Investigation of the microstructural characteristics of heated sandstone by micro-computed tomography technique

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Abstract
Heritage buildings always pose challenges due to experiencing high temperatures and pressure over time. Sandstone is one of the common sedimentary rock types used for these buildings. Therefore, it is very important to understand the microstructural variations of rocks associated with these constructions along with the mechanical variations. In this study, the microstructural and mechanical alteration of selected types of sandstones is investigated after it is heated from room temperature to 800 °C. Micro X-ray computed tomography (µXCT), X-ray diffraction (XRD), scanning electron microscopy (SEM), thermogravimetry (TG) and derivative thermogravimetry (DTG) techniques were used to identify the physical, chemical, mineralogical and microstructural changes of sandstone after different heat treatments. The mechanical alteration of the heated rock specimens was also studied using the point load index (PLI). The main changes in microstructure were observed when the sandstone’s temperature was greater than 400 °C. The total porosity measured by µXCT of sandstone increased by more than 70% at 800 °C compared to its porosity at room temperature. When the temperature increases, the open porosity increases while the closed porosity decreases. Noticeable changes in rock mineralogy were identified at temperatures exceeding 400 °C, which can be attributed to the phase transition of quartz, decomposition of feldspar and dehydroxylation of kaolinite. The TG and DTG analyses and point load index were in good agreement concerning these changes in rock mineralogy. Microstructural variation is one of the main reasons for the discrepancy in threshold values between different sandstones.

Keywords Microstructure · Micro cracks · Porosity · Mineralogy · Thermal analysis

Introduction
Sandstone is one of the most common types of sedimentary rock and is easily found. Therefore, it has a long and extensive history as a construction material in different projects such as buildings, tunnels, underground structures, pavements, warehouses and hydraulic systems (Chen et al. 2020; Gautam et al. 2016; Sirdesai et al. 2018). Sandstone also used as an aggregate in concrete (Bosnjak 2014). However, this sandstone used in concrete is highly volatile in higher temperature environments. In Australia, numerous heritage buildings are constructed with sandstone (Pitt et al. 2013; Moreira 2006; Ip et al. 2008). These buildings need to be conserved properly over time. In this conservation process, authorities always face the challenge of deciding whether the stone needs to be preserved or reconstructed (Pitt et al. 2013; Ip et al. 2008). When searching for the most suitable conservation process, it is necessary to understand how the rock becomes degraded. Thermal damage is one degradation method that so far has not been thoroughly studied. The heritage buildings in Australia are at high risk of fire damages due to bush fire (Department of Agriculture, Water and the Environment 2021). As an example, the Lithgow State Mine Museum and the Genoa Schoolhouse Museum were damaged due to the bushfire that happened in 2020 (Blue Shield Australia 2020). Understanding the thermal behaviour of sandstone would be important to decide whether fire-damaged stones should be replaced or not. This depends in part on the characteristics of the particular kind of sandstone used.

A good amount of research has already been conducted on the effect of high temperature on the engineering properties...
of various types of rocks and changes have been identified in these properties with higher temperatures (Darot and Reuschlê 2000; Dmitriyev et al. 1969; Gratchev et al. 2019; Lu et al. 2020; Pathiranagei and Gratchev 2021; Ranjith et al. 2012; Somerton 1992; Wu et al. 2019a; Yu et al. 2020). Several researchers have also studied the effect of high temperature on microstructure and mineral composition (Hajpál and Török 2004; Kong et al. 2019; Meng et al. 2019; Yang et al. 2019). These studies highlighted that changes in the engineering properties of rocks are directly governed by their microstructural properties such as porosity, pore interconnectivity and micro cracks (Isaka et al. 2019; Mahanta et al. 2020b). Thus, comprehensive studies about the microstructural properties of rocks after high-temperature treatments are necessary because of the results therefrom.

Recently, image-based digital petrophysics techniques, such as computed tomography (CT), have been extensively used to study the microstructural properties of rocks (Cnudde et al. 2006; Dong and Blunt 2009; Fan et al. 2020, 2018; Isaka et al. 2019; Jin et al. 2019). The µXCT technique has been used to find out microstructural changes (pore network changes and micro-crack propagation) in various rocks such as limestone (Mohammadmoradi et al. 2017), shale (Goral et al. 2015), sandstone (Dong et al. 2008) and coal (Zhang et al. 2019). In the past, some authors used thermogravimetric (TG) analysis to study the different aspects of thermal behaviour of rocks such as evaporation of absorbed water, phase changes of minerals and recrystallization of minerals (Gautam et al. 2019; Mahanta et al. 2020a; Sirdesai et al. 2017). The SEM technique has been recognized as a convenient technique widely used to identify rock damage and fracture properties (Sun et al. 2017; Xue et al. 2018; Yang et al. 2020; Yavuz et al. 2010; Zhang and Zhao 2013; Zuo et al. 2017).

Mahanta et al. (2020b) studied the microstructural changes and pore network configuration of three thermally treated sandstones using µXCT techniques and found that microstructural responses of these sandstones depend on the availability of clay minerals. Mahanta et al. (2020a) investigated the microstructural thermal damage and different pore characteristics of sandstone using µXCT techniques. They obtained valuable details about connected and non-connected pores and their alterations with temperature. Several authors (Fan et al. 2018; Isaka et al. 2019; Kamali-Asl et al. 2018; Sufian and Russell 2013; Zhao et al. 2017) recently studied the microstructural changes of granite under different thermal treatments. It is evident that CT imaging techniques have been comprehensively applied in recent studies to identify the microstructural properties of different rock formations in different geological environments, thus further research using this technique is essential.

Tripathi et al. (2021) studied the effects of high temperatures on the microstructural, physico-mechanical and elastic properties of sandstone. They identified that the rock strength, porosity, and Poisson’s ratio changed due to microscopic and mineralogical changes. Ersoy et al. (2021) investigated the influence of mineralogical and microstructural changes on the physical and strength properties of thermally treated clayey rocks. They concluded that the most important parameters for the behaviour of heated rocks are the mineral composition, microcrack and primary voids.

Even though changes in microstructural characteristics of rock formations under different heating/cooling treatments have been studied by several recent researchers using different techniques, there is still a huge knowledge gap concerning some microstructural properties of sandstone under high-temperature conditions. For example, Tripathi et al. (2021) stated that the threshold temperature of the same rock is found to be similar (The threshold temperature is recognised as the temperature at which the physical, mechanical and mineralogical properties of rock change significantly (Sirdesai et al. 2017; Yang et al. 2017b; Hu et al. 2018). Moreover, they emphasized that the range of threshold temperature of clastic sedimentary rock (sandstone) varies between 600 °C and 800 °C. In sharp contrast to the above statement, a range of threshold temperatures was found for sandstone, varying from 225 °C (Kong et al. 2016), 250 °C (Rao et al. 2007), 400 °C (Lan 2009; Liu and Xu 2015; Sygala and Bukowska 2019; Wu et al. 2005; Zhang and Yuan 2019; Pathiranagei et al. 2021) to 500 °C (Ranjith et al. 2012; Zhang et al. 2009). The reason for different thresholds in the same rock type is still unknown. A possible reason is that this discrepancy in thresholds may be due to microstructural defects in the rock. A detailed study of microstructural characteristics of sandstone under different high temperature and cooling treatments has not yet received much attention due to the limitations of suitable advanced instrumentation. Moreover, there is still a lack of knowledge about the influence of clay content of rock on the strength enhancement with temperature. As an example, Ersoy et al. (2021) mentioned that the strength of clayey rocks increases with rising temperature. Pathiranagei et al. (2021) observed similar behaviour for a sandstone type which has only 12% of clay. They confirmed that the mineralogical and microstructural changes of rock directly influence its strength properties. However, the clay content’s influence on rock’s strength enhancement when exposed to temperature has not yet been comprehensively studied.

There are three purposes for using µXCT analysis in this study: to understand the internal damage of sandstone caused by heating, to identify the pore structure of the sandstone and to obtain three-dimensional imaging of sandstone rock at different heating stages in a non-destructive way. The XRD analysis is important as it provides details of petrological changes of sandstone due to heat treatment. The TG and DTG analyses are useful as they give information about the
mass loss and chemical changes of sandstone at different heating temperatures. The point load index values are used in this study as they give basic idea about the strength variation of rock with temperature. Therefore, this study comprises an attempt to investigate the pore structural changes, micro-crack propagation, mineral composition changes, mechanical behavioural changes and chemical alterations of sandstone under various high-temperature treatments with the support of µXCT techniques, SEM analysis, XRD techniques, PLI analysis, TG and DTG analysis. The detailed investigation includes the observation of porosity, pore geometry and topology parameters as a function of temperatures.

**Experimental program**

**Sample preparation**

The tested sandstone sample was obtained in the Nerang area of the Gold Coast, Queensland, Australia. This sandstone belongs to the Neranleigh-Fernvale Beds and it was formed in carboniferous period. As mentioned by Willmott (1983), the Neranleigh-Fernvale Beds formed from deep-sea sediments about 290 million years ago. The results of the XRD analysis indicated that the sandstone was mainly composed of 51% quartz, 40% feldspar, 7% kaolinite and 2% illite. The sandstone is a fine-grained material with grains distributed from 0.12 to 0.25 mm. The images of the intact rock sample are indicated in Fig. 1.

A total of four cuboid samples of 1 cm height, 1 cm width and 4 cm length were prepared for µXCT tests since smaller samples are required to obtain high resolutions (Deprez et al. 2020). In addition, the XRD analysis and TG/DTG analysis were performed on powdered samples to determine the mineralogy and chemical changes of the rock specimens. To conduct the SEM analysis carbon-coated thin sections were prepared. The twenty cubic samples (5 cm×5 cm) were prepared for a point load test. The summary of sample preparation for each test is shown in Table 1.

**Heat treatment**

To prevent any thermal shock during the treatment, a slow heating rate was used. The sandstone samples were first heated in a high-temperature furnace applying a 5 °C/min increase until the nominal temperature (400 °C, 600 °C and 800 °C) was reached. Once the target temperature was reached, the samples were kept in the furnace for another 2 h under the same temperature condition for uniform heat distribution. Then the samples were cooled down to room temperature naturally inside the furnace. For this purpose, after the furnace turning off, the heated samples were allowed to be inside the furnace until they reached room temperature (letting sample to cooling down at a slow rate without any thermal damage). Previous research has indicated that 2 h would provide sufficient time to expose the rock specimens to high temperatures (Chaki et al. 2008; Ersoy et al. 2017; Glover et al. 1995; Xiong et al. 2018). Therefore, we used a heating duration of 2 h.

| Test method | Number of samples | Geometry            |
|-------------|-------------------|---------------------|
| Micro-CT    | 4                 | Cuboid samples (1 cm×1 cm×4 cm) |
| XRD         | 4                 | Powdered samples     |
| TG/DTG      | 1                 | Powdered samples     |
| SEM         | 4                 | Thin section of samples |
| Point Load test | 20                | Cubic sample (5 cm×5 cm) |

Fig. 1 Intact sandstone specimen; (a) test sample (b) closed image of sample (c) SEM image of sample
Testing procedure

The testing procedure consisted of four procedures: mineralogical observation, thermal analysis, microstructural observation and mechanical observation. Mineralogical variations were observed by using the XRD analysis. For thermal analysis, TG and DTG tests were conducted. Microstructural variations of rock sample were identified using µXCT and SEM analysis. The changes in mechanical properties were observed using the point load index. All the rock specimens were tested under dry conditions. The testing procedure for µXCT, SEM and XRD tests are explained in Fig. 2.

XRD investigation

A Philips X’Pert multipurpose XRD diffractometer (Bruker, D8 Advance Eco) with Cu K-alpha radiation (a wavelength of 1.5406Å) was used. The diffraction pattern was measured in 2θ ranging from 5 to 80° and was recorded with a step size of 0.007°. The X-ray pattern was acquired in the Sietronics data format. To convert the data format into a readable Excel format, the ConvX (Convex 2022) was used. The minerals were identified using the position (2θ) and intensity data of peaks in the XRD graphs using the origin software (Origin Pro 2022).

Thermal analysis

TG and DTG analyses were performed on the powdered samples to recognize the alterations of their mass and chemical processes with temperature. These analyses were conducted using a Netzsch STA 449F3 Jupiter simultaneous thermal analyser (QUT Engineering 2022). During the experiment, a 5 °C/min heating rate was used, and the experiment was carried out in an air atmosphere.

µXCT investigation

Image Acquisition: µXCT imaging was performed using a SkyScan 1272 (Version 1.1.19, Bruker, Belgium) scanner, to quantify and visualise the effect of high temperature on the pore structure and the crack distribution in four sandstone samples at various temperatures. The samples were scanned at 100 kV and 100μA with an exposure time of 1100 ms and 4 × 4 binning with an effective pixel size of 10um. A 0.11 mm copper filter was used with a frame averaging of 4 and 0.3° rotation step and 360° rotation around the sample. The number of total projections is 1200.

Image processing: The datasets were reconstructed using NRecon (Version 1.7.3.1, Bruker, Belgium) and InstaRecon (Version 2.0.4.6, InstaRecon, USA) using a Feldkamp algorithm. Beam hardening correction and ring artefact reduction were applied to the dataset. A combination of manual and automatic co-registration was performed in DataViewer, to align the samples within the sample group at different time points so that identical volumes would be analysed. This was necessary as the scanned field of view was smaller than the sample.

During the process of optimisation, numerous filters were applied to the datasets. These included Kuwahara, median, conditional mean and unsharp masking. On visual inspection of the filtered data (segmented and unsegmented) it was determined that there was a loss of micro crack/pore edge definition with all these processing methods. Whilst there was an overall decrease in noise within the solid components of the sample, the downside of losing the details of the air component was too significant to warrant filtering. Also, it was obvious through visual evaluation that, at the chosen segmentation threshold, any noise present within the solid portions of sandstone did not contribute to the porosity of the sample.

Pore space analysis: Results were analysed using CTAn (Version 1.18.8.0+, Bruker, Belgium) and visualised using CTVol (Version 2.3.2.0, Bruker, Belgium) software. A lower
threshold of grayscale value 52 was applied to every image and the entire cropped image was analysed for open and closed porosity. The greyscale value chosen for the segmentation was determined firstly with visual evaluation, then further verified by comparing the open porosity values from micro-CT analysis with the TG, DSC and DTG analysis. Open and closed porosity is determined automatically by the CTAn analysis software. After loading the entire dataset into the memory, a three dimensional map of all the pores was created, and any pore connected to the edge of the volume of interest was considered open.

**SEM investigation**

To identify the microstructural characteristics after this heat treatment of sandstone, a TESCAN Mira 3 scanning electron microscope (TESCAN Mira 3 2016) was used. The observations of the SEM test were collected under different magnifications (from 200 to 20,000) to analyse the variations in the microstructure.

**Point load investigation**

Using the digital point-load test apparatus (ELE INTERNATIONAL 2022), the point load tests were performed according to the AS 4133.4.1-2007 (Standards Australia 2007). Five specimens from each temperature from 25 °C to 800 °C with 200 °C increments were tested. The average point load values for each temperature were used to study the strength characteristics of the rock.

**Results**

**Effect of high temperature on rock mineralogy**

When preparing the samples for XRD, the samples were powdered after each heat treatment. Figure 3 shows the XRD spectroscopy of sandstone treated at different temperatures. As indicated in the graph, the sandstone mainly contained quartz, feldspar, kaolinite, and illite. There was an increasing trend of quartz peaks as if “new” quartz appeared and a slightly decreasing trend of feldspar peaks with the temperature increase. Moreover, when the temperature increased above 400 °C, the kaolinite peaks disappeared due to their dehydroxylation process (Pathiranagei and Gratchev 2021). These changes in the clay minerals directly affected the cementation bond between the mineral particles in sandstone. The changes in quartz and feldspar peaks mainly occurred after 400 °C due to the phase transition of quartz and decomposition of feldspar (2KAlSi3O8/2NaAlSi3O3 = K2O/Na2O + Al2O3 + 6SiO2) (Pathiranagei and Gratchev 2021). The triagonal α phase of quartz transformed to a hexagonal β phase at around 573 °C and the decomposition of feldspar occurred between 500 °C and 760 °C (Liu et al. 2019a, b). Hence, when the rock sample was heated to 600 °C and 800 °C, the quartz and feldspar peaks showed substantial changes.
Thermal analysis

Figure 4 shows the TG and DTG curves of one sample, which represents all the sandstone samples used in this study. The TG curve indicates a 0.65 mg mass loss between the temperatures of 400 °C and 800 °C. The TG curve sharply declines between 440 °C and 600 °C, which indicates the dehydroxylation of kaolinite, decomposition of feldspar and transition of quartz (Pathiranage and Gratchev 2021). Corresponding to this significant mass loss, the DTG curve shows an endothermic (energy using) peak at 486 °C, which indicates the dehydroxylation of kaolinite. These changes in the TG and DTG curves imply that the main structural damage in the sandstone rock occurred within this temperature zone. The main chemical reactions which happened during the phase transition of quartz are due to thermal expansion of the framework structure and can be explained as follows. When the temperature increases, the distance between Si–Si increases as a result of cation repulsion. Due to this process, the angle which connects the two Si–O tetrahedra increases from 150 °C (α phase) to 180 °C (β phase).

This data correlates with the results of the XRD analysis (major changes in quartz, feldspar and kaolinite peaks are obvious after 400 °C). When the temperature increased to 800 °C, more quartz, feldspar and kaolinite particles tended to react and more variations in rock mineralogy can be observed (an increase in the intensity of quartz peaks, a decrease in the intensity of feldspar peaks).

Effect of high temperature on rock microstructure

The µXCT images and SEM images of sandstone specimens subjected to different heat treatments were analysed to identify the microstructural changes of rock at higher temperatures. Figure 5 illustrates the 2D µXCT images of sandstone rock after different heat treatments. When comparing the room temperature and higher temperature images of the rock, the presence of new micro cracks at elevated temperatures can be identified. The number of micro cracks increases with the rise in temperature, the highest number of micro cracks can be identified at 800 °C due to the thermal stress that was created as a result of exposure to the high temperatures. It can be noted that significant changes in the micro-crack propagation were identified in the µXCT images of sandstone specimens above 600 °C. However, the changes in micro cracks at higher temperatures cannot be identified clearly due to the low magnification of the 2D µXCT images. Therefore, it is hard to distinguish whether these cracks are intergranular or transgranular. High-resolution SEM images of the heated sandstone will provide more information about the micro cracks.

Figure 6 illustrates the representative SEM images of the rock microstructure at different magnifications. It can be seen that the intact sample contained some pores and cracks, however, large quartz grains seem closely packed and well cemented (Fig. 6(i)) when compared to the heat-treated specimens. As indicated in Fig. 6(j) and Fig. 6(n), when the rock sample was heated to 400 °C, the finer grains became loose, and newly-formed intergranular cracks and pores can be identified (Fig. 6(b)). At 600 °C, more intergranular cracks and pores were generated. In addition, transgranular cracks also appeared (Fig. 6(c)) and the weakening of cementation between the quartz grains can be observed (Fig. 6(g)). As shown in Fig. 6(d) and Fig. 6(h), for the sample treated at 800 °C, the pore spaces between the grains, the intergranular and the transgranular cracks were increased.

Fig. 4 Thermogravimetry and derivative thermogravimetry curves of the sandstone
significantly. Figure 6(i) and (m) show the disorder flakes of clay minerals in the SEM images of the original sandstone sample. Figure 6(j) and (n) indicate the increase in disorder flakes of clay minerals which may be due to the induction of kaolinite. However, after 600 °C and 800 °C, the SEM images with magnifications of 5000 and 20,000 showed more clarity and indicated the decomposition of kaolinite (Fig. 6(k), (l), (o) and (p)).

The changes in the sandstone’s microstructure with a rise in temperature can be explained as follows. As a result of the different thermal expansion coefficients of the constituent minerals, thermal stress is generated inside the sandstone structure. When this thermal stress reaches the ultimate strength of the sandstone, micro cracks start to form. The thermal stress increases with raised temperature and this causes more micro cracks. Ultimately, when the sandstone sample is heated to a higher temperature, the intergranular and transgranular cracks are formed within the rock structure. Simultaneously, the micro pores are created inside the structure, as a result of the decomposition of clay minerals (decomposition of kaolinite) as shown in Fig. 6(g) and (h). These changes in microstructure with higher temperatures will ultimately cause the deterioration of rock engineering properties.

**Effect of high temperature on porosity**

The physical and mechanical alterations of the rock structures are either the result of the propagation of micro cracks or the changes in the chemical composition of the minerals, which cause a further increase in the pore spaces in the rock structure (Mahanta et al. 2020b). The pore spaces of sandstone specimens heated to 400 °C, 600 °C and 800 °C were analysed.

Table 2 shows the 3-D images of the pore matrix of the four sandstone samples after each heat treatment. There is a scattered distribution of pore spaces (micro cracks) at 25 °C (room temperature) and 400 °C for all four samples. However, at 600 °C and 800 °C, the pore network is denser (more connected pores) than at temperatures below 600 °C. In Table 3, open pores and closed pores of Sample 1 are indicated (green for open pores and red for closed pores). The table shows that the open pore matrix gets denser with the increase of temperature while the closed pore matrix becomes less dense with the same treatment.

Figures 7, 8 and 9 illustrate the variations in total porosity, open porosity and closed porosity over different treatment temperatures, respectively. These figures indicate a slight reduction of 1% at 400 °C for the average total porosity, compared to that at room temperature. However, the average total porosity values at 600 °C–800 °C increased by 20% and 72%, respectively, compared to the room temperature values. In particular, it was identified that the majority of the pore spaces which developed under heat treatment were open pores. At 600 °C–800 °C, a significant difference in the open and closed pores was observed. In addition, noticeable thermal damage (micro crack development) occurred at treatment temperatures greater than 600 °C.
The porosity variations with the increase of temperatures can be explained by the following. The slight decrease in porosity below 400 °C is associated with the evaporation of the bound water and attached water that are present in the sandstone sample (Fang et al. 2019; Wu et al. 2019a, b). During this temperature range, minerals undergo a certain extent of thermal expansion, which results in a reduction in porosity. Within the temperature range of 300 °C–500 °C, the escaping of crystal water and structural water cause increased porosity (Wu et al. 2019b). The changes in the DTG curve at 486 °C, correspond to the dehydroxylation of kaolinite.

In addition, the main mineral components of the tested sandstone are quartz and feldspar. The phase transition of

![Fig. 6 SEM images of sandstone after different heat treatments](image)

**Fig. 6** SEM images of sandstone after different heat treatments
quartz at around 573 °C and decomposition of feldspar in the temperature range of 500 °C to 760 °C may have mainly contributed to the porosity increment after 400 °C (Liu et al. 2019a, b; Sirdesai et al. 2017). The mass loss in the TG curve between 400 °C and 800 °C corresponds to this result.

Furthermore, the increasing trend of open porosity at higher temperatures (T > 400 °C) may be due to the generation of new pore spaces in the rock because of the formation of new micro cracks. The discrepancy in behaviour of open porosity and closed porosity at higher temperatures may be due to the chemical alteration of clay minerals (Mahanta et al. 2020b). The increase in open porosity indirectly indicates the increase of rock permeability at higher temperatures.

**Effect of high temperature on point load strength**

As indicated in Fig. 10, the average point load index value of sandstone decreases with increasing temperature. The slight decrease in point load index can be identified up to 400 °C, and then it decreases by almost 10% up to 600 °C. Relatively higher reduction (by 37%) in PLI shows up to 800 °C. It can be identified that the change in the point load index correlates with the porosity. As an example at 800 °C, it is noted that sandstone shows the highest porosity and lowest strength values. Similar behaviour was observed by other researchers such as Kong et al. (2016), Wu et al. (2005), Ersoy et al. (2007), and Tang et al. (2019), indicating the softening pattern with increasing temperatures. The strength characteristics with temperature can be explained

| Table 2 3D images of pore matrices of all the scanned sandstone samples |
| Sample 1 | Sample 2 | Sample 3 | Sample 4 |
| RT | ![3D image of RT sample 1](image1) | ![3D image of RT sample 2](image2) | ![3D image of RT sample 3](image3) | ![3D image of RT sample 4](image4) |
| 400°C | ![3D image of 400°C sample 1](image1) | ![3D image of 400°C sample 2](image2) | ![3D image of 400°C sample 3](image3) | ![3D image of 400°C sample 4](image4) |
| 600°C | ![3D image of 600°C sample 1](image1) | ![3D image of 600°C sample 2](image2) | ![3D image of 600°C sample 3](image3) | ![3D image of 600°C sample 4](image4) |
| 800°C | ![3D image of 800°C sample 1](image1) | ![3D image of 800°C sample 2](image2) | ![3D image of 800°C sample 3](image3) | ![3D image of 800°C sample 4](image4) |

Green colour- Open pores
Red colour- Closed pores
Table 3  Open pores (green colour) and closed pores (red colour) of Sample 1

| Sample 1 |          | Open porosity | Closed porosity |
|----------|----------|---------------|-----------------|
| Temperature | Pore Structure |               |                 |
| 25°C     | ![Image](image1.png) | 11.8%         | 1.2%            |
| 400°C    | ![Image](image2.png) | 13.1%         | 1%              |
| 600°C    | ![Image](image3.png) | 15%           | 0.8%            |
| 800°C    | ![Image](image4.png) | 17.7%         | 0.6%            |

**Fig. 7** The variation of total porosity (measured using µXCT technique) with temperature (The results for sample 2 at 800 °C is not available)

**Fig. 8** The variation of average open porosity (measured using µXCT technique) with temperature (The results for sample 2 at 800 °C is not available)
by denoting the porosity as follows: due to the changes in mineral properties (less compactness) at higher temperatures (600 °C and 800 °C), existing cracks are developed and new cracks are created, this increase the porosity and decreases the strength.

Discussion

Figure 11 summarizes all the changes of sandstone after different heat treatments. Even though the SEM, µXCT and XRD tests were performed at four different temperature points (25 °C, 400 °C, 600 °C and 800 °C), the summary contained additional temperature points as the thermal analysis (TG and DTG) was conducted continuously from 25 °C to 800 °C. Accordingly, 400 °C can be identified as the threshold temperature which changes the physical, mechanical and mineralogical properties of the sandstone significantly. The chemical reaction of major minerals (between 500 °C and 600 °C) and the increased rate of micro-crack development (T > 400 °C) caused significant alterations in rock microstructure at 600 °C and 800 °C.

As mentioned before, the threshold temperature of sandstone varies from 225 °C to 500 °C according to previous research works (Kong et al. 2016; Lan 2009; Liu and Xu 2015; Ranjith et al. 2012; Rao et al. 2007; Sygala and Bukowska 2019; Wu et al. 2005; Zhang et al. 2009; Zhang and Yuan 2019). As indicated in Fig. 11, it seems that the rock’s mineralogical changes and chemical reactions play a major role in the variations in the rock’s microstructure. This is proved by the findings of Ranjith et al. (2012). The sandstone they studied had 17% kaolinite and they found that major changes in its mechanical properties occurred beyond 500 °C (thus the threshold temperature was 500 °C). The main reason for this change is the dehydroxylation of kaolinite at around 500 °C which weakened the rock structure. Same kind of behaviour obtained in this study. Sandstone indicates around 10% variation in strength between 400 °C and 600 °C, which may be due to the decomposition of kaolinite and phase transition of some of the quartz minerals. The reason for the 37% strength reduction between 600 °C and 800 °C is phase transition of quartz minerals and the decomposition of feldspar. As explained by Wu et al. (2019a, b), the sensitivity of sandstone to thermal loading is governed by its porosity and thermal expansion ratio between the minerals. If the rock contains minerals which has a larger discrepancy in the thermal expansion coefficient, then it experiences severe damage (Wu et al. 2019a, b). In the studied sandstone, quartz mineral plays a major role, as it shows a comparatively larger thermal expansion coefficient than the other minerals. Therefore, the main contributor to the strength reduction of the sandstone is quartz mineral, which represents 51% of rock mineralogy.

The variation of strength with porosity shows in Fig. 12. It can be identified that rock strength linearly decreases with the porosity. The following correlation between the point load index and porosity (Eq. (1)) was obtained using a regression analysis:

$$PLI = -0.0664n + 1.9379, R^2 = 0.9973$$

Where PLI is the point load index, $n$ is the porosity of sandstone. The linear relationship between the point load index and porosity indicate that those two variables have a direct connection.

Therefore, microstructural variations can be identified as one of the main reasons for the discrepancy in threshold values between different sandstones.

Tripathi et al. (2021) observed a strength increment of clayey rock with rising temperature. Pathiranagei et al. (2021) obtained the same behaviour for the Jimboomba sandstone (12% clay). They mentioned that the mineralogical and microstructural changes of rocks directly influence its strength properties. The mineral composition of sandstone tested in this
By analysing the current test results, the main reason for the strength variation of the sandstone can be explained by the larger thermal expansion coefficient of the quartz minerals. However, the strength increment of clayey rocks may also be mainly due to the ceramic-like behaviour of clay mineral as observed by Tripathi et al. (2021).

**Conclusions**

In this study, a series of µXCT, SEM, XRD, TG/DSC/DTG, point load experiments was performed on sandstone specimens heated to a range of temperatures (400 °C,
600 °C and 800 °C). Based on the obtained results, the following major conclusions can be drawn:

1. Significant changes in the rock microstructure were observed at temperatures greater than 400 °C. Numerous new discontinuities (cracks and pore spaces) occurred in the sandstone specimens, resulting in substantial damage to the rock microstructure.

2. The µXCT data revealed that the total porosity of sandstone increased by more than 70% at 800 °C compared to its porosity at room temperature (25 °C). The open porosity of sandstone increased with greater temperatures while the closed porosity decreased.

3. The major changes in rock mineralogy occurred at temperatures greater than 400 °C when the quartz peaks increased, and the feldspar peaks significantly decreased. In addition, there was a significant decrease in the amount of clay minerals (i.e., kaolinite) at very high temperatures. These changes in the rock mineralogy correlated with the TG and DTG analyses, which indicated a noticeable mass loss after 400 °C. This mass loss was attributed to the phase transition of quartz, decomposition of feldspar and dehydroxylation of kaolinite.

4. The significant changes in rock strength (strength decreased by 37%) happened after 400 °C, which may be occurred as a result of mineralogical changes and porosity enhancement.

The outcome of this study can be used by decision makers of the heritage buildings to understand the microstructural changes of heated sandstone. From that, they can decide whether fire-damaged stones should be replaced or not.

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Declarations

Conflict of interest “The authors have no relevant financial or non-financial interests to disclose.”

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