

The use of fire at Zhoukoudian: evidence from magnetic susceptibility and color measurements

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Abstract In order to provide direct evidence for the use of fire by humans at Locality 1, Zhoukoudian, we examine the burnt and unburnt sediments of newly excavated area in Layer 4 by detailed measurements of magnetic susceptibility, color, and diffuse reflectance spectrum. Results show that the magnetic susceptibility and redness of the burnt sediments are remarkably higher than those of other areas on the same level: up to ~22 times for magnetic susceptibility and ~3 times for redness of those of the adjacent unburnt sediments. Fine-grained (superparamagnetic/stable single-domain) magnetite and hematite grains make dominant contributions to the distinctly high values of magnetic susceptibility and redness in the burnt sediments. Diffuse reflectance spectroscopy results show that

the burnt sediments contain more hematite than those of other areas and localities 2 and 3. High-temperature magnetic susceptibility measurements demonstrate that the burnt sediments have been heated above 700 °C. Those changes in low-frequency magnetic susceptibility and redness are impossibly resulted from natural fires, thus most likely signaling the human activities of controlled use of fire. However, further work is needed to confirm whether or not these heat-affected sediments were produced in situ.

Keywords Zhoukoudian · Use of fire · Magnetic susceptibility · Redness · Rock magnetism

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1 Introduction

The use of fire was one of the most important skills of hominins, which has been reported to go back at least 0.5 Ma, possibly more than 1.0 Ma [1–4]. Locality 1 of Zhoukoudian is one of the earliest sites, in which solid evidence for the use of fire was preserved [5–7]. Excavation of Locality 1 in 1920–1930s yielded the “apparently charred or partly calcined animal bones,” “antler samples,” and “black layers” [5]. Based on chemical analysis, those materials were suggested to be the “evidences of the use of fire by *Sinanthropus*” [5]. Other materials were also recovered during later excavation, e.g., burnt bones, burnt stones, and charcoal of Chinese redbud [8]. Guo et al. [9, 10] made fission track dating, and found that the sphenes in the ash samples of Layer 4 and Layer 10 were heated above the annealing temperature, further suggesting the presence of burning. Dating results of fission track, electron spin resonance, and paleomagnetism show that the age of Layer 10 is about 0.5 Ma B.P. [1, 9, 11–13]. The latest results of

cosmogenic $^{26}\text{Al}/^{10}\text{Be}$ burial dating of quartz sediments show that the age of Layers 7–10 could be over 0.70 Ma B.P. [2]. Abundant fire remains preserved in Locality 1 brought the history of human controlled use of fire much earlier at that time.

Recently, the existence of in situ burning in Locality 1 was challenged. Weiner et al.'s [14, 15] reexamination of the evidence in Layer 10 and Layer 4 indicated “the absence of ash or ash remnants (siliceous aggregates) and of in situ hearth features”, thus they concluded that “there is no direct evidence for in situ burning”, and “most of the fine-grained sediments in the site were water laid”. However, Wu [16] and other researchers [17] argued that Weiner et al. [14] only did sampling at western section of Locality 1 and “do not fully address the wealth of data collected at the entire site since the 1930s”, thus their conclusions are not convincing.

Fire causes the magnetic enhancement in soils and other sediments [18, 19], which can be attributed to the generation of ferromagnetic mineral phases like magnetite or maghemite during heating [20]. Magnetic analysis has been widely applied for studies of archaeological sites [20–24]. Results of experimental fires also show that magnetic methods can be used to effectively identify the presence of heated area, especially in early sites, where the original features of use of fire are not preserved well [25–28].

Burning also changes color of the sediments. At temperatures between 250 and 400 °C, hematite is produced by dehydration of goethite [29], which is widespread in different kinds of sediments. As a pigment mineral, the increased content of hematite causes the reddish color of the sediments. Early researches of the archaeological sites show that heating produced hematite grains are responsible for the rubification [28, 30]. Therefore, color measurements provide useful information of magnetic minerals.

During the new excavation of Locality 1 started from the year 2009 [31], a series of possible remnants of the use of fire were discovered in Layer 4 [32] of the western section. To reveal the distribution of burnt and unburnt sediments and probe into the nature of the magnetically enhanced sediments, these remnants and sediments on the same level were sampled and examined by magnetic, color, and diffuse reflectance spectrum (DRS) measurements.

2 Materials and methods

The remnants of the use of fire come from the sedimentary layer ~1 m below the top of Layer 4 at the western section. The excavation area covers ~30 m². The sediments of this level are mainly silts, which are partly cemented with calcite. Large angular breccias of limestone distribute on the excavation surface, which are likely to be the

éboulis [33]. The burnt areas distribute at the north and south ends of the excavation area. In the excavation pit N119E71 at the north, black, reddish-brown, and brownish-

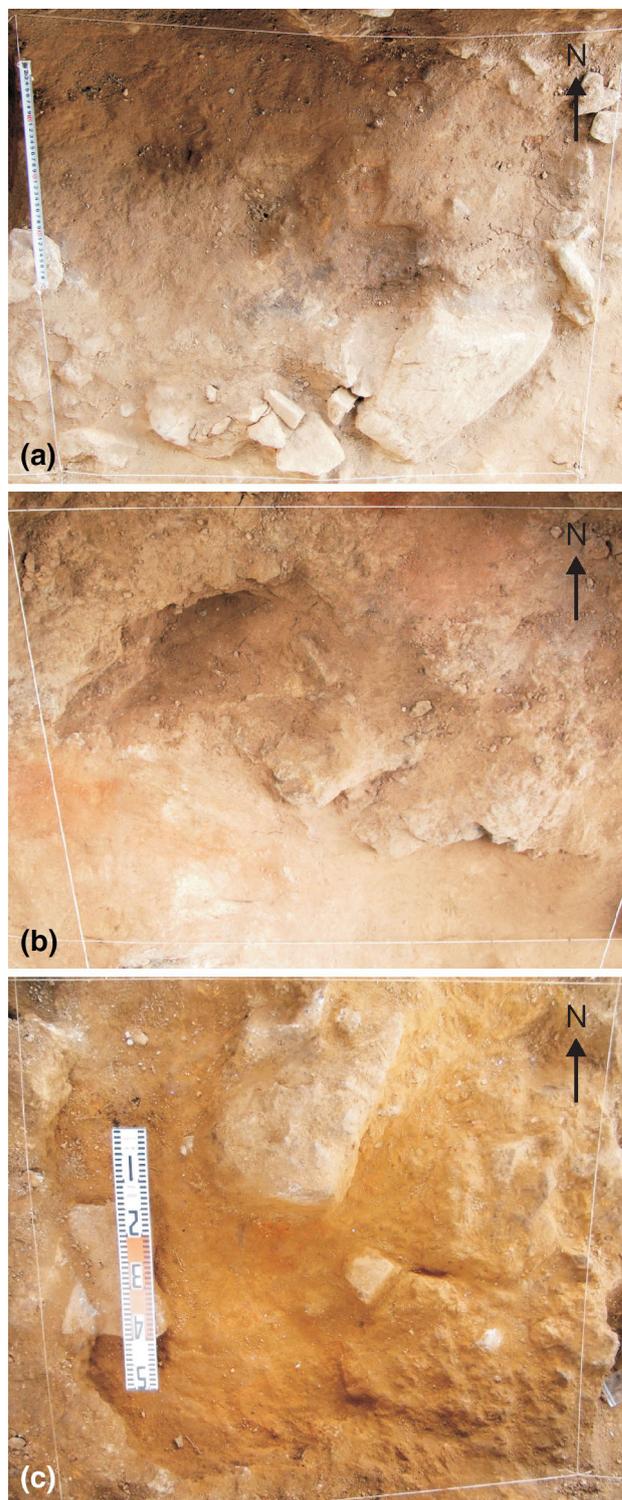


Fig. 1 Photos showing the burnt sediments in areas of H1 (a), H2 (b), and H3 (c). White lines indicate borders of the excavation pits (1 m × 1 m)

yellow silty sediments are spotted or banded, and circled by gravels with diameters from ~ 2 to 27 cm. This area is named H1 in this study (Fig. 1a). At northwest of H1 and the border of excavation pits N120E69 and N120E70, the sediments are pinkish- or light reddish-brown. This area is named H2 (Fig. 1b). At the south end of the excavation area, the sediments of excavation pit N114E69 and part of N115E69 are orange or orangey-brown, which are distinctly different from yellowish-brown sediments in the adjacent areas (Fig. 1c). This area is named H3 in this study. No distinct horizontal bedding was observed in the field.

In order to detail the sedimentary features of the excavation pits and delineate the range of the burnt area, each pit was gridded for sampling. We collected 36 samples in each pit of the burnt areas, and 9–24 samples in each pit of unburnt areas. Samples were named according to the sampling location. For example, sample ID “N119E71 C3” represents the sample collected at Row C (6 rows were divided equally and assigned A–F, respectively, from south to north of the pit) and Column 3 (six columns were numbered 1–6, respectively, from west to east) of the pit N119E71. Six natural samples were collected as control, including four topsoil samples on the Longgushan Hill, and two samples of red-hue sediments from Localities 2 and 3, respectively [34, 35].

A total of 405 samples were air-dried. Measurements of magnetic susceptibility and color were made on the samples. Magnetic susceptibility (χ) was measured with an Agico MFK1-FA Kappabridge magnetic susceptibility meter at frequencies of 976 Hz (χ_{LF}) and 15,616 Hz (χ_{HF}). The analytical precision is 0.1%. Two measures of frequency-dependent magnetic susceptibility (χ_{FD} , defined as $\chi_{LF} - \chi_{HF}$, and $\chi_{FD} \%$, defined as $(\chi_{LF} - \chi_{HF})/\chi_{LF} \times 100 \%$) were calculated from these measurements. Redness was measured by a KONICA MINOLTA CM-700d spectrophotometer. The standard deviation of chromaticity value is within ΔE^*_{ab} 0.04.

Following the magnetic susceptibility and redness measurements, typical samples were selected for DRS and high-temperature magnetic susceptibility (χ - T curves) measurements. Firstly, eight samples from the excavation pits and two from Localities 2 and 3 were selected for DRS measurements using a Varian Cary 5000 spectrophotometer equipped with a BaSO₄-coated integrating sphere and using BaSO₄ as the white standard [36]. Analytical precision of the instrument is 0.5%. Semi-quantification of goethite and hematite was processed according to the method of Torrent and Barrón [36]. Then, two samples, one from the burnt area and the other from the unburnt area were selected for χ - T measurements using an Agico KLY-3 Kappabridge with a CS-3 high-temperature furnace in an

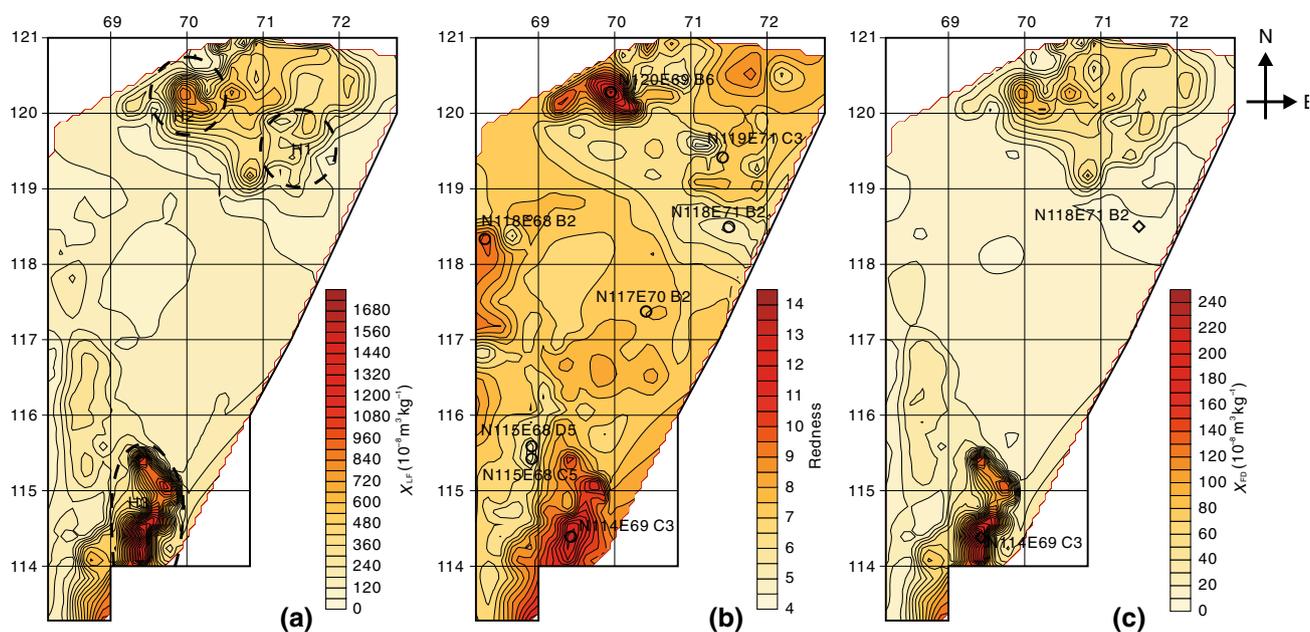


Fig. 2 Contour maps for χ_{LF} (a), redness, (b) and χ_{FD} (c) measurements. The *thick black frame* of each figure represents the range of excavation area. The *thin-line grids* show the excavation pits. The *numbers* outside the frames set the excavation coordinate, according to which the pits were named. The *thick dashed lines* in a show the burnt areas. *Black circles* in b show the locations of DRS samples (N119E71 C3, N114E69 C3, and N120E69 B6 from the burnt area; and N115E68 C5, N115E68D5, N117E70 B2, N118E68 B2, and N118E71 B2 from the unburnt area). *Diamonds* in c show locations of the χ - T samples

argon atmosphere. Each sample was separated into two parts. One was continuously exposed through temperature cycle from room temperature to 700 °C and back to the room temperature. The other was subject to a partial heating/cooling method [37, 38] with the highest temperatures from 100 to 700 °C at an interval of 100 °C.

3 Results

Contour maps for χ_{LF} , redness, and χ_{FD} are shown in Fig. 2. χ_{LF} ranges from 36×10^{-8} to $1,773 \times 10^{-8} \text{ m}^3 \text{ kg}^{-1}$. As shown in Fig. 2a, high values of χ_{LF} mainly occur at the north and south ends of the excavation area. In other areas, χ_{LF} values are relatively lower and show smaller deviations. At the south end, distribution of high χ_{LF} values is consistent with the range of H3, which was delineated during the fieldwork. The highest χ_{LF} value of the whole excavation area is found in the excavation pit N114E69, which is 22 times of that of the adjacent areas. At the north end, the distribution area of high χ_{LF} values is up to 4 m^2 , larger than that of the south end; and the values of χ_{LF} are lower than south end. The highest χ_{LF} value is $961 \times 10^{-8} \text{ m}^3 \text{ kg}^{-1}$ in H2, which is 20 times of that of the adjacent areas. The highest χ_{LF} value in H1 is only $514 \times 10^{-8} \text{ m}^3 \text{ kg}^{-1}$. As a comparison, χ_{LF} values of topsoil samples from the vicinity of Locality 1 are all lower than $180 \times 10^{-8} \text{ m}^3 \text{ kg}^{-1}$.

Figure 2b shows the contour map of redness, which ranges from 4.6 to 14.3. At the north end, the high values mainly occur in H2, consistent with the field observation. The highest value of 14.3 is at the same place with the highest χ_{LF} value of the north end. H1 shows relatively low redness values, with the highest value 8.1. At the south end, distribution of high redness values is very similar to that of χ_{LF} . The highest value of this area is 13.8, which is at the same place of the highest χ_{LF} value of the whole excavation area. Another high redness region is at the west, of the excavation pits N118E68 and N117E68. The highest value is 9.9, lower than those in H1 and H3. And χ_{LF} values of this area are low (Fig. 2a). Besides, samples of Localities 2 and 3 have quite high redness values (11.3 and 10.6, respectively), but have low χ_{LF} values (120×10^{-8} and $153 \times 10^{-8} \text{ m}^3 \text{ kg}^{-1}$, respectively).

Frequency-dependent magnetic susceptibility is widely used for detection of magnetic grains ranging across the superparamagnetic (SP) to single-domain (SD) grain size boundary [39]. As shown in Fig. 2c, the distribution of high χ_{FD} values is very similar to that of χ_{LF} . The two parameters are linearly correlated (Fig. 3a). The average and maximum values of χ_{FD} % are 11 % and 15 %, respectively, which suggests that there are considerable quantities of ferrimagnetic grains ranging across the SP to SD grain size boundary, especially in the burnt area with high values of χ_{LF} and redness.

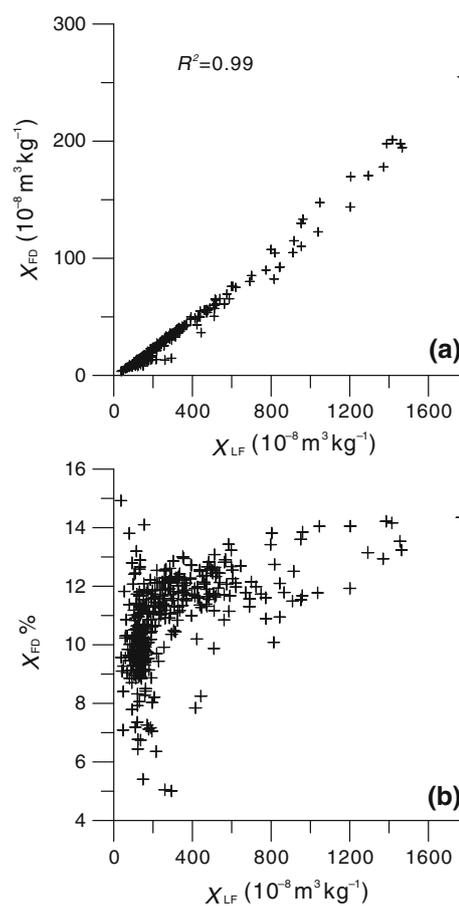


Fig. 3 Relationships between χ_{LF} and χ_{FD} (a), χ_{LF} % and χ_{FD} % (b)

Figure 4 shows the χ - T curves of sample N114E69 C3 from the burnt area H3 and sample N118E71 B2 from the unburnt area. χ - T curves of the two samples are characterized by a major decrease in susceptibility at ~ 580 °C (Fig. 4a, i), marking the Curie point of nearly stoichiometric magnetite. In addition, χ - T curve of sample N114E69 C3 from the burnt area is nearly reversible (Fig. 4a). However, χ - T curve of sample N118E71 B2 from the unburnt area is clearly irreversible (Fig. 4i) with the cooling curve above the heating curve. These behaviors suggest that almost no neo-formation of ferrimagnetic minerals during thermal treatment of the sample from the burnt area (N114E69 C3), because the iron-containing silicates/clays in that sample, which are required for generation of strong magnetic minerals, may have been depleted due to the use of fire by hominins [40]. As for sample N118E71 B2 from the unburnt area, large quantity of strong magnetic minerals was generated during thermal treatment, suggesting the presence of metastable iron-containing silicates/clays in the sample. The newly formed mineral should be magnetite according to the distinct kink of the cooling curve at ~ 580 °C.

A partial heating/cooling method [37, 38] was used to investigate magnetic mineralogical changes during thermal

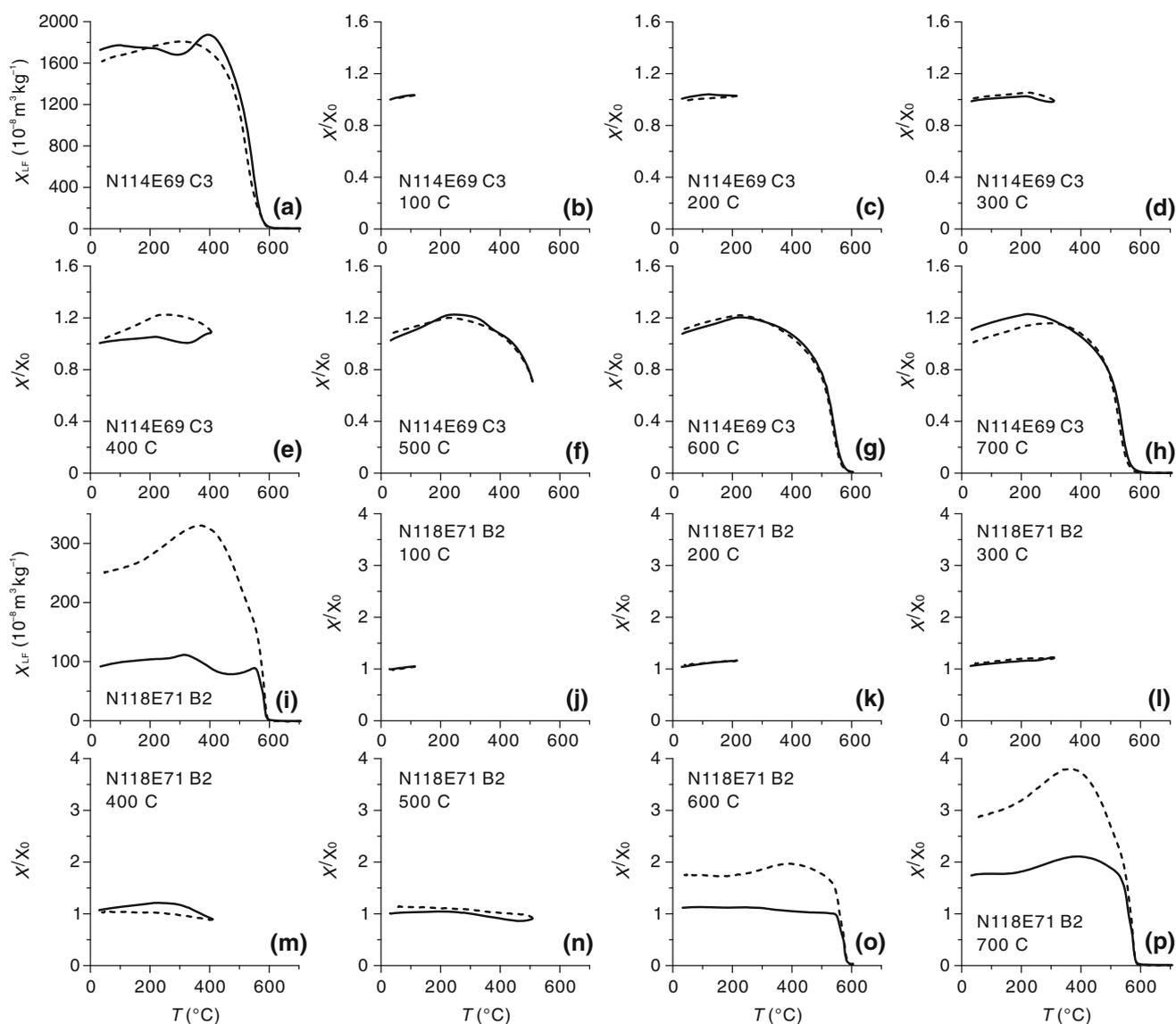


Fig. 4 χ - T curves of representative samples from the burnt area (N114E69 C3) and the unburnt area (N118E71 B2). Note that each sample was separated into two parts: one being continuously exposed through temperature cycle from room temperature to 700 °C and back to the room temperature (a, i); and the other, subject to partial heating/cooling runs with the highest temperatures from 100 to 700 °C at an interval of 100 °C (b–h, j–p). Solid and dashed lines show heating and cooling curves, respectively

treatment (Fig. 4b–h, j–p). For the sample from the burnt area (N114E69 C3), except for the 400 °C heating/cooling run, χ - T curves are nearly reversible (Fig. 4b–h). This behavior is well consistent with that of the 700 °C heating/cooling run (Fig. 4a), further indicating that almost no neoformation of ferrimagnetic minerals during thermal treatment. However, for the sample from the unburnt area (N118E71 B2), the heating/cooling runs between 100 and 500 °C are nearly reversible (Fig. 4j–n), while the 600 and 700 °C runs are clearly irreversible (Fig. 4o, p). These results indicate the presence of metastable iron-containing silicates/clays in the sample from the unburnt area, which were transformed to strong magnetic minerals during

thermal treatment. Also, the irreversible heating/cooling runs at 600 and 700 °C illustrate that the sample was not heated up to 600 °C for a certain period of time.

Diffuse reflectance spectrum results are shown in Fig. 5, which indicate that all selected samples contain goethite and hematite. Samples with high redness values (N120E69 B6, N114E69 C3, N118E68 B2, and samples of Localities 2 and 3) contain more hematite. Semiquantitative analysis of the DRS measurements shows that samples of H2 and H3 contain less goethite than hematite, and samples of H1 contain a little bit more goethite than hematite (~ 1.5 times). However, samples from the unburnt areas all contain more goethite than hematite. The goethite content of

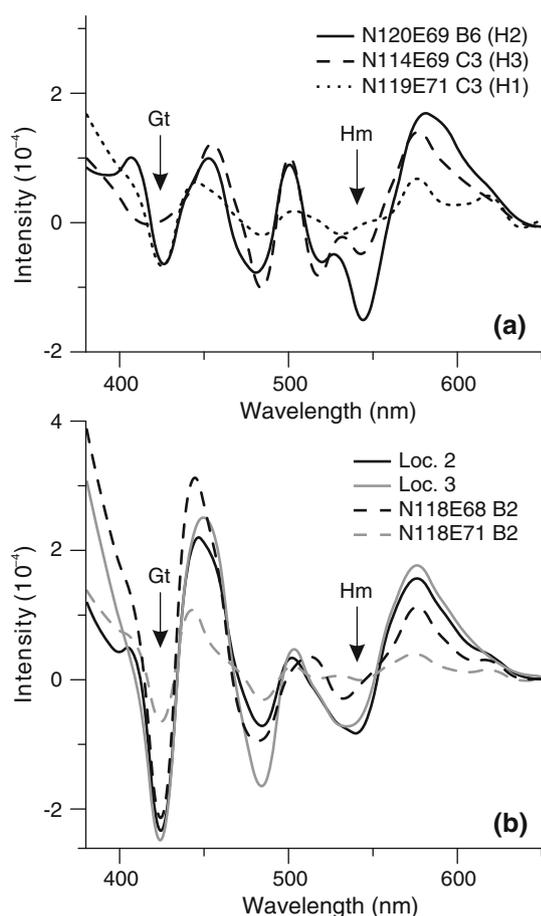


Fig. 5 The second-order derivatives of the K–M functions. Gt and Hm indicate the absorption band of goethite (~ 425 nm) and hematite (~ 535 nm), respectively [36, 41]

Localities 2 and 3 samples is much higher than that of the excavation area (except for sample N118E68 B2, of which goethite content is close to Localities 2 and 3). The hematite content of Localities 2 and 3 samples is lower than that of the burnt area, but much higher than that of other excavation area.

In summary, samples from both the burnt and unburnt areas contain magnetic minerals of magnetite, hematite, and goethite. However, samples from the burnt areas contain more magnetite and hematite. Difference of magnetic susceptibility and redness in different areas is mainly controlled by the content of magnetite and hematite. High values of magnetic susceptibility and redness in the burnt areas are, respectively, attributable to the newly formed magnetite and hematite grains during heating process.

4 Discussion

Previous researches of field experimental fires show that controlled fires (e.g., campfires, which are usually kept

burning over a certain period of time or are burnt repeatedly) generate a significant magnetic enhancement [25]. The post-burn magnetic susceptibility is at least 50 % higher than that of adjacent unburnt control samples [25], and even could be up to 50 times of the pre-burn magnetic susceptibility [20]. Natural burning processes (e.g., tree stump fires, grass fires, etc.) only make very small changes in the magnetic susceptibility of the sediments [25]. Difference between burning temperature of human use of fires and natural fires causes the distinction of magnetic susceptibilities. The highest temperature on surface of the central area of the hearth can be above 600 °C [20]. And a maximum temperature of 860 °C was recorded in the work of Berna et al. [42]. Temperatures of the periphery are much lower, usually below 300 °C [20, 21]. However, natural fires can't generate sufficient heat to produce distinct changes in magnetic mineral of the sediments because of the lower temperatures (usually below 300 °C) [25]. The Peking Man cave at Zhoukoudian was formed in carbonate rock area, where the magnetic susceptibility of bedrock is close to zero. The magnetic susceptibility of the burnt sediments is much higher than that of the bedrock, topsoils in the vicinity of Locality 1 and natural sediments of Localities 2 and 3. Therefore, sediments with extremely high χ_{LF} very likely had experienced high-temperature events. The results of χ - T curves illustrate that sediments of the burnt areas could have been heated up to 700 °C. As mentioned above, temperatures of natural fires are generally much lower. Hence, the distinct enhancement of magnetic susceptibility of the sediments of the burnt area is most likely due to human controlled use of fire.

Transformation of metastable iron-containing silicates/clays to stronger magnetic minerals (e.g., magnetite) leads to the enhancement of magnetic susceptibility [28, 37, 40]. Furthermore, at temperatures between 250 and 400 °C, goethite dehydrates and transforms to hematite [29], which causes the red coloration. Results of DRS show that all samples contain goethite. In samples of H3 and H2, the ratio of goethite to hematite is lower than 1. In sample of H1, even though the ratio is slightly higher than 1, it is still much lower than that of the samples of the unburnt area and Localities 2 and 3. These results indicate that the reduction in the goethite transformed to hematite due to heating. The newly generated hematite caused the reddish color of the rubified sediments in H1 and H2. At Localities 2 and 3, the sediments show reddish hue too, but their χ_{LF} are much lower. The reddish hues should be caused by weathering-generated hematite.

The generation of hematite during heating will not cause the distinct increase of χ_{LF} , since χ_{LF} of hematite is much lower than that of magnetite. Above 400 °C, magnetite can be generated by reduction of hematite [28, 43, 44], which apparently will increase the χ_{LF} . And the results of

χ - T curves illustrate the heating temperature could be above 700 °C.

Heating changes grain size distribution of magnetic particles. Newly generated hematite grains by dehydration of goethite are generally SP [29]. Previous studies show that large amount of ultra-fine (SD and SP) grains are generated during heating [23], and significant amount of SP magnetite grains were detected in burnt clay [30]. χ_{FD} and χ_{FD} % data indicate that the sediments of the burnt area contain large amount of magnetite grains ranging across the SP to SD boundary, which are the major contributor of the high values of χ_{LF} . These grains were very likely to be generated during anthropogenic use of fire.

5 Conclusions

We have examined the burnt and unburnt sediments of the newly excavated area in Layer 4, Locality 1, Zhoukoudian by detailed magnetic, color, and diffuse reflectance spectrum measurements. The main conclusions are:

- (1) Sediments from the burnt area have distinctly high values of χ_{LF} and redness, especially those of H3 at south end of the excavation area, whose χ_{LF} is 22 times of the adjacent areas. χ - T curves suggest that these burnt sediments could have been heated above 700 °C.
- (2) Burning caused the new formation of large amounts of magnetite and hematite grains, which are responsible for the distinctly high values of χ_{LF} and redness in the burnt area of Layer 4.
- (3) Those changes in χ_{LF} and redness are impossibly resulted from natural fires, thus most likely signaling the human activities of controlled use of fire. However, additional work is needed to confirm whether or not these heat-affected sediments were occurred in situ.

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