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THERMOLUMINESCENCE DATING OF SURFACE LITHIC ARTEFACTS FROM THE CHACABUCO VALLEY, CHILEAN PATAGONIA*

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This work presents several thermoluminescence (TL) dates of six archaeological sites located in the Andean valley of the Chacabuco River, central Patagonia. We discuss the advantages of this method to assess the chronology of human occupations, employing samples of lithic artefacts with evidence of fire exposure from surface contexts in the area. We compare the results with stratigraphic archaeological dating by ^{14}C. We discuss these results in the context of previously formulated hypotheses on the past behaviour of the human population in this area.

KEYWORDS: THERMOLUMINESCENCE, LITHIC ARTEFACTS, SURFACE RECORD, HUNTER–GATHERERS, PATAGONIA

INTRODUCTION

'I fell into a burnin' ring of fire . . .'
Johnny Cash

Although luminescence techniques are experiencing a vigorous development in the dating of sediments, monuments and other materials, this is mostly in the form of OSL (optically stimulated luminescence). Thermoluminescence (TL) has been principally focused on the dating of ceramics (e.g., Aitken 1985; Roberts 1998; Wintle 2008). This has also been the case for Chilean archaeological studies (e.g., Castro *et al*. 1979; Concha *et al*. 1980; Román *et al*. 1983; Berenguer *et al*. 1986; Deza and Román 1986; Planella *et al*. 1991; Falabella *et al*. 1993). In principle, however, it can be used with any material that contains quartz or feldspar previously heated to near 500°C, not only fired clay, but also bones (e.g., Jasinka and Niewiadomsky 1970; Chapman *et al*. 1979; Deza and Román 1991) or hearth quartz-containing stones (see Wintle 2008). This application has been extensively demonstrated in the dating of subsurface heated flints in Europe (Wintle and Aitken 1977; Valladas 1978; Huxtable 1981; among many others),

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the Middle East (e.g., Shea 2001) and other parts of the world (e.g., Richter 2007; Wintle 2008), since ^{14}C dating does not cover most of the Palaeolithic. Nevertheless, applications of the technique to surface materials are still relatively rare (e.g., Wilhelmsen and Feathers 2003).

Dating of rocks is, in fact, the only way to make this method relevant to hunter–gatherer archaeology. This is particularly important in our area, where all prehistoric peoples were hunter–gatherers and there were no sedentary-agricultural groups, until the arrival of Europeans during the 20th century.

In Chile, Román and Jackson (1998) have employed this kind of approach to date hearth rocks from deep layers of archaic shell middens on the northern coast, with results consistent with ^{14}C. Thermoluminescence (TL) dating has been carried out in the same region (Aisén; about 1700 km away) where we work (Mena and Lucero 2004; Velásquez *et al*. 2007), but it has only been applied to superficial ceramic fragments. Here, we report an experience in dating the scarce *surface-recovered* heated lithic artefacts.

The first archaeological note from the Chacabuco Valley (Fig. 1) was provided by F. Bate (1970), with a short reference to rock art (a red-painted negative hand) at Alero Gianella (RCh-1) in its middle course. For decades, no further archaeological studies were attempted, while in Argentina, systematic archaeological research with stratigraphic work was well under way (e.g., Gradín *et al*. 1979). The best known example is the Río Pinturas area, with sites such as Alero Cárdenas, Cueva de las Manos (a UNESCO site) and Cueva Grande del Arroyo Feo, which presented a sequence with nine dates between 9410 ± 70 and 7280 ± 60 BP for its first temporal block; and six from 6000 ± 60 to 3450 ± 110 BP for the second (Gradín *et al*. 1987). Obviously, since more research has now been undertaken (e.g., Aschero *et al*. 1992; Goñi 2000–2; Mengoni *et al*. 2009), the uncritical projection of this sequence to the whole region is in question. Thus, our research aims to contribute further to this discussion.

Our Pacific drainage (Chacabuco Valley) was first studied in this fashion from the late 1980s (Mena 1986; Mena and Jackson 1991), and has since been the focus of several archaeological research undertakings. Among them are a bi-national project (Mena *et al*. 2000), with excavations at small sites in the middle valley and analyses focused on lithic problems (cf., Méndez and Blanco 2001); a large distributional study comparing three valleys (FONDECYT 1990159: Mena and Lucero 2004; Quemada 2008); and, recently, field research promoted by the Centro de Investigación en Ecosistemas de la Patagonia (CIEP).

Although these previous studies have provided a fairly complete distributional perspective, the Chacabuco Valley—like most of Patagonia—lacks the straightforward cultural sequence made possible in other regions by stylistic changes in ceramics, architecture and other cultural traits. Despite the fact that some basic typological framework is often used (Bird 1938; Menghin 1952; Sade 2008), they should be treated as hypotheses that must be evaluated in each case, as they have often proved to be misleading (Borrero 1994–5). In fact, as said above, human occupation in the region is represented only by hunter–gatherer societies until recent historical times (*c*. AD 1880). Thus, the archaeological contexts that they produced reveal little differences and are largely constrained by situational conditions, such as site function or the availability of raw materials and other resources. The main point is that, although these societies undoubtedly changed through time, these changes are not easy to observe archaeologically, and a good chronological framework with which to do so is needed. In this endeavour, all dating methodologies should be considered. Radiocarbon dating has provided some basis for evaluation, but the number of excavations is still limited: meanwhile, surface materials can be a rich source of information that can only be interpreted through methods such as thermoluminescence (TL) dating.

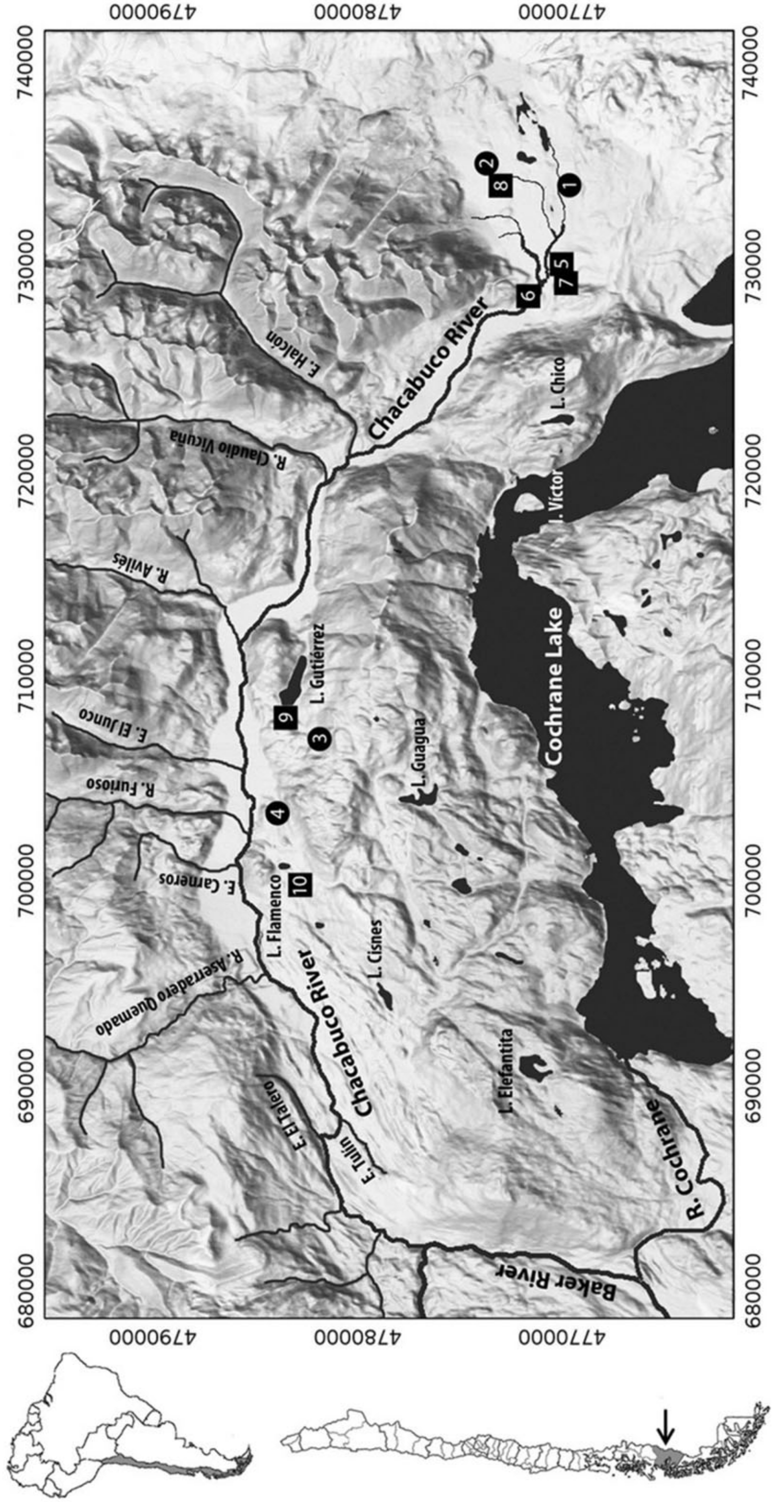

Figure 1 *A map of the Chacabuco Valley, showing the sites discussed in the text. 1, Alero Entrada Baker; 2, Los Carneros; 3, Alero Gianella; 4, Alero Blanco; 5, Upper Chacabuco 7S/3; 6, Upper Chacabuco 7S/1; 7, Upper Chacabuco 8S/2; 8, Upper Chacabuco 14N/1; 9, Middle Chacabuco 11/18; 10, Middle Chacabuco 18/9. Round markers have ^{14}C dates, while squares show the places dated by TL in this work. The arrow shows the approximate location of the main map. The map coordinates are in WGS84 datum.*

In summary, the greater part of the known Chacabuco Valley archaeological record is comprised by open-air lithic scatterings that are difficult to detect (Fig. 2) and to date, unless we rest upon the scarce finds with typologically diagnostic tools that—as we have stated—may be ambiguous.

The exceptions to this are a few sheltered sites where radiocarbon dating has proved feasible, as they present charcoal and bone that can be dated. These sites provide us with a small base of ^{14}C dates, consisting of four from Alero Entrada Baker (Mena and Jackson 1991; Méndez and Velásquez 2005) and one for Alero Gianella (Fig. 3; Fuentes-Mucherl *et al*. 2012). These can be used as general chronological references. For this work, we added one date for Cueva Los Carneros (Upper Chacabuco) and another for Alero Blanco (Middle Chacabuco), giving seven radiocarbon dates in total (see the Discussion section).

In the upper parts of the valley ('Entrada Baker'), over a hundred surface lithic assemblages have been recorded. It is assumed that they correspond to the traces of summer logistical forays from camps further east and in low basins (Mena and Jackson 1991). Examples of this may be the Ghio or Salitroso lakes in present-day Argentine territory, where large '*chenque*' cemeteries have been recorded and dated between 1000 and 350 BP (Goñi and Barrientos 2000; Goñi *et al*. 2004). In the absence of direct absolute dates, however, this remains a low-power hypothesis.

From the point of view of lithic studies, we have been able to characterize an important part of the assemblages as being composed of several raw materials, of which a large part is constituted by 'Pampa del Asador' obsidian and 'Posadas' andesite (Méndez and Blanco 2001; Méndez *et al*. 2003, 2007). These materials have known provenances in Parque Nacional Perito Moreno (Stern 1999) and in the vicinity of Cerro de Los Indios, near Lake Posadas (Guráieb 2004), both located far away (~100 km) in what is today Argentina. In the Upper Chacabuco, 43% of the sample collected consists of these exotic materials, while the rest is represented by a variety of fine-grained siliceous and basaltic rocks. Because the former rocks seem to have

Figure 2 *The middle course of the Chacabuco River, where one of the open-air TL dated sites (MCh 18/9) is located: Laguna Flamenco, south shore.*

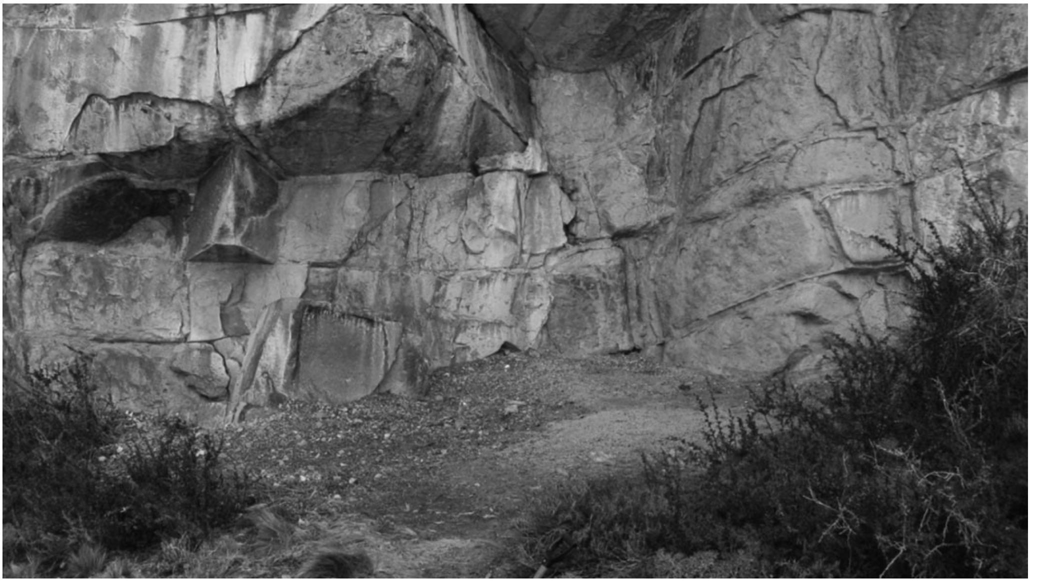

Figure 3 *The Alero Gianella Rockshelter, Middle Chacabuco, from where one of the ^{14}C dates was obtained.*

superior knapping quality, and they do not exhibit pot lids, it is possible that the samples of the last group (siliceous and basaltic rocks) employed in this work could be leftovers of pieces that were specially submitted to heat treatment to improve their fracture properties. This idea is yet to be evaluated, as there are alternative interpretations that will be further commented on in the next section.

The middle and lower parts of the Chacabuco Valley have two new radiocarbon dates that indicate occupations around the third and second millennia BP (see Fig. 6 in the Discussion section), when we have supposed that the upper portion was largely abandoned, judging from the dates and stratigraphy of Alero Entrada Baker (Mena and Jackson 1991).

The previous hypotheses should be subject to validation by alternative methods, among them the TL dating of surface lithic material, which is what we report here.

MATERIALS AND METHODS

Lithic sample selection

We analysed samples that correspond to artefacts that presented macroscopic traits indicative of thermal exposure. In particular, we looked at the presence of small pits, or 'pot lids' (e.g., Purdy 1975; Gould 1976; Miranda 2008; Frank 2011, 2012; Deal 2012), caused by the expansion of water or structures of dissimilar density inside the rock (Fig. 4). These authors and many others have found that this kind of damage requires a temperature over 450°C, making it unnecessary to resort to specific experiments or microscopic analyses. As examples, flint has been found susceptible to this kind of damage over 400°C (and especially between 600 and 800°C) by Preece *et al*. (2006); similarly, silicates are affected at about 550°C (Deal 2012), obsidian in the range of 500–600°C (Deal 2012), silcrete at over 600°C (Mercieca and Hiscock 2008), basalt starting at

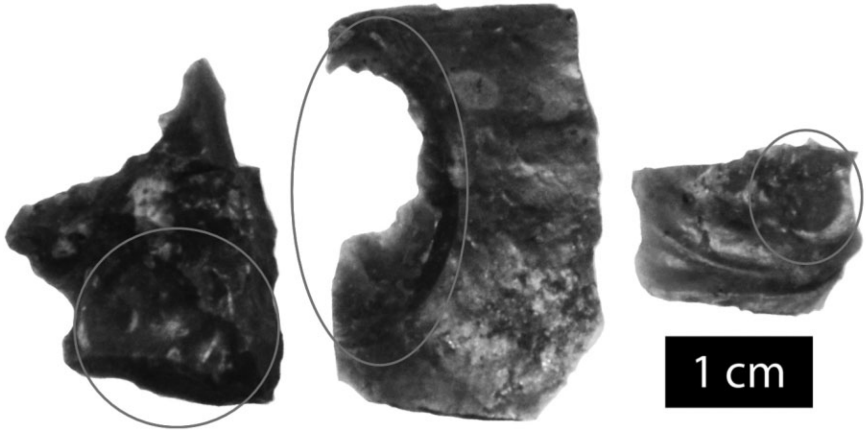

Figure 4 *Examples of pot lid damage on samples from the study area: the ovals are centred on the 'pot lids'.*

400°C (Deal 2012), mudstone at more than 500°C (Mercieca and Hiscock 2008) and LS–LF chalcedony at over 450°C (Schmidt *et al*. 2013a,b). Thus, pot lids provide unmistakable evidence of fire exposure, while signs that appear at lower temperatures (such as colour and surface alterations) are more ambiguous and harder to identify. Furthermore, the temperature needed to produce pot lids is, in almost every case in the literature, higher than the quartz trap threshold employed in TL dating (see also Richter 2007, 674).

As we have stated, it is possible that this effect may be related to intentional thermal pre-treatment to enhance the knapping quality of the rocks, but it could also be due to the simple disposal of the pieces in the fire. Another possibility is the lighting of camp-fires directly on top of previously discarded lithics. None of the pieces with this kind of damage was a finished tool; all of the analysed samples were lithic debris.

Since we could not determine by simple inspection whether or not the samples selected contained enough quartz, we first subjected them to a palaeodose reading. The eight samples analysed rendered positive results (an increased luminescence signal), regardless of whether they were sedimentary or volcanic rocks. The luminescence emission peaks were obtained at 110, 325 and 375°C as usual, for quartz grains.

The sites that yielded pieces suitable for the analysis were the following: 7S/1, 7S/3 8S/2 and 14N/1 in the Upper Chacabuco Valley; and 18/9 and 11/18 in the middle section of the valley, all about 500 m a.s.l.

Sample preparation

The lithic artefacts were fragmented by hammer strikes and the resulting interior material (fragments with cortical and exposed surfaces were discarded) was ground inside a bronze tube, by pressing with a metallic screw. Then, grains smaller than 1 mm were selected to be attacked by concentrated HCl for 72 h, with the purpose of removing potential contaminants. Before the grinding of each sample, 1–2 mm of the external layer was removed, in order to avoid the alpha contribution to the annual dose (e.g., Huxtable 1981).

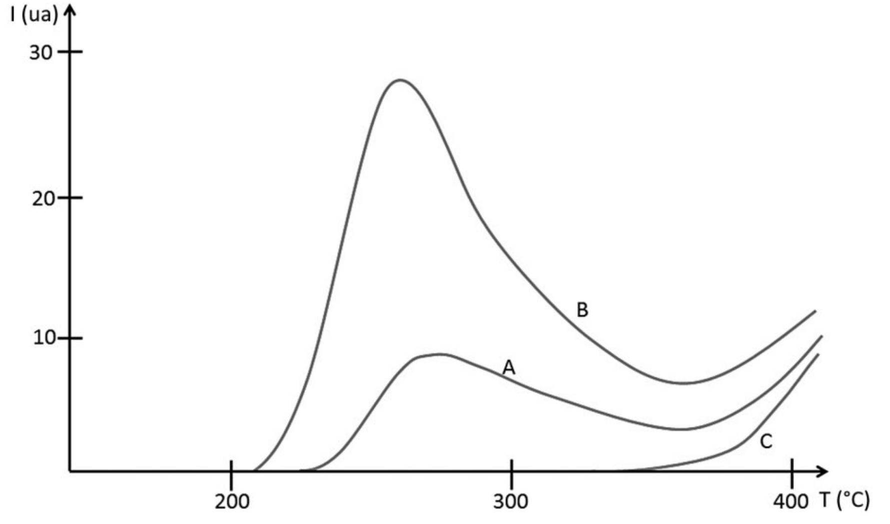

Figure 5 *An example of a TL plot from sample UCTL2053.*

Afterwards, they were treated with HF (25%) at up to 40°C for 1 h. The quartz grains that resulted from this process were subject to repeated washing with distilled water until a neutral pH was reached, and finally dried in an oven at 50°C. The quartz was sieved to obtain grains with diameters in the 75–105 μm size range.

Palaeodosage

All measurements were carried out using the equipment at the Laboratorio de Dosimetría (Physics Faculty of the Pontificia Universidad Católica de Chile), as described in earlier works (Berenguer *et al*. 1986; Ávalos and Román 1996; Román and Jackson 1998). We used a TL Harshaw 2000 AB, with a heating speed of 12°C s^{-1}, which integrates the quartz TL signals between two previously established temperatures. For the additive method and the supralinearity calculus, the chosen interval was 330–400°C, since it comprises the 375°C signal, a stable trap with a long half-life of several million years (cf., Martini *et al*. 2001). This signal is not shown on the TL curves in Figure 5. This could raise doubts about the quartz content of the samples. However, each irradiated sample gave a TL curve with a clear peak at 110°C, typical of this crystal (cf., Koul 2008; see also Göksu *et al*. 1989). Samples were irradiated using ^{90}Sr, which renders a dosage rate of 1 Gy min^{-1} with homogeneous geometry.

The equivalent *Q* dosage was determined by the plateau and additive method. For the former, we graphed the quotient (natural TL)/(natural TL + 3 Gy) in relation to temperature, as shown in Figure 5 for sample UCTL2053. In most of the studied samples, the plateau extended from 330 to 400°C, approximately. On the other hand, for the additive method, we used 2, 4 and 6 Gy as additional doses, the same being used to obtain the supralinearity of each sample.

Each sample palaeodose (*P*) corresponds to the addition of the equivalent *Q* dose and the supralinearity *I*. The equivalent *Q* dose was obtained by means of the plateau method (samples UCTL 2211, 2212, 2215, 2216, 2053 and 2054). For UCTL 2213 and 2214, the additive method was used.

Annual dose

The gamma radiation dose of the soil was calculated by thermoluminescence (TL) dosimetry using $CaSO_4$:Dy dosimeters. Four of these small discs (diameter 4 mm) were placed in plastic tubes and buried on the spot for 176 days. Dosimeters were placed on the exact location from where the analysed samples were collected. Although the latter were found lying on the surface, to avoid loss, stealing or accidental removal, the dosimeters had to be buried 10 cm below it.

The internal annual dose of the samples, on the other hand, was measured by means of similar discs introduced in the fragments obtained by grinding the lithics, which were left to stand in total darkness for 100 days.

Quartz grain sizes were considered for the annual dose calculation for each sample. Since the radiation dose is also affected by the saturation water content, we calculated it by comparing the weight of a 500 g soil sample with its dry weight using the following formula: humidity factor = 1 + 1.14[(humid weight/dry weight)] – 1) (Zimmermann 1971; see also Aitken and Xie 1990).

One of the problems regarding the surface dating of lithics is that the internal dose rate may be low. This is common for cherts and quartzites, making the cosmic dose rate a large proportion of the total dose rate and hence reducing precision. In our case, the correction of the external dosage of the ground, taking cosmic rays into consideration, was not applied because it gave a radiation dosage that was more than 20 times greater (Mackeever 1985, 278).

More recently, Richter (2007) in a work assessing methods and results for Near Eastern Palaeolithic sites, has suggested that 'The degree of influence of environmental parameters on the age result depends on the proportion of the varied parameter to the sum of all parameters . . . Of all dosimetric methods, TL on heated rock material is the least sensitive, due to the stable internal dose rate in all samples' (p. 681).

RESULTS

Table 1 shows the dates obtained through TL dating. We will discuss them in the context of the valley's prehistory in the next section.

DISCUSSION

The results indicate that the use of the thermoluminescence dating technique on surface lithic material is feasible, contributing greatly to a better understanding of the prehistoric processes in our area, as we discuss below. Hopefully, it may also contribute to the improvement of methods to study other archaeological superficial palimpsests, which occur frequently in the study of highly mobile prehistoric hunter–gatherers.

Since our experiment has a coarse-grained focus, as we try to contextualize the records across a vast area and a large span of time, with limited samples from a few sites, the results must be taken as a general guideline for the process of occupation and use of the valley by prehistoric populations. Therefore, it is not essential that materials dated on a supposedly 'contemporary' assemblage render precisely the same ages (e.g., 7S/3), as we are not interested in dealing with single events but, rather, with the general pattern of the process of human population of the valley.

Figure 6 shows the distribution of dates obtained in the Chacabuco Valley by radiocarbon/stratigraphic records and thermoluminescence/superficial *loci*. We should keep in mind the

Table 1 *Thermoluminescence (TL) dates from surface heat-damaged lithics, Upper and Middle Chacabuco Valley, Aisén*

Sample	*Valley section*	*Site*	*Q (Gy)*	*I (Gy)*	*P (Gy)*	D_{int} *(Gy yr*$^{-1}$*)*	D_{ext} *(Gy yr*$^{-1}$*)*	*D (Gy yr*$^{-1}$*)*	*BP age before 2010*	*Date*
UCTL2211	Upper	7S11	4.31 ± 0.35	0.51 ± 0.05	4.82 ± 0.40	0.12×10^{-3}	2.80×10^{-3}	2.92×10^{-3}	1650 ± 160	AD 360
UCTL2212	Upper	7S/3	7.00 ± 0.64	0.04 ± 0.02	7.04 ± 0.66	3.00×10^{-3}	2.80×10^{-3}	5.80×10^{-3}	1215 ± 120	AD 795
UCTL2213	Upper	7S/3	4.20 ± 0.33	0.13 ± 0.07	4.33 ± 0.40	0.86×10^{-3}	2.80×10^{-3}	3.66×10^{-3}	1185 ± 120	AD 825
UCTL2214	Upper	7S13	4.40 ± 0.35	0.08 ± 0.05	4.48 ± 0.40	0.22×10^{-3}	2.80×10^{-3}	3.02×10^{-3}	1465 ± 140	AD 545
UCTL2215	Upper	8S/2	6.27 ± 0.55	0.10 ± 0.05	6.37 ± 0.60	0.20×10^{-3}	3.00×10^{-3}	3.20×10^{-3}	1990 ± 200	AD 20
UCTL2216	Upper	14N/1	7.21 ± 0.50	0.51 ± 0.20	7.71 ± 0.70	0.80×10^{-3}	3.30×10^{-3}	4.10×10^{-3}	1880 ± 180	AD 130
UCTL2053	Middle	18/9	3.98 ± 0.15	0.80 ± 0.29	4.78 ± 0.29	0.68×10^{-3}	1.49×10^{-3}	2.17×10^{-3}	2200 ± 200	190 BC
UCTL2054	Middle	11/18	4.14 ± 0.21	1.00 ± 0.26	5.14 ± 0.47	1.34×10^{-3}	1.49×10^{-3}	2.83×10^{-3}	1820 ± 180	AD 190

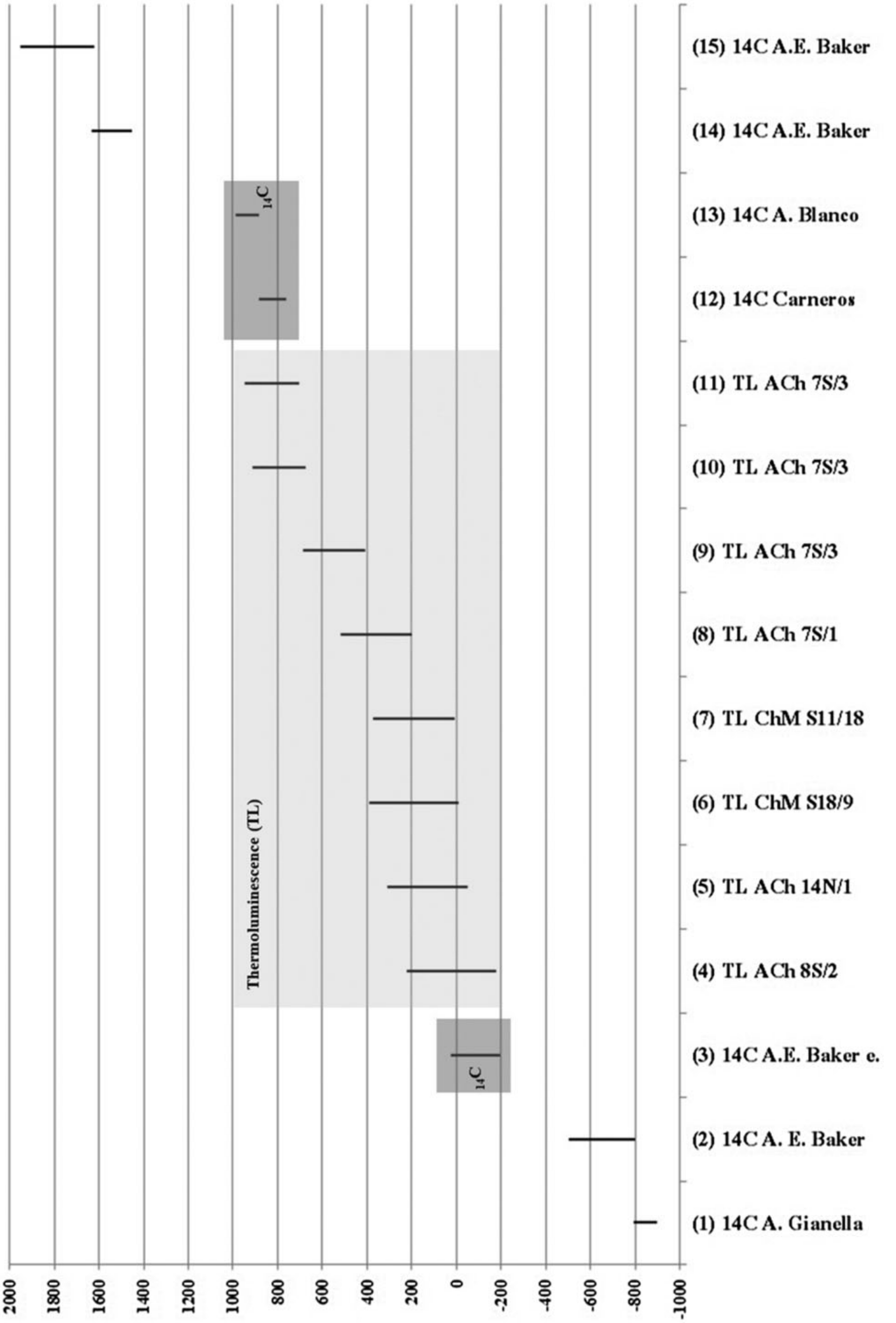

Figure 6 *The thermoluminescence and radiocarbon dates of the Chacabuco Valley.*

greater deviations of the TL dates (120–200 years) compared to those provided by the ^{14}C method, regardless of whether standard (~40–70 year) or AMS (~25 year) procedures were used. In order to standardize the timeframe to calendar years, to allow comparison with TL dates, calibration was also used: the ^{14}C dates were calibrated to calendar years (BC–AD) using Oxcal 4.1 with the SHCal04 curve.

It might have been expected, for instance, that the TL dates would be systematically younger, as they come from a non-stratigraphic context, but in fact they are not. It is noteworthy that there is little overlap between the two series, and then only in the upper and lower ranges of the TL sequence.

Also, Figure 6 reveals that open-air (TL dates) and sheltered (^{14}C dates) occupations do not necessarily coincide, and that the latter occurred before AD 1 and after AD 800. Perhaps the selection of sample material was biased towards extremes of dispersion when choosing samples to radiocarbon date from archaeological excavations. In that case, we should submit samples from the middle of the sequences to analysis. Further enquiries should attempt to recover samples from the *same levels* to be subjected to both to ^{14}C and TL dating.

Conversely, if there is no bias in the selection of ^{14}C samples, it is not clear why TL dates on open-air sites fill the hiatus indicated by ^{14}C analysis in sheltered sites so neatly. A provisional explanation may be that more favourable environmental conditions existed during the period from AD 1 to AD 800, allowing prehistoric peoples to use open-air locations more intensively during that interval.

Recent work by Mardones *et al*. (2011) conducted near the Blanco River (~150 km NNW) suggests a glacial advance prior to our TL series (*c*. 2250 BP), correlated with the millennial palynological sequence due to Moreno *et al*. (2009) near the Southern Patagonian Icefield (~370 km south). This last work proposes relations between humid phases and glacier advances at 4400–4100, 2900–1900, 1300–1100 and 570–60 BP, punctuated by openings in the woodlands, as shown by the *Nothofagus/Poaceae* ratio. If the interpretation of this phenomenon is generalizable to our study area, then our TL series fits very well into the sequence, sustaining our previous climatic explanation derived from the TL dating effort, within the 2300–1300 BP opening (Moreno *et al*. 2009, 37). Up to this point, it is fair to say that the TL dating of archaeological superficial scatterings may serve as a *proxy* for the Holocene history of the Patagonian palaeoclimate.

On a more restrictive timescale, the overall date distribution also leads us to question why we have two hiatuses not covered by the absolute dates, the first one occurring between *c*.500–200 BC and later one at AD 1000–1400. Was the valley abandoned in these periods, or is this a stochastic effect of our sampling of the valley? This question remains to be elucidated by further investigations.

It is worth noting that some surface finds in the Upper Chacabuco are somewhat older than those at the middle course of the same valley, suggesting a directionality of the peopling process of the valley. But, perhaps, soil deposition can be much faster in the forested and steeper middle regions, where soil formation seems to be more rapid and the erosive impact of the wind, milder than on upper steppes that are open, flat and largely devoid of vegetation. Thus it seems reasonable to assume that earlier materials are to be found beneath the surface at the Middle Chacabuco and that we should look for them underground.

Although preliminary, this study has accomplished our basic goal of assessing whether the TL dating of surface-recovered lithic artefacts is feasible. Since it has proven to be so, it could provide the basis for a broader research programme. Moreover, if this experiment raises archaeological, taphonomic and even palaeoenvironmental hypotheses such as those discussed above and elicits new questions, it has fulfilled an important purpose, beyond its sole methodological prospects.

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