facilitate easier operation. The parallel geometry does however generate a shear gradient along the radial direction of the device. Optical measurements were made at a distance of 7.5 mm from the centre of 30 mm quartz discs, and all reported shear rates were referenced to this position. In terms of rheology, data have been corrected using the single-point correction method to account for flow non-homogeneity in the parallel plate rheometer (see for example Macosko 1994). To allow for direct comparison between optical observations and rheological measurements, shear rates for rheological measurements ($\dot{\gamma}_R$: shear rate at the edge of sample) were carefully chosen such that the true/average shear rate in the parallel-plate rheometer matched the local shear rate as specified for optical measurements (i.e. $\widetilde{\dot{\gamma}} \approx 0.76 \dot{\gamma}_R = \dot{\gamma}_{\text{optical}}$).

Results and discussion

The formation of HBs

Figure 2 shows the time evolution of CNT micro-structure for different gap sizes but all at the same shear rate condition of 0.5 s^{-1}. For all gap sizes, the optical image before the shear flow was applied (at $t=0$) showed an isotropic assembly of diffuse CNT aggregates (Fig. 2a,d,g and j), which is an observation consistent with optical textures that have been reported before (Hobbie et al. 2003a, b; Lin-Gibson et al. 2004; Rahatekar et al. 2006). When a low shear rate was applied, the fluid motion led to
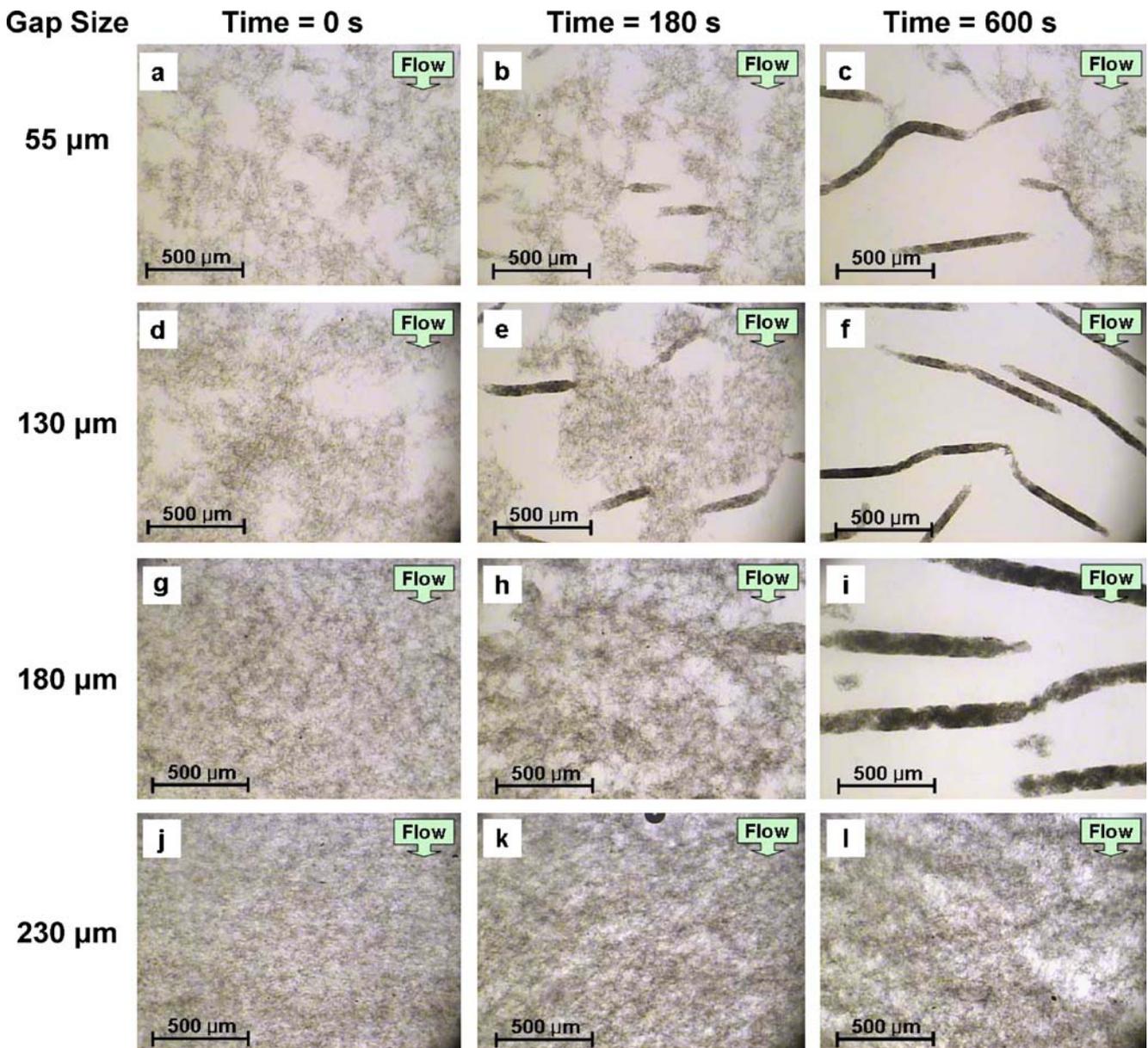
Fig. 2 The effect of time and gap size on the formation of HBs. The shear rate for all conditions was fixed at 0.5 s^{-1}. Direction of flow is vertical as indicated

particle transport and an increase in the chance of interparticle collision. The general phenomenon is known as orthokinetic aggregation (Smoluchowski 1917), which has been previously reported for colloidal suspensions (Warren 1981; Zollars and Ali 1986). In the case of CNT suspensions, collision occurred between CNTs, and diffuse aggregate structures were interlocked as shown in Fig. 2b. With the application of shear, as shown for example in the time evolution series Fig. 2a, b and c, there is a progressive evolution to form helical bands (HBs) aligned perpendicular to the direction of flow. Similar steady state optical textures have been previously reported for a CNT suspension (Lin-Gibson et al. 2004), and the observations

reported here support and extend those findings. The tumbling action of CNTs is similar to the rolling of tumbleweed in desert conditions, and the kinetics is best viewed in video mode (supplementary information movie). The movie clearly shows how HBs grow along the vorticity axis with the application of shear flow. Nucleus segments of rotating HBs are surrounded by diffuse CNT aggregates. As the base nucleus rotates in the simple shear flow, it consumes the nearby diffuse aggregates and grows in both mass and length. Although it is difficult to calculate the exact mass fraction of CNT within HB structures, it was observed that all CNT existed within the banding structures at the final stage of shear. Based on

Fig. 3 The effect of shear rate on HB formation. This figure shows the microstructure of CNT suspension sheared at a 0.5 s⁻¹, b 5 s⁻¹, c 10 s⁻¹ and d 50 s⁻¹. Temperature=25 °C, gap size=130 μm and shear time in excess of 600 s
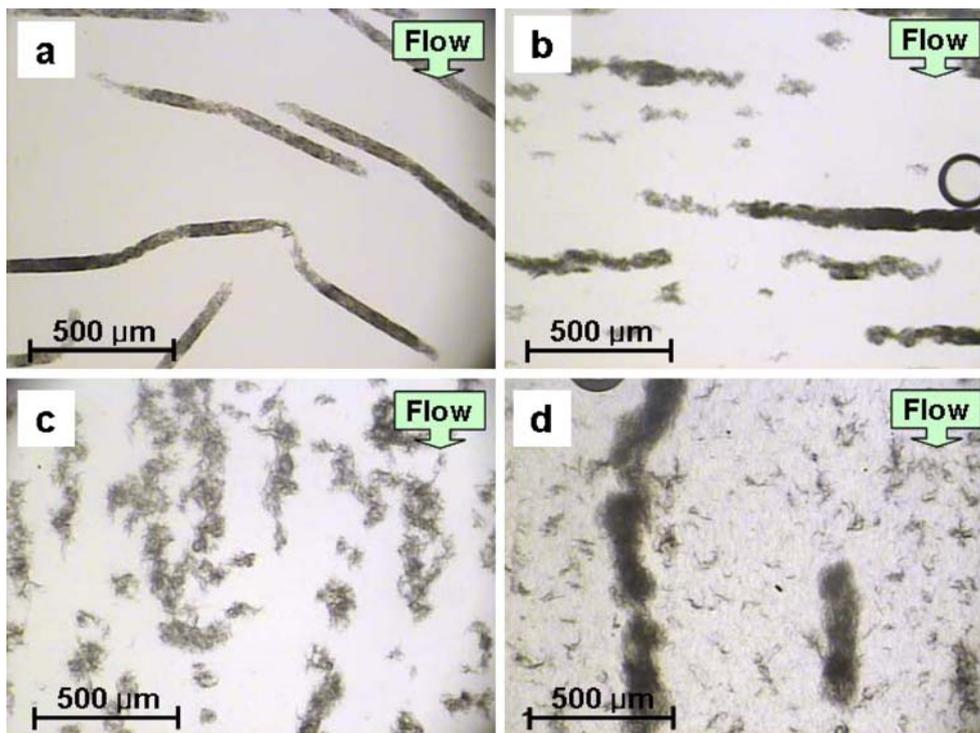
Fig. 2f, the volume fraction of HBs in the CNT suspension was calculated to be 14%. Given an initial mass fraction of 0.03% CNT and the density of epoxy to be 1.09 g/cm³, we therefore estimate the density of CNT within an HB to be 0.0023 g-CNT/cm³, which is equivalent to a mass fraction of about 0.21%.

It can be seen from Fig. 2 that the gap size at which the shear is applied influences both the formation kinetics and the final diameter of the HBs. At a shear rate of 0.5 s⁻¹ and a gap size of 130 μm (Fig. 2d,e and f), all the diffuse aggregates were consumed within 600 s of shear and formed millimeter-long HBs with a fairly uniform diameter

Fig. 4 Growth and connection mechanism of HBs. This series of still frames from the supplementary information movie shows the way in which two HBs were woven together. Gap size=130 μm; shear rate= 0.5 s⁻¹ and shear time in excess of 210 s
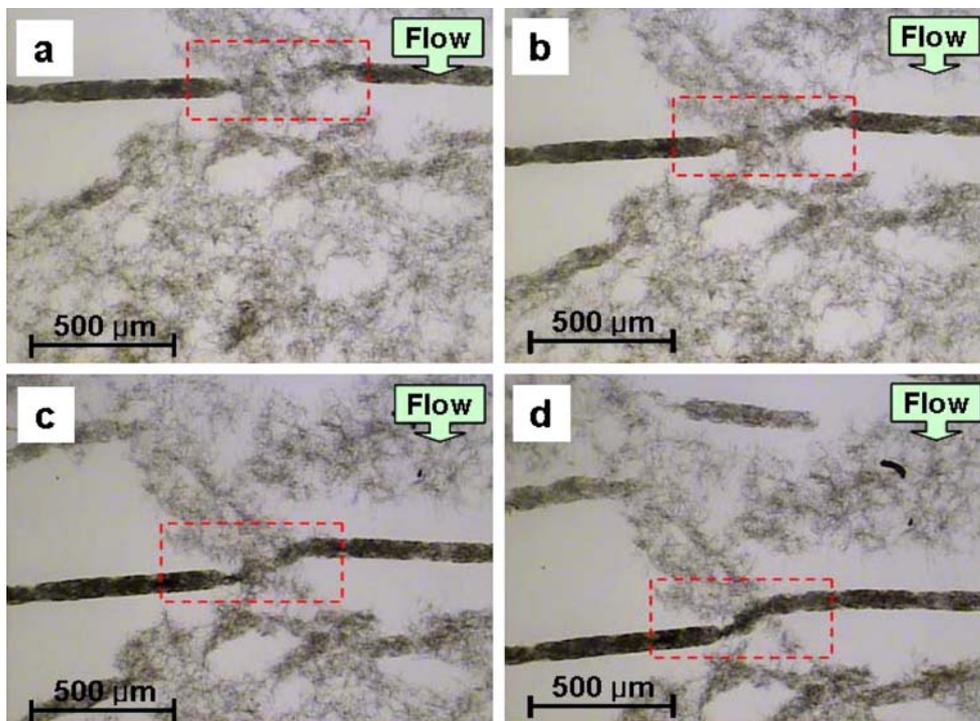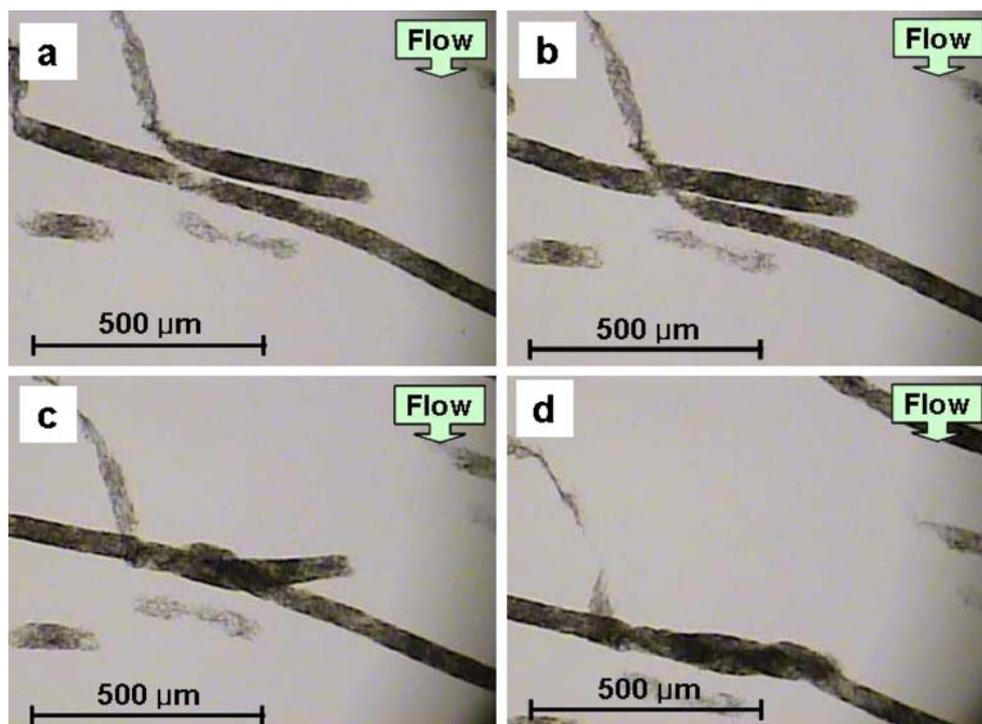
Fig. 5 Formation of a double helix structure of carbon nanotubes. When two HBs came into contact, they wrapped around each other helically and subsequently formed a double helix structure. Gap size=130 μm; shear rate=0.5 s^{-1}, and shear time in excess of 400 s

of 60 μm. As the gap size was reduced to 55 μm, the rotation speed of HBs decreased, and it was observed that a longer time was required for the complete consumption of diffuse aggregates (Fig. 2c). When a larger gap size of 180 μm was used, the diameter of HBs doubled (120 μm). With a further increase in gap size to 230 μm, optical resolution became more difficult due to an increasing depth of field; however, no clear discrete HB structures were formed within a shear time of 600 s, and there is also no optical evidence for sedimentation. Based on all these observations, it is concluded that the formation of HBs is a result of mechanical aggregation where the gap size plays an important role in constraining the aggregate to form such an anisotropic structure. Such observation is in general agreement with other studies (Lin-Gibson et al. 2004; Vermant and Solomon 2005; Hobbie and Fry 2006), and our experimental observations on the kinetics of formation (Fig. 2) tend to suggest that the presence of HBs originates dominantly from a mechanical and geometrical mechanism rather than being of rheological origin as proposed by others (Lin-Gibson et al. 2004; Montesi et al. 2004).

The effect of the magnitude of shear rate was studied with a fixed gap size of 130 μm (Fig. 3) and a shear time of 600 s. The figure shows that textures are very sensitive to shear and that the HB structures are only achieved in a specific shear rate regime. At high shear rates, the HB structure that had formed perpendicular to the shear direction broke up and formed a less clearly defined optical texture that was aligned predominantly in the direction of flow. The figures indicate that there is a shear rate pro-

cessing window for HB formation, and a perpendicular alignment of the HBs can be obtained up to approximately a shear rate of 5 s^{-1}.

Figure 4 shows in greater detail the mechanism by which a long HB can be formed. In Fig. 4a, two HBs were initially separated by diffuse aggregates. As they rotated, diffuse aggregates were drawn to the HBs, and the individual HBs were subsequently woven together to form a longer HB (Fig. 4d). In Fig. 5, a double helix formation mechanism is
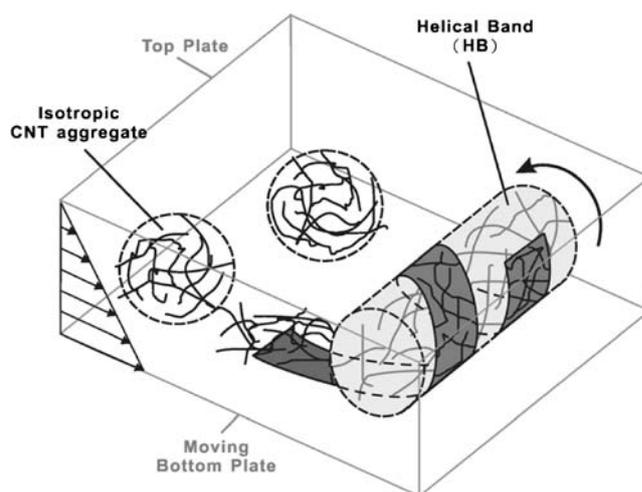

Fig. 6 Schematic diagram of HB growth. An HB nucleus rotates within the steady shear and captures initially isotropic aggregates of nanotubes. The nanotubes are then wound helically to form a HB with an axis perpendicular to the direction of flow
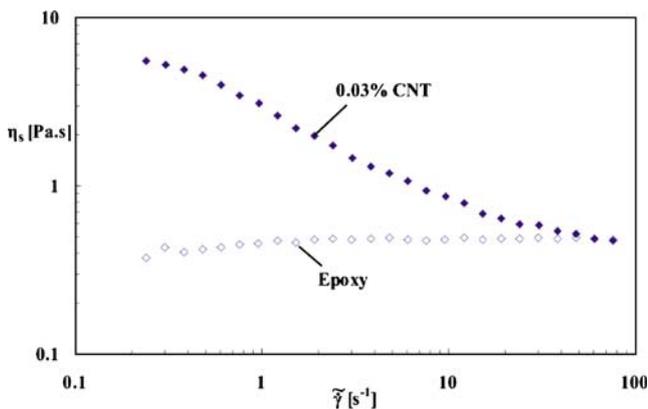
Fig. 7 Shear viscosity (η_s) of the epoxy matrix and 0.03% CNT suspension as a function of the shear rate ($\tilde{\gamma}$) applied (temperature= 25 °C and gap size=130 μm)

shown where two HBs come into direct contact at an angle (Fig. 5a); they wrap around each other and subsequently form a "double helix" structure (Fig. 5d). The process essentially mimics the spinning of two threads into a plied yarn (Morton and Wray 1962). The optical textures and kinetics described in this paper were for multi-walled CNTs suspended in an organic matrix, and we have observed similar effects for acid-treated nanotubes suspended in an aqueous matrix. We did not see the perpendicular alignment effect using an epoxy resin with a higher matrix viscosity of 10 Pa.s for a shear time of 600 s, although the effect may occur for much longer shearing times (see for example Lin-Gibson et al. 2004).

From the experimental observations made in this paper, it is possible to identify conditions for the formation of HBs. If the shear stress within the suspension is too high, CNT aggregates can break up and subsequently align in the

flow direction as described for example by Hobbie et al. (2003a) and Rahatekar et al. (2006). By reducing the viscosity of the matrix and ensuring the shear rate is below a critical threshold, HBs can form. The time for the formation of HB depends on both the shear rate and the gap size. Small gap sizes and high shear below the critical threshold resulted in HB formation, whilst large gap size and low shear resulted in no HB formation within the timescale studied. Although the effect of initial volume fraction of CNT has not been explored in this paper, Hobbie and Fry (2006) reported in a recent independent study that HBs were only formed above a volume fraction of 0.025%.

From the type of observations shown in Figs. 2, 3, 4 and 5, it is possible to develop a model of the way in which the HBs are formed. Figure 6 shows a schematic diagram that illustrates the main features. In a simple shear flow, because there is a velocity gradient, there is a relative motion between aggregates at different vertical positions within the gap, resulting in the faster moving aggregates interlocking with the slow moving aggregates and subsequently the formation a single aggregate, or an HB nucleus. As the HB nucleus rotate in the simple shear flow, individual isotropic aggregates of nanotubes are swept up in a similar way to the hand-spinning of fluffy cotton into short fibres without using a spindle (Morton and Wray 1962). Despite the fact that individual CNTs are essentially rigid, the way in which isotropic aggregates wrapped around each other has similarity to a mechanism proposed for the formation of roller-like structure in attractive emulsions (Montesi et al. 2004). Various authors have also put forward arguments to explain vorticity banding for different systems, which has been extensively reviewed by Vermant and Solomon (2005). Based on 2D video microscopy observation of polypropyl-

Fig. 8 Time evolution of flow stress (τ) and microstructure during a steady shear at $\tilde{\gamma} = 0.5\,\text{s}^{-1}$, gap size=130 μm, and temperature=25 °C. (Note: rheology data at short times have been omitted since the instrument took approximately 1 s to reach the commanded shear rate.)
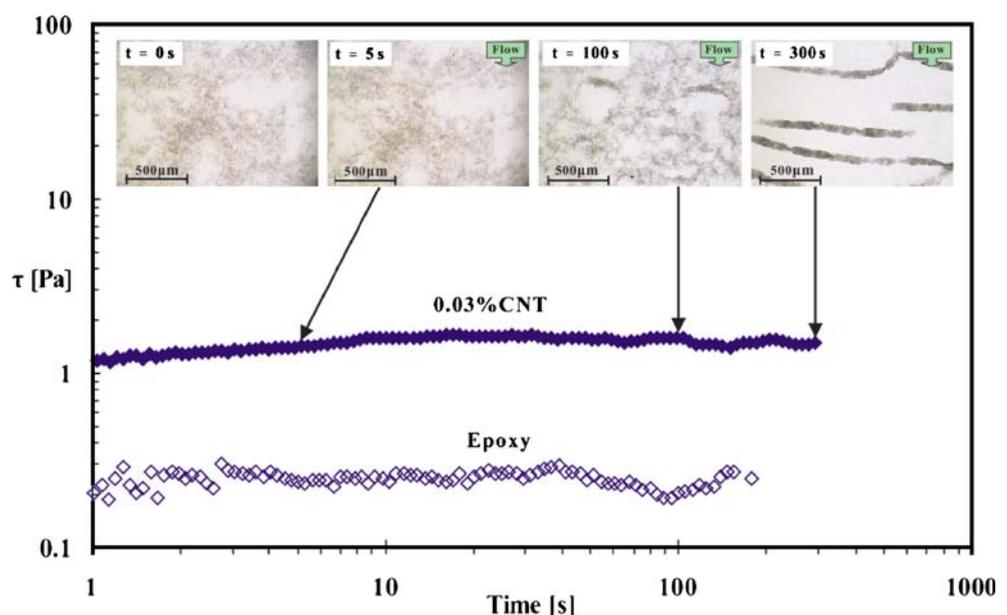
Fig. 9 Time evolution of flow stress (τ) for 0.03% CNT in epoxy using different gaps sizes (130, 180 and 230 μm; $\widetilde{\gamma} = 0.5\,\mathrm{s}^{-1}$ and temperature=25 °C). Note: rheology data at short times have been omitted since the instrument took approximately 1 s to reach the commanded shear rate
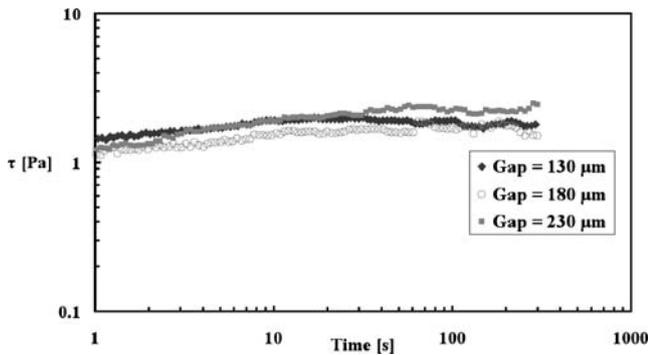
ene particles suspended in aqueous mediums, Hoekstra et al. (2003), for example, has proposed a purely hydrodynamic mechanism for the formation of anisotropic microstructures in free-surface flows. In terms of CNT suspensions, Lin-Gibson et al. (2004) proposed that roller-like organisation of CNTs is a consequence of elastic instability and that the observed anisotropic structure might have an implication on the measured negative first normal stress difference, as in the case of some emulsions. For the conditions described in this paper, it is clear that the aggregate grows as HBs in the direction of vorticity rather than increasing the aggregate size as a spherical entity. Although walls clearly provided geometric constraints that small gaps would prevent CNT

aggregates from growing in the velocity gradient direction, a detailed reason for the HB formation is not yet fully understood at this stage.

The rheology of HBs

Figure 7 compares the rheology of the CNT suspension and the suspending matrix for steady shear. The data show a low-shear-rate viscosity enhancement and progressive shear thinning where the CNT suspension asymptotes to the matrix viscosity at high shear rates. The form of data is similar to that reported for other CNT suspensions (see for example Rahatekar et al. 2006). The aspect ratio (λ) of CNTs after mixing with the epoxy was estimated to be about 300 and a suspension with 0.03% (w/w) CNT (or 0.025% by volume) is, according to Petrie (1999), in the semi-dilute regime ($\lambda^{-2} < \Phi < \lambda^{-1}$). It would therefore be inappropriate to use dilute suspension modelling (see for example Batchelor 1977) to predict any viscosity enhancement. Experimentally, the observed viscosity enhancement as shown in Fig. 7 is about an order of magnitude higher than that would otherwise be predicted by assuming a dilute and uniformly dispersed suspension of rods (see for example Burgers 1938). The time evolution of flow stress during observed micro-structure change is shown in Fig. 8. The CNT suspension was loaded into the rheometer, and when a shear rate of 0.5 s^{-1} was applied, a transient stress start-up was observed. The stress reached a steady state within 1 s, which remained essentially constant for the experiment duration of 300 s. During this period, there was a progressive change in the micro-structure, from diffuse

Fig. 10 Evolution of shear viscosity (η_s) and microstructure of 0.03% CNT in epoxy for different steady shear rates ($\widetilde{\gamma}$) at gap size=130 μm and temperature of 25 °C
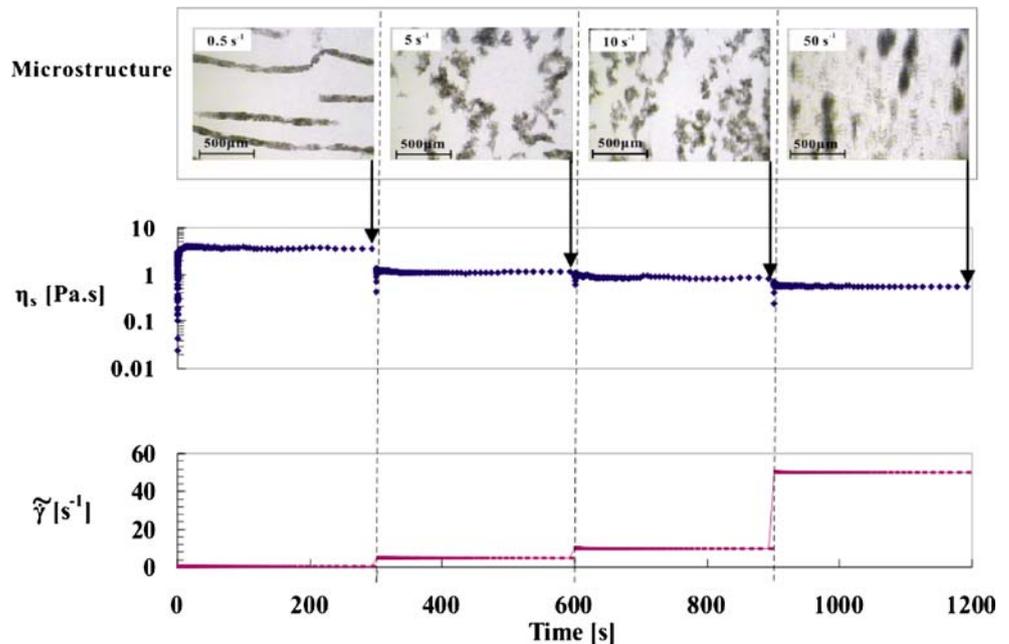
isotropic aggregates to HBs aligned perpendicular to the flow. Surprisingly, this micro-structure change did not affect the flow stress measured, and it would appear that for constant applied strain rate conditions, significant micro-structure changes can occur without changing the energy dissipation of the system. Further experiments were carried out for different gap sizes, and the flow stress was found to be the same as shown in Fig. 9. Corresponding time evolution of micro-structure for different gap sizes is given in Fig. 2.

Figure 10 shows the rheo-optical data for different steady flow conditions. As the shear rate increased, the HBs broke up to form essentially isotropic aggregates and then aggregate structures that were aligned in the direction of flow. There was an associated shear thinning of the suspension with increasing shear rate. In many current rheology/microstructure papers, rheology has been directly linked to observed micro-structure changes (see for example Varadan and Solomon 2001). In the particular case reported in this paper, a clear change in micro-structure was observed during a period where there is no rheology change. We therefore concluded that the optically observed micro-structure does not necessarily change rheology. In the case of the data presented in Fig. 10, there was a rheology change and there was also an optical micro-structure change. It is possible that the two are linked; however, we are inclined to believe that any change in rheology is linked to events below the resolution of the optical microscope (e.g. local anisotropy within the HB structure) and that the larger optically resolvable micro-structure plays a relatively weak role. It is possible that local anisotropy of the CNT suspension remained un-changed given the relatively low shear applied. This conclusion is also supported by our recent modelling work on CNT suspensions, which will be described in a future paper. As in the case of short fibre suspensions (see for example Hinch and Leal 1975, 1976; Petrie 1999), a constitutive relationship can be established to correlate stress contribution, and CNT orientations (local anisotropy) and viscosity enhancement effect of aggregating CNT suspensions have subsequently been modelled by simulat-ing the orientation of CNTs in different entanglement states. Finally, exploring local anisotropy of the HB structure is difficult, but there is a scope to UV irradiate samples that have been sheared under different conditions and then carry out nano-structure analysis using SEM or atomic force microscopy, and this is an objective for future work.

Conclusions

In conclusion, we have reported the way in which HBs can be formed in suspension using steady simple shear with a controlled gap size and a low shear rate. The way in which

the CNT aggregates are helically wound to form extended HBs is striking, and the twisting of two HBs into a double helix structure is intriguing and not fully understood. From the viewpoint of applications, the advantage of having an HB structure of CNTs has also not yet been explored; however, the anisotropic nature of the meso-structure will produce preferential conductivity along the length of the HBs and thereby provide unusual anisotropic electrical properties to the system as a whole. At present, enhancing the bulk electrical conductivity of a polymer film by the addition of CNTs is a promising potential application for CNTs (Sandler et al. 1999; Shaffer et al. 1998). For such an application, it is thought desirable to utilise CNTs as a homogeneous dispersed system, in which case, the type of meso-structures described in this paper should be avoided; however, the anisotropic meso-structures described here could potentially be used for strategic benefit. In terms of rheology, no substantial change in the flow stress was recorded during the formation of HBs, implying that optically observable micro-structure does not necessarily control the rheology. Finally, it is conjectured that the overall rheology is mainly controlled by the sub-micron CNT structures that are not optically resolvable.

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