Journal of Archaeological Science (1997) **24,** 649–657

Detecting Heat Treatment on Silcrete: Experiments with Methods

Martin Rowney and J. Peter White

School of Archaeology, University of Sydney, NSW 2006, Australia

(*Received 27 October 1995, revised manuscript accepted 4 June 1996*)

We describe experiments aimed at detecting heat treatment of silcrete, a siliceous rock of inhomogeneous lithology widely used in prehistoric Australia. Our samples ($N=66$) come from eastern New South Wales. Two methods widely used on other materials are found to be inadequate: *scanning electron microscope* (SEM) analyses allow conflicting interpretations of the same piece of material, while *colour* changes are too variable to be a reliable indicator. Two other methods are more reliable: increase in *lustre* is rare, but clearly indicates heat treatment when its thermal origin is certain; *palaeomagnetism* assessments of modern ($N=18$) and archaeological ($N=23$) samples show that while the method does not give false positives, it indicates heating and not necessarily heat treatment. A combination of palaeomagnetism and lustre assessments is recommended.

Keywords: HEAT TREATMENT, PALAEOMAGNETISM, SILCRETE, LUSTRE.

Introduction

How to detect heat treatment on siliceous lithologies effectively has been discussed since Crabtree & Butler's first attempt in 1964 using SEM. Since then many scientific techniques and observation methods have been tried with varying degrees of success. Macroscopic observations, including visible changes in the colour and surface lustre of thermally altered artefacts, are discussed by many authors. Scientific techniques tested have included SEM (Domanski & Webb, 1992, with refs), thermoluminescence (Pavlish & Sheppard, 1983), X-ray diffraction (Olausson & Larsson, 1982), petrographic thin sectioning (Purdy & Brooks, 1971), and point tensile and compression strength testing (Domanski & Webb, 1992). It is clear from these studies that there is no one single method which allows the detection of heat treatment and that various techniques can be employed depending upon the results required and the ethical position of the researchers in relation to destruction or visible alteration of artefacts.

Most researchers dealing with heat treatment have attempted to detect it on fine-grained, homogeneous siliceous lithologies like chert and flint and very few have worked with less homogeneous lithologies like silcrete. Silcrete is a hard indurated rock resulting from surficial or penesurficial silicification at low temperatures. It is essentially composed of quartz grains cemented by microcrystalline silica (SiO_2). In texture it grades from very fine-grained, like chert, to coarse-grained, like quartzite. Four classes of fabric have been identified by Summerfield (1983: table 3.2) to describe the range of variation found in silcretes. Coarse silcretes are classified as having a GS (grain-supported) fabric represented by a high percentage of skeletal grains which produce a self-suporting framework. Summerfield's plate 3.1(a) shows the skeletal grains as averaging 500 μm in length with some larger grains up to 1 mm in length. Generally, there is proportionately less microcrystalline matrix bonding the skeletal grains together. At the other end of the range, very fine silcretes are classified as having a M (matrix) fabric which is represented by few skeletal grains and a higher proportion of microcrystalline matrix. Summerfield's plate 3.1(d) shows skeletal grains which average 50–100 μm in length and quartz grains of less than 30 μm in length in the matrix. Between these two extremes is the F (floating) fabric which is characterized by medium-sized skeletal quartz grains (Summerfield's plates 3.1(b) and (c) show these to be between 100 and 500 μm in length) cemented by a microcrystalline matrix containing a high proportion of quartz grains less than 30 μm in length. The fourth class of silcrete is the C (conglomeratic) fabric which contains a higher size and proportion of detritus (including pebbles) and a matrix which can be any of the GS, F, or M types. Fabric types ranging from GS to F can be detected within a single sample or between different samples in a single outcrop area and between outcrops in different localities (for further discussion, see Langford-Smith, 1978; Summerfield, 1983: 72–3). Our observations confirm that a wide range of variation can be found between different parts of the same rock sample. Chemical composition, apart from >85% silica, is highly variable, and generally includes aluminium, iron and titanium in significant amounts (Summerfield, 1983). The bonding matrix is often

0305-4403/97/070649+09 $25.00/0/as960147

composed of cryptocrystalline silica and microquartz. Silcrete occurs widely in arid and semi-arid regions and along the humid east coast of Australia (Langford-Smith, 1978; Summerfield, 1983; Twidale, 1983). Prehistoric artefacts in these areas were often made of silcrete. Because of its inhomogeneity we believe that heat treatment detection techniques, which are useful and even reliable when applied to flint and chert, are not necessarily so for silcretes.

It is appropriate here to distinguish between the detection of heating and the detection of heat treatment. Rocks and artefacts may be *heated* intentionally or accidentally at any time, while heat *treatment* refers specifically to deliberate heating of a piece of stone to improve its flakability. Although never explicitly stated (and perhaps not perceived) by researchers studying heat treatment, there are two distinct categories of heat detection methods which give us results of differing reliability and interest. We label these categories "surficial" and "subsurficial". Surficial detection methods are those which assess the surface features of the samples as a means of determining their thermal history, while subsurficial detection methods assess thermal alterations from the internal structure of the sample. We draw this distinction specifically because we will show that surficial detection methods when applied to silcretes may detect heat *treatment* but the results are of dubious reliability, while subsurficial methods reliably detect *heating* but cannot show that this is heat treatment.

The aim of this paper is to demonstrate a combination of heat treatment detection techniques which can be used fairly reliably on inhomogeneous lithologies like silcrete and which produce archaeologically interesting results without damaging the artefacts. This is especially important in Australia where many artefacts are made in this raw material, but will be applicable to similarly inhomogeneous materials elsewhere.

Surficial Methods

Scanning electron microscope

We started our analyses with SEM determinations, since these have recently been re-promoted by Domanski & Webb (1992). SEM determinations are expected to identify changes in two phenomena: one is through the change which occurs to the fracture plane and the other is in the format of the crystal structure. Changes to the fracture plane are discussed by many authors (e.g. Olausson & Larsson, 1982; Flenniken & White, 1983; Domanski & Webb, 1992). Microstructural changes noticed by other authors include a reduction in crystal size (Crabtree & Butler, 1964; Domanski & Webb, 1992), a recrystallization of the matrix causing it to become denser (Mandeville, 1973), and a change from goethite to haematite (Schindler *et al*., 1982). The precise nature of the changes is still unknown and certainly not agreed upon.

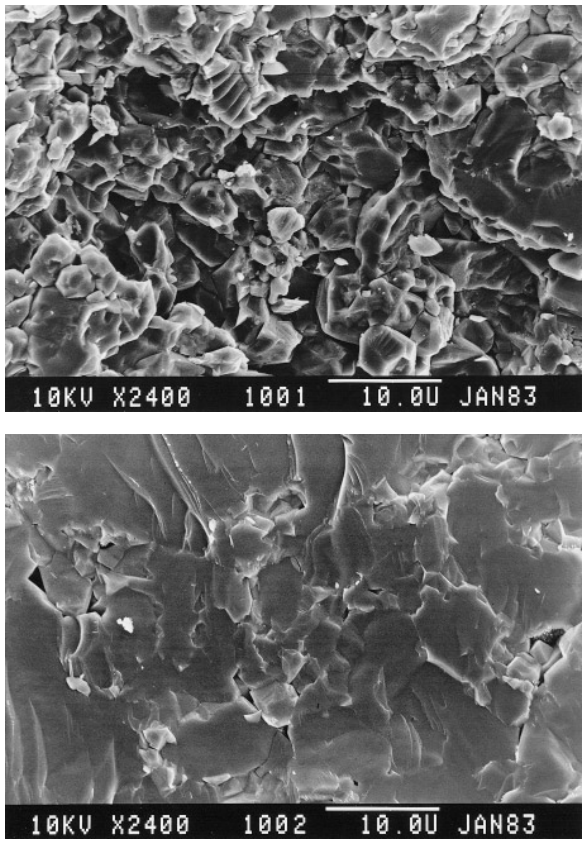

Figure 1. SEM micrographs of flakes of fine-grain (M-type) silcrete from Bass Point, New South Wales. (a): unheated; (b): the same piece after heating. Difference in the fracture plane is clearly visible (Flenniken & White, 1983: figures 1 & 2 respectively).

Our analyses focused specifically on changes to the fracture plane in light of Domanski & Webb's (1992) discussion of the change from transgranular to intergranular fracturing paths after a sample had been heated. They argue that prior to heating the fracture force will travel around individual skeletal quartz grains and through the surrounding silica matrix, while after heating, the fracture force travels through both the skeletal quartz grains and the matrix.

As a control sample, to find out how different unheated and heated samples could look when micrographed, we began by striking off another flake from both the unheated and heated halves of one of the experimental samples used by Flenniken & White (1983). The original piece of Bass Point silcrete is considerably finer grained than any other samples used in this study and is shown in Figure 1(a) (unheated) & (b) (heated) (Flenniken & White, 1983: figures 1 & 2). This sample is of an M-type fabric with a high proportion of matrix quartz grains which are <10 μm in length. As a rough guide to the variation within this sample we point out that the complete sample itself is only 60 mm long and the flake we removed from the heated section is approximately 25 mm away from the one removed by Flenniken & White (1983). Our

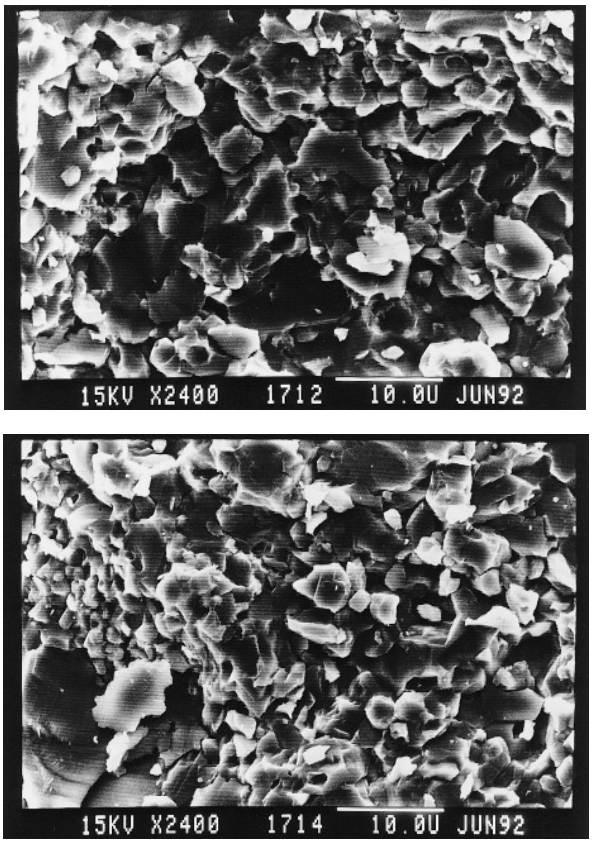

Figure 2. SEM micrographs of two further flakes from the same piece of Bass Point silcrete as Figure 1. (a): unheated; (b): heated. There is little observable difference between fracture planes.

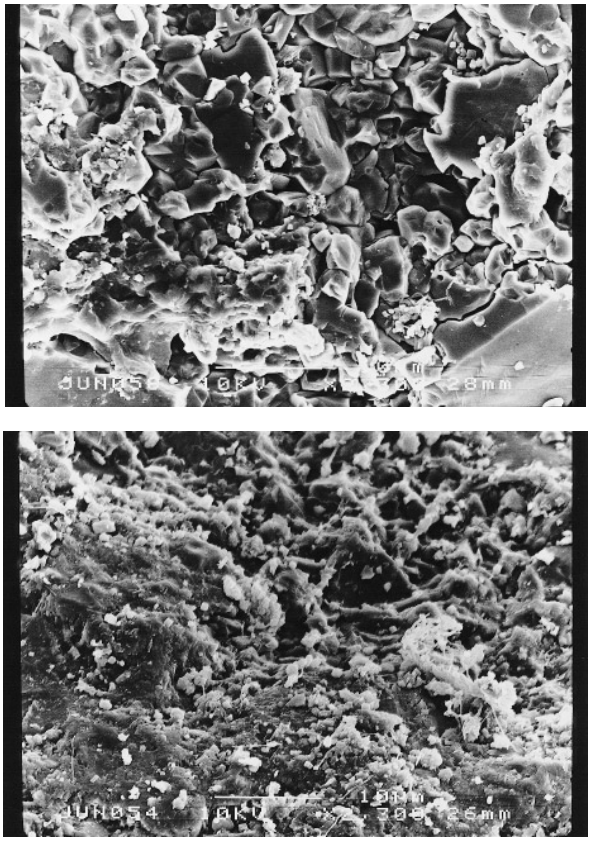

Figure 3. SEM micrographs of the unheated surfaces of opposite ends of a 4 mm-long flake of the same piece of Bass Point silcrete as shown in Figures 1 & 2. Considerable variation is apparent.

sample is only 4 mm in length while the original flake shown in Figure 1(b) is approximately 10 mm long. The original sample demonstrates clearly the expected changes after heating—the individual crystal structure is sheared away leaving an image of the outlines of macrocrystalline features by the transgranular fracture propagation. In the micrographs of our sample Figure 2(a) shows the unheated sample while Figure 2(b) is of the heated sample. Both these micrographs look very similar and resemble the unheated flake of Figure 1(a). The large flat areas of transgranular fracture force are not apparent here even though the sample is magnified to the same level. Figures 3(a) & (b) also show how much variation is found in a sample like this, as they show opposite ends of the flake we removed from the Flenniken & White (1983) unheated sample (Figure 2(a)). The keys to the similarities between the unheated and heated flakes are heterogeneity and lustre. The flake which we removed from Flenniken & White's heated sample was not lustrous and displayed no shearing of individual crystals. This suggests that heat treatment may only show up on SEM photos of samples which are glossy and not on those which are dull or lustreless.

On a number of other flakes (e.g. Figures 4(a) & (b)) we found that the larger skeletal quartz grains were subjected to shearing in both the raw and heated states making the differentiation between these states impossible using SEM. Thus we found no definitive evidence to support the theories that thermal alteration causes changes to the fracture plane or to the crystal structure.

Finally, the unreliability of SEM as a method of detecting heat treatment on silcrete can be seen in Figure 5 which shows a small area of a sample heated in a fire pit (see below). This contains regions which look like archetypal views of both the heated and unheated states. It demonstrates conclusively that using SEM it is possible to find evidence of heat treatment and also of no heat treatment on a single sample of inhomogeneous material.

Colour and lustre

These two attributes have been used by almost all researchers, so we tried to assess changes in these induced in silcrete by heating. We used 21 pieces of silcrete from Ulladulla on the New South Wales south coast, three silcrete samples from the Hunter Valley, NSW and two pieces of chalcedony from north-west Western Australia. The Ulladulla samples cover a broad range of textures from fine- to coarse-grained

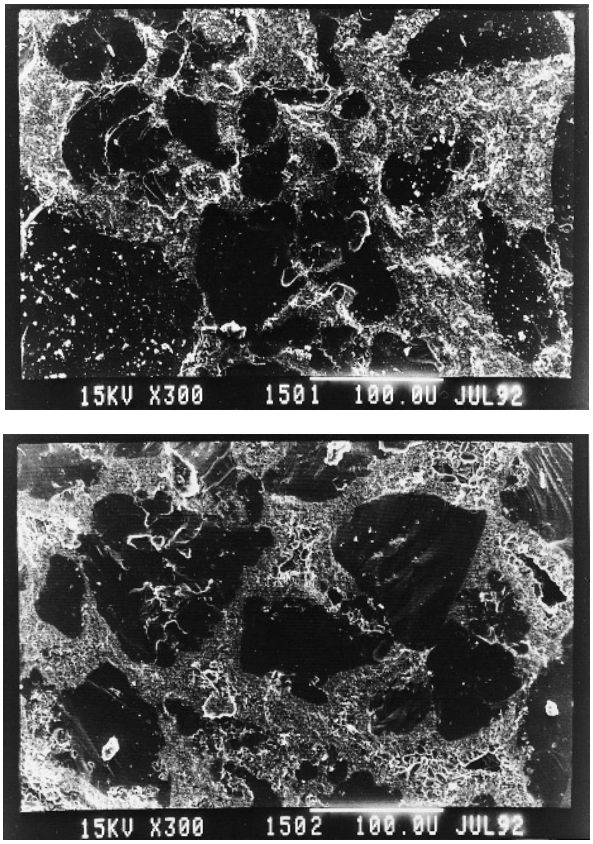

Figure 4. SEM micrographs of Ulladulla silcrete specimen 15. (a): unheated; (b): heated. Skeletal quartz grains are sheared in both states.

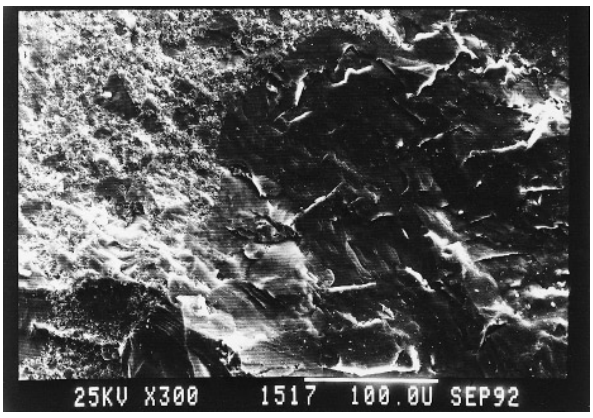

Figure 5. SEM micrograph of Hunter Valley silcrete specimen 51, flaked after heating in a fire pit. The rough end would usually be interpreted as unheated, the smooth end as heated.

and a range of very pale to strong colours including grey, red, brown and yellow. The samples were all classified from coarse- to fine-grained (a distinction made by eye) and then two of the samples were thin-sectioned to test our classifications. The coarsest sample (No. 1 in Table 1) was found to be of the GS-type fabric composed primarily of large quartz

*Table 1. Colour and lustre changes in 11 Ulladulla silcrete samples heated in a kiln. *Indicates slight lustre change, **indicates considerable lustre change*

Sample	Raw	250°C	350°C	450°C
1	10 YR 5/3	—	7·5 YR 5/4	7·5 YR 5/4
4	10 YR 6/3	—	—	—
	2·5 YR 6/2	—	—	—
	10 R 4/4	—	—	—
	7·5 YR 6/6	—	— *	—
7	10 YR 6/1	—	5 YR 7/1	5 YR 7/1
	7·5 YR 5/6	—	10 R 2·5/2 *	10 R 2·5/2 *
8	2·5 YR 5/0	—	—	—
10	10 YR 7/1	—	—	—
	10 YR 6/6	—	—	—
	10 R 4/2	—	—	—
	N4/0	—	—	—
	7·5 YR 5/6	—	10 R 3/2 *	—
15	2·5 Y 7/0	—	5 YR 6/1	—
22	5 R 4/4	—	5 R 4/4 **	— **
	5 R 6/3	—	7·5 R 3/2 **	— **
24	10 YR 6/1	—	—	—
	10 YR 7/4	—	—	—
29	N 4/0	—	—	—
	7·5 YR 6/8	—	—	—
	7·5 R 5/4	2·5 YR 4/6	2·5 YR 4/6	2·5 YR 4/6 **
	5 R 3/2	—	—	—
	5 YR 7/6	—	— *	—
31	N 7/0	—	N 4/0	—
	7·5 YR 5/4	—	7·5 R 3/2	— *
33	10 YR 7/1	5 YR 7/1	5 YR 7/1	5 YR 7/1
	10 YR 7/4	2·5 YR 5/4	2·5 YR 5/4	2·5 YR 5/4
	7·5 YR 5/4	2·5 YR 5/6	2·5 YR 5/6	2·5 YR 5/6

grains (approximately 500 μm in length) joined into a self-supporting structure by overgrowths around the grains (cf. Summerfield, 1983: plate 3.1a). The shapes of the original quartz grains were outlined by a thin iron oxide coating. Small areas of microcrystalline silica matrix were visible between the overgrowths. The second thin section was taken of one of the samples which we classified as being our 5th finest grained sample (No. 22 in Table 1). This thin section revealed the sample to be of the F-type fabric, having an average number of skeletal quartz grains in the size range of 100–500 μm and a greater proportion of matrix quartz grains which were less than 30 μm (Summerfield, 1983: plates 3.1b, 3.1c). No thin sections were taken from the finest grained sample (the Flenniken & White (1983) sample) as the SEM micrographs were used to determine the fabric type. The three Hunter Valley samples were all fine-grained and of a fairly consistent yellow-brown colour. The chalcedony samples, which were extremely fine-grained and pure white, were tested for comparisons.

The experimental samples consisted of 11 Ulladulla specimens, chosen to cover the texture and colour range. At least three flakes were removed from each sample and marked as A, B or C, denoting three different target temperatures: 250, 350 and 450°C respectively. Each of these three groups was first placed in a sand bath to control for differential heating, and then heated in a kiln for 16–18 h in total: 8 h to reach the target temperature and 8–10 h to cool to

ambient temperature. They were then examined for changes.

Out of 33 assessments of colour, only 14 changes were recorded (42%) (Table 1), and many of these were minor changes occurring in patches on the surface where sufficient iron was concentrated. Lustre was visible in only nine cases (27%) (Table 1). Lustre and colour changes did not necessarily occur on the same samples. We believe visual assessment of heat treatment is unlikely to be reliable when used alone.

Subsurficial Methods

We focus here on palaeomagnetism since, unlike SEM, thermoluminescence or thin-sectioning, artefacts are not damaged, while variation within specimens can be accommodated. Palaeomagnetism has been widely used in the study of ceramics, to determine such matters as authenticity and date (e.g. Barbetti, 1976, 1986; Barbetti *et al.*, 1980). These studies are enabled by palaeomagnetism because some of the small particles of iron oxides which are almost invariably present in soils, clays and rocks carry what are known as ''magnetic moments'' (Barbetti, 1986: 773). These magnetic moments are weak permanent magnetism oriented in a specific direction for each particle, according to that particle's shape and structure. A look at the magnetic moments of numerous particles together in one sample would show that they were generally oriented randomly. During heating, the magnetic moments of different particles will unlock from their current orientation and will oscillate from one position to the reverse, and will become locked again or ''frozen'' (Barbetti, 1986: 773) once they cool below their ''blocking temperature''. Magnetic moments of different particles unlock and relock at different temperatures. As the particle moments relock, their magnetic alignment is affected by the presence of a magnetic field—often the magnetic field of the Earth—causing the magnetic moments to tend to be aligned with it (Barbetti, 1986: 773). The result of this realignment is that more of the particles' magnetic moments are oriented in the same direction, which produces an increase in the overall strength of the magnetic moment of the sample. Demonstrating this increased magnetic strength is the key to detecting thermal alteration in stone artefacts.

We used two types of testing in our research, a preliminary one and a full one using the ''Thellier'' method. The preliminary testing procedure was tried initially on some experimental samples. When the results proved to be both interesting and non-damaging, the 23 artefacts from archaeological sites across NSW were subjected to the full palaeomagnetic test.

The preliminary test for magnetization consisted of placing each specimen in a Molspin magnetometer and reading its natural remanent magnetism (NRM) at ambient temperature, as a means of determining which specimens were most likely to have a measurable magnetism and which were not. Specimens with a weak NRM are likely to produce results which are more scattered and difficult to interpret, because the strength of their magnetic signal is hardly sufficient to override the ''noise'' appearing in the measurement procedure.

The ''Thellier'' method utilizes a process known as ''stepwise thermal demagnetization''. This is a two-part operation requiring the samples to be heated twice to the same temperature, and allows the heating temperature of the specimen to be estimated within the range 200–650°C, and the thermal or natural origin of the magnetism to be determined. The first heating and cooling is done in a specialized furnace (Magnetic Measurements Thermal Demagnetizer) which has zero magnetic field, as a means of partially removing the natural remanent magnetism (NRM) allowing the remaining partial NRM to be measured. The samples are then reheated in the furnace to the same temperature with a controlled magnetic field to impart a partial thermoremanent magnetization (TRM) which is measured in addition to the partial NRM. Vector subtraction of the partial NRM from the partial NRM+TRM will give a value for the latter. Graphing the results at different temperature intervals will give a straight line if the NRM is thermoremanent in origin (Barbetti, 1986).

Stepwise thermal demagnetization requires that the specimens are heated and cooled in the thermal demagnetizer at successive increments of 30–40°C at a rate of one heating per day over 40 days. The first heating at each temperature in zero magnetic field is followed by a heating to the same temperature in a known magnetic field. All artefacts were placed into the furnace at one time, and were heated to the following temperatures (°C) over successive periods of 24 h (heating and cooling time included): 130, 160, 190, 220, 260, 300, 340, 370, 400, 430, 460, 500, 530, 560, 590. The magnetic moment of each artefact was measured after each heating, and each measurement contributes to an overall reading of the artefact's magnetic history.

Specimens for testing

(a) Experimental specimens. We used nine pairs of specimens (heated and unheated) for the preliminary testing. Of the heated samples ($N=9$), three came from the kiln-heated samples and six (five Ulladulla silcretes, one chalcedony) from a fire pit. The fire pit consisted of a layer of soil over a bed of hot coals. Artefacts were placed on the soil, covered with another layer of soil and a fire which burned for 5 h. Artefacts were removed after 24 h of cooling. Palaeomagnetic testing on these artefacts shows they were heated to 430°C. One of the requirements of this method is that the samples be smaller than a 25 mm cube, so we cut them with a diamond saw. The sample cubes were marked with arrows to help align them correctly and were then put directly into the magnetometer.

(b) Archaeological specimens. Twenty-three archaeological samples came from five geographical areas and included specimens known to have been heated previously, as well as others which, based on their external features and archaeological contexts, were suspected of having been heated. Of the 23, eight specimens came from four artefacts from the coastal site of Bass Point as tested by Flenniken & White (1983) using SEM. Each of the four artefacts in this group had been previously cut in half during the original research with one half of each having been heated to the temperature of 275°C, while the remaining portions were kept as the testable unknowns. We were able to utilize the heated portions here as a cross-check on the results because we had the unheated pieces which retained their original natural magnetism. Five artefacts were used from the coastal site of Currarong (Lampert, 1971), where some of Hanckel's (1983) artefacts were obtained. The assemblage could be suspected of containing some heat-altered artefacts if Hanckel's results are to be taken at face value. The chosen specimens also exhibited some surface gloss, and appeared most likely to have been heat treated. Eight artefacts came from Narama 48 site in the Hunter Valley, about 80 km inland, and were included because they exhibited external signs of heat alteration, including a deep red colour and a slight degree of increased surface gloss. Two specimens were from Warkworth, near Narama, and while exhibiting no external signs of heat treatment their context suggested that they may have been heat affected.

Samples were selected to fit the 25 mm size restriction. Individual moulds were made from a silicon rubber compound (Silastic E® RTV—room temperature vulcanizing). Each mould was a 25 mm cube and was designed to hold the specimen in precisely the same position each time it was placed in the magnetometer. The decision to use silicon rubber rather than the usual plaster was so that the specimens could be removed from the moulds after each measurement without incurring any damage and then could be placed into the furnace. While silicon rubber is a messy moulding material, it was quite effective.

Results

The results of the preliminary tests on the experimental samples were quite diverse. The three samples which had been heated in the kiln to 250°C returned, as predicted, a magnetic measurement which was lower than the unheated samples. The explanation for this is that the kiln is steel framed and thus produced its own magnetic field which was lower than the magnetic field outside. Hence when the samples cooled down they incurred a reduced rather than an increased magnetic strength. The remaining six pairs tested had been heated in the fire pit and the results varied widely. Two determinations were lower than the originals. In both it appears as though the samples were heated to a temperature where either the iron oxide particles turned to haematite, which has a lower magnetic intensity, or only half the particles were unlocked during heating and relocked in an opposite direction to the upper temperature magnetic field, effectively cancelling the effect of each other out (M. Barbetti, pers. comm.). One of these samples was the chalcedony from Western Australia which from external appearances is particularly low in iron particles. Of the four samples which did give a result, the increase in strength of the magnetic field intensity was between 1·7 and 476 times.

The results of the full (Thellier method) palaeomagnetic tests on the archaeological samples are set out in Table 2. Producing an interpretable result from the data collected from the Thellier method requires the NRM to be plotted against the TRM. If the samples were heated in antiquity, joining the points on the graph for each temperature will produce "a straight line with negative slope equal to the ratio of original to laboratory magnetic fields" (Barbetti, 1976: 139). Samples where the points are distributed around the ideal straight line can be validly interpreted with a line of best fit. It is also valid to exclude low and high temperature points from the calculation of the slope where linearity is constant over a wide temperature range (Barbetti, 1976).

These results give an unambiguous result for 60% of the artefacts and an interpretable result for a further 13%. Since the method does not produce false positives, this represents the minimum number of heated artefacts. There are 10 cases (5, 10, 14, 16, 18, 19, 20, 21, 22, 24) in which the magnetic plots give us a positive indication of heat treatment, or so closely resemble heat treatment that it would be difficult to argue any other way. There are three cases (3, 4, 7) of possible heat treatment with some noise hindering a more definite interpretation. Four cases (6, 15, 23, 25) show that the samples were not heat treated, although some of these are more easily interpreted than others as noise also plays its part. Finally there are six cases (8, 9, 11, 12, 13, 17) where there is more noise than magnetic signal and thus no interpretation is possible.

The heated control sample 1 presented problems because of the size of the sample and the relatively strong background magnetic signal. There was a lot more noise in the readings than expected. Knowing that it was heated, it is possible to make a case for the magnetic results showing this, and the overall distribution of the temperature readings is consistent with heating. However, if this were an unknown sample it would probably be regarded as noise obscured. It appears as though the sample was moved during heating and thus the magnetic remanence changed direction.

The following three examples represent the three different kinds of results which may be expected from this testing method—definite evidence of heating, definite evidence of no heating and an indeterminate outcome due to noise interference.

Table 2. Results of palaeomagnetic tests on archaeological samples

Sample	Provenance	Result
1	Heated control (from fire pit)	Consistent with heat treatment
2	Unheated control	Lost in furnace
3	Narama	Looks like heat treatment—slightly noise obscured
4	Narama	Looks like heat treatment—slightly noise obscured
5	Narama	Heat treated to 430°C
6	Narama	Definitely not heat treated
7	Narama	Possible heat treatment—slightly noise obscured
8	Narama	Noise obscured
9	Narama	Noise obscured
10	Narama	Heat treated to 430°C
11	Currarong	Noise obscured
12	Currarong	Noise obscured
13	Currarong	Noise obscured
14	Currarong	Heat treated to 400°C
15	Currarong	Not heat treated—also noisy
16	Bass Point 1A	Heat treated to 370°C
17	Bass Point 1B	Noise obscured
18	Bass Point 3B	Heat treated to 430°C
19	Bass Point 3A	Heat treated to 430°C
20	Bass Point 7A	Heat treated to 430°C
21	Bass Point 7B	Heat treated to 430°C
22	Bass Point 2B	Shows previous experimental heating
23	Bass Point 2A	Not heat treated
24	Warkworth	Heat treated to 400°C
25	Warkworth	Not heat treated

Bass Point samples marked A are the untreated half of the samples marked B which were reheated to 275°C by Flenniken & White (1983) as part of a previous heat treatment detection study.

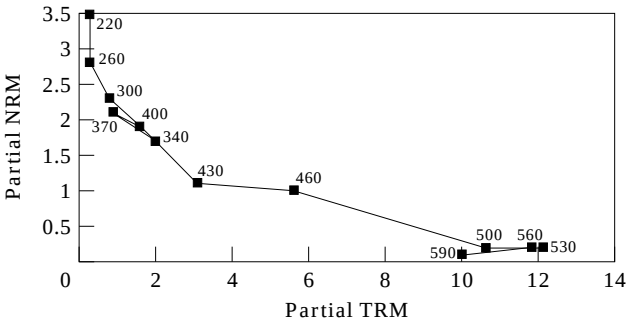

Figure 6. Relation between partial TRM and partial NRM at a range of temperatures (°C) for sample 5 (Table 2). The constant slope below 430°C indicates heat treatment.

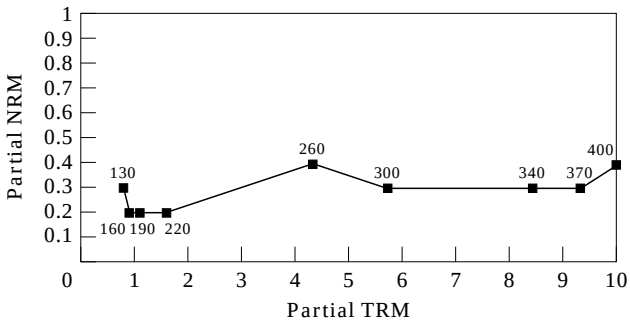

Figure 7. Relation between partial TRM and partial NRM at a range of temperatures (°C) for sample 6 (Table 2). The constant relation at all temperatures indicates absence of heat treatment.

Sample 5 (Figure 6) is the most distinct case of a clearly heat-treated artefact from this series of tests. A line of best fit through the points below 430°C gives a line of negative slope with a constant ratio between NRM and TRM. The low sloping line above 430°C is the record of the geological magnetism still remaining in the sample and unaffected by the heating which stopped in the vicinity of 430°C.

Sample 6 (Figure 7) is the most definite example we have of an artefact which has not been heat altered. While the NRM of the sample rates as magnetically fairly weak, it does acquire a sufficient magnetic TRM signal, which would have registered a thermal alteration if that had happened.

An example of a completely indeterminate outcome due to noise interference can be seen in sample 9 (Figures 8(a) & (b)). In the first graph, the magnetic readings beyond 430°C merely demonstrate the geological magnetism, while the readings below this temperature in the second graph are so randomly distributed that they can only represent noise, having no linear structure at all.

Our results have established that some form of thermal modification has been made to some of the artefacts at Narama, Currarong, Warkworth and Bass Point. However, the results we review closely are those from Bass Point where the artefacts were previously tested by Flenniken & White (1983). Three of the four artefacts tested show evidence of a thermal history and also retain all of the external indicators of heat treatment, results which corroborate those of Flenniken & White (1983). However, one artefact (specimens 22 &

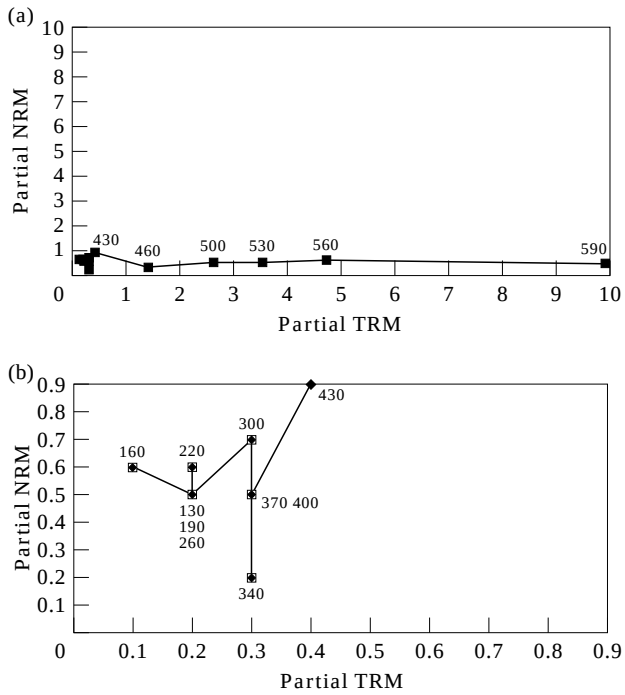

Figure 8. Relation between partial TRM and partial NRM at a range of temperatures (°C) for sample 9 (Table 2). (a): overall; (b): area below 430°C. The readings >430°C reveal geological magnetism; the random distribution of readings at temperatures <430°C indicate only noise.

23) was originally deemed to be heat treated and yet this is not apparent in its magnetic history. This result suggests that either palaeomagnetism needs to be reviewed for producing false negatives or that SEM results need to be treated with caution—as we observed earlier.

Discussion and Conclusions

Our experiments suggest that no one technique can be used to determine whether silcrete artefacts have been heat treated.

Lustre is useful for determining whether heat treatment has taken place because if it develops heating must have been prior to flaking. However, it does not always develop and can sometimes be confused with silica sheen or desert varnish. If the thermal origin of lustre can be determined, then heat treatment has definitely occurred. The major advantage of this method is that it can be observed directly on artefacts.

When applied to silcretes, SEM is not a reliable technique for detecting heat treatment because it is intrinsically able to produce false positive and negative results depending on the inhomogeneity of the raw material and how the artefact's surface area is sampled. This has been demonstrated through the inability to replicate the results of previous SEM determinations for heat treatment on the same artefacts. One of the major failings of SEM is that while it appears to be an objective technique, the results require a lot of interpretation based on subjective concepts like a smoother surface or a change in overall crystal size.

In contrast to SEM, palaeomagnetism will not produce false positives, is not subject to sampling error as the whole artefact is used, and requires only the interpretation of a graph which is usually quite straightforward. However, there are two major problems with palaeomagnetism. The first is the need to use artefacts which are small enough to fit into the 25 mm space in the magnetometer (thus avoiding cutting the artefacts) and still fill as much of this space as possible. This seriously reduces our archaeological sample choice and thus palaeomagnetism cannot be used for all artefacts on a site without damaging some of them. Unfortunately, the smaller the size of the artefacts, the greater the likelihood of noise interference as the NRM tends to be weaker in smaller samples. The second problem with palaeomagnetism is that it is able to establish only that an artefact has been heated and not to differentiate between heating before and after it has been flaked. Heat *treatment* is only useful if the heating occurred before the flaking event. Because the interpretation of heat treatment is dependent on the position of the heating in the reduction sequence we still require another method of determining the true nature of the thermal modifications.

We suggest that the most reliable and non-damaging method of determining heat treatment on silcrete artefacts comes from using a combination of lustre and palaeomagnetism. Lustre is able to tell us that the artefact was heated before it was flaked and palaeomagnetism is able to confirm that the lustre is of thermal origin and not from some other natural process. Using this approach we not only achieve reliable conclusions but also find archaeologically interesting information.

Acknowledgements

This research started as an Honours thesis by Rowney (1992), supervised by White and was supplemented by further research by Rowney, funded by the Australian Institute for Aboriginal and Torres Straits Islander Studies (Grant L93/4547). Most of the work was carried out in the N. W. G. Macintosh Quaternary Dating Laboratory, and we thank Dr Mike Barbetti and Ms Xiao Yu most warmly for their constant assistance and encouragement. We thank also Dr C. Nockolds, Tony Romeo and Adam Sikorski of the Electron Microscope Unit, and for samples The Australian Museum and Dr Laila Haglund. This paper is a joint production by the two authors, with critical input, much appreciated, from Robin Torrence.

References

Barbetti, M. (1976). Archaeomagnetic analysis of six Glozelian ceramic artifacts. *Journal of Archaeological Science* **3,** 137–151.

Barbetti, M. (1986). Traces of fire in the archaeological record before one million years ago? *Journal of Human Evolution* **15,** 771–781.

Barbetti, M., Clark, J. D., Williams, F. M. & Williams, M. A. J. (1980). Palaeomagnetism and the search for very ancient fireplaces in Africa. *Anthropologie* **18,** 299–304.

Crabtree, D. E. & Butler, B. R. (1964). Notes on experiments in flint knapping: 1—heat treatment of silica materials. *Tebiwa* **7,** 1–6.

Domanski, M. & Webb, J. (1992). Effect of heat treatment on siliceous rocks used in prehistoric lithic technology. *Journal of Archaeological Science* **19,** 601–614.

Flenniken, J. J. & White, J. P. (1983). Heat treatment of siliceous rocks and its implications for Australian prehistory. *Australian Aboriginal Studies* **1983/1,** 43–48.

Hanckel, M. (1983). *Hot Rocks from the South Coast: a Study of Thermal Pretreatment at Burrill Lake and Currarong, New South Wales*. Honours Thesis, Department of Anthropology, University of Sydney (unpublished).

Lampert, R. J. (1971). Burrill Lake and Currarong. *Terra Australis* **1**.

Langford-Smith, T. (1978). A select review of silcrete research in Australia. In (T. Langford-Smith, Ed.) *Silcrete in Australia*. Armidale: University of New England, Department of Geography, pp. 1–12.

Mandeville, M. D. (1973). A consideration of the thermal pretreatment of chert. *Plains Anthropologist* **18/61,** 177–202.

Olausson, D. S. & Larsson, L. (1982). Testing for the presence of thermal pretreatment of flint in the Mesolithic and Neolithic of Sweden. *Journal of Archaeological Science* **9,** 275–285.

Pavlish, L. A. & Sheppard, P. J. (1983). Thermoluminescent determination of Paleoindian heat treatment in Ontario, Canada. *American Antiquity* **48,** 793–799.

Purdy, B. A. & Brooks, H. K. (1971). Thermal alteration of silica minerals: an archeological approach. *Science* **173,** 322–325.

Rowney, M. (1992). *Heat Treatment on the Rocks: (Shaken not Stirred)*. Honours Thesis, Department of Prehistoric and Historical Archaeology, University of Sydney (unpublished).

Schindler, D. L., Hatch, J. W., Hay, C. A. & Bradt, R. C. (1982). Aboriginal thermal alteration of a central Pennsylvania jasper: analytical and behavioural implications. *American Antiquity* **47,** 526–544.

Summerfield, M. A. (1983). Silcrete. In (A. S. Goudie & K. Pye, Eds) *Chemical Sediments and Geomorphology: Precipitates and Residua in the Near-Surface Environment*. London: Academic Press, pp. 59–91.

Twidale, C. R. (1983). Australian laterites and silcretes: ages and significance. *Revue de Geologie Dynamique et de Geographie Physique* **24,** 35–45.