Chapter 5

A model relating grain size distribution to the rheology of dynamically recrystallizing rock materials

5.1 Introduction

From both experimental and theoretical studies, it is well established that the microstructure of a material can be extensively modified by recrystallization during deformation. A relation between flow stress ($\sigma$) and the recrystallized grain size ($d$) that develops during steady state deformation, of the type $\sigma \propto d^{1/p}$ where $p$ is a constant, has generally been found for metals and ceramics [Takeuchi and Argon, 1976; Sellars, 1978; Humphreys and Hatherly, 1996] as well as geological materials [Twiss, 1977; Drury et al., 1985]. Recrystallized grain size versus stress relations for geological materials can be used as a piezometer to estimate the paleo-stress associated with the deformation of dynamically recrystallized tectonites in nature [Christie and Ord, 1980; Etheridge and Wilkie, 1981; White et al., 1985].

Deformation in natural tectonites is often found to be localized in zones of recrystallized rock material. Several mechanisms have been proposed that may cause localization in natural rocks, almost all of them being associated with rheological weakening [e.g., White et al., 1980; Vissers et al., 1995; Jin et al., 1998]. In particular, recrystallized grain size versus stress relations have been repeatedly used in combination with deformation mechanism maps to explore the possibility of weakening and associated strain localization due to grain refinement by dynamic recrystallization [Rutter and Brodie, 1988; Karato and Wu, 1993; Govers and Wortel, 1995]. In such approach, $\sigma$-$d$ relations are considered to be independent of the active deformation mechanisms, making a recrystallization induced switch in deformation mechanism from dislocation to diffusion creep possible if the relation transects the boundary between the diffusion and dislocation creep fields. However, evaluation of theoretical models for recrystallized grain size versus stress relations against experimental $\sigma$-$d$ data (chapter 1) suggests that dynamic recrystallization and creep behavior cannot be regarded as independent. Rather, a direct relationship between recrystallized grain size and active deformation mechanisms exists, with the recrystallized grain size falling in the boundary between diffusion and dislocation creep fields [cf. De Bresser et al., 2001]. This is explained by the hypothesis that the mechanism boundary acts as an attractor for recrystallized grain size, because small grains deforming by diffusion creep tend to grow towards the field boundary, while large grains deforming by dislocation creep tend to be reduced towards that boundary by dynamic recrystallization. Hence, grain growth and grain size reduction processes will tend to balance during steady state deformation, rendering a dynamically stable recrystallized grain size set up in the neighborhood of the diffusion-dislocation creep field boundary.

One consequence of a direct relationship between recrystallized grain size and active creep mechanisms is that temperature cannot be à priori ignored, while conventional $\sigma$-$d$ relations usually do not include temperature. So far, experimental support for a temperature dependence of recrystallized grain size is limited, but some experimental evidence has been presented [Mercier et al., 1977; Ross et al., 1980; Tungatt and Humphreys, 1984; De Bresser et al., 1998]. These studies indicate that temperature only weakly influences recrystallized grain size and may be noticed only in studies that systematically explore the role of temperature. This is corroborated by the results obtained from new systematic deformation experiments on natural Carrara marble (chapter 3) and synthetic polycrystalline halite (chapter
4). A possible effect of temperature should be taken into account when using \( \sigma-d \) relations in paleo-piezometry.

Another consequence of the inferred direct relationship between recrystallized grain size and active creep mechanisms is that grain size needs to be distributed to allow grain growth and grain size reduction to occur simultaneously. Dynamically recrystallized rock materials invariably exhibit a grain size distribution that may vary considerably in width (expressed by the standard deviation of the distribution) as well as in median or average grain size (cf. chapters 3 and 4) [Michibayashi, 1993; Dijkstra, 2001]. The importance of this has been demonstrated in chapter 2, where it has been shown that variation in distribution width can have a pronounced effect on the rheology of materials. This effect cannot be ignored. Only recently though, measurement of grain size distribution instead of determination of a single, mean value has become standard [e.g. Heilbronner and Bruhn, 1998; Post and Tullis, 1999; Pieri et al., 2001a]. However, theoretical models for dynamic recrystallization as well as formulations of flow laws used so far generally regard grain size as single-valued, and consequently cannot account for variations in the width of the grain size distribution. Obviously, this gap between the demonstrated effect of grain size distribution and the practical application in \( \sigma-d \) equations that are related to rheological behavior needs to be filled.

Aim now of the present chapter is to develop a model that relates recrystallized grain size to rheology taking the distribution of grain size and temperature into account. We use the composite diffusion-dislocation creep laws derived for materials with a lognormal grain size distribution in chapter 2, and combine these with the field boundary model [De Bresser et al., 1998; 2001 – see also chapter 1]. The result is a model equation that relates median grain size and standard deviation to stress and temperature and incorporates flow parameters for diffusion and dislocation creep. The model is tested against the experimental data obtained on wet polycrystalline halite (chapter 4). The obtained model allows more reliable estimation of deformation conditions in nature since it is not hindered by inconsistencies between extrapolated flow laws and extrapolated \( \sigma-d \) relations. It thereby improves analysis of the role of grain microstructure evolution in weakening and localization.

5.2 Model Development

The model addresses a single phase polycrystalline material with lognormally distributed grain size (see chapter 2), undergoing steady state deformation by GSS and GSI mechanisms that operate as independent, parallel-concurrent processes [Poirier, 1985]. If stress is assumed to be uniform throughout the material, the composite diffusion-dislocation creep law can be written as (chapter 2)

\[
\dot{\varepsilon}_{\sigma} = \left[ \frac{A_{\text{diff}} b D_{\text{diff}} \mu}{kT} \left( \frac{b}{\exp\left(3 - \frac{m}{2} \phi^2\right) d_{\text{med}}} \right)^{m} \left( \frac{\sigma_{\sigma}}{\mu} \right) \right] + \left[ \frac{A_{\text{dis}} b D_{\text{dis}} \mu}{kT} \left( \frac{\sigma_{\sigma}}{\mu} \right)^n \right] \tag{5.1}
\]

All symbols used are defined in table 2.1 (chapter 2). If strain rate is uniform throughout the material, strain rate can be approximated by a composite flow law of the type

\[
\dot{\varepsilon}_{\text{approx}} = \left\{ \left[ \frac{A_{\text{diff}} b D_{\text{diff}} \mu}{kT} \left( \frac{b}{\exp\left(3 + \frac{m}{2} \phi^2\right) d_{\text{med}}} \right)^{m} \left( \frac{\sigma_{\sigma}}{\mu} \right) \right] + \left[ \frac{A_{\text{dis}} b D_{\text{dis}} \mu}{kT} \left( \frac{\sigma_{\sigma}}{\mu} \right)^n \right] \right\} \left\{ \frac{l}{1 - F(\sigma^*, \phi, n, m)} \right\}
\]
Recrystallized grain size distribution model

where \( \sigma^* = \sigma / \sigma_{\text{int}} \) with \( \sigma_{\text{int}} = \left( \frac{A_{\text{diff}} D_{\text{diff}}}{A_{\text{disl}} D_{\text{disl}}} \right)^{\frac{1}{n-1}} \left( \frac{b}{\exp\left[3 + \frac{1}{2} m \phi^2\right] d_{\text{med}}} \right)^{\frac{m}{n-1}} \) (5.2)

In (5.2), \( F(\sigma^*, \phi, n, m) \) represents a function with three parameters that are related to the stress exponent \((n)\), grain size exponent \((m)\) and standard deviation \((\phi)\). It needs to be incorporated to keep the error between the approximated strain rate and the true strain rate, determined numerically, at acceptable levels for high values of \( n (n=5) \) and \( \phi (\phi=1.2) \). Its exact meaning and values for \( m=2, 3<n<5 \) and \( 0<\phi<1.2 \) are given in chapter 2. Flow laws (5.1) and (5.2) describe bounds on the rate of deformation in a single phase material with a distributed grain size, in a similar manner as uniform stress or strain rate provide bounds on the rate of deformation in polyphase materials [Raj and Ghosh, 1981; Tullis et al., 1991]. Both flow laws incorporate a diffusion creep term that is dependent on the median grain size and standard deviation of the lognormal grain size distribution, and a dislocation creep term that is grain size insensitive.

As a next step, we apply the field boundary model formulated for single-fixed grain size [De Bresser et al., 1998; 2001], which assumes that the steady state grain size \( d \) that develops during dynamic recrystallization is set up in the boundary between the diffusion and dislocation creep fields. This concept is easily envisaged if a single-phase polycrystal is considered with a specific grain size distribution consisting of small grains and large grains. The small grains dominantly deform by diffusion creep and are likely to grow, while the large grains dominantly deform by dislocation creep and are likely to be reduced by dynamic recrystallization. If this rationale holds, both deformation mechanisms will have a contribution to the overall creep rate and because diffusion and dislocation creep mechanism are parallel-concurrent [Poirier, 1985], their strain rates are additive. At the boundary, both mechanisms contribute equally, but the recrystallized grain size might adjust itself to a different relative contribution of diffusion and dislocation creep [De Bresser et al., 2001], written as

\[
\dot{\varepsilon}_{\text{diff}} / \dot{\varepsilon}_{\text{disl}} = U \tag{5.3}
\]

This expression has already been used to derive a relation between stress, temperature and the recrystallized grain size (chapter 1), but can now be modified using (5.1) or (5.2) to include grain size distribution (assuming a lognormal shape of the distribution, characterized by median \( d \) and standard deviation \( \phi \) in log-space). If stress is uniform, substitution of (5.1) in (5.3) yields

\[
\left( \frac{\sigma}{\mu} \right) = \left( \frac{A_{\text{diff}} D_{\text{diff}}}{UA_{\text{disl}} D_{\text{disl}}} \right)^{\frac{1}{n-1}} \left( \frac{b}{\exp\left[3 - \frac{1}{2} m \phi^2\right] d_{\text{med}}} \right)^{\frac{m}{n-1}} \exp\left( \frac{Q_{\text{disl}} - Q_{\text{diff}}}{(n-1)RT} \right) \tag{5.4}
\]

If strain rate is uniform through the aggregate, the physical meaning of (5.3) is not straightforward, because bulk strain rate is not an independent variable but dependent on the distribution of stress in the polycrystal. The volume average of the stress in individual grains determines the overall creep rate (chapter 2) and therefore the contributions of the diffusion and dislocation creep rate to the overall creep rate cannot be simply expressed as a ratio. However, this problem only arises if both mechanisms contribute significantly to the overall creep rate. The diffusion and dislocation creep terms in (5.2) describe the pure
diffusion and dislocation creep rate if strain rate is uniform. This means that the position of lines (in \( \sigma-d \) space) parallel to the field boundary can be determined by (5.3), but that these lines may not be used to quantify the relative contribution of both mechanisms to the overall creep rate. Substitution of (5.2) in (5.1) yields

\[
\left( \frac{\sigma}{\mu} \right) = \left( \frac{A_{\text{diff}}D_{\text{diff}}}{U A_{\text{disl}}D_{\text{disl}}} \right)^{\frac{1}{n}} \left( \frac{b}{\exp \left( 3 + \frac{n}{m} \right) \phi^2 d_{\text{med}}} \right)^{\frac{m}{n-1}} \exp \left( \frac{Q_{\text{disl}} - Q_{\text{diff}}}{(n-1)RT} \right) \tag{5.5}
\]

It should be emphasized at this point that \( U \) in expressions (5.4) and (5.5) is not necessarily a constant for all deformation conditions, as \( U \) will be determined by the relative rates of grain size reduction and grain growth processes. However, for sufficiently small ranges of deformation conditions, \( U \) may be assumed constant. Full evaluation of the magnitude of \( U \) and its dependence on deformation conditions can only be performed using models that describe evolution of grain size distribution due to the different microphysical processes involved in dynamic recrystallization [e.g. Derby and Ashby, 1990; Shimizu, 1998], which is beyond the scope of this paper.

Equations (5.4) or (5.5) now form an improved relationship between recrystallized grain size and flow stress, taking into account a lognormal distribution of grain size. The relation is directly linked to the active creep mechanisms via the parameters \( n \) and \( Q_{\text{disl}} \) (for dislocation creep) and \( m \) and \( Q_{\text{diff}} \) (for diffusion creep), respectively. Note that (5.4) and (5.5) are temperature dependent as long as the two activation energy terms are not equal.

### Table 5.1 Selected experiments and grain size distribution data for experimentally deformed and fully recrystallized wet polycrystalline halite (cf. chapter 4).

<table>
<thead>
<tr>
<th>test</th>
<th>flow stress (\dagger) [MPa]</th>
<th>strain rate (\dagger) [s(^{-1})]</th>
<th>temp. (\dagger) [°C]</th>
<th>natural strain</th>
<th>median grain size [(\mu\text{m})]</th>
<th>standard deviation for ln(d)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7RS150</td>
<td>7.2</td>
<td>5.3 x 10(^{-7})</td>
<td>149</td>
<td>0.29</td>
<td>355</td>
<td>1.15</td>
</tr>
<tr>
<td>7RS200</td>
<td>7.5</td>
<td>2.4 x 10(^{-6})</td>
<td>203</td>
<td>0.30</td>
<td>269</td>
<td>1.24</td>
</tr>
<tr>
<td>7RS240</td>
<td>7.3</td>
<td>1.6 x 10(^{-5})</td>
<td>243</td>
<td>0.33</td>
<td>380</td>
<td>1.05</td>
</tr>
<tr>
<td>10RS240</td>
<td>9.5</td>
<td>1.3 x 10(^{-4})</td>
<td>243</td>
<td>0.34</td>
<td>163</td>
<td>0.82</td>
</tr>
<tr>
<td>11RS125</td>
<td>10.8</td>
<td>5.4 x 10(^{-7})</td>
<td>243</td>
<td>0.31</td>
<td>161</td>
<td>1.06</td>
</tr>
<tr>
<td>11RS150a</td>
<td>11.3</td>
<td>2.2 x 10(^{-6})</td>
<td>150</td>
<td>0.31</td>
<td>322</td>
<td>1.01</td>
</tr>
<tr>
<td>11RS150b</td>
<td>11.3</td>
<td>1.4 x 10(^{-6})</td>
<td>150</td>
<td>0.30</td>
<td>230</td>
<td>1.06</td>
</tr>
<tr>
<td>11RS175</td>
<td>10.9</td>
<td>6.6 x 10(^{-6})</td>
<td>174</td>
<td>0.30</td>
<td>202</td>
<td>0.95</td>
</tr>
<tr>
<td>11RS200</td>
<td>11.1</td>
<td>2.4 x 10(^{-5})</td>
<td>202</td>
<td>0.36</td>
<td>183</td>
<td>0.90</td>
</tr>
<tr>
<td>13RS125</td>
<td>13.3</td>
<td>1.3 x 10(^{-6})</td>
<td>125</td>
<td>0.30</td>
<td>193</td>
<td>0.97</td>
</tr>
<tr>
<td>13RS150</td>
<td>13.3</td>
<td>4.5 x 10(^{-6})</td>
<td>150</td>
<td>0.32</td>
<td>173</td>
<td>0.80</td>
</tr>
<tr>
<td>14RS100</td>
<td>13.6</td>
<td>5.3 x 10(^{-7})</td>
<td>99</td>
<td>0.29</td>
<td>158</td>
<td>1.05</td>
</tr>
<tr>
<td>14RS150</td>
<td>14.4</td>
<td>6.6 x 10(^{-6})</td>
<td>150</td>
<td>0.32</td>
<td>159</td>
<td>0.82</td>
</tr>
<tr>
<td>14RS175</td>
<td>13.9</td>
<td>5.0 x 10(^{-5})</td>
<td>171</td>
<td>0.44</td>
<td>133</td>
<td>0.93</td>
</tr>
<tr>
<td>18RS100</td>
<td>17.9</td>
<td>2.8 x 10(^{-6})</td>
<td>100</td>
<td>0.29</td>
<td>164</td>
<td>0.97</td>
</tr>
<tr>
<td>18RS125</td>
<td>17.7</td>
<td>7.4 x 10(^{-6})</td>
<td>126</td>
<td>0.34</td>
<td>136</td>
<td>0.86</td>
</tr>
<tr>
<td>18RS150</td>
<td>17.6</td>
<td>3.6 x 10(^{-5})</td>
<td>148</td>
<td>0.46</td>
<td>124</td>
<td>0.85</td>
</tr>
<tr>
<td>22RS100</td>
<td>22.1</td>
<td>7.5 x 10(^{-6})</td>
<td>99</td>
<td>0.38</td>
<td>116</td>
<td>0.93</td>
</tr>
</tbody>
</table>

\(\dagger\) Flow stress, temperature, strain rate and pressure averaged over a strain of 0.01 at the end of the test are quoted.
5.3 Evaluation of the model using data on wet polycrystalline halite

We will now test the model given by (5.4) and (5.5) against the data for wet polycrystalline halite, obtained in chapter 4. Most of the wet polycrystalline halite samples were completely recrystallized and are therefore favored above the partially recrystallized samples of Carrara marble (chapter 3) in evaluating the model. Details on the experimental method and data on wet polycrystalline halite have been described in detail in chapter 4. Only samples that have completely recrystallized and have been deformed to roughly similar strain are used (table 5.1). The standard deviations determined for the logarithmic grain size \( \log(d) \), quoted in table 4.3, are converted to standard deviations for natural logarithmic grain size \( \ln(d) \) to allow direct substitution in (5.4) and (5.5). In this way, the distributions analyzed in chapter 4 are approximated by a lognormal grain size distribution. This is a viable approach as shown by the example distribution of fig. 5.1.

![Figure 5.1 Grain size distribution for a representative fully recrystallized sample of wet polycrystalline halite compared with an ideal, continuous lognormal grain size distribution.](image)

In order to test the model, flow law parameters for pure diffusion creep and pure dislocation creep for polycrystalline halite are required (assuming that the effect of grain boundary migration on pure dislocation creep is limited). Analysis of mechanical data and microstructures of deformed dry polycrystalline halite indicates that it predominantly deforms by dislocation creep processes, without evidence for significant contribution of diffusion creep mechanisms (chapter 4). Carter et al. [1993] claim that samples of Avery Island natural halite deformed by pure cross slip controlled dislocation creep at conditions (high strain rate, high stress) similar to the conditions for which our recrystallized grain size data was obtained. Accordingly, the flow law defined by Carter et al. [1993] is taken as representing pure dislocation creep. Flow law parameters are given in table 5.2. In contrast, wet polycrystalline halite shows widespread evidence for dislocation creep as well as solution-precipitation processes. The flow law for solution-precipitation creep in dense aggregates of wet synthetic polycrystalline halite is well defined [Spiers et al., 1990; Spiers and Carter, 1998]. Using this flow law, we have shown in chapter 4 that the difference in strength between the dry and wet polycrystalline halite samples could be largely explained by the joint activity of solution-precipitation creep and dislocation creep mechanisms in the wet material, as opposed to the single dislocation creep deformation of the dry samples. The solution-precipitation creep law of Spiers and Carter [1998] thus can be taken as representative of diffusion creep in wet polycrystalline halite aggregates. Accordingly, the flow parameters are used as input for the
model (table 5.2). In order to calculate the rate constant $A_{\text{diff}}$, we assume that the grain size distribution of the material used to calibrate the solution-precipitation flow law is similar to the grain size distribution of the starting material in the present study (fig. 4.4). This is a realistic assumption as the sample preparation procedure described in chapter 4 was very similar to that used to produce the material the flow law was calibrated for [Spiers et al., 1986]. It does mean, however, that $A_{\text{diff}}$ is different for the uniform stress and uniform strain rate case, because of differences in the distribution terms containing median grain size and standard deviation (compare (5.1) and (5.2), see table 5.2).

Table 5.2 Constants and flow law parameters for solution-precipitation creep [Spiers et al., 1990; Spiers and Carter, 1998] and cross slip controlled dislocation creep [Carter et al., 1993] in polycrystalline halite used for the evaluation of the presented model against experimental data on wet polycrystalline halite.

<table>
<thead>
<tr>
<th>parameter</th>
<th>value</th>
<th>dimensions</th>
<th>source/remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_{\text{diff}}$</td>
<td>$\pi \delta D_{\text{diff}}$</td>
<td>$1.90 \times 10^{-3}$</td>
<td>- for uniform stress, calculated using the rate constant from Spiers and Carter [1998] and values for $b$ and $\mu$ from Frost and Ashby [1982]</td>
</tr>
<tr>
<td>$A_{\text{diff}}$</td>
<td>$\pi \delta D_{\text{diff}}$</td>
<td>$1.05 \times 10^{-1}$</td>
<td>- for uniform strain rate, calculated using the rate constant from Spiers and Carter [1998] and values for $b$ and $\mu$ from Frost and Ashby [1982]</td>
</tr>
<tr>
<td>$Q_{\text{diff}}$</td>
<td></td>
<td>24530 J.mol^{-1}</td>
<td>Spiers et al. [1990]</td>
</tr>
<tr>
<td>$m$</td>
<td></td>
<td>3</td>
<td>- Spiers et al. [1990]</td>
</tr>
<tr>
<td>$A_{\text{disl}} D_{\text{disl}}$</td>
<td></td>
<td>$1.87 \times 10^{-3}$</td>
<td>- calculated using the rate constant and stress exponent quoted in Carter et al. [1993] and values for $b$ and $\mu$ from Frost and Ashby [1982] for $T=100^\circ\text{C}$</td>
</tr>
<tr>
<td>$Q_{\text{disl}}$</td>
<td></td>
<td>68100 J.mol^{-1}</td>
<td>Carter et al. [1993]</td>
</tr>
<tr>
<td>$n$</td>
<td></td>
<td>5.3</td>
<td>- Carter et al. [1993]</td>
</tr>
<tr>
<td>$b$</td>
<td></td>
<td>$3.99 \times 10^{-10}$ m</td>
<td>Frost and Ashby [1982]</td>
</tr>
<tr>
<td>$\mu$</td>
<td></td>
<td>$1.5 \times 10^{-10}$ Pa</td>
<td>Frost and Ashby [1982] for $T=300$ K, small temperature dependence neglected</td>
</tr>
<tr>
<td>$k$</td>
<td></td>
<td>$1.381 \times 10^{-23}$ J.K^{-1}</td>
<td>-</td>
</tr>
<tr>
<td>$R$</td>
<td></td>
<td>8.314 J.mol^{-1}.K^{-1}</td>
<td>-</td>
</tr>
</tbody>
</table>

5.3.1 Deformation mechanism maps of grain size versus stress including a distribution term

Figure 5.2a shows the distribution data from table 5.1 plotted in a deformation mechanism map for stress versus the distribution term $\exp((3-\frac{1}{2}m)\hat{\phi})d_{\text{med}}$ for the case of uniform stress (5.1), with $m=3$ (table 5.2). Strain rate contours are indicated as well. The map is drawn for $T=150^\circ\text{C}$, but distribution data for the temperature range 125-175$^\circ\text{C}$ are included. The field boundary is defined as the region where both diffusion and dislocation creep contribute for more than 10% to the overall strain rate, indicated by the lines with $U=0.11$ and $U=10$. In addition, the position of the line where both mechanisms contribute equally to the overall strain rate ($U=1$) is indicated. All the distribution data fall in the diffusion-dislocation creep field boundary.
Figure 5.2 Deformation mechanism maps of stress versus the distribution term with strain rate contours for T=150°C. Experimental data of wet polycrystalline halite for T=125-175°C (table 5.1) are superimposed. (a) Uniform stress case with lines for U=0.11, 1 and 10 denoting 10, 50 and 90% contribution of diffusion creep. (b) Uniform strain rate case with strain rate contours for \( \phi = 0.5 \) and \( \phi = 0.8 \).

A map similar to that of fig. 5.2a can be drawn for uniform strain rate with strain rate contours that differ with standard deviation (fig. 5.2b), using the distribution term \( \exp((3+\frac{1}{2}m)\phi^2)d_{med} \) with \( m = 3 \). As shown in chapter 2, the width of the boundary region spreads out considerably with increasing standard deviation. However, the accuracy of the composite flow law given by (5.2) decreases with increasing standard deviation due to a reduced effectiveness of the error-reducing function \( F(\sigma^*, \phi, n, m) \) (as can be observed by the irregularity of the contours at \( \phi = 0.8 \)). The \( U = 0.11, 1 \) and \( 10 \) lines are not included in the map, because for uniform strain rate these are merely lines parallel to the field boundary and do not indicate the contribution of diffusion creep to the overall strain rate as in the uniform stress case (section 5.2). The map can be used, however, to show that the distribution data also falls in the region where both mechanisms contribute significantly to the overall strain rate (field boundary) for uniform strain rate, consistent with the results of fig. 5.2a. Note at this point that strain rate contours have been drawn for standard deviations (\( \phi = 0.5 \) and \( \phi = 0.8 \)) that are low in comparison with the distribution data (table 5.1), because the error in strain rate increases towards higher standard deviations. As the width of boundary region spreads with increasing standard deviation (compare contours for \( \phi = 0.5 \) and \( \phi = 0.8 \)), the data will be positioned even further into the boundary for higher standard deviations.

5.3.2 Deformation mechanism maps of stress versus strain rate

The flow laws given in (5.1) and (5.2) can be written in normalized form (chapter 2) as

\[
S = \left[ C \left( \frac{\sigma}{\mu} \right) ^n \left( \frac{\sigma}{\mu} \right) ^{0.5} \right] \left[ \frac{1}{1 - F(\sigma^*, \phi, n, m)} \right] (5.6)
\]

where \( S = \frac{\dot{\varepsilon}kT}{A_{\text{dis}}bD_{\text{dis}}\mu} \) and \( C = A_{\text{dis}}D_{\text{dis}} \left( \frac{b}{\lambda X} \right) ^m \exp \left( \frac{Q_{\text{dis}} - Q_{\text{diff}}}{RT} \right) \).
In this expression, $X = \exp[(3-m/2)\phi^2]d_{med}$ and $F(\sigma^\star, \phi, n) = 0$ for uniform stress and $X = \exp[(3+m/2)\phi^2]d_{med}$ and $F(\sigma^\star, \phi, n)$ represents the same function as in (5.2) for uniform strain rate. The advantage of (5.6) is that universal deformation mechanism maps can be constructed of normalized strain rate $S$ to the normalized stress ($\sigma/\mu$) for different values of $C$ containing the median grain size and standard deviation. This allows data of different stress, temperature and grain size distribution to be plotted in a single diagram.

Fig. 5.3a and 5.3b show universal mechanism maps drawn for wet polycrystalline halite for uniform stress and strain rate (calculated using the flow law parameters of table 5.2). For uniform stress, the position of the $U=0.11$, 0.1 and 10 lines is fixed for all standard deviations and indicate a 90, 50 or 10% contribution of diffusion creep to the overall strain rate. For uniform strain rate, the position of the $U=0.11$, 0.1 and 10 lines are not included because of reasons mentioned before. Values of $S$ for the distribution data of table 5.1 have been calculated using (5.6) and the data are indicated on both maps. The distribution data plot close to the boundary region for uniform stress and in the boundary for uniform strain rate. In addition, the slope of the best fit line through the data points is sub-parallel (slope: $4.58 \pm 0.39$) to the lines with a constant value of $U$ denoting the diffusion-dislocation creep boundary zone (slope: 5.3, equal to stress exponent $n$).

Figure 5.3 Deformation mechanism map of normalized strain rate $S$ versus normalized stress $\sigma/\mu$ with contours of the normalized parameter $C$. Experimental data of wet polycrystalline halite (table 5.1) and best fit line through the data are superimposed. (a) Uniform stress with lines for $U=0.11$, 1 and 10 denoting 10, 50 and 90% contribution of diffusion creep. (b) Uniform strain rate with $C$ contours for $\phi=0.5$ and $\phi=0.8$.

In comparing the position of the data points on the maps with that of the lines of constant $U$, a point of caution should be made. The $C$ contours on the map are calculated using best fit values for the flow law parameters (cf. 5.6). However, the position of the data points on the maps is dependent on the values of these flow law parameters, as $S$ and $C$ of the data are calculated using the same flow law parameters. The use of slightly different values for the flow law parameters, within the error bars resulting from the quality of fit of the mechanical data to the flow equations, may move the data relative to the lines of constant $U$. 
5.4 Discussion

In this chapter, a model is advanced that relates the grain size distribution that develops during dynamic recrystallization to rheology. The model uses composite diffusion-dislocation creep laws for materials with a lognormal grain size distribution. The model is based on the hypothesis that the grain size distribution of dynamically recrystallizing materials tends to organize itself in the boundary between the diffusion and dislocation creep fields (chapter 1). In this, the boundary is taken as the region where both mechanisms contribute significantly (but not necessarily equal) to the overall strain rate. The requirements for this to happen are that (1) grain size is distributed, (2) diffusion creep contributes significantly to the strain rate of (small) grains that nucleate during dynamic recrystallization, and (3) grains are allowed to grow by normal grain growth or by grain boundary migration during dynamic recrystallization. The mechanical and microstructural results obtained from experiments on natural Carrara marble (chapter 3) and synthetic polycrystalline halite (chapter 4) have shown that these requirements are generally fulfilled, at least in these two, relatively pure single phase materials.

The new set of data on wet polycrystalline halite, covering a systematic range in strain rate, temperature and stress, has been selected to test the model formulated in this chapter, taking into account the complete grain size distributions. As far as we are aware, such analysis has not been performed before, which seems a deficiency since distributed grain sizes are standard in rock and other materials. Grain size distributions are taken as approximately log-normal in our analysis, supported by measurements on a variety of rock materials [e.g., Ranalli, 1984; Michibayashi, 1993; Newman, 1994; Post and Tullis, 1994; Shimizu, 1999; Dijkstra, 2001; chapters 3, 4]. The halite data are in agreement with the model. Several points, however, need further discussion. These are (1) the consequence for paleopiezometry of incorporating standard deviation in \( \sigma-d \) relations, (2) the implications in terms of rheological weakening, and (3) the limitations of the model.

5.4.1 Consequences of incorporating standard deviation in \( \sigma-d \) relations

The present analysis differs from conventional methods used to calibrate a relation between recrystallized grain size and stress and temperature by the incorporation of the distribution parameters, standard deviation and median grain size, in the distribution term \( \exp((3 \pm \frac{\sigma}{\sqrt{2m}})\phi) d_{med} \). Because conventional relations only use a single value for the grain size [e.g., Derby and Ashby, 1990; Shimizu, 1998; De Bresser et al., 2001], paleo-stresses calculated from earlier piezometric relations may be over- or underestimated. This is illustrated in fig. 5.4, which shows the variation in estimated paleo-stress (expressed as \( \sigma_2/\sigma_1 \)) between materials with different standard deviation (\( \phi_1 \) and \( \phi_2 \)) of the lognormal grain size distribution for the uniform stress case using \( n=3 \) (e.g. olivine, see table 2.3) and \( m=3 \) (grain boundary diffusion creep). As an example, \( \phi_1 \) may indicate the standard deviation of the material used to calibrate the piezometer and \( \phi_2 \) may indicate the standard deviation of a natural tectonite to which the piezometer is applied. In this case, conventional relations that do not account for variations in standard deviation will only predict accurate paleo-stresses if
\( \phi_1 = \phi_2 (\sigma_2 / \sigma_1 = 1) \). The error in estimated stress may be up to one order of magnitude \( (\sigma_2 / \sigma_1 = 0.1) \) for realistic variations in standard deviation (e.g. \( \phi_1 = 0.8 \) and \( \phi_2 = 1.2 \)).

![Illustration of the effect of variations in standard deviation of the lognormal grain size distribution (\( \phi_1 \) and \( \phi_2 \)) on paleo-stress estimates in nature. Piezometers that do not account for differences in standard deviation may significantly over- or underestimate \( (\sigma_2 / \sigma_1 \neq 1) \) paleo-stresses of natural tectonites.](image)

If the boundary hypothesis advanced by De Bresser et al. [1998; 2001] is valid, the model given by (5.4) and (5.5) provides a physical basis for piezometers, constraining the grain size distribution to the diffusion-dislocation creep boundary. In contrast, conventional piezometers that relate recrystallized grain size to stress (and in some cases temperature) are usually fully empirical and unrelated to the active creep mechanisms. The model does not assume \( \text{à priori} \) a specific recrystallization mechanism (e.g. migration recrystallization, Derby and Ashby, [1990], or rotation recrystallization, Shimizu, [1998]). Through the underlying assumptions, the model implicitly requires the recrystallized grain size distribution to be expressed in a distribution characteristics term \( \exp((3 \pm 1/2m) \phi^2)d_{med} \) (with median grain size \( d_{med} \), standard deviation \( \phi \), and \( m = 2 \) or \( m = 3 \) for diffusion creep controlled by volume or grain boundary diffusion, respectively). Consequently, an important implication of the model is that recrystallized grain size is not uniquely related to stress and temperature, as predicted by conventional models. Instead, stress and temperature only determine the magnitude of the distribution characteristics term. This is an important issue, because the standard deviation of the recrystallized grain size distribution is often dependent on the deformation conditions (see table 5.1 and chapters 3 and 4). Conventional models cannot account for a variation in standard deviation and would predict erroneous stresses and strain rate based on a single value for grain size.
5.4.2 Implications for rheological weakening

A direct consequence of the model is that the rheology of dynamically recrystallizing materials is constrained to the diffusion-dislocation creep boundary for all deformation conditions, as was originally proposed by De Bresser et al. [2001] for materials with a single-valued grain size. Using (5.3), the steady state creep rate of dynamically recrystallizing materials (\( \dot{\varepsilon}_{rx} \)) assuming uniform stress can be written as

\[
\dot{\varepsilon}_{rx} = \dot{\varepsilon}_{\text{diff}} + \dot{\varepsilon}_{\text{dis}} = (U + 1)\dot{\varepsilon}_{\text{dis}}
\]

(5.6)

This means that for uniform stress, assessment of \( U \) and the experimentally calibrated flow law parameters for the dislocation creep part of (5.1) or (5.2) suffices to constrain the rheology of a specific material under natural conditions. As a consequence, a complete switch in deformation mechanism from dislocation to diffusion creep may not occur as recrystallized grain size is constrained to the diffusion-dislocation creep boundary. This has also been pointed out by De Bresser et al. [2001] for a single-valued grain size (chapter 1). These authors conclude that rheological weakening due to dynamic recrystallization may be limited and insufficient to cause strain localization in nature. The present model incorporates the effect of the width of the grain size distribution, and gives nuances to this conclusion. For uniform strain rate, the diffusion-dislocation creep boundary may spread out considerably with increasing standard deviation (chapter 2, see also fig. 5.2b). This means that even if grain size organizes itself in the diffusion-dislocation creep boundary, rheological weakening due to dynamic recrystallization may be larger than expected on the basis of a single-valued grain size. The amount of weakening depends on the change in standard deviation and median grain size due to dynamic recrystallization. Considering the fact that for uniform strain rate the boundary may spread out over orders of magnitude in stress for large standard deviations (chapter 2, see also Freeman and Ferguson, [1986]), weakening may be considerable without a complete switch in deformation mechanism. The uniform stress and uniform strain rate case provide bounds on the rate of deformation in real polycrystals [Raj and Ghosh, 1981; Freeman and Ferguson, 1986]. For uniform stress, (5.6) predicts minor weakening (a factor 2, if \( U=1 \) corresponding to an equal contribution of diffusion and dislocation creep, cf. section 1.4.4), but weakening may be expected to be larger if strain rate is assumed uniform. This means that rheological weakening may be expected to be larger than predicted by (5.6). Accurate assessment of rheological weakening due to dynamic recrystallization in real polycrystalline materials needs to account for distributed grain size.

5.4.3 Final remarks

The data on wet polycrystalline halite are found to be in agreement with the proposed model relating recrystallized grain size distribution to the rheology controlled by diffusion as well as dislocation mechanisms. Further experimental support for the model should come from other systematic data on the mechanical behavior and grain size distribution of single materials. In this, focus should be on calibrating microphysically based pure diffusion and dislocation creep (end-member) laws for a single material, as the model incorporates flow law parameters for both mechanisms. In addition, microphysical models for the processes involved in dynamic recrystallization, such as grain boundary migration and progressive subgrain rotation, can provide independent proof for the model. One final remark on the limitation of the model refers to the single, pure phase nature of the materials considered in the field boundary hypothesis. The requirements for the model outlined in section 5.4.1 might not be fulfilled in polyphase materials or materials with impurities inhibiting grain growth.
Chapter 5

5.5 Conclusions

1. We have proposed a model that relates the grain size distribution to rheology of dynamically recrystallizing materials. The model is based on the notion that grain size tends to organize itself in the boundary between the (grain size sensitive) diffusion and (grain size insensitive) dislocation creep fields. It uses composite diffusion-dislocation flow laws for materials with a lognormal grain size distribution, uniquely characterized by the median grain size and standard deviation of the distribution in log-space.

2. The model can be used as a piezometer that relates the recrystallized grain size distribution to temperature as well as stress. This relation can be used to constrain the rheology of rock materials in nature. It differs from conventional piezometers that uniquely relate recrystallized grain size (as a single value) to flow stress and (in some cases) temperature. Instead, it implies that the standard deviation of the distribution should be taken into account if reliable paleo-stress estimates are to be obtained from piezometers.

3. Experimental data on wet polycrystalline halite, consisting of mechanical data together with detailed analysis of the grain size distribution of fully recrystallized samples, show good agreement with the formulated model. Support from experimental data for other materials is required now to assess the general applicability of the model.

4. If the model generally holds, the earlier suggestion that dynamic recrystallization in single phase, pure materials cannot result in rheological weakening and associated localization [De Bresser et al., 2001] requires examination, since the distributed nature of grain size broadens the transition zone between in deformation mechanisms.

5. The present results highlight the need to consider grain size distributions rather than treating grain size as a single value, if accurate constraints on deformation of rock materials in nature are to be obtained.