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THE EXPERIMENTAL STUDY OF PALAEOLITHIC HEAT-TREATMENT TECHNOLOGY: A CASE FROM THE SHUIDONGGOU ROCK RESOURCES, NORTH-WEST CHINA*

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The application of heat-treatment technology on lithic raw materials is an important feature of early modern human behaviour. The evidence of heat-treated stone artefacts discovered at Localities 2 and 12 of the Shuidonggou Late Palaeolithic site, North-West China, provides an important example for studying this technology among ancient humans in Asia during the Late Palaeolithic. The mechanism and effects of heat treatment on raw materials and the role of this technology in producing stone tools were studied by means of a simulation experiment and related analytical methods. These facilitated an in-depth analysis of the heat-treatment activities of the Shuidonggou occupants and their implications for cognitive ability and survival strategies of human populations at that time.

KEYWORDS: PALAEOLITHIC, CHINA, HEAT TREATMENT, SIMULATION EXPERIMENT, MECHANICAL PROPERTIES

INTRODUCTION

In the field of Palaeolithic archaeology, *heat-treatment technology* refers specifically to the heating, insulating and cooling of stone raw materials to alter their surface and internal structure to produce preferred characteristics such as texture and colour (Zhou *et al.* 2013b). Application of this technology enabled ancient humans to improve the efficiency and quality of stone tool production and the utilization rate of raw materials (Schmidt *et al.* 2012, 2013). In the Late Palaeolithic of China, which began *c.*30 000–27 000 years ago (Gao 1999, 2013; Gao and Norton 2002) and ended with the beginning of the Holocene, heat-treatment technology was closely linked to humans' cognitive ability and social organization, because of the significant cultural input and the high level of intelligence that this technology required.

To date, various scholars have conducted extensive studies on heat-treatment techniques (e.g.,Clark and Williams 1987; Copeland 1998; Duttine 2005; Domanski and Webb 2007; Mercieca and Hiscock 2008; Brown and Marean 2009; Oestmo 2013; Zhou *et al.* 2013a,b). The three core branches involve understanding the effects of this technology on stone tool production, how it changes the characteristics of raw materials and experimental analyses.

Stone artefacts unearthed at Locality 2 (SDG 2 hereafter) and 12 (SDG 12 hereafter) at the Late Palaeolithic Shuidonggou site in North-West China exhibit clear characteristics of heat

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treatment (Zhou *et al.* 2013b) and thus provide an excellent opportunity for studying this technology. Prior to evaluating the archaeological specimens, we collected raw material samples from sources near the site for use in the experiments and related analytical tests. We designed these experiments to evaluate in detail the changes to the internal and external properties of the main types of rocks found locally (for the location of the collection of rock materials, see Fig. 1), which include dolomite, quartz sandstone, chalcedony and chert. Our goal was to understand the overall effects of heat treatment on the production of stone tools at the Shuidonggou site.

As the comparison study of the experimental and archaeological specimens has been published (Zhou *et al.* 2013b), this paper will focus on the experimental design and its implementation and, therefore, the archaeological details of the Shuidonggou sites and the discoveries of heat-treated archaeological specimens will not be elaborated. Readers interested in the background should refer to Zhou *et al.* (2013b), Li *et al.* (2013a,b), Yi *et al.* (2013), Pei *et al.* (2012) and Guan *et al.* (2011, 2012).

In addition to increasing our knowledge of the internal and external morphological changes in stones, this simulation experiment also helped us to better understand the various structural patterns in stones during the heat-treatment process. This was accomplished by precise control of the temperature used to heat the samples. Our experiment took place in sealed indoor and outdoor environments, and included sessions of both rapid and slow heating–cooling. The main observations included changes in the external properties of the materials, such as their colour, lustre and weight before and after heat treatment, as well as changes in internal properties, such as the degree of crystallization, the microstructure and microcracks.

MATERIALS AND METHODS

The design of the experiment and the application of analytical tests

The appropriate heat-treatment temperatures vary for each type of rock, while different temperatures also affect changes in the properties of the same types. Therefore, temperature control is critical in the experimental heat-treatment process (Flenniken and Garrison 1975; Price *et al.* 1982). For instance, the knapping property of fine chert will increase when this material is heated to 250°C, followed by changes in its colour. However, the colours of coarse chert only change when it is heated to 250–300°C and temperatures of 350–400°C are required to increase its knapping property (Purdy and Brooks 1971; Bleed and Maier 1980; Domanski and Webb 2000, 2007).

Based on archaeological and ethnological evidence, two open-air hearths were built on sandy clay sediment. Hearth B was an open hearth, and hearth A was a stone-lined hearth with ventilators. Some pebbles and flakes were buried in sands at a depth of 3–5 cm in the centre of the hearth to avoid rapid temperature change. The intentionally heat-treated specimens will be compared with the unintentionally heated specimens. In hearth A, the open fire lasted for 10 h, and the pebbles buried with sands cooled slowly: 16 h later, the heated specimens cooled to air temperature. In hearth B, the open fire lasted for 8 h and was then allowed to cool slowly in the open air for 6 h, until the specimens cooled to air temperature. A TM902C thermodetector was used to record the real-time experiment temperature. For further details, see Zhou *et al.* (2013b).

For the indoor segment, we used the GW-300C box-type resistance furnace for heating, which allowed for precise control of the temperature, rate and time of the treatment. The rates of both heating and cooling were relatively slow, with a cooling duration of over 10 h and a maximum temperature between 300°C and 550°C. In addition, to enhance the accuracy of this study, all raw materials were cut into small pieces for heat treatment under different temperatures, to ensure that specimens used in the later stage of examination and comparison originated from the same source.

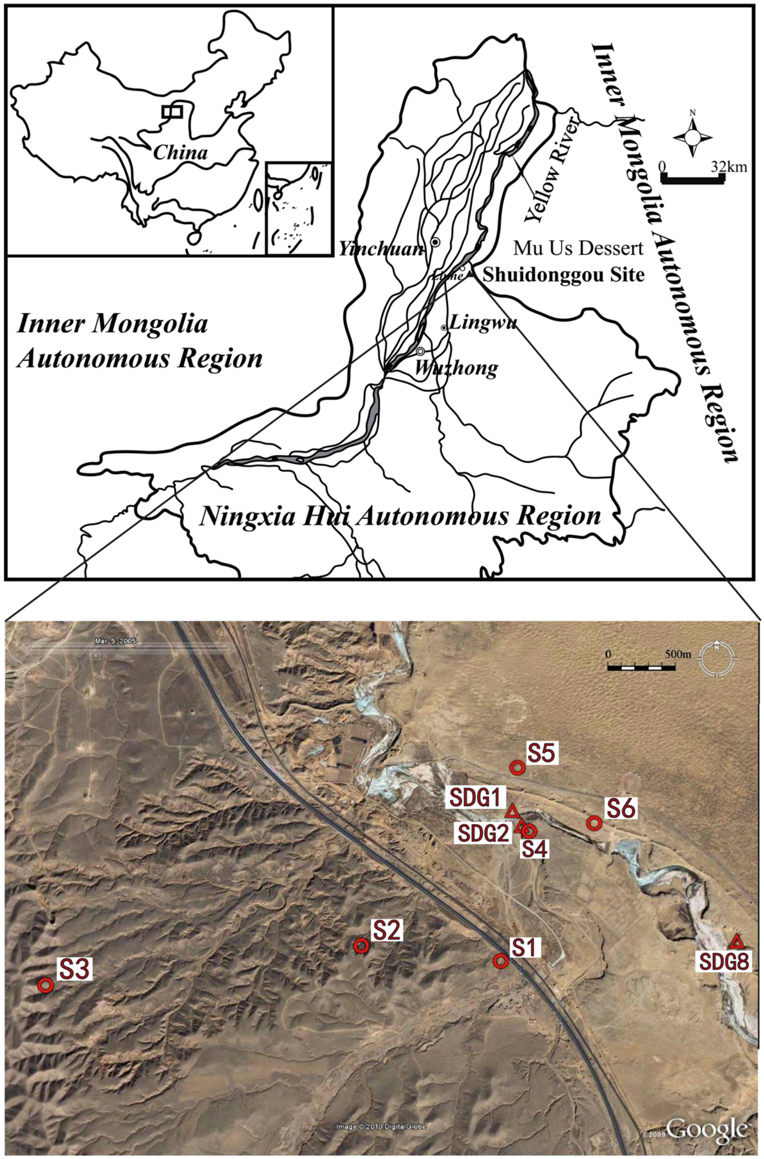

Figure 1 *The locations of the Shuidonggou site complex (SDG1, SDG2 and SDG8), and the rock-collecting areas for the heat-treatment experiment (S1–S6). S1, 38°17'24.9"N, 106°30'3.5"E; S2, 38°17'28"N, 106°29'28.5"E; S3, 38°17'20.2"N, 106°28'10.9"E; S4, 38°17'51.6"N, 106°30'11.2"E; S5, 38°18'4.8"N, 106°30'8.4"E; S6, 38°17'53.2"N, 106°30'27.7"E.*

X-ray diffraction (XRD) and X-ray fluorescence (XRF) examinations

Heat treatment may change the colour of the stone and improve its forgeability (Purdy and Brooks 1971; Collins and Fenwick 1974; Bleed and Maier 1980; Delage and Sunseri 2004), but it remains unclear how such changes are produced. We are certain that the material composition, crystal morphology and phase structure of rocks will change when heated to high temperatures (Domanski and Webb 2000). The X-ray powder diffraction (XRD) method and X-ray fluorescence spectrometry (XRF) examination enable us to better ascertain the changes in stones that occur during heat treatment.

XRF and XRD analyses were performed on 33 stone powder samples from 13 assemblages of heat-treated specimens. The assemblages included five groups of dolomites, six of chert and one each of chalcedony and quartz sandstone. The equipment used included the 800HS energy-dispersive X-ray fluorescence spectrometer and the Rigaku D/Max-2200 X-ray diffractometer, manufactured by the Japanese Shimadzu Corporation.

The uniaxial compression test

The main reason why prehistoric people applied heat treatment to raw materials was to improve their mechanical properties (Copeland 1998; Brown and Marean 2009; Zhou *et al.* 2013b, 2014). Therefore, heat-treatment studies involve tests on these properties primarily based on the following indicators: elastic constant, compressive strength, tensile strength and fracture toughness.

In a comparative study on changes in the mechanical properties of heat-treated stone artefacts, Domanski *et al.* (1994) compared these indicators in experimental specimens before and after heat treatment. They suggested that the fracture toughness exhibited relatively regular changes before and after heat treatment, and was therefore the most suitable indicator for evaluating the mechanical properties of heat-treated stone artefacts. However, these four indicators are all critical to a rock's mechanical properties, and theoretically, a higher compressive strength would prevent the expansion of cracks (i.e., greater fracture toughness). A strong linear relationship exists between a rock's compressive strength and resistance to crack expansion as well as its tensile strength and fracture toughness (Zhang 2002; Lakshmikantha *et al.* 2008; Li *et al.* 2009). Since rocks are not ideal linear elastic materials, their stress and strain curve is usually non-linear as well. Therefore, it is relatively difficult to define indicators such as elasticity modulus and the Poisson ratio (the negative ratio of transverse to axial strain) accurately, because they change with stress and strain. At the same time, as the fracture strength represents the maximum force when a specimen breaks, it is only reflective of the rock's condition at one point in time during the entire process.

In order to reduce the probability of these potential errors and obtain more objective test results, we chose to perform the uniaxial compressive strength test on our raw material samples. In addition to obtaining the compressive strength of specimens, the more important and complete stress–strain curve of stone materials can be obtained using this test. This curve helps us to understand the entire process of rock breakage under uniaxial pressure by providing information such as the brittleness and ductility of raw materials; these two factors are important for analysing the stone's breakage process, breakage patterns and characteristics. Although rock breakage is an instantaneous process under human observation, the servomechanism (an automatic device for controlling large amounts of power by means of very small amounts of power and automatically correcting the performance of a mechanism) can divide this moment into many phases, in which different rock types have different curves representing various mechanical properties. The fracture toughness measured in the study by Domanski *et al.* (1994) yielded only a peak value, and information on other phases during the process of rock breakage was overlooked.

A total of 125 cuboid samples from 31 rock specimens were prepared for this study. We collected data on the physical characteristics of rock samples, such as strength, ductility, brittleness and stress–strain curves, through the uniaxial compressive test, which was conducted at the State Key Laboratory for Nonlinear Mechanics, under the Institute of Mechanics of the

Chinese Academy of Sciences. The hydraulic servo material testing machine used was the MTS 810 model manufactured by MTS Systems Corporation and the operating ambient temperature was 15°C.

Scanning electron microscope observation

Because heat-treated rocks may present certain characteristics that are unobservable to the naked eye, a scanning electron microscope (SEM) is needed to observe the features and structure of rock crystals (Rowney and White 1997; Braadbaart *et al.* 2004). The SEM used here was a Hitachi S-3700N from the Key Laboratory of Vertebrate Evolution and Human Origins in the Institute of Vertebrate Palaeontology and Palaeoanthropology, Chinese Academy of Sciences.

The knapping experiment

This experiment involved the production of stone tools by simulating prehistoric knapping technology and behaviour. Based on these results, this study explores the effects of heat treatment on knapping and summarizes the overall changes in the characteristics of stone tools produced from heat-treated raw materials. This allows us to further discuss the significance of heat-treatment technology in prehistory. A total of 10 knapping sessions were distributed across four experimental raw material groups: eight sessions involved three groups of dolomite and two sessions were performed on one group of quartz sandstones. The raw materials used in the three dolomite groups were samples 050, 201 and 199. Three knapping sessions utilized sample 050 in an unheated state, and heat-treated at 350°C and 400°C; three knapping sessions were conducted with sample 201, unheated and heat-treated at 300°C and 350°C; two knapping sessions used sample 199, in an unheated state and heated to 400°C; and sample 146 (quartz sandstone) was used in two knapping sessions, in an unheated state and heated to 550°C. All sessions were performed with the direct percussion method, using hard hammers. To ensure objective results, all experiments were performed by the same knapper.

RESULTS

X-ray diffraction and fluorescence

Results of X-ray fluorescence revealed that dolomite from the Shuidonggou area is mainly composed of CaO and SiO_2, with small quantities of MgO and Fe_2O_3; chert is composed of SiO_2 and small quantities of CaO and Fe_2O_3; and quartzite and quartz sandstone are composed of SiO_2 and CaO, with small quantities of MgO and Fe_2O_3. Six groups of rock samples, including five of dolomite and one of chert, showed significant changes in material composition following heat treatment. Changes in material compositions among the remaining samples were comparatively smaller. The results showed that heat treatment only resulted in changes in the ratio of material compositions, but not the type. In addition, some level of regularity was also observed in altered samples: for example, when dolomite was heated to 350–400°C, its ratio of SiO_2 increased significantly, whereas when it was heated to 450°C or higher, the ratio of CaO increased significantly. During knapping, a higher siliceous content in raw materials such as chert and chalcedony facilitated the process. Therefore, the most effective heat-treatment temperature for dolomite was between 350°C and 450°C. The tendency of rocks to redden after heat treatment may be because Fe_2O_3 turns dark red as the temperature rises.

The X-ray diffraction pattern revealed that heat treatment impacted on the structure of stone materials (Figs. 2 (a)–(c)). When samples were heated to approximately 400°C, the diffraction peak intensity of silica increased significantly. As illustrated in Figure 3 (d) below, the diffraction peak position of an original sample group and two heat-treated sample groups coincided perfectly, but the diffraction peak intensity composed of different materials underwent relatively significant changes. The diffraction peak intensity of a phase is directly proportional to its percentage in the sample (Zhou and Wang 2002). The increase in the SiO_2 diffraction peak intensity reflects the increase in the degree and amount of silica in the stone materials, which enhanced their knapping properties.

According to the study by Domański *et al.* (2009), changes in the peak shape indicate alterations in the degree of crystallinity in rocks. Crystallinity is a relative value representing the ratio between the integrated intensity of the crystalline portion and the integrated intensity of the crystalline and amorphous portions when both forms are present in X-ray spectra. As the value of crystallinity can sometimes indicate the sample's degree of order, changes in crystallinity can thus indicate whether or not crystals are uniform and in order (Brindley 1980). A higher degree of crystallinity implies larger grains in samples, where the arrangement of internal particles is relatively regular and the diffraction line is strong, sharp and symmetrical; stone with poor crystallinity often contains grains that are too small and defects such as dislocation. Poorer crystallinity will also result in broader diffraction peaks. In general, the breakage of rocks actually represents the separation process of rock crystals, where force is generally conducted along the crystal's edge. Rocks that have not undergone heat treatment have crystals of various sizes, hindering the transmission of force in a regular direction. As a result, the difficulty

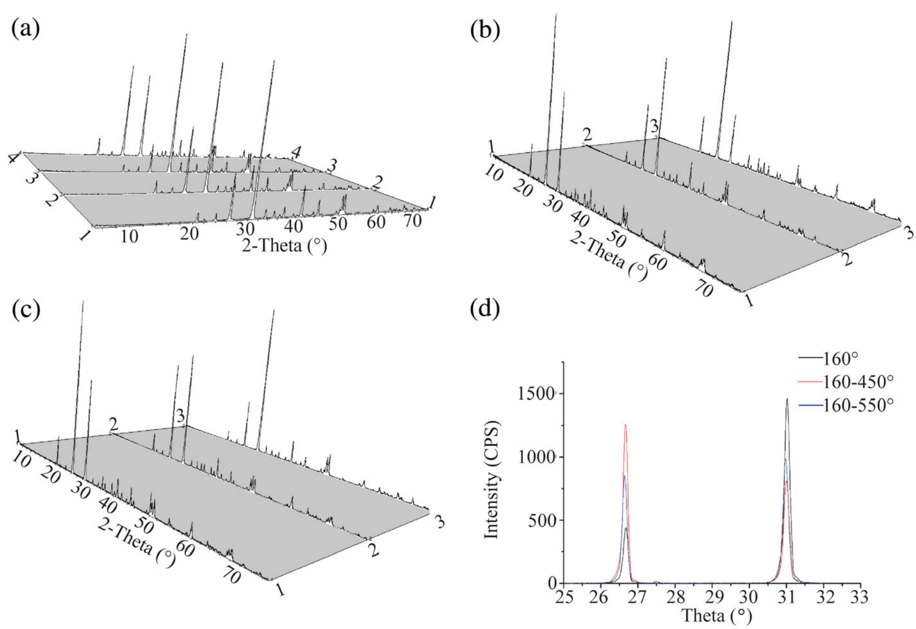

Figure 2 *The diffraction peaks of various rock samples before and after heat treatment: (a) dolomite sample 079; (b) dolomite and chert sample 150; (c) quartzite sample 160; (d) an example of the diffraction details of quartzite sample 160.*

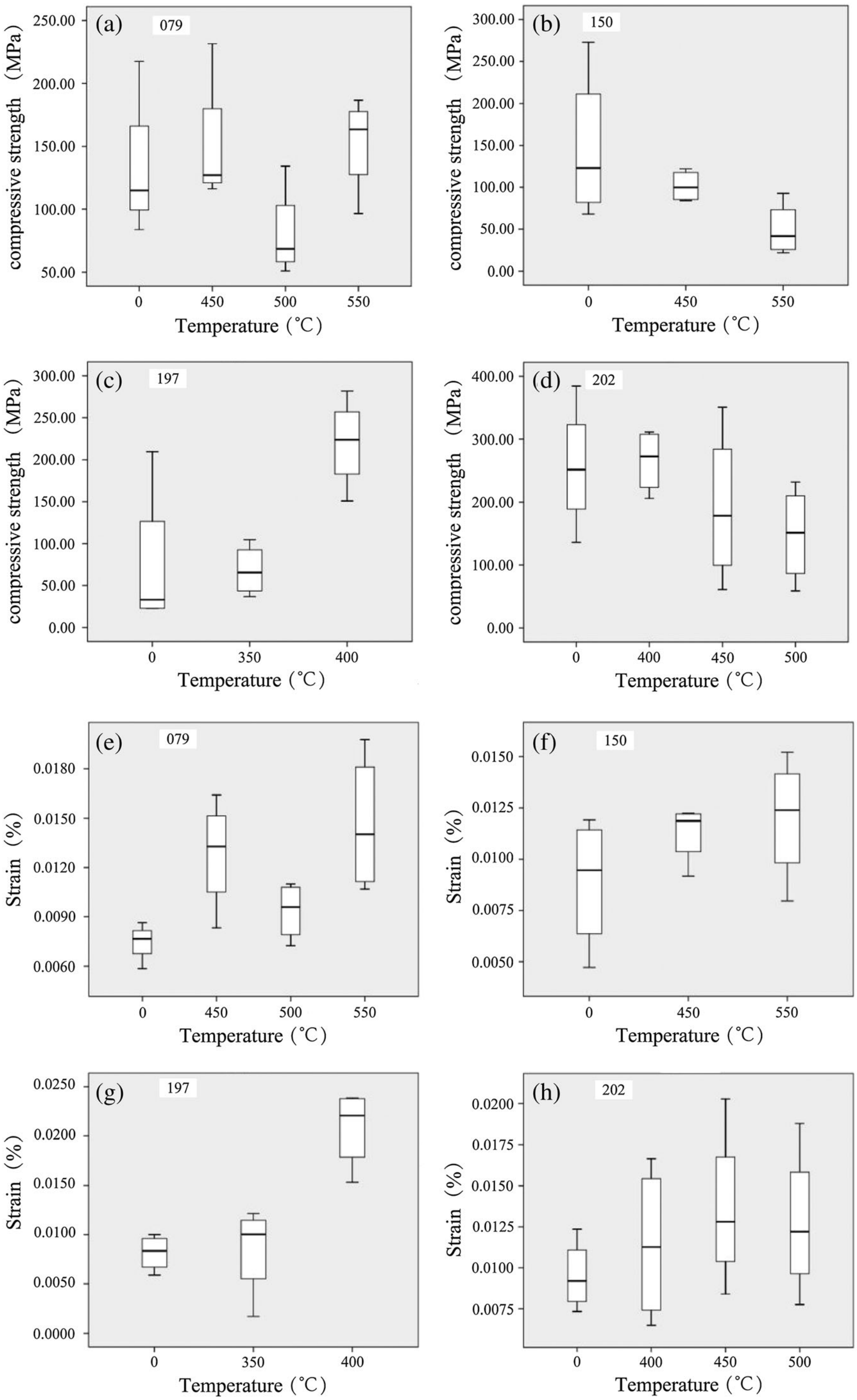

Figure 3 *The relationship between the compressive strength (calculated in megapascals; i.e., MPa), strain (calculated in %) and temperature of heat-treated samples: (a) sample 079; (b) sample 150; (c) sample 197; (d) sample 202; (e) sample 079; (f) sample 150; (g) sample 197; (h) sample 202.*

of knapping is increased and breakage is more likely to occur. The crystallinity in rocks will increase after heat treatment, facilitating better integration of crystals and creating more uniform crystal sizes, which are more favourable for conducting force. Thus, the strength of heat-treated rocks will be reduced but the ductility will be improved (Domański *et al.* 2009).

Using the MDI Jade5.0 software, we calculated the crystallization of the samples: for the specific sample results, see Table 1. In the 28 heat-treated samples, 17 saw an increase in crystallization (61%). The highest increase was 456%, the lowest was 3% and the average increment was 68%. An additional 11 heat-treated samples showed decreased crystallization. The largest decrease was 49%, the smallest was 5% and the average was 21%. Among these 11 samples, two were from quartzite and quartz sandstone, five were chert and four were dolomites. Of the 20 samples heat-treated indoors, 14 had an increase in crystallization (70%), whereas only three out of eight samples heat-treated outdoors saw an increase. The main reason for the latter result could relate to the short time span at high temperature, due to the difficulty of controlling the temperature outdoors.

Overall, most of the stones had increased quartz crystallization after heat treatment; however, changes in the crystallization of dolomite were minor and not significantly regular. Improved crystallization indicates an increase in the size of crystal grains (Domański *et al.* 2009). As stated above, crystal grains and compressive strength are inversely related. The larger the grain size, the weaker they are, therefore reducing the difficulty of knapping that particular stone.

The uniaxial compression test

Changes in the mechanical properties of stone caused by heat treatment are mainly reflected as reduced toughness and higher ductility and brittleness—and, to some extent, improved textural uniformity. Considering that the mechanical properties of stones improve significantly when they are heated at approximately 400°C, this is the most suitable temperature for heat treatment (Figs. 3 (a)–(d)). When the temperature is above 450°C, the improvement of each characteristic will be insignificant or too minimal to improve knapping; for example, the decrease in brittleness or the increase in the amount of microcracks will be small. In contrast to dolomite and chert, which had improved mechanical properties as a result of heat treatment, the quartzite found in the Shuidonggou area was proven to be unsuitable for this technique; its strength increased significantly and its ductility and brittleness reflected minimal improvement. This may be caused by the recrystallization of coarser quartzite grains, which led to closer bonds between crystals.

The variation in the uniaxial compressive strength of the dolomite, quartzite and quartz sandstone found in the Shuidonggou area is relatively large, indicating an inhomogeneous composition of particles inside the stones. The variation was greatly decreased after heat treatment, especially after being heated to about 400°C. This shows that heat-treatment technology can improve the homogeneity of stone materials.

Among the eight groups of experimental stone materials, one group had an A grade for toughness, five had a B grade and two had a C grade (Table 2). Among these, the strength of three sample groups decreased by one grade after heat treatment. Apart from quartzite and quartz sandstones, all other stones were reduced in strength to varying degrees following heat treatment.

Although stone samples experienced changes in shape and displacement deformation under the uniaxial compressive stress test, deformation can reflect the ductility of samples to a certain extent. This refers to deformation between the time when a cross-section of the stone starts to weaken and when it reaches its maximum carrying capacity; in other words, the time when the stone reaches maximum capacity, but before it starts to decrease significantly. This characteristic

Table 1 *The diffraction peaks and crystallization of the experimental rock samples*

Sample number	Temperature (°C)			Diffraction peak		Crystallization		Fitting error, R	Crystallization change (%)	
	Unheated	Indoor experiment	Outdoor experiment	Quartz	Dolomite	Quartz	Dolomite		Quartz	Dolomite
079	○	–	–	26.637	30.971	1.57	1.56	11.13	–	–
	–	450	–	26.61	30.939	3.33	1.3	11.04	112	–17
	–	451 CF*	–	26.667	30.996	2.28	1.41	12.2	45	–10
	–	550	–	26.664	30.979	3.72	1.19	10.74	137	–24
100	○	–	–	26.631	–	0.75	–	8.93	–	–
	–	450	–	26.628	–	1.12	–	12.22	49	–
105	○	–	–	26.679	–	0.91	–	12.09	–	–
	–	450	–	26.64	–	0.82	–	10.82	–10	–
139	○	–	–	26.66	–	0.76	–	10.32	–	–
	–	450	–	26.625	–	0.7	–	9.19	–8	–
107	○	–	–	26.671	–	0.76	–	10.11	–	–
	–	450	–	26.663	–	0.82	–	10.76	8	–
150	○	–	–	26.639	–	1.22	–	12.02	–	–
	–	450	–	26.598	–	1.57	–	13.85	29	–
	–	550	–	26.558	–	2.69	–	11.14	120	–
160	○	–	–	26.66	–	2	–	8.1	–	–
	–	450	–	26.67	–	1.12	–	9.11	–44	–
	–	550	–	26.645	–	2.06	–	11.66	3	–
173	○	–	–	26.702	–	0.8	–	8.81	–	–
	–	550	–	26.706	–	0.44	–	6.42	–45	–
197	○	–	–	26.693	31.001	2.25	1.54	11.42	–	–
	–	350	–	26.694	30.997	1.14	2.25	10.17	–49	46
	–	400	–	26.666	30.989	2.4	2.8	14.35	7	82
200	○	–	–	36.653	–	0.88	–	12.09	–	–
	–	550	–	26.645	–	4.89	–	9.43	456	–

(*Continues*)

Table 1 *(Continued)*

Sample number	*Temperature (°C)*			*Diffraction peak*		*Crystallization*		*Fitting error, R*	*Crystallization change (%)*	
	Unheated	*Indoor experiment*	*Outdoor experiment*	*Quartz*	*Dolomite*	*Quartz*	*Dolomite*		*Quartz*	*Dolomite*
202	○	–	–	26.713	–	1.56	–	11.04	–	–
	–	400	–	26.69	–	2.21	–	12.62	42	–
	–	450	–	26.682	–	2.17	–	10.7	39	–
	–	450 CF	–	26.704	–	1.93	–	12.18	24	–
cjss	○	–	–	26.655	–	0.57	–	7.86	–	–
	–	450	–	26.651	–	0.53	–	7.65	–7	–
ys	○	–	–	26.603	–	0.64	–	8.85	–	–
	–	450	–	26.656	–	0.66	–	9.2	3	–
003	○	–	–	26.681	31.003	1.04	2.09	9.63	–	–
	–	–	○	26.649	30.963	1.31	2.79	10.13	26	33
	–	–	–	26.669	31.004	1.27	2.57	11.17	22	23
027	○	–	–	26.666	30.977	1.82	1.88	12.28	–	–
	–	–	○	26.632	30.94	1.66	2.06	12.7	–9	–26
	–	–	–	26.687	31.019	1.73	3.88	12.74	–5	106
070	○	–	–	26.689	31.007	1.58	1.96	10.49	–	–
	–	–	○	26.764	31.083	1.31	1.89	10.23	–17	–4
099	○	–	–	26.662	–	0.47	–	6.1	–	–
	–	–	○	26.635	–	0.63	–	7.97	34	–
175	○	–	–	26.7	–	0.5	–	6.45		–
	–	–	○	26.688	–	0.4	–	6.34	–20	–
	–	–	–	26.683	–	0.42	–	5.96	–16	–

*CF, cooling fast.

Table 2 *The strength rating of stones (Zhang 2007)*

Grade	*Description of strength*	*Uniaxial compressive strength (MPa)*
A	Very high	>250
B	High	100–250
C	Moderate	50–100
D	Medium	25–50
E	Low	5–25
F	Very low	<5

is reflected in the knapping of a stone tool, as the raw material can be better controlled and can withstand repeated stress that does not exceed its strength. It is easier to control the direction of force within stone materials that have better ductility, which are more suitable for the knapping of long flakes. Figure 3 shows that the ductility of a stone improves significantly after heat treatment and that 400°C is the most suitable temperature for the rock types from the Shuidonggou area (Figs. 3 (e)–(h)).

SEM observation

In general, chert or dolomite specimens exhibit similar characteristics after heat treatment. The grains are distributed equally, mainly with a grain-based structure, and when grains were ground, they became sub-rounded. Fusion and bonding of crystals occurred, resulting in a smooth surface. In comparison, these characteristics of chert are more obvious than those of dolomite after heat treatment. It is thought that the phenomenon involving the fusion of crystals is related to the better crystallization of heat-treated stone, as indicated in the XRD test (Zhou *et al.* 2013b: see also Fig. 4).

As shown in Figure 4, dolomite sample 198 appears to have a euhedral to subhedral porphyritic structure under electron microscopy. When ground, its crystal size measured 1–5 μm and its shape became angular and sub-rounded. It has good directionality and close interlayers and is generally smoother (Fig. 4 (a)). After heat treatment at 450°C, its grain structure appears to be plate-shaped. It has a crystal size of 5–10 μm and it becomes round when ground. It has regular directionality and close interlayers. The edges of crystals begin to fuse as the boundaries are blurred and closely connected. The boundaries also appear to be flat and smooth (Fig. 4 (b)).

The knapping experiment

Tools produced using the same piece of stone for knapping before and after heat treatment have clear differences:

(1) Flakes produced after heat treatment have more structured lengths, widths and thicknesses. For example, the structured index of dolomite samples 050 and 201 is close to 1 after heat treatment at 350°C. Lower values of this index, as well as a lower index for width and thickness, indicate that they are more structured. A larger ratio of the length and width of a flake (Table 3) indicates that it is becoming narrower. This was clear when dolomite sample 050 underwent heat treatment at 350°C and 400°C; the ductility improved after treatment and the flakes ruptured further along the direction of stress.

(2) Part of the flake produced from heat-treated stone yielded a larger striking platform size than that produced from untreated stone. This is shown in dolomite and quartz sandstone. However, the increment is smaller when quartz sandstone undergoes treatment at 550°C and dolomite at

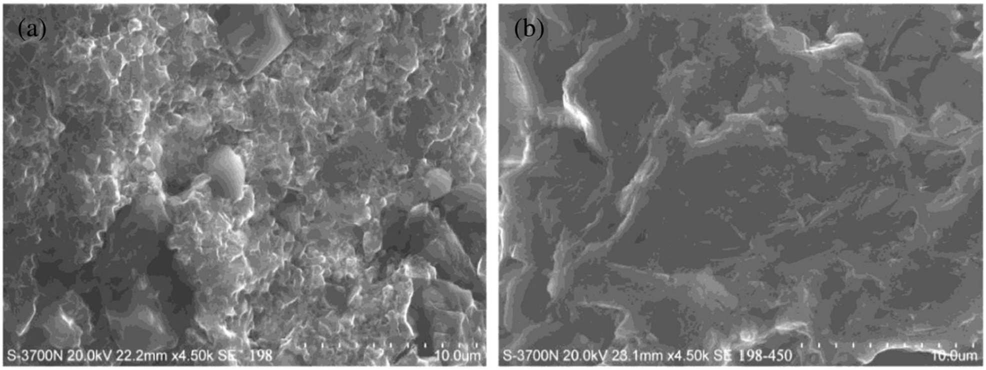

Figure 4 *A comparison of dolomite sample 198 (a) before and (b) after 450°C heat treatment.*

Table 3 *A comparison of the sizes and lengths of flake platforms with heat treatment: scale in millimetres*

	Temperature	*N*	*Minimum*	*Maximum*	*Average*	*SD*
Length	050—untreated	25	1.54	132.82	24.60	31.62
	050—350°C	41	0.77	302.84	37.63	58.79
	050–400°C	20	3.60	160.55	32.75	43.91
Length:width ratio	050—untreated	25	0.5	2.57	1.27	0.64
	050—350°C	41	0.4	3.71	1.36	0.7
	050—400°C	20	0.56	3.1	1.39	0.81

400°C. The increment is larger when dolomite is treated at 350°C. This may be because of the lower compressive strength of the stone following heat treatment. Equal force from the same source will cause a larger rupture area, due to the reduced strength and increased brittleness of the stone. Heat treatment can also cause changes in the flake platform angle. Before heat treatment, the angle of dolomite flake platforms is between 100° and 110°, with a large range of variation. After heat treatment, the angle falls to between 90° and 100°, with a smaller range of variation. Stability of the platform angle in suitable cases can guarantee a high utilization rate of the raw material core, reflecting one benefit of heat treatment.

(3) There is a much lower rate of vertical bending from flakes produced on heat-treated stone. After heat treatment at 350°C and 400°C, vertical bending of dolomite sample 050 decreased from the initial 36% to 9% and 10%, respectively. The bending angle also decreased from the average 160° to 150°. After heat treatment at 300°C and 350°C, vertical bending of the dolomite decreased from 17% to 12.5% and 3%, respectively. The average reduction in bend decreased from 160° to 155°. After heat treatment, the degree of cross-sectional bending in quartz sandstone was minimal. The degree of vertical cross-sectional bending can improve the quality of the flake, as well as providing a better surface for knapping, thus increasing the utilization rate of cores. Heat treatment effectively lowers the degree of bending of dolomite flakes, particularly at 350°C.

(4) Heat treatment will also affect the ventral side of the flake by increasing the amount of lip from around 10% to 30%. Dolomite sample 050 underwent the largest increase at 68% after heat treatment at 350°C. It is generally understood that lip is characteristic of knapping with soft hammer percussion. In an experiment conducted at Locality 8 of the Shuidonggou site that used the same raw materials as in this study, lip production rose to 4.8% when the stone was knapped with soft

hammer percussion (Wang 2010). No lip was produced on the quartz sandstone flake before or after heat treatment. Approximately 70% of the flakes extended vertically before heat treatment, whereas following treatment, this number increased to 90% or more. The percentage of feather-shaped ends on dolomite flakes increased from 40% to 60% after heat treatment. As the feather-shaped end or termination of a flake could serve as an actual or potential working edge to the ancient lithic maker, the increase caused by heat treatment could considerably enhance the efficiency of stone tool production.

(5) For prehistoric people, the main goal of obtaining flakes was to use them as sharp tools for various needs. Therefore, the flake edge length is an important indicator of whether the knapping is successful. We discovered that heat-treated flakes have longer edge lengths. For example, the length of the effective edge of the flake produced from dolomite sample 050 increased from 13.7 mm g^{-1} to 56.4 mm g^{-1} (with 350°C treatment) and 51.8 mm g^{-1} (with 400°C treatment). In sample 201, this edge length increased from 29.1 mm g^{-1} to 176.6 mm g^{-1} (with 350°C treatment) and 194 mm g^{-1} (with 400°C treatment) after heat treatment. Quartz sandstone sample 146 underwent the smallest change, at 6.45 mm g^{-1} and 6.19 mm g^{-1}.

DISCUSSION

The heat-treatment mechanism

Based on the X-ray diffraction analysis, we believe that a suitable temperature for heat treating raw materials changes the sizes and crystallization of crystal grains. At the same time, the treatment will affect the proportion of matter composition and hence achieve the purpose of improving the quality of the stone. This conclusion is also supported by the SEM analysis. As a result of this mechanism, the knapping qualities of raw materials that have a high content of siliceous and smaller grain sizes can be improved through heat treatment. Thus, it is not surprising that all of the heat-treated stone artefacts from the Shuidonggou site were chert and siliceous dolomite. The results of our simulated experiment showed that the physical characteristics of heat-treated quartzite and quartz sandstone underwent smaller changes, demonstrating the minimal effects of this process on those materials.

Because the effect of heat treatment is closely linked to factors such as the composition and crystal structure of the material, we need to carefully consider the differences between individual types of stone during experiments. Palaeolithic tools in the region around the Shuidonggou site were largely produced from gravels found along riverbanks. As these raw material sources were inconsistent, the difference in types should not be overlooked. Most of the siliceous stones, such as chert, will darken to a reddish colour following heat treatment. However, the change is not totally uniform and is less obvious when the temperature rises quickly during heat treatment. Therefore, in such cases, it would be difficult to differentiate between raw materials based on colour alone. The transformations would be even more obvious if the raw material source was different. This phenomenon also applies to the mechanical properties of raw materials. Hence, experiments are the essential foundation of studies on archaeological heat treatment.

Change in knapping efficiency

When knapping stone tools, the rock ruptures when receiving an instant impact from a sharp object (mainly stone), which acts as the origin of the force. That is followed by the expansion of microcracks throughout the rock, which is a macroreflection of the deformation and damage accumulation in its microstructure. Based on the mechanism of stone breakage, changes after heat treatment in mechanical properties such as strength, brittleness, lithology and homogeneity could effectively improve the knapping quality of the stone. Features such as bulb of percussion and *éraillure*, produced by the interaction between internal impurities of the stone and the impact force in the process of knapping, may result in irregular flakes, as well as damage on the breakage surface and the core's platform. Thus, by lowering the chances of forming a bulb of percussion, or reducing its volume and preventing the production of an *éraillure*, one can improve the efficiency of knapping stone tools and the effective flake production rate. By improving the homogeneity of the stone, heat treatment lowers the chance of producing these features.

The forms of flake termination are related to the angle and method of force. Heat treatment can effectively lower the chances of producing the non-ideal forms of termination, such as step and *outrepassé*, by improving the ductility and homogeneity of stones. This is because the impact force passes through the impurities in such stones and extends beyond a stable direction, thereby facilitating the formation of a feather-shaped termination.

The results of our knapping experiment showed that heat treatment had significant effects on siliceous stones such as dolomite. However, the changes in quartz sandstone before and after heat treatment were minimal. At Shuidonggou, we found that 350°C is the appropriate temperature for treating dolomite. The effects would be insignificant if the temperature was too low, whereas too high a temperature would damage the knapping quality of the material. Because heat treatment changes the mechanical properties of stone, the form of heat-treated flakes is more regular, with an increase in the proportion of narrow and thin flakes. At the same time, the extension of the flake is straighter, while the degree of bending decreases and the percentage of feather-shaped termination increases. Most importantly, the production rate of the effective edge of the flake increases significantly. Therefore, suitable heat treatment can effectively increase the utilization rate of stones.

We have also observed few variations in the length and thickness of heat-treated flakes. This may be related to the knapping strategy during the experiment. In order to obtain a thinner flake, the percussion points were positioned as close to the edge of the core as possible. Since the stone hammer was somewhat harder than the material being knapped, positioning the percussion points too close to the suitable edge would result in platform breakage and failure of knapping. Thus, in this study, most percussion points were away from the edge of the core platform. This caused the vertical force to be greater than the tangential force, which is an important factor in controlling the length of flakes. This is also the main reason why the heat-treated flakes did not vary much in length or thickness. When a vertical force is dominant, the change in the length of a flake before and after heat treatment is minimal; when a tangential force is dominant, it has a magnifying effect on that change. It also increases the size of the flake platform. This phenomenon also indirectly proves that the effects of heat treatment in creating longer and thinner flakes become more prominent only when the tangential force is greater than the vertical force. Thus, when a billet is used to knap heat-treated stones, the improvement in mechanical properties will increase.

Note that the same knapping experiment was performed on 10 pieces of stone that had undergone heat treatment at 450°C and 550°C. The results showed that grain-shaped structures would form in dolomite and chert when treated at 450°C and above. Compared to untreated stones, the area of breakage in these samples appeared to be significantly rougher. A large amount of coarse grains, observable with the naked eye, resulted in the ineffective conduction of force and even increased the difficulty of knapping the flakes. Because this loss of flaking ability was observed in these raw materials, no data were collected. This phenomenon shows the important contribution of controlling the temperature during heat treatment to the overall success of the process.

CONCLUSION

This simulation experiment demonstrates that colour change should be one indication for heat-treated archaeological artefacts. Although lustre is also an effective identification, it does not apply all the time on all kinds of materials. In consideration of the subjectivity of the naked eye, it is suggested that scanning electron microscopy, X-ray diffraction, thermoluminescence, palaeomagnetism or other methods should be applied to improve the credibility of archaeological heat-treatment identification (Zhou *et al.* 2013b, 2014).

The application of complex technologies represents one stage in the outcome of the evolution of human cognition and physical ability. The results reported here show that heat treatment of lithic raw materials is one such complex technology, since it requires the person to have sufficient knowledge of the stones and the confidence to control the hearth temperatures precisely, and also to be a skilled knapper. The heat-treated stone artefacts found at Localities 2 and 12 of the Late Palaeolithic Shuidonggou site demonstrate that humans already possessed the skilful technique of heat treatment at that time. Since cultural relics from the Palaeolithic are often difficult to preserve and fully understand, technical studies of these assemblages often lack sufficient detail. Simulated experiments can compensate for such deficiencies to some extent, enabling us to have a clearer understanding of technologies such as heat treatment. This knowledge is particularly valuable in interpreting the Shuidonggou site. The discovery of heat-treated stone artefacts at that site shows that different generations of occupants at Shuidonggou used complex forms of technology to enhance their survival. Moreover, this evidence indicates that Late Palaeolithic people in this part of Asia had relatively high cognitive abilities and a comparatively complex form of social organization.

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