

Experimental approaches to the study of deformation/metamorphism relationships

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Abstract

Rock deformation and metamorphism can interact at the mechanistic level in the following ways: (a) facilitation of cataclasis through the release of high-pressure fluid during dehydration and decarbonation reactions; (b) facilitation of intracrystalline plasticity through the stresses induced during solid-state volume changes; (c) enhanced deformability through the transient existence of fine-grained reaction products; (d) modification of chemical potential gradients driving diffusion if a reaction can occur along the diffusion path; (f) changes in the resistance to intracrystalline plasticity through the effect of reaction-induced changes, pore fluid pressure and chemistry on point defect chemistry of the solid phases.

Examples of experimental studies of each of these types of interaction are described. Special experimental problems arise through: (i) the effects of solid phase and pore space volume changes, and their effects on pore fluid pressure and measured strain; (ii) the effects of such microstructural changes on the determination of flow law parameters; and (iii) in many instances the need for very long duration deformation experiments if reaction kinetics are sluggish.

There is an outstanding need for experimental studies of the effects of non-hydrostatic stress on the conditions for the onset of metamorphic reactions and phase transformations, as a basis for understanding some classes of deformation/metamorphism interaction. However, it is emphasized that the threefold classification of rock deformation mechanisms into cataclastic, crystal-plastic, and diffusive mass transfer processes, established from the study of deformation of monomineralic rocks, forms an essential framework for the understanding of deformation/metamorphism inter-relationships.

KEYWORDS: deformation, metamorphism, cataclasis, intracrystalline plasticity, chemical potential gradients, diffusion.

Introduction

MOST geologically oriented experimental rock mechanics over the past fifty years has involved either monomineralic rocks or polymineralic rocks deformed under conditions which do not favour metamorphic transformations. Out of this work has emerged an appreciation that rocks may deform by three fundamental deformation mechanisms: cataclasis, intracrystalline plasticity, and flow by diffusive mass transfer.

Experimental metamorphic petrology has concentrated on programmes aimed at producing equilibrium assemblages under hydrostatic conditions. In contrast, many natural regionally metamorphosed rocks were subjected to non-hydrostatic stress fields under pressure/temperature conditions favouring metamorphic transformations, and there is growing interest in the ways in which the rock

deformation and metamorphism interact in mechanistic terms.

In naturally deformed and metamorphosed rocks, any evidence of this interaction is often obscured. This happens through the destruction of deformation-induced microstructures as a result of the development of mineralogically stable assemblages and the attainment of microstructural equilibrium under hydrostatic stress conditions at the temperature peak of metamorphism, which is often after most strain has been accumulated. Schistosity in metamorphic tectonites is frequently a coarsened mimetic growth feature superimposed on earlier, strain-induced microstructures. Experimental studies are required to fill in such gaps in our understanding of deformation/metamorphism relationships. The purpose of this paper is to illustrate some of the experimental approaches which may be adopted. These are illustrated mainly

with examples from current and recent studies made in our laboratory.

Categories of deformation/metamorphism interactions causing modification of deformability

The *kinetics* of metamorphic transformations may be affected by mechanical strain, *mineral equilibria* may be affected by deformation, and the *deformability* of rocks may be modified by mineral transformations. The most recent work dealing with the first of these topics (Snow and Yund, 1987) provides a review of previous literature on the subject. Although there have been various speculations in the literature on the significant perturbation of mineral equilibria through the effects of deformation, no systematic effects have been demonstrated, especially experimentally. In this article we will consider the third of these topics, namely the ways in which metamorphic transformations may influence deformability. Brodie and Rutter (1985) categorised the main mechanistic interactions in the following way: (a) facilitation of deformation by cataclasis through the release of pressurized fluid during dehydration or decarbonation reactions; (b) enhancement of deformability by intracrystalline plasticity, arising from stresses induced through volume changes arising from solid-solid transformations; (c) facilitation of grain-size-sensitive deformation mechanisms (e.g. diffusion-accommodated grain boundary sliding) as a result of the transient existence of fine-grained reaction products; (d) effects of variations in the magnitudes of stress-induced chemical potential gradients, which drive diffusion creep, arising from phase changes along the diffusion path; (e) modification of resistance to intracrystalline plasticity arising from changes in equilibrium point-defect chemistry as a result of changes in pore fluid chemistry.

The term 'Transformation Modified Deformability' used to describe the above group of effects is preferred to the frequently employed term 'Reaction Enhanced Ductility' (e.g. White and Knipe, 1978) and similar terms, in recognition of the fact that metamorphic transformations may not always make rocks weaker, and that transformations may not always make rocks more ductile. Ductility implies a capacity for the accommodation of a large, distributed strain, yet metamorphic transformations may modify the properties of rocks in such a way that deformation may become intensely localized into a narrow shear zone, rather than being uniformly distributed. The mineral products of a metamorphic reaction may be intrinsically stronger or weaker than the assemblage of the parent rock, provided the comparison is carried out

at the same grain size, but we will not consider this aspect further in this paper. Here we will be concerned only with the effects that arise during the course of the metamorphic transformation itself.

Examples of experimental studies

A number of experimental studies have been made of the mechanical properties of rocks under conditions favouring mineral reactions, and these illustrate most of the above types of interaction. Some of our more recent data are described in more detail.

Heard and Rubey (1966) and Murrell and Ismail (1976) studied the effect of dehydration reactions on the mechanical properties of gypsum. These tests were carried out at high strain-rates (typically less than one day duration), and under fully drained (specimen pores vented to atmosphere) or undrained conditions (specimen not vented). In the latter case, evolution of water during the dehydration reaction caused pore pressure to rise until it was equal to the applied confining pressure. In accordance with the law of effective stress, this lowered the effective confining pressure and caused weakening and embrittlement.

A similar pattern of behaviour was observed by Raleigh and Paterson (1955) and Murrell and Ismail (1976) in the deformation of serpentinite under conditions favouring dehydration. Comparable effects can also be observed in the mechanical properties of clay rocks, particularly in the case of swelling clays such as montmorillonite and vermiculite (e.g. Summers and Byerlee, 1977). Riecker and Rooney (1969) and Murrell and Ismail (1976) found similar effects in the experimental deformation and dehydration of amphibole and chlorite, respectively. All of these examples illustrate interrelationships of type (a) above.

The experimental and theoretical basis for transformation-enhanced plasticity [(b) above] comes mainly from metallurgy and materials science (e.g. Greenwood and Johnston, 1965; Poirier, 1982). Enhancement of deformability arises from the stresses induced in a polycrystal resulting from volume changes during solid-solid transformations. Gordon (1971) and Sammis and Dein (1974) described experiments on various ionic salts which were regarded as analogues for processes occurring deep in the mantle.

It is widely recognized that a sufficient degree of reduction of grain-size by tectonic or metamorphic processes may cause a switch of dominant deformation mechanism to one which is grain-size sensitive. Such processes include Coble creep and Nabarro-Herring creep, diffusion-accommodated grain-boundary sliding, cyclic plastic deformation and

dynamic recrystallization, and enhanced plasticity through diffusion of weakness-inducing point-defects from the pore fluid (e.g. Kirby, 1985). These processes are each characterized by different constitutive flow laws, and all exhibit enhancement of deformability through reduction of grain size.

Grain-size reduction may be brought about by cataclastic granulation, dynamic recrystallization during plastic flow, or through the formation of transiently fine-grained reaction products during metamorphism [(c) above]. Natural examples of the latter process facilitating deformability have been described by Rubie (1983) and Brodie and Rutter (1987). We have also been able to demonstrate this process during the experimental deformation of serpentinite at low strain rates during 'syntectonic' dehydration under controlled pore water pressure conditions (Brodie and Rutter, 1987; Rutter and Brodie, 1987*a* and *b*).

In these experiments, serpentinite cylinders were sawcut at 35° to the cylinder axis and compressed axially at temperatures ranging from 300 to 600 °C and at effective confining pressures ranging between 100 and 275 MPa. The sawcut forces the development of a shear zone in a well controlled planar orientation. A small hole drilled through the upper part of the specimen ensures that the pore fluid pressure in the vicinity of the sliding zone is known. Constant axial displacement rate, constant stress (creep) and constant strain (stress relaxation) testing techniques were used in these experiments. Activation enthalpies for flow were determined by temperature cycling during creep tests.

At temperatures below those required for the onset of the dehydration reaction, the strength of the serpentinite was almost independent of large changes in deformation rate (Fig. 1). Above the temperature necessary for breakdown of serpentinite to olivine plus talc plus water, the strength dropped rapidly as the shear displacement rate along the sawcut was lowered below 10^{-6} mm sec⁻¹ (Fig. 1). The rate of reduction of strength corresponds to linear viscous behaviour, and is independent of effective confining pressure over the range investigated.

SEM and TEM investigation revealed that deformation was accomplished by sliding along the sawcut, which became lined with a 5 to 10 µm thick layer of ultrafine-grained olivine (grain-size about 0.1 µm). Samples which were loaded without being pre-cut behaved in the same way, via the formation of a more irregular shear zone lined with ultrafine-grained olivine. Away from the shear zone, dehydration proceeds by the formation of larger (about 5 µm diameter) crystals of olivine, scattered throughout the volume of the sample. Formation of

these grains does not appear to be accompanied by any evidence of deformation except local collapse of the serpentinite matrix around the olivine nucleus. Volume changes associated with the amounts of dehydration observed cannot account for the observed total strains.

The mechanical properties and microstructures produced in these experiments are most consistent with deformation of the olivine aggregate in the shear zone by diffusion-accommodated grain-boundary sliding. This is believed to be possible at the relatively low temperatures of these experiments only because of the extremely fine grain size of the olivine formed in the shear zone, and provides an example of process (c) above. Taking into account the differences in temperature (via the activation enthalpy) and grain-size, Rutter and Brodie (1987*a*) argue that these data are consistent with the study of grain-size sensitive flow of olivine made by Karato *et al.* (1986), made using hot-pressed aggregates of fine-grained olivine.

In the shear zones, the generation and maintenance of olivine of fine grain-size during the progressive dehydration and deformation must depend upon the fact that the zone is being continuously deformed. In nature, the relaxation of the applied stress will presumably lead to static grain growth, provided the temperature is sufficiently high. It is for this reason that we regard the enhancement of deformability in this way as being a transient phenomenon.

Examples of naturally deformed rocks are frequently found in which microstructural evidence points to deformation by diffusive mass transfer, but in which the phases precipitated in relatively less stressed interfaces are different from those diffused away from relatively stressed interfaces. In such cases the deformation mechanism provides the pathway through which a metamorphic reaction is accomplished, such that reactants and products become separated. Equilibrium between solid phases exists only at one point along the diffusion path. This type of process has been termed 'in-congruent pressure solution' by Beach (1982) and corresponds to interaction type (d) above.

Because the reactants and products possess different molar volumes, it is to be expected that the chemical potential gradient which drives the diffusive mass transfer (which in turn depends on the gradient of the product of intergranular normal stress with molar volume) will be modified, leading to a change in creep rate. Rutter *et al.* (1985) argued that this effect on the creep rate will be relatively small, and illustrated it by means of deformation experiments carried out on basalt under conditions favouring hydration reactions. In these experi-

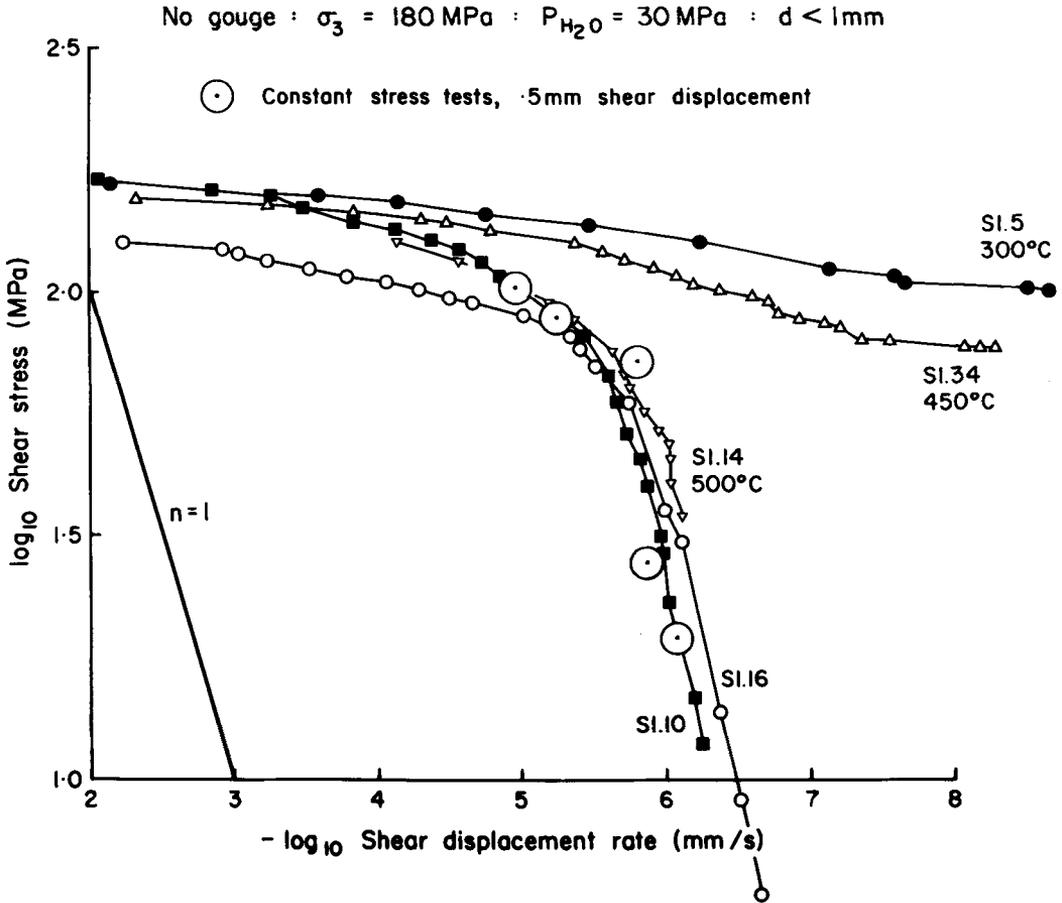


FIG. 1. Typical experimental results for the deformation of serpentinite by shearing along a sawcut made at 35° to the specimen axis. Behaviour under conditions favouring the dehydration reaction to olivine + talc + water (500°C) may be compared to that at lower temperature conditions such that dehydration does not occur. In each case total confining pressure = 180 MPa and pore water pressure = 30 MPa . The linked points show the results of stress relaxation tests at total shear displacements (d) less than 1 mm , whilst the large circles show the results of constant stress (creep) tests to larger shear displacements. At 500°C at low deformation rates rapid strength loss occurs at a rate corresponding to Newtonian viscous behaviour (a reference line of unit slope is drawn for comparison). This is attributed to the formation of ultrafine-grained olivine along the sliding surface. (After Rutter and Brodie, 1987b.)

ments, oriented overgrowths of Mg-gedrite were produced on pre-existing grains of clinopyroxene and feldspar, in the same way that oriented 'beards' of fibrous phases are often developed in extended grain boundaries in metabasic rocks subjected to syntectonic retrogressive metamorphism. In this type of deformation/metamorphism inter-relationship, the textural effects are probably more dramatic than the effect on mechanical properties.

It has been established for some silicate minerals, notably quartz and olivine (e.g. Tullis *et al.*, 1979; Hobbs, 1981, 1983; Kronenberg and Tullis, 1984; Kirby, 1984; Chopra and Paterson, 1984, Ricoult

and Kohlstedt, 1985), that the resistance to intracrystalline plastic deformation is sensitive to the composition of the pore fluid. This is thought to be in response to the fact that the equilibrium point-defect chemistry of minerals depends on temperature and the activity of the various chemical components of the pore fluid. A change in temperature, pore fluid composition or pressure results in a diffusive exchange between the pore fluid and the solid in order to restore the chemical equilibrium. Because plastic deformation kinetics may involve interactions between dislocations and point defects, the resistance to plastic flow can be affected by

changes in pore fluid pressure and chemistry brought about through metamorphic transformations [(e) above].

When water itself appears to provide the weakening defect, the phenomenon is known as 'hydrolytic weakening'. Current views on the subject are reviewed by Paterson (1987).

Design and interpretation of experiments

The experience gained from the above studies points to certain important principles in the design and interpretation of experiments aimed at elucidating the nature of deformation/metamorphism inter-relationships.

Microstructural changes. Mineral transformations taking place during a deformation experiment necessarily mean that the microstructure of the sample must change. In any constitutive law for deformation, i.e. an equation relating strain rate to stress and temperature, there is always an additional dependence on aspects of the microstructure of the material. What is perceived as steady-state flow (deformation at constant strain rate, constant stress and constant temperature) is strictly only possible if the microstructure also remains constant. Microstructural changes during deformation must result either in strain-hardening or softening.

Even in dislocation creep, the dislocation substructure evolves with strain. There may be sub-grain formation and dynamic recrystallization, and a grain shape fabric and crystallographic preferred orientation generally form progressively. In this case the effect on the flow stress is often small over the strain intervals employed in experimental deformation studies. Over larger, natural strains, however, the effects may not be negligible. Perhaps only when flow is by diffusion-accommodated grain-boundary sliding is it possible to have flow at truly constant structure, with the consequent possibility of stable flow to large strains at constant stress.

In contrast, the microstructural changes accompanying syntectonic mineral transformations may often exert a much greater effect on the flow stress over even small creep strains. In these circumstances it may be impossible to assign flow laws in the familiar forms that are frequently applied to the apparently steady flow of monomineralic rocks. In general, therefore, a determination of the relationship between strain and strain-rate must also be made (e.g. Rutter *et al.*, 1985).

When rocks deform by steady-state flow, or when microstructure varies only slowly with strain, the sensitivity of strain rate to stress can be determined experimentally using separate runs at different constant strain rate to a given level of

strain, at which point the stress is measured. Alternatively, separate constant stress (creep) tests may be carried out or the stress relaxation technique may be used. The sensitivity of strain rate to temperature (expressed by the activation enthalpy for flow) can also be determined from separate constant stress or constant strain-rate tests at different temperatures. When microstructure is strongly dependent on strain, however, stress or strain-rate cycling methods, or the temperature transient technique, must be used to extract the above parameters of the flow law.

Volume changes. Steady-state flow of rocks takes place at constant volume, but mineral transformations are generally accompanied by a volume change. If this is substantial and negative (volume reduction), in an experiment part of the volume change will be sensed as a contribution to axial strain (unless volume strain is measured separately). Care must therefore be taken to separate out the volume change effect in any study aimed at determining the effect of the mineral transformation on deformability (e.g. Gordon, 1971; Sammis and Dein, 1974).

Pore fluid pressure. Many mineral reactions involve water as a reactant, or are catalysed by water. Experimental studies of mineral equilibria have always been determined with $P_{\text{H}_2\text{O}} = P_{\text{total}}$ or with the activity of water reduced by dilution with argon or a dissolved salt (e.g. Greenwood, 1961; Barnes and Ernst, 1963). In such cases there is no effective pressure pushing the grains of the solid together. Rock deformation, however, generally takes place with pore pressure less than the least principal stress, so that there is almost always an *effective* pressure on the rock.

The mechanical behaviour of rocks is extremely sensitive to effective confining pressure, even at high temperatures if the pore pressure is high. Little is known of mineral equilibria under conditions of effective confining pressure greater than zero, and the outcome of theoretical discussions depends upon what assumptions are made regarding the state of water in intergranular regions when the rock is under effective pressure (e.g. Bruton and Helgeson, 1983; Rutter, 1983). However, from our studies of the dehydration of serpentinite, we have established that under conditions of effective pressure, the breakdown of serpentinite to forsterite plus talc plus water begins at substantially lower temperatures than under $P_{\text{H}_2\text{O}} = P_{\text{total}}$ conditions. Preliminary results are shown in Fig. 2, from which it is apparent that under lowered water pressure conditions, the dehydration reaction is favoured by an *increase* in total pressure. It may therefore be speculated that under a state of applied deviatoric stress the more highly stressed grain-boundary

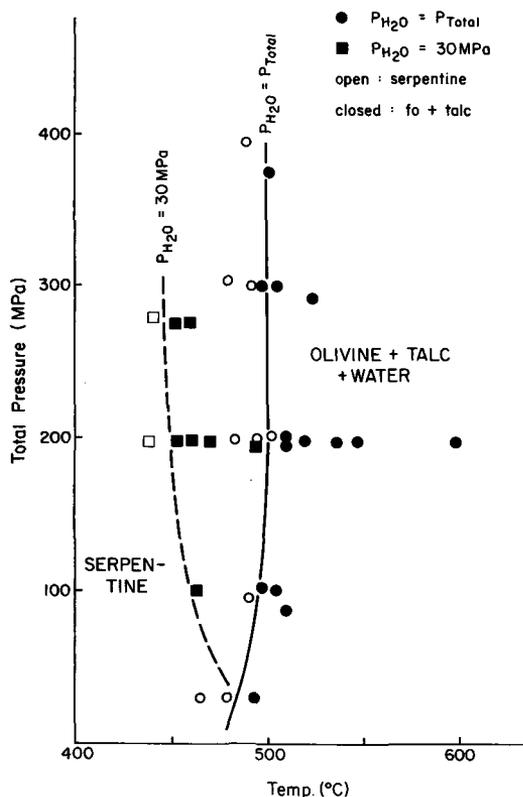


FIG. 2. Preliminary results of experiments performed to investigate the effect of pore water pressure less than total applied (solid-solid) pressure, on the onset of the serpentine breakdown to olivine + talc + water, determined using scanning electron microscopy. The experiments were carried out in a rock deformation machine on intact rock cores so that a true effective pressure could be applied. The displacement of the $P_{H_2O} = 30$ Mpa curve to lower temperatures implies that the effective water activity at the reaction sites is less than that which would arise if water pressure were equal to the total pressure, but the negative slope additionally implies that the excess of total pressure over the pore pressure influences the behaviour. (After Rutter and Brodie, 1987b.)

orientations will be more favoured sites for the dehydration reaction to proceed.

Burnley and Kirby (1982) and Durham *et al.* (1983) report experimental data on the breakdown of tremolite to diopside plus talc, and the ice I_h to ice II transformation under differential stress, from which they conclude that elevation of the mean stress during deformation brings about the mineral transformation, resulting in a change in the deformability of the rock, although the processes involved have not yet been fully clarified.

A great deal remains to be done in the investigation of the role of effective pressure on the conditions of onset of dehydration and decarbonation reactions, their kinetics, and of the textures and microstructures produced. The techniques of experimental rock deformation under conditions of controlled pore pressure are well suited to this type of study.

Pore volume. The sensitivity of the mechanical properties of rocks to variations in pore fluid pressure is of particular importance in view of the variations in pore volume which are likely to arise during a mineral transformation. If intracrystalline plasticity becomes enhanced during a metamorphic transformation, porosity will tend to collapse, leading to an increase in pore fluid pressure. The reverse will happen if porosity is created as a result of a solid phase volume decrease. The resultant fluctuations in effective confining pressure may induce changes in deformability unless rock permeability is sufficiently high, and/or the time-scale of events is sufficiently long for continuous re-equilibration of the pore pressure from the surrounding rock volume in response to such transients.

The problem is of especial importance in experimental studies owing to the short timescale. If communication is effectively lost between the pores of the sample and the external pore pressure system so that the effective pore pressure is not known, mechanical effects attributable to 'dilatancy hardening' or 'compaction softening' (Brace and Martin, 1968; Rutter, 1972, 1974) may arise and be difficult to interpret. For this reason steps must be taken in experiments to ensure that pore pressure is applied to that part of the sample within which the deformation is concentrated. Rutter *et al.* (1985), Brodie and Rutter (1987) and Rutter and Brodie (1987b) used hollow samples to increase the area over which the pore pressure was applied, coupled with the use of an inclined sawcut (which was sometimes filled with crushed mineral or rock powder) to force deformation into a restricted zone and to ensure that some porosity was present. A price must be paid for this, however, because collapse of this porosity through plasticity or filling by reaction products contributes to the measured apparent strain and modifies the microstructure during straining. If flow is by diffusion creep, the increase in effective grain contact area results in a dramatic strain-hardening effect (Rutter *et al.*, 1985).

In studies of grain-size-sensitive flow of monomineralic aggregates it makes sense to prepare synthetic samples of controlled porosity and grain-size by hot-pressing close to the melting temperature (e.g. Karato *et al.*, 1986). The attainment of

steady flow at high temperatures in such materials may be limited only by microstructural changes arising from grain coarsening. However, high-temperature sintering has the disadvantage that it may force the development of 'dry' or disordered structures, or the formation of grain boundaries contaminated by small quantities of quenched melt. It may prove possible to produce effective sintering at lower temperatures through the use of a controlled pore water pressure. Finally, the use of high-temperature sintering techniques may not always be possible for polymineralic aggregates, depending on the temperatures at which reactions take place.

Except in rare cases, it seems unlikely that satisfactory synthetic aggregates of coarse-grained aggregates of minerals for mechanical testing can be produced by hot-pressing and sintering techniques, owing to the difficulty in eliminating most of the pore spaces and to the intracrystalline damage, which might not be easily removed, done to the grains.

Time duration of experiments. Most rock deformation experiments at high temperatures are not carried out over long time periods (i.e. several weeks). Gas-pressurized rock-testing machines are not usually designed for operation for such periods. Solid-medium testing machines are capable of long periods of operation, but are best suited to measurements on very strong rocks at pressures of the order of 1 GPa and above and are not normally equipped for testing under controlled pore water pressure.

For our studies of deformation/metamorphism relationships we have used units of a fluid-medium testing machine specifically designed for this purpose. The equipment is described by Rutter *et al.* (1985) and can operate at temperatures to 800 °C, confining pressures to 400 MPa and with controlled pore fluid pressure. Constant strain-rate testing to 10^{-8} s^{-1} , true constant stress, and stress relaxation testing modes can be used. Temperature-transient tests under constant applied stress can be carried out under computer control.

Conclusions

Despite the textural and mechanical complexities of deformation accompanied by metamorphic transformations, the threefold classification of rock deformation mechanisms into cataclastic, crystal-plastic and diffusive mass transfer, established from the study of monomineralic rocks, forms a useful framework for the interpretation of syn-metamorphic deformation. There are special experimental problems, however, in studying such effects, arising from solid and pore-space volume

changes and their effects on pore pressure, and the need for long duration deformation experiments if transformations are sluggish. Microstructural changes may mean that steady-state flow is impossible, and this in turn poses practical problems for the determination of flow law parameters. There is an outstanding need for the study of mineral transformations and reactions under conditions of pore fluid pressure less than the least principal stress.

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