An apparatus to experimentally model the dynamics of ductile shear zones

Paul D. Bons a, *, Janos L. Urai b

a Victorian Institute of Earth and Planetary Sciences, Department of Earth Sciences, Monash University, Clayton, Vic. 3168, Australia
b Shell Research B.V., PO Box 60, 2280 AB Rijswijk, Netherlands

Received 7 December 1994; accepted 14 August 1995

Abstract

We present a ring-shear apparatus designed to deform soft, ductile rock analogues to arbitrarily high shear strains in simple shear, in experiments attempting to model processes that occur in natural ductile shear zones. The sintered specimen is deformed between two unsupported cylinders by two rotating grips, under a normal and shear stress up to 5 MPa and at temperatures up to 600 K. First results of experiments with octachloropropane (OCP, C₈Cl₈) and camphor (C₁₀H₁₆O) are presented.

Experiments with pure OCP samples show that a steady-state strain rate is achieved at a shear strain of around 4, when a steady-state grain shape and crystallographic preferred orientation is established. Mechanical properties compare well with those of cylindrical samples deformed in triaxial compression. Shear localisation is relatively minor. Samples of pure camphor, on the other hand, show very strong shear localisation behaviour, and mechanical properties which do not compare well with those of cylindrical samples deformed in triaxial compression.

Experiments with two-phase samples (OCP containing stronger camphor inclusions) show an initial softening of the composite, followed by hardening. This is associated with the development of a foliation, defined by camphor inclusions that stretched to lenses and layers, and the subsequent folding of this foliation at shear strains above 50. A steady-state strain rate is not reached, even at strains of 100.

The experiments produce results closely resembling the kinematics observed in natural shear zones, and give a first look into the complex changes in mechanical properties which occur at very high shear strains. They show how this technique, in conjunction with transparent deformation cells, can be used to study both microstructural and rheological development during long simple shear deformation histories.

1. Introduction

Our knowledge of the mechanical properties of rocks and the accompanying development of microstructures has increased greatly over the past decades, partly based on high-temperature rock deformation experiments, which have established reasonable bounds on in-situ flow laws for monophase rocks (Carter and Tsenn, 1987; Kirby and Kronenberg, 1987). These have been used in modelling studies (for instance, England and Houseman, 1986), often assuming a constitutive law where steady-state flow is established at shear strains around 0.1.

* Corresponding author
Ductile shear zones, characterised by strongly localised non-coaxial deformation to very high shear strains, are widespread in the crust and upper mantle (Carter, 1976; Carter et al., 1990; Twiss and Moore, 1992; Handy, 1994), and play a major role in determining large-scale mechanical and transport properties. Quantifying the rheology of polyphase rocks under these conditions, together with the microstructures which develop in shear zones, forms an important basis for a wide range of crustal deformation studies. However, experimental data on the mechanical properties of polyphase rocks at shear strains relevant to natural shear zones are relatively scarce (Paterson and Weiss, 1962; Dell’Angelo and Tullis, 1982; Jordan, 1987; Ross et al., 1987; Schmid et al., 1987; Franssen and Spiers, 1990; Tullis, 1990).

The aim of this paper is to contribute to this understanding by means of model experiments which cannot yet be performed using real rocks under the relevant conditions.

Two types of model materials are used to simulate natural deformation, in (partially) scaled experiments (Hubbert, 1937; Weijermars and Schmeling, 1986; Means, 1989). The first are materials with bulk rheological properties similar to their natural prototypes, but with fundamentally different microscale deformation mechanisms (such as putties and polymers, Weijermars, 1986). These have been used to simulate tectonic processes at different scales (Hudleston, 1973; Van den Driessche, 1986; Talbot and Jackson, 1989; Mandal and Khan, 1991). Of the second type of materials both bulk properties and deformation mechanisms (cataclastic processes, dislocation creep, grain boundary sliding, dynamic recrystallisation) are analogous, such as sand, clay, ice, sodium nitrate, camphor (Kamb, 1972; Ashby and Brown, 1981; Tangatt and Humphreys, 1984; Wilson, 1986; Means, 1989; Richard and Krantz, 1991). These are soft and ductile at low pressure and temperature, and allow a number of experiments which cannot be performed on real rocks at the corresponding (scaled) temperature and pressure. For example, the specimen can be deformed in transparent deformation cells that allow the observation of microstructural evolution by optical microscopy during deformation. Until recently, the second group has not been widely used to study the bulk rheological properties of polyphase materials (Ross et al., 1987; Bons and Urai, 1994; Tóth et al., 1994; Tullis and Wenk, 1994).

In this paper we use the second group of materials in an apparatus that enables us to use the advantages of crystalline rock analogues. We study the rheology and microstructure of two-phase materials deformed to very high shear strain, in samples large enough to study processes such as foliation development and folding. Identical samples can also be deformed in a transparent ring-shear apparatus (Jessel and Lister, 1991; Bons, 1993) under similar conditions, thus allowing a detailed study of microstructural development on the grain scale.

In this paper we first describe the apparatus in detail and discuss the interpretation of the measurements and some of its critical functions; then we present results of a first series of experiments; and finally we discuss our results and point to possible future improvements of the technique.

2. The apparatus

2.1. Shear configuration

Torsional deformation of cylindrical samples in a range of geometries and loading arrangements has been used in rock and soil deformation in the past (Griggs et al., 1960; Handin et al., 1960; Saada and Townsend, 1981), by Kamb (1972) and Bouchez and Duval (1982) for deformation of ice, by Tullis and Weeks (1986) for the study of frictional sliding and by Jessell and Lister (1991) for deformation of organic rock analogues in a transparent deformation cell. The main advantage of this type of deformation is that very high shear strains can be achieved without changing the shape of the sample. Complicating factors are the difficulty to design sample grips and lateral support.

We have chosen the hollow-cylinder configuration with inclined grips (inspired by the design of cone- and plate-viscometers) to minimise variation in stress state in the radial direction (Fig. 1). Deformation is imposed by rotating one grip with respect to the other, and simultaneously applying a normal stress to suppress dilatancy and microcracking. The sample is constrained by two unsupported, rigid cylinders with a low-friction interface to the sample.
The apparatus can contain samples of approximately 5 mm in cross-section, and apply normal and shear stresses of a few MPa at temperatures up to 600 K. The prototype which is presented here was designed to be a relatively low-cost rig, aimed to explore overall performance.

Since localisation of shear is common in natural shear zones (Berthé et al., 1979a, b; Lister and Williams, 1983; Law et al., 1984; Lister and Snoke, 1984; Hobbs et al., 1990) we chose a design which allows but does not force strain localisation. The configuration maintains a reasonably homogeneous stress state in a homogeneous sample, and allows measurement of the bulk shear strain (rate) by monitoring the rotation of the grips. This design is different from those where the outside of the specimen is forced to deform by bulk homogeneous simple shear and shear localisation is suppressed (Price and Torok, 1989), or from the common soil mechanics designs where the supporting cylinder is split at mid-height and localisation is imposed on the sample by the boundary conditions.

2.2. Description of the apparatus

A schematic drawing of the apparatus is given in Fig. 2. In the following description the parts are labelled as in the figure. The doughnut-shaped specimen (a) is held between two cylindrical steel grips (b and c), inclined as shown in Fig. 1, and with sawtooth-shaped radial grooves to hold the specimen. The lower grip is fixed to the upper base plate (i), while the upper grip is rotated to deform the specimen. Two polished confining cylinders (g and h) constrain the sample laterally; these are not fixed to the apparatus and can rotate or move in axial direction. In all experiments, the angular velocity of the outer cylinder was between 0.3 and 0.7 times that of the upper grip. The clearance between the confining cylinders and the grips is less than 0.02 mm. At high normal stress some of the specimen material can extrude through this narrow slot, but this was found to be negligible in our experiments.

2.3. Applied shear stress

The upper grip is fixed to a stiff central shaft (d) which extends downwards to below the lower base plate (j) and is vertically unconstrained. Its lateral movement is constrained by two ball-bearings to within 0.02 mm at the two base plates. This shaft is made of two parts that can be disconnected to allow loading the sample into the apparatus. A torque is applied by a dead weight (n) attached to a steel wire which is wound around one of two driving wheels (k) on the shaft just below the upper base plate. The shear stress applied on the specimen is determined by the mass of the dead weight and the radii of the driving wheel and the grips. The accuracy of the shear stress values is primarily determined by the friction at the sides of the sample and of the bearings in the system, since all other relevant parameters are precisely known ($\pm 0.001\%$). Friction at the sides of the sample is the major (as yet not fully quantified) source of uncertainty in the shear stress in the sample. A preliminary analysis of this is given in Appendix A, where it is argued that these effects were minor ($\pm$ several %). Shear stress values presented in this paper were not corrected for friction.

2.4. Measurement of bulk shear strain (rate)

The wire is led along a wheel of a potentiometer, fed by a stabilised DC source (l). Movement of the wire causes a rotation of the potentiometer’s axis, producing an output voltage which is linearly dependent on the angular orientation. This was used to measure displacement of the top grip with respect to the bottom grip, and calculate the bulk shear strain, as follows.

The output voltage was recorded by a personal computer and a chart recorder. The bulk shear strain and bulk shear strain rate in the specimen can be derived from the output voltage, the radii of the potentiometer wheel, the driving wheel and the height.
of the sample at a reference radius (see Appendix A). This set-up was chosen as a cost-effective first solution, instead of a more accurate shaft-encoder. Possible sources of error are tightening and slip of the wire at the potentiometer wheel and around the driving wheels, and the noise (\pm 0.15\%) in the potentiometer output. The uncertainty in bulk shear strain measurements was estimated to be \pm 0.0001 to \pm 0.01, depending on the choice of the wheels involved. The error in the derived strain rate can however become significant. Strain rates presented in this paper were therefore determined with a moving-window least-squares linear fit through the strain-time data (Bons, 1993), using a relatively wide window, which filters out possible high-frequency variations in strain rate. The widths of the windows (w) used are given in the caption of the graphs. The strain rates shown in the figures are believed to be accurate to within 10–20%.

2.5. Confining pressure

A confining pressure is applied by a normal force on the grips. This is needed to achieve a firm grip of the corrugated grips on the specimen and to prevent dilatancy and microcracking. The normal force is applied by pulling the central rotating shaft, attached to the upper grip, downwards with a spring (o). A plate with ball-bearings is fixed to the shaft at its lower end, below the lower base plate. The non-rotating spring rests on the plate with ball-bearings. The (calibrated) spring can be shortened with a ring, attached to the lower base plate, that can be screwed downwards. This puts the central shaft into tension, and allows the confining pressure to be controlled by adjusting the length of the spring. All experiments were done at a confining pressure of 1.9 \pm 0.1 MPa.

2.6. Temperature control

Two 75-W heater elements (f) are located in a brass block (g) below the lower grip. A K-type thermocouple (e) is located between the heater elements and the specimen. The brass block is separated from the base plate by an insulating ceramic material. An insulating cover (p) can be placed over the upper part of the apparatus. Calibration with a thermocouple inserted into a dummy sample through the upper piston showed a temperature variation of 0.2\°C from top to bottom at a set temperature of 29\°C, which was the temperature at which all experiments were done.

3. Sample preparation and observation

3.1. Starting materials

Two crystalline organic compounds were used for this study: octachloropropene (OCP) and camphor. Uniaxial compression experiments have shown that both compounds observe a power-law relation between stress and strain rate. At room temperature the stress exponents are 4.5 \pm 0.3 for OCP and 3.3 \pm 0.3 for camphor (Bons and Urai, 1994). Camphor is stronger than OCP (i.e. it deforms at a lower strain rate) at room temperature and differential stresses of 0.1–2.0 MPa.

OCP (C_{10}Cl_{8}) is hexagonal and ductile at room temperature and pressure, the melting temperature is 160\°C. Its deformation is characterised by crystal plasticity, the formation of subgrains, grain boundary migration and the development of a crystallographic preferred orientation (Means, 1983; Jessell, 1986b; Means and Ree, 1988; Ree, 1991). Kink bands or twins at high strain rates and low temperature, and diffusional creep processes at elevated temperatures have also been reported (Jessell, 1986a; Ree, 1994).

Camphor (C_{10}H_{16}O) is rhombohedral, with a phase transition to cubic at 92\°C and a melting temperature of 179\°C. Deformation is characterised by the formation of kink bands, dislocation creep, dynamic recrystallisation, the development of a crystallographic preferred orientation and the tendency to
localise strain in recrystallised areas (Urai et al., 1980; Urai and Humphreys, 1981).

Camphor (a mixture of d- and l-isomers) was obtained from MERCK (Germany). OCP was a donation by Shell Research Laboratories in Amsterdam, and purified to a melting interval of 160 ± 2°C. Further details are given by Bons (1993). The materials used are different from the OCP used by Means and Ree (1988) and Ree (1990, 1991, 1994), which has a wider melting interval, and the camphor used by Urai and Humphreys (1981) and Urai et al. (1980).

3.2. Sintering of the samples

A specimen is made by first placing the two confining cylinders in position and placing the granular sample material evenly on the lower grip. The upper grip, and the upper half of the shaft is then placed in position and attached to the lower half of the shaft. The spring is tightened to pull down the upper grip and compress the specimen. Confining pressure is kept at approximately 2 MPa by periodically tightening the spring. The temperature was kept at about 66°C in most cases to speed up the compaction. Depending on the material used it then takes about 1 to 4 days to achieve a solid, translucent, fully compacted sample and no measurable further compaction.

3.3. Observation and photography of the sample

The surface of the specimen can be observed when the outer confining cylinder is removed. This is done at the end of an experiment, or during an experiment to study the development of microstructures. OCP and camphor are hard to distinguish on a smooth surface. The OCP for experiments with mixtures of OCP and camphor (RSS and 8) was therefore mixed with a small amount of very fine graphite powder. This proved not to provide the desired contrast between the two materials and the sample surface was etched slightly with methylated spirit to make the camphor stand out as white on a black OCP background. Only very slight etching and short (≤ 15 min) exposure to air was used during experiments, to avoid significant disturbance of the sample. For a clear distinction between the phases, several hours of exposure to air is necessary, which is only permissible at the end of an experiment.

The following procedure was followed to produce a complete image of the outer surface of the specimen. About 60-degree sections of the sample were photographed, to produce about 100 size black and white negatives. 13 × 18 cm prints of these were made and digitised to 32 bpi 256 grey-scale PICT-files for a Macintosh computer. These images were then distorted numerically, to correct for the photography of a cylindrical surface. The corrected images were then merged manually on the computer display to create one image.

4. Experiments with monophase samples

Four tests are presented of deformation of pure OCP or pure camphor. The tests were aimed at: (1) comparing simple shear rheological data with those from uni-axial compression experiments; (2) exploring mechanical properties at very high shear strain; and (3) studying the distribution of strain in the sample. An overview of relevant data is given in Table 1.

4.1. Pure OCP

Three experiments were done with pure OCP at an applied shear stress of 0.23 MPa (RS1-3), and one at 0.28 MPa (RS6). Resulting strain versus time and strain rate versus strain graphs are shown in Fig. 3. Earlier triaxial compression experiments showed that OCP observes a power-law relation between stress \( \sigma_{ij} \), and strain rate \( \dot{\epsilon}_{ij} \), which can be expressed as:

\[
\dot{\epsilon}_{ij} = BT_i^{\frac{1}{n-1}} \tau_{ij} \tag{1}
\]

where \( B \) is a (mainly temperature dependent) material factor, \( T \) is the second invariant of the stress tensor and \( n \) the stress exponent (Cobbold, 1983; Schmid et al., 1987). For our experiments, Eq. 1 can be simplified to (Bons, 1993):

\[
\dot{\gamma} = 2B(\sqrt{2})^{\frac{1}{n-1}} \tau \tag{2}
\]

where \( \dot{\gamma} \) is the shear strain rate \( \dot{\epsilon}_{ij} = 2 \dot{\epsilon}_{12} = 2 \dot{\epsilon}_{21} \) and \( \tau \) the shear stress.

The predicted bulk shear rate was \( 1.0 \times 10^{-4} \text{ s}^{-1} \) at 0.23 MPa and \( 2.8 \times 10^{-4} \text{ s}^{-1} \) at 0.28 MPa, using
the values $B$ and $n$ from the compression experiments (Bons and Urai, 1994). These values compare quite well with the low shear strain measurements in simple shear. Assuming that the low strain rheology of OCP in pure shear and simple shear is not significantly different, this suggests that friction at the sides of the sample does not play an important role. This assumption needs further testing, however. For example, Franssen and Spiers (1990) have shown that depending on the exact deformation mechanisms operating, there may be a significant difference between coaxial and non-coaxial rheology, even at low finite strain.

All experiments show an approximately three-fold

---

Fig. 3. Graphs of bulk shear strain as a function of time for experiments with pure OCP. (a) RS1, (b) RS2 (interrupted for 17.5 h), (c) RS3 and (d) RS6. (e) Graph of bulk shear strain rate as a function of bulk shear strain for experiments RS1-3 (at 0.23 MPa) and RS6 (at 0.28 MPa). All four experiments show an increase in shear strain rate up to a shear strain of about 4. In all subsequent plots, $w$ indicates the width of the moving window filter used for the different datasets.
Table 1
Details on the seven experiments discussed in this paper ( lubrication at confining cylinders: O = thin machine oil; G = Apiezon AP-101 grease)

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Composition (OCP:camphor)</th>
<th>Lubrication</th>
<th>Shear stress (MPa)</th>
<th>Finite shear strain</th>
<th>Duration (s)</th>
<th>Annealing time at (\gamma = 2) Mpa</th>
<th>Interruptions</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>100:0</td>
<td>O</td>
<td>0.23</td>
<td>0–44.3</td>
<td>1.7 (\times 10^5)</td>
<td>70 h (45 at 48°C)</td>
<td>--</td>
</tr>
<tr>
<td>RS2</td>
<td>100:0</td>
<td>G</td>
<td>0.23</td>
<td>0–5.9</td>
<td>9 (\times 10^4)</td>
<td>22 h (6 at 66°C)</td>
<td>no load for 17.5 h at (\gamma = 3.2)</td>
</tr>
<tr>
<td>RS3</td>
<td>100:0</td>
<td>G</td>
<td>0.23</td>
<td>8.7</td>
<td>3 (\times 10^4)</td>
<td>14 h (11 at 66°C)</td>
<td>--</td>
</tr>
<tr>
<td>RS5</td>
<td>68:32</td>
<td>G</td>
<td>0.23</td>
<td>&lt; 3.2</td>
<td>1.3 (\times 10^6)</td>
<td>29 h (25 at 60°C)</td>
<td>(\gamma = 1.02, 2.75, 10.3, 16.2 \text{ and } 30.9), 46, 74, 78, 111, 116 and 122</td>
</tr>
<tr>
<td>RS6</td>
<td>100:0</td>
<td>G</td>
<td>0.23</td>
<td>30</td>
<td>7 (\times 10^4)</td>
<td>24 h (6 at 66°C)</td>
<td>--</td>
</tr>
<tr>
<td>RS8</td>
<td>62:38</td>
<td>G</td>
<td>0.28</td>
<td>195</td>
<td>4 (\times 10^6)</td>
<td>51 h at 29°C</td>
<td>--</td>
</tr>
<tr>
<td>RS9</td>
<td>0:100</td>
<td>G</td>
<td>0.28</td>
<td>&lt; 0.05</td>
<td>1.1 (\times 10^5)</td>
<td>no data</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.39</td>
<td>0.05–0.28</td>
<td>1.1–2.1 (\times 10^6)</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.79</td>
<td>0.28–17.27</td>
<td>2.1–2.33 (\times 10^6)</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.23</td>
<td>17.27–17.33</td>
<td>2.33–2.48 (\times 10^6)</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

The increase in shear strain rate up to a shear strain of about 4, after which the shear strain rate stabilises at a 'steady-state' value. Examination of 1-mm-thick sections, cut with a razor blade after the end of the experiment from samples RS-1 and 3 showed a grain-shape preferred orientation oriented approximately 15° to the shear plane and a crystallographic preferred orientation with basal planes parallel to the shear plane. Development of similar microstructures was observed in transparent deformation cell experiments with OCP (Jessell and Lister, 1991), and we infer that the mechanical steady-state corresponds to a stable microstructure, where lattice rotation, grain boundary migration, sub-grain formation etc. have effectively reached a dynamic equilibrium.

Experiment RS2 was interrupted for 17.5 h at a shear strain of 3.2. After the interruption the shear rate commenced at a lower value than before the interruption. This lower shear strain rate could well be associated with recovery and/or grain growth that partly destroyed the existing microstructure.

The distribution of strain was investigated in experiments RS3 and RS6. The samples were cut in half after initial compaction, and two clearly visible black planes normal to the shear direction were produced with fine carbon powder. These planes deformed into a smooth S-shape, showing that shear localised at the grips (Fig. 4a and b). At a finite shear strain of 8.7 the shear strain in the middle of the samples was 2.8 for RS3; for RS6 the values were 29 and 16, respectively.

4.2. Pure camphor

One experiment (RS9) was done with pure camphor. The sample was deformed to a shear strain of 17.3 in four steps. The shear strain rate in camphor is very low at shear stresses equivalent to those used for the deformation of OCP (between \(10^{-8}\) and \(10^{-7}\) s⁻¹). Only small strain increments could be achieved
5. Experiments with two-phase samples

Most tectonites consist of more than one mineral and probably the most exciting experiments that can be done with this ring-shear apparatus are those with mixtures of rock analogues. We present two of these here. Samples RS5 and RS8 were both made of a random mixture of fine-grained OCP and 1-4 mm³ size blocks of camphor (32 and 38 vol.%, respectively). This starting material is comparable to a quartzitic rock with feldspar clasts. The two samples were deformed to shear strains of 122 and 195, respectively. Study of the microstructural developments was the main subject of experiment RS5. The experiment was interrupted at intervals and the outer confining cylinder removed to photograph the outer surface of the sample. Disturbance of the sample for observation, however, influences the shear strain rate. The second experiment (RS8) was therefore done at approximately identical conditions, but without interruptions for observation.

5.1. Microstructural development

Figs. 6 and 7 show the developing microstructure in RS5 at eight stages. Up to a shear strain of 32 the applied shear stress was 0.23 MPa (Fig. 8). At an average shear strain rate of $2.5 \times 10^{-3}$ s⁻¹ this took fifteen days. The shear stress was then raised to 0.28 MPa to reach a high finite strain in a practical time span. The shear strain rate increased to about $1 \times 10^{-4}$ s⁻¹ and later dropped to about $5 \times 10^{-5}$ s⁻¹ (Fig. 9). The initial stage of microstructural development is characterised by the stretching of camphor inclusions and localisation of shear strain at the grips. Camphor inclusions near the grips get stretched the most and/or form long ‘tails’. Inclusions that initially touch the grips move away towards the centre of the sample. Several inclusions have stretched into layers at a shear strain of 46. No photographs were taken between a shear strain of 46 and 111, during which a complete layering or foliation was established and disharmonic folding occurred. The fold development in two camphor layers visible in Fig. 6d-f is drawn in Fig. 9a. It can be seen that folds initiate with axial planes nearly perpendicular to the shear plane. This is followed by amplification and lateral propagation of folding in one fold train. During amplification, the folds tighten and the axial planes rotate toward the shear plane.

To observe the microstructure in three dimensions, five series of photographs were made at increasing depth in the sample, by scraping off 0.5-1
mm of sample material at a time. Stronger etching was applied to enhance contrast between the phases (Figs. 10 and 11). A variety of structures can be seen: open to close overturned folds to completely transposed isoclinal folds, duplex-like stacking of highly elongate camphor lenses and pinch-and-swell structures. Different layers show different styles of folding and varying stages of fold development, reflecting the variety in layer thickness and the timing of fold-initiation in each layer. Fold axes are not straight nor parallel within one fold train (Fig. 9b). Refolding can be seen in at least one fold-train. Refolding is a logical consequence of continuous simple shear deformation. Layers are folded and become transposed to isoclinal folds with both limbs parallel to the shear plane, after which a new cycle of folding can begin as described by Mawer and Williams (1991). Folding commences by buckling of layers and cannot be described in terms of passive shearing of initial perturbations (Quinquis et al.,

Fig. 6. Photographs of the microstructure in R55, a mixture of OCP (dark) and camphor (light). Photographs are taken at different stages during progressive deformation: (a) sample before deformation, (b) $\gamma = 10.3$, (c) $\gamma = 46$, (d) $\gamma = 111$, (e) $\gamma = 116$ and (f) $\gamma = 122$. The initial stage of foliation development is characterised by the stretching of the camphor inclusions into lenses and layers, visible in (b) at $\gamma = 10.3$ and (c) at $\gamma = 46$. Foliation development is followed by disharmonic folding of the individual layers; (d), (e) and (f) show two fold-trains during progressive folding at bulk shear strains of $\gamma = 111$ (d), $\gamma = 116$ (e) and at the end of the experiment at $\gamma = 122$ (f). The distance between the grips is 6 mm and the shear sense is top to the right.
1978), which does not provide for fold amplification normal to the shear plane. This amplification can be seen in Fig. 9 and inferred from the fact that some folds almost span the entire width of the shear zone. Buckling is probably initiated by local variations in the deformation field, caused by heterogeneities in the folding layer itself and by interaction with surrounding layers, lenses and folds (Hobbs et al., 1976; Ildefonse et al., 1992; Rykkvil and Fossen, 1992).

The applied shear stress in experiment RS8 was held at 0.28 MPa throughout the deformation to a shear strain of 195. The experiment was not interrupted for observation, to allow uninterrupted measurement of the stress-strain behaviour. Only the microstructure before and after deformation is known and shown in Fig. 12. The shear strain-time and shear strain rate-shear strain data are shown in Fig. 8. Although the microstructure after deformation in sample RS8 is similar to that in RS5, there are differences. There has been less stretching of the camphor layers, less folding and a more pronounced concentration of camphor inclusions in the middle of the sample. These differences could be the result of a combination of factors: relative large size of the camphor inclusions, larger fraction of camphor (38 instead of 32 vol%), higher initial shear stress (0.28 instead of 0.23 MPa), and the interruptions of RS5, allowing for recovery of the sample. Not enough experiments have been done to decide which factor is dominant. The two experiments, however, do show that small variations in conditions may have significant microstructural effects.

5.2. Shear strain rate history

The shear strain rate curves of experiments RS5 and RS8 are remarkably similar (Fig. 8), given the differences in final microstructure. Three stages can be distinguished.

Fig. 8: (a) Graph of the bulk shear strain as a function of time for experiment RS5. The first part of the experiment was done at a shear stress of 0.23 MPa and the second part at a shear stress of 0.28 MPa. The experiment was interrupted and the outer cylinder removed several times, as indicated by the little arrows. (b) Graph of the bulk shear strain as a function of time for experiment RS8. The shear strain rate was kept at 0.28 MPa throughout the experiment and there were no interruptions for observation. (c) Combined graph of the bulk shear strain rate as a function of bulk shear strain for experiments RS5 (thin line) and RS8 (bold line). The two graphs are remarkably similar, despite the differences in deformation history, microstructure and fraction of camphor inclusions.
(1) Both experiments show an initially rapid increase in shear strain rate, up to a shear strain of about 5.

(2) Whereas the shear strain rate then fluctuates around a constant value in RS5, it gradually increases in RS8, to reach its peak at a shear strain of about 40–75. After the increase in applied shear stress in RS5, shear strain rates also reach peak values of around $1.0 \times 10^{-4}$ s$^{-1}$ at a shear strain of 60–75.

(3) After this the shear strain rate gradually decreases to almost the initial value in both experiments. There is also a noticeable cyclic fluctuation in the shear strain rate.

6. Discussion

The correspondence between predicted shear strain rates from coaxial compression experiments and measured shear strain rates in our ring-shear apparatus indicates that the most critical point in the system friction at the sides of the sides of the sample, does not disturb measurements significantly. However, the accurate quantification of friction, and its possible effect on shear rates and microstructural development needs to be critically addressed in future work.

6.1. Pure end-member samples

All four experiments on OCP showed a consistent pattern of weakening (increase in shear strain rate) with strain up to a shear strain of about 4. We interpret this weakening to be associated with the formation of a dynamic steady-state microstructure (Means, 1981), i.e., the formation of a grain-shape and crystallographic preferred orientation in OCP and possibly in camphor also.

The experiments with the pure end members OCP and camphor show that different materials can have very different shear localization behaviour. Such contexts in the tendency to localize strain are probably also common in deforming minerals, and a description of this has to be included in the material's constitutive law to allow accurate numerical modelling of geological systems.

The ring-shear apparatus enables us to quantify this behaviour and try to correlate it with the deformation mechanisms operating. It also raises the question: what is the strength of a material? The shear rate in the middle 65% of the camphor sample is more than two orders of magnitude lower than near the grips. Yet the behaviour of the sample is domi-
nated by the 35% which is deforming rapidly. Such localisation of shear frequently occurs in natural shear zones, making it difficult to determine the rheology of the whole shear zone with flow laws for individual minerals, without knowledge of the localisation behaviour.

6.2. Two-phase samples

The correlation between microstructural and rheological developments in experiment RS5 and the similarity in stress–strain behaviour between RS5 and RS8 suggest that the observed changes in strain
Fig. 11. Drawing of the microstructure at different depths (d) in sample BSS after deformation. The drawings are corrected for the curvature of the cylindrical sample surface.
rate are indeed caused by the microstructural changes (Bons and Cox, 1994). The initial increase (stage 1) in shear strain rate is also observed in experiments with pure OCP. We interpret this to be due to the formation of a grain shape and crystallographic preferred orientation in OCP and possibly in camphor as well. The subsequent slower increase (stage 2) in shear strain rate is contemporaneous with the development of a foliation; we interpret this to be the main process responsible for the observed behaviour. The formation of a foliation subparallel to the shear plane enhances the partitioning of deformation and reduces the stress heterogeneities. The soft OCP can shear relatively unhindered by the camphor inclusions. The flow properties of the foliated composite thus move towards the high strain rate Reuss or static bound (Reuss, 1929). A second contributing factor could be softening of the camphor, which takes place at a slower rate, because of the locally lower strain rate in the harder camphor. The decrease in shear strain rate (in stage 3) coincides with the folding of the foliation. The folding of the camphor layers partially destroys the foliation, effectively breaking up the planar sub-shear zones of OCP. This reduces the heterogeneity of deformation and increases stress partitioning. The properties now move towards the low strain rate Voigt or Taylor bound (Voigt, 1928).

Softening due to foliation development is likely to occur in shear zones in rocks that have no distinct initial foliation, such as for instance granitic or gneissic rocks. This softening may contribute to shear localization (White et al., 1980; Hobbs et al., 1990), but this localization will not continue in a ‘run away’ manner. Firstly, softening will stop at the upper strain rate Reuss bound once an ideal planar

Fig. 12. Photographs of the microstructure in sample RS8. (a) The sample before deformation, and (b) and (c) the sample at a depth of 0.7 mm after deformation to a bulk shear strain of 195. The sample contained more camphor than that of RS5 (38% instead of 32%) and the initial camphor inclusions were larger than in RS5. Differences in deformed microstructure with RS5 are less stretching of the camphor inclusions and a stronger concentration of camphor inclusions half-way between the grips. The distance between the grips is 6 mm and the shear sense is top to the right.
foliation has formed. Secondly, as has been observed in the experiments, the planar foliation may be perturbed by folding, reversing the softening at high shear strain. It seems likely that a dynamic steady-state is reached eventually where stretching of folds and inclusions into layers is balanced by folding of these layers. In the experiments presented here this steady-state may not yet have been reached, but with different relative rheological properties it may settle at lower finite shear strain.

The observed cyclicity of shear strain rate in our experiments remains somewhat enigmatic. The cycles do not correspond with particular orientations of the rotating parts of the apparatus, suggesting that it is caused by changes in the strength of the deforming sample itself. We suggest that the cyclicity may be caused by the interaction of clusters of camphor inclusions. The effect can be detected in the stress-strain behaviour because of the relatively small number of camphor inclusions in the sample; it is expected to disappear if samples with a larger number of camphor inclusions are used.

The segregation of camphor and OCP, particularly well developed in RS8, is a phenomenon often observed in shear flows in suspensions (so called 'wall effect'). The presence of a rigid surface near a hard inclusion in a shearing soft matrix causes a net force on the inclusion away from that surface. This force is a function of the shear stress in the soft matrix and the distance from the surface relative to the size of the inclusion (Hsu and Ganatos, 1994). Phase segregation is often observed in natural shear zones. Apart from chemical segregation processes (Robins, 1979), this mechanical segregation process may operate by enhancing existing planar variations in the phase distribution.

6.3. Suggestions for further work

The first results presented here show that a hollow cylinder apparatus can be used to investigate rheological and microstructural developments up to very high shear strain. Here we have only presented the results for one type of composite: camphor inclusions in OCP. The study of the full range of composite types (hard inclusions in soft matrix and vice versa, different rheological contrasts between the components, reaction between the phases during deformation) remains to be done. In addition, the apparatus presented here can be used to study the development of single-inclusion structures, such as α and δ clasts (Passchier et al., 1993).

Friction in the system is yet not fully constrained. Further work has to be done to determine the amount of friction and the effect on the strain and strain rate distribution in a deforming sample. Ways should be sought to further minimise this friction. Possibilities are the use of low-friction materials as confining cylinders or a stack of rings that can rotate independently instead of one cylinder.

Other additions and modifications would be the use of a transparent outer cylinder to allow observation of the sample during deformation, and the option to do constant strain rate experiments. Experience with the deformation of soft materials in the ring-shear configuration may eventually aid the construction of a similar apparatus for the ductile deformation of real rock-forming minerals to very high simple shear strain.

Acknowledgements

JLU would like to express his gratitude to Neville Carter for many memorable and illuminating discussions in the past decade, and for warm hospitality during visits to A and M.

Technical assistance of the workshop of the Department of Earth Sciences, Utrecht University, is gratefully acknowledged. In particular we would like to mention Gert Kastelein, who, with the help of Arnout van der Gon-Netsch, built the prototype, which worked right away. PDB acknowledges the financial support of the Netherlands Organisation for Scientific Research for the project. Thorough reviews by Ben van der Pluijm and Jin-Han Ree are gratefully acknowledged.

Appendix A. Friction against the support cylinders

Fig. 13 shows a schematic drawing of the ringshear set-up with the relevant parameters for the determination of the shear stress and strain and strain
rate. The shear strain ($\Delta \gamma$) is related to the voltage output ($\Delta V_p$) of the potentiometer according to:

$$\Delta \gamma = \frac{\Delta V_p + k V_i}{c r_p r_s}$$

(A.1)

$r_s$ is a reference radius of the sample at which the height is $h_s$. The radii of the potentiometer wheel and the driving wheel are $r_p$ and $r_s$, respectively. The voltage output of the potentiometer ($V_i$) is a linear function of the angular position of the potentiometer wheel ($\omega_i$), given by: $V_i = c \omega_i$. It returns to 0 after one full cycle. $k$ is the number of full cycles and $V_i$ the voltage output at $360^\circ$ rotation of the potentiometer.

The shear stress ($\tau$) on the sample (neglecting friction) is given by:

$$\tau = \frac{3 g M r_w}{2 \pi (r_1^2 - r_0^2)}$$

(A.2)

g is the gravitational constant, $M$ the mass of the dead weight and $r_0$ and $r_1$ the inside and outside radius of the sample, respectively.

Friction with the cylinders at the sides of the sample gives rise to a complicated stress field within the sample. This stress field depends on the magnitude and nature of the friction, the rheology of the sample material and the shape of the sample. If the rheology of the sample material is strain-dependent, then the stress distribution will change with increasing bulk strain. It is therefore not possible to give a simple solution for the effect of friction on the stress distribution. Friction is highest at the corners where the grips and the confining cylinders meet, if the confining cylinders are stationary and the grips move in opposite directions. Here friction increases the shear stress in the sample and shear strain rate is highest. Away from the grips the difference between the velocity of the sample surface and the cylinder decreases and the effect of friction decreases. The result is an S-shaped velocity profile in the sample at the sides. The effect of friction decreases away from the sides of the sample. The applied stress and measured strain rates are bulk-values averaged over the whole sample. The effect of friction on these values depends on the width–height ratio of the sample, here approximately 1.

It is argued by Bons (1993) that friction may reduce the effective shear stress on the sample by up to 60% in the case that the side of the sample is completely coupled to the cylinder. This would result in a strong difference in shear profile between the sides and the middle of the sample, which is not observed. We therefore conclude that friction reduces the effective stress felt by the sample by (far) less than 60%.

One way to further investigate this is to apply only a torque on the outer confining cylinder and measure the rotation rate and deformation distribution inside the sample. This will provide an indication of the magnitude of the friction as a function of slip rate. This has to be done for each sample material and at different slip rates.

References


Ree, J.H., 1990. High temperature deformation of octachloro-


