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Monitoring *in situ* stress/strain behaviour during plastic yielding in polymineralic rocks using neutron diffraction

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ABSTRACT

Attempts to use rock deformation experiments to examine the elastic and plastic behaviour of polymineralic rocks are hampered by the fact that usually only whole sample properties can be monitored as opposed to the separate contribution of each phase. To circumvent this difficulty, room-temperature, uniaxial compression experiments were performed in a neutron beam-line on a suite of calcite + halite samples with different phase volume proportions. By collecting diffraction data during loading, the elastic strain and hence stress in each phase was determined as a function of load to bulk strains of 1–2%. In all cases, the calcite behaved elastically while the halite underwent plastic yielding. During the fully elastic part of the deformation, the composite elastic properties and the within-phase stresses are well-described both by recent shear lag models and by analyses based on Eshelby's solution for the elastic field around an ellipsoidal inclusion in a homogeneous medium. After the onset of yielding, the halite *in situ* stress/total strain curve may be reconstructed using the rule of mixtures. At calcite contents of greater than 30%, the *in situ* halite response may be significantly weaker or stronger than that obtained at lesser calcite contents. The results highlight the potential that such techniques offer for developing an explicitly experimental approach for determining the influence of microstructural variables on the mechanical properties of polymineralic rocks.

1. Introduction

For the most part, rocks in the lithosphere are composed of two or more mineral phases that have different mechanical properties. The deformation behaviour of these composite materials depends on the properties of the mineral phases themselves but it is also strongly influenced by the mechanical interactions between the phases that serve to maintain strain compatibility. Thus, for example, in a two-phase material, a grain of the weaker phase that is completely surrounded by grains of the stronger phase cannot yield plastically until those neighbouring grains allow it to, and hence, its *in situ* response differs from that which it would have in a monomineralic aggregate of that phase.

Interaction effects of this kind are what it means to say that microstructure influences mechanical properties. For composite materials of coarse microstructure (i.e. where the grain size of the phases is large in comparison to the scale of a dislocation), the key microstructural variables that influence the stress distribution within the material and hence the nature of the interaction effects, include: (1) the relative proportions and spatial distributions of the phases, (2) grain shape, and (3) the presence of lattice-preferred orientations in any of the phases. A key aim of those who seek to describe the elastic and plastic behaviour of polymineralic materials is therefore to incorporate the influence of these variables into their considerations.

Numerous theoretical approaches to this matter have been developed (for extensive overviews see Nemat-Nasser and Hori, 1999; Milton, 1992). Rigorous bounds may be placed on the elastic properties of composites by assuming that in response to an applied load, each grain, irrespective of its mechanical properties, experiences the same strain thereby ensuring strain compatibility, or the same stress thereby ensuring stress equilibrium (Hill, 1952). Tighter bounds may be derived by minimizing either potential or complementary potential energy under suitable constraints (Hashin and Shtrikman, 1962a, 1963). By using the solution derived by Eshelby

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(1957) for the elastic field around an ellipsoidal inclusion embedded in an unbounded homogeneous medium, these tighter bounds may be generalized to materials in which the grains have anisotropic properties and which have grain shapes and spatial distributions that can be approximated by an ellipsoidal symmetry (Ponte Castañeda and Willis, 1995). All of these bounds can be extended to the plastic deformation case by replacing the plastically deforming nonlinear material with an equivalent linear material using appropriately defined elastic constants (Ponte Castañeda and Suquet, 1998). The most frequently used method of deriving a specific solution for the elastic and plastic properties between these bounds employs Eshelby's analysis within a self-consistent scheme (e.g. for the elastic case, Pedersen and Withers, 1992). This strategy forms the basis of the popular elastoplastic (Turner et al., 1995) and viscoplastic models (Lebensohn et al., 2007).

Greater flexibility in accounting for phase spatial distribution may be introduced by using topological parameters such as phase contiguity to describe the microstructure (Fan and Miodownik, 1992; Fan et al., 1992, 1993). Alternatively, in finite-element models, the microstructure can be described directly as a mesh with properties that vary spatially to match the elastic and plastic properties of the composite (e.g. Marin and Dawson, 1998; Han and Dawson, 2005). This descriptive flexibility, coupled with an enhanced capability in the prescription of interface and mechanical properties, has made finite-element modelling of composite mechanical behaviour increasingly popular.

These theoretical developments should not, however, obscure the fact that the modelling problem is complex and that consequently the models require validation. In particular, the impact of (1) the way in which the geometry of the microstructure is idealized, from the use of ellipsoidal inclusion-matrix geometry in Eshelby-based models to the choice of representative unit cells in finite-element models; (2) the scheme used to specify the material properties, including the choice of constitutive equation/yield criteria employed, and the linearization strategy used when modelling plastic behaviour; and (3) the way in which the behaviour at interfaces is described, all merit consideration. Evaluating the significance of these matters gives experimental work an important role, not only in validating the models but also in guiding their development.

To have greater control on the important microstructural variables, recent experimental work on geological composites has favoured the use of synthetic two-phase samples that were manufactured by sintering powders of the constituent phases mixed in the desired volume proportions. The plastic deformation behaviour of a number of two-phase systems have now been explored, including: anhydrite + halite (Price, 1982; Ross et al., 1987); calcite + halite (Jordan, 1987; Bloomfield and Covey-Crump, 1993; Marques et al., 2010); olivine + pyroxene (Hitchings et al., 1989; McDonnell et al., 2000; Ji et al., 2001); muscovite + quartz (Tullis and Wenk, 1994); calcite + quartz (Dresen et al., 1998; Rybacki et al., 2003); calcite + anhydrite (Bruhn et al., 1999; Barnhoorn et al., 2005); plagioclase + quartz (Xiao et al., 2002); plagioclase + pyroxene (Dimanov and Dresen, 2005); olivine-magnesiowüstite (Bystricky et al., 2006); muscovite + halite (Marques et al., 2011a,b). These studies highlighted the rich diversity in mechanical response that composites containing geologically important minerals exhibit. However, in conventional rock deformation tests, only whole sample properties are typically monitored during the experiment. Information about how the individual phases within the sample contribute to those bulk properties and how this might vary with microstructure is confined to inferences made after the experiment from an examination of the deformation microstructures and/or considerations based on the behaviour of the relevant phases in monomineralic aggregates. Such inferences are generally poorly constrained. What is required, particularly when the significance of microstructural variables is under investigation, is an ability to monitor the stresses and/or strains experienced by each phase within the sample during the deformation experiment.

X-ray and neutron diffraction techniques that allow non-destructive measurement of elastic strains within samples as a function of applied load (Noyan and Cohen, 1987; Hauk, 1997; Fitzpatrick and Lodini, 2003; Hutchings et al., 2005) offer a means of monitoring such *in situ* stresses and strains during a deformation experiment. For these experiments, the sample is held at constant load within an X-ray or neutron beam-line while diffraction data are collected. By collecting diffraction data at several different applied loads, the change in lattice spacings, and hence elastic strain, in each diffracting phase within the sample can be determined as a function of load. If the elastic properties of the constituent phases are known *a priori*, then the elastic strains may be converted into stresses, thereby allowing the approach to be applied to plastic as well as fully elastic deformation.

Diffraction data acquired in this way are currently applied to a wide range of mechanical behaviour and property characterization problems in the engineering sciences and, increasingly, also within the Earth sciences (Covey-Crump et al., 2003; Wenk, 2006; Covey-Crump and Schofield, 2009; Liang et al., 2009). To avoid the problems associated with interpreting mechanical data obtained from very small samples or from near-surface regions of larger samples, the X-rays or neutrons must be sufficiently penetrating to collect data from within sample interiors that are at least a few cubic centimetres in size (Schofield et al., 2003). Consequently, most work to date on samples of similar size and shape to those used in conventional rock deformation experiments (typically, right circular cylinders about 10 mm in diameter and 25 mm long) has been performed using neutrons.

Most deformation testing of this kind using polyphase materials has been conducted on engineering composites (e.g. Carter and Bourke, 2000; Oliver et al., 2004; Daymond et al., 2005). These studies have shown that the experimental data can be successfully used to interrogate the predictions of individual phase behaviour made by finite-element and elastoplastic self-consistent modelling. In the present study, we develop and extend this work to geological composites by reporting the results of deformation experiments on synthetic calcite + halite aggregates at the ISIS neutron facility, Rutherford Appleton Laboratory, U.K. Under the deformation conditions employed, the halite yielded plastically while the calcite remained fully elastic. The samples were fabricated such that phases were uniformly intermixed and any grain-shape fabrics and lattice-preferred orientations were minimized. By recovering the *in situ* behaviour of the two phases in this microstructurally simple case, our aim is to highlight the potential that these techniques offer for developing an explicitly experimental approach to examine influences on the flow properties of polymineralic rocks with more interesting microstructures. Such an approach is one that can complement and inform theoretical developments rather than being led by them. It is also one that can be used to interrogate recent advances in the interpretation of the mechanical significance of the microstructures of naturally deformed polymineralic rocks (e.g. Herwegh et al., 2011).

2. The nature of the experimental task

A common starting point for analysing the results obtained from axial loading experiments on two-phase composites of coarse microstructure, is the so-called rule of mixtures:

$$\sigma_{\text{agg}} = \phi_1\sigma_1 + \phi_2\sigma_2 \tag{1}$$