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THE USE OF THE SCANNING ELECTRON MICROSCOPE IN STUDYING THE HEAT TREATMENT OF PREHISTORIC LITHIC ARTIFACTS FROM THE NORTH FLORIDA WEEDEN ISLAND PERIOD MCKEITHEN SITE

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Abstract

In this paper, I discuss a study in which I attempted to determine whether or not the inhabitants of the McKeithen site, a Weeden Island Period mound and village complex in what is today northern Florida, employed heat treatment as part of their lithic reduction technology, and if so, at what point in the reduction sequence it was conducted. I used a scanning electron microscope (SEM) to photograph raw and heat treated control samples of northern Florida chert, then debitage and tools of chert and silicified coral from the McKeithen site which represented the range of colors and luster present in the assemblage. A comparison of the photographs of the controls with those of the archaeological specimens showed that the McKeithen knappers did heat treat some of their lithic materials. Based on this sample, I determined that about 16% of the chert and 38% of the silicified coral had been heat treated. In general, the McKeithen knappers brought potential cores to the village from nearby quarries, heat treated some of them, then detached flakes from them and reduced the flakes, and occasionally the cores, into tools. While this study largely corroborates the finding that luster is a better macroscopic indicator of heat treatment than color change, exceptions occurred which demonstrate the necessity of using instrumental techniques to accurately detect heat treatment.

Key words: scanning electron microscopy, archaeology, lithic technology, heat treatment, debitage, chert, silicified coral, McKeithen site, Weeden Island Period, Florida.

Introduction

In this paper, I discuss the use of the scanning electron microscope (SEM) in determining the heat treatment of lithic artifacts from the McKeithen site, a Weeden Island Period mound and village complex in North Florida (Milanich et al. 1984) (Figure 1). The site's lithic assemblage consists of cores, blanks, preforms, and bifacially worked triangular and lanceolate end products, along with a number of possibly utilized flakes and more than 6500 unutilized flakes which are by-products of stone tool manufacture at the site. Figure 2 summarizes the reduction technology employed by the McKeithen flintknappers. The major materials used were chert and silicified coral, both of which are locally available and occur in a variety of colors. Jasper, quartz cobble and pebble, and quartz crystal are also present in small amounts. The goal of the study was to determine whether or not the lithic materials had been heat treated; and if so, at what point in the reduction process the heat treatment was conducted.

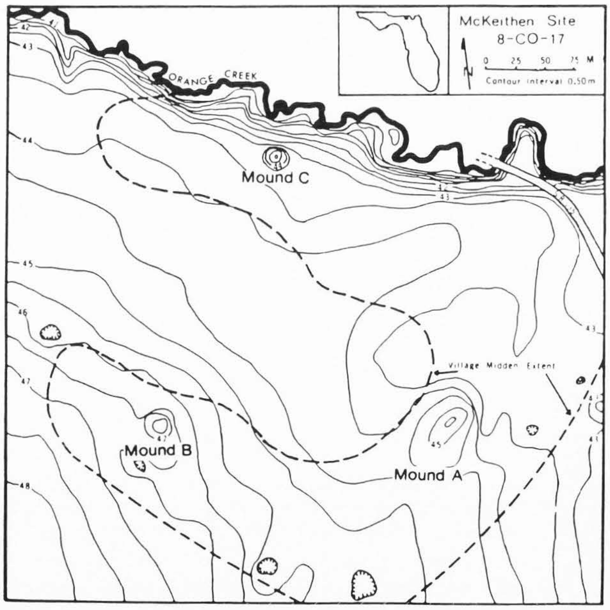


Figure 1. The McKeithen site (after Milanich and Fairbanks 1980).

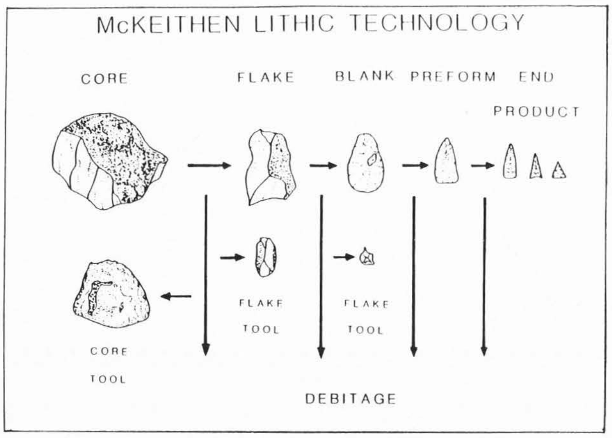


Figure 2. McKeithen lithic technology.

Heat treatment of lithic materials prior to their reduction into finished tools has been well documented ethnographically (Hester 1972; Mandeville 1973), and has been the subject of much experimental research (Crabtree and Butler 1964; Purdy and Brooks 1971; Purdy 1974; Mandeville 1971, 1973; Mandeville and Flenniken 1974; Collins and Fenwick 1974; Flenniken and Garrison 1975; Rick 1978). While heat treatment effects have been found to vary among different materials, heat treatment generally reduces the material's tensile strength, increases its compressive strength if it is cooled properly, increases the sharpness of subsequently detached flakes, and improves the material's overall flakeability. Purdy and Brooks (1971) suggest that these changes occur because heating fuses impurities in the intercrystalline matrix. This process increases the structural homogeneity of the material because upon impact the fused impurities serve as fluxes. Flenniken and Garrison (1975) suggest that while such changes in the matrix may occur, the improved flakeability results from an increase in the number of microfractures within the stone. They believe that these microfractures allow the force of a blow to travel more directly through the crystalline lattice.

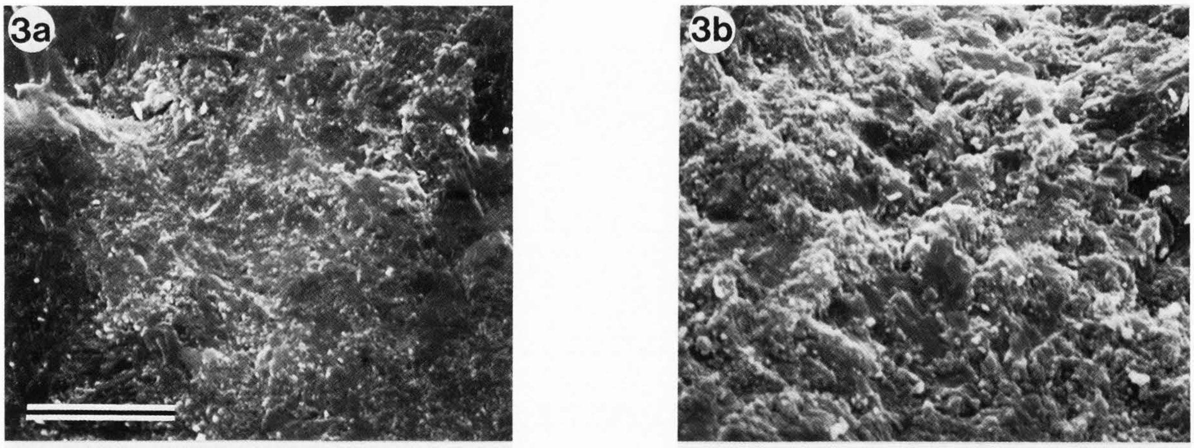
Two macroscopic indicators of heat treatment most often mentioned by researchers are color change, often to a red or pink, and increased luster. Many of the McKeithen specimens exhibit a red or pink color or lustrous appearance or both. Purdy and Brooks (1971) report that such a color change often occurred in Florida cherts heated to between 240° C and 260° C, but only when iron oxides were present in the material. A change in luster consistently occurred when the temperature reached about 350° C, making luster the more reliable indicator of heat treatment. Luster increases because light is reflected more uniformly by the smoother fractured surface (Rick 1978); however, certain materials become more lustrous than others when heat treated, and post-depositional patination can cause changes to the stone's surface which obscure or may be confused with luster caused by heat treatment.

Instrumental techniques can provide a more accurate determination of heat treatment than can attempts to use macroscopic indicators whose expression is influenced by a variety of factors. X-ray diffraction, thermoluminescence, and scanning electron microscopy have been used to detect heat treatment. X-ray diffraction has yielded generally poor results. Purdy and Brooks (1971) and Thiel (1972) both observed no change in heat treated materials. Weymouth and Mandeville (1974) noted a lowering of intensity peaks in heat treated materials, but only after the materials were heated to over 400° C, well above the temperature necessary to alter the stone. The amount of thermoluminescent energy stored in lithic artifacts has recently been used both to detect heat treatment (Pavlish and Sheppard 1983), and to determine when heat treatment took place (Purdy 1981). Scanning electron microscopy has been used by a number of researchers (Purdy and Brooks 1971; Mandeville 1973; Collins and Fenwick 1974; Rick 1978; Bond 1981; Draper and Flenniken 1984). These studies have consistently shown that freshly flaked surfaces have a smoother, flatter appearance after heat treatment due to the fracture's relatively straight path through the crystals. These differences are often quite dramatic.

Table 1. Specimens used in SEM analysis

	Material	Color	Luster
Control Specimens			
Heat Treated	Chert*	10 R 4/10	High
Raw	Chert*	10 YR 9/2	None
McKeithen Specimens			
Heat Treated	Chert*	10 R 5/4	High
	Chert*	10 R 6/4	Medium
	Chert	10 YR 9/2	High
	Chert	7.5 R 5/4	High
	Chert	2.5 Y 8/2	Medium
	Chert	10 YR 6/6	Medium
	Chert	5 Y 5/2	High
	Chert	10 YR 9/2	Medium
	Chert	10 YR 8/4	None
	Chert	10 R 8/2	Medium
Probably Heat Treated	Chert*	7.5 R 3/4	High
	Chert	7.5 R 5/6	Medium
	Chert	5 B 4/1	High
	Chert	5 Y 9/1	Medium
	Coral	10 YR 9/1	High
	Coral	5 Y 9/1	High
Raw	Chert*	10 R 7/6	None
	Chert*	2.5 Y 4/4	High
	Chert	7.5 YR 7/6	Medium
	Chert	10 R 4/6	High
Probably Raw	Chert*	10 B 7/2	Low
	Chert	10 R 4/4	Low
	Chert	10 YR 6/4	High
Indeterminate	Chert	10 YR 1/9	High

*illustrated



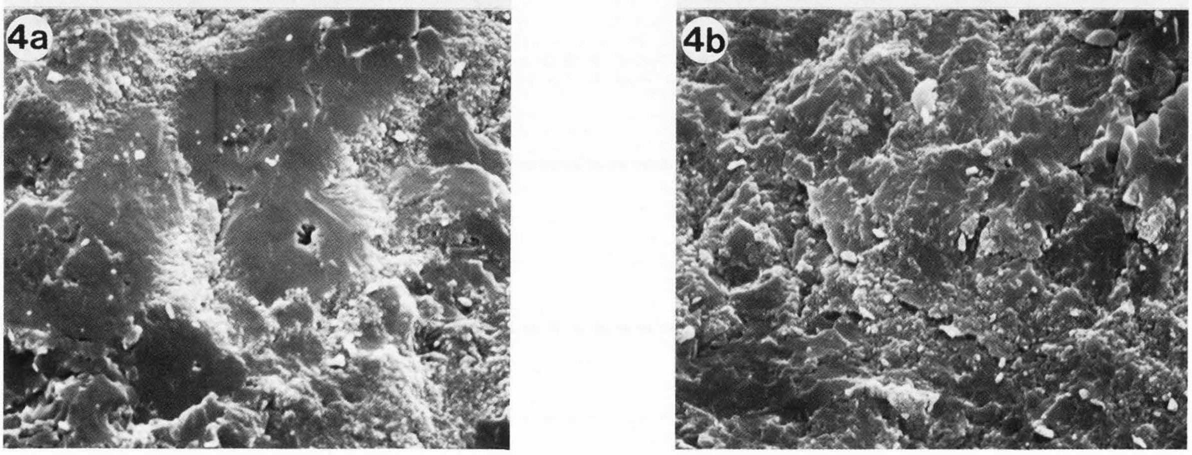


Figure 3. Control (a) heat treated, (b) raw.

Figure 4. Heat treated archaeological specimen.

All micrographs at same magnification. Bar = 10 μm.

Materials and Methods

The McKeithen lithic specimens were studied using a U1 ETEC Autoscan SEM. First, two flakes suitable for subsequent reduction into bifaces were removed from a nodule of Florida chert which was used as a control specimen. This material is comparable in color and texture to the cherts in the McKeithen collection. One flake was left unheated and one was surrounded by sand in a closed metal container and heated in an oxidizing Lindburg furnace for 10 hours at 350° C. This specimen was allowed to cool naturally. Small flakes were then removed by pressure flaking from the raw and heat treated controls, and from 24 flakes and bifaces from the McKeithen assemblage which represented the range of colors and luster present in the collection (see Table 1). Care was taken to select artifacts that had not been weathered, since weathering can cause internal changes to the stone, especially if it has been heat treated (Purdy and Clark 1979). These small flakes were removed to obtain samples which would fit into the SEM's small viewing compartment, and to provide freshly flaked surfaces which revealed the stone's interior. The flakes were affixed ventral side up to adhesive-covered stages and electrically coated with gold for nine minutes in a Technics Hummer. To ensure even coating, flakes were oriented at a 90° angle for half of the time and at a 45° angle for the other half. Each of the 26 flakes was then observed in the SEM, and an area which typified the flake's appearance was selected.

The photographs of the controls and archaeological samples were then compared. Figures 3a and 3b show that the controls exhibit a readily observable change from the raw to the heat treated condition. The raw sample has a few small, flat areas surrounded by larger areas of greater relief. The overall appearance is irregular because the flake's ventral surface consists of complete crystals sticking up amid depressions left when other crystals remained on the flake blank. The heat treated sample appears extremely flat and smooth over its entire surface because the fracture went through most of the crystals.

Figures 4a and 4b through 6a and 6b, all illustrating chert specimens, show that while some McKeithen specimens closely resemble either the heat treated or raw control, others appear to be intermediate between the two. In these cases, specimens which exhibit particularly large or frequent flat areas are interpreted as probably heat treated, while the rest are probably raw. This interpretation agrees with the findings of a similar analysis done for the National Park Service by the Washington State University Laboratory of Lithic Technology. In this study, experimental heat treatment of lithic materials from Wyoming in several cases resulted in an increase in the size and frequency of flat areas rather than a change to a flattened appearance over the entire surface. This finding suggests that heat treatment effects vary among different kinds of materials, and even among different cherts, probably due to varying degrees of homogeneity in the materials which necessitate different temperatures, different durations of heating, or both (see Rick 1978; Bond 1981).

Results

The SEM analysis of the McKeithen specimens shows that in general, especially lustrous specimens of all colors represented in the collection appear to have been heat treated, while specimens which do not exhibit marked luster, even red and pink ones, do not appear under SEM scrutiny to have been heat treated. Many flakes of heat treated chert did become red or pink. This color change either occurred throughout the stone or was restricted to the outer few micrometers which were often subsequently flaked off. Three major exceptions to these general patterns were noted. Lustrous specimens of translucent amber chert do not appear to have been heat treated. The same is true of extremely lustrous olive-green jasper which is represented by only a few flakes and a single biface. The most interesting exception is a distinctive lustrous chert found only on the floor of the structure which Milanich et al. (1984) interpret as the religious specialist's residence. This material is slightly translucent to opaque and is predominantly bright red with some areas which grade into tan. Flakes of this material do not appear to have been heat treated. Table 1 summarizes the appearance of the specimens used in the analysis. Color is described using Munsell notation. Luster is subjectively assessed because the equipment necessary for measuring the critical angle of surface reflection was unavailable.

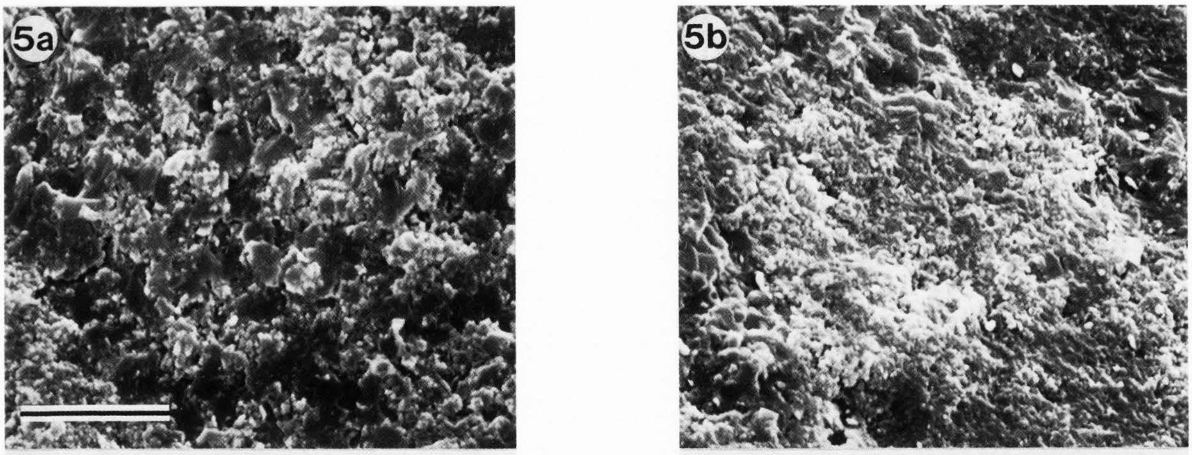
Discussion

Based on the findings from this sample, each item in the McKeithen assemblage was observed and classified as raw, heat treated, or indeterminate. Crazed and potlidded specimens were also recorded. Since control samples of silicified coral were not obtained for SEM analysis, determinations of heat treatment for the silicified coral items were based on the degree of luster, which varied greatly within the collection. Heat treatment of quartz specimens was not suspected, but based on luster they were usually recorded as indeterminate. Omitting heat damaged specimens, which are possibly the result of accidental heating, and indeterminate specimens, 16% of the chert flakes and 38% of the silicified coral flakes appear to have been heat treated. The corresponding tool percentages are 17% and 47%.

Table 2. McKeithen debitage: Heat treatment by reduction stage.

	Raw	Heat Treated	Other	Total
Chert				
Primary Decortication	93(74%)	18(14%)	15(12%)	126
Secondary Decortication	627(72%)	152(17%)	96(11%)	875
Core Reduction	20(80%)	3(12%)	2 (8%)	25
Early Shatter	801(69%)	202(17%)	156(14%)	1159
Bifacial Thinning	1967(77%)	362(14%)	219 (9%)	2548
Late Shatter	362(66%)	143(26%)	48 (8%)	553
Pressure	21(75%)	4(14%)	3(11%)	28
Indeterminate	578(70%)	95(12%)	154(18%)	827
Total	4469	979	693	6141
Coral				
Primary Decortication	15(75%)	4(20%)	1 (5%)	20
Secondary Decortication	26(39%)	28(42%)	12(19%)	66
Core Reduction	1(50%)	0	1(50%)	2
Early Shatter	36(41%)	44(50%)	8 (9%)	88
Bifacial Thinning	70(52%)	43(32%)	23(16%)	136
Late Shatter	8(27%)	18(60%)	4(13%)	30
Pressure	0	0	0	0
Indeterminate	25(56%)	10(22%)	10(22%)	45
Total	181	147	59	387

An alternative analytical method which could have been used to provide a more direct determination of heat treatment is the comparison of experimentally heated and unheated archaeological specimens. This method involves breaking archaeological specimens into halves, then heat treating one half of each specimen and leaving the other halves as controls. These specimens must be a minimum of about 1 cm in thickness to heat treat properly. Since repeated heat treatment causes no further change in the stone, the interpretation is relatively straightforward. If experimental heat treatment alters the specimen, then it was not heat treated prehistorically. If heat treatment does not alter the specimen, then it was previously heat treated. Care must again be taken to observe unweathered areas. While this method provides excellent and often unambiguous results, its destructiveness was deemed unsuitable for this study.



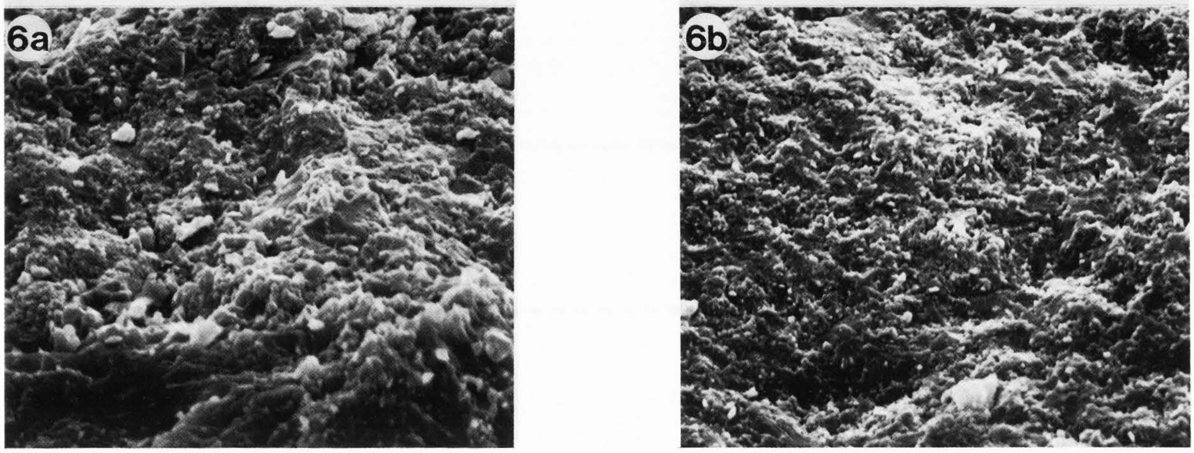


Figure 5. Probably (a) heat treated and (b) raw archaeological specimen.

Figure 6. Raw archaeological specimen.

All micrographs at same magnification. Bar = 10 μm.

Based on flintknapping experimentation and on the experimental work of a number of researchers (Newcomer 1971; Crabtree 1973; Callahan 1979; Frison and Bradley 1980; Magne and Pokotylo 1981; Gilreath 1983), the McKeithen debitage had previously been divided into eight categories. Complete flakes were labeled primary decortication flakes, secondary decortication flakes, core reduction flakes, bifacial thinning flakes, and pressure flakes. Flake fragments were labeled early shatter and late shatter. Flakes and fragments that could not be more specifically described were labeled indeterminate flakes.

Primary decortication flakes (White 1963) have dorsal surfaces completely covered with weathering surface, or cortex. Secondary decortication flakes (White 1963) have partial cortex cover on their dorsal surfaces. These two kinds of flakes were produced during both core and biface reduction. Core reduction flakes were removed from the cores by percussion both during the production of flakes to be made into tools and as a test of material quality. They are fairly large flakes which have no cortex and usually have one to three dorsal flake scars. Bifacial thinning flakes (Crabtree 1972) were struck off the bifaces following the removal of the cortex. They are considerably smaller than core reduction flakes, usually have two or more dorsal flake scars, and often have a degree of curvature. Pressure flakes, a rather subjective category, also usually exhibit some curvature and often have two dorsal flake scars whose intersection forms a central ridge.

Flake fragments considered early shatter have either a complete or partial dorsal cortex cover or are fairly large, often angular, and lack cortex. They result from core and early biface reduction. Late shatter is defined as small flake fragments which have no cortex, have three or more flake scars, and are sometimes curved. They are produced during bifacial thinning (Magne and Pokotylo 1981). Indeterminate flakes and fragments are quite small fragments or small, flat, complete flakes with one or two dorsal flake scars which could have been produced at any time during core or biface reduction.

The pattern of heat treatment by reduction stage is summarized in Table 2. The percentages of heat treated chert flakes vary little from category to category except for late shatter which shows a 9% increase over the next highest categories. The silicified coral debitage shows, with one major exception, an incremental increase in the amount of heat treatment as reduction progresses. The exception is the bifacial thinning flake category which has a much lower heat treatment percentage than is expected compared to the other categories. Crazed, potlidded, and indeterminate specimens comprise the "Other" category.

The heat treatment pattern seen in the chert debitage indicates that the McKeithen inhabitants heat treated selected cores and probably flakes, as well. This conclusion is based on the high percentage of heat treated cortical and core reduction flakes. A number of the cortical flakes have dull dorsal and lustrous ventral surfaces showing that they were in the first series of flakes removed from the core after heat treatment. The 22 chert cores in the McKeithen collection range in size from 102.5 by 76.5 by 64.7 mm to 26.8 by 23.1 by 12.1 mm. Seven of these cores appear to have been heat treated. The higher percentage of late shatter evidencing heat treatment may be the result of the material's reduced tensile strength which caused increased breakage per blow.

The silicified coral's heat treatment pattern is more difficult to interpret, and conclusions are tentative due to the small sample size. It does seem clear that a much larger percentage of silicified coral than chert was heat treated. Since Austin (1983) reports that raw silicified coral is particularly difficult to work, it is not surprising that the McKeithen flintknappers heat treated it more often. They probably heat treated silicified coral as cores, and possibly as cortical flakes. If the lower than expected percentage of heat treated bifacial thinning flakes is not the result of small sample size or misidentification of heat treatment, then it may mean that either these flakes were often being selected out, perhaps for use as flake tools or for some other purpose, or that silicified coral specimens were often removed from the site before being completed. If either suggestion is correct, then the locations of these items are unknown.

Conclusions

While this study has largely corroborated the conclusion that luster is a better indicator of heat treatment than color change, it has also revealed exceptions which emphasize the need to conduct instrumental analyses to obtain accurate results. Once accurate results are obtained, one can derive important technological and behavorial information. Scanning electron microscopy is a useful technique for heat treatment analyses because it is relatively inexpensive if the necessary facilities are available, and the interpretation of its results, while not entirely unambiguous, is relatively straightforward.

Acknowledgements

I would like to thank the Florida State Museum, and specifically Dr. Jerald T. Milanich, for the loan of the lithic materials used in this study. I would also like to thank Joyce L. Sutphin Davis, Chris M. Davitt, and Sue Backus of the Washington State University Electron Microscopy Center for sharing their equipment and expertise. Dr. Timothy A. Kohler and Dr. J. Jeffrey Flenniken of the Washington State University Department of Anthropology, provided technical, editorial, and financial assistance for which I am very grateful. Finally, I would like to thank Eileen M. Adams Draper for drawing Figure 1, and Annette Hoch-Johnson for giving me the benefit of her word processing and proofreading skills.

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Discussion with Reviewers

B.A. Purdy: I question the application of instrumentation when visual observation may have permitted conclusions that fell within the same range of error.

Author: While visual observation may in some cases provide a reasonably accurate subjective assessment of heat treatment, complicating factors such as patination and natural luster variability make instrumental demonstration of heat treatment highly desirable, especially in studies of numerous specimens. Actually, visual determination of heat treatment has been conducted on these materials (Milanich et al. 1984), and quite different results were obtained. According to previous analysis, about 39% of the chert debitage and 25% of the coral debitage in the village midden appeared heat treated, as did most of the debitage from the Mound B structure floor. The present study suggests that 16% of the chert debitage and 38% of the coral debitage in the village midden, and only 14% of the Mound B floor debitage, has been heat treated.

J. Rick: It is not clear that the range of variability of a chert material can be understood through the use of one control specimen. Are we assured that smoother surfaces do not occur in other natural specimens of unheated chert?

Author: Chert variability is a critical concern, and the use of several different control specimens would have better demonstrated the variability present in Florida cherts, and may have improved the study. Based on the results of many experiments conducted at the WSU Laboratory of Lithic Technology that employed a variety of lithic materials from Florida and elsewhere, I felt that the sample I chose adequately characterized the McKeithen materials. If additional controls had been used, I suspect that fewer specimens would have been relegated to the "probably heat treated" and "probably raw" categories.

S.Z. Lewin: I question the generalization of the observation that the degree of surface roughness at 2500X magnification is diagnostic of the occurrence of heat treatment and may not be a function of the inherent microgeneity of the rock itself.

Author: Since different cherts are affected differently by heating, there is variation in their appearance in the SEM; however, when it is evident that the observed fracture divides rather than bypasses crystals, the researcher is relatively safe in positing heat treatment for that specimen when it is compared to a control specimen of the same material, as was done in this study. Direct experimentation and SEM analysis of archaeological materials removes much of the ambiguity present in studies comparing control and archaeological specimens.

R.T. Matheny: Would not the term "anneal", meaning to heat and cool to achieve softening and to make less brittle, be more appropriate than "heat treat"? Annealing applies equally to glass treatment and is a regular term used in that industry. Annealing in metals is used the same way.
Author: The term "anneal" has been used to describe purposeful heating of lithic materials (Shippee 1963), but as Mandeville (1973) points out, such heating does not really result in a thorough softening of the material. Also, since heating reduces tensile strength, lithic materials may become more brittle. This is especially apparent when use-wear on heat treated and non-heat treated utilized flakes is compared (Towner 1984).

J. Rick: Reference is made to difficulty in successfully heat treating thin pieces of chert. I am curious about this, since I generally have had more trouble heat treating thick items than thin ones. Could it be that the author's experimental conditions caused thin flakes to heat faster, causing greater incidence of heat fracture?
Author: In a number of experiments like this one in which specimens were heated in an oxidizing furnace to between 250° C and 350° C for 8 to 12 hours and then allowed to cool gradually, thin specimens, especially ones less than 0.5 cm thick, often are unaffected rather than damaged by heating. This may result because very thin specimens have less interstitial water to lose during heat treatment than do larger specimens (J. Jeffrey Flenniken, personal communication 1985).

J. Rick: Examination of the internal faces of the flakes removed from artifacts strongly suggests that heat treatment is a permanent change, rather than a temporary one. I am not aware that this has been documented previously.
Author: The change does appear to be permanent. Flenniken and White (1983) have recently documented heat treatment of prehistoric (ca. 16,000 B.P.) artifacts from Tasmania using the SEM. The thermoluminescence studies mentioned above also suggest that heat treatment changes are permanent.

B.A. Purdy: It is true that tensile strength is reduced considerably, but I have always felt that flintknappers would alter the force of their blows accordingly; therefore, theoretically at least, higher percentages of shatter should not occur. The author's observation demonstrates, at least in this one case, that this inference is incorrect.
Author: Since there is a range of acceptable force for the detachment of specific flakes, and since the knapper must continually adjust the force of the blows to remove different amounts of mass from certain areas on the biface, it is possible that blows often exceed the minimum force necessary to remove a particular flake and therefore produce more broken flakes. It is also possible that knappers often use the same amount of force that they would use on non-heat treated flakes to remove longer flakes from heat treated materials. More broken flakes would again result.

R.T. Matheny: It seems to me that reduced tensile strength would increase the size of shatter in the form of flakes. If this is the case, then please state it or otherwise clarify.
Author: Since I am defining shatter as flakes which lack platforms, the size of shatter decreases during biface reduction because the knapper generally decreases the force of successive series of blows in an attempt to remove less mass in an increasingly controlled manner.

W.M. Hess: It would be interesting to analyze heat treated and non-heat treated specimens with x-ray microanalysis to see if elemental composition is altered with heat treatment. If alterations are evident, based upon elemental composition, the use of x-ray microanalysis may help to refine the analysis of prehistoric lithic samples. It may be interesting to attempt to correlate color with elemental composition. If specimens were mounted on carbon stubs and examined uncoated or were coated with a layer of carbon to reduce charging, elemental composition could be easily determined.
Author: Elemental analyses comparing heat treated and non-heat treated chert are presently being conducted by Donald Howes, J. Jeffrey Flenniken, and Roy Filby of Washington State University. Their neutron activation analysis does show that heat treatment causes an elemental change. Their findings will be published in the near future.

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