Thermal alterations in the poro-mechanical characteristic of an Indian sandstone – A comparative study

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\textbf{A R T I C L E  I N F O}

\textbf{Keywords:}  
Sandstone  
Temperature  
Strength  
Porosity  
Elastic modulus

\textbf{A B S T R A C T}

Knowledge of the variations caused by high temperature to the mechanical properties is a vital asset in processes such as underground coal gasification (UCG), in-situ disposal of nuclear waste and during the recovery of geothermal. The study can also provide valuable insights during the restoration of fire-damaged buildings. Due to the presence of heat, changes in rock morphology and mineralogy lead to a substantial change in the geomechanical properties of rock. Effect of high temperatures (1000 °C) on the tensile and compressive strength of a fine-grained Indian Sandstone have been analysed in this study. Induced ductility, observed at temperatures above 500 °C causes the rock to exhibit properties similar to that of a plastic material. The geomechanical properties decrease sharply beyond 500 °C, which has been referred to as the critical temperature of the rock. Beyond the critical temperature, internal structure of the rock undergoes considerable damage in the form of microcracks and fissures created due to phase transition of quartz at 573 °C and generation of thermal stress. Change in internal structure and mineralogy have been analysed by performing XRD (X-ray powder diffraction) and FDXM (Four-dimensional X-ray microscopy) studies. With the help of FDXM, change in porosity of rock has been analysed by studying the variation in connected and non-connected pores.

1. Introduction

Behaviour of rocks under thermo-mechanical conditions is of great importance for the success of processes such as underground coal gasification (UCG), disposal of radioactive waste in deep geological repositories (DGR), recovery of geothermal energy and restoration of fire-damaged buildings. The rocks in these processes encounter various degrees of thermal treatment. Thermal treatment refers to the heating regime provided to the rocks. Some processes, such as radioactive disposal are designed to operate within a temperature limit of 100 °C for a duration of several years (Verma et al., 2015; Vishal et al., 2011). However, in processes like UCG and events of building fires, temperature can reach up to 1000 °C (Hajpál, 2002; Sirdesai et al., 2015). In event of building fires, the construction materials are exposed to temperatures over 700 °C (Kourkoulis, 2007). The nature of thermal damage is governed by factors such as source and the intensity of the fire, duration of exposure, construction material and the technique used for quenching the fire (Chakrabarti et al., 1996; Freire-Lista et al., 2016). When exposed to thermal treatment, microstructure of the rock sustains considerable damage resulting from the change in the density of the microcracks. The change to the microstructure alters the physical and mechanical properties of the rock. However, the nature and the magnitude of the alteration is not constant within all rock types (Homand-Etienne and Troalen, 1984; Liu and Xu, 2015; Luo and Wang, 2011; Zhang et al., 2009). Properties such as morphology, mineralogy and chemical composition play a pivotal role in governing the post-treatment behaviour. Furthermore, the effect of thermal treatment is diverse in rocks of the same origin, as explained by Hajpál (2002) and Tian et al. (2014, 2015). Variations in the degree and nature of thermal treatment across the above-mentioned processes pose a challenge in predicting the geo-mechanical response of the rocks. It is, therefore, imperative to study the effect of temperature and duration of exposure on the rocks encountered in such processes. Changes brought about by thermal treatment need to be taken into consideration before designing structures within the strata. Properties such as porosity and permeability are crucial in processes such as radioactive disposal in DGR and UCG. Hazardous contaminants such as radionuclides and the noxious gases can migrate through the newly generated microcracks and could contaminate the underground aquifer, causing long-term large-scale disasters. Therefore, it is essential to study the effect of thermal...
treatment on the transport properties of rocks. The study will also help in formulating the protocols of repair and restoration of fire-damaged structures. Factors imperative to the stability of a structure are mainly the strength and the load bearing capacity of the building materials. Effect of fire on the structures made primarily of stone can be analysed by performing extensive laboratory investigation. Strength of the rocks exposed to fires can be predicted from the result of such studies and suitable relief protocols can be established in order to remove and replace the affected zones. Suitable and viable restoration materials can be effectively chosen by performing such studies on the potential rock specimens (Freire-Lista et al., 2016). Since strength and the geomechanical response of rocks is governed by its microstructure, detailed analysis of thermal damage can be performed by analysing the change in the internal morphology and mineralogy when subjected to high temperatures.

The present study focuses on studying the effect of high temperatures on the poro-mechanical properties of a fine-grained Indian sandstone with the help of experimental analysis such strength tests, FDXM (Four-dimensional X-ray microscopy) and XRD (X-ray diffraction). The study is a part of the ongoing research to evaluate the changes brought about in the strata in a UCG project. The samples, analysed in this study, have been tested under unconfined conditions due to the unavailability of tri-axial systems that can operate at high temperatures. With the help of a comprehensive literature survey, rocks of similar mineralogical and morphological properties were chosen for comparative analysis. A comparative analysis helps in appreciating the mechanism of thermal damage and deformation. The analysis also provides an insight into the effect of a specific regime of thermal treatment on the mechanical properties of rock.

2. Previous work

The mechanism of thermal deformations in rocks has intrigued several researchers since the beginning of the 19th century. In their research, Handin and Hager Jr studied the effects of temperature and confining pressure on deformational behaviour of rocks (Handin and Hager, 1957, 1958). Several sedimentary rock types were tested at room temperature and high temperature (300 °C). However, the aim of the study was to investigate the rock deformations occurring within the crust. Several similar studies were later performed by seismologists to gain an insight on the deep-seated crustal deformations. Research on the sustainability and feasibility of processes such as UCS and nuclear waste disposal provided a niche for further development and addition to the scientific database on the behaviour of rocks at high temperatures. Experimental studies were conducted to gain an understanding on the effect of temperature on physico-mechanical properties. The mechanism of fragmentation in several igneous and metamorphic rocks under thermal loads was investigated by studying the effect of temperature on the elastic moduli (Wingquist, 1969). The rocks were tested at various temperatures up to 815.5 °C (1500 °F) and the elastic moduli were calculated by measuring the sonic velocities. Gradual decrease in the shear modulus and the Young’s modulus up to 204.4 °C (400 °F) and a sharp decline thereafter up to 537.8 °C (1000 °F) was observed in all the samples. An increase of 28% in the Young’s modulus of quartzite at 571.1 °C (1060 °F) was reported. This increase was attributed to the α–β phase inversion in quartz observed at 573 °C.

Heating leads to the formation of thermal stresses in a polycrystalline mineral assemblage. Thermal stress is a result of the anisotropy in thermal expansion properties of different minerals. Thermal expansion of different minerals was studied by Clark (1966) and their variations can be seen in Table 1. As a result, microcracks start originating at the grain boundaries of the minerals. Generation of microcracks, the primary cause of the reduction in the elastic properties, was reported by Homand-Etienne and Troalen (1984), Zhang et al. (2005) and Freire-Lista et al. (2016) as well. Microcracking at high temperatures can be better appreciated by viewing a rock specimen under a microscope.

Table 1
Thermal expansion of various minerals (modified after (Clark, 1966)).

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Axis</th>
<th>100 °C</th>
<th>200 °C</th>
<th>400 °C</th>
<th>600 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>lc</td>
<td>0.14</td>
<td>0.30</td>
<td>0.73</td>
<td>1.75</td>
</tr>
<tr>
<td></td>
<td>lc</td>
<td>0.08</td>
<td>0.18</td>
<td>0.43</td>
<td>1.02</td>
</tr>
<tr>
<td></td>
<td>lc</td>
<td>0.05</td>
<td>0.14</td>
<td>0.48</td>
<td>0.90</td>
</tr>
<tr>
<td></td>
<td>lc</td>
<td>0.00</td>
<td>0.10</td>
<td>0.04</td>
<td>0.13</td>
</tr>
<tr>
<td>Plagioclase</td>
<td>lc</td>
<td>0.01</td>
<td>0.00</td>
<td>0.005</td>
<td>0.065</td>
</tr>
<tr>
<td></td>
<td>lc10</td>
<td>0.00</td>
<td>0.22</td>
<td>0.93</td>
<td>0.83</td>
</tr>
<tr>
<td>Calcite</td>
<td>lc</td>
<td>0.09</td>
<td>0.22</td>
<td>0.55</td>
<td>0.39</td>
</tr>
<tr>
<td>Hornblende</td>
<td>lc</td>
<td>0.00</td>
<td>0.10</td>
<td>0.04</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>lc</td>
<td>0.005</td>
<td>0.13</td>
<td>0.29</td>
<td>0.46</td>
</tr>
</tbody>
</table>

Homand-Etienne and Troalen (1984) observed thermal cracking in granites and limestones, with the help of a scanning electron microscope (SEM) and concluded that the response of a rock depends on both the mineralogy and the texture. Zhang et al. (2005) studied thermal cracking of sandstone by performing thin-section analysis coupled with heating. Growth in the length of the inherent microcracks and development of new microcracks at grain boundaries, were reported at the onset of heating. The newly generated cracks migrate and join each other to form a major fissure. The effect of cyclic thermal treatment was studied on the samples of Spanish granites by Freire-Lista et al. (2016). The granite samples, which are the widely-used construction material, were heated for 18 h at 105 °C and cooled at 20 °C for 6 h by immersing them in water. Repetitive heating and cooling (42 times) ensured that the samples were provided cyclic thermal treatment. Although the samples were exposed to mere 105 °C, repetitive thermal treatment resulted in formation of high thermal stresses within the rock specimen. Microcracks originating in and around the mineral grains were reported by observing the thin-sections of the samples before and after thermal treatment. Fresh microcracking and coalescence of pre-existing microcracks, led to a decrease in the ultrasonic wave velocities, bulk density and the dynamic Young’s modulus, whereas, the porosity of the rock samples increased. Effect of heat treatment on the microcracking behaviour can be better appreciated while studying rock types which are compact and very low inherent microcracks. Studies performed on coarse-grained marble specimens having low inherent microcracks reported the evolution of fissures at various temperatures (Peng et al., 2016). At temperatures between 200 and 400 °C, large amount of microcracks start emerging around the tightly-packed calcite grains due to anisotropic thermal expansion. However, the high thermal stresses within the structure leads to the disintegration of the grain at temperatures around 600 °C due to the formation of intragranular cracks. Distinct thermal expansion behaviour of various mineral grains were observed by Keshavarz et al. (2010) while studying the geomechanical response of African gabbro samples at high temperatures. Physical properties such as porosity and permeability are directly dependent on the microcrack network; and therefore, any alteration to the latter would effect a change in the former. While an increase in these properties has been commonly reported (Brotóns et al., 2013; Hajipavlou, 2002; Homand-Etienne and Troalen, 1984; Liu et al., 2016a; Liu et al., 2016b; Rosengren and Jaeger, 1968; Sirdesai et al., 2016b; Zhang et al., 2016; Zhang et al., 2017; Zhang et al., 2005), it has also been suggested that the newly generated cracks/pores do not always merge (Deragina et al., 1993). Chemical reactions occurring at different temperature also play a role in the generation of microcracks (Hajipavlou, 2002; Somerton, 1992). Key thermal reactions occurring in the mineral assemblage of a rock can be seen in Table 2.

Together, microcracks and chemical changes alter the mechanical properties of a rock. The formation of fissures and pores reduce the
load-bearing capacity of rocks. Stability and condition of the neighbouring strata is of immense importance in processes such as UCG and disposals in DGR. In UCG, the rocks surrounding the reactor have to encounter extreme levels of heat treatment leading to the formation of large fissures. This renders the rock loose and fragile (Den’gina et al., 1993; Sirdesai et al., 2015). Furthermore, since swelling is observed in rocks of the finer grained variety, a reactor surrounded by such rocks would be encompassed within a “swell-zone”. Swelling results from the formation of large number of interconnected pores that can constitute 40–60% of the total volume, leading to an increase in permeability (Den’gina et al., 1993). Therefore, to lower the possibility of subsidence and groundwater contamination, the extent of loosening and swelling in rocks and their relation have to be studied. The study would assist in optimising the dimension and the operations of a reactor. Since the scope of this study could be extended to examine and suggest the re-optimising the dimension and the operations of a reactor. Since the irregularities in the range of critical temperature can be caused by the mineral composition and the nature of thermal treatment (rate of heating, method of cooling, duration of exposure at target temperature), a comprehensive comparative study can help in understanding the mechanism of thermally driven alterations in the mechanical properties of the rock. A detailed overview of the rocks chosen for the comparative study has been provided in Table 3.

### Materials and methods

#### 3.1. Origin of sample, mineralogy and specimen preparation

The experimental analyses were performed on specimens of fine-grained sandstone belonging to the Upper Bhandari Group of the Vindhyan Supergroup which were collected from the Dholpur district of Rajasthan, India (Fig. 1). The sandstone is characterised by its monomineralic nature with quartz and feldspar being the dominant minerals. The mineral grains, which constitute 95% of the sandstone, are contained within a siliceous cement containing trace amounts of mica and pyroxene. The mineral grains are well-sorted, closely packed and are sub-rounded to rounded in nature. The rock is grey in colour owing to the presence of ferrous minerals in trace quantities (CGWB, 2010; DMG-Rajasthan, 2006). Sandstone belonging to this area has been extensively under loads. Post failure characteristics of the rock are highlighted on the induction of plasticity since the rocks display higher value of strain prior to failure (Keshavarz et al., 2010; Mao et al., 2009; Peng et al., 2016). The ‘strain-to-failure’ gradually increases with the increase in temperature but above a certain level of heating, the increase becomes prominent and noticeable with a reciprocated decrease in compressive strength and the modulus of elasticity, thereby marking the transition point from brittle to ductile mode of failure. The point of transition denotes the critical temperature (CT) or critical temperature zone (CTZ) beyond which all a rapid decline can be observed in the mechanical properties of the rock. This temperature indicates reduction in inter-grain cohesion and the formation of intragranular microcracks. Rock specimen beyond critical temperature appears highly broken and splintered when observed through a microscope. Rocks of the same lithology have similar critical temperature range; as has been observed for sandstone at around 600–800 °C by Hajpál and Török (1998), Ranjith et al. (2012), Liu and Xu (2015) and Lü et al. (2017). However, variations have been observed for sedimentary rocks such as limestone, mudstone and claystone. While transitions in limestones have been observed at lower temperatures (200 °C) (Zhang et al., 2017), heat treatment until 800 °C manifested no marked changes in the mechanical properties of the mudstones and claystone studied by Luo and Wang (2011) and Tian et al. (2014), respectively. Since the irregularities in the range of critical temperature can be caused by the mineral composition and the nature of thermal treatment (rate of heating, method of cooling, duration of exposure at target temperature), a comprehensive comparative study can help in understanding the mechanism of thermally driven alterations in the mechanical properties of the rock. A detailed overview of the rocks chosen for the comparative study has been provided in Table 3.

### Table 3

**Overview of the rocks reviewed for comparative study.**

<table>
<thead>
<tr>
<th>Sample (acronym)</th>
<th>Origin</th>
<th>Ref.</th>
<th>Dominant mineral</th>
<th>Cement</th>
<th>$\sigma_c$ (MPa)</th>
<th>$\sigma_t$ (MPa)</th>
<th>$E$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maukbroner (MS)</td>
<td>Germany</td>
<td>(Hajpál and Török, 1998)</td>
<td>Quartz</td>
<td>Clayey</td>
<td>43.38</td>
<td>4.54</td>
<td>–</td>
</tr>
<tr>
<td>Donzdorfer (DS)</td>
<td>Germany</td>
<td>(Hajpál and Török, 1998)</td>
<td>Quartz</td>
<td>Ferruginous-clayey</td>
<td>49.64</td>
<td>3.08</td>
<td>–</td>
</tr>
<tr>
<td>Pfzinzaier (PS)</td>
<td>Germany</td>
<td>(Hajpál, 2002)</td>
<td>Quartz</td>
<td>Ferruginous-clayey</td>
<td>84.1</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Zuxhou (XS)</td>
<td>China</td>
<td>(Zhang et al., 2009)</td>
<td>–</td>
<td>–</td>
<td>169.27</td>
<td>–</td>
<td>16.76</td>
</tr>
<tr>
<td>Qinling (QS)</td>
<td>China</td>
<td>(Li and Xu, 2015)</td>
<td>Quartz</td>
<td>–</td>
<td>59.79</td>
<td>–</td>
<td>21.06</td>
</tr>
<tr>
<td>Linyi (LS)</td>
<td>China</td>
<td>(Li et al., 2017)</td>
<td>Quartz</td>
<td>–</td>
<td>5.69</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Hawkesbury (HS)</td>
<td>Australia</td>
<td>(Ranjith et al., 2012)</td>
<td>Quartz</td>
<td>Argillaceous</td>
<td>36.51</td>
<td>–</td>
<td>2.75</td>
</tr>
<tr>
<td>Hunter Valley (HV)</td>
<td>Australia</td>
<td>(Lü et al., 2016a)</td>
<td>Quartz</td>
<td>Clayey</td>
<td>58.36</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Dholpur (DH)</td>
<td>India</td>
<td>This study</td>
<td>Quartz</td>
<td>Siliceous</td>
<td>38.12</td>
<td>4.81</td>
<td>27.83</td>
</tr>
<tr>
<td>Limestone (L)</td>
<td>China</td>
<td>(Mao et al., 2009)</td>
<td>–</td>
<td>–</td>
<td>123.44</td>
<td>–</td>
<td>17.68</td>
</tr>
<tr>
<td>Claystone (CL)</td>
<td>Germany</td>
<td>(Tian et al., 2014)</td>
<td>Clay (Illite)</td>
<td>Siliceous</td>
<td>17.41</td>
<td>–</td>
<td>1.63</td>
</tr>
<tr>
<td>Westphalia (WC)</td>
<td>Germany</td>
<td>(Lü et al., 2016a)</td>
<td>Clay &amp; Quartz</td>
<td>Micaceous</td>
<td>31.05</td>
<td>–</td>
<td>1.18</td>
</tr>
</tbody>
</table>

#### Table 2

**Key Thermal Reaction occurring in a rock (modified after Somerton, 1992).**

<table>
<thead>
<tr>
<th>Temperature range (°C)</th>
<th>Mineral</th>
<th>Reaction</th>
</tr>
</thead>
<tbody>
<tr>
<td>25–220</td>
<td>Clay minerals</td>
<td>Desorption</td>
</tr>
<tr>
<td>400–723</td>
<td>Clay minerals</td>
<td>Decomposition</td>
</tr>
<tr>
<td>573</td>
<td>Quartz</td>
<td>$\alpha\beta$ transition</td>
</tr>
<tr>
<td>700–830</td>
<td>Carbonate</td>
<td>Decomposition</td>
</tr>
<tr>
<td>790–908</td>
<td>Clay minerals</td>
<td>Decomposition</td>
</tr>
</tbody>
</table>

#### Table 3

**Overview of the rocks reviewed for comparative study.**
used as a major building material in structures of historical importance, for instance, the Red Fort, House of the President of India, the Indian Parliament and the Buddhist Stupas in Sanchi and Sarnath (UNESCO, 2015). Therefore, it is imperative to study the effect of high temperature on the geomechanical properties of Dholpur sandstone. The study can also help in developing a protocol for the restoration of these structures in cases such as building fires. Cylindrical samples were recovered from the sandstone blocks by coring perpendicularly to the bedding plane. Thereafter, the samples were cut to attain length-to-diameter ratios of 2:1 and 0.75:1 as specified for the respective compressive and tensile tests (ASTM, 2008; IS, 2001; ISRM, 1981). Grinding the ends of the samples on a face grinder ensured that the two planar ends were not only parallel to one another, but also perpendicular to the longer dimension.

3.2. Thermal treatment

The nature of thermal treatment and various experimental considerations, play an important role on the thermo-mechanical response of specimen of any rock type. Parameters governing the thermal treatment, namely rate of heating, duration of exposure at the target temperature, nature and the rate of cooling, bear a huge impact on the way the rock performs under compressive and tensile loads. Since heating leads to a change in the microcrack network, fast or slow heating of the sample would bear diverse impacts on the process of microcracking (Den‘gina et al., 1993; Sirdesai et al., 2016b; Tian et al., 2015). High rate of heating or cooling (quenching effect), lead to the formation of thermal stresses which lead to premature failure. Therefore, a slow and steady rate of heating has been proposed by many researchers while investigating the effects of temperature on the various geo-mechanical properties of rocks (Brotóns et al., 2013; Gautam et al., 2016; Sirdesai et al., 2016a; Yavuz et al., 2010). Additionally, the mechanical properties are sensitive to the duration for which the rock sample is exposed to the target temperature. While shorter durations could restrict the interaction of heat with the entire volume of the sample, exposure to longer durations leads to advanced microcracking and thermal damage. Studies conducted by various researchers have concluded that a rock sample should be exposed to the target temperature for a minimum of 20 min before conducting geotechnical tests (Dmitriyev, 1972). Some of the rocks reviewed from the literature were tested under high temperature conditions, whereas, others were tested after having undergone high temperature treatment. A detailed description of the thermal treatment provided to the rocks chosen for comparative study can be seen in Table 4.

Specimens of Dholpur sandstone tested in this study were heated for a duration of 120 h (5 days) in a high temperature furnace at a rate of 5 °C/min to a temperature of 1000 °C, at intervals of 100 °C. The dependence of geomechanical properties on the thermal condition can be studied by testing the specimen both under and after having undergone high temperature treatment. This was achieved by treating two sets of samples at a particular temperature. Each set comprised of at least three samples. The set of samples that was tested under high temperature conditions was named NC (No cooling). The other set was allowed to cool in open air for five days. Since this set was tested after having undergone the cooling treatment, it was named WC (With cooling). A detailed flowchart illustrating the thermal treatment and experimental procedure can be seen in Fig. 2.

3.3. Methodology for strength tests and strain measurement

The effect of temperature on the compressive and tensile strength was determined by performing Uniaxial Compressive Strength (UCS) tests and indirect tensile tests using a Brazilian Disc. The tests were performed both under high temperature conditions and after having undergone high temperature treatment. All the tests were performed in accordance to the guidelines outlined by ISRM (1981). For the UCS testing, the samples were loaded in compression in an Instron 5982 Universal Testing Machine (UTM) under a constant loading rate of 0.1 mm/min. The UTM has been fitted with a load cell and displacement transducer which record the load and displacement, respectively. The setup for the simultaneous heating-loading experiment along with failed Dholpur sandstone specimens that had been tested at room temperature, can be seen in Fig. 3.

The NC specimens up to 600 °C were tested for their compressive and tensile strength in an Instron Environmental Chamber as seen in Fig. 3(a). Above 600 °C, a tubular furnace, Instron SF-16, as seen in Fig. 3(b), was used for maintaining the temperature at the desired value. With the help of these furnaces, the temperature was maintained at the target value during the entire course of the experiment. Both the environmental chamber (used up to 600 °C) and the columnar furnace (> 600 °C) were used in order to maintain the testing condition at the target temperature. Before the commencement of tests, both the systems were allowed to heat-up to the target temperature. The samples were placed into the testing arrangement only after the desired temperature was achieved. No samples were placed or tested in the testing arrangement during the heat-up session. Additionally, in order to nullify the loss of heat during the transport of samples from the muffle furnace to the testing arrangement, the samples were allowed to soak at the target temperature in the testing arrangement. The surface temperatures of the samples were monitored with the help of 3 thermo-couples placed at random locations on the sample. The tests were performed once the sample attained the target temperature of testing. A
failed specimen from both compressive and tensile test that was performed at room temperature can be seen in Fig. 3(c) and (d), respectively.

Since the NC samples were tested without the use of strain gauges, the axial displacement was measured from the data obtained from the displacement transducer. By analysing this data, the axial deformation can be estimated for an NC sample. However, since the calculations include a factor of the machine stiffness, the value of the deformation would be an estimate and would not reflect the true nature of the sample. Axial and lateral deformations in the thermally treated WC samples were measured by using strain gauges. Strain gauges provide a deep insight on the deformational and the elastic characteristic of the rock. Therefore, the Young's modulus and the Poisson's ratio were calculated only for the WC samples. Young's modulus was calculated from the data obtained from the axial strain gauge, by measuring the gradient of the linear portion (elastic zone) within the stress-strain curve (see Fig. S1 in Supplementary material). In order to obtain the representative value of Young's modulus, certain protocols were established for the selection of the linear region. The lower and upper stress limits were set at 5 and 20 MPa, respectively, in order to exclude the non-linear seating and microcracking zones. Similarly, the gradient of the corresponding zone on the lateral strain curve was measured. Poisson's ratio was then calculated as a ratio of the lateral and axial gradients. Furthermore, in cases where the sample failed under 20 MPa, the stress-strain curves were analysed in detail and the interval was accordingly modified. Additionally, the failure mechanism of NC and WC specimens were studied from the load-deformation curves obtained from the UTM.

3.4. FDXM and XRD analysis

Strength and the mechanical properties are closely governed by the internal microstructure of rocks. Structural and chemical changes, enlisted in Table 2, lead to a change in the microcrack network thereby altering the rock microstructure. These changes also affect the transport properties of rock bringing about a change to the porosity and the permeability. Changes in porosity were studied by performing FDXM analysis on the rock specimen using a Zeiss Xradia 520 Versa X-ray microscope. The instrument works on the principle of absorption and attenuation in energy of the X-rays that are projected onto the sample from multiple orientations. In this study, the samples were rotated along 360°, during which 3201 projections were captured. Therefore, the effective scanning rate was 1 scan every 0.112° (360°/3201). The samples were scanned vertically which helped in acquiring the data in the form of slices, similar to the CT scanning technique. The equipment operates on the Diffraction Contrast Tomography (DCT) technique. Although the equipment can attain a true spatial resolution of 700 nm, it would require scanning the sample at a higher number of projections.

<table>
<thead>
<tr>
<th>Reference</th>
<th>Tested</th>
<th>Rate of heating (°C/min)</th>
<th>Duration at target temp. (Hour)</th>
<th>Cooling treatment</th>
<th>Max. temp. (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Hajpál, 2002)</td>
<td>After</td>
<td>a</td>
<td>6</td>
<td>F</td>
<td>900</td>
</tr>
<tr>
<td>(Zhang et al., 2009)</td>
<td>Under</td>
<td>2</td>
<td>2</td>
<td>–</td>
<td>800</td>
</tr>
<tr>
<td>(Liu and Xu, 2015)</td>
<td>After</td>
<td>10</td>
<td>2</td>
<td>F</td>
<td>1000</td>
</tr>
<tr>
<td>(Tian et al., 2014)</td>
<td>After</td>
<td>0.83b</td>
<td>2</td>
<td>F</td>
<td>1000</td>
</tr>
<tr>
<td>(Liu et al., 2016a; Liu et al., 2016b)</td>
<td>After</td>
<td>0.16c</td>
<td>24</td>
<td>F</td>
<td>1200</td>
</tr>
<tr>
<td>(Hajpál and Török, 1998)</td>
<td>After</td>
<td>a</td>
<td>6</td>
<td>F</td>
<td>900</td>
</tr>
<tr>
<td>(Ranjith et al., 2012)</td>
<td>Under</td>
<td>5</td>
<td>2</td>
<td>–</td>
<td>950</td>
</tr>
<tr>
<td>(Li et al., 2017)</td>
<td>After</td>
<td>30</td>
<td>0.5</td>
<td>F</td>
<td>900</td>
</tr>
<tr>
<td>(Mao et al., 2009)</td>
<td>Under</td>
<td>2</td>
<td>2</td>
<td>–</td>
<td>800</td>
</tr>
<tr>
<td>Current study</td>
<td>After (WC)</td>
<td>5</td>
<td>120</td>
<td>A</td>
<td>1000</td>
</tr>
<tr>
<td>Current study</td>
<td>Under (NC)</td>
<td>5</td>
<td>120</td>
<td>F</td>
<td>1000</td>
</tr>
</tbody>
</table>

F = Samples cooled in furnace.
A = Samples cooled in air at atmospheric conditions.
a = 1 h required to reach target temperature.
b = 50 °C/h.
c = 1 °C/h.
Compressive strength of the samples was calculated using the following formula:

\[ \sigma_c = \frac{P}{\pi r^2} \]

where, \( \sigma_c \) is the uniaxial compressive strength of the specimen (MPa), \( P \) is the peak load at failure (N) and \( r \) is the radius of the specimen (mm).

Fig. 4(a) represents the stress-strain curves obtained for the samples that have been tested under high temperature (NC samples), whereas the curves for the samples that have been tested after having undergone high temperature treatment (WC samples) can be seen in Fig. 4(b). The NC samples tested at different temperatures displayed a similar nature, as seen in Fig. 4(a). Following are the characteristics which can be seen in these curves:

1. All the curves display a concave-up shape from the start of the test up to the point of failure, signifying the absence of plasticity.
2. A brittle mode of failure is observed for all the samples marked by a distinct drop in the stress levels.
3. No significant post failure behaviour can be observed and no regular pattern can be seen in the strain-to-failure.
4. Since, the induction of plasticity was not seen at any temperature, no comments can be made so as to signify a temperature as the transition point. Therefore, no CTZ exists for the NC samples.

However, the nature of the curves changes drastically for the heat-treated WC samples. Following observations can be made for the WC samples:

1. The curves for the temperatures 25 to 600 °C have a similar nature and display the compression, elastic deformation and brittle failure phases. All the curves within this temperature range have a concave-up shape.
2. Between 600 and 900 °C, the curves have a concave-up shape from the initial loading point. However, the nature changes to convex-up when the rock approaches the point of failure, signifying the initiation of plasticity. Post failure behaviour can be observed for all the curves within this temperature zone.
3. At 1000 °C, the nature of the curve changes completely. The shape of the curve is convex-up from the start up to the failure point. The rock displays distinct post failure behaviour and behaves like a plastic material.
4. The strain-to-failure increases with increase in temperature.
5. The change in the nature of the curves and the drastic reduction in strengths beyond 500 °C, indicates the transition from entirely elastic to semi-plastic, thereby establishing 500 °C as the CTZ for the ‘heat-treated’ Dholpur sandstone.

While the strength of the samples tested at high temperature (NC) does not vary by a large extent till 1000 °C, the effect of heating and cooling is very profound and can be seen by a noted decrease in compressive strength of the WC samples after 500 °C (Fig. S2, Supplementary material).

4.2. Young’s modulus and Poisson’s ratio

All the heat-treated WC specimen was attached with strain gauges for the measurement of axial and lateral strains. The strain gauges were attached in a manner shown in Fig. 5(b) and the data from these strain gauges was recorded using a multichannel data acquisition system. The
system was calibrated with the UTM so as to facilitate simultaneous recording of both load and strain. The effect of temperature on the elastic modulus and the Poisson’s ratio of thermally treated WC Dholpur sandstone can be seen in Fig. 5(a) and (c), respectively. As observed in the case of compressive strength, the UCS of the sample reduces drastically above 500 °C. This can also be seen as a distinct reduction in elastic modulus and as an increase in the Poisson’s ratio at temperatures > 500 °C. The change in the geomechanical properties at temperatures > 500 °C, reconfirms the existence of a transitional point at that temperature. Additionally, as seen in Fig. 4 (b), the induction of plasticity occurs at temperatures > 500 °C. Therefore, the Critical temperature (CT) for the heat-treated WC samples, exists at 500 °C. A summary of the results from the mechanical tests have been presented in Table 5.

4.3. Indirect tensile strength test

Tensile strength of all the specimen was measured using the Brazilian Disc Method and was calculated using Eq. (2).

\[ \sigma_t = \frac{2P}{\pi Dt} \]  

(2)

where, \( P \) is the peak load at failure (N); \( D \) and \( t \) are the diameter and thickness of the disc, respectively (mm).

The effect of temperature is prominent on both the NC and the WC samples, with the tensile strength decreasing monotonically with rise in temperature. A plot of tensile strength vs. temperature for both the NC and WC specimen can be seen in Fig. S3 (Supplementary material). The tensile strength of both the specimen at 1000 °C is less than half of that at room temperature. The effect of temperature is prominent in heat-treated WC specimen, as was the case in UCS tests as well. The results of the tensile tests can be seen in Table 5. It can also be seen that the rate of decrease in strength increases after 500 °C. Thus, it can be restated that the transition temperature for the heat-treated WC samples is 500 °C.

4.4. Cohesion and internal angle of friction

The effect of thermal treatment on the cohesion and internal angle of friction can be studied by analysing the results obtained from the compressive and tensile tests using the Mohr-Coulomb failure criterion. The study involves the extrapolation of the failure envelope into the tensile region (negative-x axis), as seen in Fig. S4. Cohesion and the internal angle of friction (\( \Phi \)) are obtained by measuring the y-intercept and the angle of the tangent (Goodman, 1989; Labuz and Zang, 2012). However, it should be noted that the value obtained from such
calculated would only represent the estimated values of cohesion and angle of friction. Accurate values of these properties can be achieved by analysing the Mohr-Coulomb failure envelope with the help of triaxial tests. In the absence of experimental equipment which can perform triaxial tests at temperatures higher than 400 °C, the method presented in this study was used to calculate estimates of cohesion and angle of friction. In this study, the average values of UCS and tensile strength obtained for each temperature were plotted and corresponding cohesion and angle of friction were measured. Summary of the results can be seen in Table 5. Change in cohesion and the internal angle of friction with temperature can be seen in Fig. 6(a) and (b) respectively. The effect of temperature on cohesion is minimal till 500 °C, with an increase observed for the NC samples tested at 100 °C. Reduction in cohesion, however, occurs after 500 °C for both the NC and the WC samples. Cohesion at 1000 °C is less than one third of that observed at room temperature for the WC samples. The effect of temperature on the internal angle of friction is relatively chaotic. However, few observations can be made by analysing the results listed in Table 5 and Fig. 6(b). For the NC samples, a steady increase in the friction angle can be observed after 500 °C. The change in the friction angle of the NC and WC samples at temperatures > 500 °C is divergent in nature. None-theless, the angle of friction is higher at 1000 °C when compared to room temperature.

4.5. FDXM and XRD analysis

Mineral grains and their arrangement within the rock are susceptible to changes in pressure and/or temperature. Since the geo-technical properties of rocks are dependent on the mineral grains and their arrangement within the rock microstructure, it is necessary to study and quantify the changes caused by temperature.

The change in microcracks affects the transport properties of rock, namely the porosity and the permeability. Changes in texture of the rock microstructure and the evolution of pores and fissures were studied by performing non-destructive in-situ imaging of the rock in a FDXM instrument. The reconstructed image of all the samples studied in the FDXM analysis can be seen in Table 6a. Since the volumes of the pores is a function of the volumes of the connected (V_{CP}) and non-connected pores (V_{NCP}), phenomenon of microcracking can be analysed in detail by studying the change in V_{CP} and V_{NCP} and their ratio. The ratio, \( V_{CP}/V_{NCP} \), reflects the state of the internal texture of the rock and therefore, any change in this ratio would signify the effect of thermal treatment on the microstructure. The ratio of the connected and non-connected pore volume at each tested temperature can be seen in Table 6b.

Plots representing the effect of temperature on the total, connected and non-connected pore volumes and the porosities can be seen in Fig. 8(a) to (f). As the temperature rises from 25 to 400 °C, a distinct drop can be seen in all the measured volumes. This can be attributed to the closure of the inherent microcracks due to the dilation of the mineral grains. The closure of the microcracks continues till 400 °C. This can also be seen in Fig. 7. The microstructure appears relatively more compact than at 25 °C. The volumes increase on further heating to 600 °C. The reversal in trend is attributed to the formation of microcracks evolving from the continuous expansion of the mineral grain. The microcracks initially appear at the boundaries of the mineral grains and later appear within in the grain. Microcracking is largely governed by the anisotropy in the thermal properties of different minerals. The specimen at 600 °C appears damaged. The damaged sustained by the grains is caused due to the various chemical and phase changes listed in Tables 1 and 2. Differential thermal expansion coupled with chemical and phase changes leads to the formation of thermal stresses within the grain and the microstructure. As the thermal stress builds up, cracks start originating within the grains and the internal structure appears damaged and relatively loose. At 1000 °C, the rock encounters the highest level of thermal stress which leads to the cracking and crumbling of the grains.

On heating from 25 to 400 °C, the volumes of both the connected and the non-connected pore decrease. However, there is sharp decrease in the volumes of the non-connected pores, which can be seen in Fig. 8(c). This causes an increase in the ratio of \( V_{CP}/V_{NCP} \), signifying the reduction in the scatter of isolated pores. Even though the total pore volume at 400 °C was less than that at room temperature, the volume occupied by connected pores was four times higher than that of connected pores. Further heating beyond 400 °C leads to the generation of new connected and non-connected pores. However, a sharp decrease in the ratio suggests that the formation of non-connected pores is higher than the connected pores. As the temperature rises, due to further dilution of the mineral grains, more non-connected microcracks are created. Coalescence of microcracks occurs and this leads to an increase in the volume of the connected pores. At 1000 °C, the rock grain sustains the highest damage in the form of disintegration, giving rise of new non-connected pores. The ratio of \( V_{CP}/V_{NCP} \) at 1000 °C is slightly higher than that at room temperature. The evolution of microcracks as an effect of heat treatment and vertical cross-section of the samples can be seen Figs. S5 and S6 provided in the supplementary material.

In order to observe the chemical changes, the specimen was
analysed using an X-ray diffractometer and the results are summarised in Table 7. As stated in the section 3, Dholpur sandstone is a monomineralic rock with quartz as the dominant mineral. Microcline, a potassium rich alkali feldspar, is the second dominant mineral. The only distinct change in the chemical composition can be seen in the percentages of these minerals. Since the clay and carbonates minerals are present in trace quantities, no distinct change is observed in the XRD results. Thus, it can be established that chemical change does not contribute to the thermal deterioration of the rock specimen. Evolution of microcracks can thus be attributed to the textural changes caused by thermal expansion of mineral grains and mineralogical changes resulting from the quartz inversion. Together, microcracking and textural changes in the rock microstructure are the primary cause behind the deterioration of the geomechanical properties at elevated temperatures.

Fig. 7. Reconstructed 3D in-situ images of the scanned samples obtained from the FDXM analysis.

Table 6a
Change in effective and total porosity.

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Volumes (μm³)</th>
<th>Effective porosity</th>
<th>Total porosity</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Whole</td>
<td>Solid</td>
<td>Pore</td>
</tr>
<tr>
<td>25</td>
<td>2.50E+08</td>
<td>1.98E+08</td>
<td>5.19E+07</td>
</tr>
<tr>
<td>200</td>
<td>2.50E+08</td>
<td>2.09E+08</td>
<td>4.17E+07</td>
</tr>
<tr>
<td>400</td>
<td>2.51E+08</td>
<td>2.19E+08</td>
<td>3.20E+07</td>
</tr>
<tr>
<td>600</td>
<td>2.54E+08</td>
<td>2.16E+08</td>
<td>3.82E+07</td>
</tr>
<tr>
<td>800</td>
<td>2.48E+08</td>
<td>1.78E+08</td>
<td>6.98E+07</td>
</tr>
<tr>
<td>1000</td>
<td>2.51E+08</td>
<td>1.74E+08</td>
<td>7.76E+07</td>
</tr>
</tbody>
</table>
4.6. Study on the thermal damage

The damage caused to the rock can be quantified by studying the change in the elastic modulus of the rock. The elastic behaviour of rock at room temperature disappears progressively in the presence of temperature and the rock behaves as a pseudo-plastic material. Therefore, thermal damage was established as a function of elastic modulus (Hueckel et al., 1994a, 1994b; Mao et al., 2009). Thermal damage $D(T)$ can be calculated for the heat-treated WC samples using Eq. (5) and can be seen in Table 5 and Fig. S7.

$$D(T) = 1 - \frac{E_T}{E_0}$$

where, $D(T)$ is the thermal damage, $E_T$ is the Young's modulus of the specimen treated at temperature $T$ (GPa) and $E_0$ is the elastic modulus at room temperature (GPa).

As seen from the FDXM and XRD analyses, geomechanical and pore properties of Dholpur sandstone are largely dependent on the condition of microcracks within the rock. Since Dholpur sandstone is chemically inert, the damage caused by thermal treatment would be directly dependent on the state of microcracks at a particular temperature. In order to understand thermal damage and the various governing factors, the results of UCS, elastic properties, cohesion and total pores were analysed simultaneously. The various phenomena that affect the variation in elastic modulus and thereby the thermal damage is as follows:

1. Evaporation of pore water: heating to 200 °C leads to evaporation of free water present in the inherent microcracks. A reduction in strength and elastic properties is seen at 200 °C. Further heating to 400 °C, leads to the expansion of grains, which has a healing effect. The expanding grains fill the pores and fissures, rendering the rock relatively more compact and dense. The porosity of the rock is minimal at this temperature. A distinct increase in strength and the elastic properties is seen between 200 and 400 °C. Young's modulus at 400 °C is higher than that at room temperature. Therefore, thermal damage at 400 °C is negative.

2. Microcracking: between 400 and 600 °C, a rapid generation of microcracks occurs due to the continuous expansion and phase transition of the minerals. This leads to a loss in strength and the elastic properties. Creation of new microcracks leads to an increase in porosity. Signs of plasticity can be seen beyond this temperature zone.

3. Thermal disintegration: above 600 °C, the volume of microcracks increases sharply due to the formation of intergranular and intragranular cracks. Within this temperature range, the grain suffers substantial damage leading to disintegration. As the grain disintegrates, it loses the cohesion. The degree of thermal damage increases with increase in temperature.

In order to understand the process of thermal damage, it is important to conduct a detailed study of the rock sample at various temperature. Detailed studies on the correlation of thermal damage to the variations in geomechanical properties have been conducted (Mao et al., 2009; Tian et al., 2014; Zhang et al., 2013; Zhang et al., 2005). Such studies can later be used to design and develop numerical models for simulating reservoir conditions.

5. Discussion

Goodman (1989) studied the behaviour of rocks under compression and suggested the various stages occurring during a compression test. At the start of the compression test, applied load results in the initial closure of pores and fissures resulting in a concave-up 'seating' phase, as seen in Fig. S1 as section 'A'. The seating phase can be prominently observed in uniaxial tests due to the absence of $\sigma_2$ and $\sigma_3$ which lead to the rapid closure of the fissures (Tian et al., 2014). Beyond this phase, the load translates into a linear section on the curve, wherein the rock behaves as an elastic material, which continues until point B. At point B, the applied load generates new microcracks within the volume of the sample and causes an increase in the lateral strain. A stable growth in microcracks with corresponding increase in the lateral strain is
observed between points B and C. The yield point of the sample corresponds to point C (Bieniawski, 1967a, 1967b; Goodman, 1989). However, on the application of further load, the growth in microcracks becomes unstable and an increase in the density of microcracks is observed which results in an increase in both the axial and lateral strain. Point D represents the ultimate strength of the rock. Loading beyond point D would result in the generation of macrocracks that are formed due to the merging of microcracks. The rock loses its capacity to withstand load and further application of load would result in a monotonous increase in both the lateral and axial strain. Similar behaviour can be observed for the specimen of Dholpur sandstone at temperatures below 500 °C (Fig. 4). Both the NC and WC samples display the characteristic stages of a compressed elastic material. However, on the onset of plasticity at temperatures above 600 °C, we can see a distinct change in the nature of the stress-strain curves. The heat-treated WC specimens display characteristic plastic-like behaviour above 700 °C, thereby exhibiting post-failure behaviour.

As observed and reported by several researchers, the geomechanical response of rocks is dependent on the state of microcracks within the rock specimen. Microcracking at high temperature is governed by mineralogical and chemical changes which occur within the rock. Rock specimens rich in quartz are highly susceptible to the phenomenon of quartz inversion (Glover et al., 1995; Hajpál and Török, 2004; Lü et al., 2009; Ranjith et al., 2012). The conversion of low (α) quartz to high (β) quartz at 573 °C is known as quartz inversion. The phenomenon is a mineralogical change and is associated with a volumetric increase of 2% due to the difference in the densities of α and β quartz. The volumetric increase is associated with a linear expansion of 0.7% (Kerr et al., 2004; Schacht, 2004). Volumetric increase in the mineral grains leads to the development of microcracks around the grain. Although the effect of quartz inversion is reversible, it is governed by the environmental factors such as temperature and pressure. Slow cooling at atmospheric pressure can eliminate the effect of α-β quartz transformation without resulting in any visible change to the morphology of the specimen. However, at faster cooling rates, the microstructure of the specimen endures a large amount of shock in the form of thermal stresses, which leads to the development of microcracks. Since the WC samples were cooled at room temperature, the high rate of cooling leads to the generation of thermal stresses which in turn accelerates the process of microcracking. Results obtained from the FDXM analysis confirm the phenomenon of microcracking. Increase in pores and fissures after 400 °C leads to a decrease in the load bearing capacity of thermally treated Dholpur sandstone. Difference in the behaviour of the strength vs. temperature curves of NC and WC samples can therefore be attributed to the accelerated microcracking which occurs on cooling the samples (see Fig. S2, Supplementary material).

The strengths of most of the rock types reviewed in this article decrease with increase in temperature, as seen in Fig. 9. Formation and extension of microcracks is the primary reason for the decrease in strength (Hajpál, 2002; Hajpál and Török, 1998; Liu and Xu, 2015; Mao et al., 2009; Ranjith et al., 2012). However, the strength parameters of certain rock types such as mudstone (HvM) and claystone (WCl) display a peculiar behaviour at high temperatures. The increase in UCS, in the case of mudstone, was attributed to the coupled effect dehydroxylation reactions occurring in clay minerals and the decomposition of siderite present in the mudstone matrix, which lead to the stiffening of the mudstone specimens. Microcracking at high temperatures could not be observed in the HvM mudstone samples (Liu et al., 2016a; Liu et al., 2016b). In the case of WCl claystone, the UCS increases with increase in temperature. However, a sharp decrease can be seen after 800 °C. The chaotic nature of the trend observed for WCl claystone is governed by the volume of microcracks present inherently. Evaporation of water at lower treatment levels and decomposition of clay minerals at higher temperatures, leads to formation and closure of microcracks. The claystone, however, loses its load bearing capacity at 1000 °C due to dilatation and coalescence of microcracks (Tian et al., 2014).

With the formation and enlargement of microcracks, the cohesion decreases. This causes slippage between the rock forming minerals. Extension and coalescence of microcracks lead to a decrease in the elastic modulus of the rock. As seen from stress-strain analysis, the rock behaves like a plastic material at temperatures above 700 °C. Extensive lateral expansion under compression can also be noticed at high temperature, as seen in Fig. 5c. The formation of microcracks and the advent of plasticity damages the internal stability due to the deterioration of the mineral grains. The damage caused to the grains was observed by performing FDXM analysis. Pore volume studies were also performed with the help of this analysis and it can be seen that, the total pore volume was the least at 400 °C. The mineral grains expand which leads to the closure of microcracks. The rock becomes relatively more compact and therefore, the elastic modulus and thereby, the thermal damage is the least at 400 °C. A comparative study of the elastic and damage properties of various types of sedimentary rocks can be seen in Fig. 10.

Effect of thermal treatment on the elastic modulus of all the rocks is similar except in the case of HvM mudstone and the WCl claystone. As seen in Fig. 9, there is a huge increase in the strength of HvM mudstone and WCl claystone due to the various chemical transformation occurring in the clay minerals at high temperatures. These transformations render the rock relatively stiffer and therefore there is an increase in the elastic modulus. In general, the rocks could display any one of the following trends after high temperature treatment.

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**Table 7.** Mineralogical composition of Dholpur sandstone at various temperatures.

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Quartz (%)</th>
<th>Microcline (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>91.2</td>
<td>8.8</td>
</tr>
<tr>
<td>500</td>
<td>93</td>
<td>7</td>
</tr>
<tr>
<td>600</td>
<td>92.3</td>
<td>7.7</td>
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<tr>
<td>700</td>
<td>93.6</td>
<td>6.4</td>
</tr>
<tr>
<td>800</td>
<td>93.6</td>
<td>6.4</td>
</tr>
<tr>
<td>900</td>
<td>92.9</td>
<td>7.1</td>
</tr>
<tr>
<td>1000</td>
<td>93.5</td>
<td>6.5</td>
</tr>
</tbody>
</table>

---

**Fig. 9.** Comparative study on the effect of temperature on (a) UCS, (b) Tensile strength.
1. Increase in geomechanical properties up to the critical temperature (CT) followed by a sharp decrease as seen in the PS, XS, MS, HS sandstones, HvM mudstone and the WCl claystone.

2. Minimal change up to the CT followed by a sharp decrease as seen in the QS sandstone and both the NC and WC specimens of Dholpur sandstone.

3. Slow monotonic up to the CT followed by a rapid decrease as seen in the DS sandstone.

4. Combination of above three trends till CT followed by a rapid decrease as seen in the HvS sandstone and the XL limestone.

Since a large variation is seen in the behaviour of geomechanical properties of specimens belonging to the same rock type, it is therefore imperative to perform a detailed study on the thermo-mechanical behaviour of rocks.

6. Conclusions

The UCS and tensile strength tests which were carried out under and after high temperature treatment condition showcase the consequences of nature of thermal treatment and testing condition on the mechanical response of the Dholpur sandstone. As observed for the heat-treated With-cooling WC specimen, the nature and response of the stress-strain curves vary at different temperatures. The specimens tested at temperatures below 500 °C exhibit features characteristic to any elastic material. However, plasticity sets in at higher temperatures. This causes a sharp decrease in strength and the larger strains can be observed at relatively lower values of compression thereby exhibiting a plastic behaviour. The values of elastic modulus also increase till 400 °C and the calculated thermal damage suggests that damage is minimal at 400 °C. This can be attributed to the compaction of the sample due to the dilation of mineral grains. However, unconstrained expansion at higher temperatures leads to the formation of microcracks (intergranular and intragranular) which renders the rock relatively weak causing a loss in strength and stiffness. Microcracks were studied in detailed by performing textural (FDXM) analyses which suggest that the pores and fissures evolve as the treatment temperature rises. Increase in the volume occupied by pores, leads to an increase in porosity. Studies on porosity were conducted by analysis the volumes of connected (VCP) and non-connected (VNCP) pores. Increase in temperature up to 400 °C leads to the expansion of grains which fill in the inherent pores thereby causing a decrease in total pore volume. Both the VCP and the VNCP with increase in temperature, however there is a rapid fall in VSCP till 400 °C. The increase in VCP/VSCP ratio till 400 °C suggests that volume of occupied by the connected pores is higher than that occupied by the non-connected pores. However, the trend reverses upon further heating. This not only suggests the onset of microcracking at 400 °C but also suggests the expansion and phase transformation of grains causes the creation of non-connected cracks at a relatively higher rate. Dilatation and coalescence of microcracking adds to the effective and the total porosity of the rocks. Dholpur sandstone being chemically inert does not experience great change due to chemical reactions which occur at high temperatures. The sandstone is mono-mineralic consisting 91.2% quartz and 8.8% microcline at untreated room condition. Although, both the minerals are chemically inert at high temperature, phase transformation from α to β quartz takes place at 573 °C. Volumetric expansion associated with quartz inversion leads to the creation of gaps and crevices in and around the quartz grain. Although the phenomenon is reversible, conversion from β quartz to α quartz occurs at slow rates of cooling. Since the WC samples were cooled under room temperature conditions, the generation of thermal stresses within the microstructure leads to microcracking. This further emphasizes the claim that the damage caused in Dholpur sandstone is purely textural rather than chemical. However, no signs of plasticity could be observed in the No-cooling NC specimens of Dholpur sandstone which were tested under high temperature conditions in this study. A dip in the strength, observed after 500 °C, is followed by a rise and the strength of the NC specimen at 1000 °C is slightly lower than that at untreated room condition. However, the strain endured by the rock up to failure increase monotonically till 1000 °C.

The varied mechanical response of rocks of the same lithology calls for a detailed study before the commencement of any project. Case specific models should be prepared before designing structures with or within these rocks. Such a model would subsequently prove useful in establishing restoration protocols in fire damaged buildings.

Appendix A. Supplementary data

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.enggeo.2017.06.010.

References


