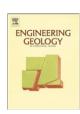


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Transformation plasticity and the effect of temperature on the mechanical behaviour of Hawkesbury sandstone at atmospheric pressure

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ABSTRACT

Uniaxial Compressive Strength (UCS) testing of Hawkesbury sandstone was carried out at various temperatures between 25 and 950 °C to explore the mechanical response of the sandstone to significant changes in temperature, as expected for enhanced geothermal energy systems, nuclear waste disposal and underground coal gasification. The UCS testing results demonstrate a mechanical dependence on temperature whereby the compressive strength and elastic modulus for the sandstone increases with increasing temperature for temperatures less than c. 500 °C and decreases with increasing temperature for temperatures greater than c. 500 °C. X-ray diffraction analyses performed on material from the failed 25 and 950 °C specimens highlights a distinct difference in mineralogy between the two specimens that has been related to mineralogical changes in the sandstone cement with heating. Progressive dehydroxylisation of kaolinite in the sandstone cement at temperatures beyond 500 °C appears to have enabled transformation plasticity, explaining the weakening and softening of the sandstone that was observed with increasing temperature beyond 500 °C. Transformation plasticity is mineralogy dependent and thus its influence on mechanical behaviour of rock will vary with bulk mineralogy and the relative distribution of mineral species. Comparison of the results from the UCS testing to those obtained from similar experimental work carried out on different sandstone units highlights variability in the response of rock to heating. The study provides a word of caution regarding the need for accurate understanding of the influence of temperature on the mechanical behaviour of the specific rock unit considered for a given elevated temperature engineering application. Such understanding requires consideration of the geological history of the rock in addition to its physical properties and mineralogy. © 2012 Elsevier B.V. All rights reserved.

1. Introduction

An appreciation of the influence of pressure and temperature on the mechanical behaviour of rock has played a vital role in the efforts of geologists and geophysicists to understand Earth structure, rock rheology and the deformation response of the Earth's crust and mantle to tectonic forces. In recent years, the search for sustainable energy alternatives to the burning of fossil fuels has stimulated further research into the mechanical behaviour of rock at elevated pressure and temperature, with a view to understanding the engineering behaviour of high-temperature environments below the Earth's surface. The mechanical characteristics of rocks can vary significantly with an increase in temperature to values expected for conventional high-level radioactive waste disposal (up to 250 °C) and conventional or hot fractured rock geothermal energy systems (up to 300 °C). The influence of temperature on the mechanical behaviour of rocks becomes yet more significant for engineering applications for which temperatures approach the melting point of rock, as in underground coal gasification (Minchener, 2005; Shoko et al., 2006; Stiegel and Ramezan, 2006), proposals for deep geological burial of high-grade nuclear waste (Gibb, 1999; Logan, 1974) and volcano flank stability (Heap et al., 2011; Voight, 2000; Voight and Elsworth, 1997; Watters et al., 2005). For engineering applications that experience significant heat input or extraction, the chief time-dependent variable will be temperature. Thus, to ensure optimal performance of these in-ground alternative energy solutions in the long term, a firm understanding of the influence of temperature on the mechanical behaviour of rock is required.

Rock testing for mechanical characterisation in engineering is typically carried out at room temperature. Direct use of mechanical properties derived from testing at room temperature to high-temperature environments would constitute a significant oversight in the engineering process. Models that account for the influence of temperature on rock behaviour must be applied before mechanical properties can be assigned to specific rock packages in engineering design for high-temperature systems.

We report on Uniaxial Compressive Strength (UCS) tests carried out on sandstone at various temperatures between 25 and 950 °C. The results of quantitative X-ray diffraction (XRD) analyses of failed samples are presented and mineralogical changes observed with heating are related to thermally-induced changes in observed mechanical behaviour. The results of the testing are compared to results obtained for similar work carried out on similar rock types. The influence of rock character

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and geological history on the mechanical response of rock to heating are considered and the specificity of the models required for effective engineering of rock units for high temperature applications is explored.

1.1. Previous work

Seismologists have long recognised variation in the manner by which rocks deform with increasing depth in the Earth's crust. The relative scarcity of earthquakes below certain depths has been related to a marked, pressure- and temperature-governed transition in rock deformation mode with depth in the crust (Sibson, 1977, 1982). Early experimental work on the influence of confining pressure on deformation mechanisms for rock demonstrated a distinct correlation between confining pressure and deformation mechanism (see Byerlee, 1968; Griggs, 1936; Mogi, 1966; Paterson, 1958). Orowan (1960) theorised that this transition in deformation behaviour with increasing crustal depth was related to inhibition of crack growth and friction-dominated (elastic) deformation mechanisms at substantial confining pressures and an associated transition to deformation by ductile mechanisms involving dislocation and/or recrystallisation. Rutter (1986) and Tullis and Yund (1987) identified a possible interstitial stage, between entirely brittle and entirely plastic deformation, involving stable crack growth and pressure-sensitive plastic deformation that proceeds by intergranular (cataclastic) flow.

Experimental work that considered the influence of temperature in addition to pressure has demonstrated that temperature will influence the pressures at which the brittle-ductile transition occurs (see Griggs et al., 1960; Heard, 1960; Tullis and Yund, 1977; Wong, 1982). The seismogenic zone extends to depths well beyond c. 10 km and thus ductile behaviour related to suppression of elastic failure mechanisms by confining stress is not of major concern for high-temperature deep Earth engineering applications (e.g. nuclear waste disposal, underground coal gasification, enhanced geothermal energy), which are limited to depths that can be easily accessed by drilling. However, studies conducted at elastic-field confining pressures have shown that temperature will also influence the brittle mechanical response of rock (see Heuze, 1983; Lockner, 1995 and references therein). For engineering applications that involve in situ variation in rock temperature at constant depth, such as heating of rock in nuclear waste disposal, underground coal gasification, heat mining in enhanced geothermal energy and volcano flank stability an understanding of the influence of temperature on the brittle response of rock is a major issue of interest. In this study, we focus on the influence of temperature on rock mechanics at low confining pressures (relevant to the elastic field).

Work carried out on the influence of temperature at a fixed confining stress within the elastic field has yielded some interesting results. For some rocks, a strengthening effect with increasing temperature has been observed (Duclos and Paquet, 1991; Rao et al., 2007). This may be related to dissipation of stress concentration at crack tips by increased plasticity (Duclos and Paquet, 1991), or closure of pre-existing cracks with thermal expansion (Dmitriyev et al., 1969). Beyond some critical temperature, rocks become weaker with increasing temperature (see Paterson and Wong, 2005 and references therein). The mechanisms responsible for this weakening of intact rock with increasing temperature (at significant temperature) have been related to thermal cracking and partial melting (Paterson and Wong, 2005) and brittle creep (Heap et al., 2009). In rock types that display strengthening with increasing temperature (at low temperatures), the temperature of the transition from strengthening (with increasing temperature) to weakening (with increasing temperature) signifies the crossover in influence of individual weakening/strengthening mechanisms. In the early literature, the majority of studies on the influence of temperature on mechanical behaviour of rock reported on the behaviour of granite during progressive melting, at temperatures ranging from 800 to 1100 °C (see Paquet et al., 1981; Rutter and Neumann, 1995; van der Molen and Paterson, 1979). Heuze (1983) and Lockner (1995) have provided reviews of information available on the topic of the influence of temperature on the mechanical response of intact rock at low confining pressures.

Tullis and Yund (1977) reported on an experimental study into the stress-strain response of granite at temperatures between 25 and 1000 °C. Their data showed a correlation between the mechanical properties of various granites and the temperature at which testing to obtain those properties was performed, whereby the compressive strength and elastic modulus decreased with increasing temperature. Duclos and Paquet (1991) reported on a series of UCS tests on partially glassy, partially crystalline basalts at temperatures between 25 and 1000 °C. They found that compressive strength increased with increasing temperature for experiments performed at temperatures between 25 and 700 °C, but that compressive strength decreased significantly with increasing temperature for experiments at temperatures greater than 750 °C. Xu et al. (2008, 2009) performed a study on the mechanical effects of temperature on granite that involved UCS testing at temperatures between 25 and 1200 °C. Their results showed that the mechanical properties of their granite varied only slightly with increasing temperature for experiments carried out at temperatures between 25 and 800 °C, but that both compressive strength and elastic modulus decreased considerably with increasing temperature for experiments at temperatures greater than 800 °C. The results of UCS tests carried out on limestone at temperatures ranging from 25 to 800 °C by Mao et al. (2009) showed a significant decrease in compressive strength and elastic modulus with increasing temperature for tests carried out at temperatures greater than 700 °C, but no significant variation in mechanical properties with increasing temperature for experiments carried out at temperatures less than 700 °C. Zhang et al. (2009) carried out UCS testing on marble at temperatures between 25 and 800 °C. They found that both compressive strength and elastic modulus displayed a general decrease with increasing temperature for the entire range of testing temperatures. Fig. 1a illustrates the manner by which UCS varies with temperature for some crystalline

Wu et al. (2005) considered the influence of temperature on the mechanical behaviour of non-crystalline (sedimentary) rocks. On the results of UCS tests carried out on sandstone at temperatures between 25 and 600 °C, they found that compressive strength decreased significantly with increasing testing temperature for tests carried out at temperatures higher than 400 °C, but that temperature did not affect compressive strength for temperatures lower than 400 °C. Elastic modulus was observed to decrease with increasing testing temperature for the entire temperature range investigated by Wu et al. (2005). Rao et al. (2007) observed strengthening and stiffening of sandstone with increasing test temperature in the results of uniaxial tests carried out at temperatures between 25 and 250 °C. A dramatic reversal of this trend was observed for temperatures greater than 250 °C, up to the maximum testing temperature of 300 °C, in the UCS experimental results of Rao et al. (2007). Zhang et al. (2009) carried out UCS tests on sandstone at temperatures between 25 and 800 °C. They found that compressive strength decreased with increasing temperature for experiments carried out at temperatures from 25 to 200 °C, then increased with increasing temperature for experiments carried out at temperatures from 200 to 600 °C. The elastic modulus values obtained from the same tests were unaffected by temperature for temperatures in the range 25 to 600 °C. However, values of compressive strength and elastic modulus were both observed to decrease significantly with increasing temperature in the results of the tests carried out by Zhang et al. (2009) at temperatures greater than 600 °C. Fig. 1b summarises variation in UCS with test temperature for some studies from the literature that considered sedimentary rock.

A general increase in 'strain to failure' was observed to accompany increases in test temperature for the UCS studies from the literature. In all cases, the increase in strain to failure with increasing testing temperature becomes exceedingly obvious at temperatures above which both compressive strength and elastic modulus decrease significantly with increasing temperature. The temperature marking the sudden transition in mechanical response (for UCS testing) can be viewed as

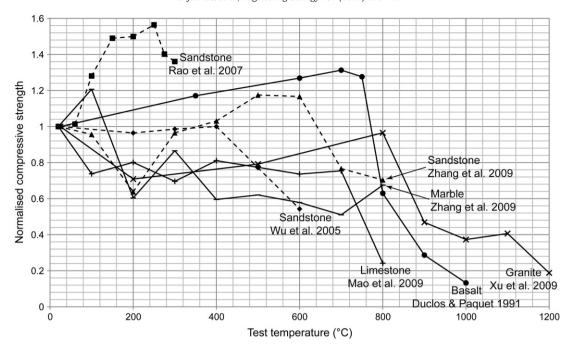


Fig. 1. Normalised compressive strength v. test temperature curves from UCS experiments reported in the literature. Continuous lines indicate crystalline samples, broken lines indicate non-crystalline (sedimentary) samples.

the temperature at which plasticity first occurs in the direction of increasing temperature. Plasticity was observed at temperatures as low as 100 °C for studies on marble (Zhang et al., 2009), between 250 and 500 °C for studies on sandstone (Rao et al., 2007; Wu et al., 2005; Zhang et al., 2009), 700 °C for studies on limestone (Mao et al., 2009); 750 °C for studies on basalt (Duclos and Paquet, 1991), and 800 °C for studies on granite (Xu et al., 2008, 2009).

2. Material and methods

The laboratory component of the study involved UCS testing on cylindrical sandstone specimens at 25, 200, 400, 600, 800 and 950 °C. The temperatures considered cover the range of temperature conditions that are likely to be encountered to significant engineering depths (of c. 5 km or more) in regions with extremely elevated geothermal gradients (such as those for which geothermal energy is an attractive proposition). The maximum value of temperature considered (i.e. 950 °C) approaches maximum relevant temperatures for elastic rock deformation in underground coal gasification and deep geological burial of high-grade nuclear waste.

2.1. Sandstone origin, mineralogy and engineering properties

All testing was carried out on homogeneous medium-grained sand-stone specimens prepared from a single, small-scale bulk sandstone sample obtained from a quarry in the Hawkesbury Sandstone at Gosford, New South Wales, Australia. The small scale of the single block from which the specimens were cored ensured maximum specimen uniformity and repeatability in the testing results. The Hawkesbury Sandstone is typically massive and consists predominantly of sub-angular quartz grains (Pells, 1977). It is medium to coarse grained and moderately to well graded (Standard, 1969). It is of Triassic age and is interpreted to comprise stacked channel deposits of a large-scale braided fluvial system that developed within the broader Sydney Basin system (Herbert, 1997; Jones and Rust, 1983; Rust and Jones, 1987). The predominantly quartz clastics occur within an argillaceous matrix and secondary silica and siderite cement is variably developed across the sequence (Pells, 1977). The average mineralogical composition for the Hawkesbury Sandstone was

given by Standard (1969) as: 68% quartz; 4% non-silica detrital grains, 20% matrix clay (70% kaolinite and 20% illite), and 8% secondary silica and siderite. The Hawkesbury sandstone has poorly connected porosity of c. 5% and bulk density of c. 2230 kg/m³ (Ord et al., 1991). UCS and elastic modulus for the Hawkesbury sandstone at room temperature is in the range 20 to 50 MPa and 2 to 6 GPa, respectively (Pells, 1977). Fig. 2a shows a view of a failed Hawkesbury sandstone specimen (from this study) that was tested at room temperature.

2.2. Specimen preparation and experimental methodology for UCS testing

To minimise durations required for heating the smallest practical specimen size was used for the testing. Bulk samples were cored and cut using diamond coring and cutting devices at the Monash University Civil Engineering Laboratories (MUCEL) to produce cylindrical specimens 23 mm in diameter and c. 46 mm in length. The ends of the samples were grinded using a face grinder at MUCEL, to produce two perfectly planar end surfaces perpendicular to the long dimension of the cylindrical specimen.

Specimens were heated in a high-temperature furnace, to their target testing temperature (25, 200, 400, 600, 800 or 950 °C) using a modest heating rate of 5 °C/min to minimise thermal shock and development of stress fractures. To ensure uniformity in temperature across the specimen upon loading, specimens were kept at the target temperature for two hours prior to testing. Two to three tests were carried out for each temperature value. Temperature was maintained at the target value during the entire duration of each test.

UCS testing was carried out at the target temperature and followed the suggested methods for UCS testing outlined in the ASTM guidelines (ASTM, 2007). The specimens were loaded in compression by a UCS testing apparatus at MUCEL (shown in Fig. 2b), employing a constant cross-head displacement rate of 0.1 mm/min, which resulted in a constant nominal axial strain rate of c. 0.22%/min. Constant-displacement loading continued until failure was observed in the stress v. nominal strain response. A load cell and displacement transducers were fitted to the loading ram to continuously record values of axial load and axial displacement, respectively.

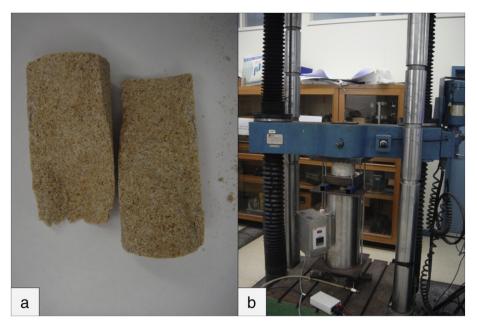


Fig. 2. Photos showing (a) failed 23 mm-diameter Hawkesbury sandstone specimen, and (b) setup for Uniaxial Compressive Strength testing at high temperatures.

2.3. Strain estimation, calculation of elastic modulus and associated limitations

The UCS testing was carried out at elevated temperature, precluding the use of strain gauges to measure specimen deformation directly. Axial deformation of the sandstone specimens was thus approximated from displacement of the loading ram. Axial strain calculated using loading ram displacement includes a component of strain related to flexing of the testing apparatus, in addition to axial strain of the specimen. The sandstone that was tested is low in stiffness when compared to the testing rig and, from our testing experience, the component of deflection related to flexing of the apparatus should be small, but not negligible. In the manuscript we refer to absolute values of strain or elastic modulus determined from the loading ram displacement data (and thus including apparatus flexure) as 'nominal strain' and 'nominal elastic modulus', respectively.

The nominal elastic modulus values discussed in this manuscript were calculated from the gradient of the steepest part of the relevant stress v. nominal strain curve that is linear over a range of at least 10 MPa (on the vertical axis), and for which the minimum value of stress was no less than 15 MPa. The minimum measurement interval (i.e. 10 MPa on the vertical axis) was chosen to ensure that the nominal elastic modulus value obtained was typical (not anomalous) and the minimum absolute value of stress considered (i.e. 15 MPa) for calculation was chosen to ensure that the nominal elastic modulus was estimated for the elastic portion of the stress v. nominal strain curve and not the non-linear portion that occurs for low load values.

A disproportionately large amount of apparatus flexure can be expected at very low values of applied load (during seating of the apparatus). Beyond significant values of applied load, change in displacement due to apparatus flexure is approximately proportional to change in applied load (i.e. the apparatus is linear elastic). Within the elastic range for the apparatus, the relative component of strain relating to apparatus flexure will be greater for rock with higher nominal elastic modulus than for rock with lower nominal elastic modulus. Thus, normalisation of nominal elastic modulus values will cause slight overestimation of nominal elastic modulus values that are low and slight underestimation of nominal elastic modulus values that are high. Normalised nominal elastic modulus values would be expected to be faithful to the true variation in elastic modulus with temperature. Despite the above-discussed

limitations on elastic modulus calculation associated with apparatus flexure, observations made regarding the influence of temperature on normalised elastic modulus values from our testing are treated as fundamental features reflective of the influence of temperature on mechanical behaviour.

2.4. Sample preparation and methodology for XRD analysis

Approximately 1.5 g of material from post-failure specimens for the 25 and 950 °C tests was ground with ethanol for 10 min in a McCrone micronising mill. Each powder slurry was then placed in an oven at 60 °C. Once dry, an agate mortar and pestle was used to homogenise each sample, before the powder was back pressed into a stainless steel sample holder for quantitative XRD analysis.

XRD patterns were recorded at CSIRO Land and Water with a PANalytical X'Pert Pro multi-purpose diffractometer using Fe-filtered Co Ka radiation, variable divergence slit, 1° anti-scatter slit and fast X'Celerator Si strip detector. The diffraction patterns were recorded in steps of 0.017° 20 using a 0.5 second counting time per step and logged to data files for analysis. The commercially-available package SIROQUANT, produced by Sietronics Pty Ltd., was used to perform quantitative analysis on the XRD data for each sample. Mineralogical abundance values determined from analysis were normalised to 100%. Unidentified or amorphous materials were excluded from the totals prior to normalisation.

3. Results

3.1. Results of UCS testing

Axial stress v. nominal axial strain plots for representative tests at each of the temperatures investigated are given in Fig. 3. The 25 to 600 $^{\circ}\text{C}$ curve set displays increasing maximum compressive strength values with increasing test temperature, a trend that is reversed for the 600 to 950 $^{\circ}\text{C}$ curve set. From Fig. 3, it can also be seen that the 25, 200, 400 and 600 $^{\circ}\text{C}$ stress v. nominal strain curves display a concave-up shape from initial loading to failure and a sudden failure, marked by a discrete stress drop after failure. On the other hand, the 950 $^{\circ}\text{C}$ curve of Fig. 3 displays a convex-up shape from initial loading to failure. The 800 $^{\circ}\text{C}$ curve displays characteristics of both the lower-temperature and higher-temperature

curve forms with a concave-up shape from initial loading, but a subtle convex-up shape for the stress v. nominal strain response during the final stages of loading prior to failure. Significant nominal strain after peak can be observed for the 800 and 950 °C (and to a lesser extent 600 °C) curves of Fig. 3, but not for the 25, 200 and 400 °C curves of Fig. 3. Nominal strain to failure increases with increasing test temperature for all curves depicted in Fig. 3.

Table 1 summarises the pertinent information of Fig. 3. The information of Table 1 confirms observations made from Fig. 3, that maximum compressive strength increases with increasing test temperature from the 25 to the 600 °C curve and maximum compressive strength decreases with increasing test temperature from the 600 to the 950 °C curve, and that nominal strain to failure increases with increasing test temperature. Table 1 also shows that nominal elastic modulus increases with increasing test temperature from the 25 to the 400 °C curve but that nominal elastic modulus decreases with increasing test temperature from the 400 to the 950 °C curve. Changes in the manner by which the mechanical properties (compressive strength and nominal elastic modulus) of the sandstone vary with increasing temperature occur in the 400 to 600 °C temperature range. We take the transition from entirely elastic to partially plastic behaviour for this sandstone to occur at c. 500 °C, for the testing conditions that were employed.

3.2. Results of XRD mineralogical analysis

The results of the mineralogical analysis using XRD methods are summarised in Table 2. From Table 2, the 950 °C specimen can be seen to contain a greater proportion of quartz, illite/muscovite, rutile and haematite relative to the 25 °C specimen. Conversely, the 25 °C specimen displays greater proportions of kaolin, smectite, goethite and anatase relative to the 950 °C specimen (Table 2). For the testing conditions considered, it is unlikely that any new quartz growth occurred. The greater quartz content for the 950 °C specimen when compared to the 25 °C specimen is likely to be related to the presence of amorphous material (that the XRD method was unable to identify) in the 950 °C specimen, which resulted in overestimation of quartz due to normalisation to a smaller total value. If this was the case,

Table 1Summary table of pertinent results from UCS testing and details for their calculation.

Temperature (°C)	Peak strength (MPa)	Nominal strain to failure	Nominal elastic modulus (GPa)	Stress range for nominal elastic modulus calculation (MPa)
25	36.51	0.0170	2.75	20-36
200	42.52	0.0197	3.30	18-28
400	60.81	0.0252	3.70	22-60
600	64.91	0.0360	2.37	30-61
800	60.99	0.0454	2.00	36-60
950	41.18	0.0587	0.97	18-28

the total amorphous material for the 950 $^{\circ}$ C specimen was on the order of 7 wt.% more than for the 25 $^{\circ}$ C specimen.

4. Discussion

The distinct difference in the shape of and nominal strain after peak displayed by the stress v. nominal strain curves for temperatures less than 500 °C when compared to the stress v. nominal strain curves for temperatures greater than 500 °C (Fig. 3) reflects the influence of thermally-induced plasticity, for temperatures greater than c. 500 °C. At the low confining pressures considered for the UCS testing, mechanisms for plasticity related to pressure-sensitive brittle–ductile transition behaviour can be excluded. However, the distinct differences in quantitative mineralogy from XRD analysis for the 25 and 950 °C specimens raise the issue of the influence of transformation plasticity. Transformation plasticity in rock refers to plastic deformation (creep) accommodated by changes in mineralogy (mineral decomposition/growth) or monomineralic phase transitions (see Poirier, 1982; Rutter and Brodie, 1995; Schmidt et al., 2003).

Temperatures on the order of 300 °C or more can initiate decomposition of goethite to form haematite (Goss, 1987) and decomposition of smectite to form illite (Huang et al., 1993). This can account for the loss of goethite and smectite and approximately equivalent gains in haematite

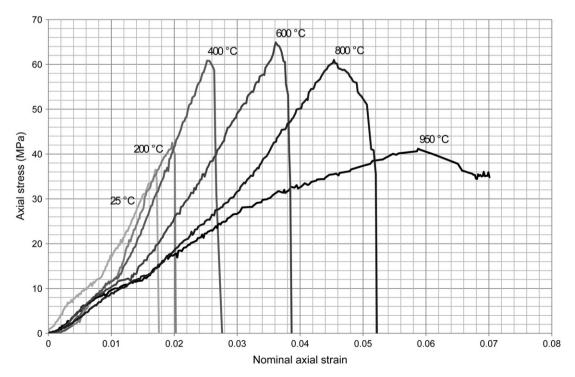


Fig. 3. Axial stress v. nominal axial strain curves for representative tests at each temperature investigated.

Table 2Summary table of mineralogical composition of specimens following UCS testing. All values in wt.%.

Testing temperature	Quartz	Haematite	Goethite	Illite/muscovite	Kaolinite	Smectite	Anatase	Rutile
25 °C	85	-	2	3	7	2	1	_
950 °C	91	1	-	7	_	-	<1	1

and illite/muscovite when the mineralogical composition from XRD analysis of the 950 °C specimen is compared to that of the 25 °C specimen (Table 2). Temperatures in excess of c. 600 to 650 °C can cause decomposition of anatase to form rutile (Czanderna et al., 1958), and can account for the higher abundance of anatase from XRD analysis of the 25 °C specimen and higher abundance of rutile in the 950 °C specimen (Table 2). Dehydroxylisation of kaolinite occurs at a rapid rate for temperatures above c. 500 °C (Bellotto et al., 1995; Ortega et al., 1993). If kaolinite is heated to temperatures beyond 1100 °C for sufficient durations, reaction will proceed beyond the dehydroxylisation process and form mullite (Gualteri et al., 1995). Thermally-induced decomposition of kaolinite and formation of mullite involves the production of an interstitial and meta-stable form of kaolin which presents as an amorphous compound in XRD analysis (Bellotto et al., 1995; Gualteri et al., 1995). The discrepancy between totals used for normalisation of the mineralogical data for the 25 and 950 °C specimens (totaling c. 7 wt.%) can be accounted for by conversion of kaolinite to amorphous meta-kaolin during dehydroxylisation of kaolinite at temperatures above 500 °C. Kaolinite is easily the most abundant secondary mineral in the 25 °C specimen (Table 2) and is likely to form a significant volume of the cement for the Hawkesbury sandstone. The coincidence between the threshold temperature for the onset of plasticity (as observed from the UCS testing programme) and the temperatures at which dehydroxylisation of kaolin becomes significant is suggestive of a causal relationship between the development of plasticity in the Hawkesbury sandstone and breakdown of cementitious kaolinite. We propose that thermally-induced plasticity (and associated weakening and softening) in the Hawkesbury sandstone for temperatures in excess of 500 °C is related to significant transformation plasticity occurring in the cement for the sandstone and dominantly driven by kaolinite dehydroxylisation.

The origin of the thermally-induced strengthening observed for temperatures less than 500 $^{\circ}$ C may be related to strain hardening by localised plasticity development, perhaps related to transformation of goethite to haematite and/or smectite to illite at more modest temperatures on the order of 300 $^{\circ}$ C.

Transformation plasticity provides a physical mechanism to (at least partly) explain the influence of temperature on the mechanical behaviour of the Hawkesbury sandstone at low confining pressures. The dependence of the process of transformation plasticity on rock mineralogy is elementary: if the mineralogy of the Hawkesbury sandstone was different one might expect a very different mechanical response to heating. Dependence between rock character and mechanical response of rock to environmental conditions is well established. The pressure and temperature conditions of the brittle-ductile transition has been shown to be highly dependent on rock type (see Paterson and Wong, 2005, Table 10, p. 215, and references therein). Research carried out on the brittle-ductile transition has demonstrated that the porosity and grain size of a given rock will influence the pressure and temperature conditions of the brittleductile transition (Fredrich et al., 1989; Rutter and Hadizadeh, 1991; Scott and Nielsen, 1991; Vajdova et al., 2004). Thus, the geological history of a region and the resultant rock properties that emerge influence its mechanical response for a given set of pressure and temperature conditions (see Wong, 1990; Zhang et al., 1993). From the variation in the thermal response of the various rock types of Fig. 1, it is clear that a model built on the expected mechanical response to heating for one rock type cannot be blindly applied to the next. One might however consider development of a general model to describe mechanical response to heating for a specific rock type (such as granite or sandstone). The validity of such an approach deserves consideration.

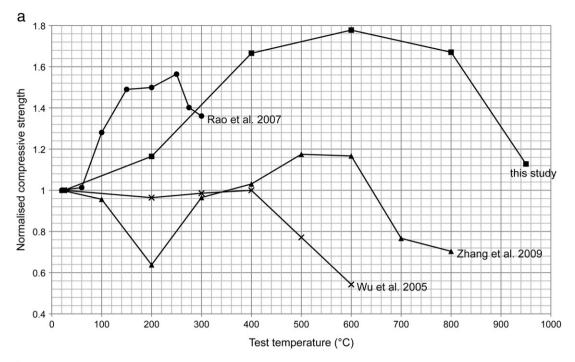
Curves displaying the influence of temperature on the normalised compressive strength and elastic modulus of the tested sandstone, and other sandstones from the literature that have been subjected to UCS testing at various temperatures, are given in Fig. 4. The results from the UCS testing from Rao et al. (2007); Zhang et al. (2009) and this study define an initial strengthening (and stiffening) and subsequent weakening (and softening) pattern (Fig. 4). On the other hand, the UCS testing results of Wu et al. (2005) show a general decrease in both compressive strength and elastic modulus with increasing temperature, from the lowest temperature tests, which is emphasised for tests carried out at temperatures above 500 °C (Fig. 4). The transition in mechanical character denoted by a negative deflection in the plots of Fig. 4 can be taken to mark a transition in deformation mechanism, with increasing temperature, for the sandstone. This deflection in the curves of Fig. 4 (the onset of plasticity) occurs at different temperatures for the various sandstones tested (250 °C for Rao et al., 2007; 400 °C for Wu et al., 2005; 500 °C for Zhang et al., 2009 and this study). The strikingly different patterns defined by the curves of Fig. 4, illustrate a variation in the mechanical response of the various sandstones tested that shows not all sandstones are 'created equal' when it comes to their mechanical response to heating.

More than crystalline rock, sedimentary rocks can display significant variability in characteristics inherited from their depositional history (detrital mineralogy, grain shape, grain size and distribution) and post-depositional experience (burial, thermal and fluid interaction history and cement mineralogy). This variability can influence the manner by which the rock responds to heating in an engineering application. The design approach for engineering applications which incorporate an understanding of expected rock behaviour at elevated temperatures needs not only consider rock type, but the material properties specific to the rock unit in question (e.g. mineralogy, porosity, density, etc.). Ideally, samples obtained directly from the unit will be tested under the range of temperatures to which the rock will be subjected so that mechanical prameters for design can be obtained. Where sampling is not feasible, a sound appreciation of the geological history of the rock mass can provide information on the conditions (pressures, temperatures, fluids) which the rock is likely to have experienced and how its mineralogy and granular properties are likely to have responded to these conditions. Expected material properties, as deduced from the likely geological history of the rock mass, can be used as a guide when applying the results of laboratory testing of the mechanical response of similar rocks to heating to estimate mechanical behaviour for the engineering application.

In addition to rock properties and mineralogy, the expected environmental conditions for an engineering application, including depth (confining pressure) and chemistry (fluids), are also likely to influence the mechanical response of rock to heating. Future work on the issue of the influence of temperature on rock mechanics could consider the influence of these additional environmental factors, by considering testing of heated specimens at a range of confining pressures and in the presence of fluids with varying chemistry.

5. Conclusions

UCS testing carried out on sandstone at various temperatures between 25 and 950 °C has revealed a distinct variation in the influence of temperature on the mechanical behaviour of Hawkesbury sandstone



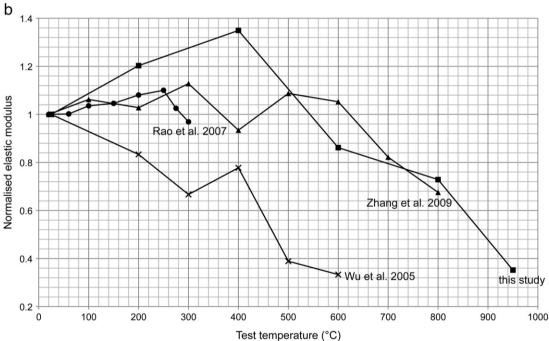


Fig. 4. (a) Normalised compressive strength v. test temperature curves, and (b) normalised elastic modulus v. test temperature curves from this study and similar studies on sand-stone reported on in the literature.

either side of a temperature of c. 500 °C. For temperatures less than c. 500 °C, compressive strength and elastic modulus were observed to increase with increasing testing temperature, whereas the opposite scenario was observed for temperatures greater than c. 500 °C. Mineralogical (XRD) analyses carried out on the post-failure samples from the 25 and 950 °C specimens are consistent with significant changes in sandstone mineralogy with heating. Dehydroxylisation of kaolinite from the Hawkesbury sandstone cement appears to be associated with the onset of plasticity and weakening and softening of the sandstone with increasing temperature, for temperatures in excess of c. 500 °C. Transformation plasticity relating to dehydroxylisation of cementitious kaolinite offers a mechanism to explain the observed

changes in mechanical properties with increasing temperature for the UCS tests at temperatures greater than c. 500 °C.

Clearly the influence of temperature in inducing transformation plasticity in sandstone will be heavily dependent on rock mineralogy and the influence of temperature on the mechanical behaviour of sandstone can differ significantly between rock units. A review of the results of some studies carried out on the influence of temperature on UCS of various sandstones supports this hypothesis. A generic model for the influence of temperature on mechanical behaviour of a rock type will not provide sufficiently accurate information for engineering design of elevated temperature underground systems. Specific models for the influence of temperature on the mechanical behaviour of the rock unit being

considered for design should be produced from the results of direct testing of the rock unit in question.

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References

- ASTM, 2007. D7012-07 Standard Test Method for Compressive Strength and Elastic Modulus of Intact Core Rock Specimens Under Varying States of Stress and Temperatures. Annual Book of ASTM Standards: American Scoiety of Testing and Materials. Philadelphia.
- Bellotto, M., Gualteri, A., Artioli, G., Clark, S.M., 1995. Kinetic study of the kaolinite-mullite reaction sequence. Part I: kaolinite dehydroxylisation. Physics and Chemistry of Minerals 22, 207–214.
- Byerlee, J.D., 1968. Brittle-ductile transition in rocks. Journal of Geophysical Research 73, 4741–4750.
- Czanderna, A.W., Ramachandra Rao, C.N., Honig, J.M., 1958. The anatase-rutile transition. Part I: kinetics of the transformation of pure anatase. Transactions of the Faraday Society 54, 1069–1073.
- Dmitriyev, A.P., Kusyayev, L.S., Protasov, Y.I., Yamschchikov, V.S., 1969. Physical Properties of Rocks at High Temperatures (translated from Russian). Nedra Press, Moscow.
- Duclos, R., Paquet, J., 1991. High-temperature behaviour of basalts role of temperature and strain rate on compressive strength and K_{Ic} toughness of partially glassy basalts at atmospheric pressure. International Journal of Rock Mechanics and Mining Science and Geomechanics Abstracts 28, 71–76.
- Fredrich, J.T., Evans, B., Wong, T.-F., 1989. Micromechanics of the brittle to ductile transition in Carrara marble. Journal of Geophysical Research 94 (B4), 4129–4145.
- Gibb, S.E., 1999. High-temperature, very deep geological disposal: a safer alternative for high-level radioactive waste? Waste Management 19, 207–211.
- Goss, C.J., 1987. The kinetics and reaction mechanism of the goethite to hematite transformation. Mineral Magazine 50, 437–451.
- Griggs, D.T., 1936. Deformation of rocks under high confining pressures: I. Experiments at room temperature. Journal of Geology 44, 541–577.
- Griggs, D.T., Turner, F.J., Hear, H.C., 1960. Deformation of rocks at 500° to 800 °C. In: Griggs, D., Handin, J. (Eds.), Rock Deformation. : Geol. Soc. Am. Mem., 79. Geological Society of America, New York, pp. 39–104.
- Gualteri, A., Bellotto, M., Artioli, G., Clark, S.M., 1995. Kinetic study of the kaolinite-mullite reaction sequence. Part II: mullite formation. Physics and Chemistry of Minerals 22, 215–222.
- Heap, M.J., Baud, P., Meredith, P.G., 2009. Influence of temperature on brittle creep in sandstones. Geophysical Research Letters 36, L19305.
- Heap, M.J., Baud, P., Meredith, P.G., Vinciguerra, S., Bell, A.F., Main, I.G., 2011. Brittle creep in basalt and its application to time-dependent volcano deformation. Earth and Planetary Science Letters 307, 71–82.
- Heard, H.C., 1960. Transition from brittle fracture to ductile flow in Solenhofen limestone as a function of temperature, confining pressure, and interstitial fluid pressure. In: Griggs, D., Handin, J. (Eds.), Rock Deformation.: Geol. Soc. Am. Mem., 79. Geological Society of America, New York, pp. 193–226.
- Herbert, G., 1997. Sequence stratigraphic analysis of early and middle Triassic alluvial and estuarine fades in the Sydney Basin, Australia. Australian Journal of Earth Sciences 44. 125–143.
- Heuze, F.E., 1983. High-temperature mechanical, physical and thermal properties of granitic rocks a review. International Journal of Rock Mechanics and Mining Science and Geomechanics Abstracts 20, 3–10.
- Huang, W.-L., Longo, J.M., Pevear, D.R., 1993. An experimentally derived kinetic model for smectite-to-illite conversion and its use as a geothermometer. Clays and Clay Minerals 41, 162–177.
- Jones, B.G., Rust, B.R., 1983. Massive sandstone facies in the Hawkesbury Sandstone, a Triassic fluvial deposit near Sydney, Australia. Journal of Sedimentary Petrology 53, 1249–1259.
- Lockner, D.A., 1995. Rock failure. In: Ahrens, T.J. (Ed.), Rock Physics and Phase Relations: a Handbook of Physical Constants. American Geophysical Union, Washington D.C., pp. 127–147.
- Logan, S.E., 1974. Deep self-burial of radioactive wastes by rock-melting capsules. Nuclear Technology 21, 111–124.
- Mao, X.-b, Zhang, L-y, Li, T.-z, Liu, H.-s, 2009. Properties of failure mode and thermal damage for limestone at high temperature. Mining Science and Technology 19, 290-294.
- Minchener, A.J., 2005. Coal gasification for advanced power generation. Fuel 84, 2222–2235.
- Mogi, K., 1966. Pressure dependence of rock strength and transition from brittle fracture to ductile flow. Bulletin of the Earthquake Research Institute 44, 215–232.
- Ord, A., Vardoulakis, I., Kajewski, R., 1991. Shear band formation in Gosford sandstone. International Journal of Rock Mechanics and Mining Science and Geomechanics Abstracts 28, 397–401.

- Orowan, E., 1960. Mechanism of seismic faulting. In: Griggs, D., Handin, J. (Eds.), Rock Deformation.: Geol. Soc. Am. Mem., 79. Geological Society of America, New York, pp. 323–345.
- Ortega, A., Rouquerol, F., Akhouayri, S., Laureiro, Y., Rouquerol, J., 1993. Kinetical study of the thermolysis of kaolinite between —30° and 1000°C by controlled rate evolved gas analysis. Applied Clay Science 8, 207–214.
- Paquet, J., Francois, P., Nedelec, A., 1981. Effect of partial melting on rock deformation: experimental and natural evidences on rocks of granitic compositions. Tectonophysics 78, 545–565.
- Paterson, M.S., 1958. Experimental deformation and faulting in Wombeyan marble. Geological Society of America Bulletin 69, 465–476.
- Paterson, M.S., Wong, T.-F., 2005. Experimental Rock Deformation the Brittle Field. Springer, New York.
- Pells, P.J.N., 1977. Measurement of engineering properties of Hawkesbury Sandstone. Australian Geomechanics Journal G7, 10–20.
- Poirier, J.P., 1982. On transformation plasticity. Journal of Geophysical Research 87 (B8), 6791–6797.
- Rao, Q.-h, Wang, Z., Xie, H.-f, Xie, Q., 2007. Experimental study of properties of sandstone at high temperature. Journal of Central South University of Technology (English Edition) 14 (s1), 478–483.
- Rust, B.K., Jones, B.G., 1987. The Hawkesbury Sandstone south of Sydney, Australia: Triassic analogue for the deposit of a large, braided river. Journal of Sedimentary Petrology 57, 222–233.
- Rutter, E.H., 1986. On the nomenclature of mode of failure transitions in rock. Tectonophysics 122, 381–387.
- Rutter, E.H., Brodie, K.H., 1995. Mechanistic interactions between deformation and metamorphism. Geological Journal 30, 227–240.
- Rutter, E.H., Hadizadeh, J., 1991. On the influence of porosity on the low-temperature brittle-ductile transition in siliciclastic rocks. Journal of Structural Geology 13, 609–614.
- Rutter, E.H., Neumann, D.H.K., 1995. Experimental deformation of partially molten Westerly granite under fluid-absent conditions, with implications for the extraction of granitic magmas. Journal of Geophysical Research 100, 15697–15715.
- Schmidt, C., Bruhn, D., Wirth, R., 2003. Experimental evidence of transformation plasticity in silicates: minimum of creep strength in quartz. Earth and Planetary Science Letters 205, 273–280.
- Scott, T.E., Nielsen, K.C., 1991. The effects of porosity on the brittle–ductile transition in sandstones. Journal of Geophysical Research 96 (B1), 405–414.
- Shoko, E., McLellan, B., Dicks, A.L., Diniz da Costa, J.C., 2006. Hydrogen from coal: production and utilisation technologies. International Journal of Coal Geology 62, 213–222.
- Sibson, R.H., 1977. Fault rocks and fault mechanisms. Journal of the Geological Society 133, 191–213.
- Sibson, R.H., 1982. Fault zone models, heat flow, and the depth distribution of earthquakes in the continental crust of the United States. Bulletin of the Seismological Society of America 72, 151–163.
- Standard, J.C., 1969. Hawkesbury Sandstone. In: Packham, G.H. (Ed.), The Geology of New South Wales. Geol. Soc. Aust., Sydney, pp. 407–415.
- Stiegel, G.J., Ramezan, M., 2006. Hydrogen from coal gasification: an economical pathway to a sustainable energy future. International Journal of Coal Geology 65, 173–190.
- Tullis, J., Yund, R.A., 1977. Experimental deformation of dry Westerly granite. Journal of Geophysical Research 82, 5705–5718.
- Tullis, J., Yund, R.A., 1987. Transition from cataclastic flow to dislocation creep of feldspar: mechanisms and microstructures. Geology 15, 606–609.
- Vajdova, V., Bond, P., Wong, T.-F., 2004. Compaction, dilatancy, and failure in porous carbonate rocks. Journal of Geophysical Research 109, B05204.
- van der Molen, I., Paterson, M.S., 1979. Experimental deformation of partially-melted granite. Contributions to Mineralogy and Petrology 70, 299–318.
- Voight, B., 2000. Structural stability of andesite volcanoes and lava domes. Philosophical Transactions of the Royal Society A 358, 1663–1703.
- Voight, B., Elsworth, D., 1997. Failure of volcano slopes. Geotechnical 47, 1-31.
- Watters, R.J., Zimbelman, D.R., Bowman, S.D., Crowley, J.K., 2005. Rock mass strength assessment and significant to edifice stability, Mt Rainer and Mt Hood, Cascade Range volcanoes. Pure and Applied Geophysics 157, 957–976.
- Wong, T.-F., 1982. Effects of temperature and pressure on failure and post-failure behavior of Westerly granite. Mechanics of Materials 1, 3–17.
- Wong, T.-F., 1990. Mechanical compaction and the brittle–ductile transition in porous sandstone. In: Knipe, R.J., Rutter, E.H. (Eds.), Deformation Mechanisms, Rheology and Tectonics, Special Publications of the Geological Society: The Geological Society, London, 54, pp. 111–122.
- Wu, Z., Qin, B.-d, Chen, L.-j, Luo, Y.-j, 2005. Experimental study on mechanical character of sandstone of the upper plank of coal bed under high temperature (in Chinese). Chinese Journal of Rock Mechanics and Engineering 24, 1863–1867.
- Xu, X.-l., Gao, F., Shen, X.-m., Xie, H.-p., 2008. Mechanical characteristics and microcosmic mechanisms of granite under temperature loads. Journal of China University of Mining and Technology 18, 413–417.
- Xu, X.-I., Kang, Z.-x., Ji, M., Ge, W.-x., Chen, J., 2009. Research of microcosmic mechanism of brittle-plastic transition for granite under high temperature. Procedia Earth and Planetary Science 1, 432–437.
- Zhang, J., Davis, D.M., Wong, T.-F., 1993. The brittle–ductile transition in porous sedimentary rocks: geological implications for accretionary wedge aseismicity. Journal of Structural Geology 15, 819–830.
- Zhang, L.-y., Mao, X.-b., Lu, A.-h., 2009. Experimental study of on the mechanical properties of rocks at high temperature. Science in China Series E-Technological Sciences 52, 641–646.