Optically stimulated luminescence dating of Palaeolithic cave sites and their environmental context in the western Mediterranean

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1. Introduction

1.1. HUMAN INTERACTION WITH PLEISTOCENE PALAEOENVIRONMENTS

The Quaternary is the most recent epoch in Earth's history and has been characterised by substantial climatic and environmental changes, the evolution of humans in Africa and their subsequent colonisation of most terrestrial landscapes. Successive cycles of global cooling and warmer interglacial phases are the core feature of the Quaternary climate and had major impact on the palaeoenvironments of terrestrial landscapes and thus the habitats of hominin populations occupying these landscapes (Klein, 2009; Lowe and Walker, 2015).

The Pleistocene is a geological period representing the earlier phase of the Quaternary that lasted from about 2.6 millions of years ago (Ma) to the onset of the most recent interglacial, the Holocene (~11.7 thousands of years ago (ka)) (Cohen and Gibbard, 2011). The substantial oscillations of global climate during Pleistocene times were mainly driven by periodic variations in the Earth's main orbital parameters – precession, obliquity and eccentricity – and their interaction with global oceanic and atmospheric circulations (Emiliani and Geiss, 1959; Hays et al., 1976; Milankovitch, 1920; Paillard, 1998). Climatic stages can be detected through variations in the content of oxygen-18 (^{18}O) over oxygen-16 (^{16}O) in marine sediment records with enrichments in ^{18}O representing cold glacial periods - when large amounts of water from the oceans became locked in ice sheets – and ^{18}O depletion reflecting warmer temperatures with decreased ice volumes during interglacial phases (Fig. 1.1.1, odd numbers represent interglacial events) (Shackleton, 1975, 1987).

Expansions and contractions of the Earth's ice sheets and glaciers influenced the rise and fall of global sea-levels (Fig. 1.1.1) and, at a more regional scale, water volume and shape of limnic and fluvial systems. Global cooling during glacial phases was associated with increasing aridification across most terrestrial landscapes with declined precipitation being mostly a consequence of reduced evaporation from the colder oceans (Bigg, 1995).

On the African continent, large rainforest areas became grassland or savanna, while grassland and savannah regions turned into desert (deMenocal, 1995; Sarnthein, 1978). The reduced vegetation cover in turn fostered wind erosion (deflation) and thus the expansion of semi-arid and arid landscapes in these regions (Darkoh, 1998). Around the Miocene/Pliocene boundary, arid conditions coupled with aeolian sand accumulation lead to the formation of the Saharan desert (Micheels et al., 2009; Schuster et al., 2006), which has since then served as a biogeographical barrier between sub-Saharan and North Africa (Lahr, 2010). It was only during the relatively short-termed periods of enhanced humidity in the Pleistocene – the 'green Sahara' events – that subtropical savannah landscapes expanded, enabling human habitation and crossings of the desert (e.g. Larrosaña, 2012; Trauth et al., 2009; Whiting Blome et al., 2012).

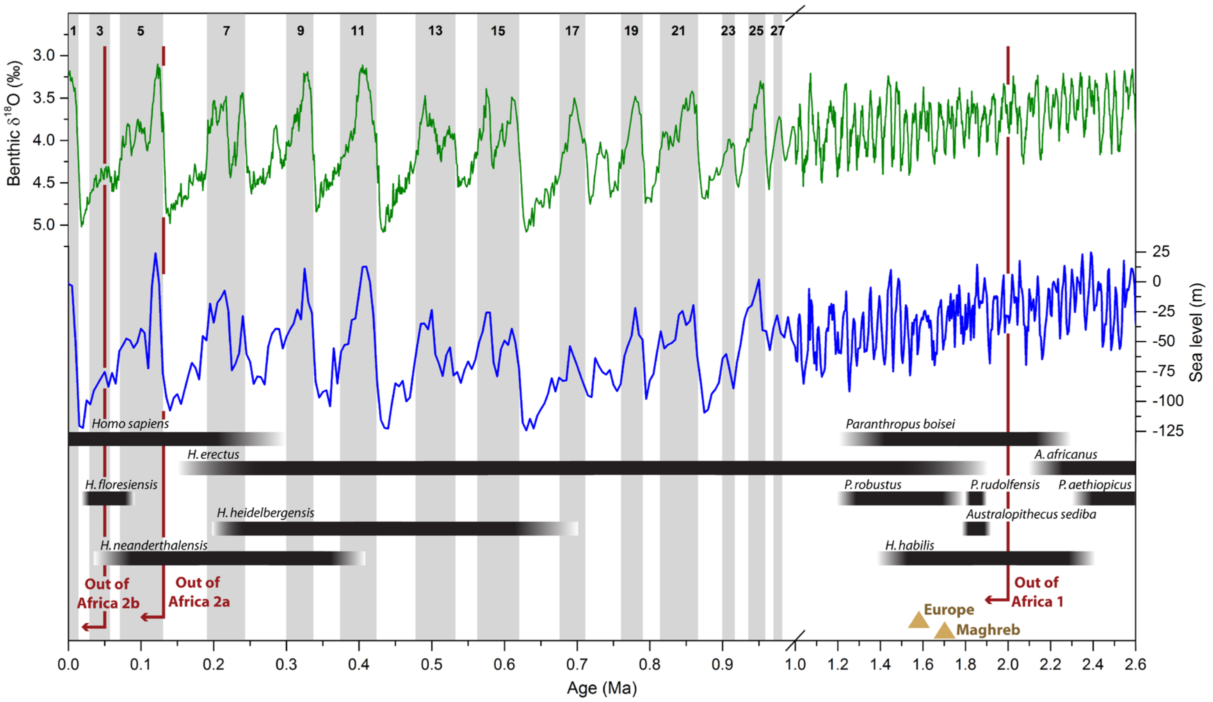

Fig. 1.1.1 Quaternary hominin evolution (after Antón et al., 2014), marine oxygen isotope record after Lisiecki and Raymo (2005) illustrating past glacial cycles and record of global sea-level fluctuations (Miller et al., 2005) with a special emphasis on the last 1 Ma, which are of particular relevance for this thesis. Highlighted are Out of Africa human dispersal events; yellow triangles represent the timing of the first early human settlements in the Maghreb and Europe.

There is a general consensus that the genus *Homo* evolved in Africa sometime between 2.5 and 2 Ma ago (Fig. 1.1.1) and then started to disperse within and out of the continent (Klein, 2009). Most of what is known about the emergence of early *Homo* comes from the East African Rift Valley (e.g. Delagnes and Roche, 2005; Partridge et al., 1995; Quade et al., 2004). Less well understood are, however, the exact timing and the routes human populations took throughout the Pleistocene to extent their range to the far southern and northern margins of Africa (Klein, 1994; Maslin et al., 2014; Raynal et al., 1995) and from the latter further into Eurasia (Klein, 2009).

In recent years, the archaeological and palaeoenvironmental record of the western Mediterranean – comprising north-western Africa and southern Iberia - has gain considerable importance in the study of human evolution (Finlayson et al., 2006; Garcea, 2012; Hublin et al., 2017). The Strait of Gibraltar has been argued by some authors to represent a potential hominin dispersal route from Africa into Europe in the Early Pleistocene at times of when the Mediterranean sea-level was significantly lowered (Alimen, 1975; Gibert et al., 2016; Sharon, 2011). Further interest in the north-western African archaeological sites has arisen in part because of evidence for an early appearance of behavioural modernity which may be linked to the dispersal of anatomically modern humans (AMH) from Africa (d'Errico et al., 2009; Klein, 2008; McBrearty and Brooks, 2000). And the south of the Iberian Peninsula, finally, has been interpreted as an ecological refugium for late Neanderthals before they eventually became extinct in the Late Pleistocene (d'Errico and Sánchez Goñi, 2003; Zilhão, 2006).

The key to understand human dispersal across and their interaction with palaeolandscapes are reliable chronologies of geological and archaeological archives which provide the framework for reconstructing the history of environmental change and human occupation patterns. Caves can safely store complex sedimentary records over millennia by providing permanent protection from sub-aerial weathering and erosion (Sasowsky and Mylroie, 2007). As they concurrently offer natural shelter, caves also often act as focus for Pleistocene human activity traceable in the stratigraphical sequences in form of artefact concentration, fire features, faunal and human remains. The preservation of disparate sources of chronological, behavioural and environmental evidence in close proximity makes such Palaeolithic cave sequences optimal archives to successfully reconstruct not only environmental and human population responses to climatic change, but also their mutual interactions throughout the Pleistocene (e.g. Barton et al., 2009; Belmaker and Hovers, 2011; Pirson et al., 2012). Quaternary dating methods enable determination of absolute ages for a variety of different materials that can be found at Palaeolithic cave sites such as bones, charcoal, heated artefacts, sediments and speleothems. Optically stimulated luminescence dating is widely applicable in such contexts, as it provides reliable estimates of the time elapsed since sediments were last exposed to sunlight.

In this dissertation, the potential of sand-sized quartz grains as luminescence chronometer to reliably establish the timing of sediment deposition and human occupation at Palaeolithic cave sites in the western Mediterranean is investigated. With this new chronological evidence, this thesis aims to contribute to an improved understanding of the significance of the region in the study of human evolution and dispersal within and out of the African continent that took place under the variable climatic conditions over the course of the Pleistocene.

1.2. The western Mediterranean perspective on human evolution

Humans evolved over Quaternary timescales, but the link between hominin evolution and climatic conditions remains unclear. In the past, the emergence of the genus *Homo* has often been linked global cooling and the subsequent increase of aridity coupled with a progressive expansion of open, grassland habitats on the African continent during the Quaternary (Cerling, 1992; Cerling et al., 2011). This hypothesis was, however, questioned by recent archaeological discoveries - including new hominin fossil evidence (e.g. Berger et al., 2010; Lordkipanidze et al., 2013) – and current synthesis of palaeoenvironmental proxies, i.e. aeolian dust, lake, faunal, stable isotopic and volcanologic records (deMenocal, 2004; Trauth et al., 2009). These evidences suggest that intentional developments of new behavioural patterns and toolkits in early *Homo* could have emerged in response to dynamic environments characterised by fluctuating moisture and aridity, shifting resource regimes and spatial heterogeneity on the African continent between ~2.5 and 1.5 Ma ago (Antón et al., 2014; Bobe and Behrensmeyer, 2004; Potts, 2012).

Irrespective of the reasons or factors eventually leading to the evolutionary success of the human lineage while other coexisting species became extinct, archaeological and palaeoanthropological evidence from the Caucasus show that hominin groups started to settle ‘Out of Africa’ - and reached the edge of south-eastern Europe - by minimally 1.85 Ma ago (Ferring et al., 2011). The opening of a corridor for hominin dispersal from East Africa into Eurasia has often been linked with the end of the humid period at the onset of the Pleistocene (2.6 - 2.0 Ma) (Prat, 2016), during which mean annual temperatures and precipitation rates in the Mediterranean region were substantially higher compared to present-day conditions (Klotz et al., 2006; Leroy et al., 2011). Out of Africa events during the Pleistocene were sporadic and multidirectional (Antón et al., 2014; Garcea, 2016) and according to Prat (2016) “did not occur on a once-off basis, but as many discontinuous occupations, over more or less distant areas and at times with episodes of turning back”. Similar to the persisting debate why people initiated these dispersals from Africa - and the role of climatic and environmental changes or social factors in the process – it remains unclear where on the African continent human groups settled that contributed to the different migration waves, which routes they took and why (e.g. Garcea, 2012; Lahr, 2010), and whether hominin settlements in Europe during the Early Pleistocene were continuous or discontinuous (e.g. Bermúdez de Castro et al., 2016; Dennell, 2003).

This thesis focusses on the western Mediterranean, a region of potentially high strategic importance for hominin dispersals out of Africa as well as for a late survival of Neanderthal populations on the European continent. With its exceptional archaeological and palaeoenvironmental record, the western Mediterranean is a key region to understand the possible role of north-western African populations in the colonisation of the European continent throughout the Pleistocene and Neanderthal displacement in southern Iberia. Of particular relevance to this work are two migration periods: The first European peopling during the Early Pleistocene (~1.6 - 0.8 Ma) and the dispersal of anatomically modern humans from Africa between 130 ka and 40 ka which lead to the eventual extinction of the indigenous Homo neanderthalensis in Eurasia (Fig. 1.1.1) (e.g. Garcea, 2016; Mellars, 2004; Prat, 2016; Villa and Roebroeks, 2014).

1.2.1. Out of Africa 1 and the early colonisation of Europe

While the earliest stone artefacts are known to occur ~3.3 Ma in Lomekwi 3, Kenya, in eastern Africa (Harmand et al., 2015), evidence for early human settlements in the Maghreb (defined here as Morocco, Algeria, Tunisia and western Libya) are not documented before ~1.7 Ma at Ain Hanech and El-Kherba, Algeria (Figs. 1.1.1 and 1.2.1; Parés et al., 2014; Sahnouni et al., 2002) and coincide with a period of increased aridity on the African continent (deMenocal, 1995, 2004). Although these early Maghrebian dates are not undisputed (Geraads et al., 2004), first hominin migrations into North Africa can be safely placed in a time period between 2.5 Ma and 1.2 Ma (Geraads and Amani, 1998; Raynal et al., 2001). The earliest archaeological sites discovered in the Levant and Europe are ‘Ubeidiya, Israel (~1.5 Ma, Martínez-Navarro et al., 2009), Pirro Nord, northern Italy (1.3 - 1.6 Ma, Arzarello et al., 2012) and Orce, Iberia (~1.4 Ma, Toro-Moyano et al., 2013), respectively (Figs. 1.1.1 and 1.2.1).

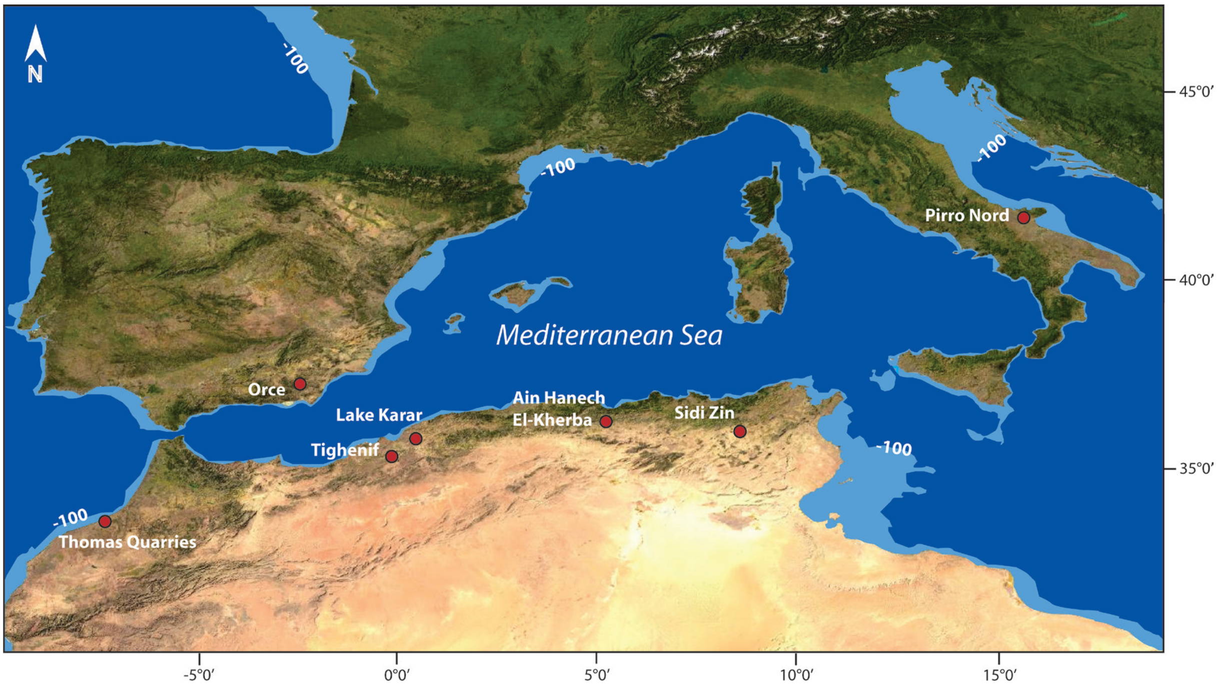

Fig. 1.2.1 Bathymetrical map of the western Mediterranean showing the present-day and the potential palaeo-coastlines during MIS 22, when sea-level was lowered by ~100 m (modified after EMODnet, 2017). Indicated are the locations of Lower Palaeolithic sites from the area mentioned in the text.

Although Early Pleistocene records of human settlements in Europe are sparse and the absolute dating of those sites highly challenging, current scientific evidence (i.e. from Dmanisi, Georgia (Ferring et al., 2011)) suggest a first colonisation of the continent by hominin populations coming from the east (Carbonell et al., 2008; Toro-Moyano et al., 2013). Researchers, however, strongly disagree about the continuity/discontinuity of those early human populations in Europe during the Early-Middle Pleistocene climatic transition (Clark et al., 2006) between 1.2 Ma and 0.7 Ma, when global climatic systems underwent major changes due to the establishment of large ice sheets in the northern hemisphere that covered major parts of the European landmass (Head and Gibbard, 2005; Maslin and Brierley, 2015; Ruddiman et al., 1989). Climate changes are likely to have caused a decrease in human population sizes with repeated episodes of local extinctions and periods of prolonged isolation in climatically favoured refugia, such as the Balkans, the Italian Peninsula and Iberia (e.g. Arribas and Palmqvist, 1999; Bermúdez de Castro et al., 2016; Dennell et al., 2011).

Directly related to this debate, is the similarly controversial question over the emergence of the Acheulian technocomplex in Europe – a distinctive stone tool industry mainly characterised by the production of bifaces (handaxes and cleavers) (Sharon, 2007) - which is placed by chronological evidence to roughly the same period of time, between ~1.0 Ma and 0.7 Ma (Sharon and Barsky, 2016). Central focus of this discussion is whether the European biface assemblages were i) a local European development or whether they originated outside of Europe ii) to the east in Asia or the Levant, or iii) to the south in Africa (e.g. Carbonell et al., 2016; Dennell et al., 2011). As sites yielding pre-Acheulian lithic assemblages in Europe are extremely rare, there is no solid scientific evidence supporting the local development theory (Sharon and Barsky, 2016). While the hypothesis of an eastern origin of the European Acheulian is primarily based on the finds from Caucasian sites (Amirkhanov et al., 2014; Gabunia et al., 2000; Lyubin and Belyaeva, 2006), more recent studies reinforce the potential significance of Northern Africa and suggest that the Acheulian in Europe may have originated through the straits of Gibraltar at times when the Mediterranean sea-level was significantly lowered i.e. during Marine Isotope Stage (MIS) 22 at ~0.9 Ma (Figs. 1.1.1 and 1.2.1; e.g. Alimen, 1975; Santonja et al., 2016; Sharon, 2011).

The strait of Gibraltar is currently ~14.5 km wide and according to Arribas and Palmqvist (1999) a sea-level drop of approximately 300 m would be required to close it, while a decreased sea-level of 100 m – as can be assumed for MIS 22 – would only narrow it (Fig. 1.2.1). Tectonic uplift during Quaternary times as evidenced by a sequence of raised shorelines in Gibraltar (Rodríguez-Vidal et al., 2004) has been used to emphasise that even the lowest Early Pleistocene sea-level lowstand would have had hardly any effect on the extent of the central channel of the Strait of Gibraltar (~5 km wide and 300 m deep) and early human crossings via that passageway are, therefore, considered highly unlikely (Derricourt, 2005; Muttoni et al., 2010; 2014). On the contrary, other authors have argued that a 100 m drop of the Mediterranean sea-level would have exposed several islands (Martinet and Searight, 1994) which together with changes in the salinity of Atlantic waters at that time (MIS 22) and the subsequently reduced intensity of the marine currents in the strait of Gibraltar (Gibert et al., 2003), would have facilitated contact between the Iberian Peninsula and the Maghreb (Lahr, 2010; Santonja and Pérez-González, 2010; Sharon, 2011).

The pre-Acheulian and Acheulian occupation record of the Maghreb is relatively sparse and many of the discovered sites lack chronological control. Acheulian lithic industries are i.e. documented at the sites of Tighenif and Lake Karar in northern Algeria as well as at Thomas Quarries in Morocco and Sidi Zin in northern Tunisia (Fig. 1.2.1; Lahr, 2010 and references therein). For these sites, researchers have made the attempt to produce absolute dates using different chronological methods in the past (e.g. Parés et al., 2014; Rhodes et al., 2006). Their results are, however, often limited by the uncertainties associated with the age estimates, the fact that only few archaeological layers could be dated for each site or were strongly disputed among the scientific community. In order to understand whether the origin of the European Acheulian lies in North African and was brought to Europe by hominin populations crossing the strait of Gibraltar, further research at archaeological sites of that time period has to be conducted at both sides of the Mediterranean, including reliable absolute dating.

1.2.2. Out of Africa 2 – success and failure of anatomically modern human dispersals from North Africa

Throughout large parts of the Middle Pleistocene, succeeding the Out of Africa 1 migration period, hominin populations evolved separately within and outside of Africa (Hublin, 2009). During MIS 5, descendants of the African clade – early anatomically modern humans – living in North Africa initiated a dispersal into the Levant (often referred to as Out of Africa 2a, ~130 ka – 80 ka (Garcea, 2012)), where the fossil remains of Skhul and Qafzeh (both Israel; Fig. 1.2.2) provide the oldest evidence of AMHs outside of the African continent at between ~135 ka and 100 ka (Fig. 1.1.1; Grün, 2006; Grün et al., 2005). Since this thesis particularly focusses on the western Mediterranean region, the southern AMH dispersal route from Africa – through East Africa into the Arabian peninsula (Armitage et al., 2011; Beyin, 2011; Lahr and Foley, 1994) – is not discussed any further. Of great importance is, however, the northern passageway which included dispersals through north-eastern Africa and the Nile corridor, the Sahara and the Mediterranean coast into the Levant (Garcea, 2016 and references therein).

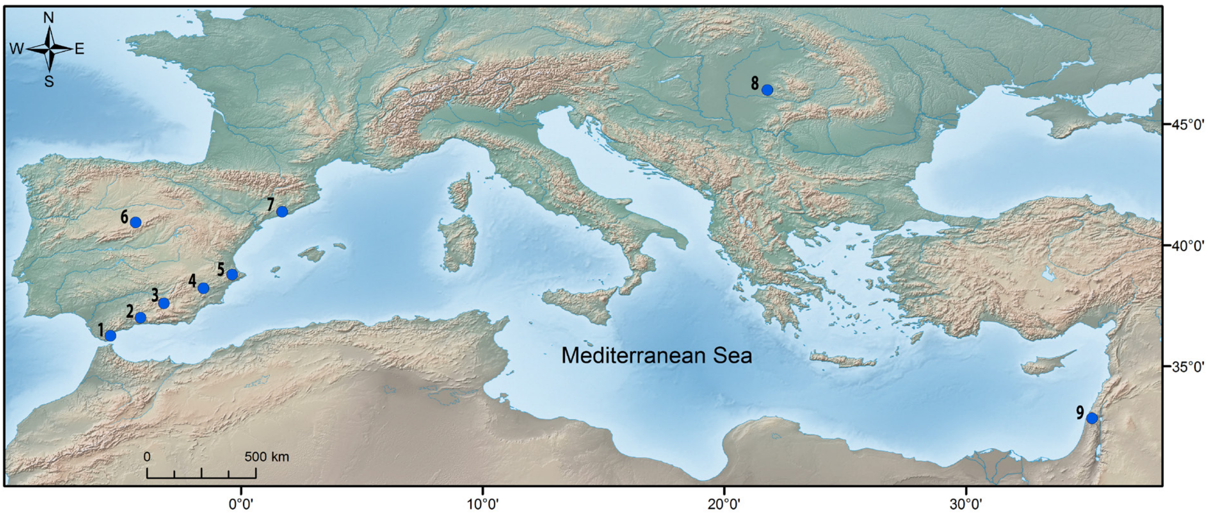

Fig. 1.2.2 Location of the Middle and Upper Palaeolithic sites mentioned in the text. 1 – Gorham's Cave; 2 – Zafarraya; 3 – Carihuela; 4 – Cueva Antón; 5 - El Salt; 6 – Abrigo del Molino; 7 – Cova del Rinoceront; 8 - Peştera cu Oase; 9 – Skhul and Qafzeh.

Climatic conditions became less hospitable - including rapid cooling and increased aridity (Rampino and Self, 1992) - between MIS 5a and MIS 4 (~74 ka) in the south-western Mediterranean basin (Cheddadi and Rossignol-Strick, 1995; Timmermann and Friedrich, 2016; Whiting Blome et al., 2012). This, together with the potential failure in the competition for resources against Neanderthals – the descendants of a western Eurasian clade living in Europe and the Levant – resulted in a massive decline of AMH populations living in the Levant (if not complete depopulation) between about 80 ka and 50 ka (Garcea, 2010a; Shea, 2003, 2010), whereas Neanderthals were still present throughout MIS 4, until about 45 ka (Bailey et al., 2008; Shea, 2008).

Another major dispersal wave was initiated by African AMHs (Out of Africa 2b, Fig. 1.1.1) after 60 ka, which eventually resulted in the displacement of Neanderthals in the Levant and a further expansion of

AMHs into other parts of Europe and Asia (e.g. Mellars, 2006; Shea, 2010; Timmermann and Friedrich, 2016). Various reasons have been proposed to explain the success of Out of Africa 2b compared to the 2a event, i.e. more favourable climatic conditions during MIS 3 (Heterington and Reid, 2010), strong demographic pressure on Neanderthal populations (Powell et al., 2009), AMH adaptation skills (McBrearty and Brooks, 2000), competitive exclusion (Banks et al., 2008) and improved technological equipment, namely projectile armatures (Lombard and Phillipson, 2015; Shea and Sisk, 2010).

While recent genetic evidence has emphasised the role of North Africa for AMH dispersals out of the continent by associating Neanderthal-AMH interbreeding - dated to between 65 ka and 47 ka - with North African populations (Pagani et al., 2015; Sankararaman et al., 2012), little is known about the advent of cultural modernity in AMHs which might be the key factor to understand the evolutionary success of our lineage over Neanderthals (d'Errico et al., 2009; McBrearty and Brooks, 2000; Vanhaeren et al., 2006). The emergence of behavioural modernity – typically associated with instances of symbolic artefacts, pigment use, engravings or formal bone tools at archaeological sites (e.g. d'Errico and Vanhaeren, 2007; Klein, 2008; Kuhn and Stiner, 2007; McBrearty and Brooks, 2000) – in the Maghreb is usually linked to the Middle Stone Age (MSA) lithic technocomplex called the Aterian. This technocomplex is primarily characterised by the appearance of pedunculated tools and bifacial foliates but also known for the presence of blades, bladelets, end-scrapers, small Levallois cores and personal ornaments (Bouzouggar and Barton, 2012). Consequently, many studies have focussed on the technological definition, the timing and the geographical distribution of the Aterian industry to investigate the advent of cultural modernity in AMH populations, and to understand the drivers of population mobilisation out of Northern Africa (e.g. Bouzouggar and Barton, 2012; d'Errico et al., 2009; Garcea, 2010b). Despite the significant progress made in this research field over the last years (see e.g. reviews by Garcea, 2016; Reyes-Centeno, 2016), we still do not fully understand where, when and why technological innovations took place and modern human behaviour emerged on the Africa continent between Out of Africa 2a and 2b and which of the proposed North African migration routes were critical for the eventual AMH dispersal into Eurasia.

1.2.3. Neanderthal habitation of southern Iberia

At the time when AMHs arrived in Europe around 50 ka (Hublin, 2012), they came in contact with indigenous Neanderthal populations living in western Eurasia (Mellars, 2004). Genetic evidence suggest that ancestors of modern humans split from the source population of Neanderthals and Denisovans – a sister group recently identified in the Russian Altai (Meyer et al., 2012) – at the beginning of the Middle Pleistocene, between 765 ka and 550 ka (Meyer et al., 2016). Early Neanderthal artefact assemblages can be classified as Acheulian, while with the onset of the Middle Palaeolithic (~300 ka, MIS 8) archaeological records are characterised by the advent of the Levallois technology (Roebroeks and Soressi, 2016). Levallois is a hierarchical core reduction strategy which entails, according to Adler et al. (2014), the "multistage shaping […] of a mass of stone (core) in preparation to detach a flake of predetermined size and shape from a single preferred surface" (see also Boëda, 1994, 1995). The first AMH fossils in Europe were found in Peştera cu Oase, Romania (Fig. 1.2.2; Trinkhaus et al., 2013). The two main specimens, a mandible (Oase 1) and a skull from a second individual (Oase 2), were directly dated and revealed for the latter a minimum age of 33.5 kcal BP (28.9 +∞/-0.2 ka BP, Trinkhaus et al., 2013), while the former gave an age close to 40 kcal BP (34.3 +1.0/-0.8 and >35.2 ka BP, Trinkaus et al., 2003) which is around the same time when Neanderthals disappeared from most archaeological records on the continent (~40 ka, Villa and Roebroeks, 2014; Zilhão, 2013).

The timing and causes for the demise of the late Neanderthals in Europe is one of the central topics in palaeoanthropology, and - since it remains unresolved - matter of an ongoing debate. Recent studies have argued that the widely assumed “superiority” of AMHs over Neanderthals – in terms of i.e. weaponry, subsistence strategies and cooperation skills (e.g. Marean, 2015 and references therein) – appear to be less profound than previously thought (Roebroeks and Soressi, 2016; Villa and Roebroeks, 2014). Scientific evidence from the last decade support this view by showing that Neanderthal populations were very similar to their AMH contemporaries in terms of diet, fire use, stone tool technologies and even symbolic behaviour (e.g. Bar-Yosef and Pilbeam, 2000; Finlayson et al., 2012; Henry et al., 2014; Roebroeks and Villa, 2011). Nonetheless, following Roebroeks and Soressi (2016), “there is no doubt that the Neanderthal phenotype ultimately disappeared through (some form of) competition with modern humans” in Eurasia within just a few thousand years, “even though the specifics of that process […] are largely unexplored”. Higham et al. (2014) recently quantified the temporal overlap between the two hominin groups on the European continent to 2,600 - 5,400 years, while emphasising that Neanderthal disappearance occurred in different regions at different times.

In the past, the south of the Iberian Peninsula has often been interpreted as an ecological refugium for late Neanderthals with the Ebro River and the Cantabrian Cordillera serving as natural barriers between the areas in the south and the rest of Europe (e.g. d’Errico and Sánchez Goñi, 2003; Zilhão, 2006), a view supported by a number of palaeoenvironmental studies (de Abreu et al., 2003; Moreno et al., 2005; Roucoux et al., 2005; Sánchez Goñi et al., 2000, 2002). Radiocarbon dates from sites like Carihuela, Zafarraya (both Spain) and Gorham’s Cave (Gibraltar; Fig. 1.2.2) placed the survival of Neanderthals in southern Iberia to post-42 ka (Fernández et al., 2007; Hublin et al., 1995; Pettitt and Bailey, 2000) and maybe even as late as 28 ka (Finlayson et al., 2006). These dates were, however, strongly disputed by Higham and colleagues, who argue that those early dates suffer from incomplete removal of contamination in the collagen (Higham et al., 2014; Wood et al., 2013). Nevertheless, there still remain reliable evidence for Neanderthal presence in southern Iberia after 40 ka i.e. Cueva Antón, shortly after 38.5 kcal BP (Zilhão et al., 2010a).

While comparatively many studies contribute to the ongoing discussion about the Neanderthal demise in southern Iberia (e.g. Bar-Yosef and Pilbeam, 2000; d’Errico and Sánchez Goñi, 2003; d’Errico et al., 1998; Finlayson et al., 2006; Higham et al., 2014; Zilhão et al., 2010b), relatively little attention has been paid to the time range preceding the last 50 ka. In part this is due to the overwhelming reliance on radiocarbon dating in archaeology which is limited to ~50 ka (Reimer et al., 2016). It is, however, exactly this period of time that has the potential to shed further light into the history of Neanderthal populations and their subsistence strategies in the region, which might eventually help us to better understand the causes of their final extinction in the Iberian Peninsula. Authors have just recently started to fill this gap by providing reliable chronostratigraphies for archaeological cave sites (Fig. 1.2.2) and their palaeoenvironmental context located mostly in the inland – i.e. Cuevá Anton (~80-35 ka, Zilhão et al., 2016), Abrigo del Molino (60-31 ka, Álvarez-Alonso et al., 2016) and El Salt (60-45 ka, Galván et al., 2014) – but also at the Mediterranean coast – Cova del Rinoceront (~210-74 ka, Daura et al., 2015) – of Iberia safely covering MIS 6 to 3.

1.3. DATING PALAEOLITHIC CAVE SITES

Our understanding of the timing of human occupation and palaeoenvironmental changes in a region largely depends on the reliability and suitability of Quaternary dating techniques which enable comparisons between stratigraphical layers on site, regional and global scale (Lowe and Walker, 2015). The most widely applied radiometric methods for dating Palaeolithic cave sites are radiocarbon, luminescence (comprising thermoluminescence (TL) and optically stimulated luminescence (OSL)), U-series and ESR (Electron Spin Resonance) (Fig. 1.3.1). These techniques allow reliable age determination of a variety of different materials, which directly (i.e. bones, charcoal, teeth and heated flint) or indirectly (i.e. sediments and speleothems) store archaeological evidences at those sites and have successfully been used in the past to i.e. date the Middle Pleistocene Neanderthal occupation in Sima de los Huesos ~430 ka (Arsuaga et al., 2014), the use of personal ornaments at MSA sites in Morocco (d'Errico et al., 2009) and Palaeolithic cave art in Iberia (Pike et al., 2012), as well as to reconstruct how wet phases in North Africa affected AMH migration out of and back to the continent (Hoffmann et al., 2016).

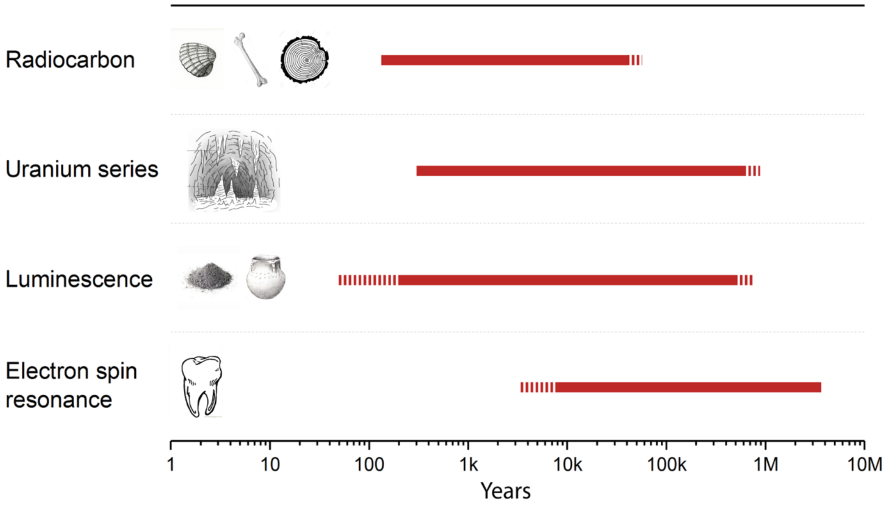

Fig. 1.3.1 The effective dating ranges of the different Quaternary dating techniques mentioned in the text after Walker (2005) and some examples of materials which are typically dated using those methods.

The radiocarbon method (for further methodological details see Jull and Burr, 2015) is undeniably of crucial importance for dating Palaeolithic sites as it provides highly precise ages for organic-bearing archaeological finds (Pollard, 2009; Taylor, 2001). It is, however, restricted by the preservation of collagen in the samples (when dating bones), an upper dating limit of ~50 ka and a calibration to cosmic ray flux through time (Brock et al., 2012; Reimer et al., 2016). While luminescence, ESR and U-series dating, on the other hand, have the advantage of covering much older time periods (usually the whole Middle Palaeolithic, sometimes even parts of the Lower Palaeolithic) by using inorganic materials for dating, determined absolute ages are commonly associated with comparatively larger uncertainties.

Out of all the above-mentioned methods, OSL dating is the least restricted as it determines the time elapsed since sediments were last exposed to sunlight. In principle, it can be used for any given archaeological site, as i) the material desired for age determination - mineral grains such as quartz and feldspar - is ubiquitous in natural sedimentary environments and ii) the only crucial prerequisites are its sufficient contact with sunlight prior to deposition and a consistent dose rate through time (Aitken, 1998). Although the accumulation age of a sediment layer, within which archaeological finds are located, is not per se identical with the age of the human activity reflected by those finds (i.e.

due to redeposition by geologic forces), OSL is a widely applied dating method for Palaeolithic sites (Jacobs and Roberts, 2007) and usually complemented by detailed sedimentological investigations of the stratigraphical context of each sample to ensure the significance of the obtained dates (Feathers, 2015).

It is now almost 20 years since Duller et al. (1999) and Bøtter-Jensen et al. (2000) presented their technical improvements of the standard Risø OSL/TL reader which extended the field of applications for OSL dating to resolve issues of i.e. post-depositional mixing in archaeological sediment layers (Jacobs and Roberts, 2007) by enabling routine measurements of the luminescence signal stored in individual sand-sized quartz grains. Before that only multiple-grain aliquots (containing many grains which are measured simultaneously) could be measured in greater quantities within reasonable times. Further favoured through the development of a new OSL measurement protocol by Murray and Wintle (2000, 2003) at about the same time – the single-aliquot regenerative-dose (SAR) protocol (chapter 1.6.5) - single-grain dating became, over the following years, an increasingly used tool for establishing chronologies of sediment deposits in archaeological contexts all over the world (e.g. Fitzsimmons et al., 2014; Guérin et al., 2012; Jacobs et al., 2011). There are, however, several aspects of single-grain quartz OSL dating – mostly concerning the variability observable in the luminescence behaviour of individual grains within one sample - which up until now remain inexplicable (Jacobs and Roberts, 2007). These include i.e. the occurrence of an extra component of random variation (called overdispersion) in single-grain distributions which restrict the precision of the final age estimates and hamper our ability to distinctly separate different age populations within one sample (Galbraith et al., 1999; Thomsen et al., 2007). Additional complications may arise when sediment samples i) are close to saturation (indicating the upper limit for age determination) or ii) yield large proportions of individual grains which emit luminescence signals unsuitable for OSL dating or no signal at all. While various alternative techniques have been proposed to extend the age range of standard quartz OSL (e.g. Ankjærgaard et al., 2016; Arnold and Demuro, 2015; Singarayer and Bailey, 2003; Wang et al., 2006), conventional multiple-grain and single-grain dating remain the first choice for establishing reliable chronostratigraphies at Palaeolithic cave sites.

1.4. RESEARCH QUESTIONS

The overview of the current knowledge on the history of hominin evolution, dispersal and displacement in the western Mediterranean region shows there are still gaps that hamper our understanding of the contribution of north-western African populations in the colonisation of the European continent and Neanderthal subsistence in southern Iberia prior to the arrival of AMHs in the region. This is despite significant progress made in terms of methodological approaches in the fields of palaeoenvironmental, geochronological and archaeological research in recent years, which have enabled more precise reconstructions of palaeoenvironmental conditions and how hominin groups adapted to those throughout Pleistocene times.

Archaeological caves can safely store records of human activity and climatic fluctuations in their stratigraphical sequences and, therefore, have the potential to provide optimal conditions for studying human-environmental interactions, patterns of human dispersal and the emergence of new lithic technologies or behavioural indicators in a certain region in the past. Fundamental to all those studies are, however, solid geochronological frameworks coupled with archaeological, geological and sedimentological analyses of the Palaeolithic sequences at the investigated sites. Consequently, this thesis addresses the following research questions:

1. *To what extent can reliable chronostratigraphies for archaeological cave sites advance our understanding of Palaeolithic human behaviour and dispersal events in the western Mediterranean?*

2. *To what extent do sedimentary cave records provide evidence of climatic changes throughout the Pleistocene in the region?*

OSL is one of the key methods for establishing absolute chronologies of Pleistocene sedimentological sequences in geological and archaeological contexts all over the world. Of particular relevance for Palaeolithic cave sites is the single-grain dating approach which allows identification of multiple discrete age populations within a single stratigraphic layer. However, the suitability of a certain sediment sample for OSL dating and, consequently, the soundness of its final calculated OSL age, largely depend on the luminescence characteristics of the individual mineral grains desired for age determination. A basic understanding of those characteristics and of potential factors which might have falsely altered the determined burial age of a sediment sample - resulting in substantial over- or underestimations - are crucial for building reliable chronostratigraphies of Palaeolithic cave sites. Therefore:

3. *What are the quartz luminescence characteristics of the investigated Moroccan and southern Iberian sediments? How variable are they at site and regional scale through time?*

4. *What are the challenges to single-grain OSL dating in Palaeolithic cave sites in the western Mediterranean?*

Necessary to answer those research questions are archaeological sites at both sides of the Mediterranean Sea that provide detailed records of Palaeolithic hominin occupation and behaviour, palaeoenvironmental changes, and contain quartz-rich sediments suitable for OSL dating. The case study sites Thomas Quarries and Rhafas in Morocco are known for their Early to Middle Pleistocene and Middle to Late Pleistocene stratigraphical sequences, respectively and, therefore, bear great potential to improve our understanding about the role of northwest African hominin populations in Out of Africa human dispersal events and for the emergence of cultural modernity in AMHs. Vanguard Cave is a Palaeolithic site at the southernmost tip of the Iberian Peninsula providing information about environmental conditions and hominin subsistence strategies during times of Neanderthal dominance in the region since the last interglacial. The next section (chapter 1.5) will briefly introduce the case study sites while in the subsequent chapter 1.6 a methodological overview on OSL dating is given.

1.5. STUDY SITES

This section aims to introduce the Palaeolithic sites studied in this thesis by giving brief overviews of their past research history and summarising their archaeological and geological context. The three main sites discussed in this thesis are located in the western Mediterranean: the cave of Rhafas and the Thomas Quarries are situated in north-eastern and western Morocco, respectively; Vanguard Cave on the other hand faces the Mediterranean Sea at the present-day shoreline of Gibraltar (Fig. 1.5.1). For a methodological study (chapter 4), OSL samples from an archaeological site in Australia, Lake Mungo, are used for comparative reasons; the site is briefly introduced in chapter 1.5.4.

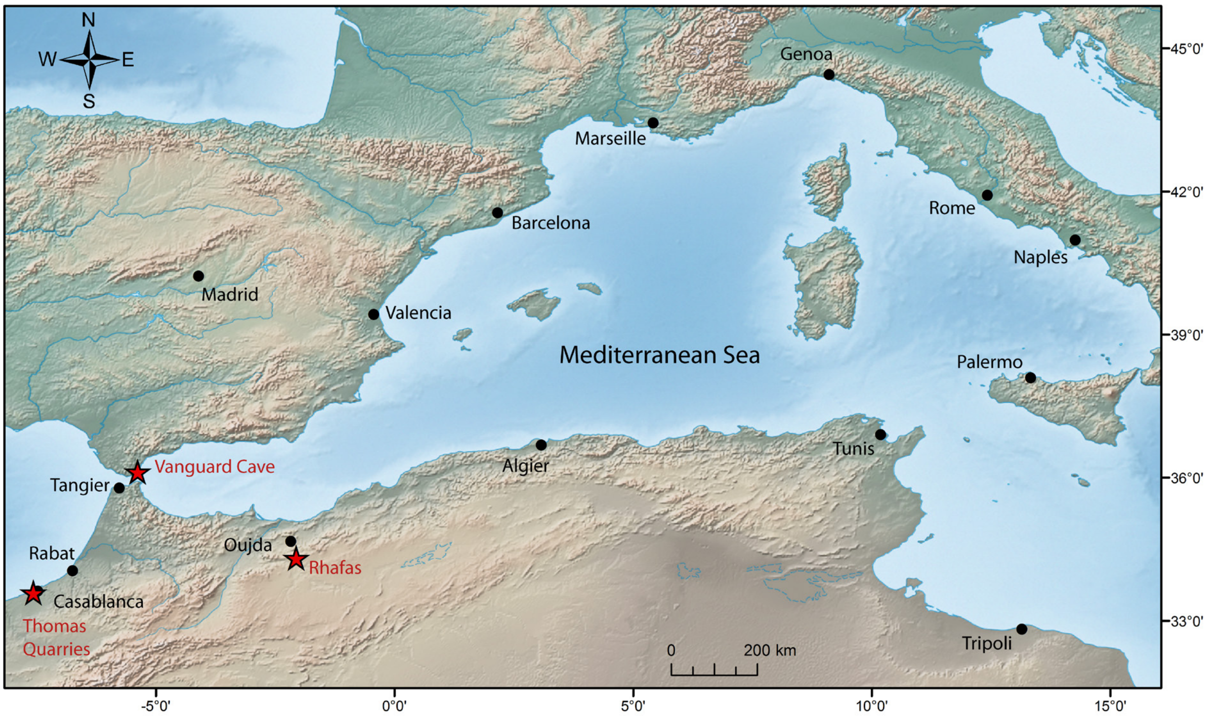

Fig. 1.5.1 Location of the three main study sites of this thesis in the western Mediterranean.

1.5.1. Rhafas Cave, Morocco

Rhafas is an inland archaeological cave site (Fig. 1.5.1) located in the Oujda Mountains in north-eastern Morocco, about 30 km south-eastwards from the city of Oujda and ~900 m above present-day sea-level (Fig. 1.5.2). Since its discovery in 1950, the site experienced several series of systematic excavations first by J.-L. Wengler (e.g. Wengler, 1993; 1997) and, since 2007, by the current excavation team headed by researchers from the Institut National des Sciences de l'Archéologie et du Patrimoine, Rabat, and the Max Planck Institute for Evolutionary Anthropology, Leipzig. With its long stratified archaeological sequence spanning the MSA (including the Aterian) through to the Neolithic, Rhafas contains valuable information about human occupation and dispersal, and cultural changes during the Palaeolithic in north-western Africa. Furthermore, the mostly aeolian sediment deposits at Rhafas – which were partly affected by post-depositional carbonate cementation – provide an important archive of past palaeoenvironmental conditions in the area on a local and regional scale.

The geology in the cave's surrounding is characterised by Palaeozoic substratum unconformably overlain by predominantly Mesozoic carbonates (Fig. 1.5.3, Talbi and Boudchiche, 2012). While the Palaeozoic units are composed of various types of metasediments, volcanic rocks and granitoids of Ordovician to Carboniferous age, the Mesozoic deposits consist mainly of Jurassic dolomite and limestone. The cave itself is situated within a limestone cliff that forms the local hilltop on the north-western slope of a prominent northeast/southwest trending valley (Fig. 1.5.2). The limestone unit unconformably overlies highly deformed meta-sediments and a coarse grained granodiorite that forms the valley floor (Fig. 1.5.3). During the Quaternary, the cave was filled with sand- and silt-rich aeolian sediments.

Since the 1990s, the lithic artefact assemblages of Rhafas, their cultural attribution and the palaeoclimatic conditions during times of human occupation of the cave in the Late Pleistocene and Holocene had been subject of numerous published studies by Wengler and colleagues (Wengler, 1997, 2001; Wengler and Vernet, 1992; Wengler et al., 2002). An absolute chronometric classification of the archaeological layers of Rhafas, however, was not realised until Mercier et al. (2007). For their study, selected samples were collected from the upper part of the cave fill sequence for thermoluminescence, OSL and radiocarbon dating and revealed ages (twelve TL, one OSL and two ^{14}C) for five archaeological layers of up to 107 ka (Mercier et al., 2007).

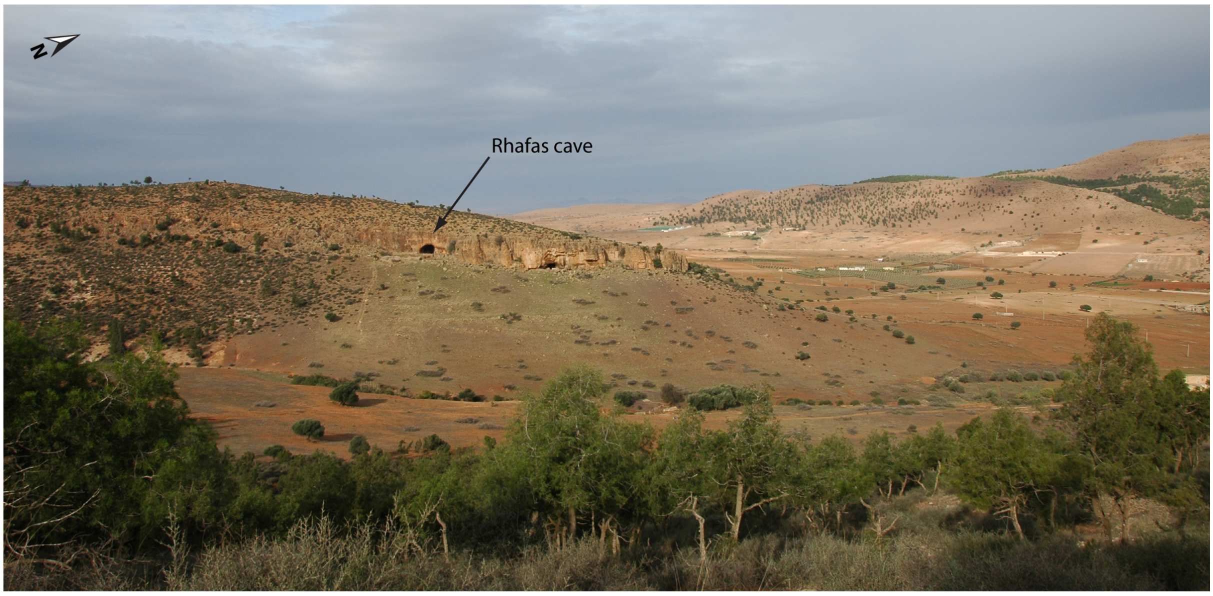

Fig. 1.5.2 Photograph of the Rhafas cave site.

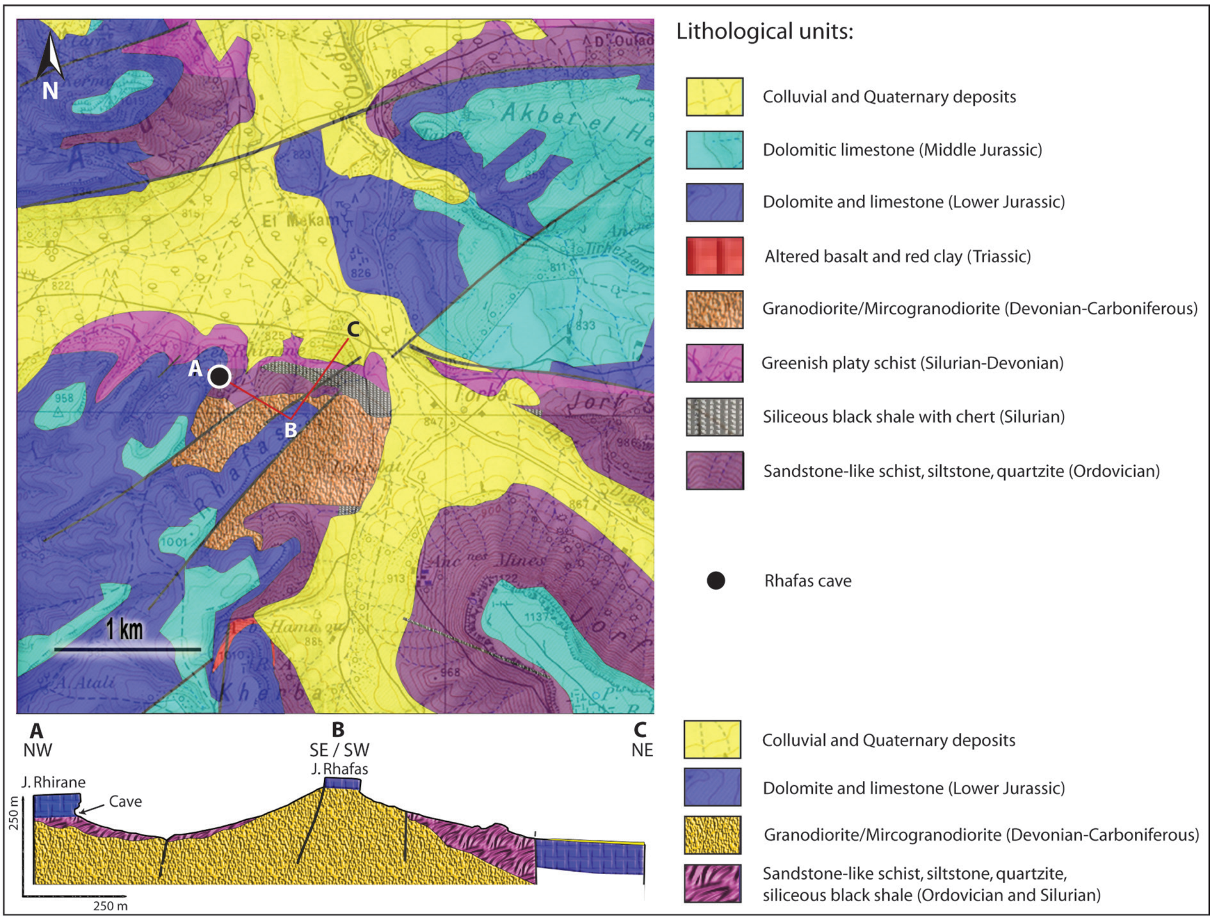

Fig. 1.5.3 Geological map and NW-SE-NE cross section of the Rhafas area (modified after Talbi and Boudchiche, 2012).

The recent field campaign included beside renewed excavations in the cave also the opening of new excavation squares on the relatively flat and terraced area in front of its entrance (Fig. 1.5.4). Geological evidence suggests that this area was formerly part of the cave itself. The large limestone boulders that are widely spread throughout the lower stratigraphical units of the terrace section are likely to represent the collapsed remains of the old cave roof. Some of the stratigraphical layers in this section are attributed to the LSA, a technocomplex which was previously thought to be missing at the site, as it is not preserved in the cave fill sequence.

Fig. 1.5.4 Photographs of the Rhafas cave site with the location of the different excavation sections.

1.5.2. Thomas Quarries, Morocco

The Thomas Quarries are a complex of Palaeolithic quarry sites (Thomas I, Thomas III and Oulad Hamida I) located in the area of Casablanca, western Morocco (Fig. 1.5.1), which is characterised by a series of large Quaternary shoreline barrier systems sub-parallel to the present shoreline of the Atlantic Ocean (Raynal et al., 2001). Since the beginning of a joint Moroccan-French research program in 1978, researchers established an extensive lithostratigraphical, biostratigraphical and archaeological framework for the area (e.g. Daujeard et al., 2016; Geraads et al., 1980; Lefevre et al., 1994; Raynal et al., 2010; Texier et al., 1994). Today the long geomorphological sequence at Casablanca is not only famous for reflecting global Quaternary sea-level fluctuations over the past 5.5 Ma (Lefevre et al., 1994; Texier et al., 1994), but also for the preservation of up to ~1 Ma old Acheulian artefacts (Raynal and Texier, 1989; Rhodes et al., 2006), and for containing rich faunal assemblages (Daujeard et al., 2012; Raynal et al., 1993) as well as Middle Pleistocene human fossils (Ennouchi, 1969; Raynal et al., 2010).

Each of the barrier systems reflect a cyclic deposition comprising underlying marine units covered by aeolian sediments and were affected by intensive post-depositional cementation (Fig. 1.5.5). The Oulad Hamida morpho-stratigraphic unit – within which the Thomas Quarries are situated - represents several major episodes of coastal sedimentation, fossilization and eventual cave development during the final Early and early Middle Pleistocene (Texier et al., 2002). The Hominid Cave (Fig. 1.5.6) at Thomas

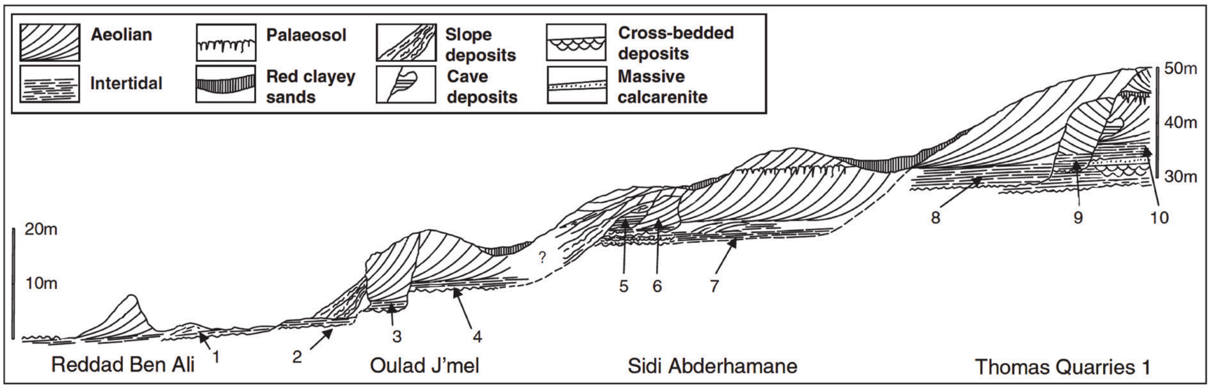

Fig. 1.5.5 Stratigraphic cross section showing the inferred relationships and relative altitudes above present day sea-level of the Quaternary sites Reddad Ben Ali, Oulad J'mel, Sidi Abderhamane and Thomas Quarries I after Texier et al. (2002) (modified after Rhodes et al., 2006). Marine units are numbered in reversed chronological order.

Fig. 1.5.6 Photograph of the Thomas Quarry I site with the location of the Locus I and the Hominid Cave.

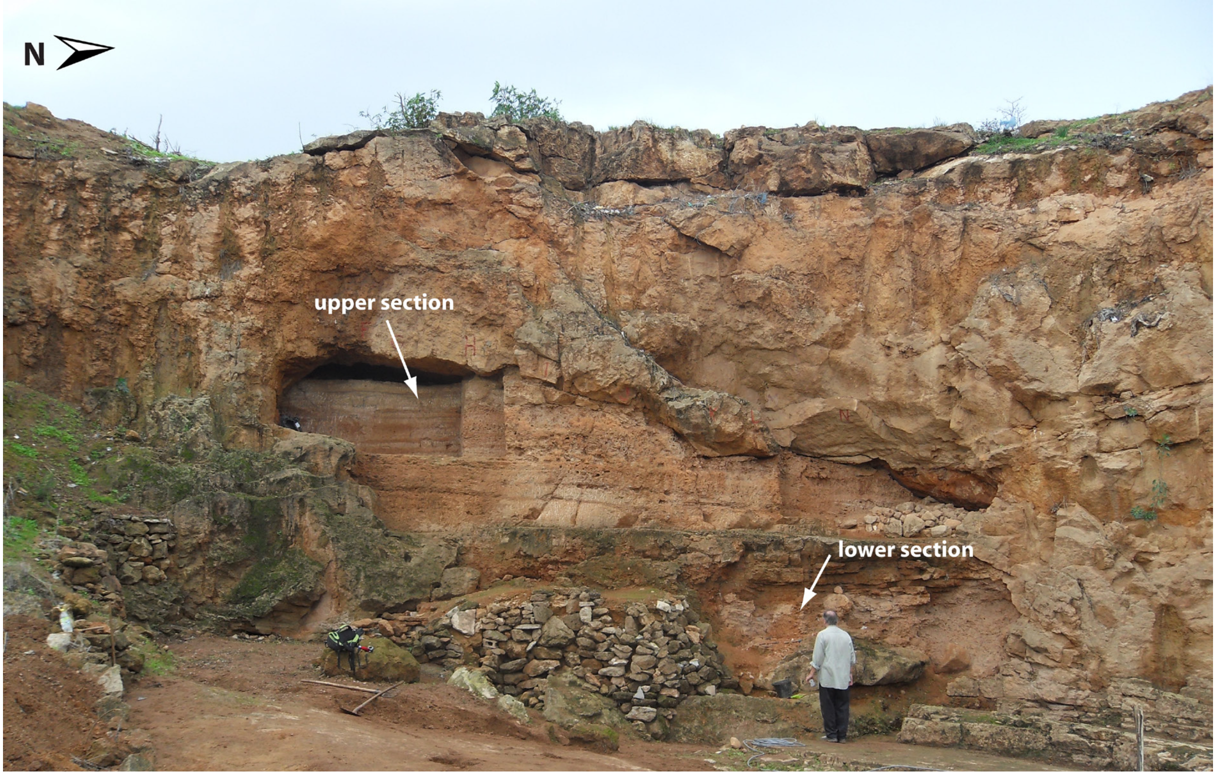

Fig. 1.5.7 Photograph of the Rhino Cave site with the location of the upper and lower cave sections.

Quarry I and the Rhino Cave (Fig. 1.5.7) at Oulad Hamida I, are cave sites filled with sediments dated to the Early and Middle Pleistocene based on litho- and biostratigraphy (Geraads, 2002) and absolute dating (between 360 and ~1 Ma using OSL, ESR, U-series and laser ablation ICP-MS) (Raynal et al., 2010; Rhodes et al., 1994; 2006). Despite the large variety of dating methods applied at the Thomas Quarries so far, sample numbers were small (ESR: four rhinoceros teeth, U-series: one speleothem age, laser ablation ICP-MS: one human premolar (Raynal et al., 2010; Rhodes et al., 1994; 2006)), and estimated absolute ages not always consistent with the previously established stratigraphical and lithological interpretation (OSL (Rhodes et al., 2006)).

1.5.3. Vanguard Cave, Gibraltar

The archaeological site of Vanguard Cave is part of a complex of limestone caves which are situated close to the present Mediterranean sea-level at the south-eastern coast of the Gibraltar promontory (Figs. 1.5.1, 1.5.8). Gibraltar is well known for its Neanderthal cave sites which preserve rich archaeological records, including human fossil remains (Busk, 1865; Garrod et al., 1928; Sollas, 1908), and provide evidence for Neanderthal habitation in the south-western extreme of the Iberian Peninsula throughout the Middle Pleistocene (e.g. Barton et al., 2013; Finlayson and Carrión, 2007; Jiménez-Espejo et al., 2013) and maybe even until 28 ka BP (Finlayson et al., 2006).

Fig. 1.5.8 Photographs of the Gorham's and Vanguard Cave sites located at the present shoreline of Gibraltar (modified after www.visitgibraltar.gi).

The Rock of Gibraltar is composed of Early Jurassic limestones and dolomites; the general shape of the promontory, however, was formed mainly as a consequence of the collision between the African and Eurasian tectonic plates in the early Miocene (Rose and Rosenbaum, 1994). In the course of this tectonic activity, the Mediterranean Sea was cut off from the Atlantic Ocean and gradually dried out during the Messinian salinity crisis (~5.6 Ma, Krijgsman et al., 1999). It was only at the beginning of the Pliocene, at 5.33 Ma, that Atlantic waters found a way through the Strait of Gibraltar and refilled the Mediterranean – an event known as the Zanclean flood (Blanc, 2002; Garcia-Castellanos et al., 2009).

Later, neotectonic uplift and eustatic fluctuations combined with surface erosional and depositional processes formed the shape of the Gibraltar promontory as it exists today with e.g. elevated marine terraces, steep cliffs and staircased slopes (Rodríguez-Vidal et al., 2004). During the Pleistocene, the east side of the Rock experienced substantial aeolian accumulation, with windblown sands filling considerable parts of the local caves (e.g. Vanguard, Gorham's and Ibex) which served as large sediment traps.

Marine highstands which are best developed to the south and the east of the Rock represent at least 12 palaeo-shorelines at heights up to 300 m above present-day sea-level (Rose and Rosenbaum, 1994). Raised terraces associated with the last interglacial – an episode which is well represented by several highstands along the southern Iberian littoral (Zazo et al., 2003; Zazo et al., 1999) - are located at 5 m (MIS 5c) and 2 - 1.5 m (MIS 5a) above sea-level at Gibraltar today (Zazo et al., 1994). No younger raised marine terraces are recorded along the Iberian coastline (Rodríguez-Vidal et al., 2004) as the sea-level significantly dropped after the end of MIS 5 until it reached a minimum (~125 m below present level, Fig. 1.1.1) during the last glacial maximum after which it raised again and reached its current height ~6.000 years ago (Miller et al., 2005).

Despite the great scientific interest on the archaeological content of the Gibraltar caves since the discovery of a Neanderthal cranium at Forbes' Quarry in 1848 (Busk, 1865), most studies in the past focussed almost exclusively on Gorham's Cave (Fig. 1.5.8, e.g. Blain et al., 2013; Carrión et al., 2008; Finlayson et al., 2006; Waechter, 1951, 1964) and it was only relatively recently (1995) that first excavations started at Vanguard Cave as part of the Gibraltar Caves Project (Barton et al., 2013; Stringer et al., 2000). Deposited on top of an MIS 5 marine terrace, the cave is filled with >17 m of sand-rich sediments which contain traces for multiple Palaeolithic occupation phases. Chronometric studies by Pettitt and Bailey (2000) and Rhodes (2013) used radiocarbon and OSL dating to provide age estimates for the Vanguard Cave profile. Unfortunately, both studies were limited by relatively small sample sizes and yielded conflicting dating results that placed the age of the uppermost stratigraphical layers to either ~45 ka (Pettitt and Bailey, 2000) or ~74 ka (Rhodes, 2013).

1.5.4. Comparative site: Lake Mungo, Australia

Lake Mungo is a presently dry lake in the Willandra Lakes Region World Heritage Area in the semi-arid zone of south-eastern Australia (Fig. 1.5.9). It preserves a unique archaeological and palaeoenvironmental record and is renowned for some of the earliest archaeological traces of AMHs on the Australian continent, including the world's oldest known cremation and ritual burial (Bowler et al., 2003; Bowler et al., 1970; Bowler and Thorne, 1976). In the past, the Willandra Lakes served as an overflow for the Willandra Creek which had its headwaters in the south-eastern Australian highlands (~1000 km to the east). During the last glacial cycle the lake lunettes experienced episodic sediment deposition, therefore providing important archives reflecting changes in lake palaeohydrologies and human activity for this time period in the area (Bowler, 1998; Stern, 2008).

Over the last decades, the Lake Mungo lunette had been subject to numerous archaeological and palaeoenvironmental studies, including absolute dating (e.g. Adams and Mortlock, 1974; Allen, 1998; Bowler et al., 2012; 2003; 1970; Fitzsimmons, 2017; Fitzsimmons et al., 2014; 2015; Olley et al., 2006). Fitzsimmons et al. (2014; 2015) published chronometric studies on the depositional history of the lunette (ca. 50–3 ka) and its archaeology based on single-grain OSL dating of sediment samples collected from the central portion of the landform. The sediments from the Lake Mungo lunette yield quartz-rich deposits well suited for OSL dating due to relatively high proportions of datable sand-sized grains which exhibit bright, rapidly decaying luminescence signals (Fitzsimmons et al., 2014). The high sensitisation of the quartz can be attributed to multiple cycles of exposure and burial within the sedimentary system before the eventual deposition at the Lake Mungo lunette (Fitzsimmons, 2011). Feldspar contamination of the quartz is low and although equivalent dose distributions for the younger, Holocene-age samples are comparatively wide - potentially related to dose rate heterogeneities within the sediments as has been reported by Lomax et al. (2007) for dune samples from the Murray-Darling Basin, south-eastern Australia - most of the age distributions are of Gaussian shape, indicating absence of post-depositional mixing in the stratigraphical layers and complete bleaching of the sediments during the last transportation process before burial (Fitzsimmons et al., 2014). The sediments from Lake Mungo, consequently, yield highly sensitised quartz well suited for OSL dating studies which have similarly been reported for various other sites from the Australian continent (e.g. Fitzsimmons et al., 2010; Pietsch et al., 2008; Roberts et al., 1999; Westaway, 2009).

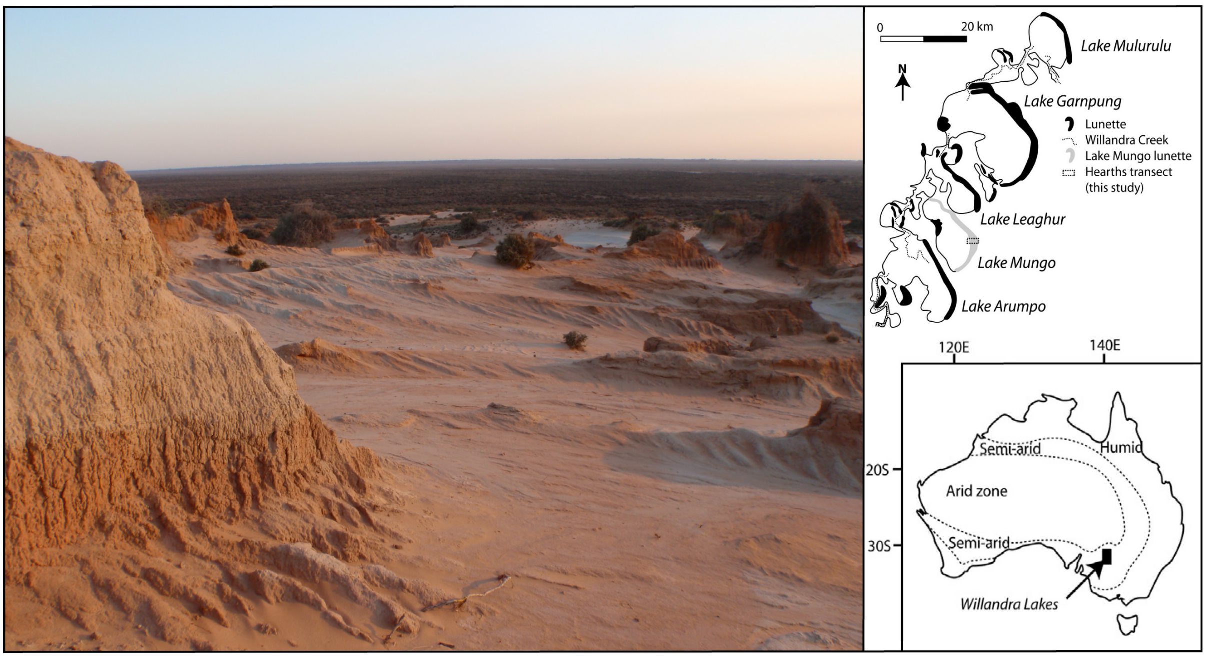

Fig. 1.5.9 Photograph and map of the Lake Mungo lunette within the Willandra Lakes Region in south-eastern Australia (modified after Fitzsimmons et al., 2014).

1.6. Optically Stimulated Luminescence dating

Luminescence is a remarkable, natural phenomenon that was already described as references to fireflies in Chinese literature about 3000 years ago (Harvey, 1957). While bioluminescence manifests in form of e.g. glow worms or luminous bacteria, more relevant to optical dating is the ‘cold light’ luminescence emitted by gems and stones which is known at least since Aristotle times (~2000 years ago) (Harvey, 1957). While the luminescence behaviour of plants, stones and animals was again described by Gesner (1555), first scientific studies on luminescence started not before the mid-seventeenth century when Robert Boyle investigated the encompassing luminescence behaviours of a ‘carbuncle’ – a diamond with the property to emit cold light; he even took the stone with him to bed, warmed it up with his body and described its elicited glimmering light (Boyle, 1664).

Scientific interest in luminescence grew after the advent of photomultiplier - a very sensitive detector of light – near the middle of the twentieth century (Aitken, 1998). In the following decades, increasing numbers of studies were performed eventually leading to the demonstration that the depositional age of sediment can be successfully determined by means of stimulation with visible light (Huntley et al., 1985). Since then, optical dating of quartz and feldspars (Hütt et al., 1988) became a widespread and powerful tool commonly used in archaeology and Quaternary geology, as it enables the reliable dating of the last sunlight contact of material - ubiquitous in nature – even if this event took place several hundreds of thousands of years ago.

1.6.1. General principles of OSL dating

Luminescence dating techniques are based on the property of naturally occurring minerals – such as quartz and feldspar - to emit measurable light signals (luminescence) when being stimulated by energy in form of either light or heat. While thermoluminescence (TL) dating determines the time since the last heating to 500°C, dating of sediments using optically stimulated luminescence (OSL) measures the time since mineral grains were last exposed to sunlight (Aitken, 1998).

This sunlight exposure – or bleaching event - happens in natural environments during erosion, transport and deposition of sediments. After burial, mineral grains store energy derived from environmental radiation - sourced from the decay of potassium (K), uranium (U) and thorium (Th) isotopes and their daughter products, and cosmic rays - within their crystal lattice (Aitken, 1998). Following the band model after Aitken (1985), electrons move short-term from the valence band to a higher energy level (conduction band) due to ionisation from the environmental radiation (Fig. 1.6.1). While most electrons drop back to the valence band, some become trapped and stored over thousands of years within the crystal lattice. The more prolonged the exposure to environmental radiation the greater the number of trapped electrons. The stability of each trap over time is indicated by its depth under the conduction band (Aitken, 1998). Electrons evict from traps in response to stimulation by heat or light (wavelength is specific depending on mineral and trap), diffuse and then recombine into recombination centres and in turn release energy – some of which is in form of light (Aitken, 1985, 1998).

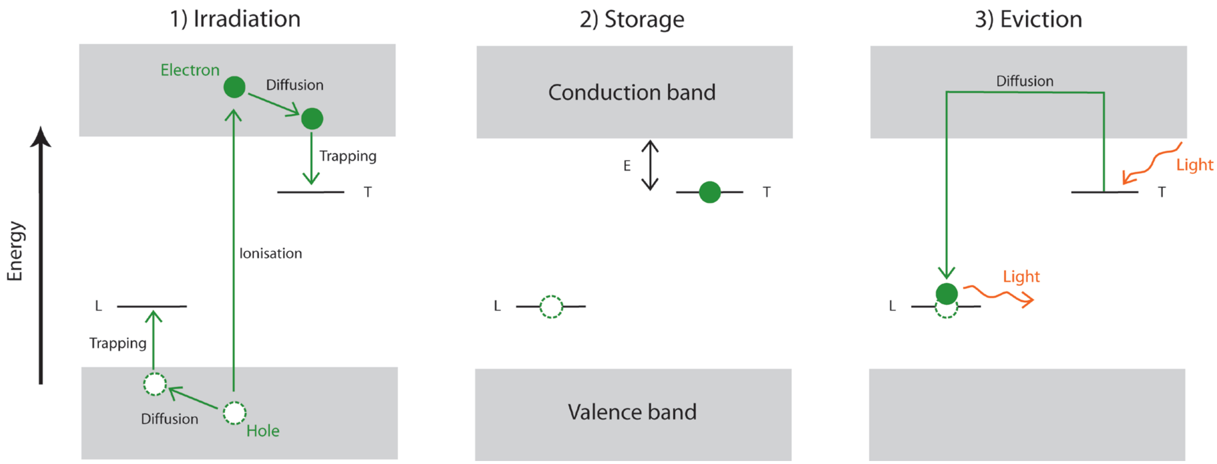

Fig. 1.6.1 OSL band model after Aitken (1985). (1) Ionisation due to exposure of the crystal to environmental radiation, with diffusion and trapping of electrons and holes at traps T and L, respectively; (2) Storage of electrons and holes in stable traps over time, the lifetime of electrons within the traps is determined by the depth E of the trap below the conduction band; (3) Eviction, and recombination of electrons with luminescence centres, L, and emission of luminescence signals in response to light stimulation. Alternatively, electrons may recombine at non-luminescent centres or deeper holes.

The number of electrons trapped in the crystal lattice of a mineral grain is proportional to the flux of environmental radiation (dose rate) it received per year since burial, until the traps approach saturation (a state where all suitable traps have become filled). Exposure to sunlight in a natural environment or intentional stimulation of the crystal by particular wavelengths of light release the stored energy in form of photons (i.e. luminescence) which can be detected by the photomultiplier in the laboratory and which is proportional to the amount of radiation the mineral received since burial. The burial age of a sediment sample can, therefore, be calculated as:

$$\text{Age (a)} = \frac{\text{Equivalent dose (Gy)}}{\text{Dose rate (Gy a}^{-1}\text{)}}$$

The equivalent dose (D_e) is defined as the laboratory radiation dose equivalent to that received in the natural environment since the last sunlight exposure, taking into account the conversion between the different energy efficiencies of environmental and laboratory induced radiation (Aitken, 1998). As age determination using OSL is limited by sample saturation, its upper dating limit in turn strongly depends on the intensity of the natural radiation in the sample surrounding.

1.6.2. Luminescence signal characteristics

Luminescence signals released in the laboratory by using intense light sources are often visually displayed as decay curves showing the intensity of the signal over stimulation time (Fig. 1.6.2a). Any luminescence signal, however, derives from multiple signal components (Bulur, 1996) - some of which might not be stable over geological times (Singarayer and Bailey, 2003) - depending on the electron traps in the crystal lattice of the mineral grains. The signal components can be differentiated into ultra-fast, fast, medium and slow (Fig. 1.6.2b) based on their length of time taken to respond to light stimulation (Bailey et al., 1997; Singarayer and Bailey, 2003). Not all components are similarly easy to bleach and each mineral grain must not exhibit every possible component (Bulur, 1996; Bulur et al., 2002). Linearly modulated (or LM) OSL measurements linearly ramp the intensity of the light stimulation source during measurement and, therefore, produce peak-shaped OSL instead of monotonically decaying OSL signals (Bulur, 1996). As the different traps contributing to the OSL signal appear as different peaks in the curve, LM-OSL measurements can be used to identify and illustrate different signal components in a given sample.

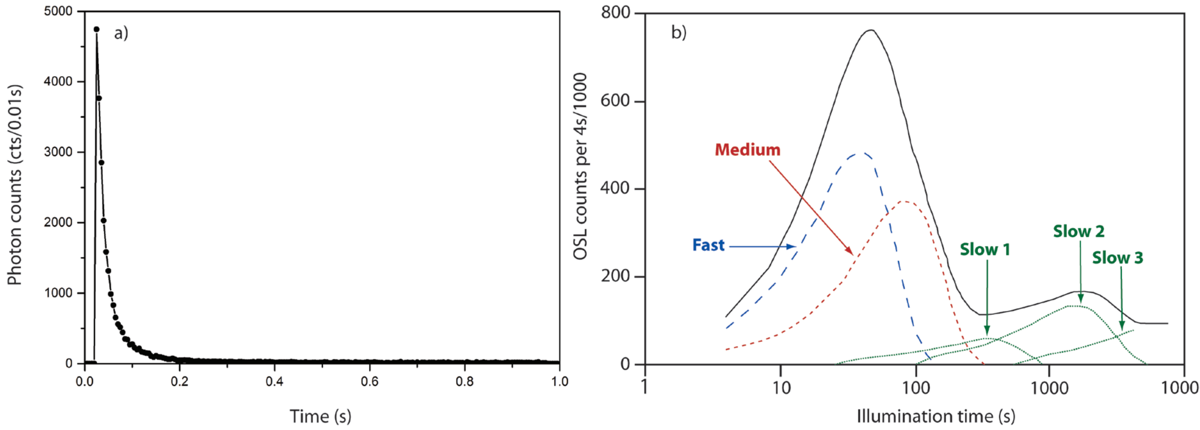

Fig. 1.6.2 (a) Schematic representation of a fast component dominated single-grain quartz luminescence signal decay curve, and (b) OSL signal component characterisation after ramped power (LM OSL) measurement (adapted from Singarayer and Bailey, 2003).

In this thesis, sand-sized quartz grains were used for age determination of the sediment samples. Quartz was chosen over feldspar for dating purposes, as it is i) ubiquitous in the studied areas, ii) more light sensitive and, therefore, readily bleachable under natural sunlight conditions (Wallinga, 2002), iii) known to produce rapidly decaying OSL signals (Aitken, 1998), iv) not suffering from anomalous fading (except volcanic quartz (Westaway, 2009)), a phenomenon which describes the loss of part of the luminescence signal with time, and v) has a less complicated internal dosimetry to account for (Huntley and Lamothe, 2001). Quartz has, furthermore, been demonstrated in previous studies to be suitable for OSL dating of Pleistocene sediments in both Morocco (e.g. Clark-Balzan et al., 2012; Jacobs et al., 2011; Rhodes et al., 2006) and Gibraltar (Barton et al., 2013).

LM-OSL measurements showed that all quartz samples in this thesis were dominated by the fast luminescence signal component (see i.e. chapter 4), which is known for being relatively easy to bleach. Light stimulations during measurements were kept under constant intensities and signals from the initial seconds of response were used for D_e determination.

1.6.3. Sample collection and preparation

Due to the light-sensitive nature of the luminescence signal stored in sediment grains, it is of critical importance to avoid any sunlight exposure of the samples before OSL measurement in the laboratory (Aitken, 1998). On this account, OSL sampling in the field was conducted – depending on the degree of cementation of the layers - by i) hammering stainless steel tubes horizontally into the freshly cleaned profile walls, or ii) collecting block samples using either hammer and chisel or a drill. Sampling tubes and block samples were quickly capped with light-proof plastic caps and covered in black, light-proof plastic bags, respectively. Sediments from the direct surrounding of the OSL sample holes were collected for subsequent determination of their radioactive element concentration in the laboratory. All samples were carefully sealed to preserve the in situ field moisture content.

In the laboratory, samples were opened and processed (Table 1.6.1) under subdued red light conditions (Fig. 1.6.3). To exclude any sediment grains from the dating process which might have been exposed to sunlight during sampling, material from both ends (1-2 cm) of each sampling tube was removed. Similarly, the outer surfaces (~1 cm) of the block samples were cut off using a circular table saw equipped with a diamond saw blade.

Table 1.6.1 Protocol for coarse-grain quartz processing in the laboratory.

Step	Treatment
1	Sample drying at 50°C
2	Wet and dry sieving to recover sand fraction (90-212 µm or 90-300 µm)
3	HCl (10%) etching to remove carbonates
4	Hydrogen peroxide (30%) wash to remove organic matter
5	Sample drying at 50°C
6	Density separation heavy minerals (Lithium heterotungstate density 2.68 g cm^{-3})
7	Sample drying at 50°C
8	Density separation feldspar (Lithium heterotungstate density 2.62 g cm^{-3})
9	Sample drying at 50°C
10	HF (40%) etching for 60 min and rinsing with HCl and purified water
11	Sample drying at 50°C
12	Dry sieving to recover the 180-212 µm sand fraction

The remaining material was weighted and subsequently dried in an oven at 50°C for calculation of the field moisture content. Coarse-grain sand (usually 90-212 µm; for some samples 90-300 µm, when only little amount of material of the desired sand fraction was available) was extracted from the sediment by a combination of wet and dry sieving. Due to intensive cementation, block samples were treated with hydrochloric acid (HCl, 10%) first to dissolve carbonates before any sieving was possible (Wintle, 1997). The isolated sand-fractions of each sample were then used for further chemical treatments, and eventual for D_e determination.

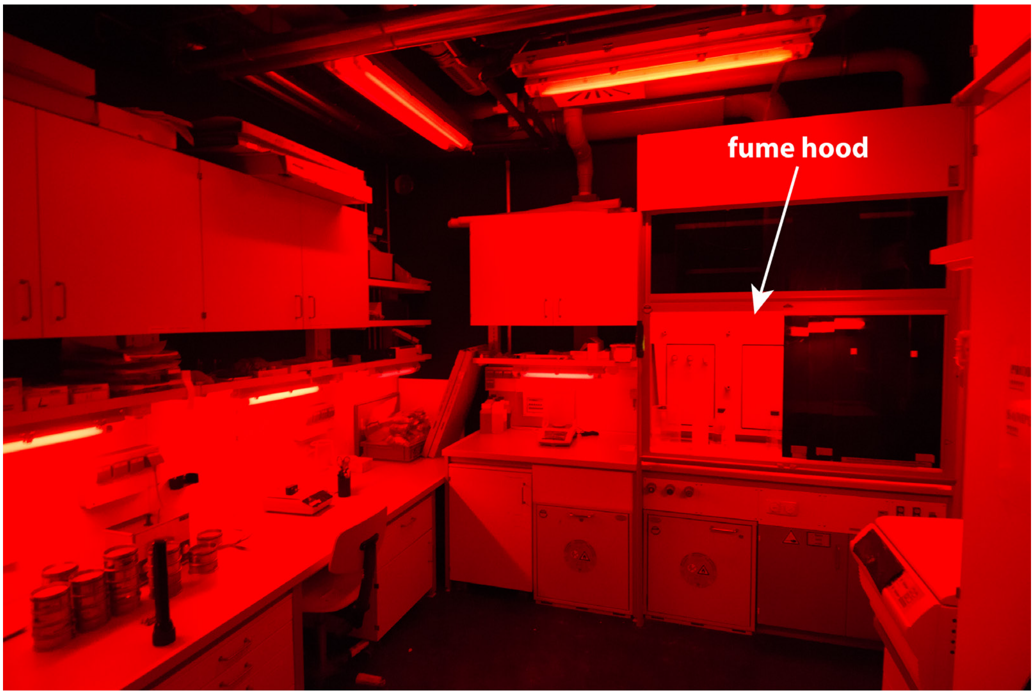

Fig. 1.6.3 The sample preparation room in the luminescence laboratory at the Max Planck Institute for Evolutionary Anthropology, Leipzig, Germany.

The material was washed with dilute HCl (10%) and hydrogen peroxide (H_2O_2, 30%) to remove carbonates and organic matter, respectively (Galbraith et al., 1999). Lithium heterotungstate, prepared to densities of 2.68 g cm^{-3} and 2.62 g cm^{-3} was used to separate quartz grains from heavy minerals and lighter feldspar grains (Aitken, 1998; Wintle, 1997). The extracted quartz was then etched with concentrated hydrofluoric acid (HF, 40%) for 60 min (Wintle, 1997) in order to remove i) the outer surface of the grains (which is affected by α radiation, chapter 1.6.9), and ii) any potentially - after density separation - remaining feldspar minerals (a chemical removal in addition to the density separation step is necessary as feldspar constitutes a solid solution spectrum of minerals with highly variable densities), which store comparatively bright luminescence signals that might otherwise significantly alter quartz OSL measurements. After etching, samples were rinsed first in HCl and subsequently multiple times in purified water to remove fluoride salts and then dried at 50°C. Laboratory processing of the samples was completed by re-sieving of the extracted quartz to recover the grain-size fraction desired for D_e determination (180-212 µm).

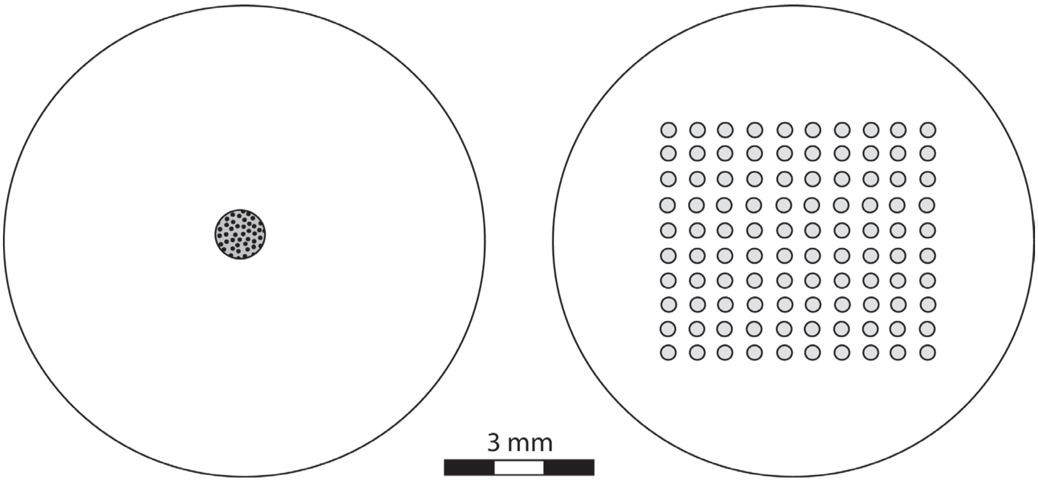

Fig. 1.6.4 Schematic representation of a 1 mm multiple-grain aliquot disc (containing ~30 individual sand grains) and a single-grain disc containing 100 holes (each being 300 µm wide and 300 µm deep).

Multiple-grain and single-grain dating techniques were used for D_e measurement. For preparation of the multiple-grain aliquots, quartz grains were mounted on stainless steel discs using silicon oil and a mask of 1 mm. Single-grain discs were loaded by sweeping individual grains over aluminium discs – containing 100 holes (each 300 µm wide and 300 µm deep) in a 10 by 10 array (Duller et al., 1999) – with a small brush (Fig. 1.6.4).

1.6.4. Multiple-grain vs. single-grain dating

Multiple-grain aliquots contain small subsamples of the desired material to be dated (Duller, 2004; Duller, 2008), and each aliquot provides an independent estimate of D_e. After measurements of many separate aliquots the D_e distribution within a sample can be assessed (Duller, 2008). The fundamental assumption behind this approach is that all sediment grains of a given sample were exposed to sufficient sunlight – as is typical for aeolian sediments - before burial to remove any trapped and stored electron charge. Godfrey-Smith et al. (1988) showed that for quartz an almost complete signal resetting (reduction of the natural unbleached optical signal to 1%) can already be achieved after 10 s of direct sunlight exposure on a clear day and even on an overcast day, samples bleach only 10 times more slowly. Given a complete signal resetting before burial, all grains would have a zero D_e at deposition and - within a homogeneous radiation environment - accumulate the same amount of charge over time. As multiple-grain aliquots usually contain between tens and about a million of individual grains (Duller, 2008) - depending on the grain-size of the measured sediment fraction and the size of the aliquot – the measured D_es represent aggregate, averaged luminescence signals (Rhodes, 2007).

Multiple-grain OSL measurements were performed in this thesis using automated Risø OSL/TL readers (DA-15 and DA-20, Fig. 1.6.5) each equipped with calibrated ^{90}Sr/^{90}Y beta sources for radiation dosing (Bøtter-Jensen et al., 2000). Stimulation light was provided by blue light-emitting diodes (470 nm wavelength) and infrared diodes (875 nm). The emitted luminescence signal was filtered by 7.5 mm Hoya U-340 detection filters (Bøtter-Jensen, 1997) before being converted from photons to an electric signal within EMI photomultiplier tubes (Aitken, 1998).

Over the last years, OSL dating of individual sand-sized quartz grains (Bøtter-Jensen et al., 2000; Duller et al., 1999; Roberts et al., 1999) has become a frequently used tool especially in archaeological contexts (e.g. Demuro et al., 2012; Fitzsimmons et al., 2014; Jacobs and Roberts, 2007; Jacobs et al., 2012; Roberts et al., 1998; Tribolo et al., 2010). For this technique Risø readers (Fig. 1.6.5) have to be equipped with a single-grain attachment (Bøtter-Jensen et al., 2003); the D_e of individual grains get measured by light stimulation from a green laser emitting at 532 nm (Bøtter-Jensen et al., 2000). Single-grain luminescence signals are often of low intensity and, therefore, more difficult to measure than multiple-grain aliquots, and as not all grains are necessarily suitable for luminescence dating (some do not exhibit luminescence signals at all (Jacobs et al., 2003, 2006; Porat et al., 2006)) (Fig. 1.6.6), this technique is rather elaborate and time-consuming (Duller, 2008). It, however, enables the identification of multiple age populations within a sediment sample, which is difficult to assess in multiple-grain dating where an averaged luminescence signal from all grains placed on a disc is recorded. Complete bleaching during the last transportation process of the sediment grains cannot always be assumed with sufficient certainty when it comes to e.g. caves (Feathers, 2002; Murray et al., 2012). Furthermore, archaeological sites are often affected by post-depositional mixing of sediments by natural processes and/or human activity (Bateman et al., 2007). In those cases single-grain dating can still provide reliable depositional ages for a sediment layer and, additionally, quantify the amount of older or younger grains in the sample, which might help to understand local site formation processes. Additional problems for OSL dating may arise from the variety of materials in the surrounding of a sediment sample each providing different dose rates (Olley et al., 1997), which often occurs in archaeological sites (e.g. Steele et al., 2016). Poor sediment sorting and high variability in grain sizes in a sediment layer can, furthermore, result in local radioactive 'hotspots' and 'coldspots' which effect dose rates at an individual grain level. Those inconsistencies in beta dose rates can be identified and visualised using single-grain dating (Jacobs et al., 2011; Jacobs and Roberts, 2007).

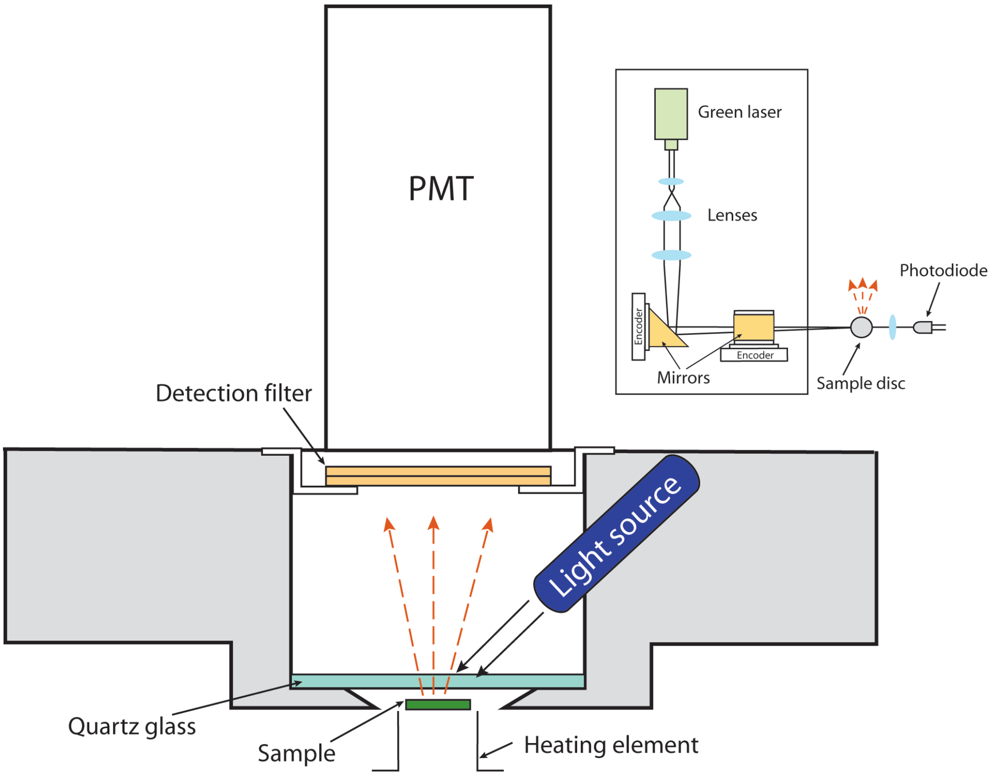

Fig. 1.6.5 Schematic representation of a Risø OSL/TL reader and a single-grain OSL unit adapted from Bøtter-Jensen et al. (2000). Optical stimulation is provided by a light source (blue LEDs, IR diodes or green laser hosted in a separate attachment) directly onto the sample which in turn emits a luminescence signal that passes through detection filters (and a quartz glass when measuring individual grains) into the photomultiplier tube (PMT). The PMT produces an output of photon counts against time (see Fig. 1.6.2a).

Since single-grain dating was first introduced to the scientific community by Galbraith et al. (1999) and Roberts et al. (1999), researchers have performed comparative studies – using both multiple-grain and single-grain dating approaches - to understand the variability observed in luminescence characteristics of sediment samples when being measured with differently sized aliquots and/or single-grains (e.g. Arnold et al., 2012; Duller, 2008; Rhodes, 2007). Rhodes (2007) showed that the brightness of OSL signals might vary considerably between grains within one sample, but also that the proportion of grains yielding detectable signals at all is highly variable between samples. Consequently, the commonly assumed averaging effect in multiple-grain aliquots can be reduced for samples which yield OSL signals dominated by just a few bright grains (Duller, 2008; Rhodes, 2007). And although, single-grain dating is often successfully applied at sites with complicated stratigraphical contexts (e.g. Jacobs et al., 2012; Roberts et al., 2000; Tribolo et al., 2010), researchers have also reported case studies for which single-grain dating was unable to overcome such issues (Guérin et al., 2012; Steele et al., 2016) or when multiple-grain results were scientifically more conclusive than those determined using single-grains (Carr et al., 2007; Guhl et al., 2013).

In this thesis, single-grain dating was used for age determination of sediments from the Moroccan sites (Rhafas and Casablanca), while at Gibraltar (Vanguard Cave) a comparative dual chronology was developed using both methodological approaches (multiple- and single-grain dating).

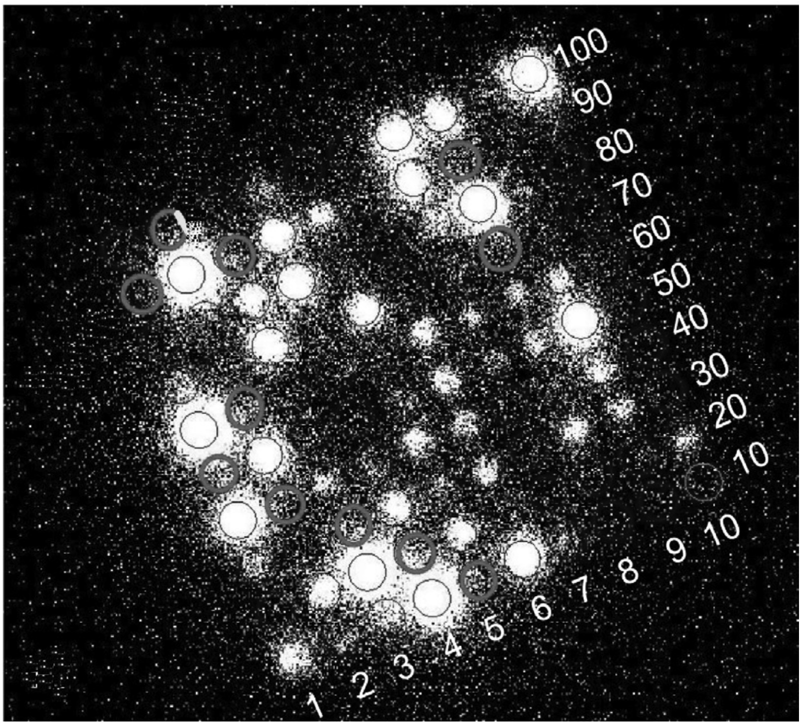

Fig. 1.6.6 EMCCD (electron multiplying charge-coupled device) image for a single-grain disc showing the luminescence signal released by each of the 100 individual grains (modified after Thomsen et al., 2015).

1.6.5. Single-aliquot regenerative-dose protocol for D_e determination

The most frequently applied protocol for optical dating of sediments, today, is the single-aliquot regenerative-dose protocol (SAR) after Murray and Wintle (2000, 2003), which was also used in this thesis for D_e determinations. Laboratory based OSL measurements generally include multiple cycles of light stimulation, heating and radiation dosing, which can cause sensitivity change (unwanted charge transfer to optically sensitive traps) in quartz minerals, resulting in inconsistent responses to light stimulation and radiation dosing and eventually problems for D_e determination. To account for this, the SAR protocol includes correction for sensitivity change by measuring small test doses in between each dose step during the protocol run (Table 1.6.2, Murray and Wintle, 2000; 2003).

Table 1.6.2 Summary of the single-grain SAR protocol used in this thesis.

Run	Treatment	Description
1	Dose (except before first run)	Radiation dose
2	Preheat (PH_1)[a] for 10s	Empties thermally unstable traps
3	Optical stimulation with IR diodes for 100s at 20°C (only for last run)	Quantifies feldspar contamination
4	Optical stimulation with green laser for 1s at 125°C	L_x[b]
5	Test dose	Allows sensitivity change correction
6	Preheat (PH_2)[a] for 10s	Empties thermally unstable traps
7	Optical stimulation with green laser for 1s at 125°C	T_x[c]
8	Start from top	

[a] PH_1/PH_2 was 240°C/200°C or 260°C/220°C; depending on the sample-specific response to preheat plateau and dose recovery preheat plateau tests (section 1.5.6).

[b] L_x is the OSL signal.

[c] T_x is the OSL response to the test dose.

The SAR protocol measures OSL signals (L_x and T_x, Table 1.6.2) derived from natural and laboratory radiation dosing in multiple-grain aliquots and single-grains. After measurement of the natural dose (N) stored in the samples, four regenerative dose cycles (R_1-R_4) with progressively increasing given doses were performed to build up reliable dose response curves (Fig. 1.6.7). The D_e of a sample is then calculated by interpolating N on the laboratory generated dose response curve. Preheats before each optical stimulation were incorporated to remove charge from thermally unstable traps that have been filled during laboratory irradiation and which may otherwise falsify the result by contributing to the recorded OSL signal (Murray and Wintle, 2000, 2003). A zero dose step (also called recuperation, R_0) – which should ideally give zero signal - was measured to assess the effect of charge transfer from deeper traps on the OSL signal caused by preheating, irradiation and optical stimulation during the previous dose cycles (Wintle and Murray, 2006).

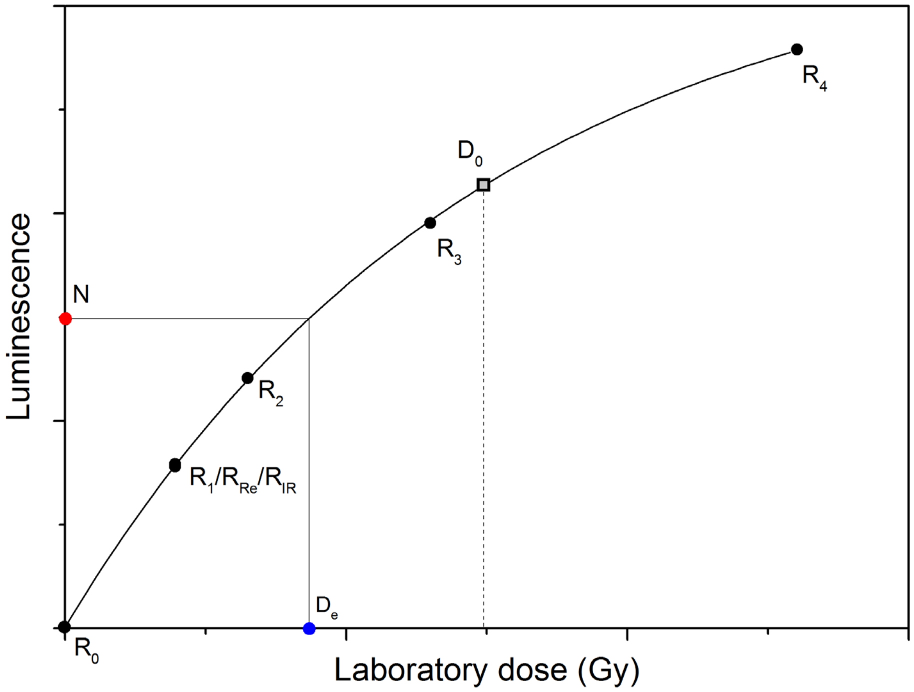

Fig. 1.6.7 Schematic representation of a sensitivity corrected SAR dose-response curve for D_e determination of a single-grain or multiple-grain aliquot with a single saturating exponential curve fit. The dose-response curve is constructed from the regeneration dose points R_1-R_4. R_0 is the OSL response to a zero dose (recuperation), R_{Re} is the recycling point which repeats R_1 at the end of the protocol and R_{IR} represents the IR depletion ratio measurement following Duller (2003). The D_e of a sample is calculated by interpolating the natural dose signal (N) of a sample on the laboratory generated dose response curve. The D_0 value characterises the rate of OSL signal saturation.

Usually sensitivity change progressively increases with each dose step in OSL protocols, which is why one of the already measured dose points is usually repeated towards the end of the protocol to check whether sensitivity change was correctly accounted for (recycling point, R_{Re}) (Murray and Wintle, 2000). The recycling point was chosen to repeat R_1 in this thesis and both values should give the same OSL signal assuming that the SAR protocol is successfully correcting for any sensitivity change in the samples. The recycling ratio is calculated according to Murray and Wintle (2000) as R_{Re} divided by R_1.

The purity of a quartz sample after chemical treatment and its potential contamination by feldspar can be examined by measuring the IR-depletion ratio after Duller (2003) in the end of the SAR protocol, which allows efficient distinction between quartz and feldspar based solely on their luminescence behaviour. The IR-depletion point (R_{IR}) is measured similarly to the recycling point, by repeating the first regenerative dose cycle. Prior to optical stimulation, however, the sample is additionally exposed to infrared light stimulation (Table 1.6.2). As quartz OSL traps are not sensitive to IR light stimulation, while feldspar traps are, the calculated IR-depletion ratio enables quantification of quartz OSL signal depletion caused by IR stimulation, or, in other words, quantification of contamination of the quartz sample by feldspar (Duller, 2003).

Age determination in OSL dating is limited by the sample-dependent signal saturation level ($2D_0$), a status which is approached when the majority of existing electron traps in the crystal lattice of a mineral are filled. OSL growth curves will reach a stable plateau when given radiation doses exceed sample saturation level.

The reliability of each measured D_e value can be assessed by testing its response to the previously outlined quality criteria (test dose, recuperation, recycling, IR-depletion, signal saturation), but also by the ability of its natural signal to be interpolated on the dose response curve and the size of the D_e error. As luminescence characteristics of quartz samples can be highly variable, quality criteria were defined individually for each site in this thesis and are discussed in greater detail in the corresponding chapters 2-5.

1.6.6. SAR performance tests

Thermal treatments (preheats) are essential to any SAR protocol (Table 1.6.2) as they empty light-sensitive shallow traps, particularly those filled by laboratory irradiations, prior to optical stimulation and measurement of the stored luminescence signal (Murray and Wintle, 2000, 2003). Preheats, however, may also cause sensitivity change resulting in erroneously high luminescence signals and, subsequently, miscounted D_es (Wintle and Murray, 2006). A thorough testing of the most appropriate thermal treatment in a SAR protocol is, therefore, required for each sample prior to D_e determination.

In this thesis, standard preheat plateau and combined dose recovery preheat plateau tests were performed on each sample. For both tests seven different temperatures - varying between 160°C and 280°C in 20°C steps – for the 10 s preheat ahead of the main OSL measurement (L_X) were tested with a fixed low temperature test dose cutheat at 160°C (Wallinga, 2002; Wintle and Murray, 2006).

For the preheat plateau test, natural D_es are measured and the ability of the test dose signal (T_X) to monitor sensitivity change should be shown as an absence of D_e dependence on the preheat temperature (Wintle and Murray, 2006). In other words, if determined D_es reach a stable plateau value independent of the applied preheat temperature then sensitivity change is correctly accounted for in the respective SAR protocols, as shown for preheat temperatures between 180°C and 260°C in the hypothetical example of a preheat plateau test in Fig. 1.6.8a. The chosen preheat temperature for the final D_e measurements should, consequently, be selected from the plateau region of the preheat plateau test (Wintle and Murray, 2006).

As the most distinct sensitivity change during SAR measurements usually occur when a sample is first heated, dose recovery tests on unheated sample material can be carried out to check whether the first sensitivity measurement (T_N) is appropriate to the preceding natural signal (L_N) (Murray and Wintle, 2003; Roberts et al., 1999). For this test, the unheated, laboratory bleached or modern analogous (with zero D_e) sample is given a known radiation dose (close to the expected natural D_e of the sample) and the SAR protocol is then run. Since a known dose is given, the ability of the protocol to accurately measure this dose can be directly tested and is mathematically expressed as ratio of measured over given dose (Fig. 1.6.8b).

Ideally, dose recovery experiments should mimic the processes of bleaching and radiation dosing in nature. This is, however, an impossible task as i) artificial bleaching sources are different to natural sunlight i.e. with respect to wavelength and light intensity, and ii) irradiation in the laboratory is administered by strong beta or gamma sources within seconds, while radiation dosing occurs slowly over millennia due to the decay of radioactive elements in natural sedimentary deposits. Researchers have performed intensive experiments in the past years using beta and gamma radiation and different kinds of bleaching sources (natural sunlight, solar simulator, blue LEDs, green laser) to test the reliability of dose recovery test results under varying experimental settings (e.g. Choi et al., 2009; Thomsen et al., 2012; 2016; Wang et al., 2011) and chapter 4 of this thesis contributes to this discussion by examining single-grain dose recovery characteristics of Moroccan and Australian samples.

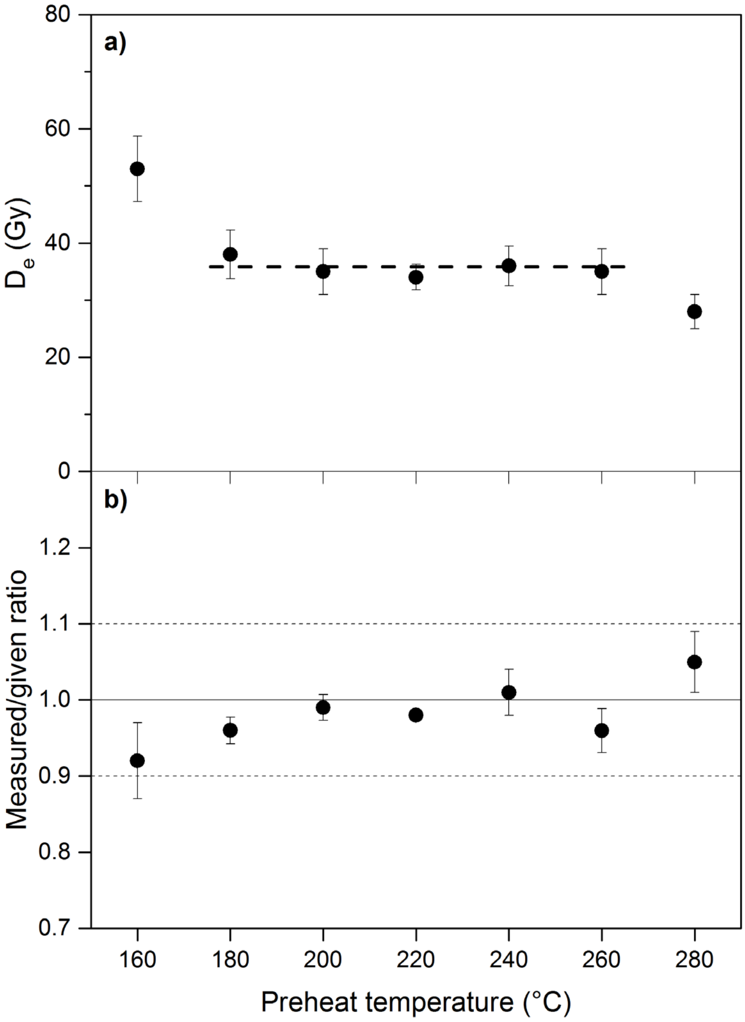

Fig. 1.6.8 Hypothetical example of a (a) preheat plateau test, and (b) dose recovery preheat plateau test as a function of preheat temperature. (a) Determined D_e are independent of the preheat temperature (preheat plateau) at 180-260°C. (b) Calculated measured/given dose ratios describe the ability of a sample to recover a laboratory given dose within acceptable ranges (2-sigma of unity).

For this thesis, standard dose recovery test are combined with a preheat plateau test to assess the ability of the respective SAR protocol to reproduce a known laboratory dose depending on a chosen preheat temperature (Fig. 1.6.8). Based on the results of the preheat plateau tests and dose recovery preheat plateau tests of each individual sample, preheat temperatures were selected for final D_e measurements.

1.6.7. Presentation of data

Radial plots – which were initially proposed by Galbraith (1988, 1990) for data presentation in fission track dating - are often used in OSL dating studies to graphically display D_e distributions of single-grains and multiple-grain aliquots (Fig. 1.6.9). Luminescence intensities of single-grains and consequently also the precision of their calculated D_es may vary greatly, which is why e.g. histograms are likely to be uninformative or even misleading for the interpretation of D_e distributions (Galbraith et al., 1999). Each data point in a radial plot represents a single grain/multiple-grain aliquot and its measured D_e can be read by tracing a line from the y-axis origin through the point until the line intersects the radial plot axis (log scale). The standard error (in %) and the precision (reciprocal standard error) of each D_e value can be read by extending a line vertically to intersect the x-axis (Olley et al., 2004). D_e values with the highest precision (and the smallest relative standard error) fall furthest to the right, whereas those measured with least precision lie furthest to the left. The y-axis extends only from -2 to +2 thereby effectively displaying the length of a 2-sigma error bar applicable to any point (Galbraith et al., 1999). The shaded region of the plot indicates those D_e values that are consistent (at 1-sigma) with the weighted mean of a discrete age population.

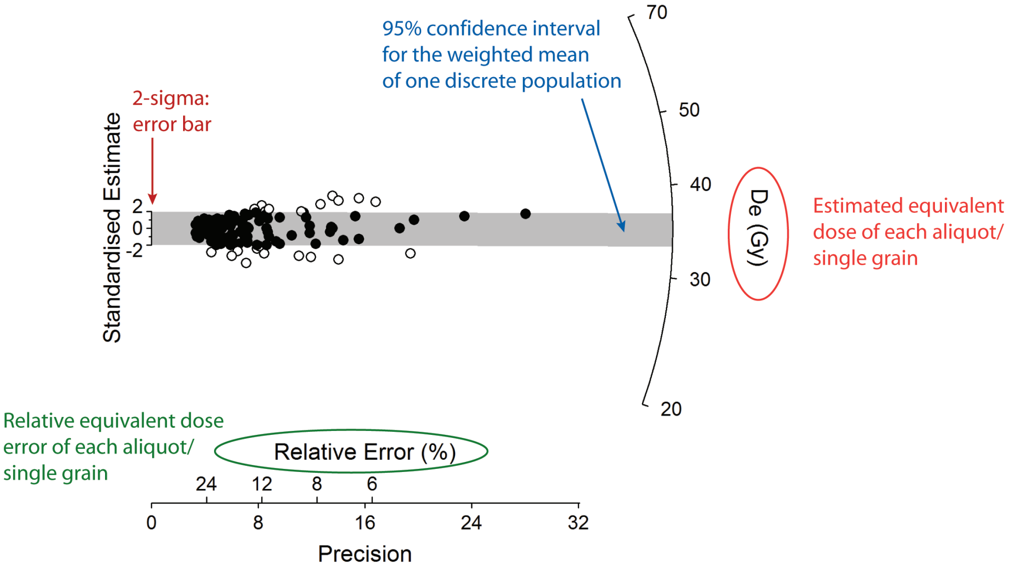

Fig. 1.6.9 Illustration of a radial plot with a hypothetical D_e distribution. The graphical display allows visual assignment of the measured D_e and relative standard error for each single-grain or multiple-grain aliquot, while at the same time visualises potential discrete D_e populations.

1.6.8. Analysis of data: age models for D_e determination

D_e distributions of sediment samples may i) contain multiple age populations (due to i.e. post-depositional mixing, roof spall in caves or incomplete bleaching), ii) show extremely wide scattering (due to heterogeneous dose rates), or iii) simply display an ideal image of a single age population. In the first two cases, single-grain dating should be favoured over multiple-grain dating approaches as it allows not only to identify and quantify those multiple age populations in a sample, but also to calculate ages separately for each of these populations.

The most commonly used statistical model to calculate the age of a single homogeneous D_e distribution (Fig. 1.6.10a) in the past years, was the Central Age Model (CAM) of Galbraith et al. (1999), which calculates a weighted geometric mean of individual D_es and gives an estimate for the overdispersion of the D_e distribution. The overdispersion is defined as the scatter beyond measurement uncertainties and allows quantification of the variability in D_e distributions (Galbraith et al., 1999). It comprises both extrinsic and intrinsic factors; the former of which can be caused by dose rate heterogeneity, incomplete bleaching and/or post-depositional mixing, while the latter arises from thermal transfer, instrument reproducibility, counting statistics or other sample-specific OSL characteristics (Thomsen et al., 2007). While researchers have recently started discussing the application of alternative mean age models such as the Bayesian central-dose model or the calculation of unweighted arithmetic means (e.g. Combès et al., 2015; Guérin et al., 2016; Thomsen et al., 2016), in this thesis, the CAM was used when D_e distributions were characterised by a single homogeneous dose population.

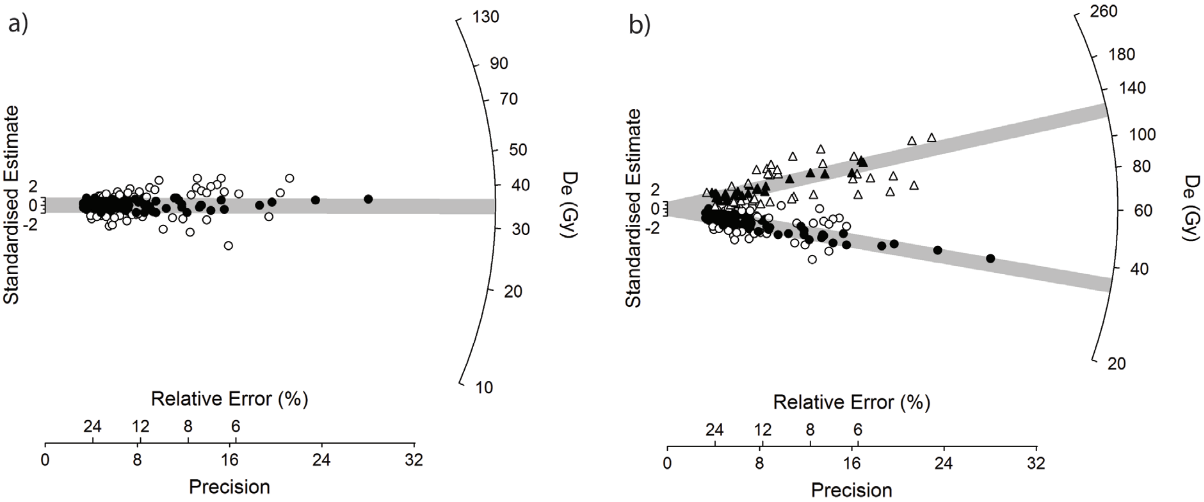

Fig. 1.6.10 Radial plots showing hypothetical examples of D_e distributions comprising (a) one discrete, and (b) two discrete age populations. For the purposes of a simplified illustration, D_es of individual grains in (b) are displayed as dots or triangles depending on the age population they are assigned to.

For samples in this thesi characterised by mixed single-grain D_e distributions (Fig. 1.6.10b), the number of discrete D_e populations (components), their relative proportion and their respective weighted mean age was determined using the Finite Mixture Model (FMM) after Galbraith et al. (1999) and Roberts et al. (2000). In the FMM, it is assumed that the log equivalent doses for single grains represent a mixture of discrete, normally distributed populations, each of which has the same relative overdispersion. The model can be run by inserting values for the number of fitted components and the overdispersion; it then uses maximum likelihood to estimate the mean D_es, their standard errors and the proportion of grains for each component (Roberts and Jacobs, 2015).

1.6.9. Dose rate determination

Equally important to the D_e determination in OSL dating is the correct assessment of the dose rate received by the sediment grains per year, as the rate at which trapped electrons are accumulated is proportional to the energy absorbed by the mineral grains from the surrounding radiation flux since its last sunlight exposure (Aitken, 1998). The annual ionising radiation arising from i) the radioactive decay of K, Th and U in the sediments, and ii) cosmic rays contribute to the total dose rate (Fig. 1.6.11) which can only be measured under present day conditions. The fundamental assumption underlying OSL dating studies, therefore, is that the total dose rate for each sediment sample remained constant with time or in other words that the present-day dose rate is the same as that in the past. Large uncertainties are incorporated into dose rate calculations to account for potential small variations in the radiation flux over time (Aitken, 1998).

Naturally occurring radiation in sedimentary bodies is mostly a result of the radioactive decay of ^{40}K - emitting β particles and γ rays - and uranium (^{238}U and ^{235}U) and ^{232}Th which both produce α and β particles as well as γ rays (Fig. 1.6.11). There is also a minor contribution from the decay of rubidium which is usually considered negligible due to its strongly absorbable, low energy β particles (Aitken, 1985, 1998).

α particle are characterised by relatively limited travel ranges within sediment samples (~20 µm, Guérin, 2015), penetrating only the outer rinds of sand-sized quartz grains (Fig. 1.6.11c). HF etching applied during chemical treatment of the samples in the laboratory removes these outer rinds of the quartz grains (section 1.6.3) and, therefore, α contribution to the total dose rate is rendered negligible. In contrast, travel ranges of β and γ particles in sedimentary bodies are 2-3 mm and up to 40-50 cm, respectively (Aitken, 1998; Guérin, 2015).

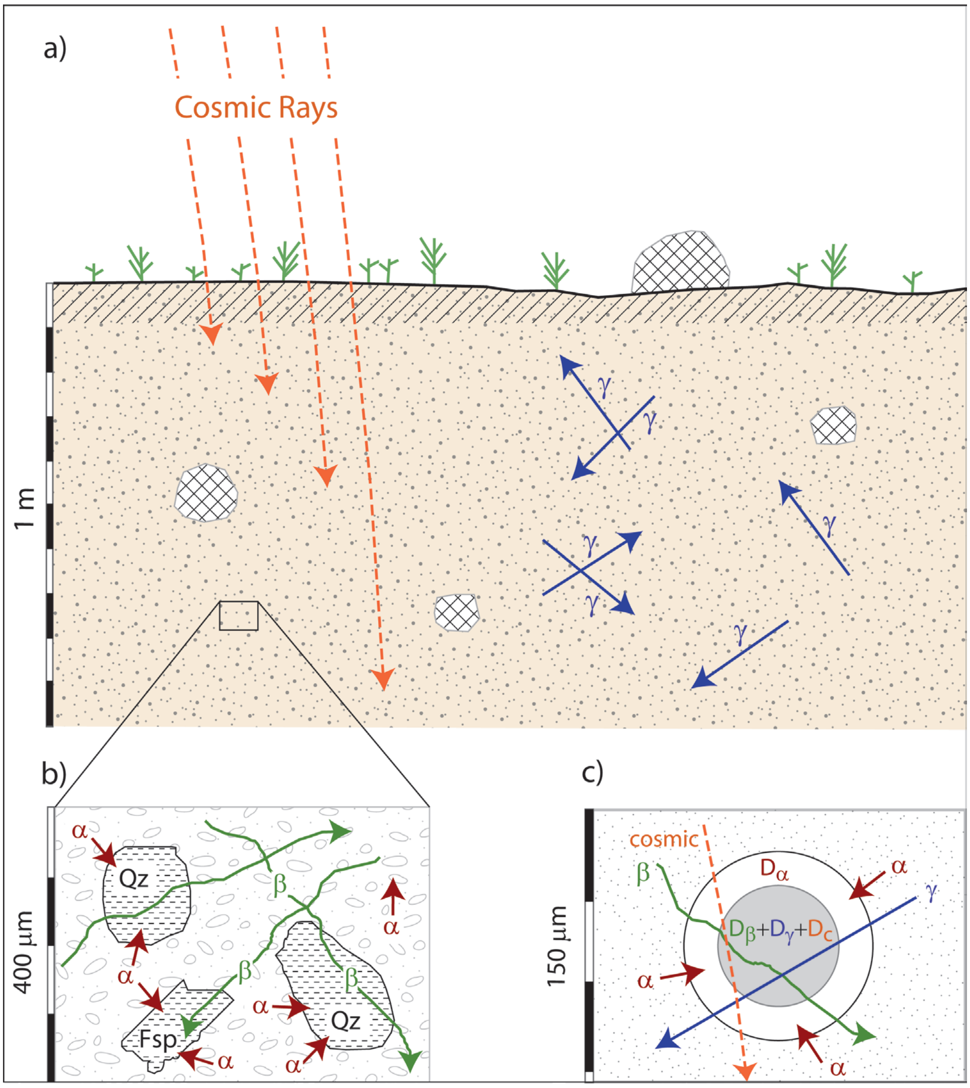

Fig. 1.6.11 Schematic representation of the sources of environmental radiation affecting individual sediment grains on different resolution levels (from low (a) to high (c)) adapted from S. Stokes in Aitken (1998) and Fleming (1970). Grains are irradiated by cosmic rays from outer space as well as alpha particles, beta particles and gamma rays originating from the radioactive decay of naturally occurring K, Th and U in the sediment. Alpha particles travel only short distances (<20 µm) compared to beta particles (2-3 mm), gamma rays (up to 40-50 cm) and cosmic rays, which is why they only penetrate the outer rind of sand-sized grains (b,c).

Sources of uncertainty in sediment dose rate determination may arise from attenuation of β particles by moisture and/or secular disequilibrium in the uranium- or thorium-series decay chain (Olley et al., 1996). Water present in the pore spaces of sediments absorbs part of the radiation that would otherwise reach individual grains (Mejdahl, 1979). Consequently, the dose rate of moist sediment is lower compared to in the same, dry material (Aitken, 1998). In this thesis, in situ and full-saturation moisture contents were taken into account to allow estimation of reliable average burial-time moisture contents for each individual OSL sample.

In radioactive decay chains, a secular equilibrium represents the condition in which activities of daughter isotopes are equal to those of the parent. Disequilibrium, hence, indicates an imbalance between daughter and parent isotope, either due to introduction of new material from an allochthonous source, or by escape or removal of the daughter isotope (e.g. gaseous escape, leaching in groundwater, absorption) (Aitken, 1985, 1998; Krbetschek et al., 1994). While the thorium decay chain is generally assumed to be in secular equilibrium due to low solubility of ^{232}Th and the short-lived nature of its unstable daughter isotopes (Fig. 1.6.12), disequilibrium in the uranium decay chain occurs more frequently – as ^{238}U produces e.g. water soluble daughter isotopes ^{226}Ra and ^{222}Rn (Fig. 1.6.12) – and can introduce substantial changes (up to ~30%) to the OSL age estimate (Olley et al., 1996; 1997). Therefore, in depositional environments where water naturally percolates through the sediment body (i.e. fluvial deposits or caves), disequilibrium is more likely to occur and should, consequently, be thoroughly assessed.

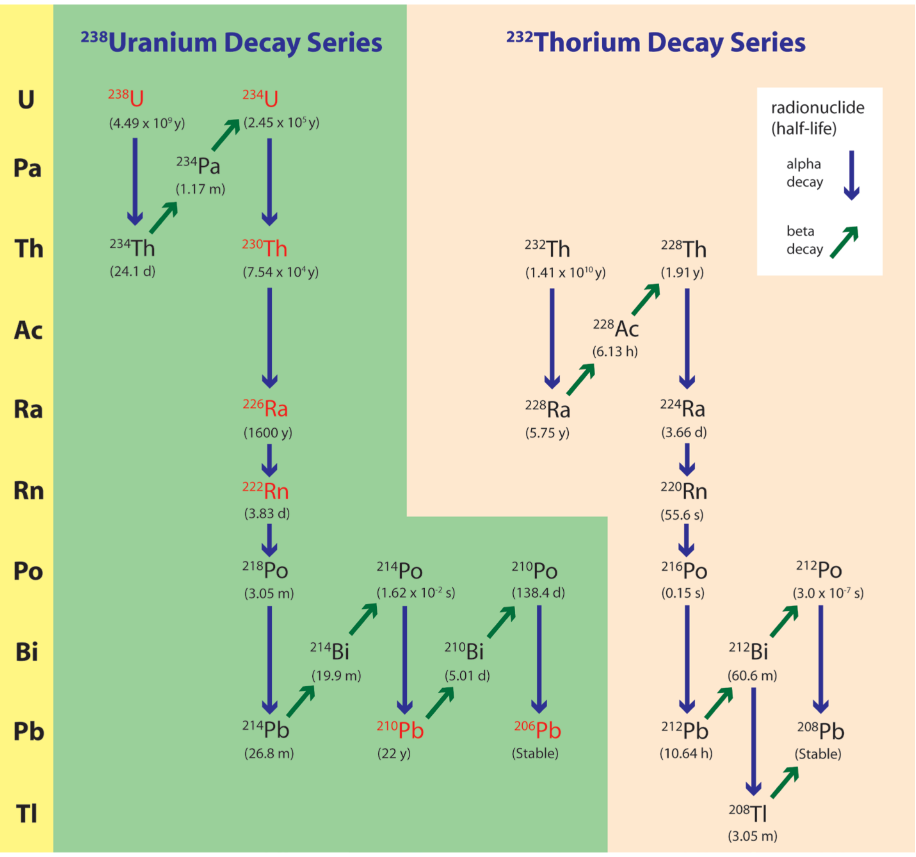

Fig. 1.6.12 Summary of the ^{238}U and ^{232}Th decay chains, including half-lives of their daughter isotopes; data from Lorenz (1983). Daughter isotopes of the ^{238}U decay chain with long half-lives for which disequilibrium might be an issue in sediments are highlighted in red.

β and γ dose rates for sediment samples can be determined by various techniques. In this thesis, dose rates were measured using primarily beta counting and in situ gamma spectrometry. Beta counting was performed on a Risø low-level multicounter system (GM-25-5) which allows β dose rate determination on small sample sizes (<10 g) (Bøtter-Jensen and Mejdahl, 1985; 1988). The system measures the total counts of β emission derived from radioactive sources in the sediment for a defined time span (usually 24h), it, however, does not give information on the concentration of the specific elements the radiation originates from. In situ gamma spectrometry (Fig. 1.6.13) allows the measurement of the natural γ radiation in a 30 cm radius sphere surrounding each OSL sampling hole in the field with a crystal detector (NaI or LaBr$_3$). This technique is particularly useful for samples from sedimentary contexts which are likely to be affected by high spatial heterogeneity in the γ radiation field, such as archaeological sites.

To allow assessment of the reliability of the calculated β and γ dose rates and to detect and quantify potential disequilibria in the Th and U decay chains (Fig. 1.6.12), the specific activities of the radioelements ^{238}U, ^{232}Th, ^{40}K and their daughter products were determined on sediment samples from the direct surrounding of each OSL sample in the laboratory using high-resolution gamma spectrometry (HRGS). The conversion factors for radioactivity of Guérin et al. (2011) were applied to calculate the corresponding β and γ dose rates, which were then compared with those derived from beta counting and in situ gamma spectrometry.

Fig. 1.6.13 In situ gamma spectrometry measurement using a $LaBr_3$ detector at an OSL sample hole at Vanguard Cave, Gibraltar.

The contribution from cosmic radiation (Fig. 1.6.11) to the total dose rate in this thesis was calculated according to Prescott and Hutton (1988, 1994) as a function of i) the site's longitude, geomagnetic latitude and altitude, and ii) the burial depth and density of the overburden for each of the dated samples. Cosmic radiation is classically grouped into a soft and a hard component, the former is absorbed within ~80 centimetres of sediment, whereas the latter is capable of penetrating much further into the ground (Aitken, 1985). While in most studies, cosmic radiation contributes only a low percentage (<10%) to the total dose rate compared to the dose rate derived from the decay of radioactive elements (Guérin, 2015), it can be 25% or more in quartz-rich sediments (e.g. Fitzsimmons et al., 2014).

1.7. OUTLINE OF THESIS CHAPTERS

The following four chapters aim to investigate the luminescence characteristics of individual quartz grains of Pleistocene archaeological cave sites in the western Mediterranean and to build reliable chronological frameworks for those sites using single-grain OSL dating to improve our understanding of the timing of hominin occupation phases and their palaeoenvironmental context at local and regional scales:

Chapter 2 (Paper 1) – Rhafas, NE Morocco – linking a long stratified Palaeolithic sequence to records of palaeoenvironmental variability and modern human dispersal across the Maghreb

Much attention has been devoted to the study of modern human origins and dispersal within and out of Africa; more recently, growing interest has been placed on the archaeological record of the Maghreb and especially the Aterian technocomplex which is often associated with personal ornaments interpreted to represent cultural modernity. Building reliable chronologies for archaeological sites containing multiple Palaeolithic stone tool industries is of critical importance to understand both timing and geographical dispersal of the emergence of modern human behaviour in Africa. The cave of Rhafas is one of the few sites in the Maghreb known to contain an exceptional Palaeolithic record spanning the MSA through to the Neolithic.

Chapter 2 focusses on the development of an absolute chronostratigraphy for the site using single-grain OSL dating. Geological and sedimentological investigations are conducted to gain insights into local site formation processes as well as Middle to Late Pleistocene palaeoenvironmental conditions in the area. The results of this multi-proxy approach allows not only to obtain reliable age estimates for large parts of the stratigraphic sequence but also to identify local processes (sediment mixing, carbonate cementation, ground water flux) which substantially affected the sediments well after deposition. This chapter provides valuable support for future OSL studies dealing with highly complicated cave settings and discusses the new ages for Rhafas in the broader chronological context of the Palaeolithic sites in the Maghreb.

Chapter 3 (unpublished study) – Casablanca, Atlantic Morocco – attempt to construct reliable chronologies for Acheulian sites close to the upper limit of quartz OSL dating

In chapter 3 the regional focus is shifted towards Atlantic Morocco where the Casablanca sites provide rich archaeological records for the study of Early and Middle Pleistocene Acheulian assemblages and their palaeoclimatic context. Eight collected OSL samples from two cave sites - which were subject to chronological investigations in the past - are used to test the potential of single-grain quartz for age determination of sediments close to the upper dating limit of this method, with the overall aim to build refined chronostratigraphies for both sites.

The OSL signal characteristics of the samples are investigated using laboratory-based experiments. It is demonstrated that while stored luminescence signals are usually bright and fast component dominated, samples are very close to saturation level and tend to fail standard OSL performance tests. The obtained test results between differently sized aliquots are often conflicting, which hampers the drawing of solid conclusions and creates more questions than initially intended to solve.

Subsequently, single-grain OSL ages are determined and critically discussed. The validity of the age estimates is highly questionable for both sites, as they are neither chronostratigraphically consistent nor in agreement with independent age controls. It is argued that while standard single-grain quartz luminescence appears to be unsuitable for the dating of these archaeological sediments, this does not necessarily applies for all quartz OSL approaches.

Chapter 4 (Paper 2) - investigating single-grain dose recovery characteristics of archaeological sediments from Moroccan and Australian sites

The question whether a specific quartz sample is suitable for OSL dating given predetermined measurement protocol parameters and whether its calculated natural D_e is, consequently, considered reliable, is commonly checked by a dose recovery test. These laboratory based experiments can, however, only mimic the processes of sunlight bleaching and radiation dosing that occur in natural environments and as yet it is unclear which effects artificial bleaching sources or differently sized recovery doses might have on the obtained test results of a sample.

Chapter 4 addresses these issues by systematically examining single-grain quartz OSL dose recovery characteristics of archaeological samples from Morocco (Rhafas and Casablanca) in comparison to those from an Australian site. This study demonstrates that dose recovery test results primarily depend on the size of the administered dose. It is furthermore shown that sample-specific responses to the chosen test parameters can significantly alter experimental results, especially in samples which underwent relatively few numbers of sensitisation cycles. As this is the case for considerable numbers of sedimentary sequences in archaeological sites – including Rhafas and Casablanca – special caution is advised for conducting dose recovery experiments on such sediments in general and more specifically for the interpretation of the obtained results.

Chapter 5 (Paper 3) – Vanguard Cave, Gibraltar – testing two OSL approaches on a high resolution sedimentary cave sequence in the context of Neanderthal occupation and Mediterranean sea-level fluctuations

Substantial amount of work on the Palaeolithic of southern Iberia has focused on the timing of Neanderthal persistence in the area as well as their potential interaction and eventual replacement by AMH. Less attention has been given to reconstruct past ecological and climatic conditions during times of Neanderthal dominance that pre-date the arrival modern human populations in the region.

In chapter 5, a chronostratigraphy for the upper part of the >17 m sedimentary sequence of Vanguard Cave is developed, a site located on top of a MIS 5 marine terrace at the present-day shoreline of Gibraltar. Sediment accumulation rates are high at Vanguard Cave, which allows a critical testing of the soundness of OSL ages derived from differently sized aliquots by means of the high resolution stratigraphical cave sequence. A dual chronology – comprising quartz single-grain and multiple-grain ages – is thus created demonstrating a high level of consistency between the two OSL dating approaches and, consequently, a great suitability of the method for age determination of the Vanguard Cave sediments. It is argued in this chapter that the stratigraphy of the cave deposits generally supports a scenario of relatively stable palaeoenvironmental conditions between MIS 5 and MIS 3 in the region. The site, thus, offers great potential for detailed studies of human-environmental interactions in southern Iberia at that time building on a robust chronostratigraphical framework.

Chapter 6 - Conclusion

The last chapter provides a synthesis of the accomplishments and conclusions of this thesis and integrates the research questions posed in the introduction.

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