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LUMINESCENCE DATING IN ARCHAEOLOGY: FROM ORIGINS TO OPTICAL

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Abstract—Luminescence dating has a proud history of association with archaeology, beginning almost half a century ago. The subsequent decades of research have seen a range of archaeometric applications of luminescence dating: from fired pottery and burnt flints to sediments incorporated into occupation deposits and earthen constructions. Important contributions have been made to topics as diverse as modern human origins, continental colonisations and the dating of prehistoric rock art. This paper provides an overview of these applications, with a particular focus on recent findings such as those from Tabun Cave in Israel, Diring Yuriakh in Siberia, and Jinmium in Australia. © 1998 Elsevier Science Ltd. All rights reserved

1. INTRODUCTION

> That they were repositories of the dead, has been obvious to all: but on what particular occasion constructed, was matter of doubt. (Thomas Jefferson, 1787, Query XI "Aborigines")

More than two centuries have elapsed since Thomas Jefferson contemplated the antiquity of prehistoric burial mounds in North America and became "the first scientific digger" in the history of archaeology (Wheeler, 1956, p. 58) when he dug a trench through a mound in Virginia and examined its internal strata (Jefferson, 1787). Such anthropogenic structures have only recently been investigated using luminescence methods (Feathers, 1997a,b; Saunders *et al.*, 1997), whose pedigree in archaeological research extends back to the pioneering work of Daniels *et al.* (1953). This team recognised the potential for dating *heated* artefacts using thermoluminescence (TL), an archaeometric tradition subsequently continued by Ugumori and Ikeya (1980) in the first application of *optically* stimulated luminescence (OSL) to archaeologically-relevant material ($CaCO_3$). Following these novel insights, TL and OSL techniques have been developed and tested extensively on a vast array of archaeological materials and across a wide range of archaeological issues; from human origins to human constructions, from rock shelters to rock art.

The aim of this paper is to present a broad overview of these archaeometric applications of luminescence dating, from its origins in pottery dating to the application of optical dating to archaeological sediments. The huge volume of literature on the subject precludes a truly comprehensive review, but I have attempted to illustrate the diversity of the archaeological applications by reference to many of the key papers published over the past few decades. While some of the more recent and controversial contributions are discussed at length, mention is also made of the less publicised luminescence studies that have laid many of the technical foundations for the dating method. Attention is drawn also to studies that have tackled a long-standing archaeological problem using a novel luminescence technique, or that have reported a technical development with implications beyond the specific archaeological application. Some contributions of luminescence dating to archaeologically-relevant issues (e.g. environmental change, faunal extinctions) are discussed briefly, as are a variety of potential avenues for profitable future archaeometric research.

The paper is organised into sections based loosely on archaeological "themes" or periods, rather than on luminescence dating methods (TL or OSL) or procedures (palaeodose or dose rate determinations). I have attempted to make the technical discussion in each section as self-contained as possible, but the details of specific luminescence dating techniques are given elsewhere in this special volume (Prescott and Robertson, 1997; Wintle, 1997) and in more than 20 reviews, several written specifically for archaeologists, published over the past two decades (Aitken, 1977, 1985, 1989, 1990, 1994, 1998; Berger, 1988, 1995; Duller, 1996; Feathers, 1997a; Fleming, 1979; Forman, 1989; Huntley and Lian, 1997; Lamothe *et al.*, 1984; Mejdahl, 1986; Mejdahl and Wintle, 1984; Roberts and Jones, 1994; Seeley, 1975; Singhvi and Mejdahl, 1985; Singhvi and Wagner, 1986; Wagner *et al.*, 1983; Wintle, 1980, 1987, 1993; Wintle and Huntley, 1982).

A variety of archaeological themes are pursued in this paper. The choice is idiosyncratic and biased towards those in which luminescence dating has thrown the greatest light on a major problem or period in archaeology. The theme of Australian colonisation is given particular prominence, not only because of my familiarity with the subject but also because TL and optical dating of archaeological sediments in Australia provides a striking counterpoint to TL dating of pottery and burnt flint, which has dominated luminescence studies in Europe and the Middle East. I have attempted to make this overview appealing to graduate students and professional practitioners in both archaeology and Quaternary geochronology, but I apologise to those whose work has been overlooked, misrepresented, or cited in an improper context.

2. MODERN TO MESOLITHIC

2.1. *Pots and potentates*

Artefacts provide crucial evidence of human activity, and the abundant remains of ancient pots and ceramic vessels are among the most valuable to archaeologists. Luminescence dating made its earliest forays into archaeology through the application of TL methods to heated pottery and ceramics from ancient Kingdoms, Empires, Dynasties and civilisations. Firing of pottery (to above 500°C) evicts the electrons trapped over geological time in the minerals which make up the body of the pot, thereby zeroing the luminescence "clock" which, for dating purposes, is based on electron traps in the temperature range 300–450°C (e.g. the 375°C peak in quartz). At the outset, pottery of "known" antiquity (established on the basis of pottery style, site stratigraphy, and uncalibrated radiocarbon (^{14}C) ages) was tested to verify the age determinations made using the infant TL methods (Fig. 1). Pot sherds up to 8000 years old from the Middle East (Iran, Turkey and the Levant), Britain (particularly from the Roman period), Italy, Denmark, Peru and Thailand were tried initially (Aitken *et al.*, 1963, 1964, 1968; Mazess and Zimmerman, 1966; Mejdahl, 1969; Ralph and Han, 1966; Ralph and Han, 1969; Zimmerman and Huxtable, 1969). The bleaching of TL signals by sunlight was explicitly reported for both $CaCO_3$ and sediment in the first of these trials (Aitken *et al.*, 1963), but Soviet scientists are generally credited with being the first to recognise the potential to date solar-reset sediments using TL (Morozov, 1968; Shelkoplyas, 1971; Shelkoplyas and Morozov, 1965). Not until the 1980s was optical dating of calcite and sediment attempted (Huntley *et al.*, 1985; Ugumori and Ikeya, 1980).

TL dating continued to develop during the 1960s and 1970s, largely through the efforts of the research team led by M. Aitken at Oxford University, and the technique became increasingly

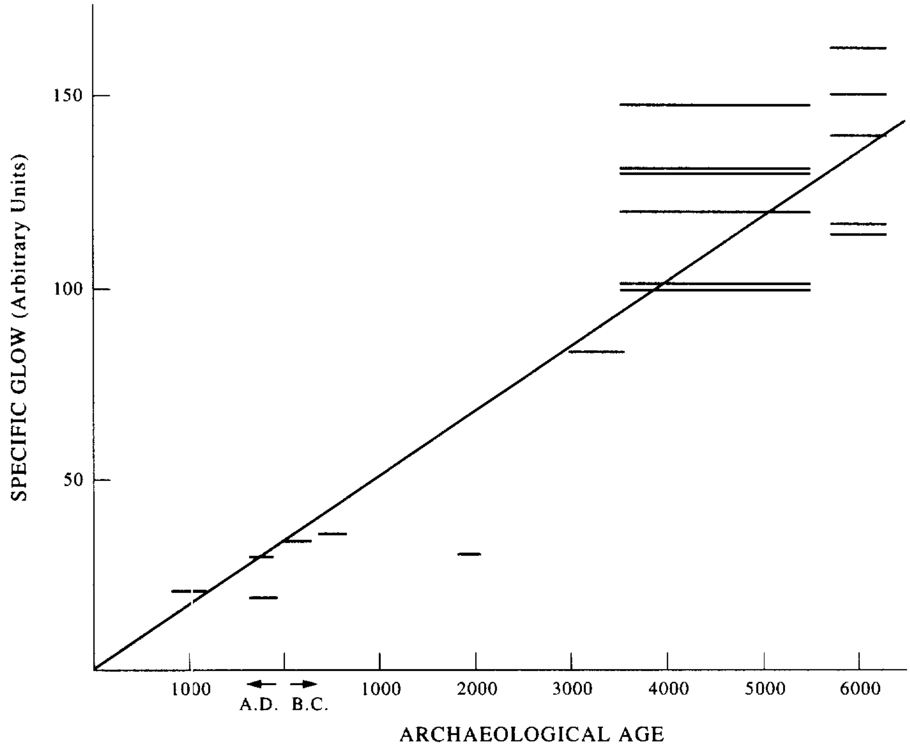

Fig. 1. An early attempt to compare the TL from 18 heated pot sherds with their known archaeological ages. The "specific glow" is defined as the natural TL (in the 350–450°C temperature region of the glow curve) divided by the product of the dose-regenerated TL and the sherd radioactivity. The specific glow is approximately proportional to age (after Aitken *et al.*, 1963).

recognised as an independent method for authenticating the age of fired pottery, ceramics and the clay cores of bronze casts (e.g. Zimmerman *et al.*, 1974). Progress was not made without the occasional stumble, such as the notorious "Glozel" controversy which threw the validity of TL dating into some doubt (see Aitken, 1985 for an historical account). But the refinement of quartz inclusion (Fleming, 1970) and fine-grain (Zimmerman, 1971) TL techniques, and the development of feldspar inclusion (Mejdahl, 1972), pre-dose (Fleming, 1973a) and zircon (Sutton and Zimmerman, 1976; Zimmerman *et al.*, 1974) dating methods, culminated in a successful series of forgery identifications, including the "Hacilar" ceramic wares from Turkey (Aitken *et al.*, 1971), and the supposedly T'ang Dynasty ceramics (Fleming, 1973b) and Hui Hsien style pottery (Fleming and Sampson, 1972) from China; additional examples are given by Fleming (1979).

As a result, TL is now used routinely in authenticity testing (Bailiff, 1994; Stoneham, 1991) and gained early service as an independent test of controversial ^{14}C age estimates. For example, TL was used to date heated materials associated with the Jomon culture in Japan, the first people in the world to make pots from burnt clay. Uncalibrated ^{14}C ages of 12,000–13,000 years for two cave sites had been hotly disputed, but were confirmed by TL dates of 12,000–14,000 years on a pot sherd (using the "subtraction" technique; Fleming and Stoneham, 1973) and four blocks of baked sandstone from a hearth (using quartz inclusions; Ichikawa and Nagatomo, 1978). At a third site, ^{14}C ages tallied with quartz inclusion TL dates on Jomon pottery from later periods (2300–7600 years BP; Ichikawa *et al.*, 1978). Other early examples of ^{14}C/TL age comparisons can be found in Sampson *et al.* (1972), Whittle and Arnaud (1975) and Zimmerman and Huxtable (1969, 1971).

2.2. *Pathfinders of the Palaeolithic*

The TL study made by Zimmerman and Huxtable (1971) of burnt clay lumps at Dolni Vestonice is notable to archaeologists in at least two important respects. First, it represents the initial attempt to extend luminescence dating into the Palaeolithic period before the Last Glacial Maximum: a crucial time period in terms of human evolution, artistic development and stone tool manufacture, in Europe as elsewhere. This push into the Palaeolithic, and beyond the limit of ^{14}C dating, became the major thrust of TL dating over the next decade, with the development and application of methods to date burnt flints (Göksu *et al.*, 1974; Wintle and Aitken, 1977) and baked sand from fireplaces (Adams and Mortlock, 1974; Huxtable and Aitken, 1977), as well as natural cave and sedimentary deposits (Wintle, 1978; Wintle and Huntley, 1979). The latter techniques were first applied in archaeological contexts by Debenham (1983) and Readhead (1982), both workers independently devising a combined additive/regenerative TL method that is now generally called the "Australian slide" method (Prescott *et al.*, 1993). An earlier version of this approach had been proposed for lava-baked quartz pebbles by Valladas and Gillot (1978), and extended to burnt flints by Valladas (1978).

The second significant feature of the Zimmerman and Huxtable (1971) paper is the comparison made between TL and ^{14}C ages for a soil layer deposited more than 30,000 years ago, from which animal and human figurines had been recovered. They showed that uncalibrated ^{14}C ages underestimated TL ages (corrected for supralinearity) by ~4000 years at ~33,000 calendar years before present. Samples of this antiquity had never before been dated using TL, so the discrepancy between the ^{14}C and TL ages was regarded as only an "apparent difference". Since then, a similar degree of ^{14}C age underestimation (of up to 7000 years) for the period 30,000–40,000 years before present has been reported on the basis of TL dating of fireplaces (Bell, 1991; Huxtable and Aitken, 1977), burnt flints (Boëda *et al.*, 1996; Mercier *et al.*, 1991) and unheated sediments (Nanson and Young, 1987; Smith *et al.*, 1997); OSL dating of unheated quartz grains (David *et al.*, 1997; Roberts *et al.*, 1994a); $^{234}U/^{230}Th$ dating of lake deposits (Peng *et al.*, 1978), coral reefs (Bard *et al.*, 1990, 1993) and carbonate tufas (Bischoff *et al.*, 1994); electron-spin resonance (ESR) dating of tooth enamel (Rink *et al.*, 1996); and geomagnetic intensity variations recorded in marine sediments (Guyodo and Valet, 1996; Laj *et al.*, 1996).

2.3. *Recent TL applications*

Luminescence dating of pottery and ceramics has played a lesser role in archaeological research since the 1980s than has TL dating of burnt flints and TL and OSL dating of unheated sediments. Nonetheless, there have been several important applications of TL dating to pottery and ceramics in archaeological contexts where no other numerical age estimates (*sensu* Colman *et al.*, 1987) are available or where the available ^{14}C chronology is ambiguous or controversial. For example, TL dating was deployed on the earliest pottery yet found in the Western Hemisphere, and compared with accelerator mass spectrometry (AMS) ^{14}C dates for associated shell and charcoal samples (Roosevelt *et al.*, 1991). The fine-grain TL technique was used to derive a date of ~7000 years for a single pot sherd from a shell midden in the Brazilian Amazon, a date consistent with the multiple AMS ^{14}C age determinations. The existence of pottery of such antiquity in this tropical forest habitat indicated that, contrary to the prevailing notion, such environments were not unfavourable for permanent settlement, population growth and cultural development (Roosevelt *et al.*, 1991).

There has also been renewed interest in TL dating of ceramics from Central and North America (e.g. Dunnell and Feathers, 1994; Feathers, 1997a,d; Feathers and Rhode, 1997; Godfrey-Smith *et al.*, 1997). At the site of St. Croix in Nova Scotia, TL dating of five sherds has produced the first numerical age estimates for the decorative styles used on the local and regional ceramics. Fine-grain TL ages of between 1150 ± 150 and 2620 ± 290 years were obtained, the oldest being in close agreement with an associated ^{14}C age of 2500 ± 120 years BP (Godfrey-Smith *et al.*, 1997). TL dating of pottery has also provided archaeologists with a chronological framework for the pre-hispanic Classic and Postclassic periods in western Mexico (Duverger *et al.*, 1993), the Ochre Colour Ware and megalithic cultures in India (Singhvi *et al.*, 1983), and Neolithic to Bronze Age sites in Sicily (Troja *et al.*, 1996), Hungary and Croatia (Benkö *et al.*, 1989), where ^{14}C dates are sparse and sometimes at odds with expectations of age based on stratigraphy and typology.

Over the timespan of the past few centuries, where the use of charcoal from long-lived tree species creates problems for ^{14}C dating of recent archaeological events, TL dating of heated materials (and now also optical dating of unheated sediments) is especially valuable. Human contact with New Zealand has been brief, extending over perhaps the last millennium (Anderson, 1991) or two (R. Holdaway, 1996). In a novel attempt to date earth ovens made by the early Maori, Fankhauser (1990) extracted quartz inclusions from oven stones and obtained TL ages (corrected for supralinearity) of up to ~770 years. In contrast, charcoal collected from these ovens gave *calibrated* ^{14}C ages that were greater by 100–300 years for long-lived tree species. This study demonstrates the necessity of matching the dating method to the archaeological event of interest: TL dating here provides a more accurate estimate than ^{14}C dating for the elapsed time since these earth ovens were last used. Understanding the relation between the *dated* event (e.g. exposure of mineral grains to heat or sunlight, or the death of a tree) and the *target* (i.e. archaeological) event is clearly central to archaeometry, and Dunnell and Readhead (1988) and Feathers (1997a) provide further discussion on this issue.

By virtue of being able to date the target event directly, TL dating has been used to maximum advantage in tests of pottery and ceramic typological sequences, and at sites where the existing ^{14}C chronologies are suspect or where artefacts are exposed on the ground surface. Surficial artefacts are often difficult to date because, unlike buried artefacts, there is a lack of associated material suitable for ^{14}C or other forms of dating. Dunnell and Feathers (1994) demonstrated how TL dating can be applied to pot sherds exposed on the ground surface in the central Mississippi Valley, and surface ceramics from the atomic test site in arid southern Nevada have since been dated by luminescence (Feathers and Rhode, 1997). In the latter study, protohistoric "brownware" pottery was dated by the fine-grain TL method, with one sherd giving a comparable OSL age from quartz inclusions dated using Duller's (1991) single-aliquot approach. The "Australian slide" method was adopted, although luminescence multiplication ("scaling") factors of up to 1.93 were required to match the dose-shifted regenerative-dose data with their additive-dose counterparts. Difficulties were encountered in the determination of the dose rate, not because the ceramics were exposed to fallout from nuclear weapons testing but because of disequilibrium in the uranium decay series. Nonetheless, the TL and OSL dates of *circa* AD 1450–1840 accorded well with age controls based on associated organics (dated by ^{14}C) and other artefacts (projectile points and historic beads).

TL and ^{14}C ages have been compared in many studies, and a few examples only are listed below to illustrate the types of heated materials and anthropogenic structures that have been investigated. Guibert *et al.* (1994a) provided confirmation of ^{14}C ages on shell, thought to be erroneously old, from fine-grain TL dates on pottery from two Neolithic cemetries in central Sudan, whereas Buschbeck and Flettner (1994) found that TL dates for the sintered remains of a Neolithic loam hut in Germany confirmed archaeological expectations that the ^{14}C ages on charcoal were too young. The latter study employed coarse-grain feldspars for analysis, as did Sanderson *et al.* (1988) and Strickertsson *et al.* (1988) in their dating of the sintered remains of 10 vitrified forts in Scotland. Both groups made corrections for anomalous fading (Wintle, 1973) but Sanderson *et al.* (1988) did not use quartz inclusions for dating because of acute supralinearity problems. The four oldest forts were TL dated to between 3100 and 4100 years, thereby extending the known timespan for these monuments. Still further north, Huxtable *et al.* (1976) obtained TL dates of 1000–400 years BC for the "mounds of burnt stones" that constitute the most numerous class of antiquity in the Orkney Islands. Fine grains were extracted from cooking stones, with those that exhibited short-term anomalous fading being rejected. The TL dates compared favourably with two calibrated ^{14}C dates of ~1180 and ~1065 years BC, at which time the mounds were built as peat-fired cooking places for boiling meat in response to the decline of forest cover in Orkney (Huxtable *et al.*, 1976).

Other monuments and settlements dated recently by TL include: three Minoan palaces in Crete (dated to 3300–3800 years using quartz inclusions extracted from the kiln walls; Liritzis and Thomas, 1980); the Terracotta Army in Xi'an, China (dated to ~2200 years using the fine-grain TL from ceramics and baked soil; Lu *et al.*, 1988); stoneware from the period of the early Thai kingdom of Sukhothai (dated using the pre-dose technique to 600–800 years; Robertson and Prescott, 1987, 1988); temples and citadels in Sri Lanka (dated to the last 2500 years using quartz inclusions in pottery and fired bricks; Abeyratne, 1994); fifteen Mesolithic to medieval habitation sites in Denmark, Sweden and Finland (dated using quartz and feldspar from ceramics and burnt stones; Mejdahl, 1989); the remains of the strategically-located Roman city of Carnuntum in Austria (dated using the fine-grain technique on ceramic tiles from public baths, a lime kiln and the fill of a post-hole; Erlach and Vana, 1988); and the foundations of the archaic fortifications on Palatino Hill in Rome itself (dated to ~2600 years using quartz inclusions from pottery; Bacci *et al.*, 1990). Even the Spanish exploration of western Canada in the 18th century AD has been the subject of TL investigation, by virtue of an olive jar hauled up from the sea-bed off the Queen Charlotte Islands and dated to 235 ± 17 years (Williams *et al.*, 1991).

An unusual final example is that of Liritzis (1994) who used TL to date the *unburnt* limestone blocks of a Mycenean wall in Greece, in this instance the zeroing mechanism being insolation at the time of construction of the wall. He took three 1 mm thick slices of calcite from the contact surface of a limestone block and determined that only the outermost layer of calcite had been bleached by sunlight during the period of wall construction; this exterior layer gave an age of 3700 ± 450 years, in accord with archaeological expectations, while the inner layers yielded geological TL signals. This method has since been extended to the dating of limestone pyramids (Theocaris *et al.*, 1997) and the temple of Apollo in Delphi (Liritzis *et al.*, 1997). (Luminescence methods could also be used to date "standing stones" and other stone arrangements, where the exterior grains on the base of the stones were last exposed to sunlight when the stones were emplaced. Potential applications include megalithic graves and monolithic structures in western Europe (W. J. Rink, Personal Communication, 1994), stone lines and cairns on the Arnhem Land plateau in northern Australia (Chaloupka, 1993, p. 237) and the variety of stone monuments (Djarlarlar), burial rings (Wundudmool) and "sneezing heaps" (Djindjudngarri) found in the Kimberley region of Western Australia (G. Walsh, Personal Communication, 1996).

2.4. *Problems and prescriptions*

There have, however, been TL studies in which difficulties were encountered in dating pottery. These problems are most often associated with the estimate of the dose rate, owing to radioactive disequilibrium in the uranium-series (^{238}U) decay chain. Even after firing (and particularly if to temperatures below 1000°C), the clay matrix of pottery and ceramics retains an appreciable cation exchange capacity, and may interact with radionuclides moving through the soil profile (Hedges and McLellan, 1976; Murray, 1981). Radon (^{222}Rn) loss is a common form of disequilibrium, particularly at sites with porous pottery (e.g. Meakins *et al.*, 1979) and igneous geology (Aitken, 1985), and was deemed responsible for the 20% age underestimate obtained from pot sherds in volcanic terrain in the American south-west (Kojo, 1991). Problems have been reported in dating pottery from volcanic islands in the south-western Pacific Ocean, but for reasons of extremely weak TL emissions from the extracted fine grains, rather than dosimetric difficulties (Prescott, 1982; Prescott *et al.*, 1982). To correct for the extreme (~50%) loss of ^{222}Rn from pottery of the Hong Kong region, the thick-source alpha counting "gas cell" system was devised (Aitken, 1978; Huxtable and Aitken, 1978) and high-resolution gamma spectrometry was later employed (Huxtable *et al.*, 1982–3; Murray and Aitken, 1982). These techniques were used to infer radon *enrichment* in surface ceramics from the Great Basin (the additional radon possibly being absorbed from the atmosphere; Feathers and Rhode, 1997), and they have been widely adopted for luminescence dating of sediments.

Another form of disequilibrium is uranium enrichment, which Guibert *et al.* (1994b) detected in ceramics from Niger, West Africa using high-resolution gamma spectrometry. Leaching of uranium from the granitic rock shelter and deposition in the sediments and ceramics was proposed as a probable explanation for the excess of ^{238}U (up to 90%) compared to ^{226}Ra. TL ages were then calculated for the ceramics assuming uranium enrichment had occurred immediately after burial or more recently, in a similar manner to early and later uranium-uptake models used in the ESR dating of tooth enamel (Grün *et al.*, 1987). Based on the data presented by Carriveau and Harbottle (1983), uranium mobilisation also appears to seriously afflict pot sherds from Ban Chiang in Thailand. The calculated TL dates (Bronson and Han, 1972; Mortlock and Price, 1980–1) make no allowance for this disequilibrium, and could be seriously in error (Aitken *et al.*, 1983; Carriveau and Harbottle, 1983).

Uranium-series disequilibria have been reported also for archaeological sediments on the karst Nullarbor Plain in South Australia. In the Allen's Cave deposit, there was a preferential *loss* of uranium (compared to ^{230}Th) in association with carbonate complexes derived from the surrounding limestone (Roberts *et al.*, 1996; Olley *et al.*, 1997a). Several other types of disequilibrium were also observed, and the evolution of the dose rate through time was modelled to improve the accuracy of the OSL dates (Olley *et al.*, 1997a). For samples of Holocene age, disequilibrium due to ^{226}Ra is of particular concern because the majority of the dose rate in the uranium chain originates from the decay of ^{226}Ra and its "daughter" products, and because the half-life of this radionuclide (~1600 years) means that even a single episode of leaching has long-lived (5–6 half-lives) consequences (Meakins *et al.*, 1978; Aitken, 1985; Olley *et al.*, 1996).

Disequilibrium in the thorium (^{232}Th) decay series is rare, or at least rarely reported. Dunnell and Feathers (1994) observed an excess of ^{232}Th over its daughters in pot sherds from south-eastern Missouri, which they attributed to leaching of ^{228}Ra; owing to the short half-life (~6 years) of this nuclide, the extant disequilibrium implies that the sherds have acted as chemically "open" systems during at least the past 30 years. In contrast, these two radionuclides were shown to be in secular equilibrium in the deposits at Allen's Cave (Olley *et al.*, 1997a).

2.5. *Let there be light!*

The use of TL methods in Mesolithic to medieval archaeology has recently been supplemented by optical dating methods. Optically stimulated luminescence (OSL, using green or green/blue wavelengths for stimulation) and infrared-stimulated luminescence (IRSL) have been applied to both heated materials and unheated sediments in archaeological contexts (the latter commonly termed "archaeological sediments" or "archaeosediments"). Given the potential for vertical displacement of artefacts in unconsolidated sedimentary deposits (Cahen and Moeyersons, 1977; Hughes and Lampert, 1977; Richardson, 1992), it is clearly preferable to date artefacts directly rather than date the archaeosediments in which they lie, all other factors (e.g. matching the dated event to the target event; accuracy and precision of the dating method; and so on) being equal. Dating of archaeosediments is especially valuable, however, when heated artefacts are unavailable or when the target events are periods of major landscape disturbance (e.g. river and hillsltope sedimentation) triggered by human activities, such as forest clearance and agriculture.

2.5.1. *Heated materials.* Bøtter-Jensen and Duller (1992) made the first attempt to date heated material by OSL, using quartz extracted from burnt stone at a Viking Age site in Sweden. The additive-dose method was used in conjunction with 24 aliquots to derive an OSL age in accord with the TL age for the site, and of similar precision. Similarly close agreement between TL and OSL dates on heated quartz has been reported by Feathers (1997a), who noted that OSL gave the more precise estimates for four out of six pot sherds from Venezuela and the American south-west. Optical dating of heated artefacts has also capitalised on the new generation of single-aliquot techniques, which offer two substantial advantages: first, the mass of sample required is reduced to a minimum, which may be critical in certain situations; and second, the palaeodose can be calculated with improved precision as each aliquot is self-normalising and as several independent estimates of the palaeodose can be obtained at least as quickly as one multiple-aliquot palaeodose estimate. Liritzis *et al.* (1994) dated two ceramic sherds using the single-aliquot additive-dose technique (Duller, 1991, 1995) and obtained OSL dates in close accord with TL dates, and of equal or better precision. Two aliquots of quartz (each weighing only 5–10 mg) were used for each sherd, one to characterise the dose-response curve and the other to describe the decay rate due to the repeated OSL measurements and preheats.

There are potential pitfalls with single-aliquots, however, particularly in the use of regenerative-dose procedures (Duller, 1991; McKeever *et al.*, 1996, 1997; Richardson, 1994; Tso and Li, 1994) which, unlike single-aliquot additive-dose methods, measure the total luminescence signal. Mejdahl and Bøtter-Jensen (1994) observed significant sensitivity changes in quartz and feldspars resulting from the erasure of the "natural" signal by TL, OSL or IRSL, and proposed a novel method of correcting for these changes using a hybrid of additive-dose and regenerative-dose protocols. Their single-aliquot/regeneration and added dose (SARA) technique is not strictly a single-aliquot method, but it does require many fewer aliquots than conventional multiple-aliquot methods and can accommodate sensitivity changes provided they are independent of the added dose. Initial tests of the SARA technique on quartz extracted from 600–1300 year-old ceramics and kiln bricks showed good agreement between OSL ages and independent age estimates (Mejdahl and Bøtter-Jensen, 1994). Another approach, requiring only three aliquots, was proposed by Tso and Li (1994) who adapted Duller's (1991) single-aliquot regenerative-dose IRSL protocol to bracket the true palaeodose in fine grains extracted from 3500 year-old Chinese pottery.

Volcanic ash (tephra) is a type of heated sediment found at some archaeological sites. Minerals in ash may include glass, feldspar and quartz, whose luminescence "clock" is reset thermally during an eruption. Volcanic feldspars suffer from anomalous fading (Wintle, 1973) but silt-sized glass shards appear to yield reliable TL and IRSL dates for tephras in the time range 500–400,000 years (Berger, 1991, 1992; Berger and Huntley, 1994). Quartz is useful in proximal volcanic contexts, and Liritzis *et al.* (1996) have recently applied fine-grain and quartz inclusion TL methods to a tephra deposited on the island of Yiali in the Aegean Sea. The four dates obtained (using the 325°C TL signal and the additive-dose method, with corrections for supralinearity) ranged from ~1000 to ~2100 years BC, but with errors that overlapped; unusually for quartz, the grains extracted from the two oldest samples exhibited significant internal radioactivity. The mean age of ~1460 years BC indicates that the hitherto overlooked Yiali eruption, as well as the destructive Thera (Santorini) eruption also in the 2nd millennium BC (Renfrew and Bahn, 1991), should be considered as a possible cause of the collapse of the Minoan civilisation in Crete. There have been few other archaeological applications of luminescence dating to tephras, although great potential exists in regions with thick accumulations of volcanic ash; examples from Papua New Guinea (Groube *et al.*, 1986; Pavlides, 1993; Pavlides and Gosden, 1994) are discussed later in connection with the human colonisation of Melanesia.

2.5.2. *Unheated sediments.* TL dating of archaeosediments was attempted in the early 1980s using techniques that were equally applicable to geological sediments (Readhead, 1982; Singhvi *et al.*, 1982), and optical dating of archaeosediments has likewise employed procedures developed originally for OSL and IRSL investigations of natural deposits. At both archaeological and geological sites, exposure to sunlight is needed to reset the luminescence "clock".

It is advantageous, however, to use optical, rather than TL, methods to date unheated archaeosediments, as the electron traps sampled in optical dating are reset by a few minutes, rather than a few hours, of solar exposure (Godfrey-Smith *et al.*, 1988). Single-aliquot methods are especially useful at habitation sites where archaeosediments in the upper few decimetres of the deposit have been mixed as a result of human activity (Chawla and Singhvi, 1989). This "zone of disturbance" is analogous to the bioturbation of soil A-horizons (Huntley *et al.*, 1983), both processes resulting in the re-exposure and mixing of surficial sediments. Conventional multiple-grain, multiple-aliquot methods will yield only an average age for those grains present at any particular depth, whereas single-grain optical dating methods (Galloway, 1996; Lamothe *et al.*, 1994; Lamothe and Auclair, 1997; Murray and Roberts, 1997, Murray *et al.*, 1997) present the opportunity to distinguish between "modern" grains that have infiltrated downwards and "old" grains that have moved upwards but have not been re-exposed on the ground surface.

OSL and IRSL methods have seen surprisingly little application to Holocene and terminal Pleistocene archaeosediments since Huntley *et al.* (1985) first tested the accuracy of optical dating on a 6000 year-old aeolian dune sequence in the Peace River valley of British Columbia, Canada. The buried soil horizons in these dunes had been dated by ^{14}C and contained artefacts such as stone tools (including projectile points) and hearths; for a 5000 year-old sample, good agreement was obtained between OSL and TL ages on quartz, but both were double the ^{14}C age and the TL age obtained from fine-grain polyminerals (Huntley *et al.*, 1983). Greater success was achieved at three archaeological sites in Morocco (Chaperon Rouge, Skhirat and Tahadart), where dune sands had been deposited between 6000 and 24,000 years ago: multiple-aliquot additive-dose OSL dates on coarse-grain quartz from these archaeosediments compared favourably with ^{14}C ages and with TL ages on pottery and burnt flints (Rhodes, 1988; Smith *et al.*, 1990a,b). The latter optical dating team also collected samples from the Upper Palaeolithic to Roman age site at Hengistbury Head in Dorest, England (Smith *et al.*, 1990a,b), an important site in terms of linking the British stone tool sequence with that found in north-west Europe (Barton and Huxtable, 1983). The quartz OSL palaeodose determinations (multiple-aliquot, additive-dose) matched those obtained by TL on sediment and burnt flint, and the ages were consistent with the ^{14}C chronology.

OSL and TL dating methods have also been used to check the ^{14}C ages obtained previously for anthropogenic earthen mounds and concentric ridges in north-east Louisiana (Feathers, 1997b; Saunders *et al.*, 1997). For such structures, luminescence methods hold the advantage over ^{14}C determinations by dating the target archaeological event: the time of mound or ridge construction. It is important to demonstrate, however, that the luminescence "clock" was reset when the earthworks were built, otherwise too great an age will be estimated. Sand-sized quartz grains were extracted from buried surfaces within mound and ridge structures, and their OSL and TL palaeodoses were determined, the latter from the hard-to-bleach (375°C) peak which had been isolated by first erasing the easy-to-bleach TL signal using green (550 ± 20 nm) light. The dose rates were derived mainly from high-resolution gamma spectrometry, which indicated no disequilibrium in either the ^{232}Th chain or, for most samples, the ^{238}U chain. The combined additive/regenerative "Australian slide" procedure (Prescott *et al.*, 1993) yielded TL ages that reflected the Pleistocene antiquity of the geologic features on which the earthworks stood, indicating that exposure to ultraviolet wavelengths of sunlight had been too brief during ridge and mound construction to even partially erase the hard-to-bleach signal (Feathers, 1997b).

OSL palaeodoses were determined using the "dose corrected" additive and regenerative single-aliquot procedures of Duller (1994, 1995), and a preheat of 220°C for 300 seconds. For each sample, the natural OSL intensity of each aliquot was plotted against its palaeodose, following Li (1994). Two samples showed an increase in palaeodose with OSL intensity, again suggestive of insufficient bleaching in antiquity (of even the most light-sensitive signal); a third sample showed no such relation and an age of *circa* 4000–5000 years was calculated for this earthen ridge. Two ages were calculated for both partially bleached samples: a maximum age was obtained from the smallest single-aliquot palaeodose (Duller, 1995) and the approximate true age was inferred from the palaeodose intercept on the OSL intensity/palaeodose plot (Li, 1994). One of the samples (a mound) gave ages of ~5500 and ~4600 years, in close accord with the ^{14}C date of 5300 years BP for charcoal from the construction fill. The other sample (a ridge at Poverty Point) yielded OSL ages of ~2100 and ~1800 years, which compare favourably with a ^{14}C date of 2100 years BP for the same level (Feathers, 1997b). A luminescence chronology for the "moundbuilders", who so intrigued Jefferson and 19th century American archaeologists (Renfrew and Bahn, 1991, pp. 26–27), now appears to be within reach using single-aliquot optical dating methods to deal with partially bleached sediments.

IRSL made its debut in archaeosediment dating at a Romano-British site near Hereford in England (Spooner *et al*., 1990). A mixture of fine-grain polyminerals was extracted from an alluvial sediment layer sandwiched between two occupation levels containing pottery, stylistically dated to 1700 ± 50 years. Ages consistent with this were obtained by IRSL (1900 ± 200 years), using 880 nm diodes for stimulation, and by OSL (1500 ± 200 years), using the 514 nm (green) line from an argon-ion laser; in each case, the additive-dose method was applied to 50 aliquots.

Infrared stimulation has since continued to be used to obtain optical dates from feldspars extracted from archaeosediments that may have been poorly bleached at deposition, and hence liable to yield gross TL age overestimates. For example, Fuller *et al*. (1994) employed a Mesolithic burial site on the banks of the River Danube in Romania to constrain the age of the overlying alluvium; this they dated using a "partial bleach" approach, to simulate the bleaching conditions experienced by fluvially transported silt grains, and obtained an IRSL age consistent with the archaeological age.

Even slopewash deposits transported distances of less than 100 m have been successfully dated using IRSL (Lang, 1994; Lang and Wagner, 1996, 1997; Wiggenhorn *et al*., 1994; Zöller *et al*., 1996). Among the remains of a "Michelsberger Kultur" hillslope settlement at Bruchsal Aue in southern Germany are two sets of defence trenches which were infilled by colluvium after abandonment of the settlement in the 4th millennium BC (Fig. 2). The IRSL ages of silt-sized feldspars obtained from these colluvial infills (~5000 years) post-date the abandonment of the Late Neolithic settlement, and indicate that the IRSL signal was satisfactorily zeroed in antiquity, despite the sediments being transported by water over a very short distance. The efficacy of bleaching was tested experimentally by Lang and Wagner (1996), who showed that the IRSL signal was effectively zeroed after 30 min of exposure to sunlight filtered through cloud and fog. A more recent sample of heated colluvium from a fireplace was also found to yield an IRSL age in accord with its stratigraphic position and a TL age determination; the IRSL age was significantly more precise than its TL counterpart, indicating the benefits of using optical dating for burnt archaeosediments. The youngest sample dated (780 ± 280 years) further demonstrates the potential for optical dating of archaeological events as recent as medieval.

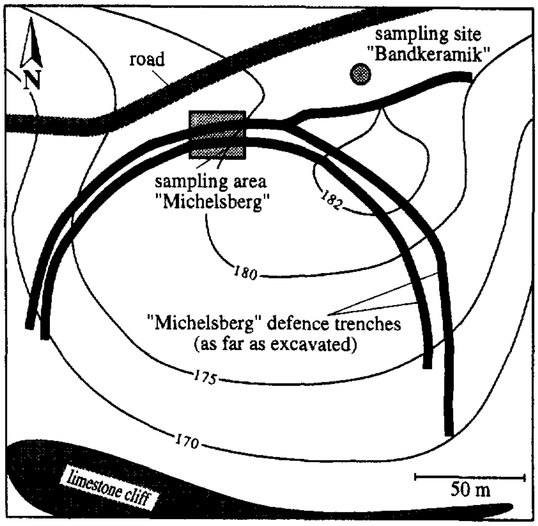

Fig. 2. Topographic map and ground plan of the "Michelsberg" defence trenches, showing sampling site locations. Contours are in metres above mean sea-level (after Lang and Wagner, 1996).

Despite these promising findings, Wiggenhorn *et al*. (1994) expressed concern that the IRSL ages may have been too young owing to interference from unstable IRSL emissions in the broad optical detection window (330–600 nm) used for dating. As a test of this proposition, Lang and Wagner (1996) extended their IRSL investigations to a nearby Early Neolithic site, where a pit had been infilled with sediment and pot sherds from the "Mittlere Bandkeramik" period (Fig. 3). Ages obtained using the broad detection band were compared with those obtained using a narrow detection band, centred on 402 nm. Only minor differences were observed, although the narrow band was recommended for future studies. The IRSL ages obtained for the pit-fill (6400 ± 800 years) and oldest colluvium (7500 ± 1300 years) were consistent with each other and with archaeological expectations, and suggest that the advent of agriculture with the "Bandkeramik" people was accompanied by accelerated soil erosion and sedimentation.

Further indications of the magnitude and timing of soil erosion resulting from ancient human activities have been gleaned from archaeosediments by

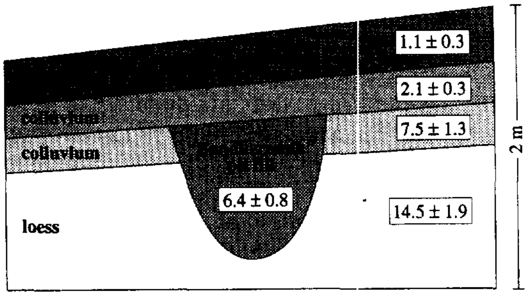

Fig. 3. Schematic cross-section of the "Bandkeramik" sampling site, showing sediment type, sample locations, and IRSL ages in thousands of years (after Lang and Wagner, 1996).

Lang and Wagner (1997) and Rees-Jones and Tite (1997a). Again using a narrow detection band (390–450 nm) for IRSL dating of fine-grain feldspars, Lang and Wagner (1997) found that the greatest rate of colluvial accumulation at a site near Wiesenbach, south-west Germany, occurred in response to intensive agriculture in medieval times, ceasing around 540 ± 45 years ago; surprisingly, rather little sedimentation was associated with farming during the Roman period. In contrast, periods of sedimentation in the river valleys of the Nene and Thames in England were found to coincide with forest clearance in the Bronze Age and intensive farming in Roman and Saxon times (Rees-Jones and Tite, 1997a). The latter results were obtained using the IRSL from fine-grain polyminerals and the, rarely exploited, OSL from fine-grain quartz. A regenerative-dose correction procedure was devised to account for the hard-to-bleach and preheat-induced IRSL signal, which constituted 7–30% of the total IRSL in these samples; by comparison, palaeodose corrections of less than 4% were applied to the fine-grain quartz OSL, for which alpha-efficiency "*a*" values of 0.032–0.043 (± 0.002) were measured.

Rees-Jones and Tite (1997a) reported less success, however, in dating colluvial deposits from an infilled Iron Age ditch in Hampshire and an 8th century AD earthwork (Wat's Dyke) in North Wales. In both cases, the IRSL and OSL ages obtained from fine-grain feldspar and quartz were much older than the archaeological evidence, insufficient bleaching of the sediments at deposition being the proposed explanation. A similar lack of success with IRSL dating of fine-grain feldspars in colluvium has been reported by Wintle *et al.* (1993). It now appears advantageous to use coarse-grain feldspars allied with single-aliquot techniques to identify those colluvial deposits that contain a proportion of poorly bleached grains (Duller *et al.*, 1995; Li, 1994; Wintle *et al.*, 1995).

In conclusion, luminescence dating methods have played a major role over the past three decades in providing archaeologists with numerical age estimates for a variety of human activities since the Mesolithic. Heated artefacts have been dated directly using TL and, increasingly, by OSL and IRSL methods, while unheated sediments in archaeological contexts (archaeosediments) have enabled human disruptions to the landscape to be discerned. These kinds of applications have also been made for time periods preceding the Mesolithic. To these we shall now turn, beginning with the debate on the origins and evolution of modern humans—an arena in which luminescence dating has made, in my view, its most spectacular and influential contributions to archaeology.

3. MODERN HUMAN ANTIQUITY AND ANCESTRY

The origins and evolution of anatomically modern humans, *Homo sapiens sapiens*, is a theme in archaeology and palaeoanthropology that fascinates laymen and scholars alike. Some of the greatest scientists of the 1860s were drawn into this debate, such as the comparative anatomist Richard Owen, the geologist Charles Lyell, and the biologists Charles Darwin, Thomas Henry Huxley and Alfred Russel Wallace (Desmond and Moore, 1991). Lyell only became convinced of a pre-glacial antiquity for modern humans in 1859 after a tour of archaeological sites in England and France, during which he acknowledged the co-existence of flint tools with the remains of extinct animals (Lyell, 1863). At the same time, the first discovery of a Neanderthal skull was made in a cave near Düsseldorf in Germany. This species of human, *Homo sapiens neanderthalensis*, added an extra element to the question of human evolution which entangled both Huxley (1863) and Darwin (1871) and which continues to intrigue to this day (Krings *et al.*, 1997).

Over the past quarter century, TL has played a central chronological role in the debate on modern human antiquity and ancestry. Stone tools are one of the most enduring artefacts found at archaeological sites (e.g. the 2.5 million year-old tools from northern Ethiopia; Semaw *et al.*, 1997) and their typological differences have been used to recognise distinctive stone tool "industries" and to construct *relative* chronologies for the emergence and spread of humans around the world. Since the work of Göksu *et al.* (1974), burnt flint (a form of chert) has taken on additional significance as a reliable medium for obtaining *numerical* age estimates using TL. Recent applications of TL dating to heated flints has forced a re-evaluation of the evolutionary relationship between anatomically modern humans and Neanderthals, and their association with particular stone tool industries, in Western Europe and the Middle East. Much of this section of the paper will review these outstanding and often controversial contributions.

Luminescence dating of unheated archaeosediments has made a similar dramatic impact on the chronology of modern human origins in regions as far flung as Africa, Siberia, Australia and the Americas. The Australian and American applications will be discussed in the next section on continental colonisations, but we shall begin here with luminescence studies of sediments from the presumed birthplace of modern hominids—Africa.

3.1. *Africa*

The point in time at which modern humans can be distinguished anatomically from their archaic ancestors is a matter of considerable interest, as it relates directly to arguments about the morphological and genetic lineage of ancient human populations. Some palaeoanthropologists (e.g. Frayer *et al.*, 1993; Templeton, 1993; Wolpoff, 1989) have argued the case for "regional continuity" over the past million years, where hominid populations in more or less geographically isolated regions of the world have evolved their own distinctive, regional traits. Other palaeoanthropologists and most geneticists (e.g. Cann *et al.*, 1987; Groves, 1994; Hammer, 1995; Stringer and McKie, 1996; Tishkoff *et al.*, 1996) have championed the "replacement" view that modern humans everywhere are derived from a common genetic stock that evolved in Africa between 100,000 and 200,000 years ago, and then swept across the globe; over time, these relatively recent migrants completely replaced the existing archaic hominid populations. It is therefore important to pinpoint when modern humans first emerged in Africa, as this will place a lower limit on the date of their departure from the continent and their dispersal around the world.

3.1.1. *Southern Africa.* Hominid remains at Florisbad spring in South Africa show a combination of archaic and modern characteristics, and may be "among the direct forerunners of anatomically modern humans" (Bräuer, 1989, p. 129). The remains were recovered from a spring vent, which presented severe problems for reliable dating. Early dating attempts were made on the overlying and adjacent peat layers, which produced ^{14}C and uranium-series ages of >42,600 years BP and >100,000 years, respectively. Recently, however, ESR dating of a hominid tooth from the spring vent assemblage and OSL dating of the adjacent peat and brown sand layers have provided finite age estimates for this specimen (Grün *et al.*, 1996). The ESR age of 259,000 ± 35,000 years (using a novel non-destructive method) compared favourably with the OSL ages of the deposits through which the spring had vented (281,000 ± 73,000 and 279,000 ± 47,000 years). The latter were obtained from quartz extracts, the gamma dose rate having been measured *in situ* and with high-resolution gamma spectrometry to check for radioactive disequilibrium in this groundwater-affected site.

To my knowledge, these are the oldest OSL ages yet reported for quartz and, despite dose-saturation problems giving rise to the wide error margins, they demonstrate that the OSL and associated easy-to-bleach 325°C TL signals in quartz (Kaylor *et al.*, 1995; Smith *et al.*, 1986; Spooner, 1994a; Wintle and Murray, 1997) are stable over at least the last 300,000 years. This finding conflicts with the upper limit of ~150,000 years suggested by Huntley *et al.* (1993a, p. 18), who observed the lack of a palaeodose plateau over the 325°C TL region in quartz sand deposited as recently as 120,000 years ago (see Huntley *et al.*, 1994a). They attributed this foreshortened plateau to instability of the 325°C TL peak but, in such young samples, an alternative explanation may be the decay of the overlapping 280°C TL peak, whose lifetime (at 15–20°C) is probably on the order of 300,000 years (Aitken, 1985; Roberts *et al.*, 1993b) or less (Murray *et al.*, 1997; Prokein and Wagner, 1994).

The date of ~260,000 years for the Florisbad hominid provides a slightly longer timeline for anatomically modern humans than that estimated by geneticists. But it supports the recent suggestion of a >250,000 years age for the transition from archaic *Homo sapiens* to the first fully modern humans, the Proto-Cro-Magnons, in the Middle East (Mercier *et al.*, 1995a,b). The skull of the "Man of Galilee" was collected from Zuttiyeh Cave in Israel and was described originally by Arthur Keith (1927), the prominent British prehistorian who was then embroiled in the "Piltdown Man" fraud. Some palaeoanthropologists (e.g. Vandermeersch, 1989) consider the skull as being the close ancestor of Proto-Cro-Magnons and, on the basis of the associated stone tool industry, would appear to correlate with an age of more than 250,000–270,000 years (Mercier *et al.*, 1995a,b). The latter age estimate is derived from TL dates obtained on burnt flints from stone tool industries throughout the Levant (see below). Both TL and OSL have therefore played their part in defining the length of time over which modern humanity has existed.

TL and optical dating have also contributed to questions concerning the Middle Stone Age in Africa. This period is crucial in terms of the evolution of anatomically modern humans, yet its chronology is poorly constrained. At Florisbad, quartz sediments from a Middle Stone Age horizon gave an OSL age of 133,000 ± 31,000 years, in close agreement with a mean ESR age of 121,000 ± 6000 years from associated tooth fragments (Grün *et al.*, 1996). This last interglacial age is similar to the ESR ages obtained for Middle Stone Age artefacts and remains of anatomically modern humans found in Border Cave and the cave complex at the mouth of Klasies River in South Africa (Grün *et al.*, 1990; Grün and Stringer, 1991). However, the Border Cave dates (including those of 45,000–75,000 years for the distinctive Howiesons Poort lithic industry) are too young by ~30% compared with amino-acid racemization dates on ostrich eggshell (Miller *et al.*, 1992); the latter suggest an age of more than 105,000 years, and possibly > 145,000 years, for the base of the sequence. Attempts to resolve this discrepancy through TL and optical dating were unsuccessful, owing to the powdery nature of the cave sediments (Wintle, 1996), but luminescence dating of the Klasies River cave sediments would appear warranted, as the ESR ages of the Howiesons Poort layers are similar to those obtained at Border Cave and may likewise be too young.

Luminescence methods have also been used to date the Middle and Late Stone Age levels at White Paintings Rockshelter, located near the Okavango Delta in north-west Botswana (Feathers, 1997c). This site is important for the early appearance of barbed bone points and fish exploitation in Africa (Robbins *et al.*, 1994). As deposits in the shelter were a mix of wind-blown and colluvial quartz sand, some being derived from roof collapse, steps were taken to determine the adequacy of sediment bleaching at deposition. The rapidly bleached OSL and 325°C TL signals and the hard-to-bleach (375°C) TL peak in quartz were examined, the latter by the "Australian slide" combination of additive-dose and regenerative-dose procedures to minimise extrapolation errors (Prescott *et al.*, 1993). He also constructed a "master" additive-dose growth curve from all samples at the site, as each exhibited a similar dose-response; this provided a second means of restricting extrapolation errors for the older samples. For the OSL analyses, Feathers (1997c) employed the single-aliquot additive-dose and regenerative-dose protocols in tandem with the "dose correction" method that Duller (1994) devised for feldspars.

A similar palaeodose was obtained from the three signals using the different methods, which was taken as indicating sufficient bleaching of the sediments at deposition (Feathers, 1997c). For example, the sample marking the transition from the Middle to the Late Stone Age yielded mean palaeodose estimates of 55 Gy (Australian slide) and 63 Gy ("master" curve) for the hard-to-bleach TL peak, 71 Gy (additive-dose) for the rapidly bleached TL peak, 56 Gy from single-aliquot regenerative-dose OSL, and three determinations of 54, 57 and 72 Gy from single-aliquot additive-dose OSL. The weighted mean of these determinations corresponds to an age of 55,400 ± 4700 years, in reasonable accord with ^{14}C and amino-acid racemization dates for the Middle to Late Stone Age transition at Border Cave (Miller *et al.*, 1992). There remains the possibility, however, of a systematic palaeodose (and thus age) overestimation if contaminant grains form a significant (> 10%) proportion of each sample and if each aliquot holds more than a few tens of grains. Contaminant grains may then be present in roughly equal numbers on all aliquots and this will go undetected, even by the method proposed by Li (1994). Only analyses using fewer than 10–20 grains per aliquot, extending ultimately to the dating of single grains (e.g. Lamothe *et al.*, 1994; Murray and Roberts, 1997), can circumvent this thorny problem.

3.1.2. *Central and northern Africa.* Katanda in eastern Zaire is another Middle Stone Age site where luminescence has been used to date the first *behavioural* traits of fully modern humans, as evidenced by bone points carved more than 90,000 years ago (Brooks *et al.*, 1995; Yellen *et al.*, 1995). TL and OSL dates were obtained for quartz grains extracted from well-sorted alluvial sands. The easy-to-bleach OSL and TL signals in quartz were exploited initially, but unusual dose-response behaviour prevented definitive palaeodose determinations (Kaylor *et al.*, 1993). Consequently, the hard-to-bleach TL peak was used for analysis, a less-than-ideal choice given its slow bleaching response to unfiltered sunlight and particularly those wavelengths that penetrate through a column of water (Berger, 1990; Spooner *et al.*, 1988). Fortunately, the TL palaeodose estimate of 170 ± 10 Gy was similar to that suggested by OSL (~190 Gy), providing some confidence that the sample had been well bleached before deposition. The TL palaeodose corresponds to an age of 82,000 ± 8000 years. The likely prospect of radioactive disequilibrium in these fluvial sediments was examined directly by using high-resolution gamma spectrometry, and none was observed in the present-day deposits.

Independent confirmation of the great antiquity of the bone harpoons at Katanda is given by the ESR age of ~89,000 years obtained from the enamel of hippopotamus teeth recovered from the same level (Brooks *et al.*, 1995). This ESR age is calculated assuming that uranium was absorbed by the teeth soon after burial, whereas the assumption of linear-uptake produces a much greater age (~155,000 years). The latter falls between mass-spectrometric U-series ages of ~140,000 and ~174,000 years on dentine from two teeth, but these samples are considered to have suffered from the loss of uranium late in their burial history, resulting in age overestimates (Brooks *et al.*, 1995). By way of contrast, uranium loss (such as that detected by Feathers (1997c) from high-resolution gamma spectrometry analyses of deposits at White Paintings Rockshelter) has a minor effect on luminescence age determinations because the bulk of the dose rate in the ^{238}U series is derived from ^{226}Ra

and its decay products (Aitken, 1985; Olley *et al.*, 1996).

The Katanda study demonstrates the value of using multiple dating methods at an archaeological site to avoid deficiencies in any particular technique. Despite the apparent concordance between the luminescence and ESR ages, however, there seem to be some problems with the original study, and further OSL analysis of the sediment samples using a single-aliquot additive-dose protocol (Duller, 1994) suggests that the material may not have been fully bleached in atiquity (Feathers, 1997c; Gibbons, 1997c). Another example of multi-method dating comparisons is the study of two adjacent fossil lakes (Bir Tarfawi and Bir Sahara East) in the Egyptian portion of the Sahara Desert (Wendorf *et al.*, 1993, 1994). Stone artefacts have been recovered from the lacustrine sediments, and a variety of numerical dating methods (^{14}C, uranium-series, ESR, TL, OSL and amino-acid racemization) have been applied to a range of materials to obtain a chronology for these archaeological and palaeoclimatic events. A neat chronology was not obtained, however, leading Wendorf *et al.* (1993, p. 558) to conclude "We expected grossly similar results and, to some extent, that expectation has been fulfilled. However, there are discrepancies between the dates obtained by different methods, and none of them appears to yield completely reliable or consistent results, although some are better than others." The latter category included optical dating methods, which provided "the most internally consistent suite of dates for the Tarfawi and Sahara East sequences." (Wendorf *et al.*, 1993, p. 560).

The luminescence chronologies for Bir Tarfawi and Bir Sahara East were obtained using sand-sized quartz grains from unheated sediments as well as burnt sediments. The unheated sediments were examined by TL (Bluszcz, 1993) and OSL (Stokes, 1993), both using the regenerative-dose method. As the initial bleaching step in this method may induce changes in TL and OSL sensitivity (e.g. Berger, 1988; Bowall *et al.*, 1987; Smith *et al.*, 1990b), the additive-dose technique was also used for a series of OSL samples. The calculated palaeodoses were found to correspond closely to their regenerative-dose counterparts (using the 514.5 nm line from an argon-ion laser for optical stimulation), demonstrating the absence of significant sensitivity change in these samples. As the TL palaeodoses were determined principally from the hard-to-bleach peak (judging by the palaeodose plateaux obtained), sensitivity change was less of a concern than adequate bleaching in antiquity. Ultraviolet laboratory bleaches of between 2 and 6 h duration, and the plateau method of Mejdahl (1988a), were applied to determine the "residual" TL level reached by the sediments at deposition. This variant of the "partial bleach" method (Berger, 1988; Wintle and Huntley, 1982) produced ages in broad agreement with the OSL ages, as did TL analysis of the 375°C TL peak in two burnt sediment layers from Bir Sahara East and one burnt core from Bir Tarfawi (Huxtable, 1993).

The burnt samples and the surrounding unheated sediments were analysed by high-resolution gamma spectrometry to check for disequilibrium in the ^{238}U decay series. None was observed, nor did thick-source alpha counting show any evidence of radon escape (Huxtable, 1993). Past episodes of disequilibrium, however, may have occurred during wetter, lake-full periods. Difficulties encountered in U-series dating of lacustrine marls clearly suggest that these sediments have not always behaved as chemically "closed" systems (Schwarcz and Morawska, 1993). TL, OSL and infinite ^{14}C dates for lake sediments, and OSL ages for overlying aeolian sands, indicate that the most recent Pleistocene wet episode occurred more than 60,000 years ago (Wendorf *et al.*, 1993, 1994). If this and earlier episodes were characterised by leaching of ^{226}Ra, for example, then such disequilibrium would since have disappeared, leaving no sign that dose rates determined from present-day radionuclide concentrations are in error. This problem plagues all luminescence dating studies, but especially those involving river and lake sediments which are proof of former hydrologic activity and which are sampled, often many millennia later, when dry.

Despite the chronological uncertainties, luminescence and other dating methods have placed constraints on the timing of archaeological and palaeohydrologic events for the duration of the Middle Stone Age at Bir Tarfawi and Bir Sahara East. The earliest deposits contain Late and Final Acheulian hand axes, followed by Middle Stone Age artefacts in deposits no older than ~175,000 years. At least five lacustrine phases occurred between then and 60,000 years ago, with firm evidence for major wet episodes in the interval 100,000–140,000 years ago. For much of the Middle Stone Age, therefore, the environment differed considerably from the desert of today, permanent or seasonal lakes being present for most of this important period of human behavioural and biological development. Which species of human inhabited these sites is not known, but the limited variation in stone tool typology and the consistent pattern of site exploitation throughout the Middle Stone Age suggest that "the acceleration of cultural development that supposedly accompanied the appearance of the modern form of human had not begun by the end of the Last Interglacial." (Wendorf *et al.*, 1993, p. 573).

The suggestion that a marked change in human behaviour can be perceived in artefacts produced only since the last interglacial is consistent with the luminescence dates of 70,000–90,000 years (or less; J. Feathers, Personal Communication, 1997) for the bone implements found at Katanda, to the south-west. It also sits comfortably with the longevity of certain lithic industries at modern hominid sites in the Middle East, to the north-east. At Tabun Cave, a mandible assigned to *Homo sapiens sapiens* was recovered from Layer C and seven associated burnt flints were TL dated to 171,000 ± 17,000 years (Mercier *et al.*, 1995b). This lithic industry persisted in the Levant for a further 80,000 years, being present at Qafzeh Cave in deposits dated to 92,000 ± 5000 years (Valladas *et al.*, 1988). Fully modern human behaviour thus appears to have developed only since the onset of the most recent glaciation, a period during which at least two hominid species inhabited Europe and the Middle East. Our understanding of the temporal overlap between Neanderthals and anatomically modern humans has been revolutionised in the past decade, due largely to the application of TL dating methods. These notable contributions are the main subject of the next section.

3.2. *Eurasia*

For hominids leaving Africa, southwest Asia has been the gateway to Europe. While *Homo erectus* may have been living 1.0–1.4 million years ago in the Jordan Valley (Bar-Yosef, 1994) and 1.6–1.8 million years ago in the Caucasus (Gabunia and Vekua, 1995), the earliest remains of *Homo* in Europe are those of *H. antecessor* from Gran Dolina in northern Spain (Bermúdez de Castro *et al.*, 1997; Gibbons, 1997b). This newly-named species may be the last common ancestor of modern humans and Neanderthals, and the fossil deposits have been dated by palaeomagnetism to more than 780,000 years (Parés and Pérez-González, 1995). The prehistory of these archaic hominids lies at or beyond the dating limit of ~800,000 years for current luminescence methods (Berger *et al.*, 1992; Huntley *et al.*, 1993a, 1994a,b). TL dates of 850,000 years for the sites of Korolevo in Ukraine and Kuldara in Tajikistan (cited in discussion by Carbonell *et al.*, 1995, pp. 828–829) should be regarded with caution until details of the methods used are made available for scrutiny. Luminescence dating efforts have instead been concentrated on the chronology of modern human evolution in this key region, with a particular focus on the eastern Mediterranean region (known variously as the Middle East, the Near East, and the Levant).

3.2.1. *The Levant.* Tabun Cave is the key site for the Late Pleistocene cultures of the Levant (Bar-Yosef, 1989). It lies on the western edge of Mount Carmel in Israel and a variety of lithic industries are represented in its deposits (Jelinek, 1982). A chronology for this important site has been erected from ^{14}C dating of charcoal and black soil (Weinstein, 1984), ESR (Grün *et al.*, 1991) and mass-spectrometric U-series (McDermott *et al.*, 1993) dating of tooth enamel, and TL dating of fine-grain quartz sediments (Bowman, 1985) and, most recently, burnt flints (Mercier *et al.*, 1995b). Particular attention has been paid to Layer C (in Garrod's scheme, equivalent to Jelinek's Unit I) which contained Mousterian artefacts together with the remains of two hominids, one a Neanderthal and the other possibly a Proto-Cro-Magnon (Mercier *et al.*, 1995b).

The first numerical age estimates for Tabun were obtained by ^{14}C which yielded finite dates of between ~40,900 and ~51,000 years BP for Layer C (Weinstein, 1984). An apparent stratigraphic inversion was indicated by a younger age of 35,400 ± 900 years BP for the underlying Layer D. These determinations lie at the limits of the ^{14}C method, however, and should be viewed as minimum ages only (Grün and Stringer, 1991). Subsequent mass-spectrometric U-series dating of dental fragments produced ages of *circa* 98,000–105,000 years for Layer C and 110,700 ± 900 years for Layer D (McDermott *et al.*, 1993). These are comparable to the ESR ages of 102,000 ± 17,000 and 122,000 ± 20,000 years for Layers C and D respectively, assuming early uranium-uptake by teeth after burial (Grün *et al.*, 1991); the linear-uptake ESR ages were substantially older, being ~119,000 years for Layer C and ~166,000 years for Layer D. The agreement between the U-series and early-uptake ESR ages was taken as providing confidence in both sets of age determinations.

Recent TL dates from burnt flints, however, seriously conflict with the ESR and U-series age estimates, Layer C being dated to 171,000 ± 17,000 years and Layer D (Jelinek's Unit IX) to 263,000 ± 27,000 years (Mercier *et al.*, 1995b). The base of the sequence (Layer E) was TL dated to 330,000–350,000 years, which is double the U-series and early-uptake ESR ages for this level. How can these differences be explained? The TL dates were obtained using well-tested methods for burnt flint (Aitken and Valladas, 1992; Bowman, 1982; Göksu Ögelman, 1986; Huxtable, 1982; Mercier *et al.*, 1995a; Valladas, 1978, 1992) which, at other sites, have produced ages in good accord with ^{14}C (e.g. Boëda *et al.*, 1996; Göksu *et al.*, 1974; Roosevelt *et al.*, 1996; Valladas, 1992; Valladas and Valladas, 1987), ESR (Mellars and Grün, 1991; Mercier *et al.*, 1993; Schwarcz *et al.*, 1988; Schwarcz *et al.*, 1989), U-series on tooth (McDermott *et al.*, 1993) and calcite (Green, 1984; Green *et al.*, 1981), OSL of unheated quartz (Michab *et al.*, 1997; Roosevelt *et al.*, 1996; Smith *et al.*, 1990b), TL of calcite (Green, 1984; Huxtable and Aitken, 1986) and quartz in heated pebbles and ceramics (Valladas, 1992; Valladas and Valladas, 1987). These comparisons cover the period 4000–200,000 years, the oldest site being Pontnewydd Cave in north Wales (Green, 1984; Green *et al.*, 1981).

Although the dates for Layers D and E extend beyond this range, ages of ~230,000 and ~285,000 years have been reported also for burnt flints from Terra Amata in southern France (Wintle and Aitken, 1977) and Belvédère in the southern Netherlands (Huxtable and Aitken, 1986), respectively. Furthermore, the trapped electrons that give rise to the flint dating peak at 370–390°C have a lifetime in excess of 50 million years (Wintle and Aitken, 1977; Bowman, 1982) and appear not to suffer from anomalous fading (Bowman *et al.*, 1982; Huxtable and Jacobi, 1982; Valladas, 1985), so the practical upper limit to flint dating is governed not by signal instability but by dose-saturation. As methods to obtain reliable age estimates for flints approaching dose-saturation have been developed (Mercier, 1991; Mercier *et al.*, 1992), an unforeseen bias in palaeodose determinations would appear unlikely. Incomplete heating of flint in antiquity will yield an overestimate of the age, but analysis of the TL glow curves (Rowlett *et al.*, 1974) and application of the "plateau test" (Melcher and Zimmerman, 1977) provide a check on this problem. The plateau test is performed routinely in flint dating (Aitken, 1985; Aitken and Valladas, 1992), leading to the rejection of a significant fraction of collected flints (Mercier *et al.*, 1995a). Partial bleaching of flint by sunlight (Wintle and Aitken, 1977; Huxtable, 1981; Bowman and Sieveking, 1983) would reduce, rather than enhance, the TL signal and thus the age. To avoid the latter problem, flints should be at least 10 mm thick and be sealed immediately in a black bag after collection; as a further precaution, and to remove the "rind" irradiated by alpha and beta particles, the outer 2 mm is removed in the laboratory (Aitken, 1985; Huxtable and Jacobi, 1982; Mercier *et al.*, 1995a).

As regards the dose rate at Tabun, Mercier *et al.* (1995b) conducted a set of careful field and laboratory analyses of the flints and the surrounding sediments. The environmental dose rate due to sediments and cosmic rays is especially important in flint dating studies because of the low internal radioactivity of flint; at Tabun, the environmental dose rate constituted 42–83% of the total (Mercier *et al.*, 1995b). The external dose rate was deduced from two independent methods: thermoluminescence dosimetry (TLD) capsules (using $CaSO_4$:Dy as the dosimeter) buried in the cave deposits for one year, and high-resolution gamma spectrometry measurements of discrete sediment samples. The combination of these methods allowed a check on the homogeneity of radionuclide distribution in the cave deposit and any extant disequilibrium in the ^{238}U and ^{232}Th decay chains. Dose rates varied by a factor of two between adjacent units, with local "hot spots" of ^{40}K concentration being partly responsible. At the western Galilee site of Hayonim Cave, neutron activation analysis of sediments in hearth-dominated layers (from which burnt flints are collected) showed a similarly non-uniform distribution of dose rate (Mercier *et al.*, 1995c).

A condition of secular equilibrium prevailed in the ^{238}U decay series at Tabun, there being no evidence of significant uranium migration nor radon escape ($^{226}Ra/^{210}Pb$ activity ratios consistent with unity) at the present day (Mercier *et al.*, 1995b). The latter result is at odds with the 10–30% emanation of ^{222}Rn inferred from "sealed" versus "unsealed" thick-source alpha counting (Bowman, 1985). If the cave deposit only began to emanate radon after being disturbed during flint collection, then the activity of ^{210}Pb (half-life of ~22 years) determined by gamma spectrometry gives a better indication of the *in situ* ^{222}Rn activity than does thick-source alpha counting, which instead indicates the post-disturbance level of ^{222}Rn escape.

The validity of the external dose rates determined by Mercier *et al.* (1995b) is most convincingly shown by their application of the "isochron test" at Tabun (Fig. 4). This method allows the external dose rate to be deduced independently, from a plot of palaeodose versus internal dose rate for a suite of contemporaneous flints. The only requirements are that the flints have received a similar environmental radiation dose and possess a wide range of internal dose rates. The isochron age calculated for the flints is then based solely on their internal radioactivity, which is unaffected by uranium-series disequilibrium and variations in soil water content (Mercier *et al.*, 1995a) and thus avoids many of the complications that plague the determination of the external dose rate. The approach was first suggested for TL by Mejdahl (1982), and was demonstrated using a variety of minerals (alkali and plagioclase feldspars, and quartz) of different grain size (0.1–2 mm diameter) extracted from a burnt stone

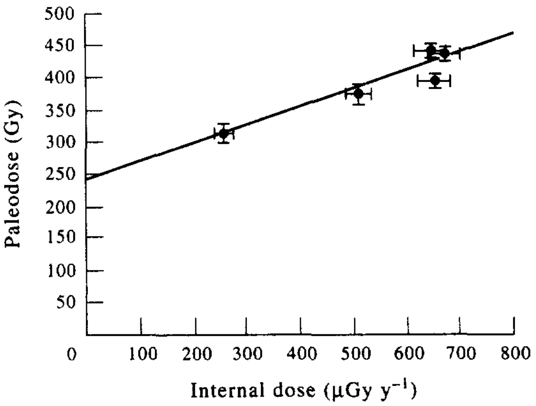

Fig. 4. Isochron plot for five burnt flints from Jelinek's Unit XI at Tabun Cave. The flint TL palaeodoses are plotted against their internal radioactivities, and a best-fit linear regression is made. The average age of the flints (287,000 ± 20,000 years) is calculated from the slope of the line, and the external dose rate (800 ± 90 µGy per year) is obtained from the intercept on the horizontal axis (from Mercier *et al.*, 1995b).

(Mejdahl, 1983). The average TL age of 916 years for six separate determinations closely matched the isochron age of 918 years, and the external dose rate measured by *in situ* spectrometry likewise agreed with the value inferred from the isochron analysis. A related approach, called the "subtraction technique", had been proposed previously for pottery (Fleming and Stoneham, 1973); with this technique, the difference in palaeodose measured from fine and coarse grains of quartz is due only to their different effective alpha dose rates, and the need for any knowledge about the environmental dose rate is eliminated.

The isochron test has since been applied to TL dates obtained from burnt flints at Qafzeh (Aitken and Valladas, 1992), Kebara (Mercier *et al.*, 1995a) and Tabun. For Jelinek's Unit XI (equivalent to Garrod's Layer E and dated to ~300,000 years), the external annual dose computed from TLD capsules and high-resolution gamma spectrometry (743 ± 42 μGy) accords closely with the estimate derived by isochron analysis (800 ± 90 μGy), and the age comparison was similarly encouraging (Mercier *et al.*, 1995a,b; Fig. 4). This result indicates that the present-day environmental dose rate is typical of the average dose rate for the last 300,000 years, so the possibility of the flint ages being distorted significantly by radionuclide leaching or ingress over this period can be discounted.

To explain the discrepancy between the seemingly reliable TL ages and the previous ESR determinations, Mercier *et al.* (1995b) compared the ^{232}Th/^{238}U ratios of the sediments that comprised the bulk of the deposit (used to calculate the TL dates) and the sediments that were adhered to the teeth used for ESR and U-series dating. This ratio is a relative measure of the heterogeneity of radionuclide distribution in sediments and, being dimensionless, can accommodate differences in absolute radionuclide concentrations; a related ratio (^{232}Th/^{230}Th) has been used to identify archaeological sediments derived from different sources (Olley *et al.*, 1997a). The sediments stuck to the teeth had generally lower ^{232}Th/^{238}U ratios than the "bulk" sediments, which they attributed to uranium enrichment in the vicinity of the teeth. The precipitated uranium will contribute an insignificant additional dose (for the observed relative enrichment), but will bias U-series dose rate determinations made from parental concentrations (e.g. by ICP-MS or neutron activation).

The ESR gamma dose rates were derived from neutron activation analysis of sediment attached to teeth, which would give an overestimate of the true "bulk" soil gamma dose rate and, thus, an underestimate of the ESR ages. This problem could have been avoided by using methods such as alpha and gamma spectrometry to directly measure the activities of ^{226}Ra and its decay products, which account for the majority of the U-series dose rate. Precipitation of uranium salts might be expected also in sediments adjacent to bone, owing to the local chemical environment created by decomposition of animal tissue (Mercier and Valladas, 1994). Consequently, the same caution applies to the determination of the U-series dose rate for sediments extracted from the bones of museum specimens for direct dating by single-aliquot luminescence methods. An additional consideration is that often only fine-grain sediments remain attached to teeth and bones in museum collections. As silts and clays tend to have a greater dose rate per unit mass than coarser fractions, then the *beta* dose rate in the original deposit may also be significantly overestimated (Mellars *et al.*, 1997).

Problems of sediment inhomogeneity should not, however, affect the mass-spectrometric U-series ages (McDermott *et al.*, 1993). To facilitate a corresponding increase in the latter, Mercier *et al.* (1995b) challenged the assumption of early-uptake of uranium by the teeth and postulated instead a linear-uptake model. This doubled the reported U-series ages and brought them into closer alignment with the flint ages. The dating discrepancies appear, therefore, to be resolved if early-uptake ESR and U-series ages at Tabun are viewed as minima (Mercier *et al.*, 1995b). This view is consistent with other comparative TL/ESR results from caves in the Levant (Es Skhul, Kebara, Qafzeh) and western Europe (Le Moustier), where TL ages agree at least as well with linear-uptake ESR ages as with their early-uptake counterparts (Mellars and Grün, 1991; Mercier *et al.*, 1993; Schwarcz *et al.*, 1988, 1989; Stringer *et al.*, 1989; Valladas *et al.*, 1986, 1987, 1988).

TL dating of sediments does, on the other hand, raise concerns about the validity of the flint dates for Tabun Cave. Two sediment samples were collected from Layer D and a mix of fine-grain minerals was extracted for TL dating (Bowman, 1985). The Tabun sediments are dominated by quartz and clay minerals (Grün *et al.*, 1991) and *quartz* (rather than feldspar) appeared to be the dominant TL mineral, yielding palaeodoses of ~240 Gy (sample TBN 4) and ~390 Gy (sample TBN 5) from a combined additive-dose and regenerative-dose procedure. Bowman (1985) was reluctant, however, to calculate their corresponding ages, owing to the extent of radon escape inferred from thick-source alpha counting. But as discussed above, the high-resolution gamma spectrometry, TLD capsule, and isochron-derived estimates of the environmental dose rate calculated by Mercier *et al.* (1995b) do not indicate significant ^{222}Rn emanation, either at the present day or during the past 300,000 years. Moreover, the "unsealed" count rates of the Layer D samples examined by Bowman (1985) correspond to uranium and thorium dose rates (using the conversion factors of Nambi and Aitken, 1986) similar those calculated by Mercier *et al.* (1995b); her potassium concentrations are likewise comparable.

Bowman's "unsealed" thick-source alpha counts and K_2O data (combined with a mean water content of 27.5% and an annual cosmic-ray dose of 70 μGy, as reported by Mercier *et al.* (1995b), and an assumed alpha-efficiency "*a*" value of 0.1) yield effective annual doses of 2.23 mGy (TBN 4) and 3.46 mGy (TBN 5). The corresponding TL dates for Layer D are ~108,000 and ~113,000 years, respectively. These dates are remarkably close to the mass-spectrometric U-series and early-uptake ESR dates of 110,700 ± 900 and 122,000 ± 20,000 years, respectively (Grün *et al.*, 1991; McDermott *et al.*, 1993), but are only half as old as the TL ages on burnt flint (Mercier *et al.*, 1995b). I cannot resolve this difference to my satisfaction. The occurrence of anomalous fading in these samples was not discounted by Bowman (1985), who measured fading of 5 ± 5% after a 12-day delay between irradiation and heating, but quartz does not generally suffer from severe fading (Aitken, 1985; Fragoulis and Readhead, 1991; Wintle, 1973).

A complex site stratigraphy may provide an alternative explanation. Mercier *et al.* (1995b) collected flints from Jelinek's excavation in the *intermediate* chamber of Tabun Cave, whereas the ESR and U-series age determinations were made on teeth collected from Garrod's excavation of the *outer* chamber; the provenance of Bowman's samples is not specified. Certainly, the cave deposits at Tabun have been periodically eroded and deformed (Jelinek, 1982), with the sedimentation being described as "chaotic" in places (Grün *et al.*,

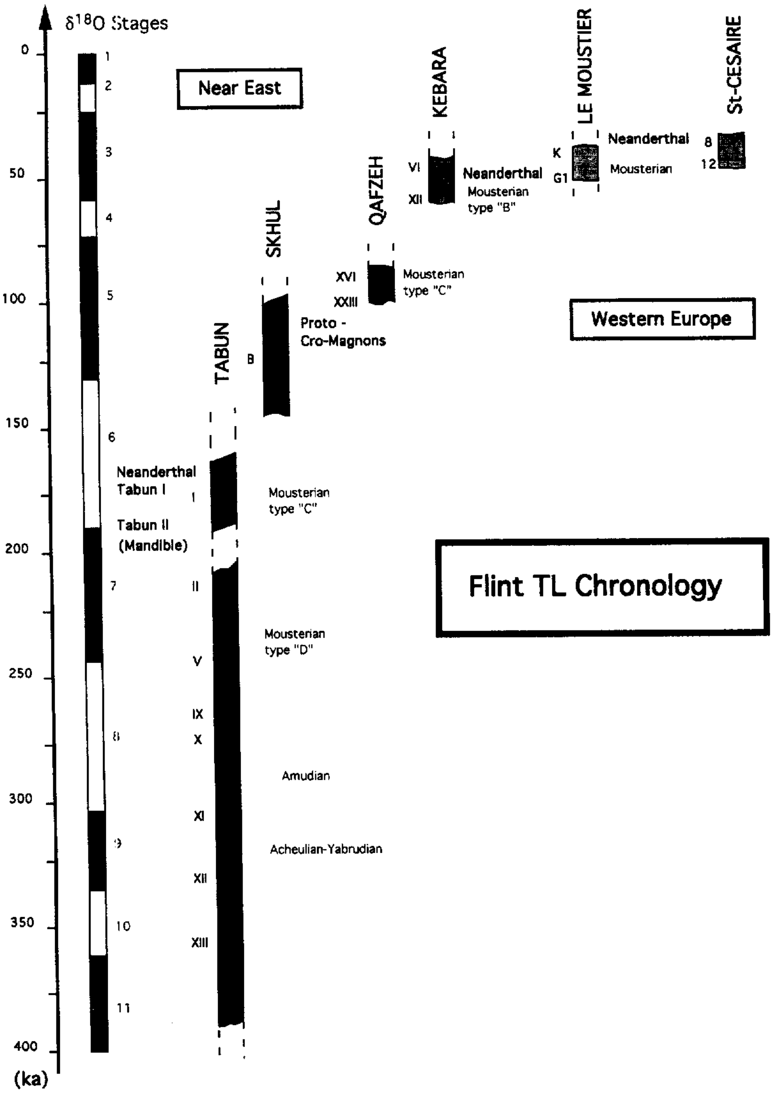

Fig. 5. Summary diagram of the flint TL chronology constructed for some key Palaeolithic sites in the Levant and western Europe. The annotations to the left of each bar indicate the archaeological levels, and the oxygen isotope scale is shown at far left. The time scale is in thousands of years (from Mercier *et al.*, 1995a).

1991, p. 233). Perhaps none of the numerical age estimates are faulty, their apparent discrepancy faithfully recording local stratigraphic complications hitherto unrecognised in the cave deposit. Differences in the ^{232}Th/^{238}U ratios of the sediments noted by Mercier *et al.* (1995b) may likewise reflect variations in radionuclide abundances between the outer and intermediate chambers. Maybe a final attempt should be made to date the sediments in *both* cave chambers, applying OSL methods to *both* the fine-sand and silt-sized quartz fractions.

I have discussed Tabun Cave at length for several reasons: it is a pivotal archaeological site for the Levant; numerical ages have been estimated using a variety of dating methods and are much older than expected by the archaeologists; and it falls into the rare category of cases where TL dating of flint appears to have produced a conflicting chronology to that obtained from ESR and U-series dating of teeth, and from luminescence dating of sediment. Despite the latter conundrum, the flint dating study at Tabun represents a fine example of the application of state-of-the-art TL methods, including isochron analysis, detailed geochemical investigation, and multiple methods of dose rate determination. These attributes have characterised the series of benchmark studies conducted by the flint dating team at Gif-sur-Yvette since their three iconoclastic papers were published a decade ago (Valladas *et al.*, 1986, 1987, 1988). An excellent summary account of these and their subsequent TL investigations of burnt flint is given by Mercier *et al.* (1995a), and their results for the Levant and western Europe are illustrated in Fig. 5.

These Middle Palaeolithic dating studies have demonstrated that both Neanderthals and modern humans inhabited the Levant between ~120,000 and ~60,000 years ago, and are associated with the same (Mousterian) lithic industry. Burnt flints associated with Proto-Cro-Magnon remains in Israel were dated to 119,000 ± 18,000 years at Es Skhul (Mercier *et al.*, 1993) and 92,000 ± 5000 years (or 88,000 ± 9000 years by the isochron method) at Qafzeh (Aitken and Valladas, 1992; Valladas *et al.*, 1988). At the nearby cave site of Kebara, the layer containing the skeleton of an adult Neanderthal was TL dated to 59,900 ± 3500 years (or 57,000 ± 8000 years by isochron analysis) and the most recent unequivocally Mousterian layer was dated to ~51,900 years (Mercier *et al.*, 1995a; Valladas *et al.*, 1987). These first reported findings of a substantial (but not necessarily continuous) temporal overlap between Neanderthals and modern humans were supported by ESR dating (Schwarcz *et al.*, 1988, 1989; Stringer *et al.*, 1989), and provided the basis for discarding ideas of a descendant evolutionary relationship between the two hominid types.

3.2.2. *Western Europe*. A radically different situation was revealed for western Europe. Evidence of modern humans here extends back only 30,000–40,000 years (Stringer, 1988; Stringer and Grün, 1991) and the remains of Neanderthals and modern humans do not occur in association with the same lithic industry (Mercier *et al.*, 1995a). The youngest dates for Neanderthal remains are those from the French sites of Saint-Césaire, Arcy-sur-Cure and Le Moustier (Fig. 5). At the latter site, TL ages of *circa* 40,300–42,500 years were obtained for layers that had yielded Mousterian tools and a Neanderthal skeleton (Valladas *et al.*, 1986), and these dates were later confirmed by ESR (Mellars and Grün, 1991). The most recent Neanderthal remains are found, however, in association with Upper Palaeolithic Châtelperronian tools at Saint-Césaire and Arcy-sur-Cure. TL dating of burnt flints from Saint-Césaire produced an age of 36,300 ± 2700 years for the hominid-bearing layer (Mercier *et al.*, 1991), and an uncalibrated ^{14}C age of ~33,800 years BP has been obtained from Arcy-sur-Cure (Hedges *et al.*, 1994; Hublin *et al.*, 1996). These two dates are comparable, if allowance is made for calibration of the ^{14}C timescale (e.g. Bard *et al.*, 1990, 1993).

In comparison, Upper Palaeolithic Aurignacian tools (thought to be produced by fully modern humans) appeared as early as 40,000 years ago in western Europe. At El Castillo Cave, L'Abreda Cave and Abric Romaní in northern Spain, uncalibrated AMS ^{14}C dates of *circa* 38,500–39,700 years BP (Bischoff *et al.*, 1989; Cabrera Valdes and Bischoff, 1989; Hedges *et al.*, 1994), ESR dates of ~39,900 years (calculated using "one-group" theory for beta dose attenuation; Rink *et al.*, 1996) and mass-spectrometric U-series dates of ~42,600 years (Bischoff *et al.*, 1994) have been obtained for the basal Aurignacian levels (which contain artefacts but not human remains). Taken together with the TL, ^{14}C and ESR results for Saint-Césaire, Arcy-sur-Cure and Le Moustier, these dates suggest that Neanderthals co-existed with modern humans for several millennia in south-west Europe (Mercier *et al.*, 1991; Straus, 1989), having been the sole occupants of the continent since at least the last interglacial (Mercier *et al.*, 1995a).

TL dating of burnt flints has also demonstrated that the Mousterian sequence at Combe Grenal, a famous rock shelter in the Dordogne district of France, spans the period *circa* 44,000–63,000 years (Bowman and Sieveking, 1983) and, therefore, marginally precedes the nearby Le Moustier sequence (Mellars, 1986). For the "Charentian" Mousterian lithic industry in the Massif Central, flint dates suggested a *minimum* age of ~78,500 years, this being the earliest known occurrence of this type of industry in France (Raynal and Huxtable, 1989). However, the Mousterian site of Pech de l'Azé IV, in the Dordogne, yielded TL dates that the authors deemed to be "too low to be acceptable" (Bowman *et al.*, 1982, p. 368), although no physical causes could be identified. Their cautious view is contingent on the climatically assigned ages for the Mousterian layers being correct. Six of the seven dated flints gave ages of ~43,000 years, a result not in conflict with the abrupt Mousterian/Aurignacian transition recorded at *circa* 40,000–43,000 years in northern Spain (Bischoff *et al.*, 1988, 1989, 1994; Straus, 1989).

The Lower Palaeolithic site of Hoxne in eastern England provides another example of flint dates that *appear* to be younger than independent age estimates: the average TL date of 210,000 ± 20,000 years for two burnt flints (Bowman, 1993) differs markedly from the mean "modelled" ESR date of 319,000 ± 38,000 years (Schwarcz and Grün, 1993). The latter was calculated from combined ESR and U-series dating of two horse teeth that exhibited late uptake of uranium. The cause of the chronological discrepancy seems to lie in the estimates of the external dose rate: the ESR study used parental radionuclide concentrations in the sediments adhered to the teeth, whereas Bowman (1993) made *in situ* gamma spectrometry measurements on the deposits adjacent to the original excavation (made several years earlier). The calculated ESR external dose rate was only 65% of the on-site determination, the latter being broadly supported by laboratory analyses of the *in situ* sediments.

Radionuclides are, therefore, distributed non-uniformly at the site or the sediments stuck to the teeth are not representative of the majority of the deposit. The second possibility has been discussed above for Tabun Cave, but in contrast to Tabun (and other sites; Mellars *et al.*, 1997), the dose rate of the sediment attached to the Hoxne teeth is *less* than that of the bulk deposit. The TL and ESR chronologies can be brought into coincidence by using the same external dose rate, but an independent evaluation of the latter cannot be reliably obtained from isochron analysis of the two flints. Bowman (1993) was unsuccessful in her attempt to obtain a third chronology by TL dating of the fine-grain sediments: the sediment date (*circa* 145,000–170,000 years by my calculation, depending on dose rate assumptions) was lower than the burnt flint ages, a result in keeping with many reports of TL age shortfalls in > 100,000 year-old samples composed of fine-grain feldspars (Berger, 1994; Debenham, 1985; Wintle, 1990).

TL techniques have also been applied recently to a fourth site in the Dordogne, the stratified cave site of Grotte XVI, where baked *sediments* from a Mousterian fireplace were dated to ~60,000 years (Rigaud *et al.*, 1995). Another TL investigation of fireplaces is currently underway at Menez-Dregan 1, a collapsed sea cave on the southern coast of Brittany (Balter, 1995; Monnier *et al.*, 1994). This site has yielded evidence of a distinctive, archaic lithic industry (the "Colombanian") and the first use of fire by Europeans (possibly *Homo erectus*). TL is now being used to check the controversial ESR dates of *circa* 380,000–465,000 years obtained from burnt quartz pebbles and sediments. The ESR palaeodoses were determined using the Al centre (Falguères *et al.*, 1991), the absence of the E' centre providing an assurance that the samples had been heated in antiquity to above 500°C (Falguères *et al.*, 1994a). A possible hearth of similar antiquity has been excavated at Schöningen in Germany, together with numerous flint tools, bones of butchered mammals, and the world's oldest hunting spears (Thieme, 1997; Dennell, 1997). TL dating of the fire-baked sediments and burnt flints would be instructive as the site has not been dated by numerical age methods.

The first appearance of another developmental trait in human behaviour, the attachment of handles to stone tools using a mastic, has also been the subject of a recent TL dating study. At the open-air site of Umm el Tlel in Syria, the presence of bitumen on stone tools has been interpreted as evidence for the manufacture of hafting material by Middle Palaeolithic people (Boëda *et al.*, 1996; S. Holdaway, 1996). A minimum age for this technological innovation was established by TL dates of ~34,000 and ~36,000 years for burnt flints from two overlying layers, and by uncalibrated ^{14}C ages for these same layers of ~32,000 and ~34,530 years BP, respectively. The dates given by ^{14}C are younger by 1500–2000 years, a shortfall similar in magnitude to previous suggestions for this time interval which were based on comparisons with calendrical age methods (e.g. luminescence, ESR and U-series) and variations in geomagnetic intensity (see Section 2.2).

3.2.3. *Archaeosediment applications.* Compared with the rich human history that TL dating of anthropogenically burnt objects has revealed for Eurasia, TL and optical dating of archaeosediments have so far played a minor role. An early attempt to date wind-blown archaeosediments was made at Plaidter Hummerich in north-west Germany, using fine-grains and a regenerative-dose procedure (Singhvi and Wagner, 1986; Singhvi *et al.*, 1986b). The *in situ* remains of a Neanderthal, together with stone artefacts, had been discovered in loess deposits, beneath a humus layer estimated to be ~80,000 years old. The archaeological horizon was underlain by loess dated by TL to ~135,000 years, so an age bracket of 80,000–135,000 years was proposed for the Neanderthal. Given the propensity for anomalous fading in feldspars (Spooner, 1994b), and the fading of ~15% exhibited over a nine-week period by a second sample from the ~135,000 year-old horizon (Singhvi *et al.*, 1986b), these ages should perhaps be regarded as minima.

This precaution was recommended by Zöller *et al.* (1991), who observed anomalous fading over a nine-month period in fine grains extracted from loess deposits at two Middle Palaeolithic sites in the Middle Rhine valley of Germany. TL ages were determined, using a regenerative-dose procedure, for the open-air sites of Tönchesberg and Remagen-Schwalbenberg. The oldest archaeological horizon at Tönchesberg was bracketed by a minimum TL age of ~120,000 years and a maximum age of ~200,000 years (from $^{40}Ar/^{39}Ar$ laser dating of the underlying volcanic tephra, e.g. Bogaard *et al.*, 1987), indicating that archaic *Homo sapiens* inhabited this region during the harsh climatic conditions of the penultimate glaciation. At Remagen-Schwalbenberg, the loess layer containing transitional Middle/Upper Palaeolithic artefacts was TL dated to 31,300 ± 2600 years and this date was supported by two uncalibrated ^{14}C ages (on snail shell) of ~28,000 years BP, making it the youngest known lithic industry transition in Germany. Taken together with the contemporaneous occurrence of Aurignacian tools elsewhere in the region and in south-west Europe, these findings suggest that a "mosaic" pattern of cultural evolution prevailed at this time (Zöller *et al.*, 1991).

Optical dating has only recently contributed to the debate on the timing of stone tool transitions and modern human occupation of the Levant and western Europe. At Holon in Israel, red loamy soil containing Lower Palaeolithic flint tools has been dated by both OSL (on quartz) and IRSL (on feldspar) to ~220,000 years, a result in agreement with the age of ~215,000 years obtained from ESR dating of bovid teeth (Porat *et al.*, 1996). This date for a Late Acheulian lithic industry is consistent with the date of ~200,000 years for Acheulian handaxes at Pontnewydd Cave in north-west Europe (Green, 1984; Green *et al.*, 1981) and with the beginning of the Middle Palaeolithic in Africa by 200,000–230,000 years ago (Wendorf *et al.*, 1993, 1994). It is, however, much younger than the flint dates of 300,000–350,000 years obtained for the Acheulian-bearing units at Tabun Cave (Mercier *et al.*, 1995b). If the flint tools recovered at Holon had been burnt sufficiently, then comparative TL dates would be instructive; further collection of material would be impractical as the site is now built over, but the environmental dose rate could be determined from previous *in situ* gamma spectrometry measurements and radionuclide analyses of the sediment samples (Porat *et al.*, 1996).

Optical dating offers the prospect of dating the termination, rather than the initiation, of the Middle Palaeolithic on the island of Gibraltar, located at the western extremity of the Mediterranean Sea. Middle and Upper Palaeolithic stone artefacts are present at Gorham's Cave and a variety of lithics occur in Vanguard Cave, deposited in a matrix of silt, sand and raised beach sediments (Rees-Jones *et al.*, 1997). The coarse-grain quartz component of these deposits has been investigated using OSL and varying the width of the stimulation waveband. Palaeodoses were determined using the multiple-aliquot additive-dose method, but no dates were calculated. For one of their samples (GOR1), the two narrowest wavebands yielded palaeodoses of 47 ± 7 Gy (514.5 nm from an Ar-ion laser) and 40 ± 15 Gy (514 ± 17 nm from a filtered halogen lamp), both of which differ markedly from the palaeodose of 13 ± 5 Gy obtained using a broader halogen-lamp passband (440–560 nm).

Their observation contrasts with my own for an Australian quartz sample (K166) examined in an identical manner (i.e. multiple aliquots, the additive-dose method, integration of the total light sum (minus background), saturating exponential curve fits (with each aliquot weighted by the inverse square of its OSL intensity), and a preheat of 220°C for 300 seconds). This sample gave a TL palaeodose of 23.1 ± 1.0 Gy (no preheat, 270–430°C plateau region), and OSL palaeodoses of 24.6 ± 0.7, 24.6 ± 1.5 and 22.9 ± 1.9 Gy using the 514.5 nm laser line and wavebands of 500 ± 40 nm and 420–550 nm, respectively (Roberts *et al.*, 1994a and unpublished data). The degree of scatter among normalised aliquots is much smaller, and the sensitivity to radiation is much greater, in the Australian quartz, resulting in palaeodose uncertainties of 3–8% for K166 compared with 15–38% for GOR1. Nevertheless, the quartz OSL exhibited by the Gibraltar coastal cave deposits appears sufficiently reproducible and dose-responsive to encourage dating of hominid activities and lithic industries at one of the most likely points of entry for early hominids into Europe (Dennell and Roebroeks, 1996).

The potential for optical dating of *unheated*, as well as burnt, flint tools has also been investigated recently (Poolton *et al.*, 1995). Flakes and grains of unheated flint from France, Israel and Florida were tested for their dose-response and OSL behaviour under 420–550 nm stimulation. The flints exhibited an extremely rapid decay of OSL when illuminated, and a growth curve was generated with added dose. The source traps for the OSL may be those associated with the TL peak observed at 180°C, no preheat having been administered. This peak showed no fading over a period of 24 h, but its long-term stability remains in doubt (given the 700-year lifetime of the 190°C trap in quartz; Aitken, 1985). The dissimilar behaviour of flint and sedimentary quartz was attributed to the existence of different crystal lattice defects in the two materials, despite both being composed largely of SiO_2 (Poolton *et al.*, 1995).

On the other hand, a rather more optimistic outcome has been reported for optical dating of quartzite pebbles from the archaeological site of Diring Yuriakh in Siberia (Richards, 1994). Quartz grains were extracted from the sun-bleached exteriors of the pebbles in an attempt to date them directly using OSL and IRSL methods, and then extend the method to quartzite artefacts. This study is discussed further in the next section, together with other contributions that luminescence dating has made to the chronology of human prehistory in the Orient.

3.3. *East Asia*

South-east Asia has vied with Africa as the favoured location for the birthplace of *Homo* since Alfred Russel Wallace returned to England in 1862, after 8 years in the Malay Archipelago, and persuaded Charles Lyell that "our progenitors" might be discovered in the caves of Borneo (Desmond and Moore, 1991, p. 523). The island of Borneo lies on the Sunda Shelf, the name given to the emergent landmass which existed to the east and south of the Malay Peninsula when sea-level has been lower; Borneo, Sumatra and Java were connected to the mainland, while the Philippines, Sulawesi and the Indonesian island chain as far east as Timor were separated by short water crossings. These water barriers, although narrow, have been sufficient to restrict the movement of land animals and birds, resulting in distinct faunal distributions on either side of "Wallace's Line" (Wallace, 1860; van Oosterzee, 1997). Borneo, Sumatra and Java lie to its west, while to the east are Sulawesi and the chain of islands stretching from Lombok to Timor. Still further east lies the continent of Australia and the islands of Irian Jaya/Papua New Guinea, which have been connected by dry land (the Sahul Shelf) at times of lower sea-level. Human colonisation of Sahul is discussed later; this section is concerned with the chronology and taxonomy of hominids in the Sunda region.

3.3.1. *The Malay Archipelago.* In the early 1890s, the Dutch palaeontologist Eugene Dubois discovered the remains of *Homo erectus* (then called *Pithecanthropus erectus*) in Java (Dubois, 1894). These fossils were promoted as the "missing link" between humans and apes, and placed this region at the forefront of the debate on hominid evolution (Lewin, 1989). The anthropological significance of south-east Asia has since been maintained, boosted by recent revelations of *Homo erectus* habitation on Java from as long ago as 1.8 million years (Swisher *et al.*, 1994) or one million years (de Vos and Sondaar, 1994), to as recently as 27,000–53,000 years ago (Bartstra, 1988; Bartstra *et al.*, 1988; Swisher *et al.*, 1996). These dates have been derived from the application of $^{40}Ar/^{39}Ar$, ESR, U-series, fission-track and palaeomagnetic methods, but luminescence dating could be used to provide additional chronological control on those sediments deposited in the last 800,000 years. For example, optical dating of the Solo River terraces in Java could show directly if the fluvial deposit and late Pleistocene *Homo erectus* fossils are contemporaneous, or if the fossils are reworked from an older deposit (as some have argued, see Gibbons, 1996c, and Grün and Thorne 1997). It could also provide an independent check on the validity of (a) the uranium-uptake models used to derive the ESR ages (which increase by 50–70% from ~27,000 years to 40,000–46,000 years under assumptions of early-uptake and linear-uptake, respectively) and (b) the application of "one-group" theory to model beta dose attenuation in enamel, which increases the quoted ESR ages by ~4000 years (Swisher *et al.*, 1996).

Homo erectus is generally thought to have lacked the necessary intellectual, linguistic and technological capacity to make sea voyages across Wallace's Line, but contrary evidence has recently emerged with the discovery of flaked stone tools on the island of Flores (Morwood *et al.*, 1997; Sondaar *et al.*, 1994). The artefacts were recovered from a fluvial sand and silt layer, whose age is estimated to be just less than 780,000 years, based on the presence of a palaeomagnetic reversal (presumed to be the Brunhes/Matuyama boundary) in the underlying deposits. If *Homo erectus* was able to make such sea crossings, then perhaps this species arrived in Australia before *Homo sapiens*, and at a much earlier date than either the currently accepted figures of 40,000–60,000 years (Allen, 1989, 1994; Roberts *et al.*, 1990a, 1994a) or the more controversial claims of *circa* 120,000–180,000 years (Fullagar *et al.*, 1996; Kershaw, 1994; Singh and Geissler, 1985).

Before such far-reaching questions are considered, however, the artefact-bearing layer on Flores must be dated directly or be bracketed by reliable age determinations. A variety of numerical dating methods are now being applied to this site: luminescence dating of the fluvial artefact-bearing sediments (whose expected age lies close to the limit of reliability claimed for current luminescence methods, e.g. Berger *et al.*, 1992; Huntley *et al.*, 1993a, 1994a,b), ESR dating of associated animal teeth, and fission-track dating of overlying and underlying volcanic tuffs. This latter study has been completed recently (Morwood *et al.*, 1998), and indicates zircon fission-track ages of *circa* 800,000–880,000 years for stone tools. This work also throws light on the role played by *Homo erectus* in the extinction of endemic island fauna, as the disappearance from the fossil record of giant tortoise and pygmy *Stegodon*, a type of elephant, appears to coincide with the human colonisation of Flores (Morwood *et al.*, 1998; Sondaar *et al.*, 1994).

3.3.2. *The Indian sub-continent.* Luminescence dating has already provided some important clues to the timing of human occupation of the Asian mainland. *Homo* may have colonised the Indian sub-continent as early as 2 million years ago, based on palaeomagnetic and stratigraphic analyses of a sedimentary sequence in northern Pakistan that has yielded *in situ* stone artefacts (Dennell *et al.*, 1988; Rendell *et al.*, 1987). These artefacts are similar in age and technological simplicity to other recent finds in Java (Swisher *et al.*, 1994) and China (W. Huang *et al.*, 1995; Larick and Ciochon, 1996), but confirmation of their great antiquity by numerical age estimates would be desirable. Luminescence methods have not been tested on these Plio-Pleistocene sediments but they have been applied to nearby late Pleistocene archaeosediments (Rendell and Dennell, 1987). TL dates of 24,000–64,000 years were obtained from fine-grain loess using the additive-dose method and, for one sample also, the "partial bleach" method (Berger, 1988; Wintle and Huntley, 1982). Anomalous fading was not detected over a 4-week period, but concerns were raised about contamination of the older loess units by poorly bleached sediments derived from local gullying. A conservative age of 42,000–45,000 years was thus proposed for the associated flaked stone tools (including blades) and the remains of a small structure, which may have been used for the processing of animal hides (Rendell and Dennell, 1987).

Remarkably few applications of luminescence dating to archaeosediments have been made in India, despite its strategic location between Africa and East Asia and the diverse types lithic industry that are poorly dated. The earliest appearance of Acheulian tools is presently placed at *circa* 540,000–670,000 years in the Pune District of west-central India (Mishra *et al.*, 1995, Mishra and Rajaguru, 1996). This figure is based on fission-track, K–Ar and ^{40}Ar/^{39}Ar ages (Horn *et al.*, 1993; Mishra *et al.*, 1995), and minimum U-series ages of 350,000 years (Mishra and Rajaguru, 1996), for a tephra that is overlain by an artefact-bearing fluvial gravel unit. Acheulian handaxes of similar antiquity had been reported previously from northern Pakistan (Rendell and Dennell, 1985), their age being constrained by the underlying Brunhes/Matuyama palaeomagnetic boundary at ~780,000 years (Shackleton *et al.*, 1990; Spell and McDougall, 1992; Tauxe *et al.*, 1992) and the onset of folding of the artefact-bearing strata at ~400,000 years.

The Indian dates and previous stratigraphic interpretations have been challenged, however, by the correlation of the tephra mineralogy, rare earth and trace element chemistry, and shard morphology with the ~74,000 year-old Youngest Toba Tuff in Indo-Malaysia (Shane *et al.*, 1995). Luminescence methods have also entered this debate by providing a TL date of ~23,000 years for the tephra (Horn *et al.*, 1993), suggesting that this unit was last reworked in the late Pleistocene. Bias in the earlier age determinations, as well as the stratigraphic inconsistencies, are consistent with fluvial reworking and detrital contamination of the tephra (Shane *et al.*, 1995, 1996). This study shows how luminescence dating of sun-bleached sediments can provide a valuable check on the stratigraphic integrity of a deposit whose age has been inferred using methods that give the time of mineral formation and not necessarily the most recent episode of deposition. The same concerns have been expressed in connection with the early hominid sites in Java (Swisher *et al.*, 1994), where ^{40}Ar/^{39}Ar dating of hornblende crystals will overestimate the true age of the fossils if the crystals have been reworked from older deposits (Gibbons, 1994b); the cystals would then date the volcanic event that reset the argon "clock" but not the target event of archaeological interest.

Acheulian tools have not been found in Sri Lanka (Kennedy and Deraniyagala, 1989), but TL dating of a later lithic tradition has proved feasible at Beli Cave (Abeyratne, 1994). This rainforest cave has yielded human remains from a level previously dated by ^{14}C to ~12,000 years BP (Kennedy and Deraniyagala, 1989), while geometric microliths have been recovered from layers with ^{14}C ages of 3000–27,000 years BP (Abeyratne, 1994). These ^{14}C ages, obtained on charcoal, were broadly confirmed by TL dating of quartz grains extracted from heated sediments, although the four Pleistocene TL dates were consistently younger than their uncalibrated ^{14}C counterparts (by 1000–3000 years, and calibration would increase this disparity). The lack of *in situ* gamma dose rate estimates may be partly responsible, if the non-uniform distribution of dose rate in the hearth-dominated layers at Hayonim Cave (Mercier *et al.*, 1995c) is any guide; so too may the repeated use of fires which would reset the TL (but not the ^{14}C) "clock" on each occasion.

The latter was considered as a possible explanation for the OSL age underestimation observed at Batadomba Cave in Sri Lanka, where ^{14}C ages on charcoal, shell and bone, and ESR ages on shell and bone, had also been obtained (Abeyratne *et al.*, 1997). The most self-consistent suite of results was generated from ^{14}C dating of charcoal and optical dating of heated quartz sediments. The OSL chronologies are, however, systematically younger than the calibrated ^{14}C ages; the greatest discrepancies (of 7000–12,000 years) occur in the lower, intensively fired levels, which were ^{14}C dated to *circa* 23,000–31,000 years. The basal level at Batadomba Cave yielded geometric microliths and human remains coated with yellow pigment (Kennedy and Deraniyagala, 1989). Given the calibrated ^{14}C age of ~31,000 years and two OSL ages of ~19,000 and ~24,000 years for this level, geometric microliths appear to have predominated at an earlier date in southern Asia than in Europe, where they did not rise to prominence until after the Last Glacial Maximum (Gamble, 1986, p. 120).

Further support for the antiquity of microlithic technology in Sri Lanka is given by the TL dates of *circa* 22,700–28,300 years obtained for *in situ* microliths in the coastal dunes at Bundala (Singhvi *et al.*, 1986a). This study is also notable in two other respects. First, it demonstrated that red iron-oxide coatings on quartz grains can dramatically reduce the bleaching of the TL signal at both the easy-to-bleach (325°C) and hard-to-bleach (375°C) peaks; exposing red-stained grains to a sunlamp for 1000 min resulted in only 30% and 20% reductions in the TL signals at 325°C and 375°C, respectively. (It should be remarked, however, that the residual TL at 325°C is probably composed mostly of the low-temperature "tail" of the 375°C signal, and that the "pure" 325°C (and hence OSL) signal may have been erased completely.) Second, it considered the possibility that uranium may be precipitated with iron in the red coatings, resulting in a time-dependent change in the dose rate as the coatings thicken with burial time. Singhvi *et al.* (1986a) concluded that the major effect of uranium precipitation would be on the external alpha dose rate, which is mostly removed during etching of the grains with HF acid. They also showed that if the red coatings were acquired *after* deposition of the grains, then reliable TL ages can be obtained from the application of "total bleach" methods to sand-sized quartz grains prepared in the standard manner (the stains being removed during acid-etching). Grains that were transported and deposited with an existing red coat, however, are apt to have been incompletely zeroed in antiquity, resulting in gross age overestimates using "total bleach" TL methods. Optical dating should be more accurate and, furthermore, as every grain in a sample is unlikely to be coated to the same thickness, single-aliquot and single-grain techniques should produce a broad distribution of palaeodoses if the grains were not bleached uniformly, as has been reported for other partially bleached sediments (e.g. Duller *et al.*, 1995; Li, 1994; Murray *et al.*, 1995; Olley *et al.*, 1997b; Wintle *et al.*, 1995).

3.3.3. *China*. The fossil record of human evolution in the more northern parts of Asia is poorly constrained by numerical age estimates. The presence of *Homo* (a more archaic form than *Homo erectus*) in southern China has recently been dated to 1–2 million years (W. Huang *et al.*, 1995), an age consistent with early hominid sites in Java and Pakistan. The ages are derived from the palaeomagnetic profile and ESR dating of tooth enamel at Longgupo Cave, where primitive stone tools have been found in association with fragmentary hominid remains and the teeth of *Gigantopithecus*, a large primate. Hominids appear to have coexisted with *Gigantopithecus* for more than one million years in south-east Asia, as *Homo erectus* and *Gigantopithecus* fossils are found together at Tham Khuyen Cave in Vietnam, in levels dated by ESR to 475,000 ± 125,000 years (Ciochon *et al.*, 1996; W. Huang *et al.*, 1995).

In northern China, a variety of numerical dating methods have been employed to provide a reliable chronology for the famous "Peking Man" site at Zhoukoudian. This cave site has yielded skulls, bones and teeth of *Homo erectus*, more than 100,000 stone tools, the remains of fossil animals, and claimed evidence (in the form of ash and hearth lenses) for the use of fire by prehistoric humans (Black *et al.*, 1933; Jia and Huang, 1990). A group of Chinese geochronologists have recently published a multi-method dating comparison for this important site, including a single TL date of *circa* 290,000–310,000 years for a baked hearth in Layer 4 (Huang *et al.*, 1993). This date agrees well with the ESR, U-series and fission-track dates for this layer, although the reliability of the TL age determination cannot be gauged as no procedural details are given. The lowest ashy layer, interpreted as a hearth, occurs at the boundary of Layers 9 and 10, the latter having a TL date of 420,000–590,000 years (cited in Chen and Zhang, 1991, again with no supporting data) and fission-track and amino-acid racemization dates of ~460,000 years. The most complete skull found at this site was retrieved from Layer 11, whose age is estimated to be 460,000–585,000 years (using ESR and fission-track methods). These deposits lie within the age range of luminescence methods and a new study has begun to date them using OSL for the unburnt sediments and TL for the baked hearth sediments; ESR dates on tooth enamel will be available for comparison (Huang and Grün, 1996).

Luminescence methods also have unfulfilled potential to date the transition between *Homo erectus* and *Homo sapiens* in China. Several uranium-series and ESR dating studies suggest that this transition took place between 200,000 and 350,000 years ago, with the possibility of coexistence of the two species (Brooks and Wood, 1990; Chen and Yuan, 1988; Chen and Zhang, 1991; Chen *et al.*, 1994; Huang and Grün, 1996). Luminescence dating methods have so far contributed little to the chronology of this crucial period of human prehistory, but they should be applied more often to check the validity of the U-series and ESR age determinations, especially given the detection of widespread "open system" uranium migration in fossil bones and teeth (Chen and Yuan, 1988).

The Hexian Man site in Anhui Province, east-central China, has yielded fossils of late *Homo erectus* and has been TL dated to less than 200,000 years (cited in Brooks and Wood, 1990 and Chen and Zhang, 1991). No details are given, however, to allow a check of the internal consistency of the TL dating procedure, although the age is not dissimilar to those obtained from associated fossil teeth by U-series (150,000–200,000 years: Brooks and Wood, 1990; Chen and Yuan, 1988) and ESR

(170,000–400,000 years: P.-H. Huang *et al.*, 1995; Huang and Grün, 1996). The cave sediments at this site have, unfortunately, been excavated completely and none remain for further TL or OSL dating (P.-H. Huang, Personal Communication, 1994). The same is not true at other cave sites, such as that near Nanjing where two skulls attributed to *Homo erectus* were discovered in 1993 (Etler, 1996); sediments and teeth have recently been collected from this site for optical and ESR dating. Another important site is that of Yunxian Man, where two skulls with a mix of *Homo erectus* and archaic *Homo sapiens* features were discovered recently on a terrace of the Han River (Li and Etler, 1992). The age of the terrace is estimated to be *circa* 580,000–850,000 years, based on palaeomagnetic analysis of the sediments and ESR dating of associated teeth (Etler, 1996; Chen *et al.*, 1997). To refine this age estimate, luminescence dating of the terrace sediments has begun.

Much also remains to be learnt about the timing of the later palaeolithic sequences in China. Some U-series ages have been obtained from sites with fossil animal remains, but the geochronologists responsible have warned that several of these may not be reliable owing to secondary mobilisation of uranium, particularly in samples from open-air sites on river terraces (Chen and Yuan, 1988). Luminescence dating of archaeosediments can play an important role at such open-air sites. For example, *in situ* stone tools were discovered in 1987 at Xiangyang, on an abandoned terrace of the Shuiyang River in Anhui Province (Fang *et al.*, 1992). ESR dating of the artefact-bearing quartz sediments has produced ages of 300,000–800,000 years for a 10 m-thick section composed of loess and alluvium (Fang *et al.*, 1992; P.-H. Huang, Personal Communication, 1996). The well-known ESR signals in quartz cannot be reset completely by sunlight (Brumby and Yoshida, 1994; Schwarcz, 1994) and only a few studies have reported ESR dating of solar-reset sediments based on these incompletely-bleached signals (e.g. Laurent *et al.*, 1994; Tanaka *et al.*, 1995; Yokoyama *et al.*, 1985). It is likely, therefore, that these age determinations are maxima, so luminescence dating methods have now been deployed at this site. TL dates for other palaeolithic sites along the Shuiyang River are cited by Fang *et al.* (1992), but procedural details are not provided. Sites throughout this region of central China also warrant further investigation, given its strategic location on the boundary between the distinctive north and south Asian lithic industries (Fang, 1994).

3.3.4. *Siberia.* Palaeolithic cultures in the far north of Asia have also come under the luminescence "spotlight" recently. Siberia is a key region in terms of human migration from Asia into North America, across the Bering land bridge that only reverted to a permanent seaway at ~11,000 years BP (Elias *et al.*, 1996). The oldest "Palaeoindian" sites in Alaska have been ^{14}C dated to almost 12,000 years BP (Hoffecker *et al.*, 1993; Kunz and Reanier, 1994), and a fluted bifacial point (a technology thought to be restricted to the Americas) has been discovered in north-eastern Siberia, beneath a tephra dated by ^{14}C to ~8300 years BP (King and Slobodin, 1996); a maximum age for the fluted point, and thus its temporal relation to lithic cultures in North America, could be established by luminescence dating of the sand deposit on which the artefact rested.

As with the date for human colonisation of the Americas (the subject of the next section), the time of initial human arrival in Siberia remains a matter of fierce debate. Some archaeologists have claimed ^{14}C dates of up to 45,000 years BP for palaeolithic sites in Siberia (see review by Dolitsky, 1985), whereas a recent evaluation of ^{14}C dates for the region place the earliest human occupation at 12,000–14,000 years BP (Kuzmin and Tankersley, 1996). Mitochondrial DNA data support the view that north-east Asia provided the founding population for North America, but indicate that probably only one "wave" of people (rather than the four waves proposed by linguists) emigrated from Siberia, at *circa* 20,000–25,000 years ago (Gibbons, 1996b).

Into this debate has stepped luminescence dating. In the mid 1980s, TL dates of 1.5 million years (with an uncertainty of ±25%) and ~2 million years were claimed by Y. Mochanov for the sites of Ulalinka and Diring Yuriakh, respectively (Ackerman and Carlson, 1991; Dolitsky, 1985, pp. 364 and 375; Mochanov, 1992). The great antiquity that these TL dates bestowed on the associated pebble tool industry led to much controversy, and the anthropogenic origin of the Ulalinka "artefacts" has been challenged. The details of the dating methods and results are not available in English, but as the dates lie well outside the demonstrated limit of reliability (~800,000 years) for current TL dating practices, they should still be regarded as questionable. Recently, however, the Diring Yuriakh site on the Lena River has been re-examined using optical methods on quartzite pebbles (Richards, 1994) and TL methods on the artefact-bearing sediments (Morell, 1994; Waters *et al.*, 1997a,b). These new results, supported by full documentation of the experimental procedures, suggest an age in excess of 110,000 years—an order-of-magnitude greater than the currently accepted chronology for human occupation based on ^{14}C determinations.

Over 4000 artefacts have been collected at Diring Yuriakh, from an occupation surface which is buried beneath a variable thickness of sediment (Fig. 6). Waters *et al.* (1997a,b) provide evidence that the covering deposit is composed of aeolian sand and

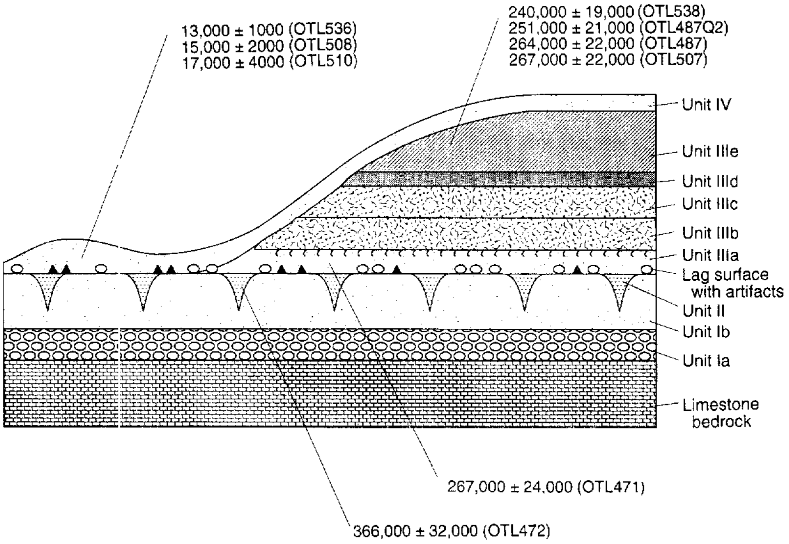

Fig. 6. Generalised cross-section of the stratigraphy at Diring Yuriakh and the associated TL ages in years (and sample codes). Solid triangles indicate the lag surface on which stone artefacts have been found (from Waters *et al.*, 1997a. Copyright 1997 American Association for the Advancement of Science).

loess, in contrast to the suggestion made by the original excavator that the artefacts are buried by alluvium (Mochanov, 1992). The artefacts are mostly made from quartzite and are also the subject of dispute among archaeologists—a natural origin being argued by some (e.g. R. Klein in Holden, 1997), while others view them as the product of human hands (e.g. Ackerman and Carlson, 1991; Mochanov, 1992; Waters *et al.*, 1997a,b). The latter group has argued that discrete clusters of unifacial choppers, cores and scrapers, flake debris, and large anvils occur on a "lag" surface of wind-abraded pebbles, produced by deflation of the underlying water-lain sands.

Owing to the lack of materials suitable for other numerical dating methods, luminescence dating was the sole technique available to determine the ages of the sedimentary units above and below the archaeological horizon. The fine-grain sediments were extracted from these units and their TL palaeodoses were determined using the additive-dose method (Waters *et al.*, 1997a,b). The TL in polymineral extracts is dominated by the signal from feldspars, but fine grains of quartz were additionally analysed for one sample. Given a subaerial mode of transport and deposition, Waters *et al.* (1997a,b) assumed that the TL signal had been reset in antiquity to a low "residual" level, which was estimated by exposing grains to both natural sunlight and an ultraviolet sunlamp. Tests for anomalous fading indicated little or no fading over periods of 1–6 months, but the TL ages would be too *young*, not too old, if long-term fading is a problem.

The environmental dose rates were deduced from thick-source alpha counting (uranium and thorium) and ICP emission spectrometry (potassium), with allowances made for the cosmic-ray dose rates and sample water contents. Measurements of the *in situ* gamma dose rate were not made but, fortunately, the annual doses show little variation ($<10\%$) within any particular aeolian unit (e.g. 2.96–3.14 mGy in Unit IV, and 3.68–3.99 mGy in Unit IIIe). The absence of significant ^{222}Rn escape was inferred from the "unsealed" versus "sealed" alpha count ratio of 0.96–1.01, but no checks were performed to detect other forms of disequilibrium in the ^{238}U chain. To overestimate the TL ages by an order-of-magnitude, however, requires that the measured (modern) dose rates be one-tenth the long-term average; neither the total loss of uranium in the distant past, nor the recent leaching of ^{226}Ra or emanation of ^{222}Rn, would produce a sufficient effect (Olley *et al.*, 1996). By my calculation, 50–70% of the total dose rate is derived from potassium, and doubling the entire U-series contribution would raise the total dose rates (and thus reduce the TL ages) by only 10–25%.

In terms of the TL measurements, the sand units immediately above and below the artefact horizon yielded palaeodoses of ~770 Gy and ~1150 Gy, respectively, corresponding to bracketing ages of 267,000 ± 24,000 years and 366,000 ± 32,000 years (Waters *et al.*, 1997a,b). The additive-dose growth curves and palaeodose plateaux are shown for both these key samples by Waters *et al.* (1997b). In both cases, the "natural" TL intensity is ~60% of the saturation intensity, so the dose-response curves require substantial extrapolations to intersect the "residual" level. Furthermore, the palaeodoses tend to rise with temperature across the glow curve region 250–400°C, rather than form a perfectly flat plateau. The palaeodoses used in the age calculations are conservative estimates, however, as they are biased towards the smaller palaeodoses associated with the TL peak at ~300°C; the residual TL levels are low (< 10% of the natural TL) at this temperature also, so the signal-to-noise ratios are optimal. Waters *et al.* (1997b) recognise the TL ages of these two samples may not be accurate, and instead suggest that the five TL dates obtained from the overlying loess (Unit IIIe) provide a reliable *minimum* age for the Diring Yuriakh artefacts.

These five dates range from 240,000 ± 19,000 years to 284,000 ± 24,000 years (the latter date is given in Waters *et al.*, 1997b, and is not shown in Fig. 6), and the dose-response curve and palaeodose plateau are illustrated for one of these samples. This sample shows a similar dose-response behaviour to the older pair discussed above, but the palaeodoses form an effectively flat plateau (at ~1000 Gy) from 250°C to at least 350°C (Waters *et al.*, 1997b), inspiring greater confidence in the age determination. Intriguingly, the fine-grain quartz extracted from this same sample gave an identical palaeodose of ~990 Gy. In my experience, this represents an extraordinarily large palaeodose for quartz grains, whether they be silt or sand sized (cf. Huntley *et al.*, 1993a, 1994a; Rees-Jones, 1995). The palaeodose plateau region for this sample (250–400°C) is also unlike that commonly reported for quartz; for a sample estimated to be ~250,000 years old, the partial or complete decay of the 280°C peak would yield a plateau that starts at ~340°C and, provided the hard-to-bleach 375°C peak had been fully reset in antiquity, extends to at least 450°C (e.g. Huntley *et al.*, 1993a).

It is possible that Waters *et al.* (1997a,b) have instead exploited the presence of micro-inclusions (presumably feldspars) within the fine-grain quartz, as has been reported for sand-sized quartz from South Australia (Huntley *et al.*, 1993b) and inferred for other silt-sized quartz data (see comments by D. Huntley following Rees-Jones, 1995). Support for this proposition is given by the findings of Richards (1994), who used the IRSL signal to date sand-sized grains of quartz extracted from the Diring Yuriakh pebbles. As quartz does not respond to infrared stimulation at room temperature (Short and Huntley, 1992; Spooner, 1994a), feldspar inclusions were deemed responsible for the IRSL signal, their presence being detected also by X-ray diffraction analysis (Richards, 1994).

The TL dates for Diring Yuriakh are spectacularly old, and require confirmation. If the overlying sediments were emplaced by water, rather than by wind, then it becomes critical to demonstrate that the sediments were well bleached at deposition; the TL ages might otherwise be considerable age overestimates. Optical dating of feldspars and feldspar inclusions in quartz grains, to exploit the rapidly bleached IRSL signal, would provide a useful check (W.J. Rink in Holden, 1997). The latter has been attempted at Diring Yuriakh by S. Forman (who found the IRSL signal was in dose-saturation; Gibbons, 1997c), and in a pilot IRSL dating study of the water-lain sands *beneath* the artefact layer (Hu, 1994). The lack of dependence of IRSL intensity on radiation dose precluded an estimate of the palaeodose from feldspar inclusions, but a minimum age of ~290,000 years was calculated for the same layer using coarse grains of potassium feldspar and assuming that these grains had been well bleached at deposition (Hu, 1994). It has been found, however, that even wind-blown sediments have not always been exposed to sufficient sunlight to reset the IRSL signal (Huntley and Lian, 1997; Lamothe and Auclair, 1997).

On the other hand, D. Huntley and M. Richards (Personal Communication, 1997) have cautioned that the Diring Yuriakh artefacts may be much *older* than the TL ages suggested by Waters *et al.* (1997a,b). They have argued that, provided the sediments received adequate solar exposure, then the TL ages measured by Waters *et al.* (1997a,b) should be treated as lower limits. This is based on the observation that feldspars suffer from long-term instability of the TL signal, resulting in substantial age underestimates (Mejdahl, 1988b). The presence of a palaeomagnetic reversal, interpreted as the Brunhes/Matuyama boundary (e.g. Alekseev *et al.*, 1990) or an earlier event (e.g. Ackerman and Carlson, 1991; Mochanov, 1992) in the sediments above the artefact-bearing layer suggests that the artefacts may be older than ~780,000 years. Although burnt flints are not available at Diring Yuriakh for comparative dating, steps toward luminescence dating of unheated quartzite artefacts have been made.

Richards (1994) made several innovative investigations on the feasibility of using TL and optical dating methods to measure the time since buried quartzite pebbles were last exposed to *sunlight*. For the Diring Yuriakh pebbles, an extended period of sub-aerial (and thus solar) exposure is indicated by their wind-abraded surfaces. Natural river pebbles were collected at night (by D. Huntley) from Diring Yuriakh, to characterise the luminescence behaviour of the local quartzite and to develop suitable dating procedures. In addition, Richards examined a pebble which had been excavated one year earlier and had since been exposed to sunlight on the ground surface. Initial petrographic and XRD analyses of three quartzite pieces revealed that two were composed wholly of quartz, whereas one also contained albite, a feldspar species which appears not to suffer severely from anomalous fading (Spooner, 1994b).

As the Diring Yuriakh pebbles are not translucent, only the external portion of each pebble would be suitable for optical dating of the solar-reset minerals. It was therefore necessary to adopt a "micro-stratigraphic" approach and remove grains from the surface of the pebbles and from increasing depths, to determine the maximum depth to which sunlight had penetrated (akin to the methodology of Liritzis, 1994). By etching the pebbles in 48% HF acid for successive periods of 30 min, Richards (1994) obtained a layer-by-layer suite of granular extracts, the silica matrix having been dissolved by the acid. He calculated that 10% of the mass removed was recovered as quartz grains, and that each "HF layer" corresponded to a thickness of ~250 μm.

The sand-sized grains from each HF layer were then examined for their TL, OSL and IRSL bleaching and dose-response characteristics. Although each quartzite behaved differently, OSL and IRSL decay curves were generated using 514 nm and 880 nm stimulation, respectively, and sunlight was

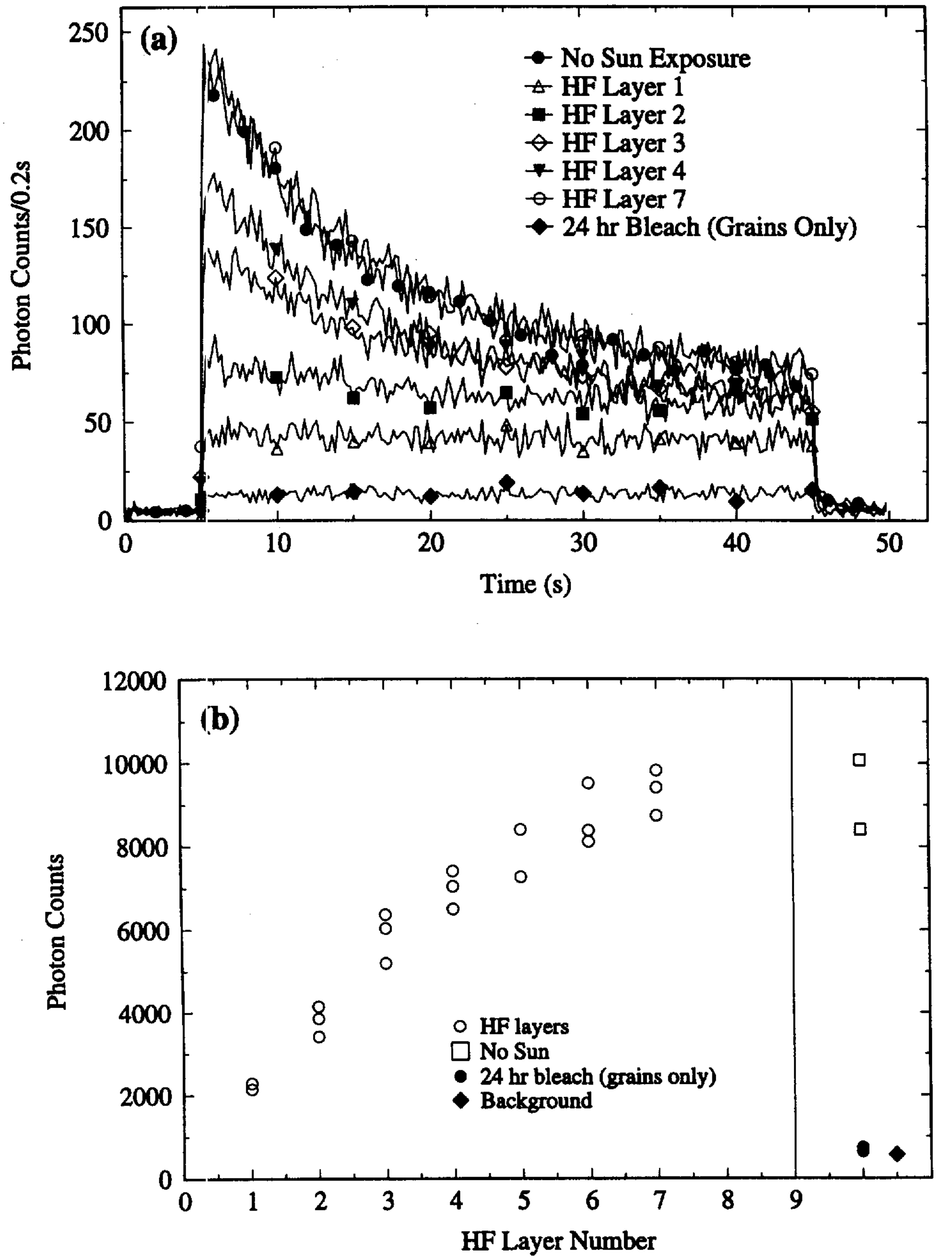

Fig. 7. (a) Shine-down curves for quartzite pebble DY-1d under 2.4 eV (514.5 nm) stimulation (22 mW cm^{-2} on 10 mg aliquots). Grains of 90–150 μm diameter were extracted from each "HF layer" after the pebble had been exposed to direct sunlight for 12.5 h and to cloudy skies for 5.5 h. The data for "no sun exposure" are from grains removed before the pebble was exposed to sunlight, and the "24 hr bleach (grains only)" data are from grains exposed to a sunlamp for 24 h. (b) The same data as in (a) but expressed as the sum of photon counts for the first 10 s of stimulation (from Richards, 1994).

found to be effective in bleaching the TL and OSL signals. For the purpose of dating the pebbles, however, it was necessary to establish the penetration depth of sunlight, so experiments were conducted with quartzite slices of varying thickness. For one sample, light transmission declined to 8% of the initial intensity using a 0.6 mm slice while, for another sample, the amount of light transmitted through a 0.25 mm slice was reduced to less than 1%. A similar result was obtained from a comparison of OSL signal intensities for individual HF layers removed from quartzite pebbles: the effect of 8–12 h of solar exposure was shown to extend through the outermost 6–8 layers (equivalent to 1.5–2 mm), with the remanent signal increasing with depth (Fig. 7). As the same treatment removed ~20% of the TL signal (at 350–400°C) in the outermost HF layer but had nil effect on the deeper layers, sun-bleached pebbles and artefacts should be dated using optical, rather than thermal, stimulation. The results of these laboratory experiments were supported also by OSL tests on an excavated pebble left exposed on the ground surface for one year. The top (exposed) surface of the pebble was bleached through to the eighth HF layer, whereas the underside of the pebble showed no bleaching of any of the outer five layers.

Richards (1994) then proceeded to examine a quartzite pebble from the archaeological horizon, using the single-aliquot additive-dose IRSL procedure of Duller (1991); regenerative-dose experiments on other quartzite pebbles had shown the OSL signal was too weak and unresponsive to different treatments, or the OSL data were too scattered, to make palaeodose determinations. Owing to the small size of the pebble, only 20 mg of 106–250 μm diameter grains could be extracted. Five aliquots were used, one as a "monitor" for signal erosion due to the repeated preheat (120°C for 16 h) and short-shine (5 s) stimulation cycles. The IRSL signal, however, remained constant with added dose, indicating the sample was in dose-saturation. A minimum palaeodose of ~400 Gy was determined by comparing the "natural" IRSL signal of each aliquot with the corresponding signal regenerated after an initial 3 h bleach (using a quartz-halogen lamp fitted with a Schott RG-715 filter). This yields a *minimum* elapsed time of 110,000 years since the pebble was last exposed to sunlight, given a total annual dose of ~3.6 mGy (deduced from thick-source alpha counting, atomic absorption and neutron activation, with adjustments for cosmic rays, beta-dose attenuation and soil water content).

The minimum IRSL age for this pebble is consistent with the TL age determinations for the enclosing sediments (Waters *et al.*, 1997a,b). The archaeological horizon at Diring Yuriakh appears, therefore, to be reliably dated to at least 110,000 years, and possibly ~260,000 years or more. These luminescence dates will doubtless make as great an impact on the palaeolithic archaeology of northern Asia as TL dating has done in western Europe and the Levant. The early human occupation of Siberia also provides an increased time-frame for these first Siberians to travel across the Bering land bridge into North America. The earliest traces of human arrival in the Americas, and the chronological contributions made by luminescence methods, are considered in the following section.

4. CONTINENTAL AND ISLAND COLONISATIONS

4.1. *The Americas*

This paper began with a quote from Thomas Jefferson concerning the age of some archaeological structures in Virginia. His interests extended also to the antiquity of the indigenous American people (Jefferson, 1787), the so-called "Palaeoindians". The time of their arrival in the Americas, and the number of founding populations, is still being hotly contested two centuries later by archaeologists, linguists and geneticists (Bednarik, 1989; Bray, 1988; Gibbons, 1993, 1996b; Gruhn, 1988; Lynch, 1990; Marshall, 1990; Meltzer, 1989, 1995). Perhaps the only point of agreement is that the "Clovis" culture was present in North America by 11,000–11,500 years BP (e.g. Haynes, 1993; Holliday, 1997; Taylor *et al.*, 1996). A recent multi-method dating study of a Palaeoindian campsite in the Brazilian Amazon has shown that people were present also in South America by this time (Roosevelt *et al.*, 1996).

4.1.1. *Pre-Clovis colonisation.* Earlier human occupation has been claimed for sites such as Meadowcroft Rockshelter in Pennsylvania (Adovasio *et al.*, 1978; Adovasio *et al.*, 1990), Monte Verde in Chile (Dillehay and Collins, 1988), Guitarrero Cave in Peru (Lynch *et al.*, 1985), Taima-taima in Venezuela (Bryan *et al.*, 1978), and a numer of sites in Brazil, including Alice Boër (Beltrão *et al.*, 1982; Danon *et al.*, 1982), Pedra Furada (Bahn, 1993; Delibrias *et al.*, 1988; Guidon and Delibrias, 1986; Guidon and Arnaud, 1991; Guidon *et al.*, 1994) and Toca da Esperança (de Lumley *et al.*, 1988). At each of these sites, except the last, ^{14}C determinations have been made on organic remains. The reliability of these ^{14}C ages and their association with the cultural remains has been disputed at some sites (e.g. Meadowcroft Rockshelter; Tankersley and Munson, 1992, and rejoinder by Adovasio *et al.*, 1992), while the human manufacture of flaked stones has been queried at others (e.g. Pedra Furada; Meltzer *et al.*, 1994, and reply by Parenti *et al.*, 1996). The latter is an archaeological not a chronological issue, but luminescence dating has been deployed at remarkably few sites to check ambiguous ^{14}C dates and provide a more detailed chrono-stratigraphy.

Luminescence methods could be used to good effect at the river terrace site of Monte Verde, which Meltzer (1995) considered to be the most viable pre-Clovis candidate. The oldest cultural layer is sandwiched between fluvial sand and redeposited volcanic ash units, and has yielded one finite ^{14}C age of ~33,400 years BP and an infinite determination of > 33,000 years BP (Dillehay and Collins, 1988). Given the latter result, and the potential for trace amounts of modern carbon to greatly reduce the apparent ^{14}C ages of samples older than 30,000 years (Aitken, 1990), the lowest cultural layer at Monte Verde may be of similar antiquity to the basal levels at Pedra Furada, dated to > 48,000 years BP (Guidon and Arnaud, 1991; Guidon *et al.*, 1994). To resolve this uncertainty, the ^{14}C ages of the lowest cultural layer and the upper ~12,500 years BP level (the latter now generally accepted as pre-Clovis: Gibbons, 1997a; Adovasio and Pedler, 1997; Meltzer, 1997; Meltzer *et al.*, 1997) could be checked by optical dating of the adjacent sediments.

Luminescence dating could also be deployed to advantage at the Brazilian cave site of Toca da Esperança, where flaked stone tools have been found in association with faunal remains (de Lumley *et al.*, 1988). Three bones from the artefact-bearing layer at the base of the deposit were dated to *circa* 200,000–300,000 years using alpha and gamma spectrometry U-series methods. Provided these dates are accurate, this is by far the oldest archaeological site yet discovered in the Americas. The site has been given little credence by most archaeologists, but if humans were present in Siberia more than 260,000 years ago (Waters *et al.*, 1997a,b) then perhaps the U-series dates for Toca da Esperança are not so outlandish. Optical dating of the basal clayey sand layer could be used to check the U-series dates, including those determined by non-destructive high-resolution gamma spectrometry; recent applications of the latter technique to hominid bone have yielded dates of variable quality (e.g. Barton and Stringer, 1997; Berzero *et al.*, 1997; Latham, 1997).

TL dating of heated chert (i.e. flint) artefacts from the Shriver site in Missouri (Reagan *et al.*, 1978) represents, to my knowledge, the earliest involvement of luminescence methods with the American colonisation question. Burnt cherts from the top and bottom of a Palaeoindian fluted point horizon yielded TL ages of 10,640 ± 1000 and 14,800 ± 1500 years. Few procedural details are given, other than the palaeodoses were determined from thin polished slices (following Göksu *et al.*, 1974) and the annual environmental dose (1.78 mGy) was deduced from calcium fluoride dosimeters buried on site for six months. The Palaeoindian horizon overlay the lowest cultural layer, which contained a different lithic assemblage composed mainly of unifacial tools and flakes. A wide range of TL ages were obtained for these artefacts, which Reagan *et al.* (1978) interpreted as the result of insufficient heating in antiquity. Although the lowest lithic culture was not dated directly, the overlying TL ages indicate that initial site occupation must be older than 15,000 years, possibly dating back into the last glaciation (Reagan *et al.*, 1978). As TL dating of cherts was then a new development and no material was available for ^{14}C dating, this site has received scant attention from archaeologists (MacDonald, 1983; Stanford, 1983), even though there now seems little reason to doubt the accuracy of the published TL ages.

TL dating of heated lithics has also been exploited at the Brazilian sites of Alice Boër, Pedra Pintada and Pedra Furada. The southern site of Alice Boër was examined initially as ^{14}C dates of up to 14,200 years BP had been obtained previously from the upper cultural layer (Beltrão *et al.*, 1982; Danon *et al.*, 1982). Heated cherts were again used, although only 20% of the total number tested proved suitable for dating. The internal radioactivity of the cherts was measured and the environmental dose rate was determined from *in situ* gamma spectrometry and from lithium fluoride and calcium fluoride dosimeters. Checks for anomalous fading were made, and none was detected after a period of 3 months. TL ages of 2000–11,000 years were obtained for Layer III, but the artefacts from the deepest levels of this layer had not been heated sufficiently to reset the TL clock. These ages are consistent with the ^{14}C determinations, and suggest that the lower cultural stratum (Layer V) might have been deposited more than 20,000 years ago (Beltrão *et al.*, 1982). Numerical age estimates for the latter await the deployment of AMS ^{14}C methods and a renewed search for burnt cherts (Danon *et al.*, 1982), but it would now seem prudent to add TL and optical dating of sediments to this list.

TL dating of sediments received early application to Palaeoindian sites in San Diego, California (Berger and Huntley, 1986). Burnt clay from a hearth preserved in terrace sediments of the Otay River was dated to ~14,000 years using conventional TL techniques for pottery, while a date of ~20,000 years was obtained from unheated sediments associated with human occupation of the Scripps Tower site. The latter date was considered unreliable, as it had been obtained from a buried Ah soil developed on mixed colluvial/aeolian sediments and as it conflicted with a ^{14}C date of ~9900 years BP for the same occupation. At a third site (Fletcher Wash), artefact-bearing gravels were bracketed by TL dates of ~9000 and 28,000 years. These dates were also treated with caution as they had been determined from slopewash and alluvial sediments whose TL signals may have been incompletely reset by sunlight at the time of deposition.

To diminish the latter problem, optical dating of sediments has been exploited recently in the search for the first Americans. At the famous Clovis site

(Blackwater Draw No. 1 locality) in New Mexico, aeolian deposits containing Palaeoindian artefacts have been dated by ^{14}C and OSL methods, the latter employing coarse-grain quartz stimulated by an Ar-ion laser (Stokes, 1992). The ^{14}C chronology obtained for the youngest artefact-bearing unit indicated a terminal date for deposition of either *circa* 6200–6500 or 7900–8800 years BP (Haynes and Agogino, 1966; Stokes, 1992). Support for the younger determination was given by an optical date of 5950 ± 600 years for the base of the overlying unit, the dose rate being deduced from thick-source alpha counting (uranium and thorium) and flame photometry (potassium). The lower units with "Folsom" and "Clovis" artefacts were not optically dated, but any such attempt may be foiled by reworking of the sediments and associated artefacts via the action of ancient groundwater springs and trampling by mammoths.

4.1.2. *The early Amazonians.* The ideal combination of optical dating of sediments, TL dating of heated lithics, and AMS ^{14}C dating of short-lived plant remains (such as fruit seeds) has been successfully employed at Caverna da Pedra Pintada in the Amazonian rainforest (Michab *et al.*, 1997; Roosevelt *et al.*, 1996). The 56 AMS ^{14}C dates ranged from 10,000 ± 60 years BP to 11,145 ± 135 years BP, which corresponds to a calibrated age range of *circa* 10,500–14,200 years. The TL dates for 10 burnt artefacts (composed of quartz sandstone) were divided into two groups: *circa* 9500–11,900 years (seven specimens) and *circa* 14,800–17,500 years (Michab *et al.*, 1997). All 10 artefacts were dated using methods described previously (Mercier *et al.*, 1995a; Valladas, 1992), and no cause could be found to reject the three oldest TL ages. The three optical (OSL) dates centered on the period between 11,400 and 12,000 years ago. The OSL palaeodoses were determined using a combined additive/regenerative single-aliquot procedure with sand-sized quartz grains that had possibly been heated in antiquity (Michab *et al.*, 1997). Attempts to use the hard-to-bleach 375°C TL signal in the sediments were again unsuccessful: the regenerative-dose palaeodoses were 45% greater than their OSL counterparts, as a result of the TL signal having been incompletely bleached or heated in antiquity (Feathers, 1997a; Michab *et al.*, 1997).

The environmental dose rates were calculated from high-resolution gamma spectrometric analyses of collected sediment samples, and no disequilibrium in either the ^{238}U or ^{232}Th decay chains was detected. On-site measurements of the environmental dose rate were not made at Pedra Pintada, but isochron analysis of the group of seven burnt lithics lends support to the laboratory determinations of the external dose rates and to the TL ages of the individual specimens: the measured and isochron estimates of the annual external doses are 336 ± 22 and 333 ± 30 μGy, respectively, while the corresponding ages are 10,570 ± 800 and 10,580 ± 660 years (Feathers, 1997a; Michab *et al.*, 1997; Roosevelt *et al.*, 1996). The three older lithics (14,800–17,500 years) in the same layer were postulated to be remnants of an earlier period of site habitation which had been incorporated into the artefact assemblage during the subsequent occupation (Michab *et al.*, 1997). The quartz sediment dates (~17,000 years) determined using the hard-to-bleach TL signal could be interpreted as supporting this theory. If the sediments deposited at the site had been fully bleached ~17,000 years ago (when the site was first occupied), but only a brief exposure to sunlight had occurred during later sedimentation events, then only the easy-to-bleach TL and OSL signals would record the time since the most recent episode of insolation.

The firm chronology established at Pedra Pintada shows the unequivocal presence of humans on the Amazon River by the terminal Pleistocene. The relation of these forest dwellers to the Clovis people is, however, more contentious (Gibbons, 1996a; Fiedel, 1996; Roosevelt, 1996). There would appear to be no climatic or biogeographic reason to exclude the possibility of earlier occupation of the rainforest which has persisted throughout the last 40,000 years, the Last Glacial Maximum being characterised by a cooling of 5–6°C and an increase in rainforest species diversity (Colinvaux *et al.*, 1996; Stute *et al.*, 1995).

The site of Pedra Furada offers the strongest case for 50,000 years or more of human colonisation of the Americas (Bahn, 1993; Delibrias *et al.*, 1988; Guidon and Arnaud, 1991; Guidon *et al.*, 1994; Parenti *et al.*, 1996). Forty-six of the 55 ^{14}C determinations made on abundant pieces of charcoal are currently accepted by the excavators, the oldest sample returning an uncalibrated AMS age of >48,000 years BP. Like Caverna da Pedra Pintada, this large rock shelter has been painted, and fragments of painted rock were found in deposits dated to ~32,000 years BP, with a pictograph (two red painted lines) at the ~17,000 years BP level (Guidon and Delibrias, 1986). The 5 m-deep sandy deposit also contains ~160 hearths and more than 7000 stone artefacts, the basal assemblage being made of flaked quartz and quartzite.

Critics have argued that iron pigments from the nests of mud-daubing insects could have created the pictograph (Bednarik, 1989), that the operation of geological processes could have produced chipped stones, and that natural bush-fires could have formed the hearths and introduced the charcoal (e.g. Lynch, 1990; Meltzer *et al.*, 1994). These possibilities have been addressed by the excavators (Guidon and Arnaud, 1991; Guidon *et al.*, 1994), with TL analysis of hearth stones providing one line of evidence against the bush-fire argument (Parenti *et al.*, 1990, 1996). The method proposed by Valladas (1981) to determine the temperature reached in prehistoric hearths indicated that 78% of quartz cobbles and 14% of sandstone fragments from a hearth feature had been heated to above 450°C. This is considerably greater than the maximum mean surface temperature reached during natural fires in Wyoming (225–250°C; Bellomo, 1993) and Western Australia (290°C; Koch and Bell, 1980). Ground surface temperatures of 550°C have been reported, however, for bush-fires in the open forests of northern Australia (Braithwaite and Estbergs, 1985), although such high temperatures are reached only briefly during the passage of the fire. Moreover, as the immediate sub-surface sediments are heated to less than 100°C (e.g. Koch and Bell, 1980), natural bush-fires are unlikely to be of sufficient severity or duration to erase the high-temperature TL of the interior portion of a cobble-sized stone. Further support for an anthropogenic origin for the hearths is given by the spatial distribution of heated stones: these were clustered on one side of a hearth (^{14}C dated to ~42,400 years BP), suggestive of site cleaning and re-use, rather than being distributed uniformly as might be expected if they had been heated by a bush-fire (Parenti *et al.*, 1990, 1996).

TL dating of the Pedra Furada hearth stones is in progress, and optical dating of the sediments is also being considered (F. Parenti, Personal Communication, 1996). Any application of optical dating will need to take account of *in situ* rubble disintegration, which would liberate unbleached grains into the deposit. Multiple-aliquot dating procedures would be susceptible to sample contamination by these unbleached grains, and could yield considerable age overestimates (Roberts and Jones, 1994). Single-aliquot or single-grain optical dating methods should be employed under such circumstances (e.g. David *et al.*, 1997) to ensure accurate age determinations.

4.1.3. *Man meets beast.* Luminescence methods also have unrealised potential to elucidate the timing of human arrival in the Americas and the extinction of many species of large mammal (the so-called "megafauna"). If Pedra Furada was first occupied by humans more than 50,000 years ago, then it becomes less likely that megafaunal extinctions in the terminal Pleistocene were due mainly to hunting by the first Americans (Bray, 1986). The impact of massive climatic change has been implicated in North America, the homeland of the "overkill" hypothesis (Martin, 1973, 1990), by the lack of synchroneity between human arrival and mammalian extinctions (Grayson, 1989, 1991). In northern Peru also, U-series dating of fossil bones has indicated that extinction of the megafauna occurred 15,000–16,000 years ago, coincident with deglaciation and the onset of aridity, but predating the arrival of people, in this region (Falguères *et al.*, 1994b).

Humans were hunting big game in Venezuela by 13,000–14,000 years BP, however, as shown by the presence of a quartzite projectile point in the pelvis of a juvenile mastodon at Taima-taima (Bryan *et al.*, 1978). Both human predation and climatic change may have played a role in the demise of the South American megafauna since the Last Glacial Maximum (Dillehay *et al.*, 1992), but if people first arrived tens of millennia earlier then perhaps there was a period of faunal attenuation, as yet unrecognised and undated, that set the stage for their post-glacial collapse. Faunal remains are not preserved in the acid sediments at Pedra Furada (Guidon *et al.*, 1994), but rich and diverse megafaunal assemblages have been found in association with stone tools in several neighbouring limestone caves (Parenti *et al.*, 1996). As organic material is too scarce for ^{14}C dating (Parenti *et al.*, 1996), luminescence methods could be applied to these megafauna-bearing sediments, as well as those at Toca da Esperança (de Lumley *et al.*, 1988), to determine the late Pleistocene chronology of mammalian extinctions in Brazil.

TL dating has been deployed recently in this capacity in North America, where the poorly preserved remains of an adult mastodon were found in 1973 in a loess section on the banks of the Missouri River (Berger, 1996; Chapman, 1975; Dunnell and Hamilton, 1995). Accelerator ^{14}C dating of the bone collagen fraction had provided two dates of ~35,800 years BP, and TL dating of the immediately underlying loess gave a "partial bleach" age of 41,700 ± 6100 years. The difference of ~5900 years is consistent with other uncalibrated ^{14}C age comparisons for this time period (see Section 2.2). The TL sample was obtained from the sediment "pedestal" attached to the plaster-cast field specimens held at a museum, the find site having been completely destroyed. The uranium-enriched bone was shown to have a negligible effect on the gamma dose rate (Berger, 1996), and the substantial difference in ^{238}U concentrations in the bone and TL samples (~10.6 and ~3.1 ppm, respectively) argues against significant uranium enrichment of the sediment close to the bones (cf. Mercier and Valladas, 1994; Mercier *et al.*, 1995b). The apparent association of the mastodon with a flake knife-scraper made of chert (Chapman, 1975, pp. 53–54) opens up the intriguing possibility that people were present in North America, and hunting big game, by 42,000 years ago—a situation reminiscent of Australia, the subject of the next section.

4.2. *Australia*

> The question has frequently presented itself—Are there any geological traces of man on this Continent, such as exist in other countries, and whereby the presence of a former race, or the antiquity of the present fast disappearing

one, can be traced? The answer given by those most competent to judge is—No! (Etheridge, 1890, p. 259)

To what cause, it may be asked, is due the extinction in Australia of the genera *Diprotodon*, *Nototherium*, *Thylacoleo*, *Phascolonus*, *Thylacinus*, *Sarcophilus*, *Palorchestes*, *Procoptodon*, *Pachysiagon*, *Protemnodon*, *Sthenurus*, with the larger species of existing genera of Kangaroos and Wombats? No adequate cause suggests itself to my mind save the hostile agency of man. (Owen, 1877, v. 1: viii)

The antiquity of the human colonisation of Australia, and its link with the extinction of the giant marsupials, reptiles and birds, has been speculated upon for more than a century. Since this time, much has been learnt on both matters. The view expressed by Etheridge (1890), the palaeontologist to the Australian Museum, was finally abandoned in the 1960s when ^{14}C age determinations pushed a human presence back towards 40,000 years (see reviews by Jones, 1989, 1993). Luminescence dating has, over the past decade, extended this timeframe still further, often fomenting controversy equal to that surrounding claims for pre-Clovis sites in the Americas. Applications have been concentrated at sites where other numerical dating methods cannot be applied, owing to the paucity of suitable materials, and where the practical limits to ^{14}C dating have been reached. The scarcity of heated flints has prevented direct dating of stone artefacts, but the dating of archaeosediments has made as great an impression on Australian archaeology as flint dating has in the Middle East and Europe. A discussion of these and other luminescence contributions to Australian archaeology is presented in this section; site locations are shown in Fig. 8.

A century of research has produced no concensus, however, on the contention by Owen (1877), a prominent anatomist and adversary of Charles Darwin, that human predation was the primary cause of megafaunal extinctions. Some researchers hold that extinction followed immediately after, or within a few millennia of, human landfall (Flannery, 1990, 1994; Jones, 1968), whereas others consider that megafauna and humans co-existed for tens of millennia (Diamond, 1989; Merrilees, 1968; Wright, 1986a) with climate change, or a combination of factors, dealing the mortal blow (e.g. Dodson, 1989; Gorecki *et al.*, 1984; Horton, 1984).

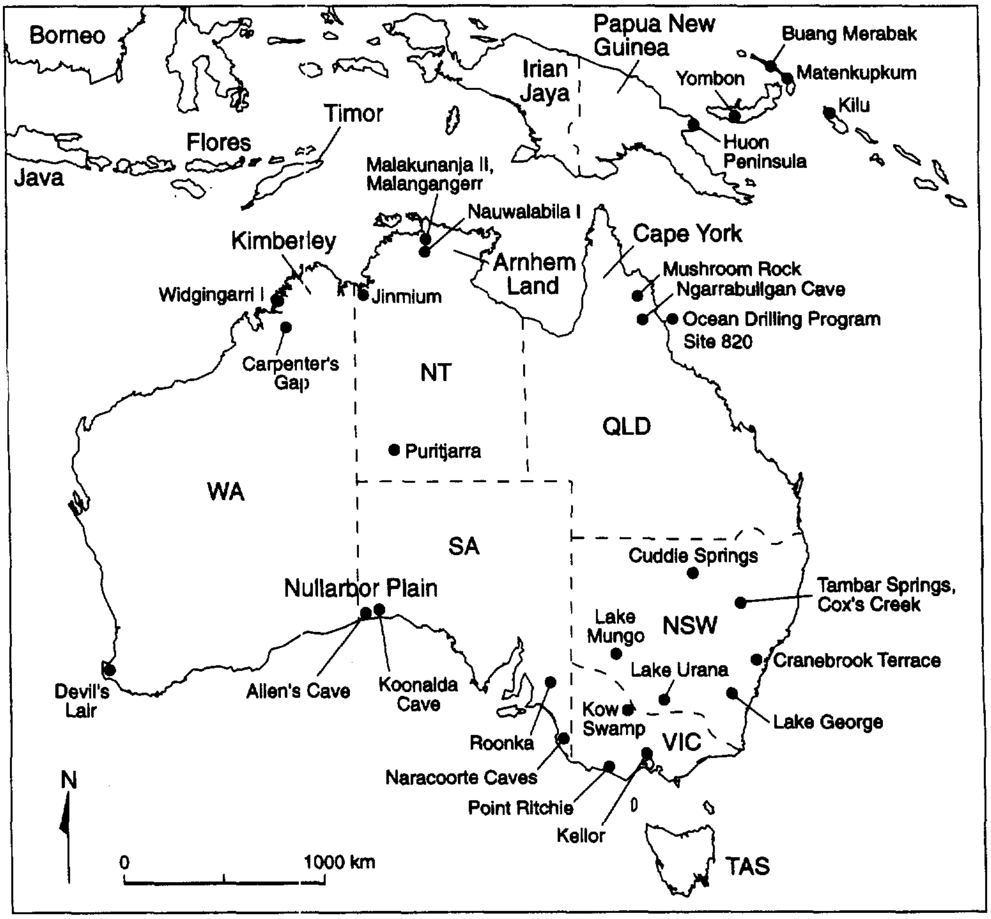

Fig. 8. Location map of archaeological and megafaunal sites discussed in the text.

As faunal attenuation over several glacial-interglacial cycles may have preceded the final extinction events, it is crucial to investigate the time period beyond the range of ^{14}C dating. Faunal remains suitable for direct dating by ESR, U-series or amino-acid racemization are not found at every site in Australia, so luminescence methods are particularly well placed to provide the "baseline" chronology for Pleistocene faunal extinctions; this chronology can then be compared with that of human colonisation, based on ^{14}C and luminescence ages. Within this archaeological context, some recent and forthcoming applications of luminescence dating to megafauna-bearing sediments are mentioned below.

4.2.1. *Lake Mungo*. The first archaeological site to capture the attention of luminescence daters was Lake Mungo, which at times during the Pleistocene formed part of a chain of freshwater lakes along Willandra Creek in south-western New South Wales (Bowler, 1971). Long crescent-shaped aeolian dunes (or "lunettes") developed along the eastern shorelines of these lakes, to which people and a variety of fauna (including megafauna) were attracted during more humid periods. Severe erosion of the Mungo lunette (at a section known as the "Walls of China") in the late 1960s revealed animal fossils, as well as stone artefacts, fireplaces, shell middens and the cremated remains of two people (Bowler *et al.*, 1970). Soon after these sensational discoveries, the "Mungo I" female was dated by ^{14}C to 25,500 ± 1000 years BP, and the skeleton of a third anatomically modern human (named "Mungo III") was located in ochre-stained grave fill (Bowler and Thorne, 1976). A ^{14}C date of ~33,000 years BP was obtained from freshwater shells, which lay alongside stone artefacts on a deflated surface, and charcoal from several Aboriginal fireplaces yielded ages of *circa* 26,000–31,000 years BP, during which period a local geomagnetic polarity excursion also occurred (Barbetti and Allen, 1972; Barbetti and McElhinny, 1972; Bowler *et al.*, 1972).

The global significance of the Mungo finds presented an ideal opportunity to test TL dating methods on the baked hearth sediments and ovenstones. The first TL application was made to baked sand from a hearth dated by ^{14}C to ~31,000 years BP. Both fine-grain and quartz inclusion methods were attempted, although the quartz samples failed the "plateau test" for constancy of palaeodose above 350°C (Adams and Mortlock, 1974). This implies that the 375°C TL peak in baked quartz sand had not been thermally erased in antiquity, whereas the same signal had been reset in coarse-grain quartz extracted from the ovenstones. The latter result was reported by Huxtable and Aitken (1977) who computed an average TL age of 33,500 ± 4300 years from 7 determinations; the corresponding average ^{14}C age, using the Libby half-life, is 28,650 years BP. TL ages obtained by Huxtable and Aitken (1977) using their fine-grain samples were considered to be overestimates, owing to spurious TL from carbonates. Again in contrast, Adams and Mortlock (1974) reported no such problems and produced a fine-grain TL date of 30,400 ± 3300 years, in close accord with the quartz TL ages of Huxtable and Aitken (1977) and the ^{14}C date of ~31,000 years BP for this hearth. This good agreement would appear to be fortuitous, as Bell (1991) subsequently reported that these fine-grain sediments suffer from anomalous fading of 10–30% over a period of 3 months. He managed, however, to obtain TL dates from baked quartz sand from four fireplaces (mean ^{14}C age of 28,220 years BP), including the one that had frustrated Adams and Mortlock (1974). Neither an explanation for this discrepancy, nor the temperature regions of his palaeodose plateaux, are supplied by Bell (1991), whose arithmetic mean age of 33,500 years is identical to that derived independently by Huxtable and Aitken (1977). Both of these studies show that uncalibrated ^{14}C determinations underestimate TL ages by ~5000 years at *circa* 33,500 years ago, a finding that was first noted by Zimmerman and Huxtable (1971) and which has since been broadly confirmed by other calendar-year dating methods (see Section 2.2).

The lunette sediments at Lake Mungo also formed the basis of Readhead's (1982) pioneering efforts to apply TL dating techniques to unburnt archaeosediments in Australia. Readhead (1982, 1988, 1990) used sand-sized quartz grains together with an early version of the "Australian slide" combination of "total bleach" additive-dose and regenerative-dose protocols. A series of aeolian sand samples were collected from the three major sedimentary units exposed in the "Walls of China" section: the stratigraphically youngest Zanci unit, the underlying archaeological Mungo unit, and the basal, culturally-sterile Golgol unit. Dose rates were calculated from laboratory measurements only, using "sealed" versus "unsealed" thick-source alpha counting to gauge the degree of radon escape; 0–26% emanation was observed, whereas none was detected by thick-source alpha counting of the baked hearth sediments from the Mungo unit (Bell, 1991). Readhead (1982, 1988) obtained TL dates of ~22,000 years for the Zanci unit (compatible with the ^{14}C ages for this layer) and *circa* 24,500–29,300 years for the upper part of the Mungo unit, but reliable TL ages could not be determined for the Golgol unit samples, owing to inflexions in their dose-response curves. He later ventured estimates of *circa* 220,000–290,000 years for the Golgol sediments (Readhead, 1990). These ages are considerably greater than those of *circa* 140,000–180,000 years obtained recently (Oyston, 1996) for the base of the lunette using the same TL dating protocols.

Oyston (1996) also applied the "selective bleach" TL method (Franklin and Hornyak, 1990; Prescott and Fox, 1990; Prescott and Mojarrabi, 1993; Prescott and Purvinskis, 1991; Prescott and Purvinskis, 1993) to quartz sand grains from the Mungo and Zanci units; the easy-to-bleach 325°C signal being subtracted (selectively bleached) from the total TL signal by exposing aliquots to the >475 nm component of sunlight. This approach is akin to optical dating and exploits the most light-sensitive signal, which has a much greater probability of having been zeroed in antiquity than the harder-to-bleach signals additionally sampled by total bleach TL methods. The improved accuracy is shown clearly in the dating of the Zanci and upper Mungo units, for which total bleach ages were greater than the selective bleach (and ^{14}C ages) by 12,100 and 9400 years, respectively (Oyston, 1996). The selective bleach procedure was also used to bracket the age of the Mungo III burial: an age of 43,300 ± 3800 years was determined for the lower part of the Mungo unit (into which the burial had been dug) and an age of 24,600 ± 2400 years was obtained from the upper Mungo unit sediments, which capped the burial. These age constraints for the Mungo III burial indicate that it is contemporaneous with, or older than, the Mungo I skeleton, as anticipated by Bowler and Thorne (1976). No other Australian site boasts such ancient human remains (Brown, 1992; Pardoe, 1995).

The Lake Mungo lunette is probably the most securely dated archaeological site in Australia. Radiocarbon methods have been applied to a variety of materials (freshwater shells providing the more reliable results for samples older than 20,000–25,000 years BP; Bowler and Wasson, 1984) and comparable TL dates have been obtained from the 375°C and 325°C glow peaks in baked and unheated quartz, respectively; *in situ* measurements of the dose rate and a direct demonstration of secular equilibrium in the ^{238}U decay series would further strengthen the TL claims.

4.2.2. *Roonka and Urana*. Prehistoric human burials, fireplaces and stone artefacts have also been yielded at Roonka, a site on the Murray River in south-eastern South Australia (Pretty, 1988). Radiocarbon dates of up to 18,000 years BP have been obtained from fireplaces, with a late Holocene example producing matching ^{14}C and TL ages of ~1000 years BP, the TL date being obtained from heated calcrete ovenstones (Prescott *et al.*, 1983). Spurious TL halted any attempt to date the calcite directly, but acid dissolution of the ovenstones produced a sufficent quantity of fine-grain minerals and sand-sized quartz for dating. A second fireplace was TL dated to *circa* 2000–2500 years, in contrast to the charcoal ^{14}C date of ~11,300 years BP (Prescott *et al.*, 1983). The source of this discrepancy is unclear but, as the fireplace was situated on a stratigraphic unconformity, the TL and ^{14}C dates may relate to different events; subsequent TL dating of the unheated dune sands beneath the fireplace produced ages of *circa* 8000–14,500 years (see below), comparable with the ^{14}C age for the charcoal. An oven mound on the Riverine Plain in the central Murray valley has since been TL dated to ~2650 years, using fine grains extracted from baked clay balls used as heat-retainers during cooking (Downey and Frankel, 1992); this date confirmed three calibrated ^{14}C determinations on charcoal of *circa* 2600–2750 years.

TL dating of *unheated* sediments also received early application at Roonka (Prescott, 1983), where cultural material and the remains of 216 people (most of which are late Holocene in age) have been excavated from aeolian sand dunes bordering the river (Pretty, 1988). On the west bank, the dune is underlain by a culturally-sterile "terra rossa" soil. This *terminus post quem* for human occupation was dated to 65,000 ± 12,000 years using sand-sized quartz grains and a variant of the "Australian slide" method (whereby the second glow data, rather than bleached grains, were used to construct regenerative-dose growth curves). On the east bank, the poorly preserved skeletons were overlain by dune sand dated to 14,500 ± 2000 years, although Prescott (1983) raised doubts about the degree to which the TL signal had been zeroed at deposition. Exposing the acid-etched quartz grains to direct sunlight for a minimum of 60 h reduced the TL signal to a much lower "residual" level than that attained by grains collected from the surface of a spoil heap formed 5 years earlier. Using the latter residual level instead of the former reduced the age of the sample to ~8000 years, more in keeping with the ^{14}C date of ~9200 years BP for the oldest burial on the west bank. Palaeodose plateaux extended from 300°C to 420°C in both cases, however, which superficially suggests that both the 325°C and 375°C peaks had been reset in antiquity.

A clue to the solution of this riddle was given by Prescott and Purvinskis (1991, 1993), who used the "selective bleach" method to abstract the "pure" 325°C signal from two near-modern Roonka samples. They found that the 325°C signal had been fully zeroed but that a significant residual TL signal remained at temperatures as low as 250°C due to the hard-to-bleach component. A palaeodose plateau extending to temperatures as low as 260–280°C can be expected in quartz if both the easy-to-bleach and hard-to-bleach signals have been fully bleached in antiquity (e.g. Chawla *et al.*, 1992; Roberts *et al.*, 1990a, 1994a, 1996). A plateau starting at temperatures above 300°C implies differential bleaching (or differential thermal stability, in much older samples) of the two components, with the hard-to-bleach signal being incompletely zeroed. The fact that the plateau extends as low as 300°C does not mean it includes the 325°C peak: it shows only that the same palaeodose is obtained from the low-temperature "tail" and the peak of the 375°C signal.

The difference in the residual signals measured for the acid-etched and spoil heap grains can be satisfactorily explained by the yellow-brown coatings on the hominid-bearing dune sands at Roonka. If the grains had been transported with their extant coatings, then the hard-to-bleach signal is unlikely to have been reset completely at deposition (see Singhvi *et al.*, 1986a), and the spoil heap grains provide the best estimate of the residual level. The residual level attained by the etched grains would be appropriate only if the grains had been stained after deposition: if this were true, and solar exposure had been prolonged, then the hard-to-bleach signal should also have been fully reset, resulting in a palaeodose plateau extending to 270°C. The lack of such a plateau indicates that the grains were stained prior to transport or that the unstained grains were exposed only briefly to sunlight. In either case, the 8000 year-old TL date is preferred for the Roonka burial, although this may still be an overestimate (the "plateau test" having failed). Optical or selective bleach TL dating methods should be able to resolve this issue and provide reliable age estimates for the fossil hominid levels at this important site.

Human remains have also been discovered in the lunette at the southern end of Lake Urana, which is located on the Riverine Plain ~300 km south-east of Lake Mungo (Page *et al.*, 1994). The skull and post-cranial remains are those of an adult female, similar in morphology to the Mungo I female, but no evidence was found of grave excavation. Two large grindstones and numerous quartzite artefacts were later recovered from the site, but the absence of charcoal deposits precluded ^{14}C dating. TL samples were collected from the dull orange aeolian sand units above and below the skeleton, to bracket its age. Sand-sized quartz grains were dated using the combined additive/regenerative "total bleach" method (Readhead, 1982, 1988), but no correction was made for the residual TL of a surface sample (Page *et al.*, 1994). As discussed above, large age overestimates have been made for hominid-bearing aeolian sands at Lake Mungo (Oyston, 1996) and Roonka (Prescott, 1983) using the total bleach method in tandem with the residual TL level reached by acid-etched grains. The two dates of $25,600 \pm 7300$ and $32,400 \pm 8000$ years (Page *et al.*, 1994) should be regarded, therefore, as *maximum* age estimates; no reassurance is given by the palaeodose plateaux, which begin only at 300°C for both samples, and no independent age control is available at this site.

OSL or selective bleach TL methods could again be used to eliminate this chronological uncertainty, and determine if the Urana hominid is contemporaneous with the Mungo I specimen. Kow Swamp, a site ~200 km south-west of Lake Urana, has yielded skeletons of a more archaic type of hominid (Thorne and Macumber, 1972). This site is presently being considered for optical dating, to check the validity of the early Holocene ^{14}C determinations made previously (T. Stone, Personal Communcation, 1996).

4.2.3. *Graveyards of the giants.* South-eastern Australia also boasts a wealth of fossil megafaunal material that could shed light on the timing of extinction events. Many of the lunettes bordering the lakes along Willandra Creek (e.g. Mungo) and other nearby lakes (e.g. Menindee, Tandou, Victoria) contain abundant faunal remains, as well as recording the palaeohydrology of the region (Bowler, 1971, 1976; Bowler and Wasson, 1984; Hope, 1982; Hope *et al.*, 1983). These sites will shortly be sampled for luminescence dating, in a joint project between the author and T. Flannery, a specialist in extant and extinct Australasian mammals. Samples have already been collected from two sites (Cuddie Springs, Tambar Springs) in northern New South Wales (Fig. 8), where artefacts have been found in association with megafaunal remains (Dodson *et al.*, 1993; Furby *et al.*, 1993; Wright, 1986a). For the samples examined thus far, additive-dose OSL methods have produced comparable palaeodoses using multiple and single aliquots of quartz (Murray *et al.*, 1997), indicating that problems of insufficient bleaching do not appear to plague these water-lain sediments.

A single-aliquot check of multiple-aliquot OSL palaeodose determinations shall be made also on the megafauna-bearing sediments collected from Cox's Creek, where the almost complete skeleton of a *Diprotodon* was excavated in 1979 from an eroding stream bank (Wright, 1986a), and Naracoorte Caves in south-east South Australia, which contains Australia's largest known collection of Pleistocene fossil vertebrates (Wells *et al.*, 1984). The 100 and 200 μm-diameter quartz grains in the entombing sand deposit at Cox's Creek have yielded preliminary OSL dates of ~75,000 and ~95,000 years, respectively, while provisional OSL ages in excess of 170,000 years have been estimated for the Victoria Cave deposit at Naracoorte. At the latter site, however, it remains to be shown that the sediment entered the deep cave system in a fully bleached state and has not been periodically transported and redeposited by underground streams. Single-aliquot methods should be able to detect infringement of uniform bleaching *before* the sediment entered the cave, but they cannot provide any information on subterranean remobilisation. In some chambers, the contemporaneity of the megafauna and the cave deposits can be independently tested by U-series dating of speleothems and ESR dating of fossil teeth (K. Moriarty, Personal Communication, 1996).

For a more complete picture of megafaunal attenuation and extinction during the latter half of the Quaternary, sites have been selected from around the continent to accommodate possible differences in timing between different climatic and ecological zones. Several cave and open-air sites across the south of the continent will be investigated, as shall a number of previously identified fossil deposits in the deserts of outback Australia (Tedford and Wells, 1990; J. Magee, Personal Communication, 1996). Only those sites with megafauna in articulated anatomical position will be sampled, to exclude the possibility that the skeletal remains have been reworked from older deposits. Whenever feasible, luminescence dating of megafauna-bearing sediments will be complemented by direct dating of fossil remains, such as the eggshell of *Genyornis* (a genus of extinct giant flightless bird), using AMS ^{14}C, U-series and amino-acid racemization methods (e.g. Magee *et al.*, 1995; Miller *et al.*, 1997), and ESR and U-series dating of tooth enamel.

Fossils from several sites have already been described and some museum specimens have a sufficient thickness of sediment attached to the outside or inside of bones for the application of single-aliquot optical dating methods; enough sediment is occasionally fastened also to artefacts stored in museums (R. Jones, Personal Communication, 1997). The principal beneficiaries of such methods are sites that have been destroyed or are inaccessible. A major disadvantage, which applies also to the dating of museum collections of teeth by ESR and pottery by luminescence, is the lack of environmental dosimetry data for the original surrounding deposit and the possible bias introduced by estimating this dose rate from the sediment adhered to the specimen (e.g. Mellars *et al.*, 1997; Mercier *et al.*, 1995b). But if more than one mineral, or size fraction of the same mineral, can be extracted from the residual sediment, then application of "isochron" or "subtraction" techniques should eliminate the need for any knowledge of the external dose rate (see Section 3.2.1).

4.2.4. *The first Australians.* In contrast to southern and central Australia, fossil remains of megafauna and humans are much less abundant north of the Tropic of Capricorn. The weathering regime of high temperature and humidity, and the naturally acidic monsoonal rainfall (Likens *et al.*, 1987; Noller *et al.*, 1990), act against the long-term preservation of organic materials such as bone and charcoal. Stone artefacts are preserved in tropical environments, however, and these form the basis for understanding the archaeology of northern Australia, the region through which the first human arrivals most likely entered the continent (Jones, 1989). The initial colonists may have travelled from south-east Asia along the chain of Indonesian islands to Timor, and then negotiated the wide sea crossing to the north of Western Australia, a region known as the Kimberley. Or they may have journeyed through to Irian Jaya/Papua New Guinea and then made their way to far north Queensland, through the Cape York Peninsula, or to Arnhem Land at the "top end" of the Northern Territory. This route necessitated only a short sea passage during interglacial periods, while the exposed Sahul Shelf functioned as a land bridge around the time of the glacial maxima.

The identification of the region of first landfall lies beyond the reach of ^{14}C methods, however, due to the paucity of organic material in the basal cultural levels of the deepest archaeological sequences in northern Australia (e.g. Malakunanja II, Nauwalabila I, Mushroom Rock). By the late 1980s, the chronology for continental colonisation rested on ^{14}C dates (on charcoal) of up to 38,000 years BP from southern sites, such as Lake Mungo, Upper Swan (Pearce and Barbetti, 1981) and Devil's Lair (Dortch, 1979), and on ^{14}C dates (on marine shell) of up to 33,000 years BP from sites in island Melanesia, to the north-east of Australia (e.g. Allen *et al.*, 1988; Wickler and Spriggs, 1988). Some archaeologists (e.g. Allen, 1989) considered these dates to reflect initial occupation of the continent, whereas others (e.g. Jones, 1982, 1989) viewed some of these dates as minimum age estimates, owing to sample contamination by modern carbon. The existence, or otherwise, of a ^{14}C "glass ceiling" at *circa* 35,000–40,000 years BP continues to be a source of lively debate (e.g. Allen, 1994; Allen and Holdaway, 1995; Chappell *et al.*, 1996a; Roberts *et al.*, 1994b). The application of TL dating to archaeosediments was proposed by Jones and Johnson (1985) as a means to resolve this chronological conundrum and to obtain basal ages for the north Australian sequences. As a consequence, a TL dating project was begun by the author and R. Jones in 1988 (Jones, 1989, p. 762), and the third member of the team, M. Smith, joined the following year. Since then, TL and optical dating methods have been deployed at archaeological sites in Arnhem Land, Cape York and the Kimberley, with the recent TL claims for Jinmium rock shelter (Fullagar *et al.*, 1996) being the latest to spark controversy over the early human occupation of northern Australia.

4.2.5. *Western Arnhem Land.* The first sites to receive attention lay at the foot of the 200 m-high escarpment that marks the western edge of Arnhem Land, ~250 km east of Darwin. Denudation of the escarpment and adjoining plateau has resulted in the formation of sand "aprons" along the base of the cliff (Nott and Roberts, 1996; Roberts, 1991, published *in toto* as Roberts *et al.*, 1991). At the head of two sand aprons lay the rock shelter sites of Malangangerr and Malakunanja II, both of which had been excavated previously and had yielded ^{14}C dates of *circa* 18,000–23,000 years BP for the non-basal levels (Kamminga and Allen, 1973; White, 1971). In 1988, sand-sized quartz grains were extracted from these archaeosediments and TL dates were derived using a combined additive/regenerative procedure to determine the palaeodoses and high-resolution gamma spectrometry to estimate the environmental dose rates (Roberts *et al.*, 1990a). Thick-source alpha counting of samples

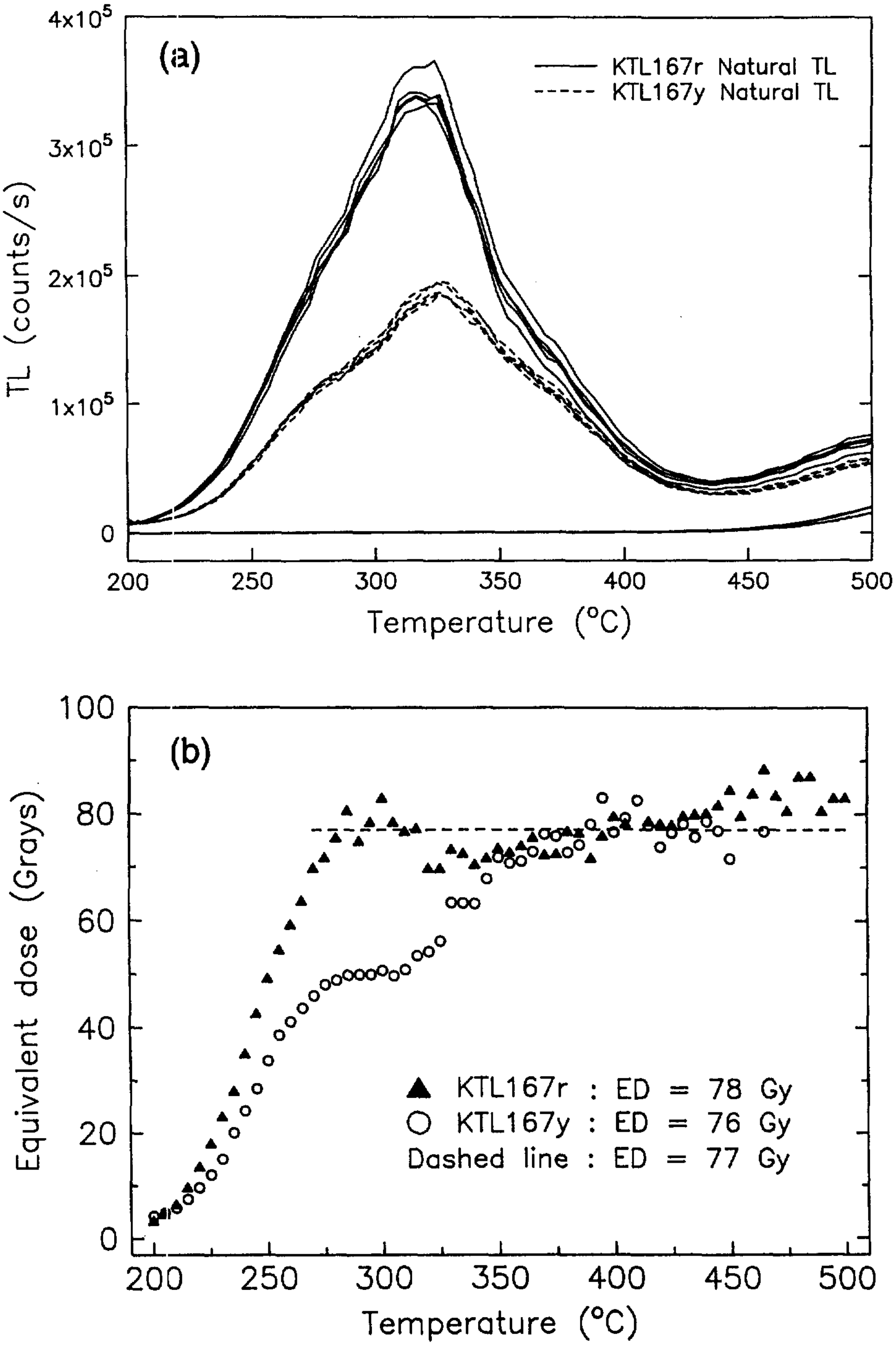

Fig. 9. (a) TL glow curves for natural aliquots of Malakunanja II sample KTL167, prepared under red (solid lines) and yellow (dashed lines) laboratory illumination. Heating rate is 2.5 K s^{-1}. (b) Palaeodose "plateau test" for aliquots of sample KTL167 processed in red (solid triangles) and yellow (open circles) light. Note partial erasure of the 325°C peak, and failure of the plateau test, under yellow illumination (from Roberts, 1991, and Roberts *et al.*, 1991).

before and after acid etching was used to deduce the magnitude of the internal dose rate, but no *in situ* measurements of the external dose rate were made. The laboratory measurements showed, however, that the dose rate did not change abruptly between levels at any particular site, and that a condition of secular equilibrium prevailed in the uranium and thorium decay chains. The palaeodoses were determined from interpolation of the regenerative-dose growth curves, as the latter coincided with the shifted additive-dose data, indicating that no significant sensitivity change had occurred upon sunlight or sunlamp bleaching.

An interesting anomaly was reported, however, for the palaeodose plateaux of samples processed under yellow (>470 nm) laboratory illumination. These showed a distinct "double" plateau, the first at 260–320°C and the second at 340–500°C. Only the latter plateau produced TL dates in correct stratigraphic order, but the failure to obtain the same palaeodose from both the 325°C and 375°C regions raised grave concerns that the easy-to-bleach signal had been partially reset, either in the field or in the laboratory. Other samples processed in this laboratory show similar plateaux characteristics (e.g. Murray *et al.*, 1992; Nanson *et al.*, 1993), and a double plateau had also been reported previously for quartz sediments collected from the Spencer Gulf in South Australia (Smith *et al.*, 1982); in the latter instance, the lower plateau (associated with the 325°C peak) was thought to represent the last time that the marine sediments had been transported underwater, while the last exposure to unfiltered sunlight was recorded by the upper plateau (associated with the 375°C peak). For the archaeological samples, various possible mechanisms to preferentially reduce the 325°C palaeodose were considered (Roberts *et al.*, 1991. 1993a): partial bleaching of the TL in water-transported grains or oxide-coated grains exhumed by termite or human activities; thermal erasure of the 325°C peak, and subsequent increase in TL sensitivity, due to bushfires; thermal instability of the 325°C peak at ambient temperatures of 25–30°C (the modern range of mean annual temperatures across tropical northern Australia) giving rise to a shortened trapped-electron lifetime; and inadvertent bleaching during sample collection or laboratory preparation.

Cautions had earlier been expressed about the use of yellow filters for laboratory illumination (Smith, 1988; Spooner and Prescott, 1986), but preliminary tests had given no indications of laboratory bleaching (A. Murray, Personal Communication, 1987). Nonetheless, acting on the suggestion of B. Smith and A. Wintle, red filters were installed and the key samples from Malakunanja II were processed under the >590 nm transmitted light (Roberts *et al.*, 1990a). Furthermore, to facilitate a direct comparison, one sample was split into two portions: one portion was processed in red light (KTL167r) and the other in yellow light (KTL167y). The results obtained were striking. The red-lit samples invariably passed the "plateau test", giving palaeodose plateaux that began at 260–270°C and extended across both the easy-to-bleach and hard-to-bleach TL peaks. For KTL167, a "double" plateau was obtained from the yellow-lit portion and only the palaeodose for the upper plateau agreed with that derived from the long plateau (270–500°C) exhibited by the red-lit portion (Fig. 9b; Roberts *et al.*, 1991); a comparison of the natural glow curves shows the magnitude of reduction (but not elimination) of the 325°C signal due to a few hours of yellow illumination [Fig. 9(a)].

This experiment demonstrated that yellow filters can result in the inadvertent bleaching of the 325°C signal and a foreshortening of the plateau temperature region, but that if red filters are installed then the palaeodose plateau should start at ~260°C for quartz samples that have not been partially reset since deposition. The plateau region will be affected slightly by heating rate, the 325°C peak shifting to lower temperatures at slower heating rates. A difference of ~10°C might be expected by heating at 2.5 K s^{-1} (as used for the Malakunanja II samples) instead of 5 K s^{-1} (Spooner, 1994a: Fig. 2), the latter rate being used for the Nauwalabila I and Allen's Cave samples described below; these were processed in red light and showed a prominent natural TL peak at 325°C, with plateaux beginning at 265–270°C (Murray *et al.*, 1997; Roberts *et al.*, 1994a, 1996). In well bleached samples older than the last interglacial, however, the plateau may not start until higher temperatures (e.g. Huntley *et al.*, 1993a, 1994a; see Section 3.1.1). The Malakunanja II sequence is mostly too young to check the generality of this observation, but the basal TL sample, prepared under red light and dated to ~110,000 years, has a plateau which begins only at 325°C (Roberts *et al.*, 1990a); palaeodoses in the 285–310°C region are ~15% smaller (Roberts *et al.*, 1991). Some of these points will arise later, in discussion of the TL dates for Jinmium (Fullagar *et al.*, 1996).

TL dating of the archaeosediments at Malakunanja II indicated that humans first occupied the site between 61,000 ± 10,000 and 52,000 ± 8000 years ago, the older date being associated with the lowest stone artefact (Roberts *et al.*, 1990a). The random and systematic uncertainties are here added in quadrature, following the standard practice in luminescence dating (Aitken, 1985); in Roberts *et al.* (1990a), the uncertainties were summed arithmetically, this being the more conservative approach to total error estimation by assigning a maximum limit to the systematic error, rather than assuming a Gaussian distribution (Roberts *et al.*, 1990b; Roberts and Jones, 1994). As there is only 10 cm depth of sand deposit between these two sample mid-points, the possibility of downward displacement of artefacts cannot be discounted (e.g. Cahen and Moeyersons, 1977; Hughes and Lampert, 1977; Richardson, 1992), even though such a mechanism seems inadequate to account for the presence of artefacts which differ markedly in size and density (e.g. silcrete flakes, pieces of dolerite and ground ochre, including a lump of high-grade haematite, a grindstone, and numerous quartzite and quartz artefacts) in the zone of first occupation.

A secure *terminus ante quem* for occupation is given, however, by the concentration of artefacts in a small pit feature overlain by sediments dated to 45,000 ± 7000 years. A weighted least-squares linear regression was also performed on the nine Pleistocene TL dates in the sequence to obtain a second estimate of the age of the lowest artefact: this was determined to be 49,900 ± 4700 years (Roberts *et al.*, 1990b), the data being consistent with a linear rate of sand accumulation (as had been noted previously for unoccupied sand aprons in the region; Roberts *et al.*, 1991). Independent support for the TL dates in the upper 2 m of deposit is given by their comparability with ^{14}C ages: one TL date of 15,000 ± 2000 years was bracketed by calibrated ^{14}C ages of 14,700–17,000 and 17,500–18,200 years (2σ range, following Stuiver and Reimer, 1993), while a second TL date of 24,000 ± 4000 years matched, within the estimate of uncertainty, a calibrated ^{14}C age of 20,600–22,300 years (Roberts *et al.*, 1990b; Roberts and Jones, 1994). On the basis of these results, we proposed that this part of northern Australia had been colonised by 50,000 years ago.

The culturally-sterile layers at Malakunanja II were dated to between ~110,000 years and ~65,000 years, which suggested to us (Roberts *et al.*, 1990a) that people did not arrive in this region during the penultimate glacial maximum, when lower sea-level might have facilitated open-water crossings from the north-west and Australia could have been reached by land bridge from Irian Jaya/Papua New Guinea. But could we have missed evidence of older occupations in western Arnhem Land? It is unlikely that the first colonists from Indo-Malaysia used tools made exclusively from perishable materials, such as wood and bamboo, as stone artefacts have been found at early Pleistocene sites in south-east Asia and at middle Pleistocene sites on Flores (see Section 3.3). Early habitation sites may exist on the now-submerged Sahul Shelf, and new excavations at Malakunanja II may reveal a large rock shelter hidden behind the 4.6 m-deep sand apron. If such a shelter exists, then an older occupation may be recorded only in its deepest recesses, with subsequent infilling of the shelter having forced the later human occupants onto the sand apron, where our excavation was placed. As occupation debris is commonly found near the entrances to rock shelters, however, it seems improbable that all traces of human activity would be restricted to the deepest recesses (M. Smith, Personal Communication, 1997).

Certainly, the Malangangerr rock shelter, situated 10 km north-east of Malakunanja II, warrants further study. The basal sediments in the shelter were TL dated to 32,000 ± 7000 years and an overlying unit was dated to 16,000 ± 4000 years, the latter being slightly younger than, but within two standard errors of, the calibrated age range of 20,300–27,000 years for four ^{14}C samples (Roberts and Jones, 1994). The basal age may, however, represent only a *minimum* age for occupation as the lowest artefacts rest directly on bedrock and, thus, could be a "lag" assemblage buried by much younger sediments (as may also have occurred at Pedra Pintada; see Section 4.1.2). The base of the adjoining sand apron was TL dated to ~120,000 years, similar to the date of initiation of the Malakunanja II apron, so future archaeological excavations should perhaps focus on the sedimentary sequence immediately outside the Malangangerr shelter (Roberts and Jones, 1994).

To test our theory that people first arrived in this region between 50,000 and 60,000 years ago, we engaged luminescence dating methods at a second deep archaeological sequence at the foot of the Arnhem Land escarpment: the Nauwalabila I (Lindner Site) rock shelter in Deaf Adder Gorge, 70 km south of Malakunanja II (Roberts *et al.*, 1994a; Roberts and Jones, 1994). A series of ^{14}C determinations had already been made at this site (Jones and Johnson, 1985) and additional ^{14}C dates were obtained to compare against luminescence dates for the upper 2 m of the 3 m-deep sand deposit. Sand-sized quartz grains were extracted to determine the TL and OSL palaeodoses using the multiple-aliquot additive-dose protocol (the OSL aliquots being held at 220°C for 300 s before 514.5 nm illumination). As at Malakunanja II, the environmental dose rate was estimated from high-resolution gamma spectrometry, which again showed no evidence of radionuclide disequilibrium in the ^{238}U or ^{232}Th decay chains. On-site dosimetry measurements were not taken, but laboratory analyses of the bedrock and compacted rubble in the basal quarter of the deposit were made to reconstruct the "infinite matrix" gamma dose rate experienced by the two lowermost samples. These analyses indicated that, despite the heterogenous texture of the deposit, the rubble and bedrock had similar radionuclide activities to the neighbouring sand samples and, therefore, the calculated dose rates should be accurate (Roberts *et al.*, 1994a).

The presence of decomposed rubble posed a much greater threat to the accurate determination of the palaeodose in the two lowest samples. Sand grains liberated by the disintegration of buried rubble will not have been exposed to sunlight since the time of rock formation, resulting in a luminescence signal that is either dose-saturated or, if the ambient temperature of burial is sufficiently high (e.g. Prokein and Wagner, 1994), in a less-than-saturated condition of dynamic equilibrium. Even at a burial temperature of 30°C, the 325°C and 375°C TL peaks in quartz have sufficient lifetimes (> 4 million years, calculated from the values listed by Aitken, 1985, p. 272) for late Quaternary sediments to remain in dose-saturation and, hence, to appear essentially unbleached. Mixing such grains (e.g. through bioturbation) with a population of recently and completely bleached grains will produce grossly inflated TL and optical dates if multiple-grain, multiple-aliquot methods are employed.

A low level of rubble contamination (e.g. less than 10%) will likely result in aliquots containing unequal numbers of contaminant grains: multiple-grain OSL aliquots that have been short-shine normalised should produce multiple-aliquot growth curves that exhibit increasing relative scatter in OSL intensity with added dose (Rhodes, 1990), while a suite of single aliquots (each composed of many grains) should show a correlation between the measured palaeodoses and the corresponding natural OSL intensities (Li, 1994). Single-grain analyses would permit the contaminant grains to identified directly, and are the only solution to the problem of high-level (e.g. more than 10%) contamination. The latter would result in contaminant grains being present on *each* multiple-grain aliquot that consisted of more than a few tens of grains; and a Gaussian distribution is rapidly generated as the level of contamination increases. Severe contamination might be expected close to decayed rubble but would not be detected using the approach of Rhodes (1990) or Li (1994) if each aliquot was uniformly contaminated. Dating of single grains or single-aliquots composed of few grains (e.g. 10 or less) should be used in such instances; otherwise the only means of recognising contamination by rubble or saprolite (*in situ* weathered bedrock) is from a comparison of luminescence and independent age estimates.

Although these concerns were raised by Roberts and Jones (1994), single-aliquot and single-grain methods of palaeodose determination were not available in 1991, when the Nauwalabila I optical dating study was begun. Sediment samples were collected from several levels, including one above and one below the rubble and sand unit that contained the lowest assemblage of artefacts. The latter is similar in composition to that found at Malakunanja II, and included flakes of quartz, quartzite and chert, a grindstone and scraper, and pieces of dolerite and abraded haematite. The rubble unit was not directly sampled for luminescence dating, but the possibility remained that the two adjacent samples may be contaminated. Conventional multiple-aliquot methods were employed for TL and OSL palaeodose determinations, each aliquot holding ~4000 quartz grains. Examination of the dose-response curves showed no evidence of increasing relative scatter with added dose, implying that low-level contamination was not a problem. The possibility of high-level contamination was checked indirectly, by dating the near-basal sediments of the sand apron immediately outside the rock shelter. These sediments have been deposited directly onto bedrock and are not subject to rubble contamination. The OSL date of 58,300 ± 5800 years for the inception of the sand apron coincides with the OSL date of 60,300 ± 6700 years for the start of sediment accumulation in the rock shelter, indicating that the latter date is not grossly distorted by severe rubble contamination (Roberts *et al.*, 1994a, p. 582); a direct test of this proposition is planned using single-grain dating methods (Murray and Roberts, 1997). The indurated nature of the Mesoproterozoic sandstone bedrock might explain the absence of rubble and saprolite contamination at Nauwalabila I. Similar good fortune is expected at Malangangerr and at Malakunanja II, where the lowest artefacts are, moreover, safely positioned 2 m above the nearest rubble or bedrock.

The ~60,300 year-old sample underlies the basal occupation level at Nauwalabila I, while the overlying sample gave an OSL date of 53,400 ± 5400 years. Furthermore, the lowest artefacts at Nauwalabila I are tightly bound within the sand and interlocked rubble unit, so we can discount the possibility of their post-depositional movement, unlike at Malakunanja II. This age bracket for the zone of initial occupation almost exactly matches that obtained at Malakunaja II, reinforcing our earlier conclusion that first settlement of western Arnhem Land occurred between 50,000 and 60,000 years ago.

At the three archaeological sites investigated in the region, rubble contamination appears to be insignificant, and three main lines of evidence argue against insufficient bleaching in antiquity. First, the near-surface samples yielded small TL and OSL palaeodoses: 1–2 Gy and ~0.16 Gy, respectively, the latter corresponding to an age of only 290 ± 60 years. Second, the palaeodose plateaux of the red-lit TL samples (which includes the pair at Nauwalabila I) incorporate both the 325°C and 375°C peaks, which implies a sufficiently prolonged solar exposure to have reset the hard-to-bleach signal as well as the easy-to-bleach signal. Third, there is good agreement between luminescence dates and calibrated ^{14}C dates for charcoal from the upper occupation levels at Malakunanja II and Malangangerr (see above); the same is true also at Nauwalabila I where TL and OSL dates of *circa* 13,500–14,600 and 28,000–30,000 years compare favourably with calibrated ^{14}C ages of 10,500–13,500 (2σ range) and *circa*

24,000–27,000 years, respectively (Roberts *et al.*, 1994a; Roberts and Jones, 1994).

A third possible mechanism to obtain age overestimates is thermal transfer of charge from light-insensitive traps to light-sensitive traps during the OSL preheat treatment. Thermal transfer effects, and the means to overcome them, have been discussed recently for potassium feldspars (Huntley and Clague, 1996) and quartz (Rhodes and Bailey, 1997). Berger (1995, p. 95) suggests that thermal transfer may account for the non-zero OSL age of the near-surface sample at Nauwalabila I, and he also comments that the rise in OSL palaeodose with increasing illumination time (for the ~30,000 year-old sample) may indicate the presence of poorly or non-bleached grains. We did not anticipate a zero age for the near-surface sample which was buried to a depth of 1–6 cm and, therefore, had been exposed for some time to the surrounding environmental radiation field and cosmic rays (Roberts *et al.*, 1993b, 1994a). The OSL age for this sample instead accords well with the age expected from the rate of sediment accumulation deduced from the Holocene ^{14}C chronology and the depth at which glass shards appear in the deposit (Jones and Johnson, 1985). Also, as just discussed, contamination of the older samples would appear to be minimal, and the operation of non-first-order kinetics during illumination (i.e. complex interactions arising from optical transfer of charge) is a more likely cause of rising palaeodose (see Roberts *et al.*, 1994a).

The magnitude of thermal transfer of charge due to a preheat of 220°C for 300 s was examined explicitly by Roberts *et al.* (1993b, pp. 51–52). Negligible transfer in the ~30,000 year-old sample was inferred from two experiments. First, preheating of the "natural" aliquots caused a *decrease* in OSL intensity (consistent with thermal erosion of part of the OSL signal) and not a transfer-induced increase, such as that reported by Godfrey-Smith *et al.* (1988). Second, natural aliquots that had been bleached for 20 h and then preheated gave rise to an OSL signal three orders-of-magnitude smaller than that produced by natural aliquots which had been bleached, given a dose of 11.2 Gy and then preheated. The resulting thermal transfer in the bleached-then-preheated aliquots is thus equivalent to ~0.01 Gy, which is a trivial correction to the measured palaeodose for this sample (~24 Gy) and is an order-of-magnitude less than the palaeodose reported from the near-surface sample. Furthermore, this bleached-then-preheated value is directly comparable to the first point on the dosed-bleached-preheated growth curve which may be used as a non-zero OSL baseline (Huntley *et al.*, 1993b; Huntley and Clague, 1996), an approach strongly advocated by Berger (1995, p. 95).

If the western Arnhem Land dates are not too old, might they be too young? To make the dates consonant with initial colonisation during the penultimate glacial maximum requires that the palaeodoses be greatly underestimated and/or that the dose rates be significantly overestimated. The TL and OSL palaeodoses are unlikely to suffer from anomalous fading, as none was detected in samples that were dosed and then stored at room temperature for periods of up to 400 days (Roberts *et al.*, 1990a, 1994a). The high-resolution gamma spectrometry facilities are calibrated against international standards, rendering a factor-of-two error improbable, and the thickness of the rock shelter roof was factored into the estimate of the cosmic-ray dose rate (which would otherwise have been overestimated). The luminescence dates will increase by ~0.8% for each 1% rise in sample water content, so the *circa* 60,000 year-old dates at Nauwalabila I and Malakunanja II would increase by only 7000 and 10,000 years, respectively, if the samples had been *permanently* saturated since deposition; this proposition is hardly credible for a habitation site on highly porous and permeable sands which, if saturated, would turn into a slurry.

The western Arnhem Land sites have been discussed in detail because they represent still, in my view, the oldest reliable evidence for human occupation of the continent. Malakunanja II and Nauwalabila I also contain the earliest evidence for the use of pigments (presumably for rock art or body decoration), in the form of red and yellow ochres, and haematite "crayons" with ground facets and striations, excavated from the 50,000–60,000 year-old levels (Morell, 1995; Roberts *et al.*, 1990a, 1994a). Recent claims have been made for earlier landfall and rock art in the Kimberley region of northern Australia (Fullagar *et al.*, 1996), but the TL dates for Jinmium rock shelter which support these claims are of questionable validity (see below). Before scrutinising the Jinmium data, however, it is pertinent to mention the optical dates obtained from archaeosediments at two painted rock shelters in Cape York: Ngarrabullgan Cave (David, 1993; David *et al.*, 1997) and Mushroom Rock (Wright, 1971b; Morwood and Hobbs, 1995; Morwood *et al.*, 1995b).

4.2.6. *Cape York*. The basal strata of the 40 cm-thick deposit in Ngarrabullgan Cave have been intensively dated by AMS and beta-counting ^{14}C methods, with 20 dates (18 from individual pieces of charcoal) currently available for the ~32,500 years BP unit; an underlying stratum has yielded a ^{14}C age of >37,200 years BP, although this may represent an outlier of the ~32,500 years BP suite of finite age determinations, which spans the period *circa* 28,800–36,100 years BP (David *et al.*, 1997). Sand-sized quartz grains were extracted from the basal strata but, as the sample was positioned only 5 cm above the bedrock floor, concerns were raised about the possibility of contamination by saprolite. Consequently, additive-dose OSL palaeodoses were determined using not only conventional multiple-aliquot procedures (short-shine normalisation, preheat of 220°C for 300 s, signal integration over the full period of stimulation (minus background) by a halogen lamp filtered to 500 ± 40 nm) but also the newly-devised single-aliquot protocol (Murray *et al.*, 1997); the latter involved a "preheat plateau" test (Aitken, 1994) using 24 multiple-grain aliquots, and a series of brief stimulations by 420–550 nm "green" light. The environmental dose rate was estimated from *in situ* and high-resolution gamma spectrometry, the two measures being accordant and secular equilibrium being observed. The average palaeodose of 48 ± 2 Gy for the six aliquots in the preheat plateau region (280–300°C for 10 s) showed agreement with the multiple-aliquot palaeodose of 47 ± 4 Gy, indicating that low-level contamination of the sample by decomposed rubble or saprolite is not prevalent (David *et al.*, 1997). Single-aliquot and single-grain analyses of this sample using a novel regenerative-dose protocol (Murray and Roberts, 1997) have confirmed these findings (J. Olley, Personal Communication, 1997). High-level contamination can also be discounted, as the corresponding weighted mean OSL age (34,700 ± 2000 years) is a few millennia older than the average ^{14}C age of ~32,500 years BP, as expected for uncalibrated ^{14}C determinations of this antiquity (see Section 2.2).

Although Ngarrabullgan Cave is the oldest human occupation site so far recorded in Cape York, Mushroom Rock boasts one of the deepest archaeological deposits in Australia, containing stone artefacts to depths of 4.4 m (Morwood *et al.*, 1995b). Optical dating of the Mushroom Rock sediments has, unlike Ngarrabullgan Cave, revealed severe problems of contamination of the lowest artefact-bearing levels. Radiocarbon determinations had been made previously on charcoal preserved in the upper 2 m of deposit, and TL dates had been obtained from the quartz sediments (using a combined additive/regenerative approach and thick-source alpha counting; Morwood *et al.*, 1995b). The first warning signs were given by the discord between the ^{14}C and TL dates for the habitation deposits: calibrated ^{14}C dates of ~7700 and ~2350 years were poorly matched by TL dates of ~11,400 and ~6800 years, respectively. In the underlying carbon-sterile deposits, a TL date of 37,400 ± 3900 years was obtained from a depth of 4 m (i.e. above the level of the lowest artefacts). Owing to the uncertainty surrounding the validity of these TL dates (Morwood *et al.*, 1995b), an optical dating study was initiated by the author, R. Jones and M. Morwood in 1994 to confirm or refute the apparent great antiquity of the archaeological sequence.

The OSL palaeodoses were obtained, in the first instance, from multiple-aliquot additive-dose procedures applied to coarse-grain quartz (identical experimental conditions to those used at Ngarrabullgan Cave), and the environmental dose rates were also determined using the same methods, and with the same outcome, as at Ngarrabullgan. An optical date of ~90,000 years was indicated for the level of the lowest artefact. This remarkable finding was soon thrown into doubt, however, by an apparent age of ~60,000 years for a sample only 15 cm higher up the profile. As no stratigraphic unconformity was visible, the difference in age between these two closely-spaced samples seemed at odds with the rapid rate of sedimentation (1 m per 4000 years) implied by the ^{14}C chronology for the upper levels. The lowermost sample had been collected from only 50 cm above the top of a strongly mottled zone, which is either saprolite or, from its micromorphology (J. Magee, Personal Communication, 1994), an ancient colluvial or alluvial deposit. Mixture of the latter with recently bleached sediments was considered as the probable cause of the problem, the samples nearest the mottled zone being contaminated to a greater degree than those further away.

At that stage, true single-aliquot methods had not been devised for quartz, but an attempt was made to examine the palaeodose distribution for individual grains using a simple regenerative-dose technique (see Murray *et al.*, 1995; Olley *et al.*, 1997b). The "natural" OSL intensities of (preheated) single grains were measured, beta doses were then administered, and the total OSL signals were again measured (after a second preheat); the ratio of the first and second light sums multiplied by the given dose provides a first-order approximation of the palaeodose (the non-linear dose-response of quartz, and possible sensitivity changes, preventing an accurate determination). Single grains from the "60,000 year-old" sample proved, unfortunately, to be too dim, so the process was repeated

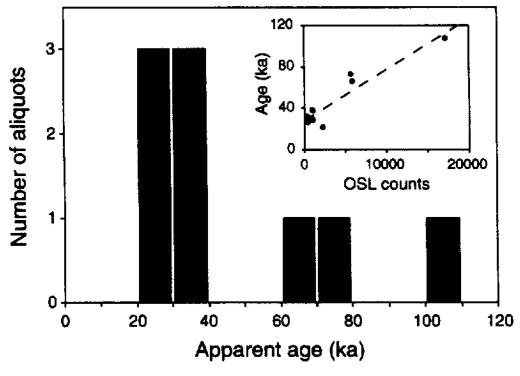

Fig. 10. Frequency distribution of apparent OSL ages (in thousands of years) obtained using single aliquots of quartz from Mushroom Rock, Cape York. Each aliquot holds 10 grains. The inset figure shows the sum of OSL counts (minus the background count rate) for each aliquot plotted against its apparent age. Note the increase of OSL intensity with age, indicating the presence of poorly bleached grains on at least some of the aliquots.

on single aliquots, each composed of 10 grains. This number was chosen to increase the OSL intensity, yet maintain a low probability of contamination. The apparent palaeodoses and corresponding ages (calculated using the mean dose rate) for nine aliquots showed a wide distribution: six aliquots clustered between *circa* 21,000–38,000 years, while the remaining three aliquots produced apparent ages of 66,000, 72,000 and 108,000 years (Fig. 10). Furthermore, the oldest ages are associated with the brightest aliquots (Fig. 10, inset), as predicted for aliquots contaminated by poorly bleached grains (Li, 1994). For this sample, the youngest group of apparent ages (mean ~30,000 years) may represent an uncontaminated population (although this remains to be shown), whereas the older aliquots contain one or more contaminant grains.

Optical dating of the lowest artefact-bearing levels at Mushroom Rock is in progress, using single-aliquot additive-dose (Murray *et al.*, 1997) and regenerative-dose (Murray and Roberts, 1997) protocols for quartz. Multiple-aliquot TL and OSL methods will clearly generate considerable age overestimates, as initially suspected from the mismatched ^{14}C/TL dates for the overlying deposits. A date of 30,000–40,000 years for first occupation of this rock shelter would be consistent with other evidence in Cape York, such as the ^{14}C date of ~32,000 years BP for the lowest artefacts at the nearby site of Sandy Creek 1 (Morwood and Hobbs, 1995; Morwood *et al.*, 1995a), and the ^{14}C and OSL ages for Ngarrabullgan Cave (David *et al.*, 1997).

4.2.7. *Jinmium.* With these findings in mind, it is appropriate to review the TL evidence for human occupation of Jinmium rock shelter (Fullagar *et al.*, 1996). Claims for occupation before 116,000 ± 12,000 years, and possibly as early as 176,000 ± 16,000 years, made media headlines around the world, as did the dates of *circa* 58,000–75,000 years for buried circular engravings (pecked cupules) on the rock shelter wall and on a fallen sandstone fragment (Bahn, 1996; Holden, 1996). When R. Fullagar first told me of these extraordinary results in 1994, I recounted the problems encountered at Mushroom Rock and suggested that optical dating of the Jinmium archaeosediments be

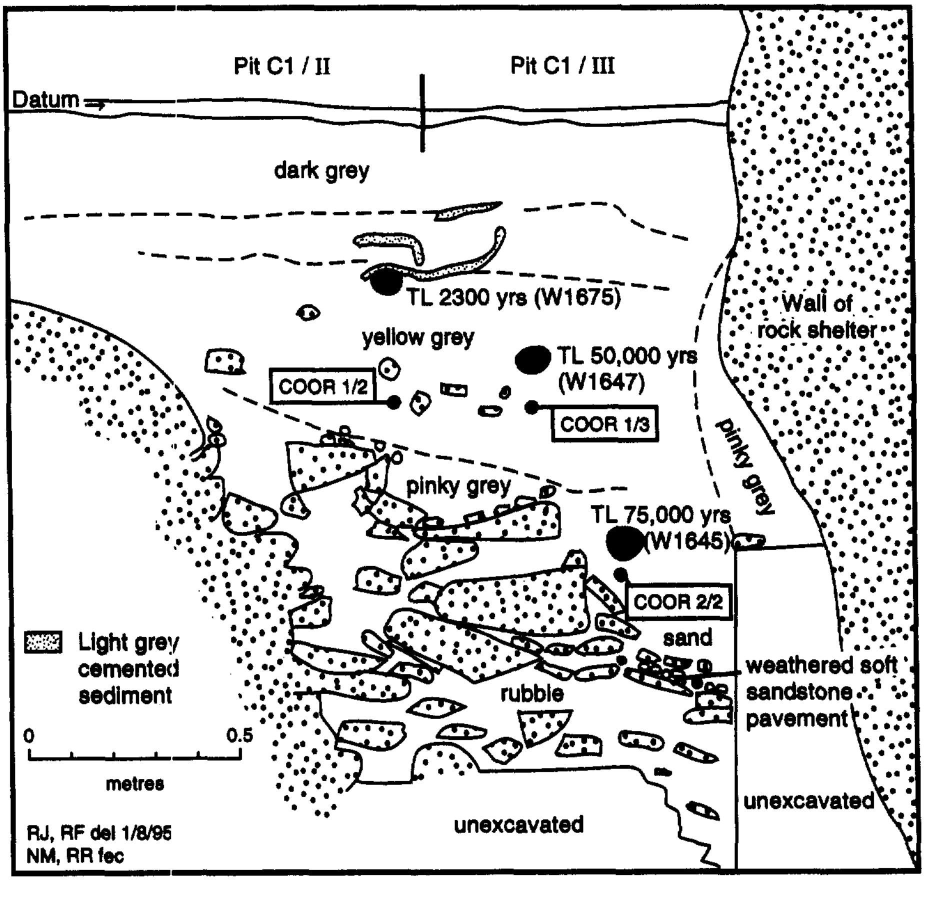

Fig. 11. Stratigraphic section of the north face of Trench C1, squares II and III, at Jinmium rock shelter. Locations of TL samples (including W1647) are shown with their published ages, and locations of OSL and elemental carbon ^{14}C samples are indicated by their COOR field codes.

carried out to check the TL dates; the latter had been obtained from quartz sand grains using a combined "total bleach" additive/regenerative multiple-aliquot procedure (each aliquot holding ~3000 grains, heated at 5 K s^{-1}) and thick-source alpha counting. Fresh sediment samples were later collected by the author, R. Jones and R. Fullagar, and *in situ* measurements of the gamma dose rate were made. Dating of the Jinmium deposit using state-of-the-art OSL and AMS ^{14}C methods is underway (Gibbons, 1997c), the latter exploiting the procedures developed by Bird and Gröcke (1997) to extract elemental carbon from the traces of soot preserved in marine cores. In the absence of these alternative chronologies, what do the data published by Fullagar *et al.* (1996), my field observations at Jinmium, and experience gleaned from luminescence studies of other Australian quartz sediments reveal about the probable accuracy of the TL ages?

Several lines of evidence suggest that the TL dates published for the key samples at Jinmium (i.e. those from Trench C1, squares I to III; see Fig. 11) are substantial overestimates of the true ages of sediment deposition. From an examination of their TL data, the Trench C1 samples do not present an internally consistent picture of having been well bleached in antiquity. The natural TL glow curve shown for sample W1647 [Fig. 12(a)] shows no evidence of a TL signal at 325°C, despite having supposedly been buried for 50,000 years. The glow curve is dominated instead by the hard-to-bleach peak at 375°C and is comparable to the TL signal that typically remains after selective bleaching (e.g. Prescott and Purvinskis, 1991, 1993; Spooner *et al.*, 1988). When the "plateau test" is performed, the Trench C1 samples have foreshortened plateaux that *begin* at 375°C, whereas the samples collected from the sand apron outside the rock shelter (e.g. W1752) exhibit plateaux extending over the temperature region 300–500°C. Sample W1752 has a prominent natural 325°C signal [Fig. 12(b)], but as the plateaux for this and other sand apron samples do not extend to 270°C, they cannot include all of the 325°C TL peak and, thus, should not be used to infer adequate bleaching in antiquity. If the Jinmium sand apron is constructed in the same manner as those in western Arnhem Land, however, then the constituent sand grains are apt to be well bleached at deposition; perhaps the use of yellow laboratory illumination, which caused a "double plateau" in the Arnhem Land samples, is partly to blame for the plateaux starting at only 300°C. The conspicuous failure of the Trench C1 samples to pass the "plateau test" is more alarming. By starting at 375°C, these plateaux do not *even* include all of the 375°C TL peak and, thus, strongly indicate this hard-to-bleach signal was not completely reset in antiquity.

Fullagar *et al.* (1996, p. 768) explain the foreshortened plateaux in terms of "partial re-exposure *following* deposition rather than insufficient exposure *prior to* deposition" (their emphases). This explanation is inadequate, however, as a brief re-exposure would zero only the easy-to-bleach (325°C) signal and, upon reburial, a substantial hard-to-bleach (375°C) signal would be retained. In such instances, the elapsed time since last exposure to

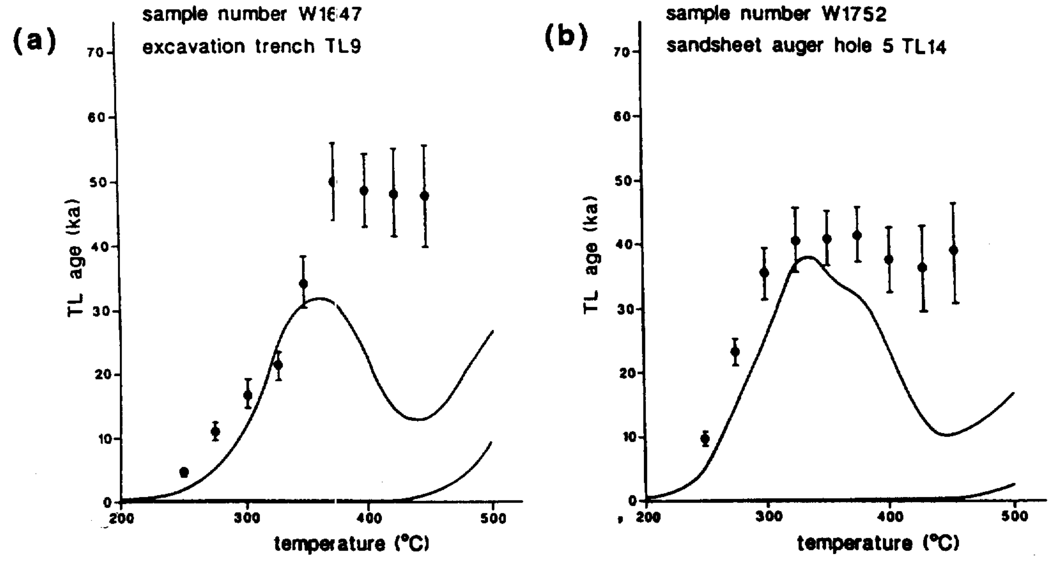

Fig. 12. (a) TL age "plateau test" for Jinmium sample W1647, with the natural TL glow curve (solid line) shown for a heating rate of 5 K s^{-1}. This sample was collected from the archaeological excavation (see Figure 11) and exhibits a foreshortened plateau. (b) Plateau test and natural TL glow curve (solid line) for sample W1752. This sample was collected from the sand apron (at 4 m depth) outside the rock shelter, and has a longer plateau . TL ages are in thousands of years (after Fullagar *et al.*, 1996).

sunlight can be accurately determined only from the easy-to-bleach signal. In addition, the re-exposure argument is difficult to reconcile with the observed increase of TL age with depth. For the dates to remain in correct stratigraphic order requires that quartz grains be raised to the ground surface, exposed to sunlight for a *brief* period (so as not to reset the 375°C signal), and then returned to the *same* level from which they originated. Such a process of reworking seems unlikely. If grains from deep in the section had been redeposited at a higher level, and near-surface grains had been redeposited further down the profile, then the entire deposit would be characterised by a single, average age.

A first-order approximation of the time since sample W1647 was last exposed to sunlight can be estimated from the size of the easy-to-bleach TL signal [Fig. 12(a)]. The natural glow curve for this sample is composed mainly of the residual 375°C peak but, as this sample has been buried for at least 1800 years (as indicated by the ^{14}C date on charcoal from an overlying level), some fraction of the TL signal at 325°C must represent the "pure" 325°C peak that has been regenerated since the last bleaching event. Based on the data presented in Spooner *et al.* (1988) for selectively bleached quartz, and the probable contribution of the 375°C peak to the natural TL at 325°C for sand apron sample W1752 [Fig. 12(b)], we might expect that 10–50% of the TL at 325°C is due to regrowth of the "pure" 325°C peak. As Fig. 12(a) shows the TL age at 325°C to be ~21,000 years, then this fractional adjustment implies that only 2100–10,500 years have elapsed since the easy-to-bleach signal was last reset by sunlight. A similar argument was presented in February 1997 by N. Spooner at the Sixth Australasian Archaeometry Conference in Sydney, the details of which are being prepared for publication with J. Prescott (Personal Communication, 1997; Gibbons, 1997c).

These ages are much younger than the 50,000 years claimed for this sample by Fullagar *et al.* (1996), but they are consistent with the rate of sand accumulation outside the rock shelter and with the lack of field evidence (such as a stratigraphic unconformity) for a hiatus of ~40,000 years at ~50 cm depth; the latter was inferred by Fullagar *et al.* (1996) on the basis of a 2300 year-old date for a TL sample positioned ~20 cm above the supposed 50,000 year-old level. The foreshortened TL age plateaux are not illustrated for the deeper samples, so the above fractional adjustment cannot be applied directly to recalculate their TL ages from the "pure" 325°C signal. Nonetheless, assuming the same proportional error applies throughout the profile, the ~116,000 and ~176,000 year-old samples may be as young as 5000–24,000 and 7000–37,000 years, respectively.

This explanation for the TL age overestimates at Jinmium fails, however, to account satisfactorily for why the 375°C peak is so poorly bleached in the rock shelter deposits, yet is adequately bleached on the adjacent sand aprons. The aprons, as in western Arnhem Land (Roberts *et al.*, 1991), are composed of sand grains eroded from the local sandstone bedrock outcrops and transported by slopewash (Fullagar *et al.*, 1996, p. 766). This process usually results in the 325°C and 375°C peaks both being reset before the sediments are finally buried (Roberts *et al.*, 1990a, 1991, 1994a). In both regions, modern grains are deposited without surface stainings or oxide coatings, the latter being acquired post-depositionally on the sand aprons; the Jinmium rock shelter samples are, moreover, only lightly stained. An alternative explanation for the TL age overestimates involves a combination of two factors: contamination of the Trench C1 samples by decomposed rubble and inadvertent bleaching of all samples during laboratory pretreatment. This is my preferred interpretation of the Jinmium data, as it better fits the geomorphological evidence and additionally accounts for the slightly foreshortened plateaux of the Jinmium sand apron samples and other quartz samples processed by the same laboratory.

The Jinmium rock shelter is composed of Upper Carboniferous sandstone and conglomerate, which weathers readily and produces rubble that is easily cut with a hand trowel. A similar situation prevails at Mushroom Rock, whereas the Arnhem Land sites are characterised by indurated Mesoproterozoic sandstone bedrock. Disintegration of buried rubble at Jinmium will liberate dose-saturated (i.e. effectively "unbleached") grains, which can then become mixed into the sediment column through bioturbation. The most contaminated sediment samples are apt to be those collected closest to the rubble. As the amount of rubble increases with depth at Jinmium, the ages of the basal samples are likely to be the most distorted and those higher up the profile should be less severely contaminated, giving rise to a series of ages that are in correct stratigraphic order but are all biased to some extent. The adjacent sand aprons are not contaminated by rubble, so their TL ages should be more accurate.

The degree of contamination of the Trench C1 samples cannot be evaluated from the published data, but one feature is clear—the saturated 325°C signal associated with any contaminant grains is largely absent in the Trench C1 samples, yet a 325°C peak is present in the sand apron samples. In both cases, multiple-grain aliquots and yellow laboratory light filters were used. The latter may provide a solution to this puzzle. If the well bleached quartz grains extracted from the sand apron behave in a similar fashion to their Arnhem Land counterparts, then their preparation under yellow illumination could reduce the natural 325°C signal by ~50% [see Fig. 9(a)]; some 325°C signal will remain, but a large proportion will be inadvertently bleached in the laboratory. Variations in the magnitude of this effect are expected between samples, owing to differences in bleaching behaviour and duration of illumination (e.g. Spooner and Prescott, 1986; Roberts *et al.*, 1991). The foreshortened plateau (starting at ~325°C) of sand apron sample W1752 is consistent with this proposition, the lack of a "double plateau" perhaps indicating a greater degree of inadvertent bleaching.

The glow curve of the rock shelter sample W1647 [Fig. 12(a)] can be likewise explained. If this sample consisted *entirely* of contaminant grains, then its TL signal should be fully saturated with a dose of ~250 Gy in both the 325°C and 375°C peaks. The latter will not be affected by yellow illumination, so its palaeodose of only 50 Gy indicates that 20%, or less, of the grains on each aliquot of this sample are contaminants. Consequently, if the 325°C signal was inadvertently bleached in sample W1647 by the same absolute amount as in sample W1752 (whose palaeodose is almost double that of W1647), then the "pure" 325°C peak would be almost completely erased, as is observed [Fig. 12(a)]. Older samples in the Jinmium sequence may have some natural 325°C peak remaining, but these samples, together with all contaminated Trench C1 samples, will fail the total bleach "plateau test" because the "pure" 325°C signal has been completely or partially reset and the 375°C signal is derived from a mix of bleached (i.e. sand apron) and unbleached (i.e. rubble) grains. The true age of sample W1647 depends critically on the degree of rubble contamination. A late Holocene age is calculated assuming each multiple-grain aliquot is composed of 20% rubble grains, whereas an age of 40,000 years is derived for 5% contamination.

The foregoing two interpretations of the Jinmium data have been proposed to explain the predominance of the hard-to-bleach TL signal and the foreshortened palaeodose plateaux in the Trench C1 samples. Both interpretations imply that the published TL ages are too old, a conclusion supported by three independent forms of geomorphological evidence: the lack of a stratigraphic unconformity between the closely-spaced 2300 and 50,000 year-old levels; the absence of post-depositional red staining of the Trench C1 sand grains compared to that developed in the supposedly coeval sand aprons; and the apparent sedimentation rate inside the rock shelter being an order-of-magnitude less than the rates for the adjacent sand apron and square C1/IV, excavated only 10 m outside the shelter.

The degree of age overestimation is contingent on the mechanism invoked to explain the inadequate "bleaching", be it through insufficient sunlight exposure of *all* the grains at deposition or through rubble contamination in tandem with yellow laboratory illumination. The former runs counter to geomorphological expectations, but the latter necessitates two compounding influences, neither of which are yet proven for Jinmium. Price (1994) reported that several quartz samples yielded the same palaeodose from the TL signals at 325°C and 375°C, indicating that his yellow laboratory lights are safe. This evidence is insufficient, however, as all sample preparations were conducted in yellow light, so the "pure" 325°C peak may have been bleached to a greater or lesser extent in every sample before the first TL measurements were made; the obtainment of similar palaeodoses at 325°C and 375°C could be argued to show only that the low-temperature "tail" and the peak of the hard-to-bleach signal contain the same palaeodose. Fullagar *et al.* (1996, p. 758) claim support for the TL dating procedures used at Jinmium from the studies of Nanson *et al.* (1991) and Shepherd and Price (1990). The plateaux published for these samples begin at 350°C and 275°C, respectively: the latter is an acceptable plateau, the former is not. Such sample-to-sample differences prevent me from making a definitive judgement on the issue of laboratory illumination, but a convincing test would involve a comparison of samples prepared under red and yellow light (e.g. Fig. 9).

Optical dating of the Jinmium sediments, using fresh samples prepared under red light, should show which (if either or a combination) of the above two interpretations of the TL data is correct. If the first interpretation is correct (i.e. solar resetting of the 325°C peak but not the 375°C peak), then multiple-aliquot OSL methods should yield the true palaeodose. On the other hand, these procedures will yield palaeodose overestimates if the samples are contaminated with rubble (the pretreated grains not being inadvertently bleached by red light); the dose acquired by the non-contaminant grains since last exposure to sunlight can, in such circumstances, only be revealed by single-aliquot or single-grain protocols. These predictive tests are currently being performed on the Jinmium samples.

4.2.8. *The hand of man?* TL dating has also featured in previous proposals for human occupation of Australia prior to the last interglacial. At Point Ritchie, on the coast of Victoria, an unusual shell deposit (showing traits of an Aboriginal midden) and discoloured cobbles (suspected of being oven stones) were investigated using a variety of numerical dating methods (Prescott and Sherwood, 1988; Sherwood *et al.*, 1994). The shell bed (composed chiefly of *Turbo undulatus*) had yielded finite ^{14}C dates of *circa* 25,000–42,000 years BP from charcoal, calcrete, rhizomorphs, and calcite and aragonite from *T. undulatus* shells. A total bleach TL date of ~80,000 years (later revised to 67,000 ± 10,000 years) was obtained from unheated quartz sediments extracted from the poorly cemented shell-bearing calcarenite, while quartz grains separated from the presumed hearth stones (found in the same stratigraphic layer) gave ages of *circa* 155,000–170,000 years, assuming the TL signals were zeroed by heating. As these ages are much greater than that obtained for the sediment, it was concluded that the discoloured cobbles were not burnt sufficiently to reset the TL "clock" and, therefore, cannot be classed as unequivocal artefacts (Sherwood *et al.*, 1994). If sunlight is the resetting mechanism, then the non-zero "residual" TL level reduces the dates to *circa* 115,000–125,000 years, indicative of a last interglacial age for the sediment from which the cobbles formed. As a similar or younger age for the *T. undulatus* shells was inferred from amino-acid racemization and ESR analyses (Goede, 1989; Sherwood *et al.*, 1994), this supposed "midden" may have been formed naturally during high sea-levels of the last interglacial.

Other claims for human landfall in Australia at around the time of the penultimate glacial maximum are based on marked changes in the types of vegetation and the increased abundance of charcoal in pollen cores (Singh and Geissler, 1985; Kershaw, 1994). These changes have been attributed to the deliberate ignition of bushfires by humans, although no associated archaeological evidence has yet been found. In addition, numerical dating methods have not been used to date the crucial horizons in either the Lake George core (from south-eastern New South Wales; Singh and Geissler, 1985) or the Ocean Drilling Program Site 820 marine core (from the edge of the continental shelf off north-east Queensland; Kershaw *et al.*, 1993). These cores have been correlated with the oxygen isotope deep-sea record, although sedimentation rates are variable at both locations (Singh *et al.*, 1981; Peerdeman *et al.*, 1993). A linear extrapolation of the ^{14}C age-versus-depth curve derived from the upper part of the Lake George core indicates an age of ~60,000 years at the depth of the supposed penultimate glacial maximum (Wright, 1986b). A TL study of this 18 m core was undertaken by Mortlock and Price (1984). The natural TL of quartz sand grains, collected at depth intervals down the core, showed a steady increase in the 375°C signal intensity to a depth of ~4 m, followed by a 4 m section of fairly constant TL, and then a renewed increase in signal intensity down the lowest 10 m of core. TL ages were not calculated, but the uniform TL signal for the middle section of core was tentatively attributed to a large and sudden depositional event. As the major change in pollen type and charcoal abundance occurs at a depth of ~4 m, confirmation of their findings could resolve the chronological uncertainties of the Lake George core. Optical dating of single grains of quartz sand from these lake and marine cores is planned to obtain numerical age estimates for the key horizons.

4.2.9. *Kimberley to Koonalda.* At the present time, therefore, claims for occupation of Australia prior to 60,000 years are unsupported by compelling archaeological or chronological proof. The oldest reliable dates are those of 50,000–60,000 years from western Arnhem Land, although two rock shelter sites in the Kimberley region of Western Australia hold promise of great antiquity. Uncalibrated ^{14}C dates of up to 39,700 ± 1000 and 28,060 ± 600 years BP have been obtained from the non-basal cultural levels at Carpenter's Gap 1 and Widgingarri 1, respectively (O'Connor, 1995; Veth, 1995), and OSL dating of quartz sediments from these and the underlying deposits is in progress. South of the Tropic of Capricorn, no archaeological site has yet been dated to more than 45,000 years by ^{14}C or luminescence methods (bearing in mind that uncalibrated ^{14}C ages of ~38,000 years BP correspond to calendar-year ages of 40,000–45,000 years; see Section 2.2). As with the sites in northern Australia, the author is involved in a program of luminescence dating of southern sites that have yielded ^{14}C dates in excess of 30,000 years BP. Confirmation or refutation of the existing ^{14}C chronology by luminescence methods will provide a test of the various theories proposed for the rate of continental colonisation and the routes taken by these first arrivals—whether they clung to the coastline and inland waterways (e.g. Bowdler, 1977), spread rapidly through all major ecological zones (e.g. Jones, 1989), or took further tens of millennia to settle the southern fringe and arid interior of Australia.

Devil's Lair is one of the oldest archaeological sites in south-west Western Australia, having yielded ^{14}C ages in excess of 30,000 years BP for first occupation (Dortch, 1979; Dortch and Dortch, 1996). Optical dating of these cave deposits is being attempted using single-aliquot, as well as multiple-aliquot, methods to circumvent problems of incomplete bleaching of the sediments washed into the cave. The short distance (~15 m) from cave entrance to deposition site should not thwart dating at Devil's Lair, but the much longer (~150 m) entrance slope into Koonalda Cave, on the karst Nullarbor Plain, was held responsible for OSL age overestimates at the "Gallus site" (Roberts *et al.*, 1996). Sediment samples collected from the latter excavation (Wright, 1971a) produced multiple-aliquot quartz OSL dates of 60,000–70,000 years, compared with calibrated ^{14}C ages of *circa* 21,000–28,000 years for multiple charcoal samples. The OSL date of ~9200 years for a fluvial sample deposited recently on the cave floor further demonstrated the inadequacy of sediment bleaching in this deep cave system. Aeolian sediments may enter the doline in a fully bleached state but they acquire a significant palaeodose *en route* to the cave floor owing to their intermittent storage along the entrance slope and their remobilisation by rivulets in the dimness of the cave. Furthermore, the degree of age overestimation will vary from sample to sample, depending on the specific history of the transport route and cumulative residence time. Palaeodose determinations on individual grains from a single habitation level may show significant differences between grains, but identification of the "true" palaeodose requires that at least one of these grains was transported rapidly to the site of final deposition—a requirement that may not be satisfied—and that all other factors contributing to the observed spread in single-grain quartz palaeodoses (Murray and Roberts, 1997; Roberts *et al.*, 1997; J. Olley, Personal Communication, 1997) are held constant.

4.2.10. *Allen's Cave.* Optical dating of archaeosediments proved more successful at Allen's Cave, located ~70 km west of Koonalda Cave. This shallow rock shelter has been studied in detail for the variety of radionuclide disequilibria observed in the ^{238}U chain (Olley *et al.*, 1997a; Roberts *et al.*, 1996) and for the application of multiple-aliquot (Roberts *et al.*, 1996), single-aliquot (Murray *et al.*, 1997) and single-grain (Murray and Roberts, 1997) methods of palaeodose determination. Charcoal samples from a well-preserved hearth had been dated previously by ^{14}C to *circa* 9300–9500 years BP (2σ calibrated age range of *circa* 10,000–11,000 years), and the quartz sediments collected from 5 cm beneath this hearth yielded closely matching OSL dates of 10,100 ± 600 (multiple-aliquot, additive-dose), 9700 ± 600 (single-aliquot, additive-dose), 10,300 ± 500 (single-aliquot, regenerative-dose), 9400 ± 700 (single-grain, additive-dose) and 10,300 ± 700 (single-grain, regenerative-dose) years. The same coarse-grain quartz gave an additive-dose TL date of 11,100 ± 900 years (plateau region 265–380°C, which includes all of the dominant 325°C peak) and ESR dates of *circa* 9800–13,300 years using three newly discovered light-sensitive ESR signals, observed at g = 1.9870, 1.9842 and 1.9162 at a temperature of 77 K (Yoshida, 1997). The excellent agreement found between these ^{14}C, OSL, TL and ESR age determinations is further enhanced by the young OSL age of 210 ± 30 years obtained from the *near-surface* sediments. These data, in stark contrast to those for Koonalda Cave, provide compelling evidence for adequate bleaching of the aeolian sediments deposited in Allen's Cave.

The additive-dose and regenerative-dose single-grain OSL dates are calculated using the average palaeodose from 28 grains (~22 Gy) and 25 grains (~24 Gy), respectively, although the individual palaeodoses show a broad, quasi-Gaussian distribution with standard deviations of 5–6 Gy for both data sets (Murray and Roberts, 1997). A major cause of this spread is probably the variable beta microdosimetry experienced by the grains, some of which may be embedded in aeolian sediments whereas others may be surrounded by carbonate derived from the local limestone. High-resolution gamma and alpha spectrometric analyses of the "pure" carbonate and aeolian fractions have shown that their annual beta doses differ sufficiently (~0.14 and ~2.6 mGy, respectively) to account for the observed spread in palaeodose (Olley *et al.*, 1997a). The magnitude of heterogeneity in the beta dose rate will vary from location to location, but is apt to plague all sedimentary deposits and, hence, complicate the interpretation of single-grain palaeodose distributions. Nonetheless, a population of well bleached grains with variable microdosimetry might be expected to produce an approximately normal, uni-modal distribution, whereas the inclusion of poorly bleached grains should give rise to a skewed or multi-modal distribution.

In addition to the inhomogenous distribution of radionuclides through the Allen's Cave deposit, several types of disequilibria were measured in the ^{238}U decay series (Olley *et al.*, 1997a). Some of these disequilibria, such as the excess of ^{230}Th over both its parent ^{238}U (by up to 450%) and its daughter ^{226}Ra (by up to 190%), cause time-dependent variations in the dose rate during the period of sample burial. The evolution of the dose rate through time was therefore modelled, and thorium isotope ratios (^{230}Th/^{232}Th) were used to demonstrate that the deposit is composed of sediments derived from two distinct sources. The near-surface sediments are derived from an unidentified source and exhibit the greatest excesses of ^{230}Th, as well as ^{210}Pb excesses from atmospheric fallout. The bulk of the deposit (from 10 cm depth to the base of the excavation at 510 cm) has ^{230}Th/^{232}Th ratios comparable with those of the sediments on the surrounding Nullarbor Plain, and ^{210}Pb/^{226}Ra ratios consistent with an average of 0.74 ± 0.03, implying ~25% radon escape irrespective of depth.

The lowest artefact and hearth at Allen's Cave have been dated to 39,800 ± 3,100 years, using multiple-aliquot additive-dose OSL procedures (Roberts *et al.*, 1996). An independent check on this date is forthcoming via AMS ^{14}C dating of elemental carbon extracted from the basal hearth (using the protocols devised by Bird and Gröcke, 1997). This OSL date is the oldest yet reported for human occupation of the Australian arid zone, slightly preceding the TL and calibrated ^{14}C dates of 30,000–35,000 years for first human presence at Lake Mungo (see Section 4.2.1) and Puritjarra rock shelter in the "red centre" (Smith *et al.*, 1997).

4.2.11. *Puritjarra.* The application of TL methods to unburnt archaeosediments at Puritjarra began in 1987, after publication of the first set of ^{14}C ages for this site (Smith, 1987). A decade of research at Puritjarra has inspired the development of the "selective bleach" TL technique and has revealed some interesting discrepancies between the ^{14}C chronology derived from charcoal and the luminescence dates obtained from heated and unheated quartz sediments. Both methods agree on a date of ~35,000 years for initial habitation of the rock shelter, with the underlying culturally-sterile sediments being TL dated to ~45,000 years using both total bleach and selective bleach procedures in combination with the additive/regenerative "Australian slide" technique (Smith *et al.*, 1997). Within the cultural levels, however, the ^{14}C and TL chronologies diverge markedly. Both sets of dates are in correct stratigraphic order, but the age-versus-depth curve for the ^{14}C dates is slightly concave in shape, whereas TL dates indicate a linear rate of sediment accumulation (Fig. 13). The two data sets agree best where their age-versus-depth curves intersect: at the 35,000 year-old level and near the ground surface (where selective bleach TL yielded a zero age for grains removed by sticky tape from the modern surface). At intermediate depths, the selective bleach TL ages are 5000–10,000 years older than their *calibrated* ^{14}C counterparts.

In their search for an answer to this riddle, Smith *et al.* (1997) canvassed a series of possibilities, including contamination of ^{14}C samples by modern carbon, vertical displacement of artefacts and charcoal, and large-scale disturbance of the deposit by soil fauna or humans. Although contamination of the older ^{14}C samples remains a possibility, such an explanation does not account for the large age differences in the Holocene and terminal Pleistocene levels. The stone artefact typology and the phytolith (polymerised biogenic silica) record of the Last Glacial Maximum support the ^{14}C chronology, so a number of options were explored to explain the aberrant TL ages. Clay coatings on the quartz sand grains were considered as a possible cause of incomplete bleaching in antiquity. For the Holocene levels, the selective bleach ages were younger than their total bleach counterparts but still too old compared to the ^{14}C ages. The two TL methods, supplemented by OSL dating at three different laboratories (Adelaide, Oxford and Simon Fraser), gave similar results for the Pleistocene sediments. Beta microdosimetry effects (due to the clay coatings), water content variations, and radioactive disequilibrium were also considered, but were found to be inadequate to reconcile the ^{14}C and luminescence chronologies. The only remaining possibility would appear to be contamination of the deposit by *in situ* disintegration of the local Devonian sandstone: as few as 5% contaminant grains would bring the discordant TL ages into alignment with the calibrated

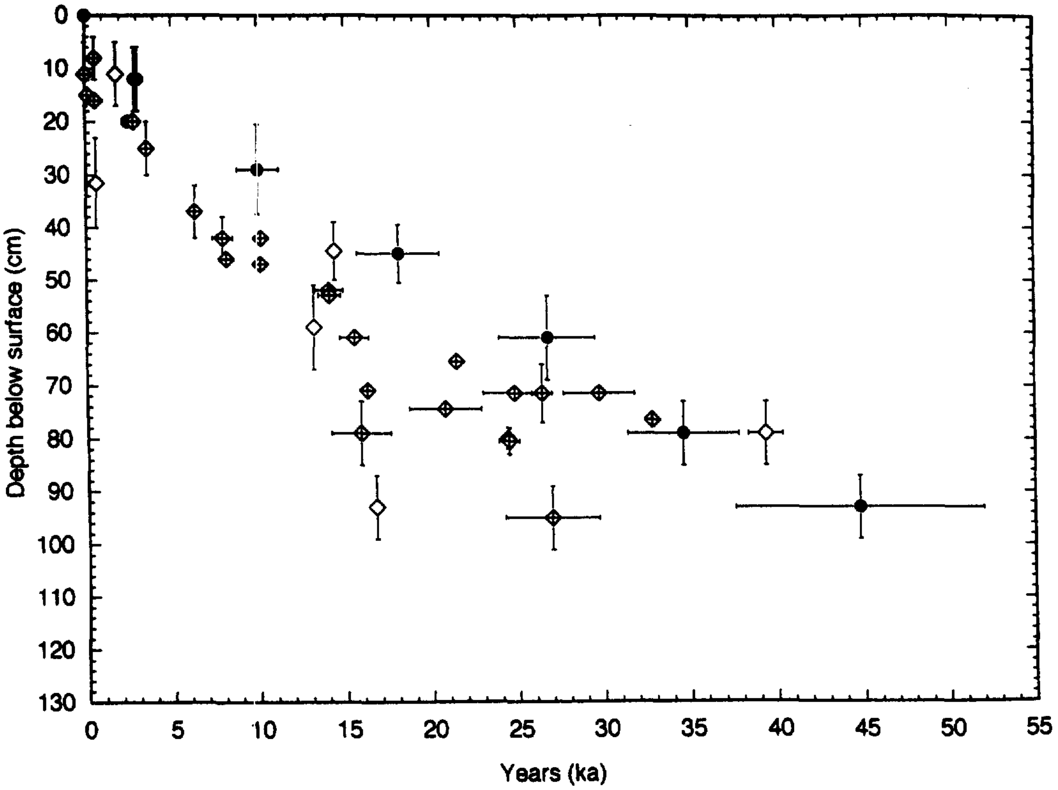

Fig. 13. Age-versus-depth plot of calibrated ^{14}C determinations and "selective bleach" TL ages from Puritjarra rock shelter. TL ages are shown by solid circles, AMS ^{14}C ages by open diamonds, and radiometric ^{14}C ages by crossed diamonds. Ages are in thousands of years. The depth range is indicated for each sample, together with its 2σ age uncertainty (from Smith *et al.*, 1997).

^{14}C ages (Smith *et al.*, 1997), and the use of single-aliquot OSL methods has been implemented to identify such material (M. Smith, Personal Communication, 1996).

4.2.12. *Terrace teasers.* Along the humid-temperate fringe of south-eastern Australia, human arrival as early as 40,000–47,000 years has been claimed by Nanson *et al.* (1987), on the basis of TL and ^{14}C dates for the Cranebrook Terrace river gravels from which stone tools had been collected a decade earlier (Stockton and Holland, 1974). The *in situ* status of the artefacts has been questioned, however, and the acceptance of these dates has been postponed until controlled excavations can locate artefacts in primary stratigraphic position (Allen, 1989; Jones, 1989). The age of the river terrace was constrained by four fine-grain TL dates of *circa* 41,000–47,000 years, one sample giving the same age by "partial bleach" and "total bleach" methods (Nanson and Young, 1987). These ages are consistent with the seven oldest finite ^{14}C determinations on wood and charcoal (*circa* 37,000–42,000 years BP), although contamination of the submerged logs by younger carbon in the groundwater can be inferred from the five ages of *circa* 27,000–34,000 years BP and the obtainment of older ages with more rigorous pretreatment of selected samples (Nanson and Young, 1987).

Human arrival by 45,000 years ago has also been suggested from the degree of pedogenesis of the artefact-bearing "D Clay" at Keilor, near Melbourne (Bowler, 1976). Optical dating of these river terrace sediments is planned (J. Allen, Personal Communication, 1995) to provide a further test of the present picture of continental colonisation—that northern Australia was settled 10,000–20,000 years earlier than in the south. Across the Sahul Shelf in Melanesia, a region possibly traversed by the first Australians, early occupation sites have also been investigated by luminescence methods. These studies are discussed in the next section, together with those pertaining to the timing of settlement of the remote Polynesian islands in the Pacific Ocean, some of the last places on the planet to be colonised by people.

4.3. *Melanesia and Polynesia*

Melanesia can be broadly defined to encompass those islands running eastward from Irian Jaya/Papua New Guinea to Fiji, while Polynesia includes those islands falling within the triangle created by Hawai'i, Easter Island and New Zealand. Irian Jaya/Papua New Guinea and Australia formed a single landmass during glacial periods of lowered sea-level, but the human colonisation of islands further east required open-sea voyages of several tens to hundreds of kilometres. First landfall in Australia may also have been made by accomplished seafarers, whose deliberate ocean crossings might have been prompted by the sighting of natural bushfires, ignited by lightning strikes, which can create smoke plumes that are visible at sea-level from distances of up to 260 km (Dortch and Muir, 1980). Crossings to the eastern end of the Solomon Islands can be made with at least one island always in sight. Once east of this island chain, however, the sea gaps extend to more than 300 km, making this truly the realm of the ancient mariners.

4.3.1. *The first Melanesians.* The oldest dates for initial occupation of Melanesia are those from the Huon Peninsula, on the north-east coast of Papua New Guinea (Fig. 8). A number of stone tools, including "waisted axes" that may have been hafted, were found in volcanic tephras deposited on a raised coral reef terrace (Groube *et al.*, 1986). This terrace (labelled IIIa) was dated by alpha-spectrometric U-series methods to *circa* 45,000–53,000 years (Chappell, 1974), but this timespan has since been revised to *circa* 52,000–61,000 years, on the basis of mass-spectrometric and new alpha-spectrometric U-series determinations (Chappell *et al.*, 1996b). TL dating was also undertaken, using silt-sized quartz grains extracted from the set of three heavily weathered tephras. Ages of *circa* 42,000–60,000 years were derived from the palaeodoses (350–475°C plateau region) and the measured radionuclide concentrations in completely dry sediment (Groube *et al.*, 1986). The most important artefacts, including waisted axes, were recovered from the two lowest tephras, which both gave TL ages of ~60,000 years. As typically observed for quartz, no anomalous fading of the TL signal was observed over a period of one month, but unusually for quartz, and especially for fine-silt grains, the dose-response curves were reported as being linear, even though palaeodoses of ~100 Gy were obtained. Perhaps feldspar inclusions were responsible for the TL signal; this possibility will be examined directly by optical dating of the sediments collected from this site by the author (Roberts *et al.*, 1994b).

Time-dependent variations in the dose rate gave much greater cause for uncertainty in these TL age estimates. The ingrowth of ^{230}Th in the neighbouring coral reef would give rise to a measured U-series dose rate that is higher than the long-term average (and, thus, yield TL ages that are too young), but the measured potassium concentrations posed a more serious problem. These were an order-of-magnitude lower than found in other tephras in the region, which Groube *et al.* (1986) attributed to leaching during strong weathering of the tephra. Using the higher potassium concentration reduced the TL ages of the lowest two tephras to ~37,000 years, but these represent *minimum* ages because they are calculated for zero water content in the tephra. Given the wet conditions typical of tropical Melanesia, Groube *et al.* (1986) concluded that the tephras are at least 40,000 years old (but did not quote the water-adjusted ages), consistent with the U-series dates for the underlying coral reef. For a water content of 25%, the minimum ages of the basal tephras are ~47,000 years (using the higher K values, and assuming an alpha-efficiency "*a*" value of 0.1 and equal activities of uranium and thorium), and these ages increase to ~76,000 years using the measured K concentrations. A *maximum* age of 61,400 ± 600 years for the artefact-bearing tephras is indicated, however, by the latest mass-spectrometric U-series date for the supporting reef (Chappell *et al.*, 1996b). The probable age of the waisted axes from the lowest pair of tephra beds is, therefore, *circa* 47,000–61,000 years, a time period remarkably similar to that for first human occupation of western Arnhem Land (Roberts *et al.*, 1990a, 1994a).

Other sites of comparable antiquity have not been discovered in Melanesia, the next most ancient archaeological sites being dated by ^{14}C to no more than 36,000 years BP. One such site (Lachitu) lies on the coast 800 km north-west of the Huon Peninsula, but the others are found on the islands of New Britain and New Ireland, which constitute the Bismarck Archipelago (Gosden, 1995). These islands face the Huon Peninsula but they were never joined by a land bridge during the Pleistocene, a sea crossing of less than 50 km being needed to make landfall on New Britain. Here, in the highland rainforests at Yombon, chert artefacts have been found sealed beneath tephra layers (Pavlides and Gosden, 1994). Lumps of charcoal associated with the lowest artefacts have given three ^{14}C ages of *circa* 32,600–35,600 years BP, and this clay layer is unconformably overlain by a series of Holocene tephras. Calibration of these dates gives an age for artefact deposition of perhaps 38,000–42,000 years, although contamination of the samples cannot be dismissed given the proximity of the dates to the ^{14}C "glass ceiling" and their procurement from charcoal in a region with an annual rainfall of more than 6 m.

A short distance to the east of New Britain lies New Ireland, where the basal cultural deposits at the cave sites of Buang Merabak and Matenkupkum (Fig. 8) have produced ^{14}C dates of ~32,000 and *circa* 31,000–33,000 years BP, respectively (Allen, 1991; Allen *et al.*, 1988; Gosden, 1995). The latter dates were obtained on marine shells from the dense shell midden which forms the earliest occupation layer at Matenkupkum; the *dated* event, in this instance, coincides with the *target* event. These ^{14}C ages may also be subject to contamination, as suggested by the increase in age of ~3000 years when the same shell species was prepared in a nitrogen environment to minimise uptake

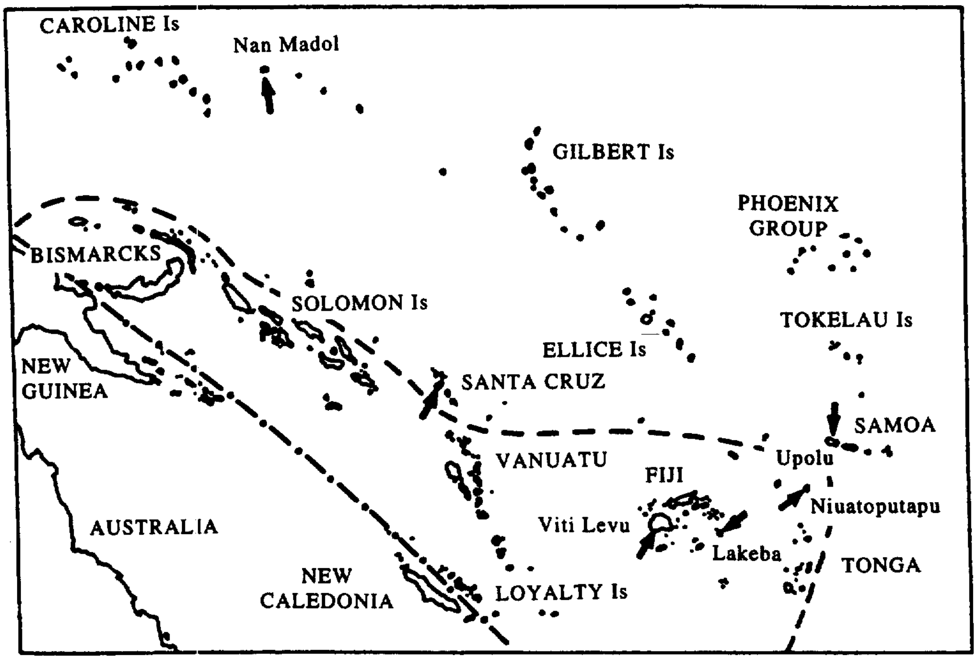

Fig. 14. Location map of the south-western Pacific Ocean, with arrows showing sites from which pottery samples have been obtained for TL dating. The dash–dot line marks the approximate eastern boundary of continental rocks, and the dashed line indicates the "andesite line". Islands to the south and west of this line are characterised by andesitic rocks of relatively low uranium and thorium content, while islands to its north and east are based on basaltic oceanic volcanoes or coral built on volcanic basement (from Prescott *et al.*, 1982).

of modern atmospheric carbon (Allen, 1994). To check for ^{14}C age underestimation at the New Ireland and New Britain sites, optical dating of sediments is foreshadowed (J. Allen, Personal Communication, 1995).

4.3.2. *The peopling of Polynesia.* Other types of artefacts have been examined by luminescence methods, an early attempt being made to obtain TL dates from the distinctive decorated pottery associated with the "Lapita" culture (Prescott, 1982; Prescott *et al.*, 1982; see Fig. 14). There is fierce disagreement among archaeologists, linguists and geneticists about the place of origin of the Lapita culture (be it south-east Asia or the Bismarck Archipelago), the rate of spread of Lapita people across Melanesia and into Polynesia, and their ancestral relationship to the Polynesians (e.g. Allen, 1991; Bellwood, 1991; Diamond, 1988; Gibbons, 1994a; Gosden *et al.*, 1989; Terrell, 1989). Lapita pottery has been found as far east as Fiji and the western Polynesian islands of Samoa and Tonga, and some of these sites have been dated by ^{14}C to as early as 3500 years BP.

TL analysis of Lapita pottery was recognised as a means of dating the target archaeological event, rather than relying on association-based ^{14}C chronologies (Prescott, 1982; Prescott *et al.*, 1982). Of the pot sherds examined, however, most were composed of volcanic minerals ill-suited to TL dating, and only a few sherds yielded fine-grain potassium feldspars and coarse-grain quartz. The latter category includes pottery from Viti Levu, the main island of the Fiji group, which has a core of relict continental rock. Some of these sherds proved difficult to date because of the coralline setting, which results in a low environmental dose rate (and the relatively larger contribution from cosmic rays), weak TL emissions (owing to the small accumulated doses), and the prevalence of spurious TL from $CaCO_3$. Dates were, nonetheless, obtained for several sherds from two Fiji sites (Naqarai and Yanuca). TL dating of one sherd confirmed its age based on ceramic type, both estimates being significantly younger than the *calibrated* ^{14}C determinations on charcoal. The latter were made at the Gakushuin (GaK) Laboratory, whose dates for Pacific and Asian samples are frequently anomalous when compared with ^{14}C dates from other laboratories (Spriggs and Anderson, 1993). Two other Lapita sites on Viti Levu (Sigatoka and Natunuku) were sampled recently by the author and A. Anderson, to attempt optical dating of the beach-dune deposits that have previously yielded ^{14}C dates suggestive of early colonisation (Davidson *et al.*, 1990). Low environmental dose rates (as shown by *in situ* gamma spectrometry) may again be problematic, but quartz OSL emissions should be much brighter than the "thermally quenched" 325°C TL signals (Aitken, 1994) and the IRSL emissions from fine-grain preparations contaminated by $CaCO_3$ can be normalised successfully (Li and Wintle, 1994).

Obsidian is a glass of volcanic origin that was widely traded in Melanesia and Polynesia, and by prehistoric people in other regions of the world (see Renfrew and Bahn, 1991). Although the timing of the volcanic event which formed the obsidian may bear no relation to that of artefact manufacture, TL has been used to supplement the many chemical methods of source identification. Pieces of obsidian from a single source typically exhibit small variations in their natural and laboratory-dosed TL signals, but between-source variations are sufficient to permit characterisation of obsidian quarries in New Zealand, North and Central America, the Mediterranean and Kenya (Carriveau and Nievens, 1979; Göksu and Türetken, 1979; Huntley and Bailey, 1978; Leach and Fankhauser, 1978; Rendell *et al.*, 1985). TL has yet to be used to characterise obsidian in Melanesia, but given the stratigraphic sequences of ashfall layers and soils that occur throughout this region, a detailed chronology could be obtained by TL dating of the heated glass shards (e.g. Berger, 1991; Berger, 1992) or quartz grains (e.g. Liritzis *et al.*, 1996), and by optical dating of the unheated sediments. Some West New Britain sites have ^{14}C age control for several of the Holocene tephras (Machida *et al.*, 1996; Pavlides, 1993). Direct dating of the volcanic eruptions remains a priority, however, to establish their impact on human activities in this important source area for chert and obsidian, and possible Lapita "homeland" (Gosden *et al.*, 1989). To this end, luminescence samples were collected recently from one such site (Namundo), where Lapita pottery and obsidian tools occur between tephras which are believed to be older than 2000 years BP (R. Torrence, Personal Communication, 1997).

4.3.3. *New Zealand.* Luminescence methods have also been largely overlooked in attempts to date palaeoenvironmental evidence for human colonisation of the remote Melanesian and Polynesian islands. Anthropogenic disturbance of island ecosystems may be inferred from vegetation changes documented by pollen and charcoal records (e.g. Kirch and Ellison, 1994; but see Anderson, 1994), animal extinctions after human landfall (e.g. Diamond, 1991; Steadman, 1995), and the introduction of non-domesticated animals transported by humans (e.g. Flannery *et al.*, 1988). Fossil remains of the commensal Pacific rat (*Rattus exulans*) have been found throughout the region, with AMS ^{14}C dates of up to ~2000 years BP being reported recently for rat remains in New Zealand (R. Holdaway, 1996). These bone-gelatin dates are much older than the first unequivocal archaeological evidence for human colonisation at ~850 years BP (Anderson, 1991), and are considered to reflect the introduction of rats by transient human visitors more than 1000 years before the permanent Polynesian settlement of New Zealand (R. Holdaway, 1996). The validity of these early ^{14}C dates has been challenged by Anderson (1996), who argues instead for rat-bone contamination. To address the chronological uncertainty, further AMS ^{14}C analyses are being done and, in collaboration with R. Holdaway, sediment samples have been collected for optical dating from the key rat-bone sites.

A resolution to this matter is vital not only for Pacific prehistory, but also for models of island zoogeography as, on the current evidence, the rat-induced extinctions of small native animals should have preceded the elimination of larger fauna by human predation and habitat destruction (R. Holdaway, 1996). Luminescence methods should be used more often to construct Quaternary chronologies for palaeontological sequences, given their frequent employment as proxy archaeological data (e.g. Diamond, 1991). Optical dating methods also deserve more widespread application to hitherto disregarded areas of archaeometric research, such as the dating of prehistoric art. To my knowledge, only a few luminescence studies have contributed to the latter theme—studies in which, as discussed in the following and penultimate section of this review, the dating methods used are fit to be acclaimed "state of the art".

5. PAINTINGS AND PETROGLYPHS

One of the most striking features that distinguishes modern humans from other hominids and the rest of the animal kingdom is the ability to transform thoughts into pictures (Valladas *et al.*, 1992). These pictures may be produced by an *additive* process, such as painting, drawing, or the application of a residue such as beeswax, or they may be created by a *reductive* process, such as engraving, pounding or pecking (Bednarik, 1993). The latter group of pictures are termed "petroglyphs". Until the advent of numerical dating methods, rock art and other rock markings could be placed only in a relative age sequence, based on criteria such as style, content (e.g. extant or extinct animals), order of superposition, and degree of weathering. Much effort has, therefore, been made to directly date rock pictures, and luminescence dating is being used increasingly to assist in this challenging endeavour.

5.1. *Petroglyphs*

The direct dating of petroglyphs has been especially intractable, because the reductive process involved in their manufacture leaves behind no substance to signify "time zero" (Bednarik, 1993). Markings are generally too shallow to distinguish differential production rates of cosmogenic radionuclides (e.g. ^{36}Cl, ^{10}Be, ^{26}Al) in the deepest part of the petroglyph and on the adjacent rock surface (J. Stone, Personal Communication, 1995). Geological micro-erosion methods have been used for some engravings (Bednarik, 1992, 1995b) and, for petroglyphs covered by mineral accretions, minimum ages have been reported using cation-ratio and AMS ^{14}C methods on the inorganic and organic components, respectively (e.g. Dorn *et al.*, 1988, 1989; Nobbs and Dorn, 1993; Watchman, 1993a,b). Individual quartz or feldspar grains trapped in these precipitate "skins" may be amenable to optical dating (Bednarik, 1996), provided that modern sunlight does not pentrate far through the opaque rock "varnish".

Luminescence methods have recently contributed, albeit indirectly, to the debate over the antiquity of the Côa petroglyphs in north-eastern Portugal. On stylistic grounds, these pecked figures are assigned an Upper Palaeolithic age by some archaeologists (e.g. Zilhão, 1995), whereas micro-erosion studies of the petroglyphs and AMS ^{14}C dating of organic inclusions in the overlying silica accretions suggest an age of less than ~3000 years (Bednarik, 1995a,b; Watchman, 1995). A late Holocene age is supported by preliminary IRSL dates of ~1000 years and *circa* 4000–6000 years for the fluvial terrace sediments that underlie the site (M. Lamothe in Watchman, 1995). These IRSL ages were obtained using single-grain methods for feldspars (Lamothe *et al.*, 1994), which revealed a population of uniformly well bleached, young grains (M. Lamothe, Personal Communication, 1995). Moreover, if some of the grains had been poorly bleached at deposition then their apparent ages would have been too *old*, rather than too young.

These late Holocene age determinations have, however, proven highly controversial (Fischman, 1995; Zilhão, 1995). A second set of similarly recent AMS ^{14}C ages has been explained in terms of sample contamination by younger carbon (Dorn, 1997a; see also Dorn, 1997b), and it now appears that the rock panels bearing the petroglyphs were available for inscription during the Upper Palaeolithic, as shown by the late Pleistocene "exposure ages" determined using ^{36}Cl (Phillips *et al.*, 1997). A moderately stable landscape is also suggested by the latter study, although the frequent floods that sweep through the Côa gorge might account for the recent deposition of the inset river terraces. Direct dating of the Côa petroglyphs seems, therefore, the only way to resolve this chronological dispute—perhaps optical dating of the overlying silica skins should be attempted, using the "micro-stratigraphic" approach of Richards (1994).

Optical dating methods have been exploited recently to *directly* date an unusual type of petroglyph: the White Horse chalk figure at Uffington, near Oxford (Rees-Jones and Tite, 1997a,b). This elegant figure was made on a hillside by cutting a

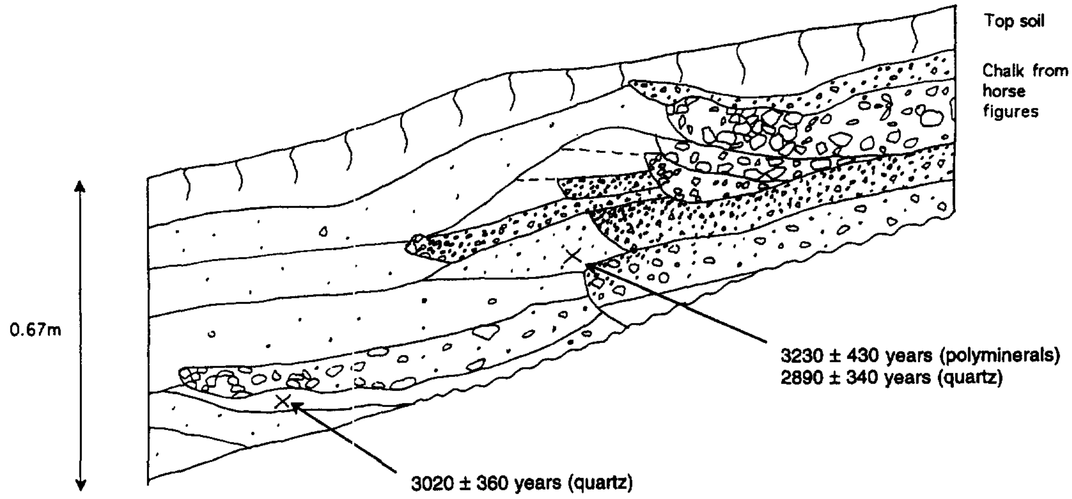

Fig. 15. Stratigraphic section at the belly of the Uffington White Horse, showing the sample locations and the dates obtained from fine-grain quartz (OSL) and polymineral (IRSL) extracts (after Rees-Jones and Tite, 1997b).

series of ditches and packing them with chalk. It is the only prehistoric equine hill-figure in Britain and, on stylistic evidence, was thought to be either Celtic (late Iron Age) or Saxon in age. OSL and IRSL dates were obtained from fine-grain colluvial sediments that had washed onto the figure and been subsequently buried by chalk added to the figure during repairs (Fig. 15). Several aspects of this novel dating problem were of concern to the geochronologists: adequate bleaching of the sediments at deposition; the magnitude of the palaeodose correction needed for such young samples; the difficulty in measuring accurately the dose rate in a low-activity material such as chalk; and the uncertainty surrounding the water content.

Reassurance that the sediments were well bleached at deposition is given by the close correspondence between the IRSL date (3230 ± 430 years) and the two OSL dates (2890 ± 340 and 3020 ± 360 years), as quartz and feldspar bleach at different rates (Godfrey-Smith *et al.*, 1988). All three ages are, however, very sensitive to small variations in the annual dose: this is less than 0.5 mGy, of which the cosmic-ray dose rate must contribute a sizeable (but unreported) proportion. The water content is too low to introduce any significant bias, while any inhomogenity of the gamma dose rate in this shallow (< 70 cm thick) deposit and any disequilibrium due to leaching of uranium (as observed in other carbonate-rich deposits, e.g. Olley *et al.*, 1997a) should be adequately accommodated by the use of *in situ* gamma spectrometry. The dates of ~3000 years place the construction of the White Horse in the Bronze Age or early Iron Age, older than originally anticipated by the archaeologists but coincident with other evidence for intense Bronze Age activity in the region.

5.2. *Paintings*

These innovative applications of optical dating to petroglyphs have only recently been matched by optical dating of prehistoric paintings. Accelerator ^{14}C dating has been used for pictures that contain organic materials such as charcoal (Clottes *et al.*, 1995; David, 1992; McDonald *et al.*, 1990; Valladas *et al.*, 1992), blood proteins (Loy *et al.*, 1990), plant fibres and other organic binders (Russ *et al.*, 1990; Watchman and Cole, 1993), and beeswax (Nelson *et al.*, 1995). AMS ^{14}C methods have also been applied to the organic fraction of mineral accretions formed on top of rock paintings in northern Australia (Morwood *et al.*, 1994; Watchman, 1993a,b; Watchman *et al.*, 1997). To date the *target* (archaeological) event by ^{14}C requires that the carbon "clock" starts to tick at the same time as the picture is created, a condition which may not be met if ancient charcoal is used for drawing (Bednarik, 1996) or if the source of the organic matter is ambiguous (e.g. Loy, 1994; Nelson, 1993) or subject to contamination by younger carbon (e.g. discrepant ^{14}C dates for the same picture; McDonald *et al.*, 1990).

Optical dating could, in principle, be used to determine the age of rock paintings at sites exposed to indirect or direct sunlight. The surface grains on the rock shelter would be effectively "zero-age" immediately prior to painting, but a thick application of paint or charcoal might be sufficient to seal a few grains from further sunlight exposure. These grains would then accumulate a radiation dose, which could be determined using single-grain optical dating methods. Even pictures in *caves* may be amenable to optical dating, because torches were used to illuminate caves for painting and drawing (e.g. Clottes *et al.*, 1995). Exposure to the light from a naked flame held close to the rock surface immediately before the application of paint or charcoal might be sufficient to reset the luminescence "clock" of the surface grains; this proposition is easily tested experimentally. Optical dating holds the advantage over ^{14}C methods in these situations, as the *target* event (the date of application of paint) is contemporaneous with the optically dated event (the time since the painted grains were last exposed to light). In practice, a "micro-stratigraphic" strategy (e.g. Liritzis, 1994; Richards, 1994) would need to be adopted, to demonstrate that the sun-lit (or torch-lit) grains had been reset initially and not bleached subsequently by light transmitted through the paint.

Optical dating may also be suitable for quartz and feldspar grains embedded in opaque mineral skins, and other accretions, which overlie and underlie paintings in rock shelters: minimum and maximum ages, respectively, are thereby obtained for the target event. The fossilised remains of insect nests represent one such type of "accretion". Northern Australia has some of the most spectacular, and possibly among the earliest, rock art in the world (e.g. Chaloupka, 1993; Flood, 1997; Walsh, 1988; Watchman, 1993b). The nests of mud-wasps are often built on top of these rock paintings

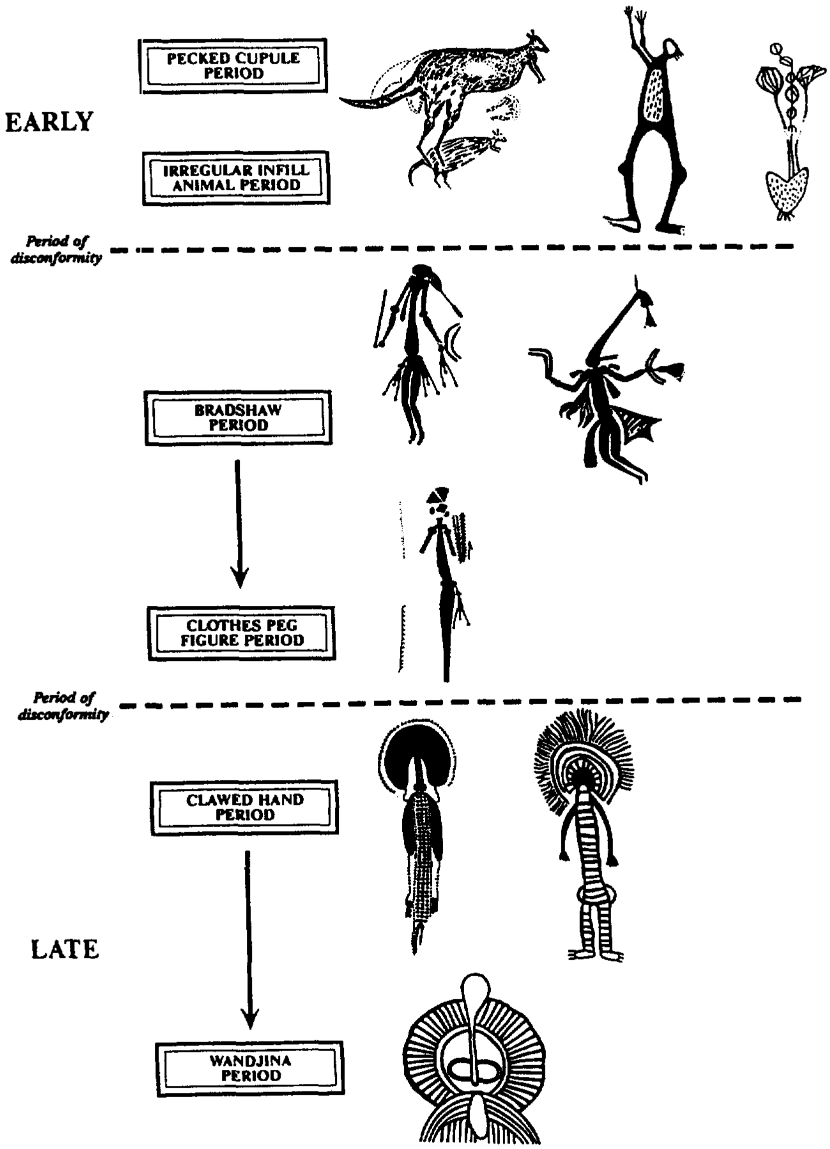

Fig. 16. Simplified version of the Kimberley rock art sequence proposed by Walsh (1994). Note the hiatus anticipated between the "Wandjina" and "Bradshaw" period motifs, and that predicted between the "Bradshaw" and "Irregular Infill Animal Period" paintings (after Walsh, 1994, and Morwood *et al.*, 1994).

(Naumann, 1983; Waterhouse, 1991), while the "stumps" of some nests have been painted over. By dating the remnants of nests above and below a painting, its age can be bracketed. This approach has recently been taken by Roberts *et al.* (1997), who extracted sand-sized quartz grains from mud-wasp nests in the Kimberley region of northern Western Australia. These muddy sediments would have been collected by wasps from the banks of streams or pools, and then carried to the rain-protected ceilings and recesses of rock shelters where the nests are constructed. Final solar exposure of the quartz grains thus occurs when the mud nest is built, an archaeologically "instantaneous" event.

To permit optical dating of the grains concealed within the body of a nest, centimetre to decimetre sized nests were removed at night using only red (> 590 nm) light (Roberts *et al.*, 1997). Five of the nests examined were built over paintings, whose relative ages could be estimated from a superimposition sequence of motif styles (Morwood *et al.*, 1994; Walsh, 1994; see Fig. 16). Three lightly-cemented nests were constructed on parts of "Wandjina" figures (which are believed to be Holocene in age) and two petrified nests were built over the head-dress of a human figure, possibly related to the more archaic "Bradshaw" style. Two other nests, not clearly associated with any paintings, were also collected: a heavily cemented nest in the "Wandjina" rock shelter and a modern nest (< 2 years old), the latter providing a check that the quartz grains had been bleached completely when the nest was made.

Initial tests were conducted on the depth of penetration of modern sunlight into one of the "Wandjina" nests. The largest nest collected was divided into seven portions, the first being composed of the outer few millimetres of nest mud and successive portions consisting of mud hidden deeper within the nest; the "core" portion had been attached to the shelter wall. The palaeodose of each portion was determined using conventional multiple-aliquot additive-dose OSL procedures, and concordant palaeodoses were obtained from two portions examined using a single-aliquot additive-dose protocol (Murray *et al.*, 1997). The outer layer yielded a palaeodose of only 0.14 ± 0.02 Gy (corresponding to an age of ~110 years), the next four portions had palaeodoses of 0.35–0.43 Gy (giving a weighted mean age of 270 ± 20 years), and the palaeodoses for the innermost two portions jumped to ~0.85 Gy (equivalent to 610 ± 40 years). These "micro-stratigraphic" data are interpreted as showing that modern sunlight penetrated only as far as the outer layer, and that two "generations" of nest are preserved: a ~610 year-old basal portion and a ~270 year-old central portion. This finding is consistent with the observation that mud-wasps prefer to build nests near existing nests and on the stumps of abandoned nests, rather than on a bare rock surface (Naumann, 1983; Waterhouse, 1991). Although some grains will have been exposed to sunlight for ~340 years on the stump of the earlier nest, most will have been derived from the unbleached interior of these remnant portions. A more enigmatic result is the non-zero palaeodose obtained for the outer portion, and for the core of the modern nest (which gave a palaeodose of 0.15 ± 0.02 Gy). Further investigations should reveal whether this represents an artefact of thermal transfer, the inclusion of a small proportion of incompletely bleached grains, or another cause.

The two nests associated with the possible "Bradshaw" figure were dated using only the single-aliquot protocol, owing to the paucity of grains for comparative multiple-aliquot analysis. The core of the larger nest yielded a palaeodose of ~33 Gy, for which the above non-zero "residual" OSL palaeodose is a trivial fraction. The optical date of 17,500 ± 1800 years provides a *minimum* Last Glacial Maximum age for the underlying painting, making it the world's oldest dated painting of a human figure. The second nest associated with this painting demanded a novel optical dating approach, because of its small size. Quartz grains were extracted by disaggregation of the whole nest and, as there were too few grains for multiple-grain analysis, individual grains were dated using additive-dose and regenerative-dose single-aliquot protocols (Murray and Roberts, 1997; Roberts *et al.*, 1997). Most grains gave palaeodoses of 14–59 Gy, forming broad, approximately Gaussian, frequency distributions. The spread in the distributions probably arises from grain-to-grain differences in dose-response behaviour and beta microdosimetry, as reported for other analyses of individual quartz grains (Murray and Roberts, 1997; J. Olley, Personal Communication, 1997). A few grains had weak natural OSL signals, presumably because they are insensitive to radiation or are derived from the sun-bleached surface of the nest. These "dim" grains were further analysed using only the regenerative-dose protocol, in which the total light sum is measured. Palaeodoses in the range 0.5–8 Gy were obtained. Such values are intermediate between those expected from grains exposed to modern sunlight (~0.15 Gy) and the > 14 Gy palaeodoses obtained from the majority (75%) of the single grains examined. These grains are thought to have originated from the "twilight zone" of the nest, where the growth in palaeodose is offset by partial bleaching from silica-filtered sunlight. The equilibrium conditions existing in this twilight zone remain an area for future investigation. Grains with palaeodoses of > 14 Gy yielded regenerative-dose and additive-dose optical dates of ~16,400 years (average of 15 grains) and ~23,800 years (average of 21 grains), respectively (Roberts *et al.*, 1997). The latter determination is influenced by the inclusion of three grains that contain about twice the average palaeodose; their omission would reduce the mean age to ~21,200 years. Despite these uncertainties, the single-grain OSL dates confirm the Pleistocene antiquity of the underlying painting and demonstrate the utility of single-grain analyses in situations (e.g. the dating of painted nest stumps) where sample material is too sparse for multiple-grain methods.

A Pleistocene age for this painting, and a young age for two other nests, is independently indicated by the radionuclide data (Roberts *et al.*, 1997). Nests built during the last 500–600 years exhibit a 40–110% excess of ^{226}Ra over its parent ^{230}Th, whereas secular equilibrium prevails in the Pleistocene nest mud. If the latter had been deposited with a similar initial excess of "unsupported" ^{226}Ra, then equilibrium would have been attained after a period of 8000 years (5 half-lives of ^{226}Ra). This represents a *minimum* age for the nest mud, and hence for the underlying painting, as secular equilibrium could have been maintained thereafter for an indefinite period. At the other end of the age spectrum, the optical dates of < 200 years for two nests are supported by the presence of a 160–270% excess of ^{210}Pb on the sediments: this excess is probably due to atmospheric fallout and indicates that the nests are built from mud collected within the last 100 years (5 half-lives of ^{210}Pb). AMS ^{14}C determinations on the pollen grains extracted from these nests also give dates that are indistinguishable from modern (Roberts *et al.*, 1997).

As with the White Horse study, the mud-wasp nests presented a challenge to the accurate determination of the dose rate. The gamma-ray contribution from the wall of the rock shelter, and the dose due to cosmic rays, was estimated by *in situ* and high-resolution gamma spectrometry, and from calcium fluoride TL dosimetry capsules placed on-site for one year. The beta dose rate, almost all of which is derived from the nest mud, was deduced from XRF and alpha spectrometry, while U and Th concentrations in the acid-etched quartz grains were measured by laser-ablation ICP-MS. Disequilibria in the ^{238}U decay series included radon gas escape of ~20% in a recent nest, and the aforementioned "unsupported" excesses of ^{226}Ra and ^{210}Pb. Potassium concentrations exhibited the greatest variation from nest to nest, however, ranging from 20 Bq kg^{-1} (in the modern nest) to 500 Bq kg^{-1} (in the Pleistocene nest mud). Given these findings and the need for single-aliquot or single-grain methods of palaeodose determination, optical dating of mud-wasp nests and sediment accretions created by other animals (e.g. ants, termites, mud-nesting birds) is never likely to be straightforward.

Many mud-wasp nests also contain fossil pollen, spores and phytoliths (Roberts *et al.*, 1997). These not only provide palaeovegetation information for the "snapshot" of time represented by a nest, in a similar manner to the palaeoecological data obtained from stick-nest rat middens (Pearson and Dodson, 1993), but they also are amenable to AMS ^{14}C dating (Brown *et al.*, 1989; Kelly *et al.*, 1991), thereby providing a possible cross-check on the optical dates. Roberts *et al.* (1997) report ^{14}C/OSL age comparisons for recent mud-wasp nests, but the Last Glacial Maximum nests, which are of most interest to archaeologists, yielded an insufficient mass of pollen grains and phytoliths for AMS ^{14}C dating (although enough could be extracted for palaeoenvironmental work). The potential for luminescence dating of phytoliths certainly warrants closer attention.

5.3. *Phytoliths*

Rowlett and Pearsall (1993) made a pioneering, and seemingly successful, attempt to date burnt and unburnt phytoliths using TL. Phytoliths are composed of microcrystals of biogenic silica (but are often described as being amorphous in structure) and thus, Rowlett and Pearsall (1993) surmised, they could be as amenable to luminescence dating as quartz and chert. Given their small size (< 200 μm diameter) and translucence, I can envisage *three* mechanisms by which the luminescence signal could be reset: by secretion of the phytolith in the plant, by exposure to sunlight after plant decomposition (and during any later episodes of insolation), and by heating in anthropogenic or natural fires. Burnt phytoliths from two archaeological sites in coastal Ecuador were investigated using a regenerative-dose procedure and ~0.8 g aliquots. The procedural details are sketchy, however, and no TL growth curves or palaeodose plateaux are given. Nonetheless, the phytoliths appear to luminesce and respond to applied radiation. TL dates of *circa* 1000–3000 years BC were derived from the 350–380°C glow curve region, which agreed with the ages assigned to the associated ceramics and ^{14}C samples. TL signal integration over the 200–380°C region gave rise to similar results, although the TL in the 200–275°C region appears to be thermally unstable (Rowlett and Pearsall, 1993).

Intriguingly, slightly *younger* ages were obtained from the unburnt phytoliths (Rowlett and Pearsall, 1993), which raises the prospect that a light-sensitive signal may be present and this was partially bleached during laboratory pretreatment (no special illumination precautions having been taken). Phytoliths may thus exhibit luminescence signals upon thermal *and* optical stimulation. The electron traps involved in luminescence production need to be investigated, but perhaps, in the new millennium, optical dating of individual phytoliths will be a complementary technique to optical dating of single grains of quartz and feldspar. Even in the absence of a light-sensitive signal, TL dating could be usefully applied to unburnt phytoliths whose TL "clock" starts when the silica microcrystals are formed; TL analysis may be feasible when AMS ^{14}C dating is not, because the latter exploits only the tiny amounts of occluded carbon. Using mud-wasp nests as an example, the phytoliths concealed in the mud are likely to be contemporaneous with the time of nest construction (AMS ^{14}C dating shows this is true for pollen grains deposited in modern nests; Roberts *et al.*, 1997). The palaeodose acquired by the phytoliths since crystallisation could thus provide a check on the solar-reset OSL palaeodoses in the quartz grains, but their common dose rate precludes an independent age comparison.

5.4. *The dating palette*

The above discussion has focussed on experimental applications of luminescence dating to rock art. The expression "necessity is the mother of invention" can be justly applied to these studies, each of which required a novel approach. Optical dating of fine-grain quartz from the White Horse, and single-grain dating in the Côa valley and northern Australia, are not without their problems, and the dose rate determinations for the mud-wasp nests proved unusually troublesome. But further testing of these methods, and continued creative applications, should elevate luminescence dating to a more prominent position in the field of rock art dating. New possibilities, such as luminescence dating of phytoliths and optical dating of individual grains hidden beneath mineral accretions or paint, could be investigated. The time of emplacement of prehistoric stone arrangements can also be established using the "micro-stratigraphic" luminescence approach of Liritzis (1994), who determined the date of construction of a Mycenean wall (see Section 4.2.5); in these situations, the surface grains on the undersides of the stones are last exposed to sunlight when the stones are emplaced.

The contributions made to rock art dating by traditional multiple-aliquot luminescence methods should not, however, be overlooked. Considerable controversy has been generated from the *indirect* dating of petroglyphs and ochres in northern Australia (Bahn, 1996; Holden, 1996; Morell, 1995). Lumps of red and yellow ochre and abraded haematite were found at Malakunanja II and Nauwalabila I in quartz sediments dated to *circa* 50,000–60,000 years using TL and OSL, respectively (see Section 4.2.5). Whether these pigments were used for rock art or body decoration is unknown, but their presence in the initial occupation levels implies that rock paintings may have been made in western Arnhem Land more than 20,000 years before the earliest dated paintings in Europe (Clottes *et al.*, 1995). It is doubtful, however, that paintings could remain visible after many tens of millennia in an open rock shelter, given the natural processes of weathering of the paint and rock substrate, and the formation of mineral accretions. Taphonomic problems are equally likely to hinder the long-term survival of cave paintings (Clottes *et al.*, 1995).

Petroglyphs are a more durable type of rock art. Circular rock engravings (pecked cupules) are widespread across northern Australia and they are considered to predate paintings in the Kimberley region (Morwood *et al.*, 1994; Walsh, 1994; see Fig. 16). In the sandy deposits at Jinmium rock shelter, a fallen fragment of sandstone marked with pecked cupules was buried by sediments TL dated to *circa* 58,000–75,000 years (Fullagar *et al.*, 1996); 58,000 years represents a minimum age for the cupules, which could have been made many millennia before the fragment fell from the shelter wall. The lowest pecked cupules engraved on the wall also occurred between these chrono-stratigraphic levels, while pieces of ochre were recovered from the underlying deposits, dated to more than ~75,000 years. But the TL ages would appear to extend the timeframe for rock art in Australia, almost doubling its duration if the maximum age for the pigments is accepted. But the TL ages for the Jinmium deposits, and the antiquity of the rock art contained therein, are likely to be gross overestimates for the reasons I have described at length in Section 4.2.7.

Appropriate luminescence methods are, nonetheless, a valuable tool in the arsenal of the rock art chronologist. They can be used to good effect to constrain the ages of pieces of pigment, painting or petroglyph that may have fallen into an archaeological deposit. Not only can optical dating be applied to the art-bearing sediments, but TL dating of associated heated artefacts can provide additional age control. Both strategies were employed at Pedra Pintada in Brazil (Michab *et al.*, 1997; Roosevelt *et al.*, 1996). The continuation of such applications, and the development of innovative methods for the *direct* dating of paintings and petroglyphs, promises to make rock art research an exciting new theatre of luminescence activity.

6. RETROSPECT AND PROSPECT

Luminescence dating can claim a place in archaeometry that stretches back nearly half a century. In this overview, I have attempted to cover some of the earliest applications and several of the latest contributions to world archaeology. A few studies have been discussed in detail, particularly the recent controversial findings from Tabun Cave in Israel (Mercier *et al.*, 1995b), Diring Yuriakh in Siberia (Waters *et al.*, 1997a,b), and Jinmium rock shelter in Australia (Fullagar *et al.*, 1996). These TL contributions to the story of human antiquity and ancestry are complemented by the novel deployment of optical dating methods to anthropogenic earthen mounds (Feathers, 1997b), quartzite pebbles (Richards, 1994) and rock art (Rees-Jones and Tite, 1997a,b; Roberts *et al.*, 1997).

There are also archaeometric applications of luminescence dating that have *not* been canvassed in this review. TL investigations of fossil bone, tooth and shell (Christodoulides and Fremlin, 1971; Driver, 1979; Jasinska and Niewiadomski, 1970), archaeometallurgical slags (Elitzsch *et al.*, 1983), ancient man-made glass (Sanderson *et al.*, 1983) and calcite deposits in caves (e.g. Aitken and Bussell, 1982; Debenham, 1983; Debenham and Aitken, 1984; Wintle, 1978) have been largely discontinued during the last decade, in favour of flint and sediment dating. Our current knowledge of the potential and pitfalls of these applications is well summarised by Aitken (1985) and little more can be added here. The call by Wintle (1980) for further studies of calcite and biological materials remains as appropriate now as it was then, given their undiminished archaeological importance.

What are the new archaeological frontiers for luminescence dating, as we approach the 21st century? A reliable method to date individual grains of quartz and feldspar would allow a variety of contentious issues to be addressed, such as the bioturbation of archaeological deposits, the dating of rock art and standing stones, and the identification and rejection of "contaminant" grains. Such a move would bring luminescence dating into line with other numerical dating methods, which have already reaped the benefits from using small samples (Wintle, 1996). Novel materials, such as phytoliths, and the extraction of tried-and-tested minerals from unusual contexts (e.g. quartz grains from mud-wasp nests and quartzite artefacts) should be further investigated, as should the pursuit of new signals and procedures to allow luminescence dating to contribute to questions of early Quaternary prehistory. The direct dating of heated artefacts, the historical foundation of luminescence dating, will continue to play a major role in archaeology. But while developments of long-range and high-precision luminescence chronologies pose the hardest challenges, they also offer the greatest prospects to open new windows onto our human past.

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