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# FIRST INSIGHTS INTO THE TECHNIQUE USED FOR HEAT TREATMENT OF CHERT AT THE SOLUTREAN SITE OF LAUGERIE-HAUTE, FRANCE*

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*The earliest evidence of flint and chert heat treatment was found in the ~21.5–17 ka old European Solutrean culture. The appearance of pyrotechnology as part of the production of stone tools has important implications for our understanding of Upper Palaeolithic technological evolution and the specific adaptations during the last glacial maximum in Europe. However, the techniques and procedures used to heat-treat rocks during the Solutrean remain poorly understood. No direct archaeological evidence has so far been found and the most promising approach is to understand these techniques by determining the parameters with which flint and chert were heated at that time. In this study, we investigate the heating temperature of 44 heat-treated laurel-leaf points from Laugerie-Haute, using a non-destructive technique based on infrared spectroscopy. Our results document that most of the artefacts were heated to a narrow interval of temperatures between 250 °C and 300 °C. This indicates a standardized technique that allowed to created similar conditions during successive heating cycles. The implications of these results for our understanding of the technical complexity during the Solutrean must be discussed in the light of different heating techniques used at different places and periods.*

*KEYWORDS:* LAUREL-LEAF POINTS, *FEUILLES DE LAURIER*, HEAT TREATMENT PROCEDURE, SOLUTREAN, INFRARED (IR) SPECTROSCOPY

## INTRODUCTION

At the end of the Lithic Technology Conference held in Les Eyzies-de-Tayac (Dordogne, France) in November 1964, François Bordes stressed in his synoptic paper (Bordes 1967) that (as translated) ‘the [finding] of thermal treatment of flint in American [lithic] industries and during the Palaeolithic has been one of the most original contributions’ (op. cit. 47). At that time, Don Crabtree’s innovative contributions to the subject of lithic heat treatment (Crabtree 1964; Crabtree and Butler 1964) had stimulated debate on the subject in Europe. From 1956, Jacques Tixier had started to experimentally heat-treat European flint and chert to prepare it for pressure retouching (Inizan and Tixier 2001) and, a few years later, heat treatment was archaeologically recognized in several Solutrean deposits in south-western France: at Laugerie-Haute (Bordes 1967, 1969) and at Le Placard (Inizan and Tixier 2001). Thus, the Upper Palaeolithic Solutrean was the first European context to have yielded traces of flint and chert heat treatment. The terms ‘flint’ and ‘chert’ are sometimes used interchangeably throughout the archaeological literature

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and their distinction is only geologically relevant. As both rocks are mineralogically the same, we henceforth only use the term chert. For a long time, the Early Solutrean (21.5–19.5 ka) was also considered the oldest culture in which intentional heat treatment of rocks for stone knapping was practised (Tiffagom 1998). Although older cases of silcrete heat treatment, dating from the South African Middle Stone Age (MSA), are known today (Brown *et al.* 2009), stone knappers from the Solutrean still appear to have been the first to heat-treat finer-grained rocks such as chert. The difference between chert that was heated during the Solutrean and the coarser-grained silcrete that was heat-treated in the MSA is of major importance for our understanding of the implications of heat treatment at these periods. For example, silcrete can be heat-treated in domestic fires (Schmidt *et al.* 2013b) and the so far available data on the MSA demonstrate that this early heat treatment relied on direct heating in open fires (Schmidt *et al.* 2015). Chert, on the other hand, calls for a procedure that involves a lower temperature and slower heating rates (Schmidt *et al.* 2011, 2012), and therefore greater investment in time and resources (Schmidt *et al.* 2016). The reason for this is that chert shatters into unusable pieces when heating temperatures are too high or heating rates too fast (Schmidt 2014). This was already noticed by the first experimenters attempting to heat-treat chert (Crabtree and Butler 1964) and the theory of sand-bath heating was used to interpret the heating technique used in the Solutrean (Inizan and Tixier 2001). Techniques such as a sand bath or ‘Polynesian oven’-like structures rely on indirect heating and involve insulating materials such as sand in their heating environments. The sand insulation creates the slow heating rates and low temperatures that allow unwanted overheating of the rocks to be avoided (Mandeville and Flenniken 1974; Griffiths *et al.* 1987). The downside of heat treatment in insulating heating environments is that it is time-intensive, because the stones must be placed under the fire before it is lit and they can only be taken out when it has burned out and the sand bath has cooled down. Hence, the procedure requires an investment in time of up to a full day or even more (Schmidt *et al.* 2016). The technique used for heat treatment in the Solutrean, its cost for the stone knappers and the degree of standardization of the procedure are therefore of major interest for our understanding of the earliest known chert heat treatment. No direct archaeological evidence that would document a heating technique in the Solutrean has yet been found, and the best possible approach to the question appears to be to determine the heating parameters used to treat chert. In this paper, we investigate the temperatures used for heat treatment in the Solutrean, to obtain a first data set that allows us to interpret the Solutrean heating environment and technique. For this study, we use material from one of the Solutrean sites where heat treatment was first recognized: Laugerie-Haute. The site has yielded a rich assemblage of bifacial points knapped from heat-treated chert and is therefore historically and technically an ideal site for this first study.

## MATERIALS AND METHODS

### *The site*

Laugerie-Haute in Les Eyzies-de-Tayac (Dordogne) is one of the few French Solutrean sites that have yielded complete sequences covering all stages of this culture, from the Proto-Solutrean to the Upper Solutrean (Peyrony 1924; Peyrony and Peyrony 1938; Bordes 1958, 1969). It was among the first sites in the Perigord that were archaeologically exploited and it was therefore the centre of attention of many pioneers of the field. One of these was Otto Hauser, who led excavations in different parts of the site, unearthing a series of hundreds of bifacial points or laurel-leaf points (*feuilles de laurier*) (almost all fragmentary) that are now curated at the Musée national de Préhistoire (MNP) des Eyzies. Our study is based on some of these laurel-leaf points.

### *The archaeological samples and their origin*

We analysed 44 fragments of laurel-leaf points from the MNP's Hauser collections. There are no detailed descriptions by the excavator that would allow to accurately locate these pieces within the deposits of Laugerie-Haute. However, one detail that Hauser mentioned in his original work (Hauser 1911) may provide an indication about their possible origin. According to his description, Hauser found a 'rich Magdalenian' (op. cit. 15) in the eastern part of the deposit (op. cit. Nos. 8–10), to which he gave the local name of *Laugerie-Haute*. In the opposite part of the site, in the west (op. cit. Nos. 11–16), he found a (as translated) 'magnificent Solutrean with laurel-leaf points and beautiful shouldered points' in a location that he called *Laugerie intermédiaire* (Hauser 1911). On the basis of these indications, the 44 analysed bifacials are likely to originate from the western part of the site. An additional indication comes from Philip Smith's Ph.D. thesis on the Solutrean of France (Smith 1966). He could reconstruct that Hauser had excavated all levels above level 11 (numbering of Bordes/Smith) in this western part of the site (op. cit. 90). A photograph showing Hauser's excavations in the western part of the cave is published in Cleyet-Merle (1990). Although level 11 was considered by the excavators as the one where the 'true laurel-leaf points' begin to appear, the overlying levels 10, 9 and 8 also yielded rich Solutrean assemblages and abundant bifacial points. Level 9, for example, yielded 40 bifacials (43.2% of all recovered tools), some of which are large. This precision may be of importance because it allows us to tentatively correlate level 9 with Peyrony's level H" (Peyrony 1924; Peyrony and Peyrony 1938), which has yielded the large laurel-leaf points. The most likely origin of the 44 analysed bifacial fragments is one of these layers.

François Bordes published the first evidence of heat treatment in the Solutrean (Bordes 1969) during his works on Laugerie-Haute in collaboration with Smith. The artefact upon which he built the argument in his paper is a fragment of a laurel-leaf point that broke during its manufacture. The piece was not unearthed during Bordes' excavations themselves but belongs to the MNP's Hauser collection. It is made from a translucent tertiary chert from the Aquitaine basin (locally called *silex calcédonieux*). Unfortunately, we were unable to find this piece in the MNP's collections and include it in our analyses. Bordes might have kept the piece in his house, separate from the main collection. However, we were able to find 44 laurel-leaf fragments from the Hauser collection that were all knapped from the same translucent tertiary chert. We chose these pieces for our analysis, among numerous heat-treated laurel-leaf points made from various rock types, because they are all knapped from a single raw material and are thus comparable for spectroscopic temperature determination (Schmidt *et al*. 2013a), and because most of them show unambiguous macroscopic indicators of heat treatment (glossy post-heating removal scars, and the contrast between glossy and matt removal scars). Because these artefacts do not have individual numbers, we randomly attributed consecutive numbers to the samples for our analyses (Table 1).

### *Geological samples*

A set of reference samples of the same chert as the laurel-leaf points—that is, samples that never had been heated—was needed for our study. To establish a 'calibration series' for the determination of the heating temperatures of the 44 artefacts, we also experimentally heat-treated one of

Table 1 *Macroscopic observations of the archaeological samples and the values of the absorbance ratio: –, absence; int., intermediate. The geological sample used for the experimental heating series is marked with an asterisk. Numbers under 'Texture': 1, very fine-grained appearance; 2, fine-grained appearance; 3, slightly coarse-looking*

*Geological reference samples*

| *Texture* | *4545/4469 cm$^{-1}$ ratio* |
|---|---|
| 2 | 0.7218 ± 0.01 |
| 2 | 0.7161 ± 0.01 |
| 2 | 0.7018 ± 0.01 |
| 2 | 0.7149 ± 0.01 |
| 2 | 0.7156 ± 0.01 |
| 2* | 0.7243 ± 0.01 |
| 2 | 0.7221 ± 0.01 |
| 2 | 0.7126 ± 0.01 |
| 2 | 0.7172 ± 0.01 |
| 2 | 0.7071 ± 0.01 |
| 2 | 0.7225 ± 0.01 |

*Experimental heating series*

| *Temperature (°C)* | *4545/4469 cm$^{-1}$ ratio* |
|---|---|
| Unheated | 0.7303 ± 0.01 |
| 200 | 0.7431 ± 0.01 |
| 250 | 0.7584 ± 0.01 |
| 300 | 0.7986 ± 0.01 |
| 350 | 0.9236 ± 0.01 |
| 400 | 1.0223 ± 0.01 |

*Archaeological samples*

| *Short sample number* | *Thickness (mm)* | *Texture* | *Gloss contrast* | *Gloss intensity* | *Heat-induced fractures* | *4545/4469 cm$^{-1}$ ratio* |
|---|---|---|---|---|---|---|
| 26 | 12.33 | 1 | Yes | Int. | – | 0.7360 ± 0.01 |
| 3 | 4.96 | 2 | – | Int. | – | 0.7445 ± 0.01 |
| 35 | 8.2 | 3 | Yes | Int. | – | 0.7465 ± 0.01 |
| 43 | 6.14 | 2 | – | Int.– strong | – | 0.7473 ± 0.01 |
| 40 | 4.25 | 1 | – | Weak | – | 0.7475 ± 0.01 |
| 8 | 8.85 | 2 | – | Int. | – | 0.7485 ± 0.01 |
| 13 | 7.8 | 2 | – | Weak | – | 0.7513 ± 0.01 |
| 1 | 5.63 | 2 | Yes | Int. | – | 0.7519 ± 0.01 |
| 12 | 8.48 | 2 | – | Int. | – | 0.7519 ± 0.01 |
| 11 | 6.35 | 2 | Yes | Weak | – | 0.7521 ± 0.01 |
| 44 | 6.64 | 1 | – | Strong | – | 0.7526 ± 0.01 |
| 36 | 5.53 | 2 | – | Low | – | 0.7551 ± 0.01 |
| 24 | 5.45 | 2 | – | Int. | – | 0.7590 ± 0.01 |
| 21 | 7.58 | 2 | – | Int. | – | 0.7594 ± 0.01 |
| 30 | 6.12 | 2 | – | Int. | – | 0.7598 ± 0.01 |
| 23 | 7.47 | 3 | Yes | Strong | – | 0.7600 ± 0.01 |
| 42 | 8.37 | 2 | Yes | Strong | – | 0.7602 ± 0.01 |
| 28 | 5.72 | 2 | – | Int. | – | 0.7603 ± 0.01 |

(*Continues*)

Table 1 *(Continued)*

*Archaeological samples*

| *Short sample number* | *Thickness (mm)* | *Texture* | *Gloss contrast* | *Gloss intensity* | *Heat-induced fractures* | *4545/4469 cm$^{-1}$ ratio* |
|---|---|---|---|---|---|---|
| 29 | 6.81 | 2 | – | Int. | – | 0.7606 ± 0.01 |
| 15 | 6.57 | 2 | Yes | Strong | – | 0.7606 ± 0.01 |
| 39 | 7.72 | 3 | Yes | Int. | – | 0.7613 ± 0.01 |
| 37 | 6.05 | 2 | – | Int. | – | 0.7613 ± 0.01 |
| 33 | 8.86 | 3 | Yes | Strong | – | 0.7618 ± 0.01 |
| 22 | 6.91 | 3 | Yes | Int. | – | 0.7622 ± 0.01 |
| 2 | 15.3 | 2 | – | Int. | – | 0.7622 ± 0.01 |
| 5 | 6.33 | 2 | Yes | Strong | – | 0.7631 ± 0.01 |
| 20 | 9.01 | 2 | – | Int. | – | 0.7659 ± 0.01 |
| 10 | 8.6 | 2 | – | Strong | Yes | 0.7660 ± 0.01 |
| 7 | 6.88 | 2 | – | Int. | Yes | 0.7662 ± 0.01 |
| 19 | 6.45 | 3 | – | Int. | – | 0.7669 ± 0.01 |
| 9 | 6.73 | 3 | – | Weak | – | 0.7670 ± 0.01 |
| 4 | 6.81 | 3 | Yes | Weak | – | 0.7676 ± 0.01 |
| 16 | 8.05 | 2 | – | Int. | Yes | 0.7704 ± 0.01 |
| 18 | 7.89 | 3 | – | Weak | – | 0.7704 ± 0.01 |
| 6 | 10.19 | 3 | – | Weak | – | 0.7717 ± 0.01 |
| 34 | 7.85 | 1 | – | Strong | – | 0.7718 ± 0.01 |
| 25 | 7.39 | 2 | – | Strong | Yes | 0.7792 ± 0.01 |
| 32 | 5.64 | 3 | – | Weak | – | 0.7800 ± 0.01 |
| 14 | 9.36 | 2 | Yes | Int. | – | 0.7807 ± 0.01 |
| 17 | 8.42 | 3 | – | Weak | – | 0.7821 ± 0.01 |
| 41 | 9.52 | 2 | Yes | Strong | Yes | 0.8040 ± 0.01 |
| 31 | 5.16 | 1 | – | Strong | – | 0.8064 ± 0.01 |
| 27 | 8.16 | 2 | – | Int. | – | 0.8152 ± 0.01 |
| 38 | 5.5 | 2 | Yes | Strong | Yes | 0.8199 ± 0.01 |

these reference samples at various temperatures. On the basis of petrographic criteria established during earlier works (unpublished), the most likely origin of the artefacts' translucent chert is a sector called La Forêt de la Bessède. This sector, located 20 km south-west of Les Eyzies, is home to lacustrian tertiary formations that contain abundant nodules of translucent chert. The samples were collected by one of us (AM) within this sector, close to the town of Monpazier, from a massive layer of translucent chert dating to the Upper Oligocene. Individual nodules with variable sizes, ranging from 10 cm to 1 m, and with variable textures, ranging from apparently fine-grained to relatively coarse-looking pieces, can be found within this layer. The average size of these nodules lies somewhere close to 20–30 cm in length and 10–20 cm in thickness. No correlation between nodule size and texture could be noted. A publication dedicated to this and other outcrops in the region is being prepared (Morala in press). Four pieces of the tertiary chert were collected from this primary outcrop. These were further sectioned to produce 11 flakes that could be compared with the 44 archaeological samples. These are the geological reference samples.

### *Methods and experimental set-up*

In a first step, all archaeological samples were inspected for heat-treatment proxies. Three proxies were observed, as follows. (1) *Gloss contrast* is the simultaneous presence of matt pre- and shinier post-heating removal scars on a single artefact. The presence of removal scars from before and after heat treatment is the most secure macroscopic criterion for identifying heat treatment of stone artefacts. (2) *Gloss intensity* is a qualitative estimation of the overall magnitude of the surface lustre of all removal scars on an artefact. The identification of weak, intermediate or strong surface gloss on an artefact may allow us to estimate whether or not it was knapped after heat treatment. (3) *Heat-induced non-conchoidal (HINC) fracture*s (Schmidt *et al.* 2015) are surfaces produced by failure during heating (overheating; Schmidt 2014). In the case of chert, such fracturing during heat treatment often leads to the artefact being discarded, because it becomes unsuitable for further knapping. However, if the HINC-fracture surface is not invasive and does not render the piece entirely unsuitable for knapping, reduction may be continued after the overheating event. Surfaces due to heat-induced fracturing can be recognized on chert through their strong surface roughness, the presence of angular surface features and the absence of signs of conchoidal fracture.

In a second step, we conducted archaeometric analyses of the heating temperatures using infrared light transmission through the artefacts. The theoretical background and detailed experimental set-up of the analyses are explained in Schmidt *et al.* (2013a), and only the information that is absolutely necessary for understanding the method is repeated here. The analyses rely on the measurement of the transmission of near-infrared radiation through the thin part of lithic artefacts (typically, near the cutting edge). The non-destructive measurements result in an infrared absorption spectrum between 4000 and 4800 $\mathrm{cm}^{-1}$ that contains an absorption band caused by SiOH. The shape of this absorption band (measured as the ratio between the linear absorbances at 4545 $\mathrm{cm}^{-1}$ and 4469 $\mathrm{cm}^{-1}$, the short notation for the ratio being 4545/4469 $\mathrm{cm}^{-1}$) is partly influenced by the quantity of water held in the open pore space of the samples. The mechanism behind this is the chemical interaction of this pore water with surface SiOH (hydrogen bonding). More pore water causes a shift to lower frequencies, while less pore water causes a relatively larger component at higher wavenumbers (Schmidt *et al.* 2011). The shape of the band is therefore an indirect measure of the quantity of water in open pores and, if all available pore space is completely filled with water, also of the volume of open pore space of the sample itself. When chert is heat-treated, it gradually loses such open pore space (Roqué-Rosell *et al.* 2011; Schmidt *et al.* 2012; Milot *et al.* 2017). Schmidt *et al.*'s (2013a) method aims at detecting past heating through the measurement of a sample's pore space with respect to the pore space of another sample of the same rock type that was surely never heated. The two samples compared in this way, the one tested for past heating and the reference, must undergo an identical protocol that allows for total filling of their open pore space with deionized or distilled $H_2O$. A higher value of the 4545/4469 $\mathrm{cm}^{-1}$ ratio in the tested sample, as compared with the same ratio for the reference sample, indicates that the former was subjected to heating in the past. The heating temperature can be estimated by combining these measurements with measurements of experimentally heat-treated reference samples of the same rock. A reference sample is progressively heated to different temperatures, rehydrated using the same protocol and then analysed for its 4545/4469 $\mathrm{cm}^{-1}$ ratio after each temperature step. The comparison between the ratio values of the archaeological samples and the ratio of the reference allows us to estimate the temperature range to which the archaeological sample was heated.

To apply this method to the Solutrean artefacts from Laugerie-Haute, all archaeological samples were heated to 110 °C for 24 h to dehydrate their open pore space and then rehydrated in deionized $H_2O$ for 24 h at 70 °C and 1 bar. Geological reference samples were hydrated along with the 44 archaeological samples, applying the identical protocol. One of the 11 reference flakes (the sample that had produced the highest ratio value) underwent experimental heat treatment to estimate the heating temperature of the archaeological samples as explained above. The sample was heated in an electrical furnace to 200 °C, 250 °C, 300 °C, 350 °C and 400 °C, with a heating rate of 20° $h^{-1}$. After each temperature step, the sample was cooled to room temperature overnight to avoid fracturing induced by excessively fast cooling and then rehydrated in deionized $H_2O$ for 24 h at 70 °C and 1 bar to saturate its open pore space with water. No fracturing of the sample was observed using this protocol.

### *Analytical equipment and experimental error*

The infrared transmission was recorded at normal incidence using unpolarized light from an Agilent Cary 660-IR FT–IR spectrometer. Spectra were acquired between 4000 and 4800 $cm^{-1}$, with a resolution of 8 $cm^{-1}$. The infrared light was directly transmitted through thin parts of the samples fixed in the spectrometer's sample chamber. The diameter of the IR beam was cut to 7 mm by a circular diaphragm. No other sample preparation was necessary and the analyses of all archaeological samples remained non-destructive. The baseline used for the measurement of the 4545/4469 $cm^{-1}$ ratio was a straight line between the lowest two points on either side of the SiOH absorption band (see Fig. 2 below). Experimental errors were estimated to a fixed ratio value of ±0.01 by repeating the analysis (mounting, analysing and unmounting) of a single sample 20 times at different spots (one flake of our geological reference chert of a size of ~6.5 × ~4 cm). This measuring error is most probably due to sample heterogeneities.

## RESULTS

### *Description of the artefacts*

The dimensions of the 44 laurel-leaf fragments are variable. Most of them are preforms, with only a few traces of an elaborate finish. Some, however, were retouched and appear to be finished objects that had broken during use or handling. The textural appearance of the removal scars on the objects indicates a preselection of rather fine raw materials for knapping. Three raw material classes can be distinguished (Table 1), slightly coarse-looking ($n$ = 12), fine ($n$ = 27) and very fine ($n$ = 5), documenting a slight preference for fine and very fine materials, although slightly coarse rocks were not discarded. Significantly courser-grained rocks can be found at the same outcrop, but these were not used for knapping laurel-leaf points.

Although the 44 pieces are fragmented, we tried to estimate their approximate initial length: less than 10 cm in length ($n$ = 18), between 10 and 15 cm ($n$ = 15 + possibly 2) and more than 15 cm ($n$ = 6 + possibly 3). Nine pieces are proximal or distal fragments, 19 pieces are mesial parts with both opposite flanks preserved, 11 pieces are mesial parts with only one lateral edge preserved and five pieces are either mesio-proximal parts or mesio-distal parts (Fig. 1). We also tried to understand the cause of breakage of the artefacts. Most of them appear to have broken during retouch ($n$ = 25 + possibly 4). Others may have broken during heat treatment ($n$ = 3 + possibly 8) or use ($n$ = 4), as indicated for some by glossy fracture surfaces that result from the breakage of the artefact ($n$ = 6).

Heat treatment proxies can be observed on most of the 44 pieces in the form of intermediate to strong overall gloss of the fracture surfaces (Table 1). In some cases ($n$ = 13), intensely glossy post-heating scars cover the entire artefact, documenting intense retouch after heat treatment. Fifteen pieces clearly show the contrast between glossy post-heating removal scars and relatively

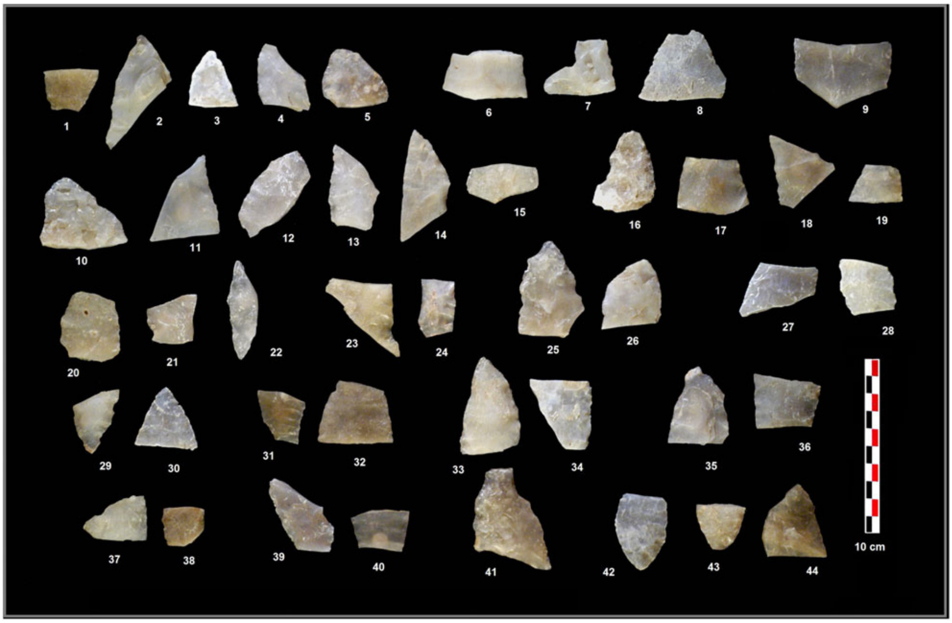

Figure 1 *Photographs of the 44 analysed laurel-leaf points. The numbers on the plate are the short sample numbers used throughout in Table 1 and the other figures (photograph by Ph. Jugie MNP). [Colour figure can be viewed at wileyonlinelibrary.com]*

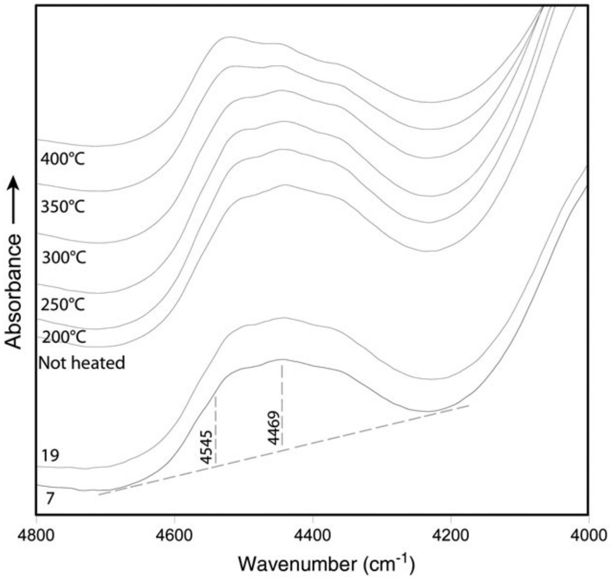

Figure 2 *Near-infrared transmission spectra between 4000 and 4800 cm$^{-1}$ and the baseline for the measurement of the 4545/4469 cm$^{-1}$ ratio (broken lines). The measured spectral range contains a SiOH combination band. The six spectra on top correspond to the experimental heating series of one of the geological reference samples. Note the gradual shift of the band to higher wavenumbers with rising heating temperature. The two spectra in the lower part correspond to archaeological samples 7 and 19. Spectra are vertically offset for readability.*

more matt preheating removal scars, documenting that knapping continued after the heat treatment. Six pieces show signs of heat-induced fracturing that most probably took place during heat treatment. In one of these cases, the identification of post-heating scars (unambiguously revealed through gloss contrast) documents that knapping continued after the heat treatment, in spite of the breakage.

### *Ratio values and heating temperatures*

Figure 2 shows the spectra of the geological reference sample that was experimentally heat-treated to successive temperatures and two spectra of archaeological samples for comparison. The SiOH band of the experimentally heated sample shifts progressively to higher wavenumbers with rising temperature. The 4545/4469 cm$^{-1}$ ratio values deduced from the band allow estimation of the heating temperatures of the archaeological samples. These ratio values are listed in Table 1. Figure 3 is a plot of the ratio values of the 44 archaeological samples compared to the ratio values of the unheated reference flakes. The reference samples have values between 0.702 and 0.724, setting the range of ratio values of the unheated tertiary chert to between ~0.69 and ~0.73 if measurement error is taken into account (the grey bar in Fig. 3). The dispersion of the ratio values of these unheated reference samples confirms that the estimated measurement error of ±0.01 is correct: the mean of all unheated ratio values is 0.7160 and their maximum is 0.7243—that is, it lies 0.0083 above the mean; all values lower than the mean, except one, also fall within the –0.01 lower error bar. Archaeological samples all have higher ratio values than the reference samples (between 0.74 and 0.81), confirming that they were heat-treated. This identification is further strengthened by the presence of gloss contrast (an unambiguous indication of knapping after heat treatment) on several of the artefacts that produced the lowest 4545/4469 cm$^{-1}$ ratio values (Table 1). Thus, the range of measured archaeological ratio values is not caused by heat-treated and not-heated artefacts, but the different values may correspond to either macroscopic variability of the raw materials that were not assessed by us—although no correlation between raw material texture types (Table 1) and

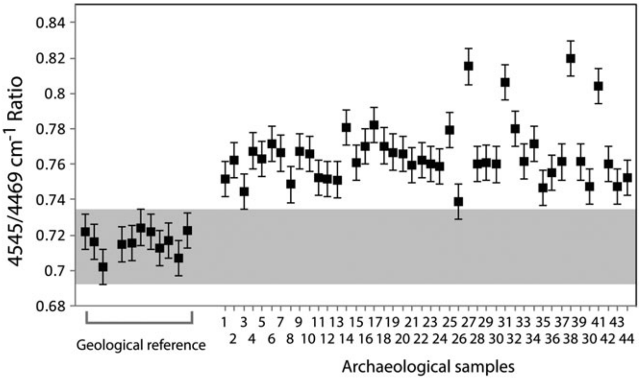

Figure 3 *A plot of the values of the 4545/4469 cm$^{-1}$ ratio obtained from Solutrean samples. Archaeological samples are named using the sample numbers shown in Table 1. The ratio values of the unheated geological reference samples are displayed on the left of the graph. The range of values produced by these unheated samples is marked by a grey bar. The archaeological samples are clearly distinguished by their 4545/4469 cm$^{-1}$ ratio values and can be identified as heat-treated.*

ratio values can be noted—or different temperatures used for heating. Pieces with lower values show no traces of overheating (Table 1). All artefacts with signs of thermal fracturing have values higher than 0.77, indicating that only the higher end of the temperature range caused overheating during heat treatment. The absence of a clear correlation between the ratio value and the overall intensity of gloss observed on the artefacts implies that gloss on the samples is not a good criterion for the estimation of the heating temperature for this type of chert.

The ratio values of the experimentally heat-treated reference sample increase gradually after each temperature step. It is interesting to note that the geological reference sample used for the experimental heating series produced a ratio value of 0.7243 ± 0.01 during its first measurement (comparison with the archaeological samples), but a value of 0.7303 ± 0.01 when reanalysed for the heating experiment (after a second dehydration at 110 °C and rehydration in water, but before heat treatment). This 0.006 higher ratio value falls within the estimated measurement error and might be explained by the supplementary step of dehydration/rehydration or spectral acquisition on another spot of the sample (sample heterogeneity). As it stands, this deviation confirms the validity of the estimated ±0.01 measurement error and the temperature estimation resulting from this experimental heating series can be expected to be reasonably accurate within this error. Figure 4 is a plot comparing the 44 archaeological ratio values with the values obtained by this experimental sequence. The comparison shows that most of the samples were heat-treated with temperatures between 250 °C and 300 °C, a minor part of the samples between 200 °C and 250 °C and only four samples were heat-treated slightly but insignificantly above 300 °C.

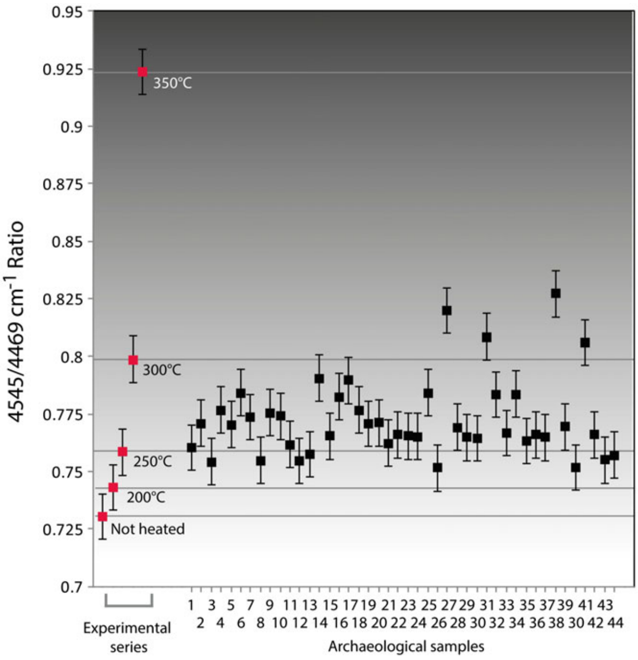

Figure 4 *A comparison between the 4545/4469 cm$^{-1}$ ratio values of the archaeological and experimental samples for the heating temperature estimation of Solutrean artefacts. The ratio values of the progressively heated geological reference samples are displayed on the left of the graph. Temperatures, as calibrated by this experimental series, are shown as horizontal lines. The experimental temperature calibration allows us to estimate the heating temperature of most of the archaeological samples between 225 °C and 300 °C. [Colour figure can be viewed at wileyonlinelibrary.com]*

## DISCUSSION

In their initial study, Schmidt *et al.* (2013a) recommended using this method to include unheated reference samples coming from the same site, to evaluate the difference in the 4545/4469 $\mathrm{cm}^{-1}$ ratio value between unheated and heated samples. However, in the current context—that is, artefacts not directly resulting from recent excavations but from old museum collections—this recommendation cannot be followed so easily. We therefore chose to directly analyse the unheated raw material used to manufacture these artefacts. Indeed, taphonomic factors in Laugerie-Haute may have influenced the value of our samples' 4545/4469 $\mathrm{cm}^{-1}$ ratio (see, for example, the mechanism in Fernandes *et al.* 2012), although the magnitude of such a potential influence remains totally unknown. The absolute temperature values of the archaeological pieces may therefore be slightly overestimated but their relative dispersion is not affected by this.

The estimated heating temperatures mostly fall between 250 °C and 300 °C. Although eight samples appear to have been heated to temperatures below 250 °C, the heating temperature range of the measured Solutrean artefacts appears to be well calibrated. The temperatures of the four samples heated to higher temperatures are still close to 300 °C. This upper limit close to 300 °C may have been imposed by the thermal stability of this type of chert, as indicated by the fact that some pieces heated to the higher range of temperatures fractured during the treatment.

Different heating environments and procedures produce a wide range of temperatures in heat-treated samples. Direct heat treatment in open fires, for example, produces temperatures between 300 °C and 550 °C (Schmidt *et al.* 2015). A sand bath or a similar underground heating structure allows stone to be heat-treated with a range of temperatures from 200 °C to 400 °C (Mandeville and Flenniken 1974; Griffiths *et al.* 1987; Eriksen 1997; Brown *et al.* 2009), depending on the firewood used and the nature/thickness of the insulating sediment. This rules out the possibility of heat treatment in open-air fires during the Solutrean at Laugerie-Haute. This is further strengthened by the homogeneity of the heating temperature of most of the Solutrean artefacts. The heating temperatures do not deviate from the mean by more than ±25 °C. Thus, the technique used allowed for good standardization of the heating process.

Concerning the heating environment or procedure possibly used during the Solutrean, our results demonstrate two things. (1) Heat treatment in the Solutrean was not simply by heating in domestic fires. Heat treatment in open fires, without a heating environment that made it possible to prevent the stones from reaching excessive temperatures, would undoubtedly have resulted in higher and less calibrated heating temperatures. (2) Heat treatment in Laugerie-Haute relied on a standardized procedure that allowed similar conditions to be created during different heating cycles. The Solutrean hunter–gatherers undoubtedly had a good knowledge of fire and its effects on objects and the environment.

## CONCLUSION

Our analyses of the 44 laurel-leaf points from Laugerie-Haute confirmed that all the pieces that we had selected for macroscopic heat treatment proxies were indeed heat-treated by the Solutrean hunter–gatherers. The technique used to heat-treat the chert was highly standardized and allowed reproducible conditions. The implications of these results for our understanding of the Solutrean culture are of great importance, because they highlight the ability of these men and women to repeatedly initiate and control technical processes that most probably were relatively complex (for the concept of technical complexity, see, e.g., Bettinger and Eerkens 1997).

All of the laurel-leaf fragments analysed by us were made from a single raw material, one for which the heating parameters can be easily identified macroscopically. Future studies should focus on broadening this field of observation to include other raw materials. For some of these chert types, the identification of proxies for heat treatment is difficult (e.g., black–grey flint from the Senonian) and the spectroscopic determination of heat treatment might allow light to be shed on some of the questions associated with these materials. Further studies on the percentage of successful heating cycles applied to all materials from Laugerie-Haute should provide a good approximation of the level of know-how and skill of the Solutrean knappers and cast further light on their degree of technical standardization.

Another perspective in the context of heat treatment during the Solutrean is to study its role for the *chaînes opératoires* associated with the production of bifacial points. Was it strictly associated with the last stages of shaping of such points, preparing chert for final retouch or even pressure flaking? Or was it used for all sorts of tool types during the Solutrean? The prevalence of heat treatment is another important question that needs thorough investigation. Was it preferentially applied to particular raw materials or was it a sort of blanket strategy applied to all materials at a given stage of manufacture? Although the European Solutrean culture was the first to have yielded clear evidence of heat treatment and although it documents the earliest cases of heat treatment of chert, the study of these processes is still in its infancy.

As it stands, heat treatment in the Solutrean of south-western France appears to have been a standardized and consistent technique used to produce laurel-leaf points.

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