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COMPARATIVE HEATING EXPERIMENTS ON FLINT FROM THE CÔTE CHALONNAISE, BURGUNDY, FRANCE

ABSTRACT: During the last 50 years, heating experiments on different variations of siliceous rocks have been conducted. In the beginning, the primary focus was on North American variations, whereas varieties deriving from sites in Europe were largely disregarded. The aim of this article is to contribute to filling in some of the gaps in current knowledge through experimental heating of flint from the argiles à Silex of the Côte Chalonnaise in southern Burgundy, France. Samples of the raw material were exposed to different temperature levels for a specified time span under controlled conditions in a muffle kiln. The goal of these experiments was to define the features of raw and heated flint and to find relationships between the appearances of those features at different temperatures. Apart from global observations and characteristics occurring under the exposure to heat, we show a new comparative method concerning the colour change of the samples. Hereunto, we use the RGB-colour values of specific parts of the samples (before and after the heating) and visualise the colour change in tree-dimensional vector graphics. The results indicate that nearly all of the pieces' surfaces show a colour shift. This experimental research indicates a trichotomy of the heated material. In the lower temperature spectrum (200°C and 250°C) the pieces are nearly in the colour range of the raw pieces, the middle ones (300°C and 350°C) show a colour shift (reduction in brightness and a shift into the spectra of higher red and blue content) as well as fragmentation. In the higher temperature range (above 350°C) the pieces tend to be more greyish and are often complete disintegrated.

KEY WORDS: Use of fire – Middle and Upper Palaeolithic – Flint from the argiles à Silex – Germolles – Grotte de la Verpillière I and II – Saône-et-Loire – Colour measurement – RGB values

INTRODUCTION

Artefacts bearing evidence of exposure to heat are far from rare in the Palaeolithic record. As today, heat and light were essential to hominine's life in Palaeolithic times. Around MIS 9 (ca. 337 to 300 ka BP) the first clear evidence for the use of fire can be seen in Europe (Roebroeks, Villa 2011). The use of fire for a variety of

purposes is a common feature at sites dating to the Middle and Upper Palaeolithic.

The works of Don Crabtree in the 1960s sparked interest in Palaeolithic research on the heat-treatment of siliceous lithic raw materials for technological improvement (Crabtree, Butler 1964). In the following decades many other researchers explored the effects of heat on the quality of siliceous rocks for better knapping conditions (e.g. Beauchamp, Purdy 1986, Bleed, Meier 1980, Domanski, Webb 1992, Gould 1976, Griffiths *et al.* 1987, Johnson 1985, Melcher, Zimmerman 1977).

Research on the effects of heat on silicates in prehistory was developed on lithic raw materials used by Palaeoindian peoples from North America (Crabtree, Butler 1964, Gould 1976, Hong, Shen 2009, Luedtke 1992, Speer 2010) or Australia (Domanski, Webb 1992, 2007, Domanski *et al.* 1994, Mercieca 2000, Mercieca, Hiscock 2008, Webb, Domanski 2008). In Europe, siliceous rocks from England (e.g. Griffiths *et al.* 1987, Luedtke 1992), Poland (Bobak *et al.* 2008, Domanski *et al.* 2009) and Germany (e.g. Weiner 1985, Weiner 2012) were also tested. In France and Spain, there is evidence of heat-treated lithic raw materials for laurel-leaf points of the Solutrean (Aubry *et al.* 2003, Bordes 1969). The tested raw materials from France are mostly derived from sources in the southwest and the Paris basin (e.g. Inizan *et al.* 1977, Tixier, Inizan 2000), and no data on the effects of heat treatment on lithic raw materials from southern Burgundy (i.e. Dept. *Saône-et-Loire*) appears to exists.

In this article, we will use the word "Silex" as umbrella term for siliceous rocks (materials containing SiO_2) like flint and chert (both biogenic sedimentary rocks) or quartz or quartzite (metamorphic rock), as used in German literature (Floss 1994).

Research on unmodified flint from the *argiles à Silex* (henceforth abbreviated as FAS) in southern Burgundy has focused on the distribution and raw material properties, as well as the establishment of a representative sample of raw material types (*Lithothek*), and was recently conducted by the *Groupe de Recherche Archéologique de Tournus* (*G.R.A.T. Tournus*, see also Rué 2000). For the southern part of *Saône-et-Loire* (*Mâconnais*) research about distribution and material properties was made first by one of the authors (Floss 2003). Up to now, no analysis of burnt FAS is documented. Our research question was therefore to define and compare the properties of raw and heated FAS in the greater area of *Chalon-sur-Saône* (*Côte Chalonnaise*) in Southern Burgundy, France. Our experimental study was based on the following questions: Are the characteristics of burnt FAS clearly distinguishable from those of the unheated material? Is it possible that lithic material heated at a low temperature shows diagnostic features? Are differences in heating temperature observable on these raw materials?

Explanations for the presence of burnt Silex in Palaeolithic sites

There are several possible explanations for the presence of heated Silex in archaeological assemblages. High temperatures can be used to alter the chemical and physical structures of lithic raw materials to lower the fracture toughness for better knapping properties (Beauchamp, Purdy 1986) or to change the colour (Inizan *et al.* 1993, Kenoyer *et al.* 1991). This intentional alteration is commonly called heat treatment (Bleed, Meier 1980, Crabtree, Butler 1964, Domanski *et al.* 1994, 2009, Griffiths *et al.* 1987, Mercieca, Hiscock 2008). Secondly, natural processes like forest/bush fires (DeBano *et al.* 1998) or volcanic activity (e.g. Luedtke 1992) can create patterns diagnostic of heating. We would call this natural burning or natural heat influence. Thirdly, Silex can be thrown into fireplaces by hominines to get rid off waste (Sergant *et al.* 2006). Fourthly, a fire may be constructed on a place where Silex artefacts happened to lie, which would also result in heating. We would call this a palimpsest (Bailey 2007, Henry 2012, Lucas 2010). This list in not exclusive, but includes the primary sources of heated lithic materials encountered in the archaeological record.

Evidence of fire in the *Grottes de la Verpillière* I and II

The adjacent cave sites of the *Grottes de la Verpillière* I and II (abbreviated as VP I and VP II) are located in a Jurassic rock-formation near the small village of *Germolles* (*commune Mellecey*, Dept. *Saône-et-Loire*, France). VP I has been known to archaeologists since the end of the 19th century for its richness in late Middle and Early Upper Palaeolithic artefacts (e.g. Desbrosse *et al.* 1976, Dutkiewicz 2011, Floss 2005, Frick 2010, Méray 1876). Our team discovered the VP II (50 m to the south) in 2006. Since then new excavations are being conducted in both caves under the direction of one of the authors (Floss 2006, 2007, 2008, 2009, 2010, 2011, Floss *et al.* 2013a, b). The deposits span from the late Middle to the middle Upper Palaeolithic (for the sites see also *Figures 1, 2*).

As 98% of the exploited lithic material in both caves is flint from the *argiles à Silex* (Floss *et al.* 2013a, b), this was the raw material selected for our heating

experiments. While there are a number of direct and indirect forms of evidence for the use of fire at Palaeolithic sites, the on-going excavations in the intact late Middle and Early Upper Palaeolithic layers of the VP I and VP II (Floss 2006, 2007, 2008, 2009, 2010, 2011, Floss *et al.* 2013a, b), have yielded surprisingly little material that is visibly affected by heat. Burnt Silex is very rare in both caves, as is burnt bone. In the intact layers from the VP I, for example, in the campaign 2012 we detected 25 obviously burnt Silex artefacts (unburnt, n = 564); in VP II we detected 32 burnt Silex artefacts (unburnt, n = 513). VP I yielded 50 fragments of charcoal. In contrast, 549 charcoal fragments were found in VP II (mostly very small fragments). For VP I, the richness of tools made from Silex and bone from old excavations (before the year 2006) is now well

documented (Dutkiewicz 2011, Floss 2005, Floss *et al.* 2012, Floss *et al.* in press, Frick 2010, Frick *et al.* 2011). It is important to note that the site was also used at the beginning of the 20th century to define the Aurignacian (Breuil 1911), mostly because of its numerous carinated pieces. From the back dirt of the old excavations (between 1868 and 2005) we have additional evidences for the fire use, like a potential fire-starter (marcasite nodule with trace marks; see Fig. 53 in Floss 2009) and a small amount of burnt Silex and bone (Floss 2006, 2007, 2008, 2009, 2010, 2011). In the Upper Middle Palaeolithic layer (GH 3) of VP II, we can see a clear horizontal distribution of very tiny charcoal pieces, which occur in clusters (see *Figure 3*). The distribution of burnt bone and Silex is not as obviously clustered as the charcoal. The Upper Middle Palaeolithic layer (GH
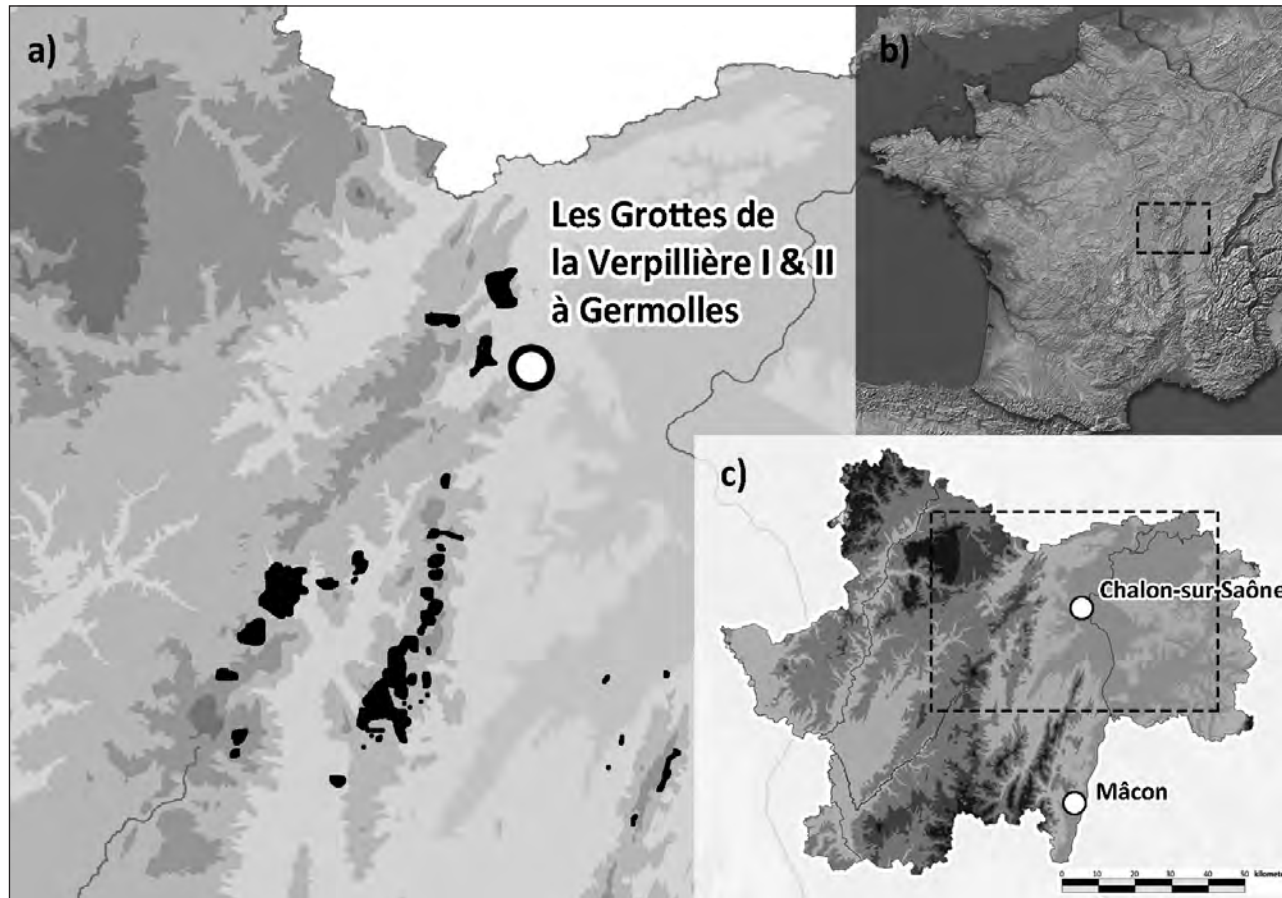

FIGURE 1: Area of work with a) the known source distribution of Flint from the *argiles à silex* in the *Côte Chalonnaise* (black spots, see Rué 2000) and the position of the *Grottes de la Verpillière I* & II in Germolles (circle), b) location of the department of *Saône-et-Loire* in France (SRTM-NASA map from 2003), c) location of the *Côte Chalonnaise* in the department of *Saône-et-Loire*. Map designed by J. A. Frick, base map by C. T. Hoyer, see, Floss *et al.* in press).
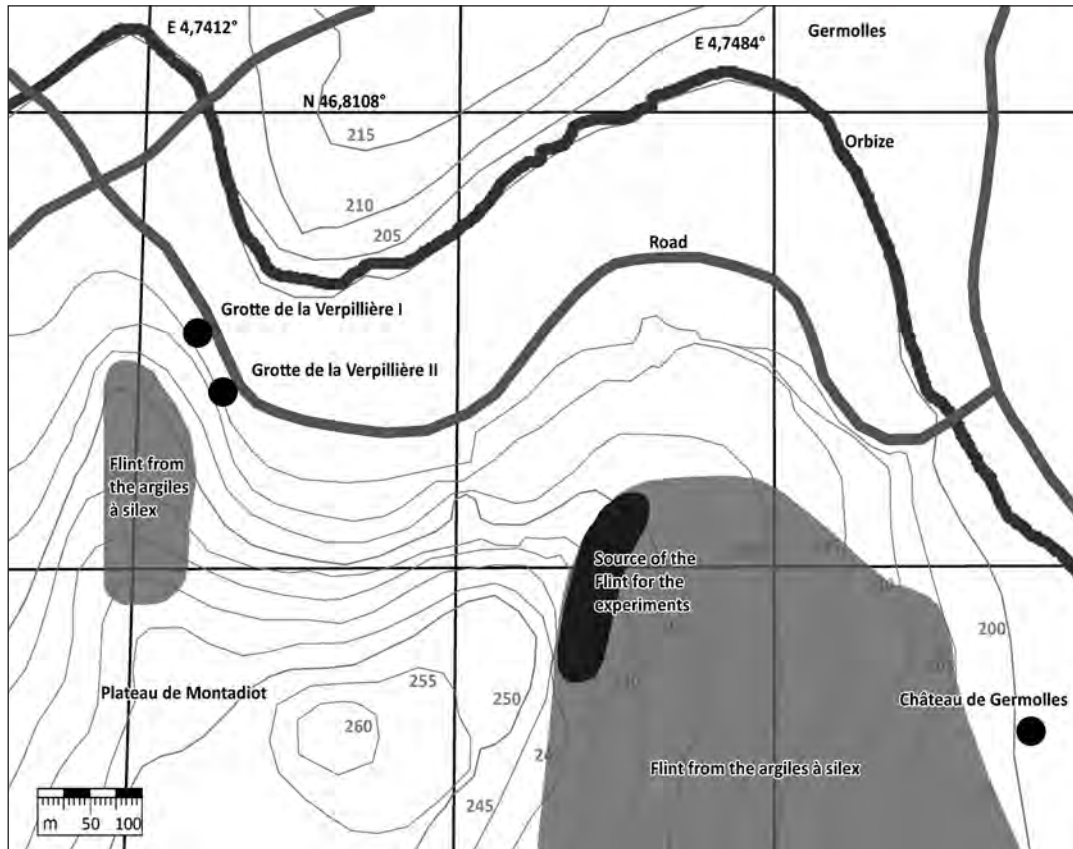
FIGURE 2: The inner logistic area of the *Grottes de la Verpillière* I & II. In shaded grey: The known sources of the Flint from the *argiles à Silex*; in black: the source of the material used in this experiments; black dots: position of the *Grottes de la Verpillière* I & II as well as the *Château de Germolles*. Map designed by J. A. Frick, base map: *carte topographique* TOP 25, Beaune-Chagny from the *Institut geographique national*, IGN 2005 and GoogleEarth map from 2011).

15) of the VP I yielded a little depression of around 20 cm in diameter with some small and one big piece of charcoal (*Figure 4*), but the sediment around showed no influence of fire (Floss 2010, 2011, Floss *et al.* 2013a, b).

While there are many ways to detect or confirm the use of fire in a site, visual identification of burnt or heated materials during excavation or artefact analysis is of central importance. The absence of a comparative or reference collection for heated lithic materials of the FAS in the region makes the positive identification of heated lithic material challenging. One of the purposes of the heating experiment, therefore, was to improve identification of heated lithic materials through the documentation of the effects of heat on the local raw materials and to build up a reference collection (*Lithothek*).

COMMONLY RECOGNISED FEATURES OF BURNT OR HEATED SILEX

The commonly described features occurring from the heat exposure of siliceous rocks are colour change, unique breakage patterns, and development of lustre. These features can be observed in both intentional and non-intentional heating. However, differences in the intensity and the specific appearance of these features can occur based on the material itself and its contact conditions to the source (direct/indirect, fast/slow and high-heat/low-heat). Here only a brief overview is given to summarise the general observations and alterations, as a foundation for the discussion of our experimental results below.
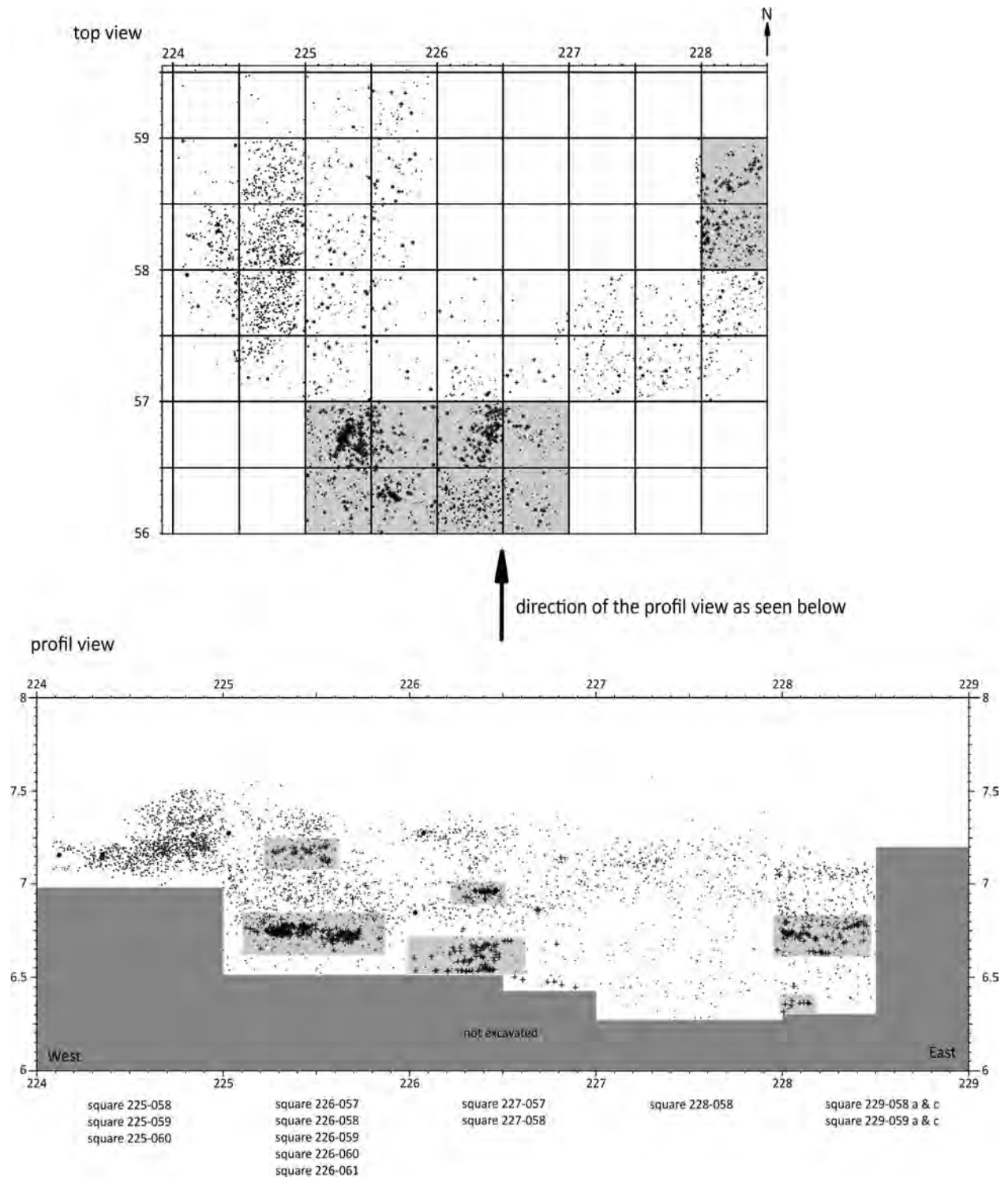
FIGURE 3: Single finds distribution of GH 3 at the *Grotte de la Verpillière* II (campagne 2010–2012). Top: top view of all single finds (Silex, bone, tooth, ivory, charcoal) from the GH 3, the dense accumulations of charcoal (crosses) are marked as light grey areas. Bottom: profile projection of all single finds from the GH 3, the layered accumulations of charcoal are marked as light grey areas. Designed by J. A. Frick, base plot by C. T. Hoyer.
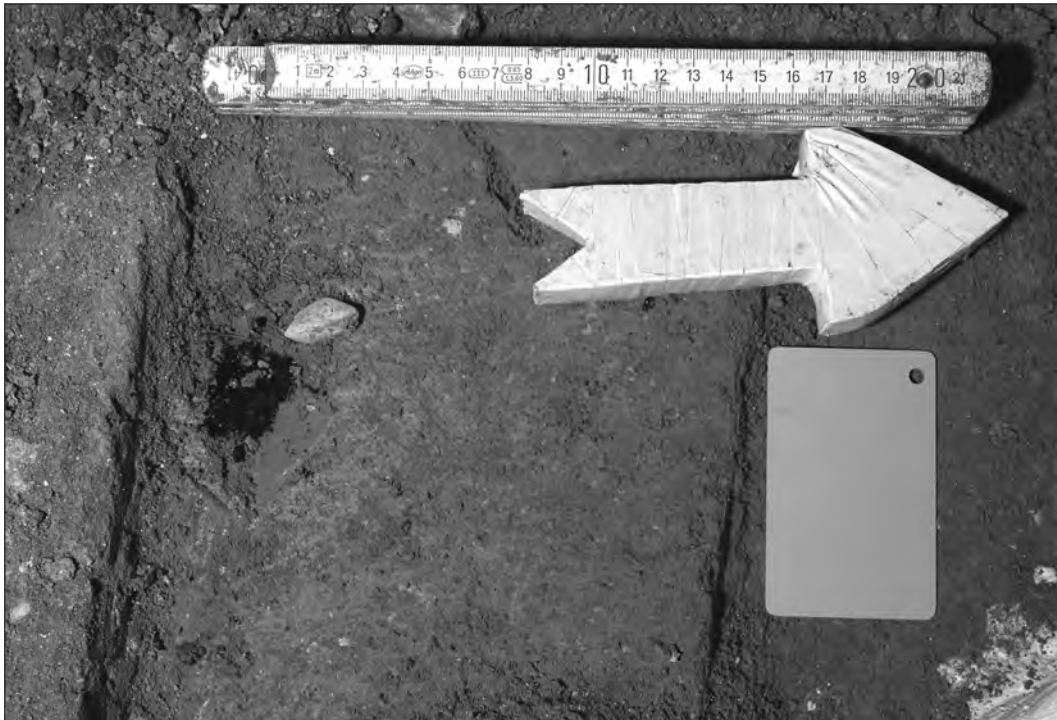
FIGURE 4: Depression in the sediments of GH 15 with charcoals from the *Grotte de la Verpillière* I (campagne 2010). Photo by E. Dutkiewicz.

Colour shift

First of all, a more or less slight shift of colour can be observed during the heating process, mostly in a kind of a rubification of the material (shift to red, either in the whole specimen or localised parts of it). This effect is due to the oxidation reactions of iron oxides present in the silicate, mainly the transformation of goethite ($HFeO_2$) to hematite (Fe_2O_3) (Gualtieri, Venturelli 1999). This transformation is especially apparent in yellow and brownish parts of the flint, which show an obvious colour-shift to a (dark)-red or pink coloration depending on the quantity if iron present (Luedtke 1992).

Breakage patterns

Another well-known feature produced by heating is the occurrence of pot-lid structures (Patterson 1995, Richter *et al.* 2011). These round-to-oval spalling patterns occur only during the heating process and not during re-cooling to room temperature (similar spallings can also occur during frost processes through expansion of water and change of the aggregate state to ice, but are not part of this paper). The negatives as well as the pot-lids show a "point of departure" (bulb) of the detachment in the centre of the structure. The diameter of pot-lid structures range in general from 1 to 60 mm (Patterson 1995). Two variations can be observed. The first variation could be described as "true" pot-lid fractures, consisting in pot-lid negatives on the pieces as well as their corresponding flakes, as described above (Patterson 1995, Richter *et al.* 2011). The second group is formed by round or oval cracks in the surface (sometimes overlapping each other) occurring in the above mentioned size range, but without full detachment of the flakes (Richter *et al.* 2011), which we will call circular breakage patterns. As far as is known to the authors, there is still no consensus on the conditions that produce either of these features (i.e. such factors as temperature, exposure time, velocity, and chemical contents or water). But the expansion of water (crystallisation water) through changing the aggregate state to gaseous seems to be an important factor.

Another well-known feature of heated silicate material somewhat similar to the fracture patterns of pot-lid structures is the crazing effect. In contrast to the pot-lid features, crazing patterns show a kind of net-structure of "irregular hairline surface cracks" (Patterson 1995), but without breakage. Depending on the duration and/or intensity of heating, this kind of thermal damage affects

only the exposed surface of the specimen. A variation on this effect called "pseudo-crazing" is also recognised, where "the pattern involves only colour-change without actual surface cracks" (Patterson 1995). Crazing can also extend deep into the material structure to the point of complete failure, resulting in rectangular blocky and granular debris or leads deep into the material structure to the point of complete disintegration (Patterson 1995). The full breakage of the heated material in the form of any kind of fragmentation or disintegration is widely attributed to very high temperatures, to thermal-shock effects due to expanding water enclosed in the silicate (Luedtke 1992, Rottländer 1989) or to tension variations in the lattice of the material itself (Weiner 2012). For the primary breakage patterns, a more detailed consideration will be given below. Finally, the relationships between specific breakage patterns and conditions such as temperature, duration of heat exposure and, of course, the raw material, remain to be discussed in a broader comparative context.

Lustre

Finally, even though there is an on-going disagreement concerning this topic, we do not wish to neglect the occurrence of changes in lustre of heat-exposed siliceous surfaces (Griffiths *et al.* 1987, Purdy, Brooks 1971, Rottländer 1989, Schindler *et al.* 1982, Weiner 2012). The question is, if the lustre develops through the heating process or other surfaces get dull through heating. In any case, there seems to be a common consensus about the macroscopic appearance, referring to a "waxy lustre" (Patterson 1995), generally observable on fresh flake negatives after the exposure to fire or heat (Collins, Fenwick 1974, Patterson 1995, Rick, Asch 1978, Weiner 2012).

MATERIALS AND METHODS

Sources

For the experiments, we used local flint found in the very proximity of the VP I and II caves. More specifically, these sources were outcrops in the forest and on cow pastures at a distance of around 100 to 500 m from the caves (see *Figure 2*).

Under the humus, there are clayish sediments from erosion processes dating to the Eocene epoch (Rat 2006). These contain siliceous nodules: the so-called flint from the *argiles à Silex* (Collenot 1876, Dewolf 1970, 1976), which is a residual Silex. Build in the Upper Cretaceous (Campanian), the sediments containing flint were eroded in the Eocene and exposed mostly on the tops of the hill

ranges between *Mâcon* and *Chalon-sur-Saône* (Rat 2006, Rué 2000; see, also *Figure 1*).

Appearance

The FAS often contains cracks, micro-cracks, fissures, and fractures, sometimes secondary healed (silicified) cracks. Zones of calcite crystals can be seen (pers. comm. P. Schmidt) where the degree of silicification was too low. The translucence of the material ranges from clear/glassy in dark brown and black colours to opaque in grey, greenish, bluish and brown-orange colours, with a prevalence of grey variants. The more homogeneous the interior of a nodule is, the better is the knapping quality in general. But the nodules are not exclusively either of good or bad quality. Many of the tested nodules contained areas of excellent quality and those of low quality. The cortex is commonly smooth and on average between 1 and 2 mm thick. The colour of the cortex ranges from light grey to dark grey, with some brown variants. We also observed an orange banding directly under the cortex with a high content of iron-oxide (see *Figure 5*).

Procurement and knapping

During the 2012 excavation season at *Germolles*, nodules of the local FAS were collected and knapped to produce simple flakes, bladelets, and cores. To provide a sampling of Middle and Upper Palaeolithic lithic industries, we knapped with different techniques (hard quartzite hammers and soft antler billets, as well as antler punches). The aim of the production process was to have lithic cores and blanks with and without cortex from local material of differing quality. One group of blanks (flakes, bladelets and blades) was unmodified, while another was modified by surface and edge retouch to create, for example, bifacials and scrapers.

Uncontrolled test heating at a fireplace

To gain preliminary insight, the first knapped series was used in less controlled pilot experiments in the fireplace in the garden at the excavation house (this series was "less controlled" in the sense that the open fire did not allow for the level of control of experimental conditions that kiln-firing presented, see *Figure 6*). The macroscopically visible cracks (presenting the possibility of embedded water) and crystallised areas (calcite) in the nodules led us to heat the flint embedded in ashes under the fire (see *Figures 6, 7*). This lessened the risk of injury in the event of bursting during heating. Altogether three different series of knapped lithic objects were heated in this manner (example, see *Figure 7*):
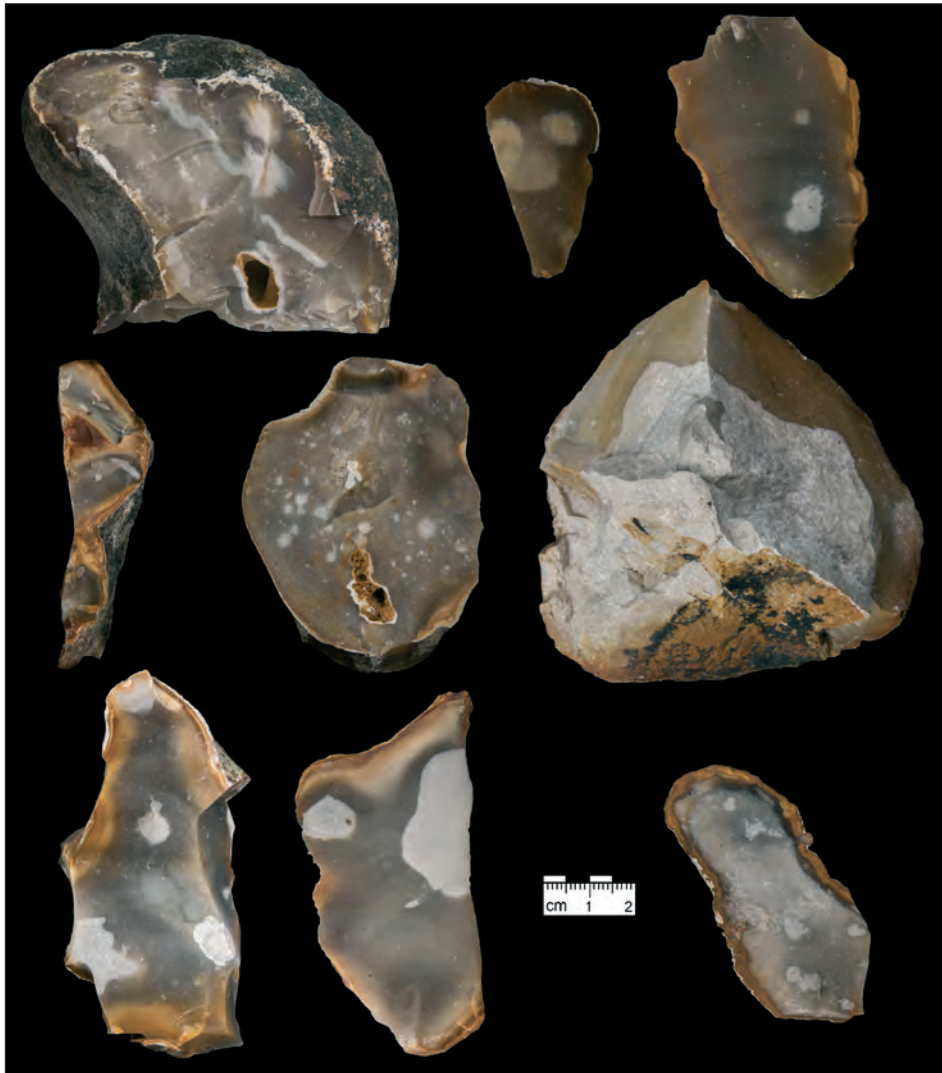
FIGURE 5: Lithic raw material variation of the experimental used Flint from the *argiles à silex* from the source showed in *Figure 2*. The most visible characteristic is the rust-coloured band below the cortex and grey spots with slightly coarser material. The centre of the nodule in the mid right contains calcite. Illustration by J. A. Frick.

1. This series contained 17 pieces (two cores and 15 flakes) selected to represent raw materials of different quality. The pieces were laid on the cleaned fireplace surface in two parallel rows from the centre of the fireplace to the periphery and covered with ashes. Above this setting a fire burned for eight hours and cooled for approximately eleven hours. After cooling, the sediments were sieved (mesh size of 1.5 mm) to recover the fragments of Silex.

2. This series contained 29 pieces (one core and corresponding flakes, blades and bladelets), representing a complete knapping sequence. The pieces were lain on the cleaned surface of the fireplace concentrically from the centre to the periphery of the burning zone and covered with ashes. Above this setting coals from the nearby fire were lain, which smouldered for approximately 17 hours. The sediment was sieved the following day (also see Fig. 6 and 7).

3. The last series contained eight pieces of roughly the same size (one core and seven flakes). These were lain onto the cleaned surface and covered with cold ashes. Coals were placed on the ashes for a period 17 hours. The sediment was sieved the following day.
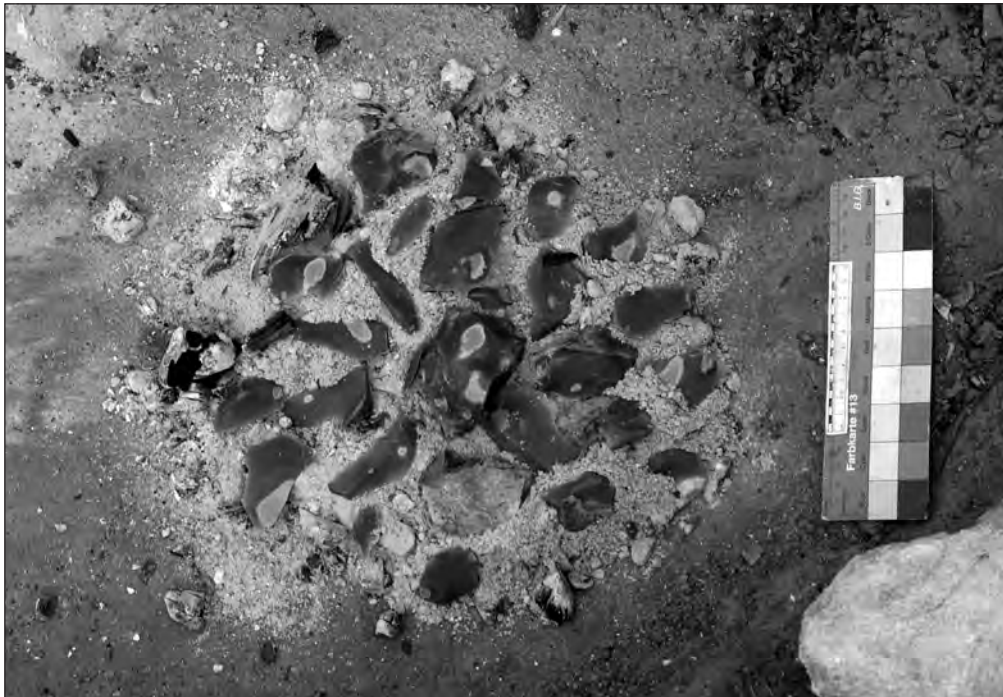
FIGURE 6: Uncontrolled heating experiment in the fireplace of test group GER12.EXP.4.2 that was a knapping sequence. The picture shows the setting of the pieces before covered with coals. Photo by J. A. Frick.
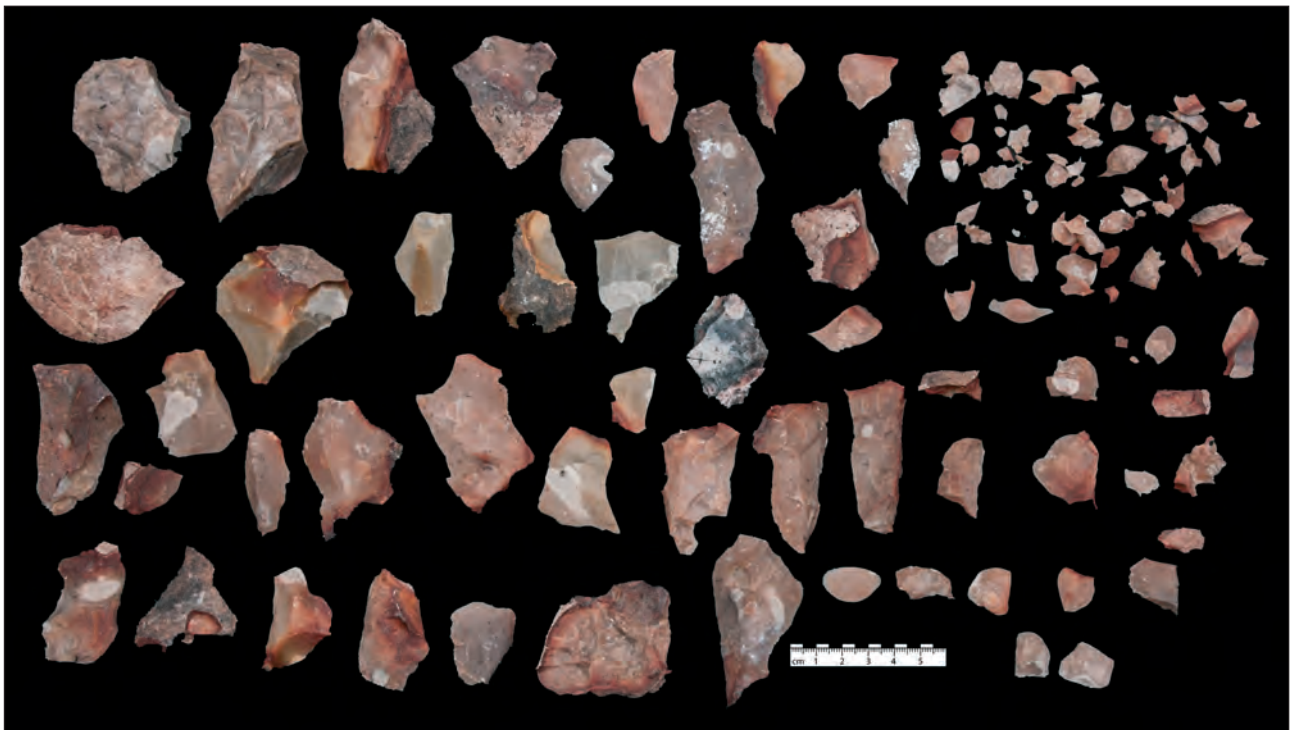

FIGURE 7: Knapping sequence showed in Fig. 6 after the heating under coal in the fireplace. Photo by J. A. Frick.

TABLE 1: Overview of all heated pieces, including amount and weight.

Sample	Number GER12.EXP.	Max. temperature (°C)	Number of pieces before burning	Number of pieces after burning (in the bowl)	Complete number of pieces after burning	Weight of sample (in the bowl) before burning, complete weight before burning	Weight of biggest piece in the bowl after burning	Weight of the sample in the bowl after burning	Weight of the pieces distributed in the oven, but connected to a sample	Complete weight after burning
Test groups	KG 1	300	44	44	44	16.1	16.1	16.1	0	16.1
	KG 2	300	1	8	8	46.6	37.2	46.6	0	46.6
	KG 3.1	450	1	Very much	Very much	347.1	14.5	113.5	286	512.7
	KG 3.2	450	1	Very much	Very much	91.7	27.5	45.8		
	KG 3.3	450	1	Very much	Very much	90.6	24.7	67.4		
	KG 4.1	> 400								
	KG 4.2	Around 400		These are the uncontrolled heating experiments, no data collected						
	KG 4.3	< 400								
Sample group 1	UG 1.1	200	1	1	1	50.3	50.2	50.2	0	50.2
	UG 1.2	200	1	1	1	23.8	23.7	23.7	0	23.7
	UG 1.3	200	1	1	1	27.5	27.7	27.7	0	27.7
	UG 1.4	200	1	1	1	69.5	69.3	69.3	0	69.3
Sample group 2	UG 2.1	250	1	1	1	24.1	24.1	24.1	0	24.1
	UG 2.2	250	1	1	1	26.8	26.8	26.8	0	26.8
	UG 2.3	250	1	1	1	36.6	36.5	36.5	0	36.5
	UG 2.4	250	1	1	1	72.2	72.1	72.1	0	72.1

TABLE 1: Continued.

Sample	Number GER12.EXP.	Max. temperature (°C)	Number of pieces before burning	Number of pieces after burning (in the bowl)	Complete number of pieces after burning	Weight of sample (in the bowl) before burning, complete weight before burning	Weight of biggest piece in the bowl after burning	Weight of the sample in the bowl after burning	Weight of the pieces distributed in the oven, but connected to a sample	Complete weight after burning
Sample group 3	UG 3.1	300	1	1	1	18.3	18.3	18.3	0	18.3
	UG 3.2	300	1	2	2	51.9	49.2	51.6	0	51.9
	UG 3.3	300	1	3	1 > 1 cm, chips 0.1 g	34.3	34.1	34.2	0	34.2
	UG 3.4	300	1	3	5	68.5	58.6	63.6	4.6	68.2
Sample group 4	UG 4.1	350	1	12 > 1 cm, chips 2.2 g	12 > 1 cm, chips 2.2 g	129.3	76.6	126.9	0	126.9
	UG 4.2	350	1	2 > 1 cm, chips 0.5 g	6 > 1 cm, chips 0.5 g	79.1	10.9	18.7	59.2	77.9
	UG 4.3	350	1	3 > 1 cm, chips 0.1 g	6 > 1 cm, chips 0.1 g	15.1	4.7	7.3	7.5	14.8
	UG 4.4	350	1	2 > 1 cm, chips 0.1 g	3 > 1 cm, chips 0.1 g	24.6	20.4	22.3	1.5g	23.8
Sample group 5	UG 5.1	200	1	1	1	35.1	35	35	0	35
	UG 5.2	200	1	1	1	19.9	19.9	19.9	0	19.9
	UG 5.3	200	1	1	1	18.1	18	18	0	18
	UG 5.4	200	1	1	1	23.1	23.1	23.1	0	23.1

TABLE 1: Continued.

Sample	Number GER12.EXP.	Max. temperature (°C)	Number of pieces before burning	Number of pieces after burning (in the bowl)	Complete number of pieces after burning	Weight of sample (in the bowl) before burning, complete weight before burning	Weight of biggest piece in the bowl after burning	Weight of the sample in the bowl after burning	Weight of the pieces distributed in the oven, but connected to a sample	Complete weight after burning
Sample group 6	UG 6.1	250	1	1	1	35	34.8	34.8	0	34.8
	UG 6.2	250	1	1	1	73.5	73.5	73.5	0	73.5
	UG 6.3	250	1	1	1	7.2	7.2	7.2	0	7.2
	UG 6.4	250	1	1	1	14.8	18.8	14.8	0	14.8
	UG 6.5	250	1	1	1	21.4	21.4	21.4	0	21.4
Sample group 7	UG 7.1	300	1	2 > 1 cm, chips 0.1 g	5 > 1 cm, chips 0.1 g	145	128.7	129	14	143
	UG 7.2	300	1	1 > 1 cm, chips 0.1 g	1 > 1 cm, chips 0.1 g	6.9	7.1	7.2	0	7.2
	UG 7.3	300	1	1	1	7.2	7	7	0	7
	UG 7.4	300	1	2 > 1 cm, chips 0.1 g	2 > 1 cm, chips 0.1 g	23.1	22.4	22.8	0	22.8
Sample group 8	UG 8.1	350	1	10 > 1 cm, chips 0.1 g	16 > 1 cm, chips 0.1 g	65.5	16.8	44.2	19.5	63.7
	UG 8.2	350	1	7 > 1 cm, chips 0.4 g	8 > 1 cm, chips 0.1 g	27.9	9.1	26.4	1.2	27.6
	UG 8.3	350	1	1 > 1 cm, chips 0.1 g	1 > 1 cm, chips 0.1 g	15.2	15.1	15.2	0	15.2
	UG 8.4	350	1	1 > 1 cm, chips 0.1 g	1 > 1 cm, chips 0.1 g	5.5	4.8	4.9	0	4.9
	UG 8.5	350	1	2 > 1 cm, chips 0.1 g	3 > 1 cm, chips 0.1 g	6.2	2.3	3.6	2.3	5.9

The first two series showed the common features of burnt Silex (cracks, fragmentation, rubification and pot-lids; see one unburnt series on *Figure 6* and the same burnt on *Figure 7*). The third series, however, showed no significant features indicative of heating.

Sample's pre-treatment, measurement and collection of data

While the first experimental series yielded satisfying qualitative results, the second series was designed to yield quantitative data under the controlled conditions of heating in a muffle kiln. Before the heating process, we took digital photographs of the samples (see below), weighed them (Sartorius® BL 3100, accuracy of measurement 0.1 g) and measured the dimensions (length and width) with callipers. Each sample was given a number (GER 12 EXP.UG xxx and KG xxx). Each of the eight sample groups (Untersuchungsgruppe 1 to 8, UG 1 to 8) contained one to five pieces (n = 33). Each individual sample was placed in a separate heat-resistant ceramic bowl for heating. The sample groups contained

products of knapping sequences as well as randomly selected blanks. Two groups were heated at a time (see *Table 1*).

Heating procedure

We performed the heating procedure in the muffle kiln (Naber® N 60 H, manufactured in 1985, see *Figure 8a*) of the geographical institute at the University of Tübingen. The muffle kiln's automated timing guaranteed a uniform process of heating for maximum comparability for the results for each sample group. The heating process was comprised of the following stages: 1. Documentation of the position in the kiln of each piece in its bowl (drawings and digital pictures); 2. Continuous gradual heating up to the maximum temperature; 3. Holding of the maximum temperature for one hour; 4. Continuous gradual cooling to room temperature; 5. Documentation of the pieces with digital photography directly after removal from the kiln (see Figure *8b, c*). All samples were later weighted and photographed again. For highly fragmented pieces (distributed into the kiln),

FIGURE 8: Heating procedure. a) Muffle kiln used for the controlled experiments, b) one series of pieces before heating, c) another series after heating, in the bigger heating bowl there are the pieces distributed into the kiln. Illustration by J. A. Frick.

sorting back to the sample groups and refitting were undertaken. The digital images serve to document the condition of the samples and to perform the colour measurements described in the following section.

Colour measurement and RGB-values

Common methods for the measurement of colour are the use of a colour chart to compare the colour of the object with a colour defined in the reference (e.g. *Munsell® Color Chart*), and the use of optical and electronic equipment to measure colour values (measuring of colour values in a pixel-based system such as computer software or measuring the emission of light with a spectrometer). In archaeology, *Munsell® Soil* or *Rock Color Charts* are commonly used to describe colours (Gerharz *et al.* 1988). But as a function of available light and subjective colour vision, the comparative process is still rather subjective and introduces the possibility of inconsistent determinations. The advances in digital photographic technology provide the possibility to measure colour in a standardised and objective manner without high-cost laboratory equipment like spectrometers or colorimeters. We therefore performed the colour measurement with the help of a digital camera, a consistent and defined light source (two daylight lamps

with 5400 K, 125 W and 6000 Lumen) and widely available photo-editing software (see below). This method is often used in the food processing industry (e.g. Leon *et al.* 2006, Mendoza *et al.* 2006, Yagiz *et al.* 2009), in the study of soils (Luo *et al.* 2008) or to detect differences in snow cover (Hinkler *et al.* 2002).

Colour management systems exist in two types of systems: additive (on screens and other digital displays, e.g. RGB, L*a*b* or HSB) and subtractive (on printers, e.g. CMYK). Digital cameras and computer monitors use the additive system to display colours (where R = 0, G = 0 and B = 0 results in black; R = 255, G = 255 and B = 255 results in white, see *Figure 9*). Understanding this system aids in the interpretation of the colour data that will be discussed below.

For the reasons described above (low cost, practicality, and consistency of results), we chose to measure the RGB colour values captured by a digital camera. If pictures are taken under consistent light conditions it is possible to precisely measure the colour change before and after heating by comparing the same area of an experimental sample. One of the primary advantages of using RGB-values is that the method expresses colour change with mathematical vectors, which are ideal for comparison (Aznaveh *et al.* 2009).
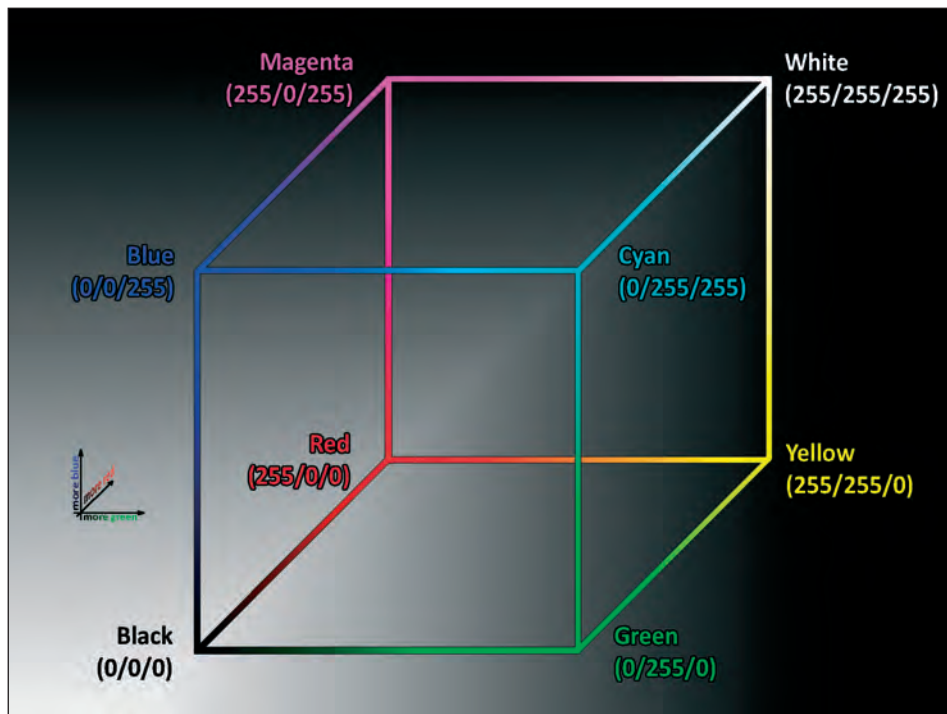

FIGURE 9: RGB spectrum, illustrated as a cube. Illustration by J. A. Frick.

We made high-resolution images (raw data in *.dng) in the colour space of AdobeRGB® (1998) under consistently controlled conditions (two daylight lamps with 5400K, a grey card, a Pentax® K200D digital camera with a Sigma® DC 17–70mm 1:2.8–4 Macro HSM lens, remote control, and tripod) of every sample before and after the heating process. Comparing the RGB-values in pictures taken under these controlled conditions is a highly accurate method for assessing the colour change due to heating.

The digital pictures were all imported, tagged and automatically white-balanced (using the grey card as a reference) in a database with Adobe® Photoshop Lightroom 4. No other changes were made to the images. The colour space of the digital camera, Adobe® Photoshop Lightroom and Adobe® Photoshop CS6 was set to AdobeRGB® (1998).

We used the "colour pipette" tool in Photoshop to measure the RGB-values. For every sample, we performed eight measurements (each 5×5 pixel) on the largest fragment remaining in the bowl. Photoshop measures the colour of every pixel of the 5×5 pixel area and provides a mean, so small inconsistencies (e.g. dust on the lenses or tiny patches of colour invisible to the naked eye) can be ignored. The same positions were measured on pictures taken before and after the heating process (see *Figure 10*). All colour values collected were recorded in an MS Excel® database.

Measurement of colour change

To quantify the colour change, simple position vectors were used because RGB values are orientated in a three-dimensional coordinate system, or vector space. By subtracting the measured "before" values from those measured after heating (see *Figure 10*), a numerical value is achieved for the colour shift. If the resulting vector is of negative value, this corresponds to a darker colour shift in the measured region. If the resulting vector shows positive values, the colour has shifted to the brighter regions of the vector space.

We processed the collected raw data of the RGB values in GoCAD®. The axes of the coordinate system match the three colours of the RGB-scheme (red, green and blue) and can be seen by the arrows in the lower left corner of each diagram. Overall, the pattern suggested by the "before" and "after" values (*Figure 11a–c*) indicates a shift of the spectrum of colours from green into a more reddish area, with a more consistent ratio of blue. A general trend of decrease in the brightness level can also be observed.

Measurement of fragmentation

Fragmentation is a common feature of burnt siliceous rocks, and was therefore another aspect we attempted to quantify in our experimental samples. To that end, we measured the weight of the samples and counted the number of pieces before and after burning (pieces stayed in the
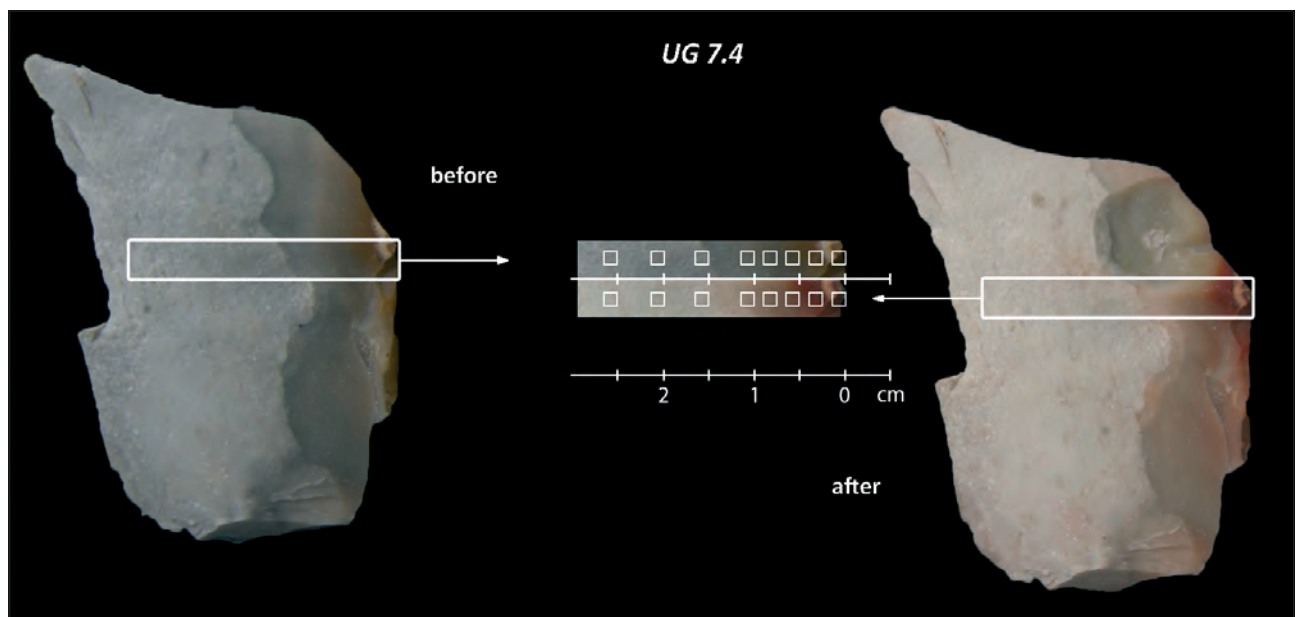

FIGURE 10: Procedure of colour measurement before and after heating. Here illustrated one piece from sample group 7 with 300°C (left, piece before heating; middle, scale of the measurement areas; right, piece after heating). Illustration by J. A. Frick.
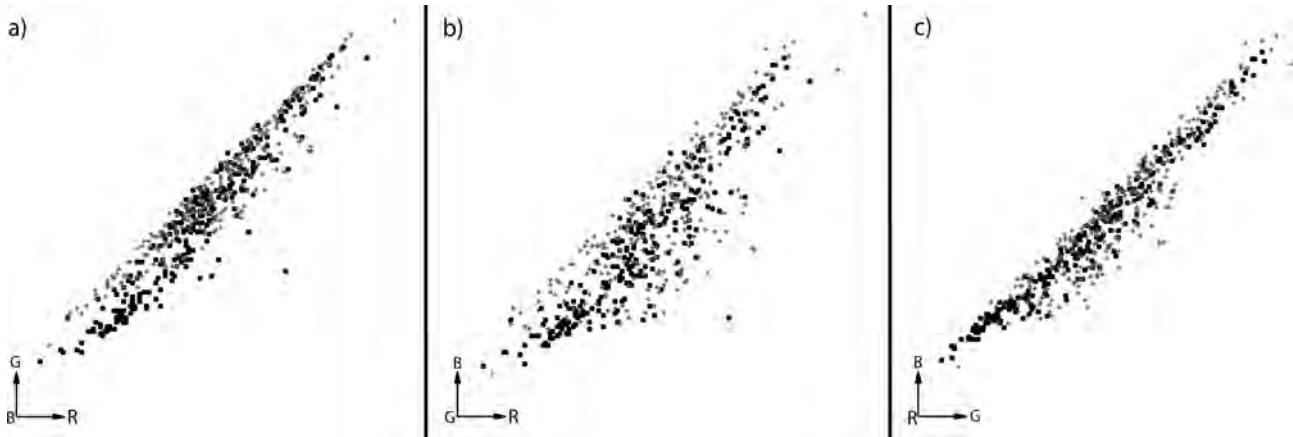
FIGURE 11: Three-dimensional graphic of all starting (before, grey crosses) and end (after heating, black dots) values indicates a shift from the green into the more reddish area with a more consistent ratio of blue. The trend of a brightness decrease can be observed. Illustrated in the view directions, a) spectrum between green and red, b) spectrum between blue and red and c) spectrum between blue and green. Illustration by C. T. Hoyer.

heating bowl as well as spread into the kiln; see *Table 1)*. We also counted the breakage pattern types and established two degrees (one for fragmentation and one for distribution).

Analysis of breakage patterns

In general, there is a notable difference in the breakage patterns of knapped and heated silicates. In the knapping process, the force used to fracture the material acts from the outside in one direction (in the case of a hand-held core) or two directions (use of an anvil in bipolar knapping). In heating and cooling processes, the splitting force originates from the inside and is completed by a process of expansion, mostly that of water (e.g. Rottländer 1989). If the material is brittle, such expansion leads to fracture.

According to the most commonly described thermal breakage-pattern types (see, e.g. Patterson 1995, Purdy 1971, 1974, Rick, Asch 1978, Rick, Chappell 1983, Weiner 2012), we established the following reference list for the comparative analysis (see *Figure 12*):

Complete disintegration of the heated piece. This tends to happen when the piece is "overheated," and causes granular, blocky heat debris (Patterson 1995). If a piece is fragmented through exfoliation of pot-lids this can also lead to a complete destruction.

Circular breakage patterns of non-detached pot-lids. These are roundish cracks that can lead later to the detachment of pot-lids from the surface (Richter *et al.* 2011). There is a clear distinction observable between this pattern and the crazing.

Pot-lid fractures. These heat-flakes are of round or oval shape and have a bulb in the centre of the "ventral face". Further pot-lid fracture can also occur on the "ventral face" of other pot-lids (Patterson 1995, Purdy 1975, Rick, Asch 1978). If a pot-lid is big enough other pot-lids can also be detached from its "dorsal face".

Flake splitting. Separation of a flake into two pieces horizontally at the reference plane (thought surface between the ventral and dorsal surface of a flake).

Transverse fracture. Splitting of pieces transverse to the reference plane. These breakage surfaces are often wavy. The difference between transverse fracture and split fractures from an exterior force is often difficult to see (Patterson 1995).

Surface Crazing or craquelage. These are irregular hairlines that are often explained as a result of material shrinkage. When they penetrate beyond the material's surface, it seems that these patterns lead to a blocky and rectangular disintegration.

Fragmentation and distribution through explosion

To quantify the degree of fragmentation and the degree of distribution of the heated pieces, two ratios were established. The degree of fragmentation (DoF) is the ratio obtained by dividing the mass of the sample fragments remaining in the bowl by those spread out in the kiln: $DoF = M_{oven} / M_{bowl}$. The DoF is always > 0. If the value is zero, nothing is fragmented and all stayed in the bowl. The degree of distribution (DoD) of the pieces of one sample is measured by dividing the mass in the bowl before the heating from the mass after the heating: $DoD = M_{after} / M_{before}$. The DoD ranges from zero to one. Zero means that there was no distribution out of the bowl. One means that all is fragmented or distributed.
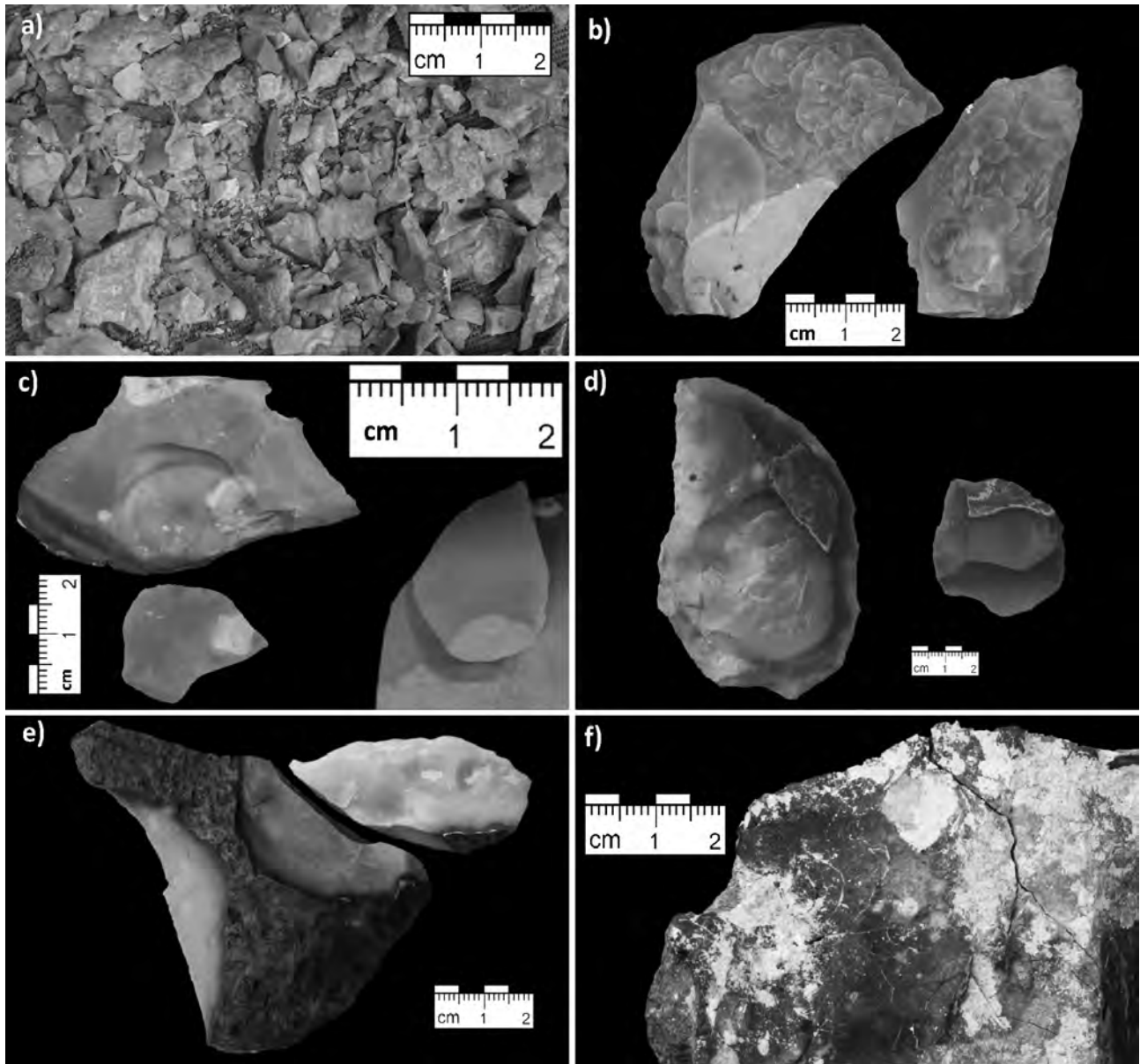
FIGURE 12: Observed breakage pattern types at the controlled and uncontrolled heating series. a) Complete disintegration (sample KG 3.1 at 450°C), b) circular breakage pattern (sample KG 4.2 at < 400°C), c) pot-lid fracture (sample KG 4.1 at < 400°C and UG 7.4 at 300°C), d) flake splitting on an experimentally knapped Keilmesser and its corresponding "flake" (sample UG 7.1 at 300°C), e) transversal fracture (sample UG 4.2 at 350°C), and f) surface crazing (sample KG 4.3 at < 400°C). Illustration by J. A. Frick.

COMPARISON AND RESULTS

In some aspects, the studied Silex shares features observed for other variants of the material found in the literature. Our main focus was the measurement and description of colour change and fragmentation patterns in heated FAS from the *Côte Chalonnaise*. In brief

conclusion, we can state that all commonly described breakage pattern types (Patterson 1995) were observed in our experimental samples (see *Figure 12*). With the naked eye, rubification is clearly visible on the samples tested. The graphics, however, show a precisely defined colour change with a starting- and end-value within a vector space. The shift of the colour spectrum tends

from green into reddish with a relatively consistent ratio of blue, in combination with a decrease of brightness.

Comparative patterns at different temperatures

Here we summarise the observed patterns of appearance, fragmentation and colour change in the samples at temperatures of 200°C, 250°C, 300°C, 350°C and 450°C.

At 200°C

No obvious changes were observed with the naked eye on the sample groups heated at the lowest temperature of 200°C (UG 1 and UG 5). Only in direct comparison of the photographs and RGB values before and after heating was a slight change in colour observed. Apart from this, one would not be able to easily distinguish the pieces burnt at 200°C from samples of fresh and unburnt raw material (see also *Figure 13*). There was no explosive distribution into the kiln.

In these sample groups (see *Figure 14a, b*), changes in the colour values are seen by comparison of the RGB-values, but differ between the two sample groups. Sample group UG 1 (*Figure 14a*) shows a shift in the red ratio, which is split up in two clusters. While measurement points with a formerly higher ratio of red got brighter and more reddish under the influence of heat, the points with a mid-to-low range in the red ratio tended to get darker and less red in their hue or colour shades. Sample group UG 5 (*Figure 14b*), however, shows an overall shift in the percentage of red and a significant rise in brightness. The difference might due to originally different hues in both sample groups.

At 250°C

At the temperature of 250°C (sample groups UG 2 and UG 6), however, a visible colour-shift to pink and especially reddish colours on the edges of the pieces as well as right under the cortex can be observed. The inner

FIGURE 13: Illustration of the slight shift in colour at 200°C. a) piece before heating, b) piece after heating, c) spectrum between green and red, d) spectrum between blue and red and e) spectrum between blue and green. Illustration by J. A. Frick.
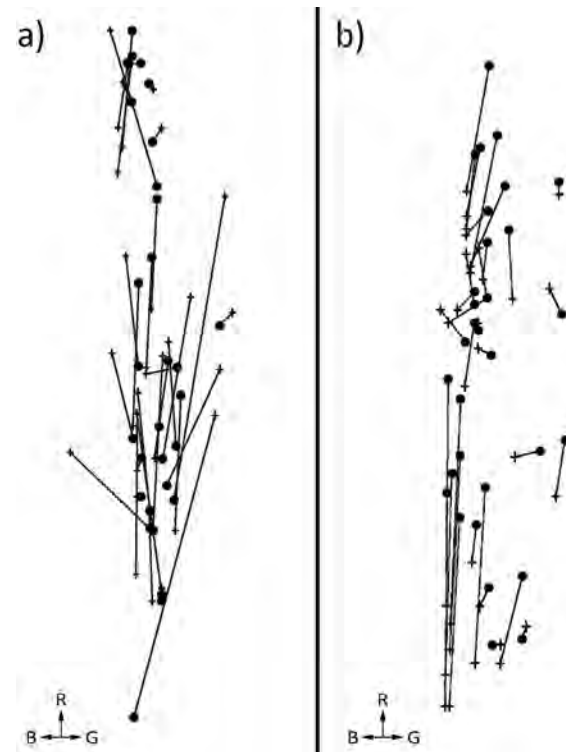

FIGURE 14: Three-dimensional graphic of 200°C. a) Sample group 1, b) sample group 5. Crosses, value of a single measure point in the RGB-space before heating; dots, value of a single measure point in the RGB-space after heating; connecting lines, shifting-vector of the change in the material colour from before to after heating). Graphic by C. T. Hoyer.

greyish parts show no visible colour alterations, but the surfaces seem a little more dull (only for the homogenous areas). At this temperature, as at 200°C, the samples underwent almost no visible fragmentation. Only some very small pot lids (< 1 mm) from the edges of the pieces could be recognised. There is also no distribution in the kiln and no other heat-produced features can be observed. In both sample groups a decrease of intensity in the red spectrum and in brightness is visible. Furthermore, a slight shift of the hues in the green direction is recorded (*Figure 15a, b*).

At 300°C

At a temperature of 300°C (sample groups UG 3, UG 7, KG 1 and KG 2) the alterations become highly visible. Most of the edges and the cortex-adjacent areas show a dark red colour after heating, and some of the inner areas become more pink. Furthermore, small cracks and a few circular cracks begin to occur and the first pot-lids are evident (5 to 20 mm in diameter). While the homogeneous glassy surfaces remain dull, the pot-lid

negatives and the "ventral faces" of the pot-lids show a kind of lustre. It is also at this temperature that the size of the sample pieces starts to play a distinct role in the process, in that larger pieces are much more subject to surface detachments or even fragmentation (in five of eight of the larger samples). In contrast, smaller and particularly flatter pieces were not affected by crazing, cracking, breaking or exfoliation (e.g. the bladelets of KG 1). The loss of material in the bowl is zero or close to zero. There is little to no mass distribution visible in the kiln. In sample group 3 (*Figure 16a*) a shift of the reddish parts in the blue direction and of the less reddish parts in the green direction is visible. In general the colours get darker and the values in the red spectrum decline. In sample group 7 (*Figure 16b*), redness and brightness intensify, while neutral and darker values are boosted in the direction of black (*Figure 16a, b*).

At 350°C

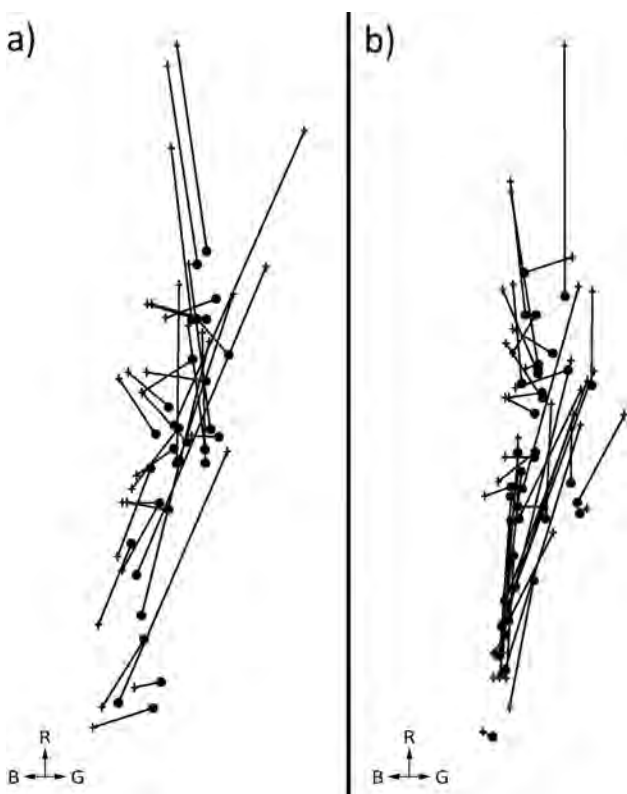Raising the temperature to 350°C (sample groups UG 4 and UG 8) resulted in even more heat-induced

FIGURE 15: Three-dimensional graphic of 250°C. a) Sample group 2 and b) sample group 6 (symbols like in *Figure 9*). Graphic by C. T. Hoyer.
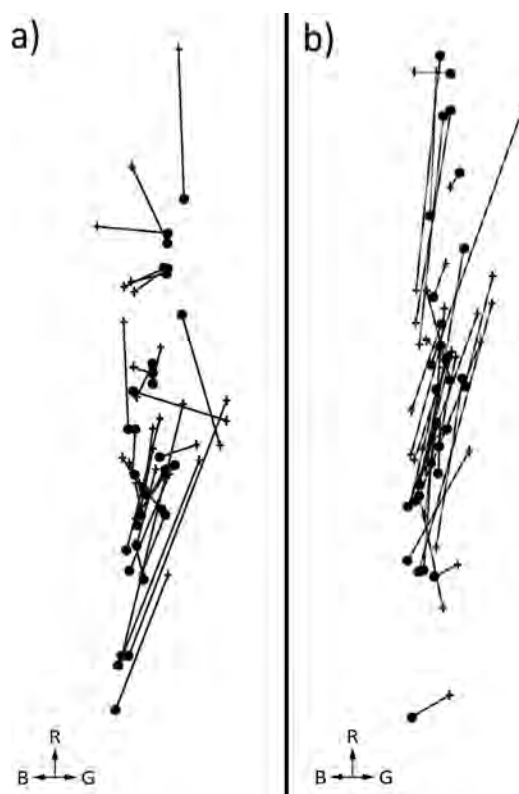
FIGURE 16: Three-dimensional graphic of 300°C. a) Sample group 3 and b) sample group 7 (symbols like in *Figure 9*). Graphic by C. T. Hoyer.
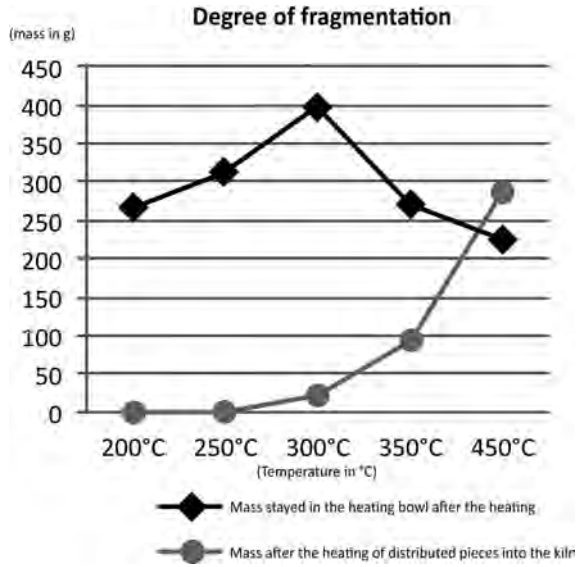
FIGURE 17: Degree of mass fragmentation after the burning process in the muffle kiln. The diagram shows the mass stayed in the heating bowl and the mass distributed out of the heating bowl into the kiln. Above 350°C more mass is distributed into the kiln than stayed in the heating bowl. Diagram by J. A. Frick.

alterations of the material. An observable colour shift to dark red and red is strongly present, especially for the edges and the cortex-adjacent parts, but also for large

areas of the rest of the surfaces. Grainy spots of the material that were formerly greyish, appeared more bleached. There is an increased amount of pot-lid flaking (100% of all pieces) observed as well as of circular cracks (88.96% of all pieces). Flake-splitting and transverse fractures are visible in 66.72% of the pieces. Crazing or hairline cracks cannot be observed, but the samples show a high fragmentation rate. At this temperature, 66.72% of all pieces suffered complete disintegration. Dull surfaces except for lustre on the pot-lid negatives remains constant. Pertaining to the degree of mass fragmentation, more material stayed in the bowl than scattered in the oven (DoF = 0.33). The DoD at this temperature is 0.23 (*Figures 17, 18*). Both sample groups show the most significant change in colour compared to those heated at lower temperatures. In both groups, a broad shift into a very dark reddish spectrum with a tendency into blue is clearly visible (*Figure 19a, b*). Only in sample group 4 do some of the values show a rise in brightness to a bright red colour (*Figure 19a*).

At 450°C

Finally, the maximum experimental temperature of 450°C (sample group KG 3) revealed a surprising characteristic concerning colour change in the material. The strong rubification is reduced again primarily to the edges and the cortex-adjacent areas while the rest attends toward a more grey spectrum. The surfaces are covered
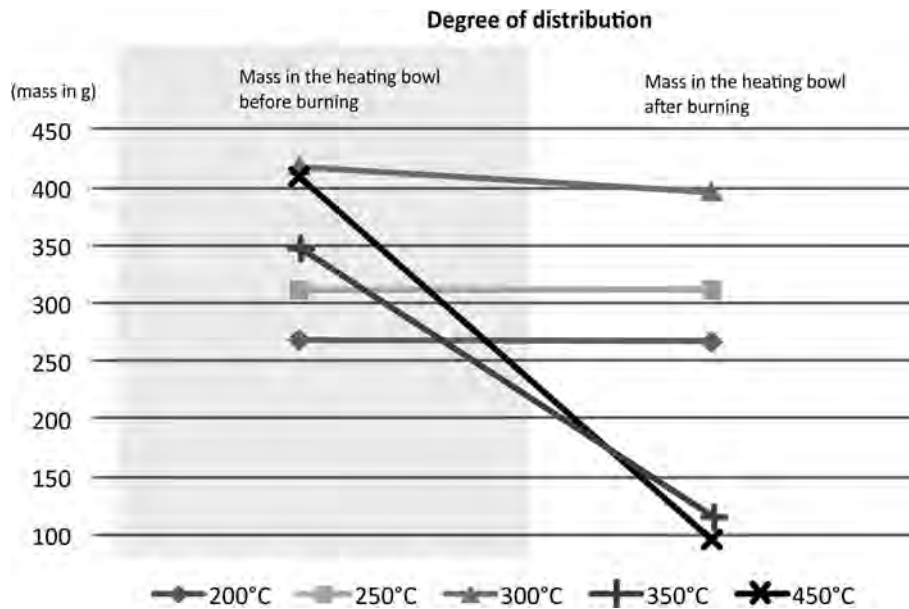

FIGURE 18: Degree of spatial mass distribution. Here the mass before and after heating is compared. Below 350°C the mass in the heating bowl stayed nearly constant. Diagram by J. A. Frick.

with pot-lid exfoliations and circular cracks as well as angular cracks that penetrate deep in the material. Flake-splitting and transverse fragmentation are also visible (see *Figure 12*). Altogether, complete disintegration resulting in a granular fragmentation of the material is observed. No increased lustre is observable on the craggy surfaces. The DoF at this temperature reaches 1.27, which means that more material was fragmented and spread into the oven than stayed in the bowl. The DoD at 450°C (DoD = 0.33) shows the same, which means that a substantial loss of mass occurred (*Figures 17, 18, 20* as well as *Table 2*). For this temperature, no RGB graphics are available, because this was a test group (KG 3).
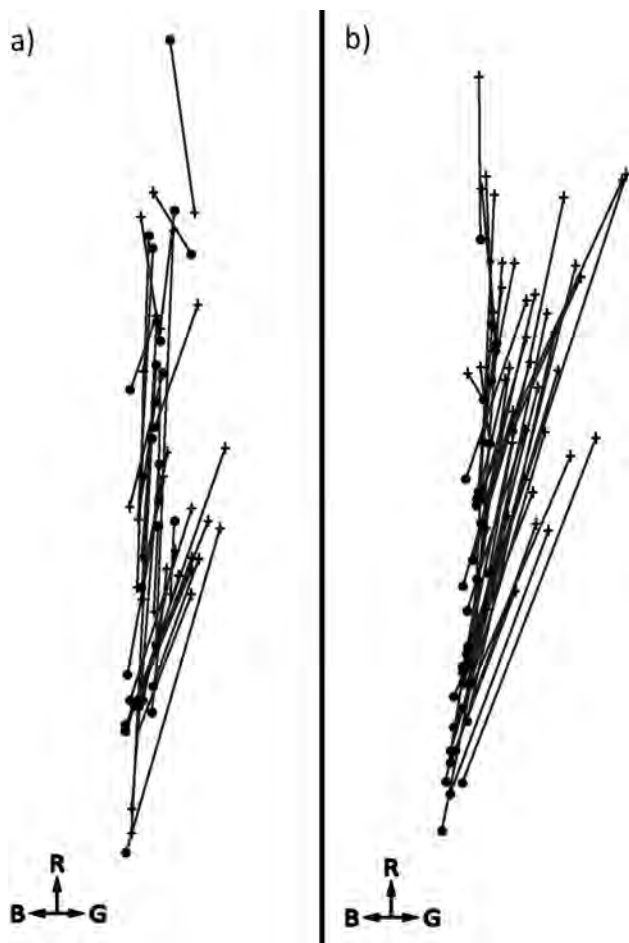
FIGURE 19: Three-dimensional graphic of 350°C, a) sample group 4 and b) sample group 8 (for symbols see *Figure 9*). Graphic by C. T. Hoyer.

DISCUSSION

Our experiments tried to characterise the results of heating at several temperatures on a specific variety of flint (FAS) found in eastern France. We have to take the following points into account for practical use in the field:

When we macroscopically compare the appearance of our samples before and after heating, above 350°C the complete disintegration is apparent. We also can observe visible colour changes (rubification on the edges) and fragmentation starting above 200°C to such an extent that heat influence could also be detected directly in the field. For lower temperatures without any visible fragmentation, the colour changes are so slight (sample groups of 200°C) that they are hard to see with the naked eye, even in direct comparison (*Figure 13*), but evident in the RGB-value spectrum (*Figure 14*).

In trying to detect heat influence on pieces in the archaeological filed work we must bear in mind the combination of colour change and fragmentation. It seems helpful for every excavator to be familiar with the major steps of heat-influenced alteration on Silex. Based on our results, we postulate a suitable trichotomy into temperature spans for the FAS material identifiable in the field:

In the lower temperature span (< 250°C) pieces are nearly in the colour range of the raw pieces without any fragmentation influenced by heat.

The medium temperature span between 250°C and 350°C is characterised by a clear colour change (reduction in brightness and a shift into the spectra of higher red and blue content) with fragmentation patterns visible.

The upper temperature span (> 350°C) contains pieces which are often almost or completely disintegrated and tend to have a more greyish colour.

While the stages two and three are clearly identifiable in the fieldwork, the stage one is only detectable in RGB-values comparison with the colour range of raw pieces.

CONCLUSION

This research represents a further contribution to a more general and global understanding of how different silicates behave under the influence of heat/fire, and to the building of a broad basis for comparative research with archaeological material subjected to heat.

Most broadly, we were able to demonstrate the occurrence of commonly described heat alteration patterns from slight colour-shifts and pot-lid exfoliation up to
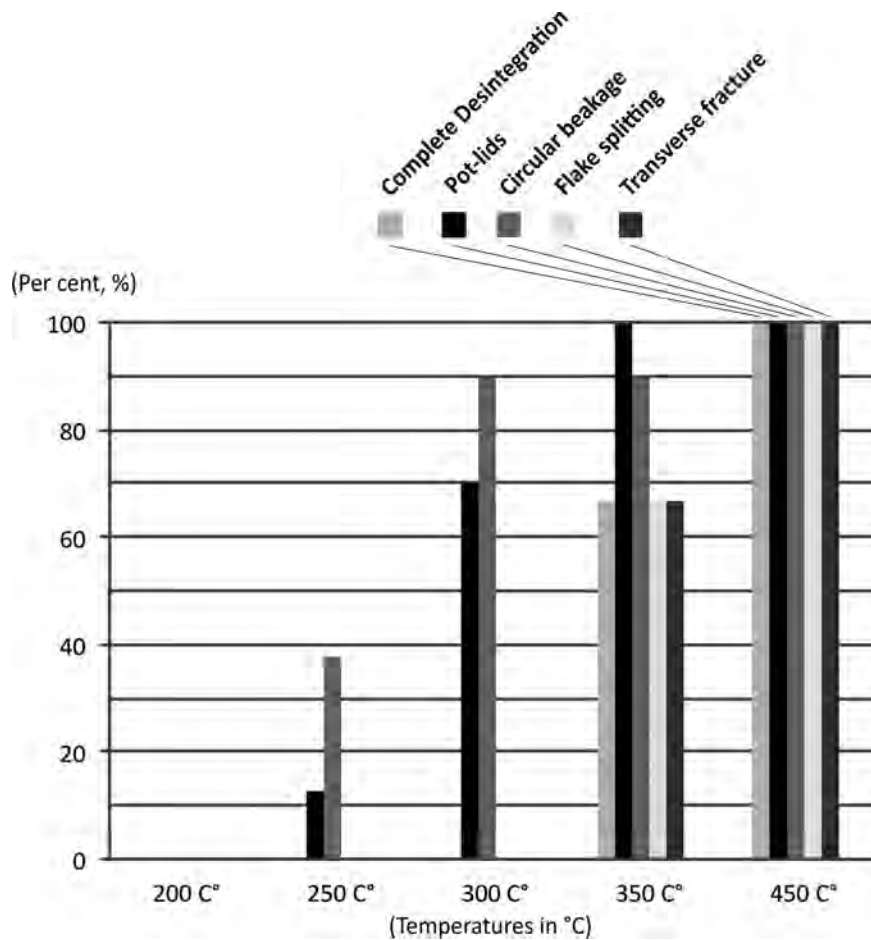
FIGURE 20: Diagram of observed breakage pattern types per temperature. The meaning of the columns is indicated for the sample groups heated till 450°C. Only the samples with 450°C shows all types on all heated pieces and the samples with 200°C show none. Diagram by J. A. Frick.

TABLE 2: Observed comparative patterns for the studied material at different temperatures.

	Rubification	Lustre	Circular breakage	Pot-lids	Crazing	Cracking	Fragmentation and disintegration
200°C	-	-	-	-	-	-	-
250°C	+	-	-	+	-	-	-
300°C	++	+	+	+	-	+	+
350°C	+++	+	++	++	-	+	++
450°C	++	-	+++	+++	-	+	+++

complete disintegration of the material's inner structure, depending on specific temperature levels (see also *Table 2*). In comparing the measurement points nearest to the cortex or the surface of the pieces (M1; *Figure 21a, b*) from all sample groups, there is a clear tendency that these areas become darker in colour shade without much change in the hue. The turn into the red spectrum (paired with increasing or decreasing brightness of the colour shades) is more visible in the inner sectors of the pieces, for example at measure point M7 (*Figure 22a, b*).
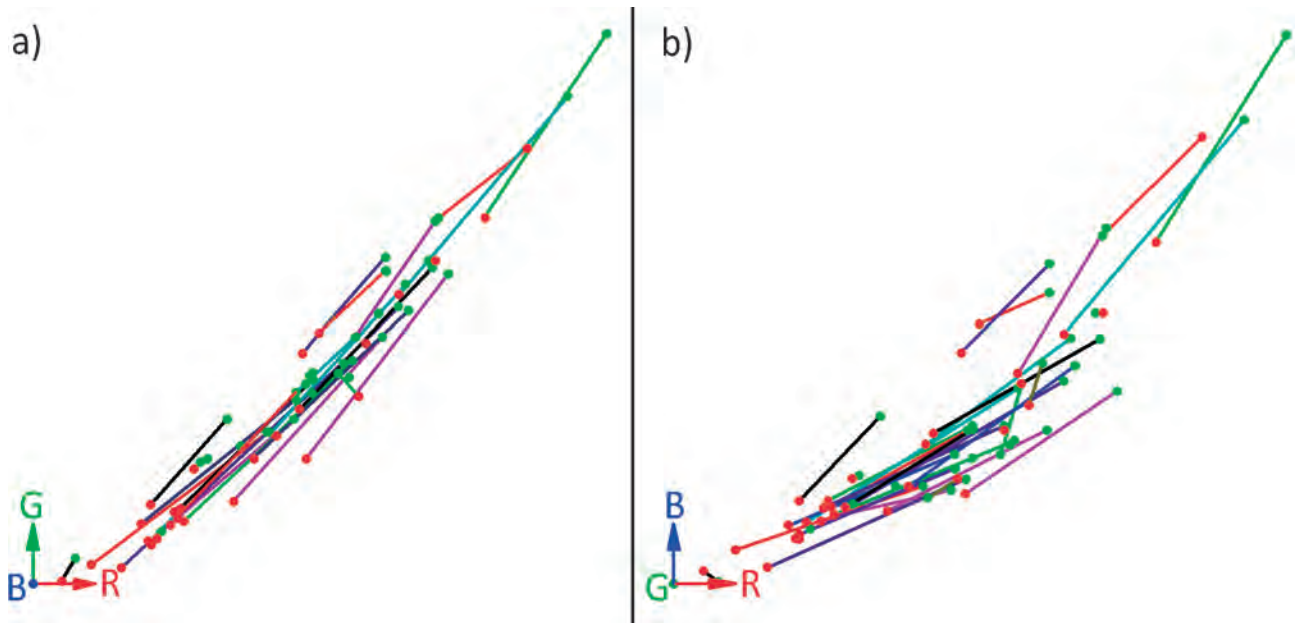
FIGURE 21: Comparison of measurement point 1 (most nearby the cortex) of every sample group. a) Spectrum between green and red b) spectrum between blue and red (sample group 1 at 200°C in blue; sample group 2 at 250°C in magenta; sample group 3 at 300°C in green; sample group 4 at 350°C in red; sample group 5 at 200°C in yellow; sample group 6 at 250°C in cyan; sample group 7 at 300°C in black and sample group 8 at 350°C in blue-violet. Graphic by C. T. Hoyer.
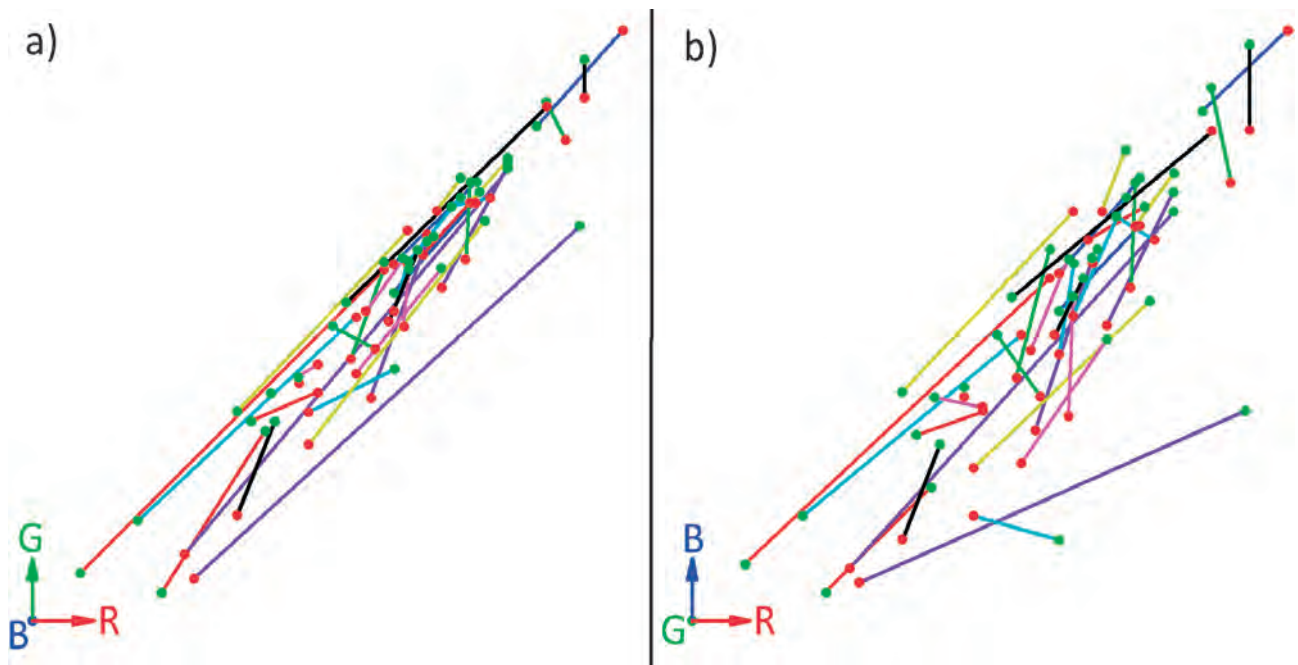

FIGURE 22: Comparison of measurement point 7 (inside the pieces) of every sample group (symbol meaning see *Figure 21*). Graphic by C. T. Hoyer.

With particular regard to the material recovered in the course of our own excavations in southern Burgundy, we are now able not only to confirm the presence of fire at the sites, but also make reasonable estimates of the duration and maximum temperature of the heating of archaeological materials. Considering the complete fragmentation into small pieces of large parts of our samples at 450°C, we are now also conscious about potential problems, especially in successful Thermoluminescence dating projects, concerning the maximum exposure-temperature and the minimum sample-size needed (Richter 2011). Therefore, regrettably, this material does not seem suitable for TL dating techniques.

Our research also demonstrates that in ashes or under a fireplace, lower temperatures could be created (e.g. Sergant *et al.* 2006) that leave no direct evidence of heating on FAS, but that even lower temperatures affect the FAS in ways that are detectable, though perhaps not recognisable in the field. Therefore additional scientific methods (ESR, Magnetometry or IR-spectroscopy) seem to be necessary for a successful detection of hearth structures, or at least for an increased amount of evidence of fire, preserved in Middle and Early Upper Palaeolithic sites.

ACKNOWLEDGEMENTS

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