THERMAL ACTIVATION CHARACTERISTICS AND THERMOLUMINESCENCE OF CHERT FROM THE RED WING, ONTARIO REGION, AND ITS PUTATIVE HEAT TREATMENT IN PREHISTORY

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Abstract: One of the still-outstanding questions in New World archaeology is whether prehistoric toolmakers here heat-treated chert raw material prior to the manufacture of stone tools, as had been demonstrated for a number of cultural loci in the Old World. This question is of particular relevance to our understanding of the technological behaviour of the Early Paleoindian people of the Parkhill complex, which has been dated between 10,400 and 11,300 ^{14}C years ago. To address it, we studied chert samples from in situ geological contexts, from reworked contexts such as glacier-plucked surface scatters or creek gravels, and from a nearby archaeological context. The samples from archaeological contexts have been flaked in antiquity, subsequently buried, and recovered during an archaeological excavation.

We compared the thermoluminescence (TL) properties of these unknowns with those of experimentally annealed cherts. The control samples, collected and flaked from the lowest of four chert layers at the Fossil Hill Formation outcrop, were annealed for four hours at 300°C, 400°C, and 600°C. Our investigation focused on the TL sensitivity of the 100°C TL peak, which is not present in natural TL but is easily observed by prompt TL following beta or gamma dose irradiation. This peak undergoes a greater sensitivity change than the high-temperature TL following heat treatment, therefore it may be considered a far more sensitive paleothermometer. The thermal activation characteristic (TAC) of the cherts was also examined. In addition, we studied the natural TL and dose response of the high-temperature TL of the cherts, and their response to illumination by natural light.

On the basis of these investigations we conclude that prior heat treatment is readily detectable in Red Wing chert, however that it has not taken place in the archaeological material we examined. This conclusion is confirmed by the excessive apparent ages of the archaeological cherts, which are an order of magnitude higher than any reasonable archaeological estimates for the presence of humans in the New World. Chert self dose rates were based on ICPMS-determined U and Th radioisotope chain concentrations, and XRF-determined K concentrations.

In addition, we found that the high-temperature TL signal in chert is sensitive to reduction by exposure to natural light. This may possibly yield a spuriously lower TL signal in surface-collected archaeological material than in self-same geological samples collected recently, and may thus lead to an erroneous conclusion of past heat treatment. A correct procedure for the accurate detection of chert heat treatment in antiquity is proposed.

1. INTRODUCTION

The case for heat treatment to improve the flaking properties of raw chert (also frequently referred to as flint in much of the literature) in preparation for lithic tool manufacture in antiquity has been made in a convincing manner for European and Levantine archaeological deposits (for example, see Valladas and Valladas, 1987; Mercier *et al.*, 1992; Valladas, 1985; 1992; Mercier *et al.*, 2003). The situation is far less convincing in the New World, and in spite of an early report to the contrary (Pavlish and Sheppard, 1983) anecdotal evidence (M. Lamothe, personal communicat, 2003) indicates that chert material recovered from archaeological sites in Canada does not appear to have been heated. This question is perhaps of special importance in Paleoindian studies in North America because of the alleged importance of high quality toolstone and a sophisticated technology for the support of a far ranging mobility, widespread colonization and successful cultural-ecological adaptations to different environmental zones.

We therefore undertook to examine the thermoluminescence (TL) and dose response properties of natural and experimentally heat treated chert from the Fossil Hill Formation, Ontario, and to compare these to the TL of chert flakes manufactured in antiquity and recovered from the Fisher archaeological site near Parkhill, Ontario.

Fossil Hill Formation chert was a highly preferred toolstone that was transported upwards of 200 km from its geological source as part of a pattern of seasonal land-use in southwestern and south central Ontario (Storck and von Bitter, 1989; Storck and Tomenchuk, 1990; Storck, 1997). It is found within a 2.5 – 6m thick, dolomite unit of the Niagara Escarpment of Middle Silurian age, and occurs at 4 of 47 known chert outcrops in southern Ontario (**Fig. 1**). It is known, both colloquially and in the literature, by a number of names, including White; Collingwood; White Collingwood; Bruce; Amabel; Manitoulin; in addition to Fossil Hill. Its fresh appearance is white, bluish white, or grey, commonly speckled by Fe and Mn oxides; at Locality 6 it's grey, green or pink, and mottled. Once oxidized, it acquires rusty colour speckles and staining in shades of yellow, green, red; the hydrated white patina on very old pieces is also stained by buff to red colours.

The Fisher archaeological site, along with several other Paleoindian sites, is situated on a relict shoreline of glacial Lake Algonquin (**Fig. 2**), at 43 °12' N, 81°4' W, 22 km east of Port Franks, Ontario. It was a manufacturing site for fluted projectile points, and contains numerous debitage flakes, discarded broken points, and bifacial performs. Based on artifact typology of diagnostic lithic tools with their characteristic affinities to the Clovis and

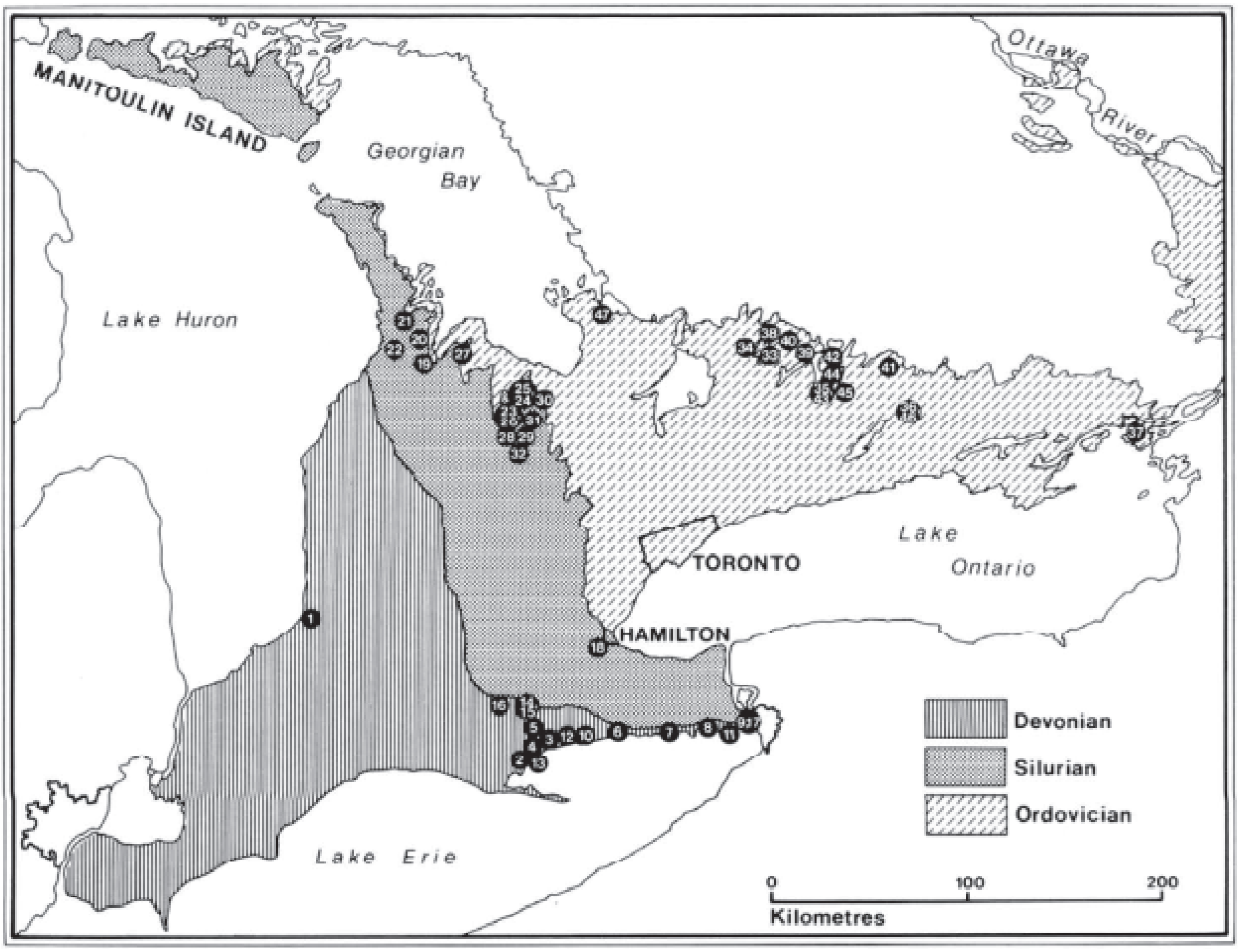

Fig. 1. Chert source locations in southern Ontario. Fossil Hill Formation outcrops are found at localities 23 to 26, centre of Figure.

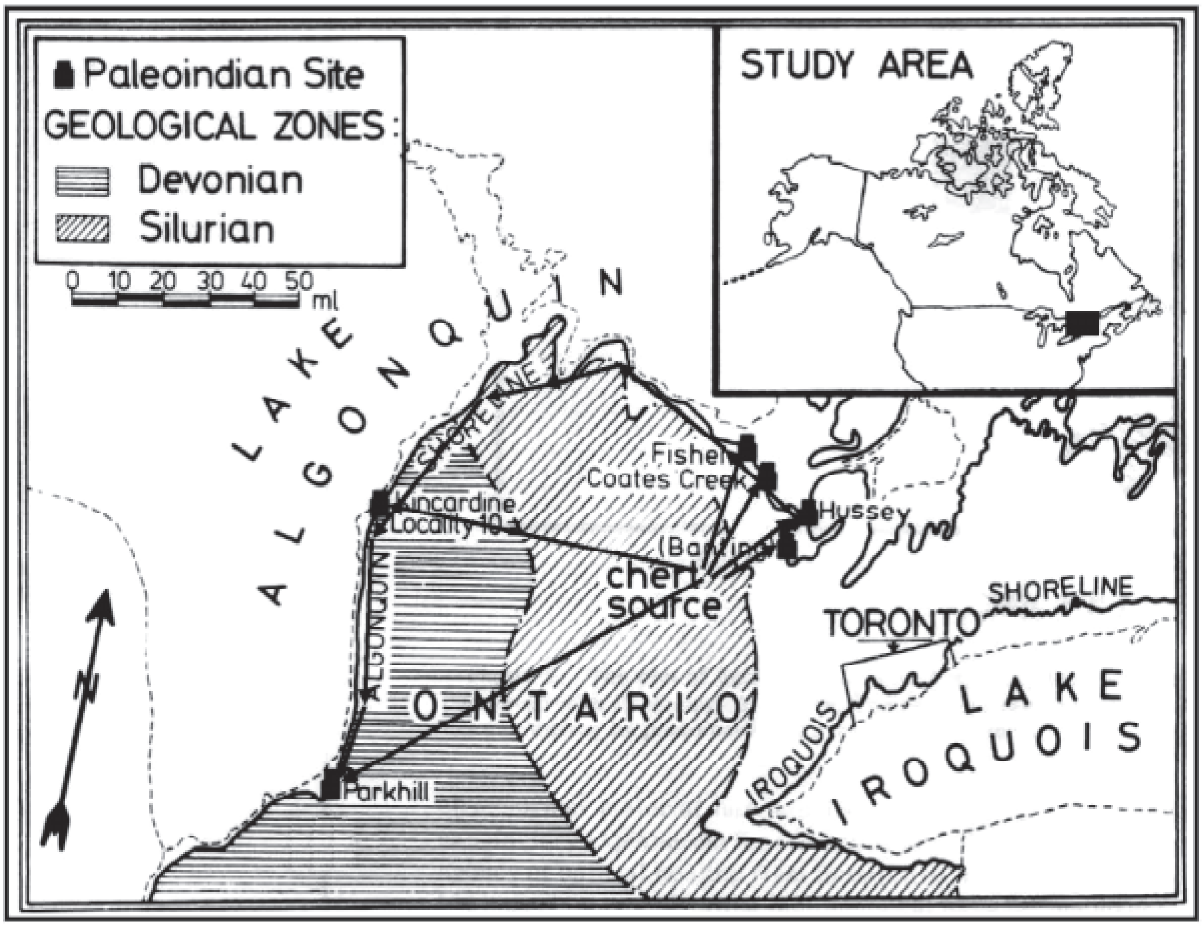

Fig. 2. *Possible means of transport of raw chert material from source area to the Fisher and other archaeological sites. A coastal route along the shoreline of paleolake Algonquin may have been more practical than a direct boreal forest route.*

Folsom fluted point typologies, the site's palynology and geomorphology, including evidence that the lowermost occupations on the Fisher site were associated with the so-called Main stage of glacial Lake Algonquin/Ardtrea, the site is dated to 10,500 years B.P. (Roosa, 1977). It was likely occupied sometime between 11,300 and 10,400 radiocarbon years B.P. (Storck, 1997).

2. METHODOLOGY

To obtain indubitably unheated control samples, the authors went to the geological source, a Fossil Hill Formation chert outcrop located near Red Wing, southern Ontario, known as Locality 1 (Storck and von Bitter 1989: 168). This same exposure is designated locality 23 in Eley and von Bitter (1989). Though there are four chert layers in the outcrop, samples were selected from the lowermost layer, immediately adjacent to the Red Wing Paleoindian quarry site. Samples of bedded chert were levered out of the outcrop using geological hammers, crowbars, and chisels, and their orientation relative to the parent rock was marked. Fossil Hill Formation outcrops are found at locations 23-26 in **Fig. 1**.

Archaeological material for this study was selected from amongst the large number of flakes in the Fisher site archaeological collection. All three archaeological flakes were excavated from the plough zone of Area B of the Fisher site, south of the city of Collingwood in the southern Georgian Bay region of Ontario. Area B is one of 19 artifact concentrations occupied by Early Paleoindian Parkhill complex people (Storck, 1997). Evidence from artifact fragments and debitage indicates that Area B was a location where a considerable amount of knapping activity occurred at an early stage(s) in the tool making process (Stewart, 1997). The three flakes all appear to be biface retouch flakes from either preforms or finished tools that were being resharpened or reconfigured.

To determine the chert's optimum heating temperature range, thermogravimetric analyses (TGA) of non-archaeological chert samples were performed. Sample preparation consisted of procuring fresh flakes by hard-hammer percussion, then snapping off their distal ends so as to avoid any contamination that may have been left behind by the hammerstone on the striking platform. These flake distal ends were hand-crushed in an agate mortar, followed by a finer grind in an Al-O mortar. Microscopic examination revealed no metallic contamination at this point. The ground sample was exposed to a heating rate of 5°C min^{-1} under nitrogen, in a Perkin-Elmer TGA 7 thermogravimetric analyzer calibrated with the magnetic transitions of Nicoseal and Perkalloy, in the Chemistry Dept. of the Univ. of Toronto. To ensure reproducibility, each measurement was carried out in duplicate. The results indicate an optimum heating temperature of between 500°C and 600°C, with a water loss peak occurring not much below the latter.

Annealing of the cherts was performed under air, using a 91 cm long, three-zone Lindberg Pyrolysis Oven Model No. 55035 with a 4.1 cm internal diameter, and Thermocraft control system Model 3D1-50-115 (UP27), with K-type thermocouples and independent oven temperature control. Chert samples were heated in quartz pyrolysis tubes, 91cm long, with a 2.54 cm external diameter. Temperature was ramped to the desired setpoint at 60°C/hr, and the setpoint was maintained for 4 hrs. Setpoints of 300°C, 400°C, 600°C, and 900°C were used, with a fresh aliquot for each anneal.

3. THERMOLUMINESCENCE AND THERMAL ACTIVATION CHARACTERISTICS

10 cherts were subjected to TL and TAC analysis. With the exception of the experimentally annealed cherts, samples for TL were prepared from as-found pieces. These were: 2 *in situ* geological samples removed from the Fossil Hill Formation outcrop; 2 field samples from local secondary geological contexts (glacially or erosionally reworked); 3 archaeological flakes from the Fisher archaeological site, thus Paleoindian in age. The 3 experimental samples consisted of aliquots of a natural *in situ* geological sample collected as described above, and annealed as described above for 4 hrs at 300°C, 400°C, and 600°C.

At Dalhousie, all handling of the flints was done under low level, dark orange lighting. Because all samples submitted had been handled under ordinary light prior to arrival at Dalhousie, an effort was made to remove the light-exposed surface of each flake. The outer surfaces were removed by grinding with a corundum dremel bit. Since the flakes were much too thin to remove 2 mm from each surface, we removed 10-20% of each flake's mass. This may not have removed all the material affected by daylight, but as the flakes appear to be quite opaque, we feel that it minimized the proportion of the light-exposed material with respect to the unexposed inner portion.

Each flake was washed and dried, gently hand-crushed in a 10-ton hydraulic press, and sieved to obtain 90-180 mmm grains. This fraction was etched for 10 min in 10% HCl, washed and dried. Grains were deposited on 1-cm Al disks, using masses of 8-10 mg per disk. TAC analysis to 650°C was performed on stainless steel disks.

Irradiations were performed using ^{90}Sr beta irradiation, at 1.05 Gy/min. TL glow curves were acquired in a dry N_2 atmosphere, using a heating rate of 3°/s, with an end point of 500°C. To remove the contributions due to blackbody radiation at high temperature, reheats were subtracted from each glow. Blue-violet TL emission was detected through Schott BG39 and Kopp 7-59 glass filters.

4. RESULTS AND DISCUSSION

The first glow natural TL curves of *in situ* (geological outcrop) and secondary context field samples, and of the archaeological flakes, are shown in **Figs 3 and 4A**, respectively. In both cases, the cherts show that the range of natural TL intensities can vary by a factor of two. We also observed that a reduction in the natural TL intensity of a geological chert is observable after an exposure to as little as 1 h of ordinary daylight, and that a significant reduction is obtained after a 10 h exposure (**Fig. 4B**). Thus, one may expect that archaeologically manufactured flakes which tend to be thin and therefore readily penetrated by sunlight, might show an apparent TL signal which is significantly lower than that of unexposed geological material from the same source. Simple TL intensity differences are thus an insufficient criterion on which to base a postulate whether a chert has or has not been heated in antiquity.

Fig. 5 permits a comparison of the natural TL of two archaeological cherts with the residual TL of two cherts which had been experimentally annealed at 300°C and 400°C, and with second-glow TL after a regeneration dose of 15 Gy. It is apparent that even the least severe of our annealing treatments (4 h at 300°C) is sufficient to erase the 340°C TL peak and results in only a residual shoulder on the high-temperature side of this peak (the fact that this is a remnant of a heat treatment is readily deduced on the basis of the shift of this little peak to a higher temperature that the peaks of first glow natural TL of chert). After the application of a regeneration dose of 15 Gy to an annealed chert, the 340°C TL peak is recovered at its expected position. We chose 15 Gy as the appropriate regeneration dose on the basis of a literature review of the relevant radioisotope concentrations in chert, and confirmed our estimates by geochemical analyses of our chert samples (**Table 1**). Based on the measured values,

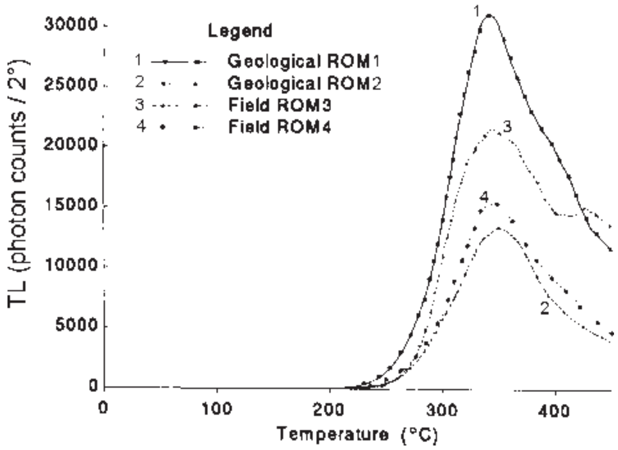

Fig. 3. Natural TL of cherts from geological sources only. Each glow curve in this and subsequent graphs is an average of two measurements.

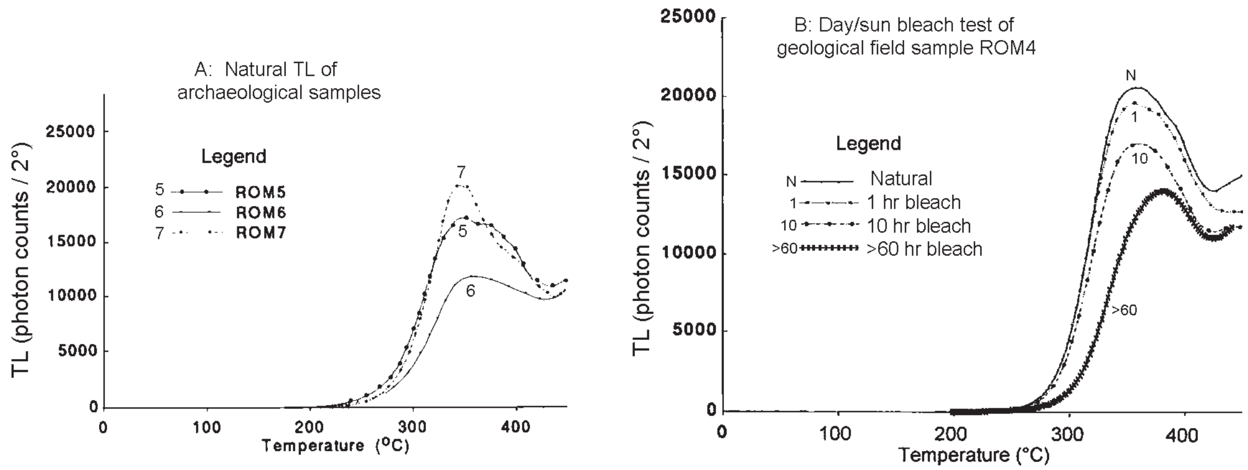

Fig. 4. A) Natural TL of chert flakes from the Fisher archaeological site. The TL intensities of these glow curves are approximately similar to those of the geological cherts. B) Bleaching of the natural TL of a geological chert by ordinary daylight (45°N, Halifax, NS). Light sensitivity of the TL signal is apparent after only 1 hr of exposure. Each glow curve is an average of 4 measurements.

Table 1. *Radioisotope concentrations for selected chert samples, and self dose rates based on the quoted values.*

Sample	K	Rb	Th	U		Self dose rate, (Gy/ka)	
	(%)	(ppm)	(ppm)	(ppm)	self alpha	self beta	self gamma
ROM 2	0.054	1.000	0.244	0.320	0.161	0.099	0.062
ROM 3	0.063	0.585	0.190	0.373	0.177	0.113	0.067
ROM 3	0.063	0.601	0.191	0.339	0.163	0.108	0.063
ROM 6	0.054	0.529	-	0.345	0.172	0.103	0.065
ROM 6	0.054	0.367	0.256	0.351	0.175	0.104	0.066
ROM 7	0.046	0.566	0.407	0.637	0.311	0.143	0.104
ROM 7	0.046	0.579	0.392	0.674	0.324	0.148	0.107

Notes:

1. *Outcrop* dose rate: self alpha+beta+gamma + cosmic. Cosmic dose rate ≤ 0.16 Gy/ka for burial depths ≥ 2 m. Accrued dose over 10 ka is 4.8 to 7.3 Gy, based on above values.
2. *Archaeological* dose rate: self alpha+beta + environmental gamma + cosmic. Environmental gamma dose rate = 1 Gy/ka for a low radioisotope, limestone/dolostone regolith of 1% K, 8.5 ppm Th, 2.5 ppm U. Cosmic dose rate is 0.204 Gy/ka for burial depths of ~0.2 m. Accrued dose over 10 ka is 14.6 to 16.7 Gy.
3. *Excluding variations in moisture content, the standard deviation associated with dose rates based on the above values is <5% of quoted value. A soil moisture content variation of 10% would increase the standard error in the calculated dose rates to ~12%.*

we calculated that an *outcrop* chert bed overlain by = 2 m of bedrock absorbs a dose of 4.8 to 7.3 Gy during a period of 10,000 years. On the other hand, a fist-sized chert fragment which might have been naturally or anthropogenically deposited in a soil such as that of an archaeological site, would be exposed to a higher flux of gamma and cosmic radiation, due to the fact that radioisotope concentrations of soils are much higher than those of lithic chert, and the cosmic dose rate decreases exponentially with burial depth. A chert pebble in a secondary context would therefore absorb a dose of 14.6 to 16.7 Gy during the same 10,000 years. Cobbles or larger blocks of chert erosionally dislodged from the outcrop would absorb intermediate doses (7 to 15 Gy) during the same period. Thus, 15 Gy represents the dose which would have been accrued by an archaeological chert if it had been deposited at the Fisher site by Paleoindians 10,000 years ago. In the case of an aboriginally heat treated chert, the 15 Gy dose would have been the total accrued dose since the last heating event, thus it would result in a TL glow curve of very low intensity (curve 5 in **Fig. 5**; **Fig. 7**; column C

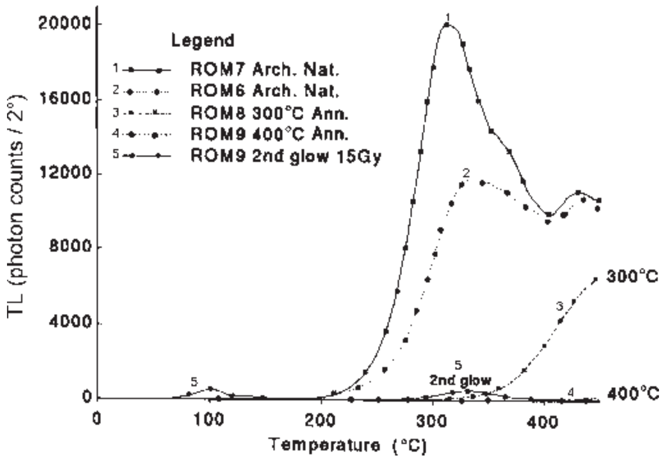

Fig. 5. Reduction of the natural TL of geological cherts by annealing for 4 hrs at 300°C and 400°C. TL glow curves of two chert flakes from the Fisher archaeological site (1,2) are included for comparison. For the sake of clarity, the TL glow curve of chert annealed at 600°C is not shown; as expected, it was indistinguishable from background.

of **Table 2**). If the 15 Gy was absorbed by a previously unheated or naturally bleached chert, the 10 ka Holocene dose would be negligible in comparison with the natural or residual geological dose (**Fig. 6**; column B compared with column A in **Table 2**).

On the basis of their natural TL levels, the N+15 Gy TL, and the regenerated TL of the 340°C peak after a second dose of 15 Gy (**Table 2**) we determined that none of the geological or archaeological cherts analyzed in this study have been previously heated. A comparison of the regenerated and natural TL intensities permits a first order estimate of the apparent minimum "age" of a chert to be made. In all geological and archaeological cherts, the minimum apparent "ages", based on a ratio of 2nd/1st glow TL, are far in excess of what is accepted about the timing of human arrival in North America. Because the ratio assumes linear growth, it provides only a lower bound to dose saturation. We also caution that such a discrimination should not be practiced in the Old World, where deliberate use of fire by hominids might pre-date 300 ka (Weiner *et al.*, 1998). In Ontario however, which was fully glaciated and could not have been occupied by humans prior to 14,000 calendar years ago, the first:second-glow TL ratio provides a rapid discriminatory test. An apparent "age" of 20 ka is produced by the ratio for chert 8, due to the previously-mentioned residual shoulder left by the 300°C annealing. However, even this value is in excess of what can reasonably be accepted as the occupation of a Paleoindian site in Ontario, and, at any rate, the temperature position of the residual peak gives it away.

Table 2 also shows that cherts exposed to prolonged heating at 400°C or higher temperatures acquire a substantial sensitization of both the 100°C and 340°C TL peaks. To confirm the sensitization, we examined the thermal activation characteristic (Valladas, 1983) of the 100°C peak in the cherts (**Fig. 7**). Most cherts (geological and

Table 2. *Intensities of the high temperature and 100°C TL peaks, for each of the cherts studied.*

	A:	B:	C:	D:	E:
ROM chert sample number	N 1st glow, 340°C pk	N+15Gy 1st glow, 340°C pk	15 Gy 2nd glow, 340°C pk	Apparent natural "age", (ka)	15 Gy 1st glow, 100°C pk
1: Outcrop geological	32,000	32,200	280	1,140	240
2: Outcrop geological	15,000	15,500	80	1,875	125
3: Field geological	13,500	12,000	110	1,230	230
4: Field geological	21,500	23,000	430	500	160
5: Archaeological	17,500	17,500	420	420	240
6: Archaeological	12,000	12,500	500	240	150
7: Archaeological	20,200	20,300	280	720	160
8: Annealed 4 hrs @ 300°C	500	950	240	20	230
9: Annealed 4 hrs @ 400°C	0	500	530	0	500
10: Annealed 4 hrs @ 600°C	0	1400	700	0	780

Notes:
1. *Each value is the average of two measurements.*
2. *Since flint yields a highly reproducible TL signal, the approximate error associated with the above data is ~5% of value quoted.*

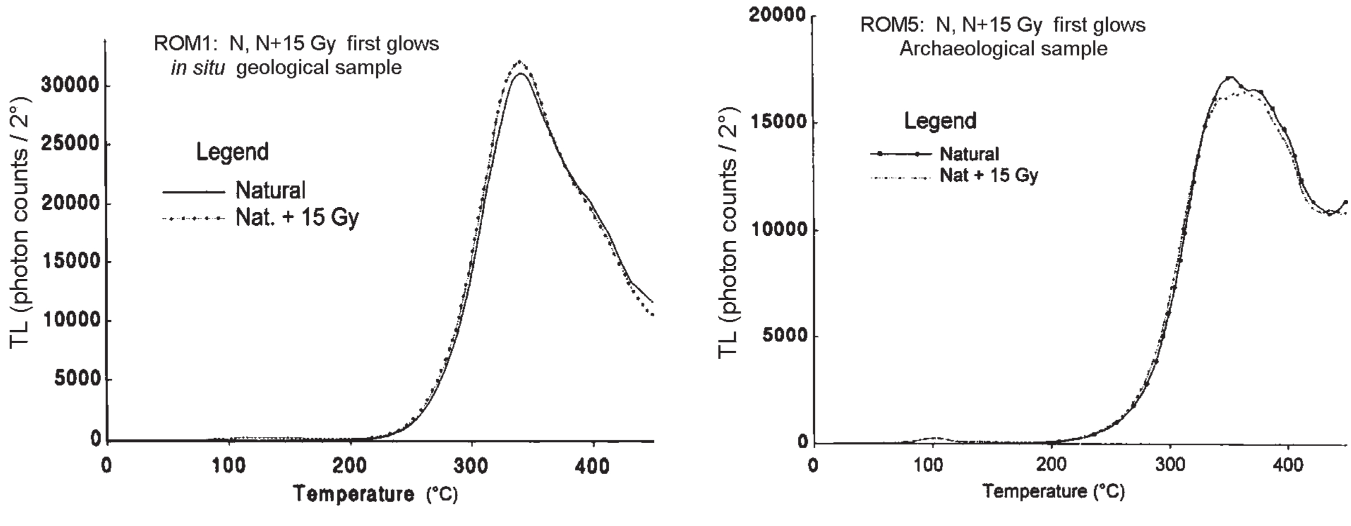

Fig. 6. *Natural and N+15 Gy glow curves of geological and archaeological chert samples. It is apparent that the added dose has a negligible effect on the natural TL levels.*

archaeological) yielded essentially identical TAC curves, thus confirming that the thermal history of the archaeological cherts does not differ from that of the geological samples. The TAC of the chert annealed at 300°C also does not differ from those of untreated cherts. However, as expected, the TAC's of the cherts which had been annealed at higher temperatures (400°C and 600°C) are elevated. Finally, we observe that sample 6 (ROM6, archaeological) yielded a TAC which is dramatically different from the others'. It is possible that this flake is composed of chert not from the Fossil Hill Formation, although it appears to be geochemically similar (**Table 1**).

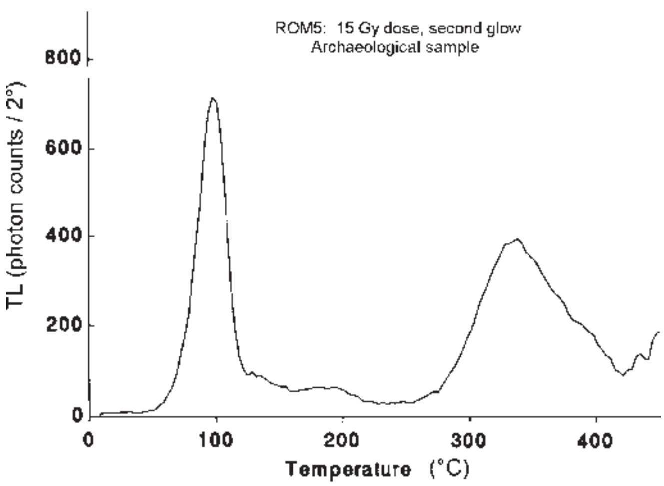

*Fig. 7. Typical second glow TL of the geological and archaeological cherts. Experimentally annealed cherts (400°C and 600°C) yielded slightly higher glow curves: see column C of **Table 2**.*

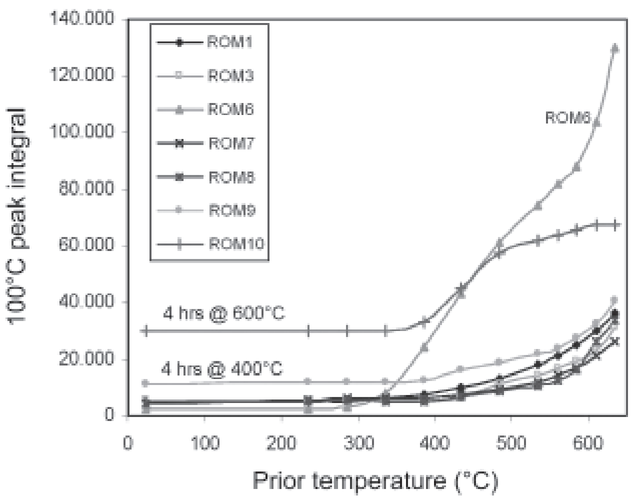

Fig. 8. Thermal activation characteristics of geological, archaeological, and annealed cherts. The TAC's of cherts ROM2, 4, 5 were indistinguishable from those of ROM1, 3, and 7, and are thus omitted for the sake of clarity. Only the experimentally annealed cherts show elevated initial TAC values, and a sensitivity increase. The TAC of ROM6 is clearly distinct from all others. This chert flake from the Fisher site, is likely from a different geological source than the Fossil Hill Formation (for example, the Annabel Formation, which is visually very similar), and thus shows different TL properties.

5. CONCLUSION

1. Even a relatively low-temperature annealing treatment, i.e., 4 h at 300°C, is sufficient to remove the very intense natural 340°C TL peak of chert. None of the three archaeological cherts examined here have experienced even this kind of heat treatment, let alone a more severe heating. Thus, we conclude that there is no evidence for deliberate heat treatment of the archaeological cherts.
2. A 15 Gy dose, appropriate to a 10 ka archaeological site, yields second glow TL which is <5% of the natural TL in all geological and archaeological samples. Conversely, an archaeologically-appropriate dose rate of 1.5 Gy/ka applied to the natural TL results in an unreasonably high minimum apparent TL age for all chert samples.
3. Instead of heating, the observed differences in the natural TL of outcrop, field, and archaeological samples may be explained by natural variations in the TL sensitivity, and by the reduction of the natural TL by exposure to daylight and sunlight. The latter is especially relevant in archaeological contexts, where small, thin manufactured flakes were likely left lying on the surface until buried by natural processes. We anticipate that exposure to natural, full spectrum unrestricted daylight and sunlight during warmer seasons would reduce the natural TL of manufactured flakes even more quickly than observed in our test, given that mostly indirect natural light was available for the experiment.

Our conclusion that Early Paleoindians of the Parkhill complex did not anneal Fossil Hill chert during tool manufacture is directly contradictory to Pavilish and Sheppard (1983) who reported that manufactured flakes from the Parkhill site in southwestern Ontario (which gives the Parkhill complex its name) that Early Paleoindian people there did, in fact, perform heat treatment on chert. After reviewing their methodology and data, we feel that their conclusions are unsupportable for the following reasons. Firstly, their TL analyses were not quantified with respect to a dose which is expected to accrue in a flake during burial for 10 ka in an archaeological soil. Secondly, a negligible reduction of a natural chert's TL signal due to light exposure was assumed, an assumption which we have invalidated here. Since our study verified the lack of heat treatment in only three archaeological samples, we recommend a verification study of further quantitative TL analyses on a much larger cohort of archaeological cherts.

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