Thermal alterations in experimentally-flaked stone tools from Olduvai Gorge and their relevance for identification of fire in the Early Stone Age

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Abstract

The use of fire represents a landmark development in the technological evolution of the genus *Homo*. However, the earliest use and control of fire is challenging to identify in the archaeological record. Olduvai Gorge in Tanzania presents some of the best preservation and volume of sites in the Oldowan and Acheulean, but has not yet shown any clear indication of the presence of fire in lower or middle Pleistocene deposits. Through the use of visual observation and optical and scanning electron microscopy, this study identified signature features of thermal alteration in experimental stone tools of quartzite, lava and chert collected from Olduvai Gorge to establish how fire-modified rocks may potentially be identified in the archaeological record using a non-destructive methodology that can be replicated in future research.

Keywords: stone tool thermal alteration; Olduvai Gorge; hominin-controlled fire; scanning electron microscopy

1. Introduction

The controlled use of fire as a technology represents a significant milestone in the hominin evolutionary lineage, as a marker of progress in cognition and a necessity in the cooking and processing of dietary materials (Aiello and Wells, 2002, Aiello and Wheeler, 1995, Carmody and Wrangham, 2009, Pennisi, 1999, Wrangham, 2017, Wrangham and Carmody, 2010). However, the identification of fire in the early archaeological record remains a challenge. The association of early *Homo* and the use of fire is tenuous at best, with extensive use of fire being more generally associated with later members of our Genus , and becoming widespread only during the Middle Palaeolithic / Middle Stone Age (Roebroeks and Villa, 2011). The earliest evidence for the control of fire is strewn across a nearly one million year time span, with claims from 1.5-0.8 Ma across East and Southern Africa (Clark and Harris, 1985, Hlubik, et al., 2017, Beaumont, 2011, Gowlett, et al., 1981, Chazan, et al., 2017, Ecker, et al., 2018).

Evidence of fire has traditionally been identified through stratigraphic evidence of hearths, fire pits, as well as burnt bones and lithic materials. In the African record, lithics in particular have been the subject of extensive research with regard to purposeful heat-treatment from the Middle Stone Age, to determine the benefits of heating to improve fracture mechanics and knapping properties (Domański and Webb, 1992, Domański, et al., 1994, Mourre, et al., 2010). However, the focus has primarily been on chert and other siliceous materials that are more intensively used during the Later Stone Age / Upper Palaeolithic (Domański and Webb, 2007), and rarely employed in pre-Middle Stone Age contexts. Chert, however, was only available during a short time and in limited quantities at Olduvai Gorge (Kimura, 1999, Stiles, et al., 1974, Stiles, 1998, Proffitt, 2018, Ludwig and Harris, 1998, Hay, 1976) , rendering the classically recognised features of heating in lithics

(Crabtree and Butler, 1964) less accurate a signifier for the long Oldowan and Acheulean sequence, in which lavas and quartzite are the predominant raw materials (Leakey, 1971; Hay, 1976).

Heat dehydrates and produces changes in lithic material properties. Scanning electron microscopy (SEM) provides structural information of the surface of a sample with high resolution. In this pilot study, scanning electron microscopy was used to capture and evaluate the effects of heating on experimentally thermally altered chert, quartzite, and lava experimental artefacts from Olduvai Gorge at sequentially- increased temperatures and durations of exposure. These images were assessed using grey level histogrammetry, which produces a relative measure of roughness on a material surface. It provides a method of evaluating heating of stone tools without damage to archaeological material. As quartzite and lava are more frequently represented in the Early Stone Age of East Africa than chert, the possibility of identifying thermally modified lithics is dependent on new methodologies that go beyond traditional studies focused on heating of siliceous rocks. The present study utilises these techniques to assess the potential for the identification of thermally altered archaeological material from the Early Stone Age at Olduvai Gorge and determine the viability of the method for further research on a larger sample size.

2. Materials and Methods

2.1 Materials

The sample was selected from knapping experiments conducted at the University College London Lithics Laboratory, using raw materials sourced from Olduvai Gorge, which follow experimental protocols and nomenclature detailed elsewhere (de la Torre, et al., 2013, de la Torre, et al., 2018, Proffitt and de la Torre, 2014, Byrne, et al., 2016). Twelve flakes, ranging from 19.5mm to 40.5mm length, 9.0mm to 27.0mm width were selected, with four flakes from each raw material (see details in Table 1). The chert flakes from Exp. #41 were originally sourced from the MNK outcrop in the Side Gorge. Quartzite flakes from Exp. #14 were sourced from Naibor Soit, to the north of the Main Gorge. The lava flakes from Exp. #17 derive from basalt cobbles collected from a contemporary fluvial conglomerate at Olduvai Gorge. The details of each of these experiments may be found in de la Torre et al. (2013, 2018), Proffitt and de la Torre (2014), and Byrne et al. (2016).

2.2 Preparation

Samples were prepared for initial analysis through measurements and digital macroscopic photography. Imaging of the samples was performed using a Dinolite Premier optical microscope in a relatively low magnification range to complement the high magnification of scanning electron microscopy (SEM) used in this experiment. Optical microscopy was used to capture the crystalline structure of each sample, as well as any existing fractures, identifiable features, or discolouration, prior to and following thermal exposure, over a magnification range of 50 to 230X.

The scanning electron micrographs were captured on a Zeiss EVO 25 microscope. Micrographs were captured at up to four locations on each specimen. Images were taken over a magnification range of 100 to 10,000X. Imaging of the specimens was repeated following the heating process.

Samples were not coated in gold prior to the use of scanning electron microscopy (SEM) due to the intent to produce a method of analysis that would be replicable on archaeological material that could not be modified in this manner due to its palaeoanthropological value.

2.4 Heating

The samples were exposed to heat following the experimental design standardised for evaluating heat-treated lithic materials (Purdy and Brooks, 1971; Domański and Webb, 1992; Mercieca and Hiscock, 2008; Schmidt et al., 2012; Domański et al., 2009). The samples were divided into four groups, as shown in Table 1. The groups were heated to 300°, 400°, 500°, and 600° C respectively, with each raw material type represented in the lower temperature groups, and lava and quartzite only in the 600° group. Chert was excluded from the 600° C group due to the predicted prevalence of fracturing at high temperatures. This range was within the natural range of temperatures produced by controlled wood-burning fires, which reaches upwards of 800° C (Goudie, et al., 1992). The experiment was conducted using a Lenton electric furnace in the UCL Institute of Archaeology Furnace Laboratory, with all samples placed on an asbestos tile, with each sample separated from one another by approximately five centimetres distance. The furnace was equipped with a fan system to aerate the samples.

In each group, temperature increased gradually at a rate of 50° C per hour, until reaching the target temperature. Once the target temperature was achieved, the furnace remained at that temperature for a duration of one hour, at which point it was allowed to cool completely overnight before the pieces were removed.

2.5 Analysis

2.5.1 Optical and Scanning Electron Microscopy

Following the heating of the samples, each sample was analysed visually, using optical microscopy and SEM image analysis techniques. Optical analysis was performed using a 10x magnification lens, as well as comparative analysis on the digital images of the specimens before and after heating, to identify differentiation in colour, fracturing, texture, and structure.

Images captured on the Dinolite Premier optical microscope were compared with one another to assess the extent of thermal damage and fracturing on each piece. Additionally, the samples were assessed to determine whether a change in colour had taken place on the individual grains, inclusions, and instances of concretions on the samples' surfaces.

SEM image analysis was performed on the micrographs of each specimen, prior to and following the heating process. This involved both qualitative observations of the images and quantitative evaluations of the crystal structure visible in the micrographs. Each sample was imaged at the same location on the material surface prior to and following heating.

Grey level histograms evaluate the topography of a grey-scale image as a measure of roughness or disruption of a material's surface (Haha, et al., 2007). Using the United States National Institute of Health software program ImageJ, each pixel is assigned a value from 0, representing black, to 255, indicating white (Rasband, 2014). The histogram function calculates the number of pixels at each value and produces a visual representation of frequency. Thresholds may be assigned to differentiate between particular regions of the surface as needed. Surfaces that are relatively smooth appear as sharp peaks, while those indicating differentiation cover a wider range of values. This technique has previously been demonstrated on SEM micrographs of non-lithic materials (Yang and Buenfeld, 2001, Risović, et al., 2008, Turner–Walker and Syversen, 2002). For the stone tools in this study, the technique was used to determine whether the average roughness of each sample's topography was changed following thermal alteration. The interpretation of the grey level histograms is dependent on the relative brightness and contrast of the images, as well as the necessary consistency of the SEM used, and as such these results must be interpreted with attention to these caveats. Histograms were produced for each micrograph. The data were assembled in Microsoft Excel, where pre- and post-thermal alteration images were analysed at each level of magnification and between specimens and raw material types.

. As the loss of molecularly bound water has been indicated to be the cause of altered fracture toughness and tensile strength (Schmidt, 2014, Schmidt and Fröhlich, 2011, Schmidt, et al., 2012), the presence of hydrogen or silanoles (SiOH) within silicate rocks, in addition to the presence of impurities such as iron, is suggested to be an influence on the parameters required for scanning electron microscopy (SEM) imaging of the raw materials.

Post-alteration raw material in this experiment required higher levels of vacuum pressure and closer focal distances to image the specimens clearly, as well as the presence of visible charging effects at lower magnifications than were present in the preliminary imaging. Values were assessed by quantifying the change in focal length when viewed on the SEM, as a measure of the dehydration of the specimens during heating. These indicators, while suggesting that some change has taken place, cannot quantifiably measure the degree of change experienced by each sample (see data in Table 1).

3. Results

3.1 Macroscopic Changes

3.1.1 Chert

The chert exposed to high temperatures during this study reflected physical alterations believed to be standard following thermal alteration, including fracturing and pot-lidding. However, development or deepening of colour and formation of lustre on the surface did not take place.

Chert showed no visual indication of colour change at 300° or 400° C, as shown in Figures 1 and 2. The 500° C sample (Exp41-86) exhibited a grey tone on the exterior surface. Fracturing increased with higher temperature exposures and was most prevalent at 500° C (Figure 2). The extent of the damage suggests that the Olduvai Gorge chert undergoes initial fracturing between 300° and 400° C with more extensive fracturing at higher temperatures, a result in agreement with previous studies on chert and flint from other regions (Domański and Webb, 2007, Purdy and Brooks, 1971).

The interior of the chert experienced a textural change above 400° C, having a dusty or chalky consistency, presumably from exposure to the heat during fracturing and pot-lidding rather than heat conduction. The external surface did not have macroscopic transformations in texture. The chalky areas were noted to be substantially less reflective than the exterior fracture surface of artefacts.

No lustre or gloss was present on the exterior surface of any of the chert samples, differentiating the Olduvai Gorge material from previously reported characteristics of thermal alteration in chert and flint. As this lustre has been shown to develop over extended periods of exposure to high heat (Rick and Chappell, 1983), lustre was unlikely to develop given the short exposure period of one hour at the maximum temperature used for each sample during heating.

3.1.2 Lava

The lava samples experienced the greatest degree of colour change of the three raw materials following the process of thermal alteration, as shown in Figure 1. The changes observed were dependent on the temperature the sample was heated at, such that darkening and reddening was more prevalent at higher temperatures. There were no macroscopic indications of fracturing or structural damage to the lava samples at any of the temperatures tested.

A number of concretions were exhibited on sample Exp17-56 that were present prior to the experimental process (Figure 2). Comparison with the images taken prior to heating indicated that the concretions had not undergone structural change, as they remained the same size and shape. Digital photography and microscope images of the concretions following heating showed the brightness of the concretions in contrast with the reddened lava surface.

Table 1 indicates a loss of mass of 1% in each of the lava samples post-alteration, in a range of 0.01-0.05g. This loss is attributed to the presence of existing labels on the pre-alteration samples, which were destroyed during the heating process. Neither the chert or quartzite samples had labels prior to the beginning of this experiment and as such were not subject to this influence.

3.1.3 Quartzite

The quartzite samples surface became more opaque following heat exposure, having a cloudy appearance without any colour change. Following heat treatment at 400° and 500° C, samples Exp14-67 and Exp14-69 were identified as having small red accumulations within the quartzite. These accumulations were below the surface and were visible to the naked eye, though microscopic imaging provides a more detailed representation (Figure 2). These features are found to an extent in the pre-heating microscopy. However, they are more clearly visible under minimal magnification following the experimental heating process. This may be due to the clouding of the quartzite following heating, allowing the accumulations to become more conspicuous.

Cracking of the quartzite samples was not extensive, but was easily discernible (Figure 2E and 2F). It was present on all specimens heated at and above 400° C, though the thinner samples Exp14-67 and Exp14-69 (4.5mm and 5mm, respectively) exhibited more frequent instances of fracturing than the more sizable samples Exp14-37 and Exp14-70 (9mm and 7mm in thickness). The cracking in the thinner samples was substantial, presenting fractures upwards of 4mm in length. Unlike the chert samples displaying extensive fracturing, the quartzite samples retained their structural integrity despite the cracking. These findings are in contrast with the conclusions of Goudie, et al. (1992), who suggest heterogeneous macrocrystalline material such as quartzite should not experience fracturing as severely as cryptocrystalline materials . However, the size of the pieces is likely to influence and lower the threshold at which the materials experience structural damage (Mercieca and Hiscock, 2008).

Heating produced a noticeable change in the quartzite texture, with a loosening and/or detachment of approx. 1 mm crystalline fragments on the surface, occurring at a higher frequency on samples Exp14-67 and Exp14-69.

3.3 Scanning Electron Microscopy

3.2.1 Visual Comparison

Visual comparison between the micrographs of each sample before and after thermal alteration provides clearer visualisation of the topography, fractures, and change on the surface of artefacts. Our results however do not indicate the presence of recrystallisation upon heating as hypothesized elsewhere (Domański and Webb, 1992, Domański, et al., 2009).

While macroscopic fracturing was present in the chert samples heated to 400°, no microfracturing was imaged (see Supplemental Figure 1). The highest peak temperature sample in the chert (Exp41-86 at 500° C) indicates macroscopic and microscopic fracturing that is apparent at all magnifications utilised for this experiment, as shown in Figure 2 (see also Supplemental Figure 2). There appears to be no recrystallisation or reshaping of the topography present on any of the chert specimens (see Supplemental Figure 3). Microfracturing was present at higher magnifications on the higher temperature lava samples (Exp17-49 and Exp17-56) indicated in Figure 3 (see also Supplemental Figures 4 and 5). These fractures were not visible through digital photography or optical microscopy. Regarding quartzite, at low magnifications of 100-500X the quartz crystal boundaries are visible, as are cleavage surfaces. Figure 4 indicates damage on sample Exp14-67, heated to 400° C. Minimal difference to the crystal structure following the heating process was identified in the lower temperature samples Exp14-37 and Exp14-67. Exp14-69 was heated to 500° C, nearing the phase transition temperature of 573° C (Rios, et al., 2001, Tucker, et al., 2001). Surface-level smoothing is present in quartzite sample Exp14-70, with fewer ridges on the surface topography visible along the crystal surface, shown in Figure 4. Supplemental Figure 6 indicates fracturing in Exp14-70 following heating to 600° C.

3.2.2 Grey Level Histogrammetry

Disruption of the surface topography was indicated at 5,000X on the chert sample (Exp41-86) heated to 500° C. However, this is likely due to the presence of fracturing in the post-alteration micrographs. At 10,000X, the grey level histograms of this specimen showed no substantial difference (see Figure 5). This result suggests the absence of surface level structural change in the chert samples tested during this experiment. The lava samples revealed more substantial smoothing effects on the topography than the chert.

The lower temperature quartzite samples tested, Exp14-37 and Exp14-67 at 300° and 400° C, showed no differentiation between the grey level histograms before and after thermal alteration (Figure 5). This was expected, as quartzite at this temperature experienced no visually discernible change through macroscopic observation or optical microscopy.

At the highest peak temperature reached (600° C), each of the histograms of sample Exp. 14-70 show higher frequencies of a smaller breadth of pixel values on the post-heating micrographs than those taken of the sample prior to experimental heating, as shown in Figure 5. This is visible at 100X, 500X, and 1000X magnification of the sample surface. These results suggest a smoothing of the crystalline surface structure has taken place.

It should be noted, however, that errors in the production of the histograms may be produced due to the blurring of charging effects, as well as differences between models of SEM and other disturbance to the material during the production of the micrograph.

4. Discussion

An initial evaluation through macroscopic observation, optical microscopy, and SEM has been made to capture the transformations in each of the most common Olduvai lithic raw materials following heating. The use of SEM on non-microcrystalline material as a test for thermal alteration has proven successful in identifying the microfracturing from heat exposure, which is most prevalent in the lava materials. Quantitative analysis of the micrographs through the use of grey level histograms present results that are in alignment with the visual observations: the most substantial differentiation occurs in the lava samples, where macroscopic colour change was present and microfracturing was visible on the SEM imaging. Some differences are also observed in the high-temperature exposed quartzite samples, where surface level smoothing was documented in the micrographs; minimal smoothing to the surface is documented in the chert.

The experimental design for this study prioritised the preservation of lithic samples, such that the methodology could be replicated on archaeological artefacts, and therefore did not include gold coating for improvement of imaging. This decision contributed to the substantial influence of charging effects on the samples during the capturing of the micrographs, as well as reducing the level of magnification that could be used. In the analysis of the micrographs, measures of change in the average size of the crystals within the stone were limited to grey level histograms. The utilisation of additional methods to determine the presence of thermal alteration in lithic materials has the potential to expand on the results of this study. These include Fourier-transformed infrared spectroscopy (FTIR), petrographic colourimetry, and the tests of mechanical change previously discussed.

The use of a furnace for this experiment introduced limitations on assessment of the effects of flames, embers, and variation of heat within a fire, such that these effects could not be evaluated. However, the furnace supplies heat at a consistent rate of increase and is comparatively uniform in its heat distribution, such that each sample underwent the same conditions and the constraints of the experiment were replicable across sample batches. Further studies with larger sample sizes could assess what differentiation is produced between samples of the same raw material in the more variable environment of a controlled fire.

The physical transformations recorded during this study provide notable differences from previous research on the effect of heat exposure on chert. The absence of reddening on the surface is a variation from the standard features of thermal damage described by Crabtree and Butler (1964) and Rick and Chappell (1983), though the pot-lidding and fracturing previously described were produced also in our experiment.

The mass of the samples showed minimal reduction following thermal alteration (Table 1). The absence of weight loss in our experiments following heat treatment is in agreement with previous studies (Clemente-Conte 1997, 527; Schmidt *et al.* 2012, 141; Haaland *et al.* 2017, 84).

Although a variety of lithic material types have been shown to present thermal alteration in varying manners based on mineralogical composition (Oestmo, 2013, Hajpál and Török, 2004), our results highlight the challenges in identifying thermal alteration using mineral composition criteria. The absence of recrystallisation in the Olduvai chert specimens is supportive of a loss of molecularly bound water in the microcrystalline structure (Schmidt, 2012). The expansion and evaporation of this water is attributed to be the cause of microfracturing in the chert structure, as well as any loss of mass in this material. Heated lavas have been demonstrated to be the most clearly identifiable of the specimens tested during this study, due to the extent of the colour change observed macroscopically, and the microfracturing documented in SEM imaging. Quartzite samples are more challenging to identify quantifiably using SEM image analysis or visual indicators, though microscopic smoothing was present on sample Exp14-70.

The evidence of fire in the early archaeological record is fragmentary, with no single site providing a comprehensive demonstration of its presence before 1.0 Ma. The Koobi Fora artefacts identified as fire-modified lavas exhibited pot-lidding and an absence of colour change on the material surface, while chert specimens were found to be reddened in contrast with the original tone of the source material (Hlubik, 2018). In an experimental replication study using Gombe and Asille basalt (Hlubik, et al. (2017), pot-lidding was present while colour change was absent; this fracturing may be due to the higher temperature of 800° C and the longer exposure time of six hours at peak temperature. In our Olduvai experiment results, however, reddening but no potlid fracturing is observed in the lava materials, and there is an absence of colour change in the chert. In contrast, the basalt and trachyte samples from Gadeb 8E (Clark and Harris, 1985, Barbetti, 1986) represent a similar suite of features in heated lavas as were identified in the Olduvai Gorge materials, through red discolouration following heating to an estimated 500° C and brightening and yellow discolouration of concretions on the surface.

The results of this study lay the foundation for identifying fire- modified rocks in the Early Stone Age record at Olduvai Gorge, should it be present. The demonstration of the lack of colour change in the chert and few macroscopically identifiable features in the quartzite samples, in addition to the challenges evaluated in comparative analysis with analogous artefacts, renders visual identification of burnt archaeological lithics from these materials functionally difficult. In turn, the presence of colour change identified in the lava samples tested for this experiment are likely to be heavily obscured by weathering processes. As quartzite is more prevalent as a raw material for stone tool production at Olduvai Gorge , the absence of clearly identifiable features of fire considerably reduces the possibility of finding the effects of fire on materials found at this Early Stone Age locality.

5. Conclusions

Thermally- altered lithic materials in the early archaeological record are present only in limited circumstances and quantities, to the extent that their presence is not definitive as a diagnostic feature for hominin-controlled fire. Determining whether purposeful control of fire took place necessitates a greater volume of combined archaeological evidence than is currently available in the Early Stone Age record. The archaeological material present of this period, including lithics, ash lenses, and soil micromorphology, indicate the presence of fire in the palaeo-landscape (Hlubik, et al., 2017, Berna, et al., 2012), but are insufficient to form conclusions as to hominin control of fire. As identification of hearths in contrast with bushfire-produced alteration of sediments is contentious, and ash lenses are far less prevalent in East African sites than their South African counterparts, conclusions on control of fire require strong evidence to be reported. At present, no conclusive method exists by which to identify whether thermal alteration of fire-modified rock was produced by hominin-controlled fires or natural bushfires.

While the artefacts selected for our experiments correspond to most representative raw materials available at Olduvai Gorge, the sample is still relatively small, and several other rock types exist in the Olduvai Pleistocene sequence. Future research should consider the use of standardised blocks of raw material which would provide more consistent measurement of fracture threshold temperatures, as well as affording the opportunity for tests of tensile strengths and other measures of fracture mechanics as indicated in Domański and Webb (1992). Further, a larger sample size would allow comprehensive statistical analysis of difference within and between raw material types in addition to confirmation of the results found in this pilot study. Given the long archaeological sequence available at Olduvai Gorge, there is potential for an expansion of future studies beyond replication of Oldowan flakes to be inclusive of Acheulean assemblages from Middle/ Upper Beds II,, III and IV, such that they correlated with Acheulean findings (e.g., Alperson-Afil, 2008) which present conclusive evidence of controlled used of fire.

As the results of this study indicate, at Olduvai Gorge visual identification of chert or quartzite material that has undergone thermal alteration is especially challenging, while lava materials are more conspicuous. These challenges are accentuated when considering that our preliminary assessment of archaeological materials show that patination and weathering may disrupt the potential for preservation of the signifiers identified in this study. Research in this field of study is ongoing, and is likely to continue as analytical methods become further refined to produce conclusive results.

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Table 1: Experimental artefacts studied in this paper. Changes in sample mass and SEM focal length following experimental thermal alteration to corresponding temperatures. Samples excluded from measures of focal length were imaged on a different SEM and therefore were not included for this comparison.

Figure 1: Samples used in this study prior to and following thermal alteration. Temperatures and time of exposure to heat may be found in Table 1. A-D) basalt, E-H) chert, I-L) quartzite.

Figure 2: Optical microscope imaging indicates macroscopic transformations to each of the samples following exposure to heat. A) Sample Exp.41-37 showed extensive fracturing following exposure to 400° C. B) Exp.41-19 indicated a reddening on the surface following exposure to 300° C. C) Concretions on Exp.17-56 before exposure to heat, D) Same feature on Exp.17-56 following heating to 600° C. E) Red accumulations within the quartzite sample Exp.14-69. F) Cracking in the quartzite sample Exp14-69 following heating to 500° C.

Figure 3: Sample Exp.41-40 (A and B) showed no evidence of recrystallisation following heating to 400° C. A) surface prior to heating, B) same surface following exposure to heat at 400° C. C) and D) indicate smoothing effect on lava sample Exp.17-56 (C: sample before heating. D: after heating to 600° C). There are fewer ridges visible on the surface of Exp.17-56 at equivalent magnifications.

Figure 4: Quartzite sample Exp.14-70 prior to heating (A) and following thermal alteration past the phase transition point to 600° C, where smoothing effects were present (B).

Figure 5: Grey level histograms indicating surface level changes. Each data curve represents the grey level pixel values of an SEM micrograph plotted against the number of occurrences

for each value. Chert (A and B) shows no indication of recrystallisation or smoothing following heating to 500° C. The lava sample in (C and D) illustrates the effects of microfracturing on the grey level histogram, as the surface is indicated to be rougher following exposure to 500° C. Figures 5E and 5F shows show smoothing effects following heating to 600° C. Figures 4G and 4H show smoothing effects in quartzite following heating to 600° C.

Supplemental Material:

Supplemental Figure 1: Sample Exp.41-40 showed no evidence of recrystallisation after heating to 400°C. Image A represents the sample prior to heating, B shows the same sample following exposure to heat. This chert sample showed no visually identifiable indicators of recrystallisation or other changes in the crystalline structure.

Supplemental Figure 2: Fracturing was present on chert sample Exp.41-86 following heating to 500° C when viewed using SEM at 3000X. At the macroscopic level, the fracturing was extensive and had caused structural damage to the sample.

Supplemental Figure 3: The 500° C chert sample Exp.41-86 showed no recrystallisation or visually distinguishable change in the relative shape and size of crystals on the material surface. Image A represents the sample before heating, and B shows the surface following heating.

Supplemental Figure 4: Microfracturing was present in both the micrographs prior to (A) and after (B) heating to 400° C on lava sample Exp.17-36. Both micrographs represent the sample at 1000X.

Supplemental Figure 5: Fracturing was apparent on the lava sample Exp.17-56 after being heated to 600°C. A) Sample at 500X, B) Sample at 1000X.

Supplemental Figure 6: SEM imaging indicating fracturing corresponding with the macroscopically identifiable indicators of damage to the quartzite sample Exp.14-70 following heating to 600°C, at 500X.